

# PAPERmaking!



*The e-magazine for the Fibrous Forest Products Sector*



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***The Paper Industry Technical Association***

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The Paper Industry Technical Association (PITA) is an independent organisation which operates for the general benefit of its members – both individual and corporate – dedicated to promoting and improving the technical and scientific knowledge of those working in the UK pulp and paper industry. Formed in 1960, it serves the Industry, both manufacturers and suppliers, by providing a forum for members to meet and network; it organises visits, conferences and training seminars that cover all aspects of papermaking science. It also publishes the prestigious journal *Paper Technology International* and the *PITA Annual Review*, both sent free to members, and a range of other technical publications which include conference proceedings and the acclaimed *Essential Guide to Aqueous Coating*.



## Influence of fiber fractionation on kraft paper properties of European black pine and European aspen

Sezgin Koray GÜLSOY and Ayben KILIÇ PEKGÖZLÜ.

In this study, the kraft pulps of European black pine (*Pinus nigra Arn.*) and European aspen (*Populus tremula L.*) were fractionated according to fiber length in a Bauer McNett classifier and effects of fiber fractionation on paper properties were investigated. Bauer McNett screens used for European black pine and European aspen were 16, 30, 50, and 100 mesh and 30, 50, 100, and 200 mesh, respectively. The handsheet surface of each fraction was observed by field emission scanning electron microscopy (FE-SEM). The results showed that handsheet properties were statistically significantly affected by fiber fractionation. The effect of fiber fractionation on tensile and burst indices of handsheets depended on the wood species. However, tear index, apparent density, and surface roughness of handsheets showed similar trends in the two species.

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## Influence of fiber fractionation on kraft paper properties of European black pine and European aspen

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**Abstract:** In this study, the kraft pulps of European black pine (*Pinus nigra* Arn.) and European aspen (*Populus tremula* L.) were fractionated according to fiber length in a Bauer McNett classifier and effects of fiber fractionation on paper properties were investigated. Bauer McNett screens used for European black pine and European aspen were 16, 30, 50, and 100 mesh and 30, 50, 100, and 200 mesh, respectively. The handsheet surface of each fraction was observed by field emission scanning electron microscopy (FE-SEM). The results showed that handsheet properties were statistically significantly affected by fiber fractionation. The effect of fiber fractionation on tensile and burst indices of handsheets depended on the wood species. However, tear index, apparent density, and surface roughness of handsheets showed similar trends in the two species.

**Key words:** Bauer McNett, European aspen, European black pine, fiber fractionation, paper properties

### 1. Introduction

Fiber dimensions have a remarkable influence on the papermaking potential of pulp. The paper properties (strength, surface roughness, porosity, density, etc.) are significantly affected by fiber length, fiber width, cell wall thickness, fiber flexibility, and fiber collapsibility (Pulkkinen et al., 2006). Hardwood fibers have been generally used to achieve good surface properties, while softwood fibers are used for high strength. Therefore, fiber sources used in pulp mill have been appropriately selected according to the quality requirements of the final product.

Fiber fractionation means separation of mixed fibers into two or more parts based on properties such as length, flexibility, and coarseness (Gooding and Olson, 2001; Sood et al., 2005). In mill scale, pulp can be fractionated in hydrocyclones, or in pressure screens using slotted or holed screen plates/baskets (Asikainen et al., 2010). However, Bauer McNett or Clark fiber classifiers are used in the laboratory. The traditional approach in fiber preparation is to use the fibers collectively without fractionating them. Although this approach facilitates the process design, it ignores the opportunity to use the natural advantages of the individual fiber fractions (Gooding and Olson, 2001). On the other hand, fractionation of pulp furnish offers the potential to produce customer-valued products from fiber sources. Thus, papermakers can produce paper with optimum properties for specific applications by controlling

process variables such as the refining conditions, use of additives, and dewatering conditions at the wet end (Sood et al., 2005; Azizi Mossello et al., 2010b).

Abubakr et al. (1995) in recycled fiber, Demuner (1999) in bleached eucalyptus kraft pulp, Reyier (2008) in Norway spruce (*Picea abies*), and Hafrén et al. (2014) in mixed softwood (lodgepole pine, Sitka spruce, Western balsam fir) studied the effects of fiber fractionation on paper properties. Huang et al. (2012) investigated the fiber morphology of each fraction after fiber fractionation of Jack pine (*Pinus banksiana*) thermomechanical pulp. However, there are no published data related to effects of fiber fractionation on paper properties of European black pine (*Pinus nigra* Arn.) and European aspen (*Populus tremula* L.). In this scope, the objective of this study was to determine the effects of fiber fractionation on handsheet properties of European black pine and European aspen.

### 2. Materials and methods

The wood samples of European black pine and European aspen were obtained from Bartın Province in Turkey. They were debarked and chipped into approximately 3.0–1.5–0.5 cm in size. Chips were air-dried and stored with less than 10% moisture content until used.

Table 1 shows the kraft pulping conditions of European black pine and European aspen. Kraft pulping was done in an electrically heated laboratory cylindrical type rotary

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digester of 15 L. Chips (750 g, oven-dried basis) for each cooking experiment were cooked in the digester. After cooking, pulps were washed with tap water to remove residual liquor. After washing, pulps were disintegrated, washed with tap water, and screened on a slot screen of 0.15 mm (TAPPI T 275).

The Bauer McNett classifier (model with 4 classifier chambers) was used for fiber fractionation of kraft pulps according to TAPPI T 233 cm-06. In fiber fractionation of European black pine pulp, R16 (1.190 mm, retained fibers of a 16-mesh screen), P16/R30 (0.595 mm, i.e. passed 16 mesh, retained 30 mesh), P30/R50 (0.297 mm), and P50/R100 (0.149 mm) classifier screens were used. In fiber fractionation of European aspen pulp, R30, P30/R50, P50/R100, and P100/R200 classifier screens were used. Fiber morphology of each fiber fraction was determined with a light microscope. Fiber dimensions of each fractions were measured (n: 100). The aspect ratio (fiber length/fiber width) and flexibility ratio [(lumen width/fiber width)  $\times$  100] were calculated using the measured fiber dimensions.

Handsheets of 75 g/m<sup>2</sup> from each fiber fraction, made with a Rapid-Kothen Sheet Former (ISO 5269-2), were conditioned (TAPPI T 402). Tensile index, tear index,

burst index, and apparent density of the handsheets were measured according to the T494, T414, T403, and T220 TAPPI standards, respectively. Also, roughness of the handsheets was determined according to the ISO 8971-2 standard method.

The handsheets of each fiber fraction were coated with gold (80%) and palladium (20%) using a sputter coater (Quorum Q150 T) and were observed by field emission scanning electron microscopy (FE-SEM) (Tescan MAIA3 XMU) operating at 10 kV. The coating thickness was approximately 10 nm.

The data of handsheet properties for each fiber fraction were subjected to analysis of variance (ANOVA) and Duncan test at 0.05 probability level. Different lowercase letters used in figures denotes that the difference in the average values of properties among the compared groups was statistically significant.

### 3. Results and discussion

The results of Bauer McNett and fiber morphology for European black pine and European aspen are shown in Table 2. It can be seen that the fiber length and fiber width of both species decreased with increasing screen mesh.

**Table 1.** Kraft pulping conditions of European black pine and European aspen.

Conditions	European black pine	European aspen
Active alkali (%)	20	16
Sulfidity (%)	25	20
Temperature (°C)	170	
Time to max. temperature (min)	90	
Time at max. temperature (min)	60	
Total cooking time (min)	150	
Liquor/chip ratio	4/1	

**Table 2.** The results of fiber fractionation of European black pine and European aspen.

Wood species	Fiber fractions	Fiber ratio (%)	Fiber length (mm)	Fiber width (μm)	Double wall thickness (μm)	Lumen width (μm)	Aspect ratio	Flexibility ratio
European black pine	R16	65.8	3.32 $\pm$ 0.02	40.40 $\pm$ 0.1	21.20 $\pm$ 0.3	19.20 $\pm$ 0.2	79.70	47.52
	R30	17.2	2.75 $\pm$ 0.04	39.00 $\pm$ 0.1	18.25 $\pm$ 0.2	20.75 $\pm$ 0.3	72.56	53.21
	R50	7.6	2.13 $\pm$ 0.04	37.10 $\pm$ 0.5	20.35 $\pm$ 0.1	16.75 $\pm$ 0.1	62.26	44.15
	R100	3.9	1.33 $\pm$ 0.02	35.10 $\pm$ 0.2	19.25 $\pm$ 0.3	15.85 $\pm$ 0.2	38.46	44.16
	R200 + fines	5.5	-	-	-	-	-	-
European aspen	R30	38.1	1.27 $\pm$ 0.01	25.0 $\pm$ 0.1	11.35 $\pm$ 0.1	13.65 $\pm$ 0.2	50.40	54.60
	R50	30.6	1.09 $\pm$ 0.01	24.5 $\pm$ 0.1	11.50 $\pm$ 0.1	13.00 $\pm$ 0.1	45.31	53.06
	R100	25.9	0.89 $\pm$ 0.02	23.7 $\pm$ 0.1	11.80 $\pm$ 0.2	11.90 $\pm$ 0.1	38.40	50.21
	R200	2.1	0.60 $\pm$ 0.01	21.4 $\pm$ 0.1	12.20 $\pm$ 0.1	9.20 $\pm$ 0.2	32.24	42.99
	Fines	3.3	-	-	-	-	-	-

Also, increased screen mesh resulted in lower fiber aspect ratio. The weight percentage of long fiber fractions was 83% and 68.7% for European black pine and European aspen, respectively.

Fiber fraction had a statistically significant effect on tensile index ( $P < 0.05$ ). Also, effect of fiber fraction depended on the wood species (Figure 1). Tensile indexes of R16, R30, R50, and R100 fractions in European black pine kraft pulp were determined as 39.71 Nm/g, 45.30 Nm/g, 43.96 Nm/g, and 34.86 Nm/g, respectively ( $P < 0.05$ ). These results can be explained by long and stiff fibers of lower screen numbered fractions, which have poor bonding characteristics (Huang et al., 2012). Tensile index depends on bonding ability of fibers (Rydholm, 1967; Levlin, 1999; Dutt et al., 2009; Jahan and Rawshan, 2009). Flexible fibers produce large contact areas for fiber-

to-fiber bonding. In European aspen samples, tensile indexes of R30, R50, R100, and R200 fractions were found as 33.22 Nm/g, 38.47 Nm/g, 42.20 Nm/g, and 46.85 Nm/g, respectively. This result can be attributed to increasing vessel element numbers in high screen mesh fractions. Thin-walled vessel elements collapse during papermaking, and their wide surface increases the interfiber bonding. On the other hand, it can be ascribed to the high apparent density of handsheets of high screen mesh numbered fractions. Higher density indicates better interfiber bonding in the sheet. A positive correlation between screen mesh number and tensile index has also been reported in previous studies (Reyier, 2008; Hafrén et al., 2014).

The relationships between the fiber fraction and tear index of handsheets are presented in Figure 2. As can be seen in Figure 2, the tear index of handsheets

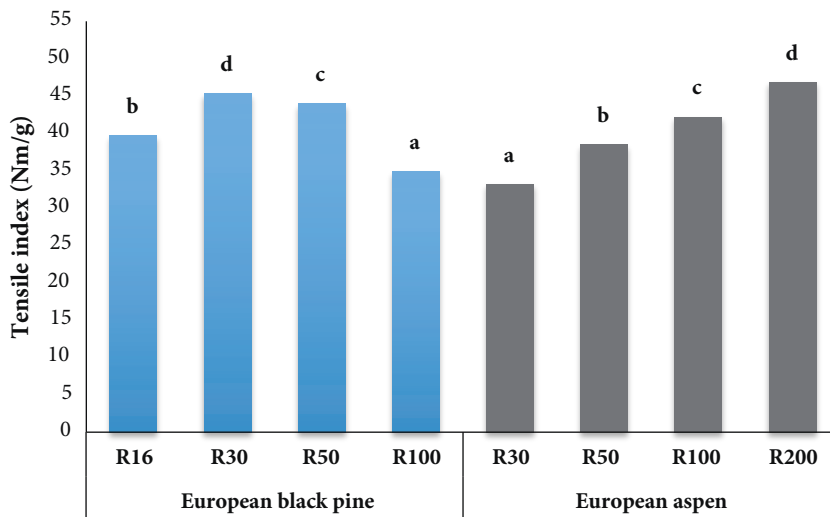


Figure 1. Effect of fiber fractionation on the tensile index of handsheets.

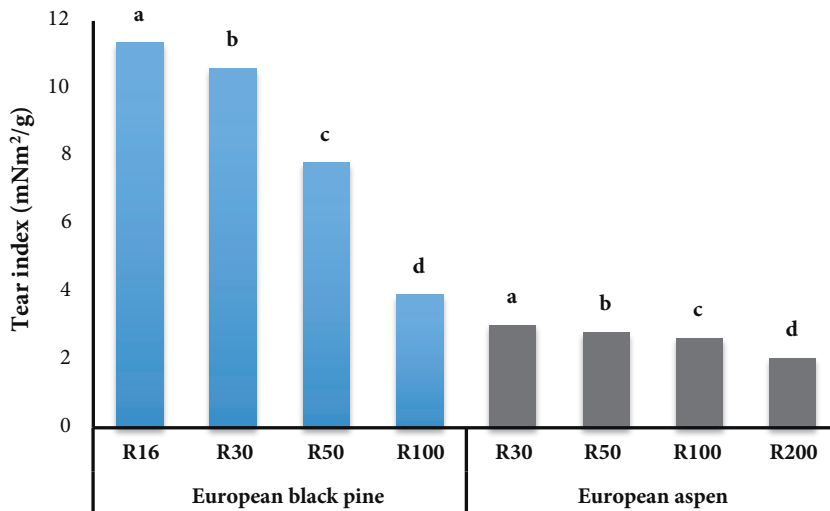


Figure 2. Effect of fiber fractionation on the tear index of handsheets.

of both species decreased significantly ( $P < 0.05$ ) with increasing screen number. The highest tear index values were determined in the R16 fraction at  $11.35 \text{ mNm}^2/\text{g}$  for European black pine and in the R30 fraction at  $3.03 \text{ mNm}^2/\text{g}$  for European aspen. Increase in tear index with decreasing screen mesh numbers could be attributed to a positive correlation between fiber length and tear index (Casey, 1961; Horn, 1978; Seth and Page, 1988; Mohlin, 1989; Horn and Setterholm, 1990; Seth, 1990; Scott et al., 1995; Retulainen, 1996; Levlin, 1999; Shin and Stromberg, 2005; Azizi Mossello et al., 2010a), and also higher aspect ratio of longer fiber (Rydholm, 1965; Shakhes et al., 2011) (Table 2). In addition, the increase in fiber flexibility (a higher sheet density and better interfiber bonding) causes a higher tear index (Bronkhorst and Bennett, 2002). On the other hand, the decrease in tear index with increasing screen mesh numbers could be ascribed to increasing vessel elements numbers with decreasing screen mesh numbers. The vessel elements are generally short and thin-walled, with pitting and open ends (Li et al., 2012). The vessel element-rich fractions cause a decrease in the tear index compared to that of vessel element-poor fractions ([http://www.eucalyptus.com.br/capitulos/ENG04\\_vessels.pdf](http://www.eucalyptus.com.br/capitulos/ENG04_vessels.pdf)). Abubakr et al. (1995) noted that tear index of long fiber fractions in recycled pulp fractionation was higher than that of short fiber fractions.

The relationships between the fiber fractions and burst index of handsheets are presented in Figure 3. Burst index of European black pine handsheets decreased with increasing screen mesh ( $P < 0.05$ ), while burst index of European aspen handsheets increased with increasing screen mesh ( $P < 0.05$ ). The lowest and highest burst index values of European black pine samples were determined in R100 and R30 fractions as  $1.43 \text{ kPa m}^2/\text{g}$  and  $1.99 \text{ kPa m}^2/\text{g}$ , respectively. This result can be explained by fiber

flexibility differences between R100 and R30 fractions (Table 2). Also, it can be attributed to decreasing fiber length with increasing screen mesh (Table 2). The lowest and highest burst index values of European aspen samples were found in the R30 and R200 fractions at  $1.12 \text{ kPa m}^2/\text{g}$  and  $1.90 \text{ kPa m}^2/\text{g}$ , respectively. The high burst index with rich vessel element fractions can be ascribed to improved fiber bonding due to collapsed vessel elements during papermaking. In recycled pulp fractionation, higher burst index of long fiber fractions than short fiber fractions was reported by Abubakr et al. (1995).

As can be seen in Figure 4, apparent density of handsheets in both species was positively correlated with increasing screen mesh ( $P < 0.05$ ) (Figure 4). These results can be explained by short and narrow fibers of higher screen numbered fractions, which give a compact structured paper due to more fibers per area. The lowest apparent density values were determined in the R16 fraction at  $470 \text{ kg/m}^3$  for European black pine and in the R30 fraction at  $580 \text{ kg/m}^3$  for the European aspen. A positive correlation between apparent density and mesh screen was also reported by Reyier (2008). It is known that the relationship between bulk and apparent density is negatively correlated. Demuner (1999) noted that the fine fractions produced sheets with lower bulk than the coarse fractions.

The results indicated that roughness of handsheets increased with increasing screen mesh ( $P < 0.05$ ) (Figure 5). Roughnesses of R16, R30, R50, and R100 fractions in European black pine kraft pulp were determined as 1566 mL/min, 1228 mL/min, 1053 mL/min, and 826 mL/min, respectively. In European aspen samples, roughnesses of R30, R50, R100, and R200 fractions were found as 1275 mL/min, 891 mL/min, 654 mL/min, and 536 mL/min, respectively. These findings can be explained by shorter

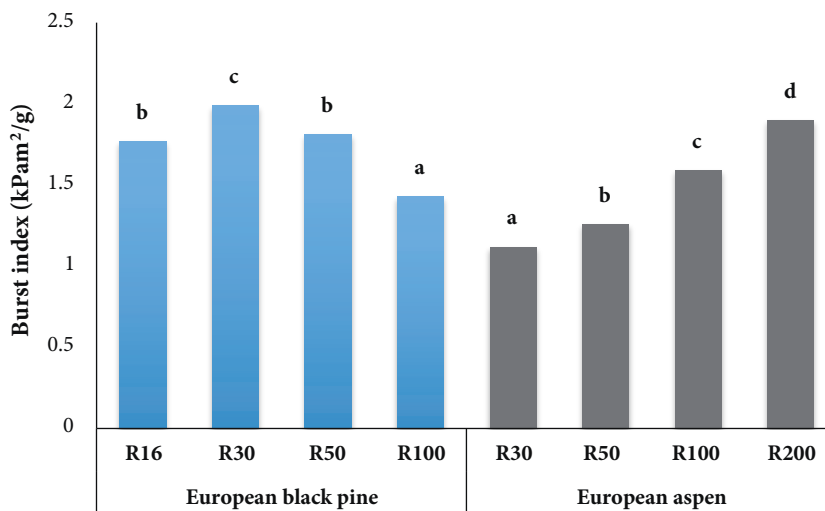


Figure 3. Effect of fiber fractionation on the burst index of handsheets.

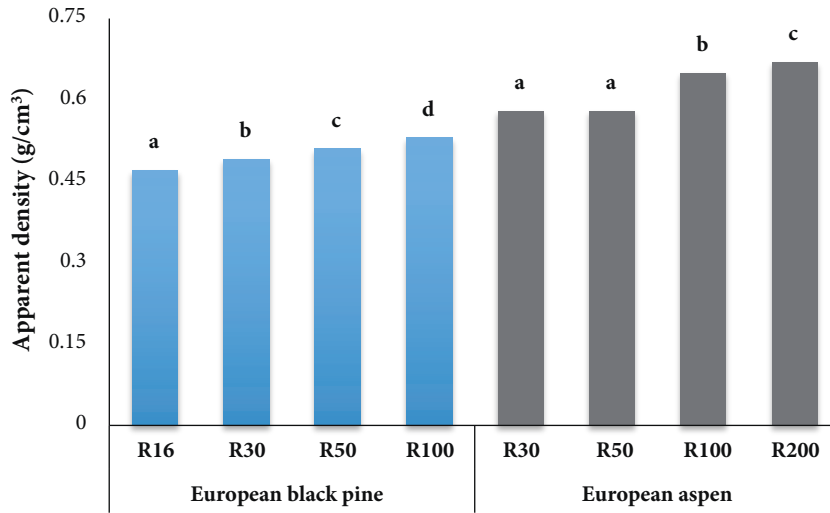


Figure 4. Effect of fiber fractionation on the apparent density of handsheets.

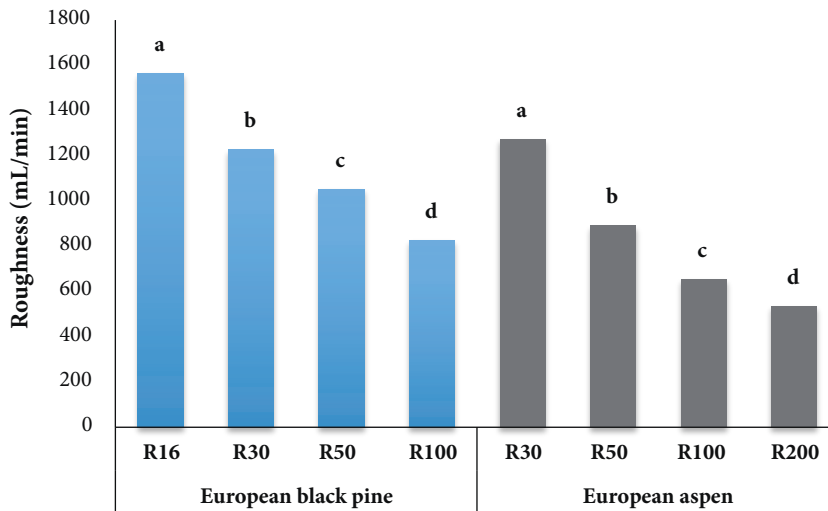


Figure 5. Effect of fiber fractionation on the roughness of handsheets.

and finer fibers of high screen mesh fractions (Table 2; Figures 6 and 7). This result can also be attributed to the action of vessel elements in fractions of European aspen samples that are rich in vessel elements (Malik et al., 2004). Demuner (1999) reported that the smoothness of fine fractions was higher than that of coarse fractions. FE-SEM handsheet micrographs of each fractionation of European black pine and European aspen are shown in Figures 6 and 7, respectively.

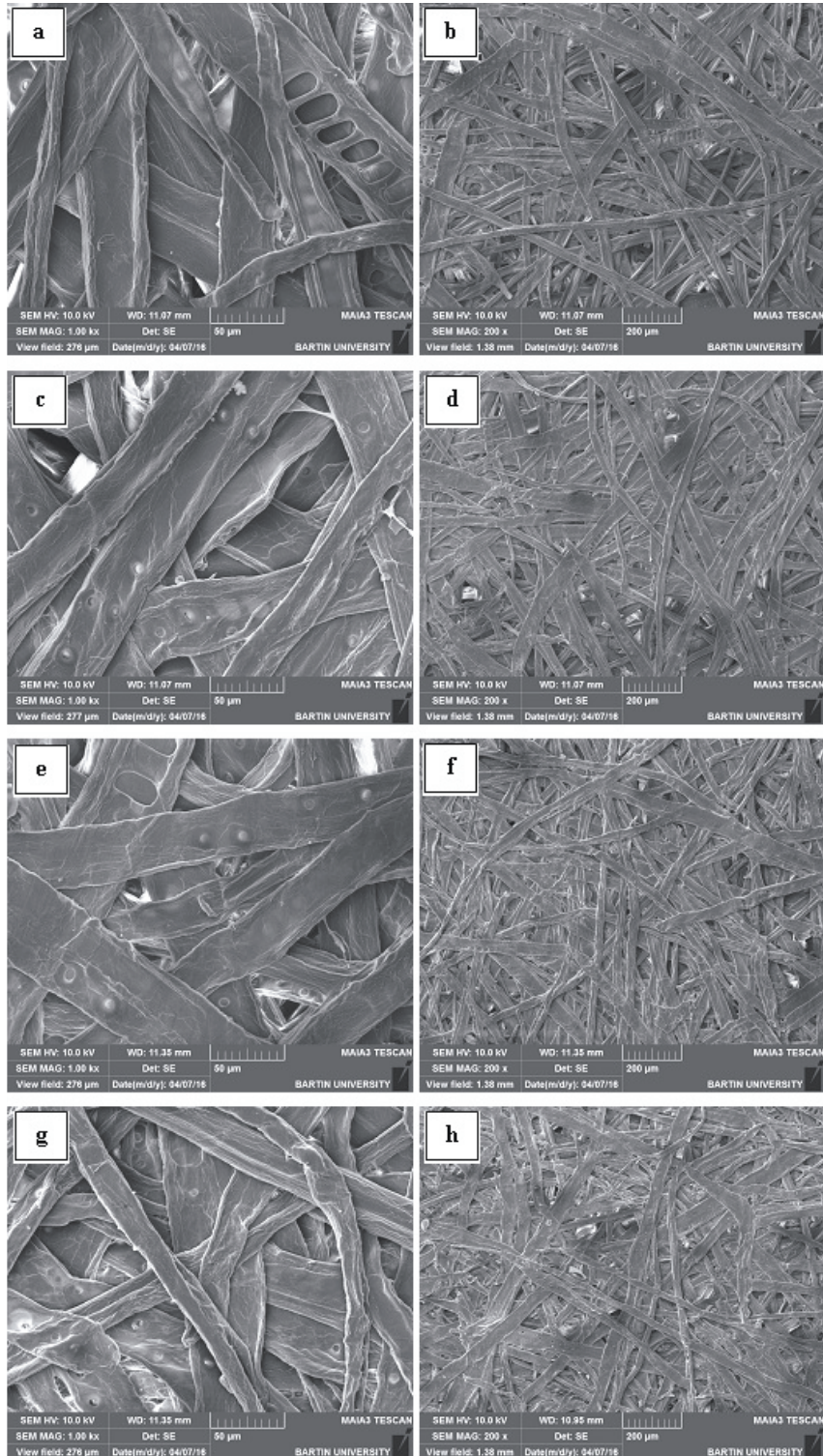
In conclusion, the results of this study have shown that the handsheet properties were statistically significantly affected by fiber fractionation. In European black pine samples, tensile index, tear index, burst index, and roughness of handsheets decreased with increasing screen mesh number. In European aspen samples, tear index and roughness of handsheets decreased with increasing screen mesh number, while tensile index and burst

index increased. Apparent density of handsheets in both species was positively correlated with increasing screen mesh. Fiber fractionation may not be technically feasible for mill-scale paper production, but papermaking from unfractionated fibers ignores the opportunity to use the natural advantages of the individual fiber fractions. Also, selective refining of fractions results in paper quality improvements. More studies related to effects of fiber fractionation (especially the effect of vessel element-rich and element-poor fractions) on paper properties of other lignocellulosic materials have to be carried out.

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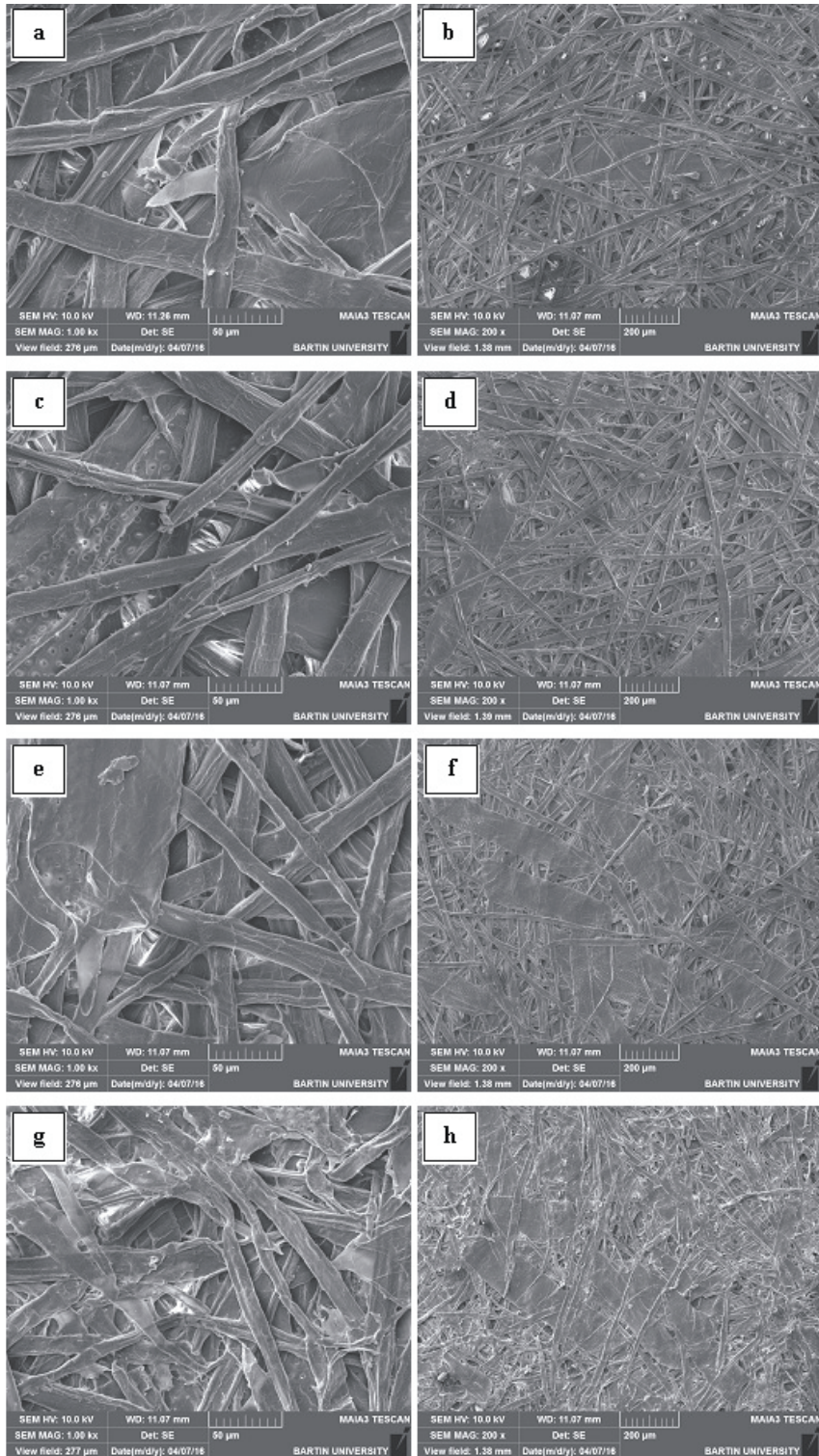
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**Figure 6.** FE-SEM handsheet micrographs of each fractionation of European black pine kraft pulp: a, b) R15; c, d) R30; e, f) R50; g, h) R100.





**Figure 7.** FE-SEM handsheet micrographs of each fractionation of European aspen kraft pulp: a, b) R30; c, d) R50; e, f) R100; g, h) R200.

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## Characterizations of El Minia limestone for manufacturing paper filler and coating

Gaber M.A. WAHAB.

This study introduces a contribution of using the El Minia carbonate filler pigment for paper making. El Minia limestone samples were grind to very fine powder ranging from 2 to 10 $\mu$ m, for utilization in paper filler/coating industry, with using testing techniques; X-ray fluorescence (XRF), X-ray diffraction (XRD), Scanning electron microscopy (SEM). The limestone assessment includes more examinations to confirm the suitability of studied samples for alkaline paper manufacture such as, chemical analysis and physical properties, brightness, refractive index, oil & water absorption, moisture content, water soluble, surface area and soundness tests as per paper industry standards.

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## Full Length Article

Characterizations of El Minia limestone for manufacturing paper filler and coating<sup>☆</sup>

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## ABSTRACT

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## 1. Introduction

Calcium carbonate “ $\text{CaCO}_3$ ”, is one of the most important and useful materials in many industries. It is extremely common and found throughout the world in sedimentary rocks. It comprises more than 4% of the earth's crust. Calcium carbonates natural forms are chalk and limestone, produced by the sedimentation of the shells of small fossilized snails, shellfish, and coral over millions of years. Although all three forms are identical in chemical terms, they differ in many other respects, including purity, whiteness, thickness and homogeneity.

The paper industry uses limestone-based product to manufacture fillers and coating pigments. Calcium carbonate pigment is used for filling and coating, for example in making of printing papers and board.  $\text{CaCO}_3$  valued worldwide for high brightness and light scattering characteristics, as well as it is used as an inexpensive filler to make bright opaque paper. Also it helps to produce papers with high whiteness and gloss and good printing properties.

In 2008, world production of paper and paperboard was 380 million tons according to Food and Agriculture Organization (FAO). Over 90% of paper and paperboard is produced in Asia, Europe and North America. Asia is the biggest producer with 34% of all production and Europe and North America are trailing with 30% and 29% respectively.

There are different grades of paper used for many purposes, for example: coated, uncoated, bond, note book, offset, index, newspaper, computer, copier, gloss, picture and inkjet papers.

The main types of mineral filler for acid papers are talc, hydrous kaolin, calcined kaolin, precipitated silica's /silicates (PSS), and titanium dioxide. For neutral/alkaline papers, talc, hydrous kaolin, calcined kaolin, PSS, titanium dioxide, ground calcium carbonate (GCC), and precipitate calcium carbonate (PCC) are used. The estimated productions of some types of paper and paperboard in 2008 were illustrated in (Fig. 1).

Kaolin, calcium carbonate (GCC and PCC), and talc are the most widely used mineral fillers, with regional variations depending on local resources available (Fig. 2). Filler pigments must have a high degree of whiteness, a high index of refraction, small particle size, low solubility in water, and low specific gravity. It is also important that the filler be chemically inert to avoid reactions with other components in the sheet and in the papermaking system. The filler should contain a minimum of impurities, and the grit content must be low to avoid excessive wear of the wire and other processing equipment such as cutting blades. Furthermore, unless the filler has very unusual properties, it must be inexpensive [1–6].

The annual Egyptian production of paper in Egypt about 150,000 tons while the domestic consumption about 650,000 tons, to compensate the difference between production and consumption, there is a large import quantities cost a lot of hard currencies. The percentage of filler used to produce different types of paper products are indicated in Table 1.

There are many Egyptian companies for paper making, the most important production companies ranked as annual production is

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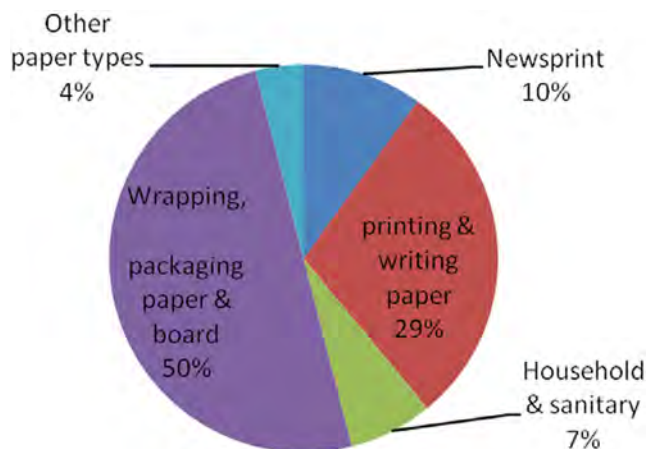


Fig. 1. Global production of paper and paperboard grades in 2008 (Finnish forest Industries 2009).

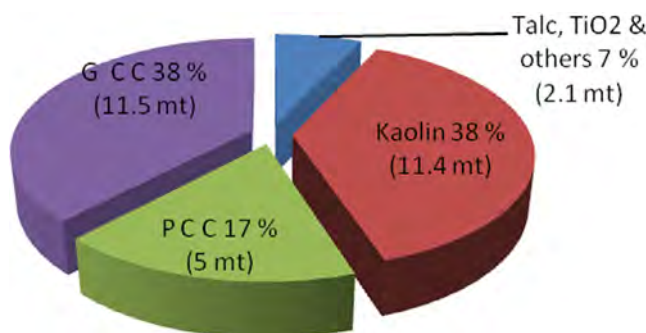


Fig. 2. Breakdown of filler pigment used for paper making at 2002 (Harris 2004).

Table 1  
Paper product and filler contents.

0–15%	Newsprint
20–32%	SC gravure paper
6–10%	LWC base paper
8–15%	Wallpaper
5–10%	Mechanical catalogue
5–20%	Wrapping base paper
10–25%	Wood free printing paper
10–25%	Wood free writing paper
2–10%	Corrugated board
2–10%	wallpaper board

Qena company with annually production about 120,000 tons, Al Ahlya (EMAC) with annual production 60,000 tons, and Rakta company produces 30,000 tons [7].

This article aims to contribute the utilization of El Mina huge quantities of high grade calcium carbonate and its suitability for making of filler/coating of paper and paperboard and stop the loss of hard currency used for importing the same ore.

## 2. Geological setting

El Minia-Maghagha area has a rectangular shape and lies on the Eastern side of the Nile River. It is located between latitude 28° and 28°40'N and longitudes 30° 50' and 31° 30' E.

The exposed carbonate rocks of El Minia-Maghagha areas includes five units of Middle Eocene age. These units are composed mainly of limestone [8,9]. The oldest exposed unit is El Minia Formation which is composed of white, hard, and fossiliferous lime-

stone, this unit is conformably overlain by snow white, soft, fossiliferous limestone of Samalut Formation. Samalut Formation overlain by creamy white, micritic, marly limestone of the Maghagha Formation. This unit is conformably overlain by brownish yellow, sandy, fossiliferous limestone of Qarara Formation. The top most part of Qarara Formation includes nodular, chalky, limestone of Fashn Formation. The studied sample was collected from El Minia and Samalut formations for laboratory examinations (Fig. 3).

## 3. Materials and methods

Thirty samples were collected from different localities along the area in between El Minia city and Beni Khalid to represent the studied limestone, the representative samples were ground to very fine particles for measuring the physical properties, chemical analysis, and X-ray diffraction, and SEM studies.

Carbonate samples were subjected to specific tests and evaluation compare with the international specification and standard to determine their suitability for alkaline paper and paperboard filler/coating manufacture.

The assessment of ground calcium carbonate characterizations were conducted according to the following standards: Specific gravity ASTM D 153, Oil absorption ASTM D 234 and ASTM D 281, Water absorption, Moisture content ASTM D 280, Particle size, Particle shape, ASTM E 70 of Hydrogen ion concentration, ASTM D 2196 for Matter soluble in water: max 1%, Hardness, Appearance & Color of powder, Brightness, purity as CaCO<sub>3</sub>: 95%, Refractive index, SEM, chemical analysis, XRD, and soundness test. Standard properties of ground, precipitated and kaolin ores utilized in paper making are listed in Table 2 as a references for studied sample.

## 4. Results and discussions

The physical and chemical assessment tests were accomplished for El Minia carbonate samples as follows:

Calcium carbonate filler pigment represent a considerable part of paper manufacture, the amount of filler vary from 5% to 30% of the whole finish. Several reasons why fillers are used in paper-making, the main reasons are their low cost compared to fibers “The price of bleached chemical fiber is roughly five to seven times as much as filler prices” and their ability to improve optical properties in the final product. Fillers can also improve surface properties of paper and by that have a positive effect on the printability of the final product [10,11].

Also fillers can improve the surface properties of paper or paperboard as well as have positive effects on the opacity, brightness and colour. Opacity is increased because of filler particles scatter light very well [12].

Fillers also have a smoothening effect on the paper surface, because small filler particles settle in between of fibers they together form a smooth paper surface, which is required in roto-gravure printing. Although fillers are needed for good printing image, excessive amount of filler will compromise the paper surface strength.

The chemical analysis of samples collected from El Minia areas were examined to ensure that the limestone samples used as a pigment are inert, stable and not contain detrimental impurities. The chemical analysis results revealed that the major elements is CaO, accordingly the CaCO<sub>3</sub> content ranging from 99.30 to 99.65%. The physical and chemical analysis was conducted at the Egyptian Petroleum Research Institute and Egyptian Mineral resources Authority “Central laboratories Sector”, and the results obtained are illustrated in Tables 4 and 5.

These testing for calcium carbonate powder were carried out according the following techniques and testing:





a- Outcrop of limestone



b- Quarry of chalky limestone



c- Cutting of limestone building blocks



d- location of limestone quarry's

Fig. 3. Field photos showing outcrop and quarry of pure limestone at El Minia East Nile Valley.

Table 2

Properties standard limits of Kaolin, PCC and GCC fillers used in paper manufacture.

Property	Kaolin	PCC	GCC
Brightness	80–85%	90–97%	> 90–96%
Particle size	2 $\mu\text{m}$	Manufacture fine	Required grinding
Opacity	Excellent	high at high load	Moderate at high load
Loading level	20–30%	Limited to 20%	20–30%
Sheet strength	Good	Moderate	Excellent
Bulking	Moderate	Good	Good
Absorption	Low	High	Low
Chemical reactivity	Inert	Unstable in acid	Unstable in acid
Flexibility	Filler/Coating	Mainly filler	Alkaline - filler/coating
Processing	Extensive	Energy extensive	Grinding/sizing
Availability	Restricted	Satellite plants	Geologically plentiful
Price	Low (N. America)	Based on cost	Low (Europe)

Table 3

Brightness of El Minia calcium carbonate.

Limestone samples	Brightness (z-direction)
Sample 1	91
Sample 2	93.5
Sample 3	90.5
Sample 4	91.5

#### 4.1. Specific gravity

The representative samples of limestone measured according to [13] and the results are ranging from 2.6 to 2.7 g/cm<sup>3</sup> as indicated in Table 4., as per standard the low specific gravity is preferred for paper manufacture.

#### 4.2. Oil absorption

The test were carried out using linseed oil mixed with carbonate powder and the specific paste obtained at the ratio of oil quantity is ranging from 32 to 36 g/100 g (g of oil/g of powder), as per [14,15].

#### 4.3. Moisture content

The moisture content of limestone powder was determined at 105 °C and the results ranging from 0.05 to 0.07% and considered low percentage moisture according to [16].

#### 4.4. Particle size

Calcium carbonate pigments for paper making occurs in the form of a fine powder or lumps by using crushing equipment's was ground to very fine grains. The recommended size of limestone powder pigment shall be in limit of 2–10  $\mu\text{m}$  as shown in (Fig. 4). The vast majority of particles are much smaller in size than 10  $\mu\text{m}$ ; there is some evidence that a mixture of particle sizes is desirable for increased durability, reduced absorption and reduced permeability of the film, also the particle size distribution graph (Fig. 5), showing that the 80% of analyzed sample is less than 10  $\mu\text{m}$  in size.

**Table 4**

Physical properties of limestone filler pigment.

Ore Type	Physical Properties					
	Sp. Sp. gravity g/cm <sup>3</sup>	oil absorp. %	Moisture %	pH	Acid soluble	hardness water soluble
Limestone sample 1	2.70	33	0.05	8.5	99.0	3.0 0.96
Limestone sample 2	2.69	32	0.06	9	99.0	3.0 0.86
Limestone sample 3	2.72	36	0.05	8	98.0	3.0 0.95
Limestone sample 4	2.70	34	0.07	9	98.5	3.0 0.93

**Table 5**

Chemical compositions of limestone at some localities of El Minia.

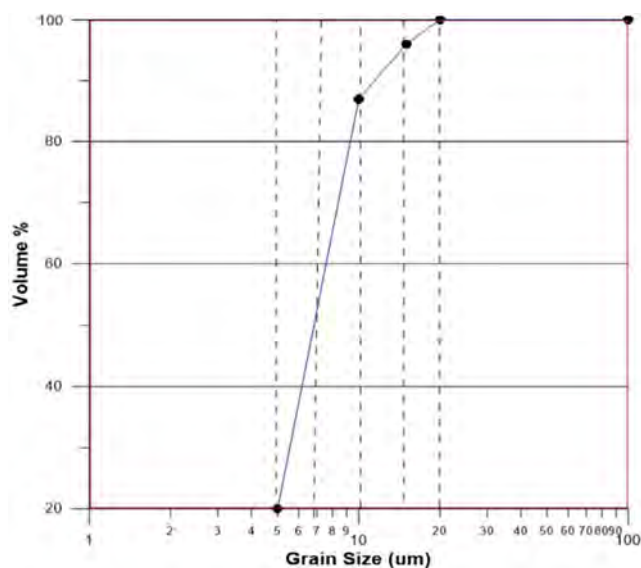
Ore Type	Chemical composition					
	CaO	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MgO	H <sub>2</sub> O	L.O.I
Limestone sample 1	55.61	0.11	0.12	0.07	0.20	43.80
Limestone sample 2	55.70	0.14	0.16	0.11	0.11	43.70
Limestone sample 3	55.56	0.10	0.07	0.13	0.10	43.75
Limestone sample 4	55.67	0.11	0.13	0.11	0.10	43.80



(a) Grinding machine of limestone



(b) Final product of grind limestone

**Fig. 4.** (a) Grinding machine of limestone (b) Final product of grind limestone.**Fig. 5.** Particle Size Distribution.

#### 4.5. Particle shape

Particle shape and size influence on the properties of carbonate powder pigment such as consistency, oil absorption, hiding power of paper coating and filler, the carbonate examined grain shape is rounded and sub round.

#### 4.6. Hydrogen ion concentration (pH value)

The pH test carried out to collected sample as per [17] to measure the value of alkaline or acidity of carbonate powder and the results shows that pH is 8.5%, it means the pH range is alkaline.

#### 4.7. Matter soluble in water

The carbonate powder pigment shall be insoluble in water, except traces of soluble salt. The test result indicates that the amount of soluble matter is 0.4%, meanwhile the standard limit as per [18], shall be not exceed than 1% as shown in Table 4.

#### 4.8. Moh's hardness

The hardness of carbonate powder utilized in paper filler and coating is most important for the paper product and production equipment (wear on wire, doctor and slitter wearing), the limestone studied samples hardness is 3 as per Moh's scale (1: talc to 10: diamond).

#### 4.9. Appearance & Color of powder

The color of ore powder is useful in identify the pigment into white or colored pigment, however the history of mineral formation. The visual inspection of limestone sample is milky white to white due to high purity of calcium carbonate content.



Fig. 6. Brightness of El Minia carbonates.

#### 4.10. Brightness of El Minia calcium carbonate

In paper industry the high dry brightness is preferred to produce the high quality paper. The limestone were crushed to very fine size and tested using Dr. Lange equipment (Fig. 6), and the results achieved the requirements of paper making brightness value ranging from 91 to 93.5% as shown in Table 3.

#### 4.11. Purity as $\text{CaCO}_3$ : 95%

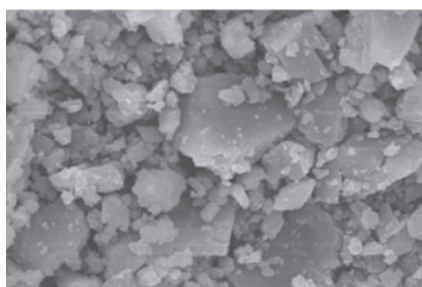
The  $\text{CaCO}_3$  content in studied samples are ranging from 99.30 to 99.65%, which called high purity limestone as per [19].

#### 4.12. Refractive index

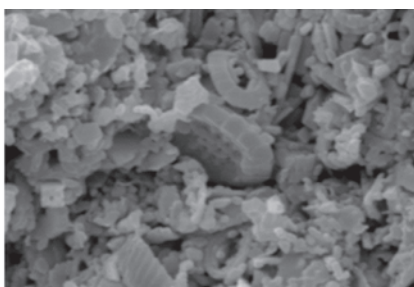
Refractive Index is the difference between the speed of light in a vacuum and the speed of light in the gemstone. As light passes through a gemstone, it slows down because a gemstone is denser than air. The angle of refraction in the gemstone determines its RI [20]. The RI is easily measured using a refractometer. Limestone fillers are mainly composed of strongly birefringent calcite mineral with refractive indices ranging from 1.49 to 1.65.

#### 4.13. Specific surface area

Specific surface area is measured by the nitrogen adsorption method (BET: Brunauer, Emmet, Teller). The particle fineness, the particle size distribution, and the particle morphology are, depending on the structure, indirectly reflected in the specific surface area of the filler. Finer, non-structured fillers exhibit a higher specific surface than coarser ones. There is, a direct correlation between the specific surface area of filler and, the internal sizing agent demand. An internal sizing agent is applied to the wet end in order to make the paper more hydrophobic. The specific surface area of regular paper fillers ranges between 2.5 and 14  $\text{m}^2/\text{g}^{-1}$ , while fiber fines show specific surface areas of 6–8  $\text{m}^2/\text{g}^{-1}$ . The surface area



(a) SEM for El Minia limestone



(b) SEM for el Minia Chalk

Fig. 7. (a) SEM for El Minia limestone (b) SEM for el Minia Chalk.

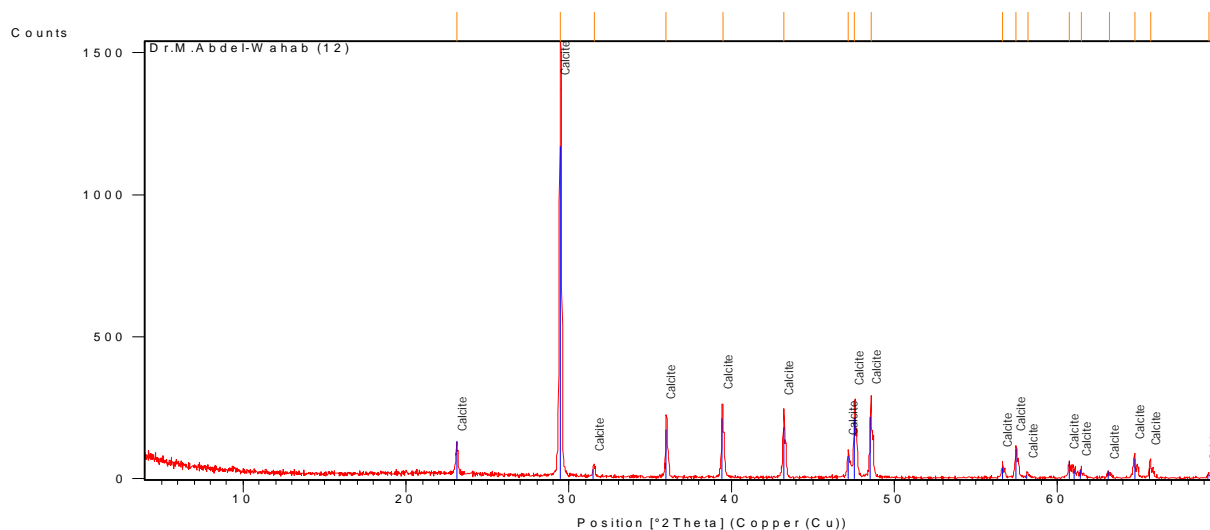


Fig. 8. XRD of Limestone at Samalout area.

for calcium carbonate powder ranging from 2 to 12 m<sup>2</sup>/g<sup>-1</sup> as per [21].

#### 4.14. Scanning electron microscope (SEM)

The structure of fillers can be observed and characterized best by scanning electron microscopy (SEM). The particle morphology has an influence on light scattering via the number and size of air microvoids in the sheet. For different morphologies, there is a different optimum for light scattering in terms of particle size. The particle morphology has an impact also on the packing of the filler particles in the flocculates usually formed during the papermaking process.

The crystallite habit of natural ground CaCO<sub>3</sub> filler is rhombohedral Fig. 7. For high brightness demand, GCC fillers based on limestone and marble are preferred by the paper industry. Lower

brightness chalk is used as filler in the production of regular newsprint.

#### 4.15. Chemical analysis

The XRF analysis of collected limestone revealed that the major element is CaO 55.70; accordingly the CaCO<sub>3</sub> is 99.65% as indicated in Table 5. These results reflect that the studied ore possess high purity and suitability for industrial proposes and paper making.

#### 4.16. X-Ray diffraction

Four samples were analyzed by XRD and the data of identified minerals of powder samples were plotted in the following diagrams:

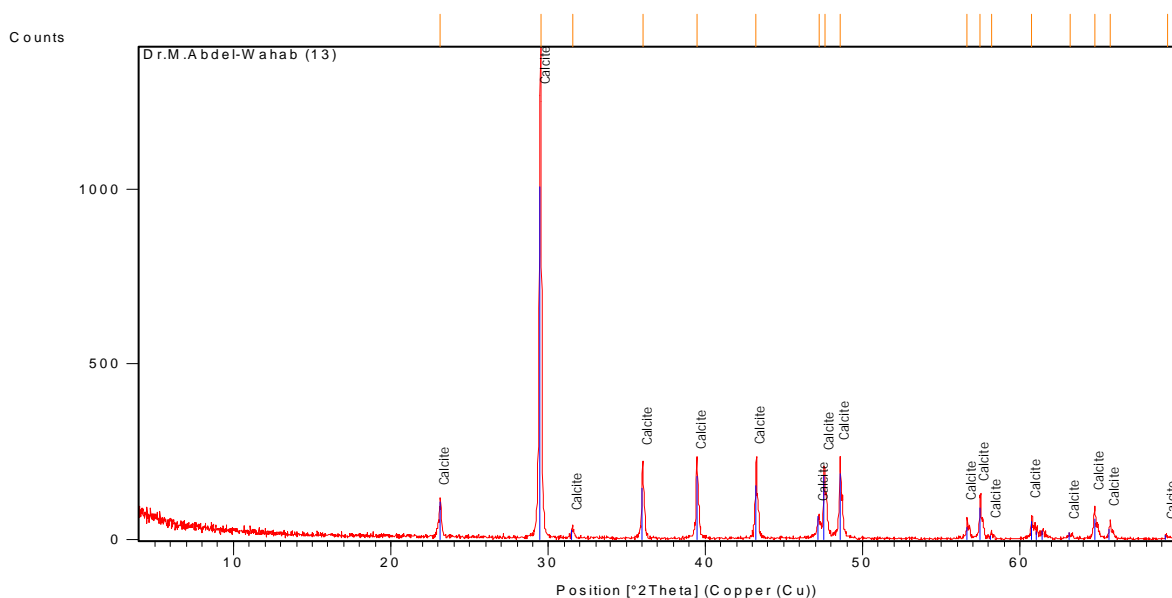


Fig. 9. XRD of Samalout Limestone.

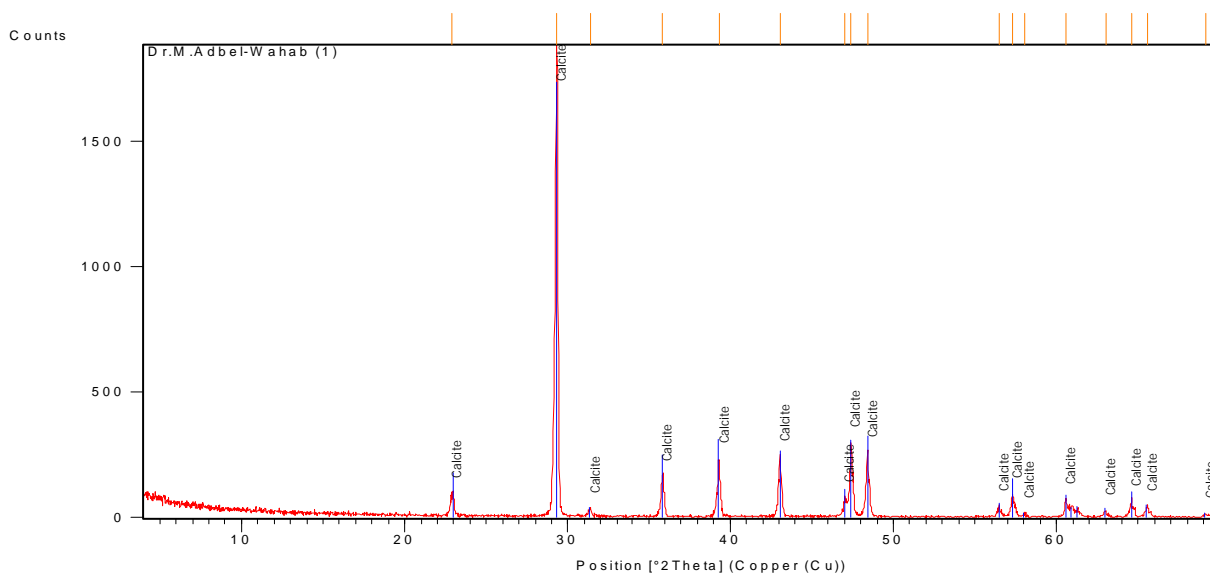


Fig. 10. X-ray diffraction of Beni Khalid carbonate.

The XRD analysis of studied samples were collected from Samalout areas indicates that the calcite mineral is the most predominant minerals of limestone ore with average ratio 99.5% as shown in (Figs. 8 and 9).

The XRD of Beni Khalid limestone (Fig. 10), shows that the samples are composed mainly of calcite, the XRD results complies with chemical composition analysis that the  $\text{CaCO}_3$  is 99.30%.

#### 4.17. Soundness test

Residues, impurities, contamination, can be coming from origin, processing the industrial mineral, transportation, and from other sources (run-ability in paper machine or coating or calendaring causes streaks and breaks, which are expensive), the test was carried out according to [22], and the obtained results are ranging from 10 to 12% loss of impurities, meanwhile the maximum standard is 15%.

### 5. Conclusions

This study indicates some positive conclusions:

- Presence of huge outcrop calcium carbonate reserve in El Minia areas can be mined open cast with low cost.
- El Mina limestone possesses high purity  $\text{CaCO}_3$  “99.65%” content suitable for industrial purposes, further more paper filler/-coating pigment.
- The calcium carbonate brightness reading ranging from 91 to 93.5% and suitable for paper pigment fillers.
- The more investigation and cooperation with paper Manufacture Company for trial testing using local ores are required.

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## In Situ Production and Application of Cellulose Nanofibers to Improve Recycled Paper Production

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The recycled paper and board industry needs to improve the quality of their products to meet customer demands. The refining process and strength additives are commonly used to increase mechanical properties. Interfiber bonding can also be improved using cellulose nanofibers (CNF). A circular economy approach in the industrial implementation of CNF can be addressed through the in situ production of CNF using side cellulose streams of the process as raw material, avoiding transportation costs and reducing industrial wastes. Furthermore, CNF fit for use can be produced for specific industrial applications. This study evaluates the feasibility of using two types of recycled fibers, simulating the broke streams of two paper machines producing newsprint and liner for cartonboard, to produce in situ CNF for direct application on the original pulps, old newsprint (ONP), and old corrugated container (OCC), and to reinforce the final products. The CNF were obtained by 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO)-mediated oxidation and homogenization at 600 bar. Handsheets were prepared with disintegrated recycled pulp and different amounts of CNF using a conventional three-component retention system. Results show that 3 wt.% of CNF produced with 10 mmol of NaClO per gram of dry pulp improve tensile index of ONP ~30%. For OCC, the same treatment and CNF dose increase tensile index above 60%. In both cases, CNF cause a deterioration of drainage, but this effect is effectively counteracted by optimising the retention system.

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The Paper Industry Technical Association (PITA) is an independent organisation which operates for the general benefit of its members – both individual and corporate – dedicated to promoting and improving the technical and scientific knowledge of those working in the UK pulp and paper industry. Formed in 1960, it serves the Industry, both manufacturers and suppliers, by providing a forum for members to meet and network; it organises visits, conferences and training seminars that cover all aspects of papermaking science. It also publishes the prestigious journal *Paper Technology International* and the *PITA Annual Review*, both sent free to members, and a range of other technical publications which include conference proceedings and the acclaimed *Essential Guide to Aqueous Coating*.

## Article

# In Situ Production and Application of Cellulose Nanofibers to Improve Recycled Paper Production

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**Abstract:** The recycled paper and board industry needs to improve the quality of their products to meet customer demands. The refining process and strength additives are commonly used to increase mechanical properties. Interfiber bonding can also be improved using cellulose nanofibers (CNF). A circular economy approach in the industrial implementation of CNF can be addressed through the in situ production of CNF using side cellulose streams of the process as raw material, avoiding transportation costs and reducing industrial wastes. Furthermore, CNF fit for use can be produced for specific industrial applications. This study evaluates the feasibility of using two types of recycled fibers, simulating the broke streams of two paper machines producing newsprint and liner for cartonboard, to produce in situ CNF for direct application on the original pulps, old newsprint (ONP), and old corrugated container (OCC), and to reinforce the final products. The CNF were obtained by 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO)-mediated oxidation and homogenization at 600 bar. Handsheets were prepared with disintegrated recycled pulp and different amounts of CNF using a conventional three-component retention system. Results show that 3 wt.% of CNF produced with 10 mmol of NaClO per gram of dry pulp improve tensile index of ONP ~30%. For OCC, the same treatment and CNF dose increase tensile index above 60%. In both cases, CNF cause a deterioration of drainage, but this effect is effectively counteracted by optimising the retention system.

**Keywords:** nanocellulose; cellulose nanofibers; recycled paper; mechanical properties; drainage; retention; circular economy

## 1. Introduction

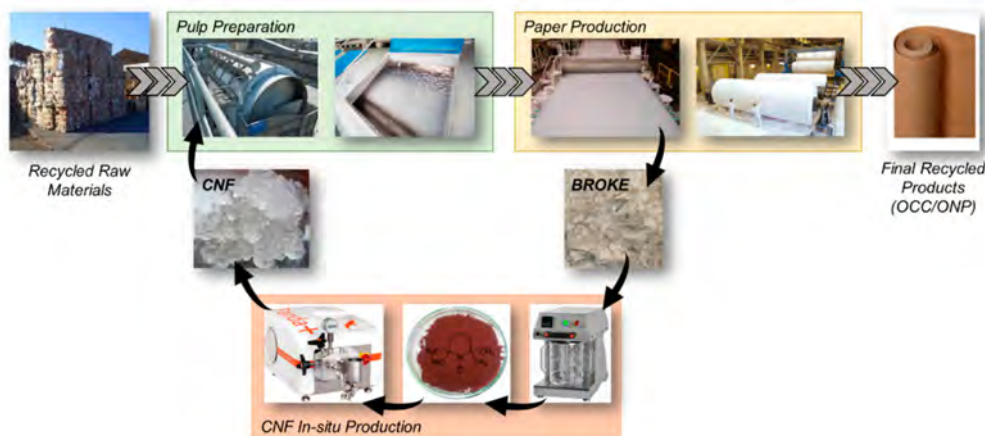
Papermaking is an industrial sector characterized by its commitment to develop sustainable production processes [1,2]. In Europe, 52.4% of the papermaking industry's raw materials come from recovered paper, which corresponds to a paper recycling rate of 72.3% [3]. Nevertheless, the quality levels required in the utilization of secondary fibers are continuously increasing according to the customer demands. Besides, paper consumption has decreased due to the replacement of paper by other supports for the information, causing cost pressures in the paper and board industry. Despite the fact that natural and synthetic strength additives are commonly used in recycled paper, the main source of complaints is still the poor tensile strength. Therefore, other strategies to improve interfiber bonding have been explored, and the use of cellulose nanofibers (CNF) is a promising alternative to increase mechanical properties of recycled products with some additional advantages, such as their renewable nature, biodegradability, high surface area, and high availability.

CNF have gained more attention due to their high strength and stiffness joined to the low weight [4,5]. For these reasons, CNF is promising in multiple sectors such as papermaking [6], composites [7,8], cement [9], packaging [10], electronic devices [11], coatings, biomedicine [12], or automotives [13]. Regarding the papermaking industry, CNF can improve paper quality, and many studies have shown that their addition to the pulp suspension increases the mechanical properties of the recycled paper [6,14–16]. The majority of CNF applied in papermaking as strength additive are produced from virgin pulp [17–19], but also from pulps from valueless agriculture residues [15,20,21]. Recently, nanocellulosic materials from papermaking streams such as solid waste from a dissolving cellulose pulp mill have been studied. Jonoobi et al. (2012) [22] produced and characterized these nanofibers as a potential biobased nanomaterial for different applications, but they did not study their use as reinforcement agent in the papermaking process. On the other hand, Campano et al. (2016) [23,24] isolated cellulose nanocrystals (CNC) from recycled newspaper and evaluated their effect on the recycled paper enhancement achieving increments of up to 30% in the tensile strength index when 3 wt.% of CNC was added into the recycled pulp; long pulping times and a polyacrylamide-based retention system were used. In situ produced bacterial cellulose (BC) in recycled pulps has also achieved increments in both tensile and tear indexes of 12.2% and 14.2%, respectively, when BC was produced in agitation culture [25]. However, to the best of our knowledge, there are no studies of the use of CNF produced in situ from recycled paper, simulating the broke streams of the recycled papermaking process, and, subsequently, evaluating their effect as strength agent in the same industrial process.

Large-scale production of CNF is still very limited and produced from virgin pulp. Therefore, the industrial implementation of CNF in the papermaking industry is still a challenge [26]. The main drawbacks to use CNF in large volume applications, such as papermaking, is their cost due to both the high amount of energy required and transportation, the difficulties in producing uniform nanocellulosic particles and the difficulties associated with both dewatering and pumping [6,27]. Some of those drawbacks can be addressed and eventually avoided through the implementation of a circular approach that will lead to an even more sustainable papermaking process. In order to achieve this, one of the key points is the in situ production of CNF (Figure 1) using process and waste cellulose streams, such as fines-rich streams, coming from the filtering of the screw presses or from the white-waters, dry and wet broke from the paper machine, and rejects from the flotation processes. Some of the advantages associated with this approach are the increase in the yield of the process, the avoidance of drying and/or transportation costs, and the decrease of waste generation. Moreover, the in situ production of CNF would help the papermakers to determine the relation between the minimum CNF quality and the needs required for a certain recycled paper product, allowing the online control of the properties of the CNF and their adjustment to the production needs. In addition, the negative effects related to the dispersion of the CNF in the pulp suspension would be also avoided. Furthermore, CNF could also be sold in the local market as additive for other industries. In this way, the benefit of this industrial symbiosis will allow papermakers to afford the cost of CNF production.

Therefore, the objective of this study was to evaluate the feasibility of using two different types of recycled pulps—Old Newsprint (ONP) and Old Corrugated Container (OCC)—with 14 wt.% and 11 wt.% ash content, respectively, to simulate the broke streams of paper machines, produce in situ CNF, and study its direct application on the recycled pulp suspension to reinforce the final product, recycled newsprint, and recycled cartonboard, respectively. The production of CNF was studied at different TEMPO (2,2,6,6-tetramethyl-1-piperidinyloxy)-mediated oxidation levels (2.5, 5, 10, and 15 mmol of NaClO per gram of pulp) before the homogenization mechanical process. TEMPO-mediated oxidation is the most common pretreatment to facilitate cellulose defibrillation reducing the energy consumption in homogenization. The obtained CNF were characterized, and several doses of CNF (1, 2, and 3 wt.%) were added to the recycled pulp to evaluate their effect in terms of paper strength enhancement, using a three-component retention and drainage system (TRDS) containing cationic polyamine as coagulant (C), cationic polyacrylamide as flocculant (PAM), and hydrated bentonite (B). The mechanical properties measured on handsheets include tensile strength index, tear strength index, and porosity in

the case of addition of CNF from recycled ONP. For CNF from recycled OCC, bursting strength index and short-span compressive strength (SCT) were also measured to evaluate the recycled cartonboard enhancement. Also, the effect of both kinds of CNF on retention and drainage was studied, using different CNF doses and TRDS.

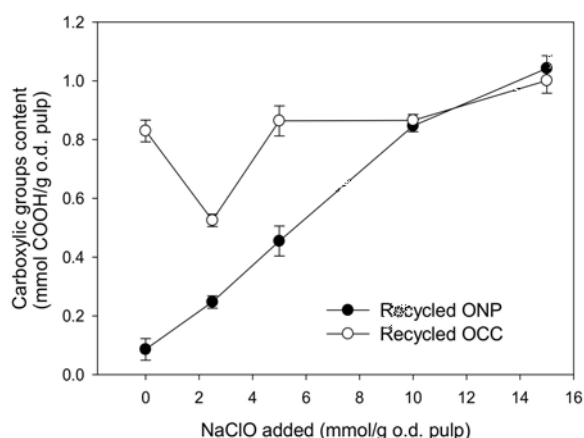


**Figure 1.** In situ production of cellulose nanofibers (CNF) from recycled papermaking streams to be used as additive in the process.

## 2. Results and Discussion

### 2.1. Characterization of CNF from Recycled ONP and Recycled OCC Pulps

The content of carboxylic groups of the recycled ONP was very low—below 0.2 mmol of COOH/g of dry pulp—compared to the content of carboxylic groups of recycled OCC (~0.85 mmol of COOH/g of dry pulp). In the case of recycled ONP, the COOH level increased linearly with increasing oxidation treatment up to a dose of 15 mmol of NaClO/g of dry pulp (Figure 2). In the case of recycled OCC, the first addition of NaClO decreased the content of carboxylic groups but higher additions produced an increase of the values, obtaining similar content of carboxylic groups to recycled ONP. This was probably due to the higher content of lignin and other impurities present in the recycled OCC (Table 1) that consume part of the NaClO [21].

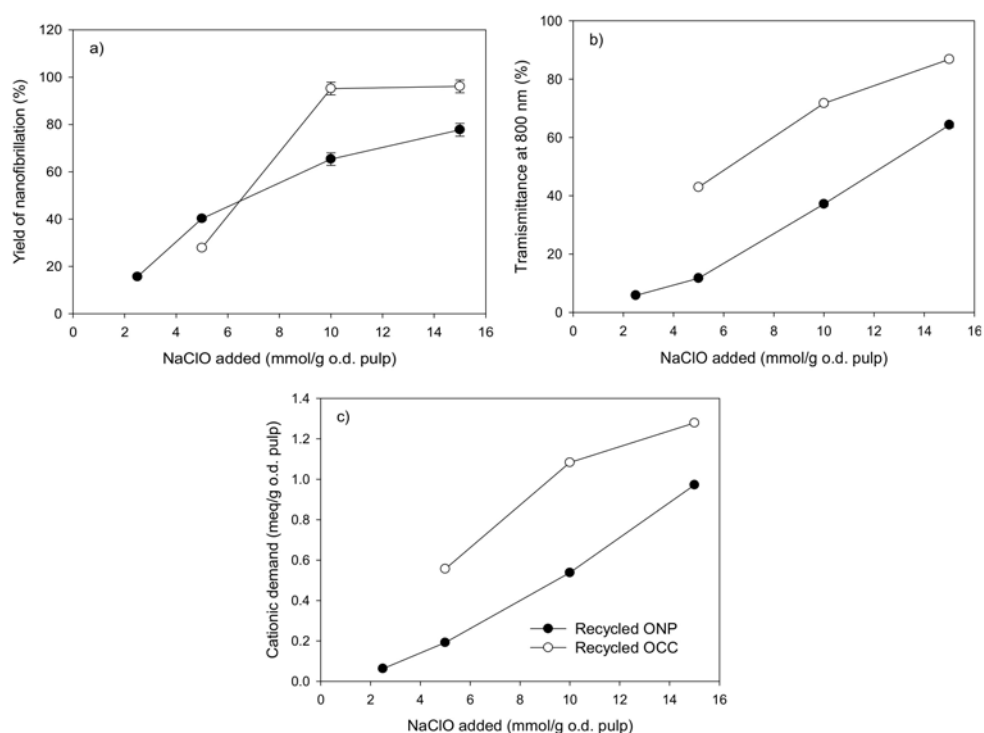


**Figure 2.** Carboxylic groups in recycled old newsprint (ONP) and old corrugated container (OCC) pulps oxidized by NaClO in presence of 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO).

**Table 1.** Morphology of recycled old newspaper.

Item	Units	Recycled ONP	Recycled OCC
<b>Fibers</b>			
Length weighted in length	( $\mu\text{m}$ )	861	1054
Average width	( $\mu\text{m}$ )	21.4	22.2
Coarseness	(mg/m)	0.141	0.159
Microfibrils	(%)	1.72	1.35
Broken ends	(%)	37.2	34.1
Average angle	( $^{\circ}$ )	130.3	133.5
Kinked fibers	(%)	13.70	13.54
Average curl	(%)	5.89	5.64
<b>Pulps</b>			
Kappa index		40	72
Fibers	(number $\times 10^6/\text{g}$ )	15.82	11.80
Aggregates	(number/g)	98,837	92,667
Fines	(number/g)	118,646	92,322

The yield of nanofibrillation increased with the degree of oxidation, reaching values close to 80% and 100% when 15 mmol of NaClO per gram of dry pulp was used to produce CNF from recycled ONP and recycled OCC, respectively (Figure 3a). As expected, catalytic oxidation with TEMPO and NaClO had an important impact on the transmittance and on the cationic demand of the CNF suspensions, obtaining higher values when the degree of oxidation applied to the recycled pulp increased (Figure 3b,c). The production of CNF from recycled OCC pulps with a degree of oxidation of 2.5 mmol of NaClO per gram of pulp was not possible due to the obstruction of the homogenizer, requiring a minimum degree of oxidation (5 mmol of NaClO per gram of pulp) to carry out the mechanical treatment.



**Figure 3.** Characterization of CNF from recycled ONP and recycled OCC pulps oxidized by NaClO in the presence of TEMPO. (a) Yield of nanofibrillation, (b) transmittance at 800 nm, and (c) cationic demand.



Results showed that CNF produced from recycled OCC with 10 mmol of NaClO/g of dry pulp or a higher dose of NaClO are more nanofibrillated than CNF produced from recycled ONP, although the chemical pretreatment and homogenization conditions were the same. This effect could be related to the higher amount of lignin of recycled OCC compared to ONP, as shown in the Kappa index values (Table 1). Delgado-Aguilar et al. (2016) [28] and Ferrer et al. (2012) [29] reported that lignin content is a key factor for the nanofibrillation process, obtaining better results when a certain amount of lignin is presented in the pulp. They reported that a good balance between hemicellulose and lignin content facilitates the mechanical defibrillation of the cellulose fibers due to increased swelling caused by hemicelluloses and the formation of mechanoradicals stabilized by residual lignin [28–30]. Probably, the amount of hemicelluloses in CNF suspensions from recycled ONP, which has a negligible quantity of lignin, was not enough to reach the higher nanofibrillation yields and transmittances values of the CNF of recycled OCC, even though they had the same carboxylic content at the higher oxidation degrees. In addition, the lower amount of aggregates in the recycled OCC pulp (6.2% less) (Table 1) could also help to improve the defibrillation process.

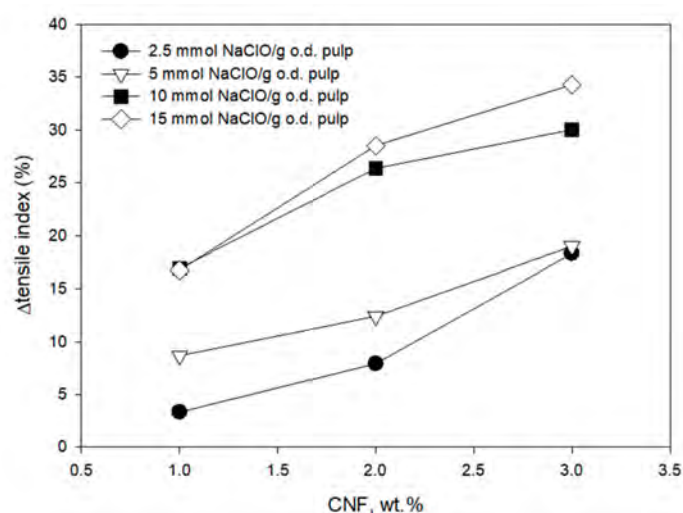
## 2.2. Effect of CNF Content on Recycled Paper and Cartonboard Properties

Once the feasibility of producing CNF from recycled fibers was established, they were applied in mass to evaluate their effectiveness as a reinforcing agent in the recycled pulp (ONP and OCC) using a TRDS for retention of the CNF. The presence of 3 wt.% CNF produced from recycled ONP with low levels of oxidation (2.5 and 5 mmol of NaClO/g of dry pulp) increased the tensile index of the recycled pulp by 18%. CNF with higher degrees of oxidation (10 and 15 mmol NaClO/g of dry pulp) increased the tensile index by 18% and more than 25% using 1 and 2 wt.% of CNF, respectively (Figure 4). A higher increase in the tensile strength (28.5% and 34.5% with 2 and 3 wt.% CNF, respectively) was obtained with the addition of CNF produced with the higher oxidation degree (15 mmol of NaClO/g of pulp), these being the highest nanofibrillated fibers in terms of nanofibrillation yield and transmittance. The CNF obtained from the recycled ONP pulp, oxidized with 10 mmol of NaClO/g of pulp, appears to be adequate for the improvement of mechanical properties of recycled paper, as there is high tensile index enhancement, similar to that obtained with 15 mmol of NaClO/g of pulp (only 5% less), but requiring 33% less NaClO. Results showed that there is a relationship between the quality of the CNF and the improvement in the mechanical properties, but this relationship is complex and it is not proportional. In other studies the quality of the CNF did not have a simple direct relation on the mechanical properties of the recycled paper [21] due to the influence of other factors, such as the dispersion of the CNF in the pulp suspension, the interaction between the CNF and the rest of the components present in the pulp, as well as the flocculation processes that may occur and affect the uniformity of the final paper [31]. This shows the importance of the application methodology for the CNF industrial application.

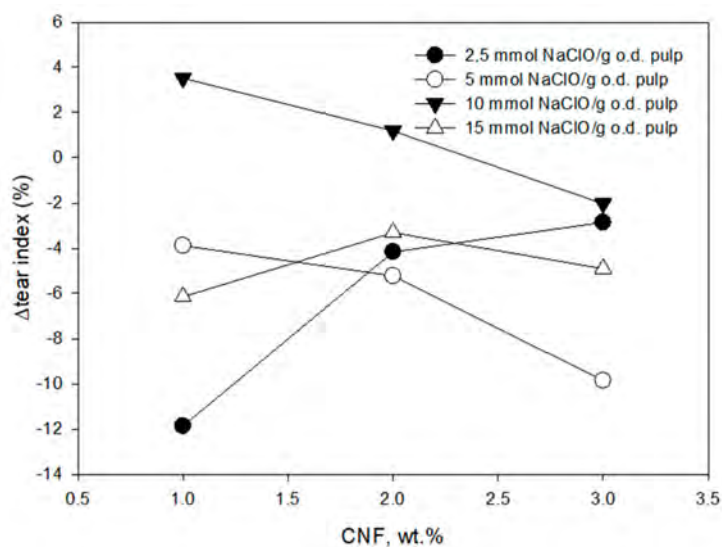
The effect on the tear index is not very significant although there were improvements achieved at 10 mmol of NaClO/g of pulp for 1 and 2 wt.% of CNF (Figure 5). As expected, the porosity of the handsheets is lower when the amount of CNF increases and the lowest values of porosity were obtained at high TEMPO-mediated oxidation level due to the high yield of the nanofibrillation achieved at these conditions (Figure 6). Both an increase in CNF content and a higher fibrillation produces a higher block of pores that reduces the porosity. The reduction of porosity produce a higher interaction in the hydrogen bounding improving the tensile strength. However, the tear strength does not show a clear trend due to the dependence of several factors. On the one hand, the tear index improves with the hydrogen bounding that increases with CNF content and the block of pores. However, the same property is reduced with short fibers that facilitate the tear of the sheet.

Comparing the improvement in tensile strength with published data, available only for CNF produced from virgin and agriculture residues pulps, the conclusion is that the effect depends on both the type of CNF and the used retention system. Merayo et al. (2017) [32] used bleached Eucalyptus pulp to produce CNF (TEMPO-5 mmol of NaClO/g of pulp and six steps of homogenization at 600 bars)

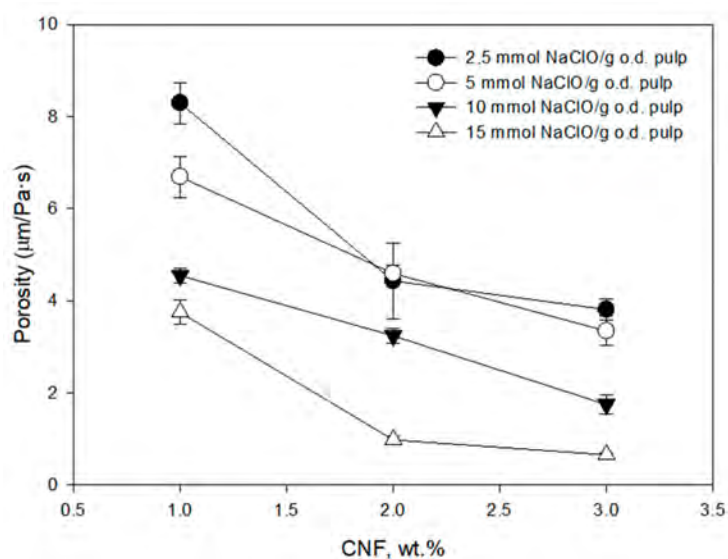
and the tensile index of newsprint increased around 60%, using 3 wt.% of CNF and the same retention system as in this study. In a similar study, Delgado-Aguilar et al. (2015) [16] obtained an increase up to 52% when cationic starch was used as retention system; although the real improvement by the 3% CNF addition was lower since cationic starch is also a strength agent. When corn stalk pulp was used to produce CNF (TEMPO-15 mmol of NaClO/g of pulp and six steps of homogenization at 600 bars) tensile index of newsprint also increases by 60% using the same TRDS as in this paper [32]. On the other hand, when 3 wt.% of CNF (obtained by bleaching with 8 wt.% NaClO/g of pulp and a homogenization cycle of three steps at 300 bars, three steps at 600 bars, and three steps at 900 bars) from sawdust of pine; Eucalyptus and triticale were used with the same TRDS and the tensile index of newsprint increased by 15%, 8%, and 3.5%, respectively [33]. Even more interesting is the comparison of the obtained data with the use of CNC, produced and applied to the same recycled newspaper. In this case, similar increments up to 30% in the tensile strength index were achieved when 3 wt.% of CNC was added into the recycled pulp using a similar polyacrylamide-based retention system [24].



**Figure 4.** Effect of CNF dose and TEMPO-oxidation degree on tensile index increment of the recycled ONP paper using a three-component retention and drainage system (C-PAM-B).

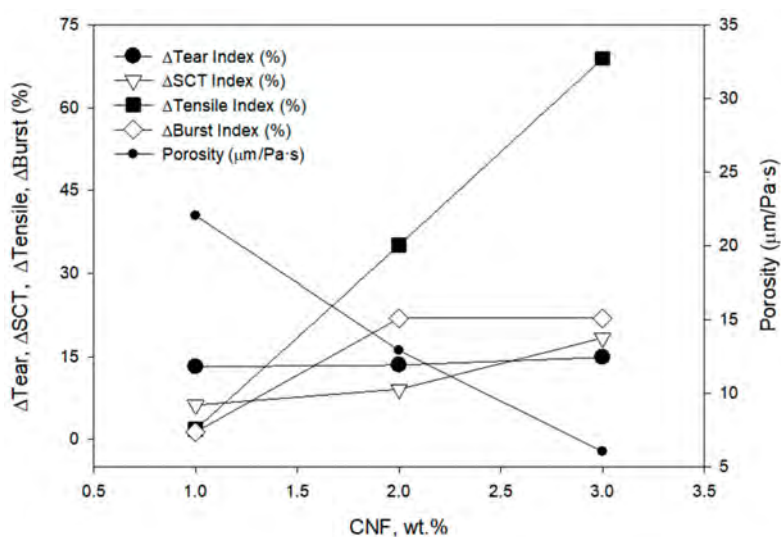


**Figure 5.** Effect of CNF dose and TEMPO-oxidation degree on tear index increment of the recycled ONP paper using a three-component retention and drainage system (C-PAM-B).



**Figure 6.** Effect of CNF dose and TEMPO-oxidation degree on porosity of the recycled ONP paper using a three-component retention and drainage system (C-PAM-B).

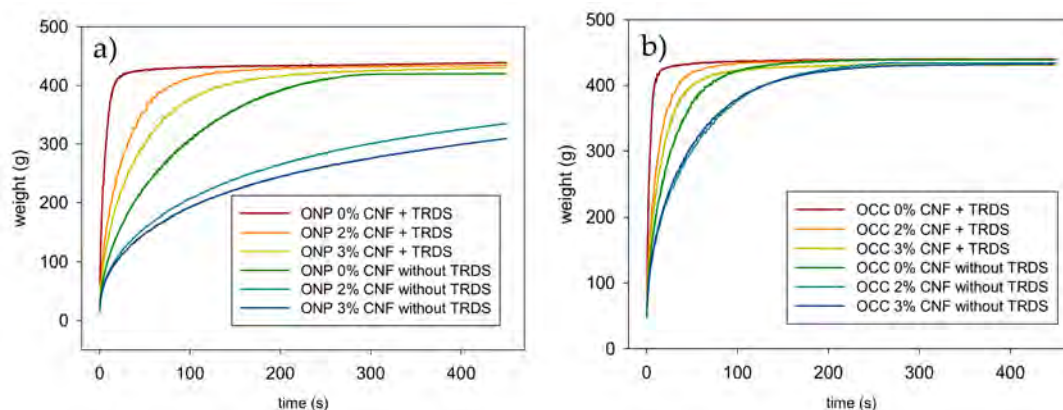
CNF from recycled OCC were prepared by TEMPO-mediated oxidation using 10 mmol of NaClO per gram of pulp before the homogenization because those were the optimal mechanical properties of the handsheets obtained previously for recycled ONP. The effect of the CNF on the short-span compressive test (SCT) and burst index of the cartonboard—the same raw material as the one used to prepare CNF, besides tensile index, tear index, and porosity—was studied at 1, 2, and 3 wt.% doses of CNF. The retention and drainage system C-PAM-B was added to the pulps, and handsheets were formed and characterized. In this case, 3 wt.% CNF increased the tensile index above 60%, and decreased the porosity, as it was expected (Figure 7). Burst index and SCT increased above 15% for a 3 wt.% CNF dose. The mechanical properties obtained are similar than the results of Balea et al. (2016) [14] that used virgin pulps, namely bleached Eucalyptus and pine, to produce CNF using cationic starch as retention system. They applied 3 wt.% CNF reaching only 20–25% tensile index improvement; however, they achieved 30% and 37% increases in burst and tensile indexes, respectively.



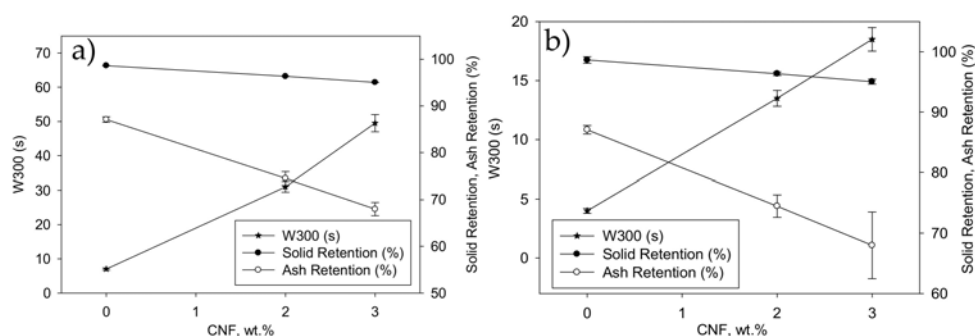
**Figure 7.** Effect of CNF dose on mechanical properties of the recycled OCC paper using CNF produced from recycled OCC using 10 mmol of NaClO per gram of pulp before the homogenization and a three-component retention and drainage system (C-PAM-B).

### 2.3. Effect of CNF on Retention and Drainage Process

The effect of the addition of CNF was assessed on the retention and drainage process of recycled ONP and recycled OCC with and without a retention system. In both cases, drainage effect was studied only with CNF oxidized with 10 mmol of NaClO/g of pulp before the homogenization. Figure 8 shows the drainage curves of experiments performed with and without TRDS for recycled ONP (Figure 8a) and recycled OCC (Figure 8b). To compare drainage results, drainage time was calculated when 300 g of water were drained (W300). Figure 9 shows the effect of CNF dose on the W300 time, the total solid retention and ash retention in recycled ONP and OCC pulps (Figure 9a,b, respectively) using TRDS.



**Figure 8.** Effect of CNF dose and three-component retention and drainage system on the drainage process. (a) ONP and (b) OCC.



**Figure 9.** Effect of drainage time (W300), solid retention, and ash retention of the handsheets with different doses of CNF and TRDS. (a) Recycled ONP pulp and (b) recycled OCC pulp.

In general, the drainage time of the recycled ONP pulp was lower than OCC in all conditions studied, probably due to the higher amount of fines that the recycled ONP pulp has compared to the recycled OCC pulp (Table 1). Cellulose fines usually consist of a very complex and heterogeneous set of materials, thus a certain fraction of fines is similar to cellulose microfibrils [19]. The drainage results obtained in this study are according to Taipale et al. (2010) [19] and Johnson et al. (2016) [34], which demonstrated that lower content of fines in the pulp decreased the dependence of the drainage time with the CNF content. In both recycled pulps, higher doses of CNF absorbed more water in the pulp, making more difficult the drainage process, thus increasing the W300 by 334% and 77% when 3 wt.% CNF from recycled ONP and recycled OCC were added into the pulp in absence of TRDS. However, this effect can be reduced by 48% and 30%, respectively with the incorporation of TRDS as retention agent. These results are according to several authors, which demonstrated that the addition of CNF into a pulp suspension gets worse the drainage rate but this effect can be counteracted by the addition of different retention systems [19,21,32,34].

For total solids retention, determined through gravimetric analysis, the addition of both CNF reduced their retention, reaching a minimum of 95% when a 3 wt.% of CNF was added to the recycled pulps. Finally, the trend of the ash retention was similar as that of total solids retention, decreasing as CNF content increased. Both recycled ONP and OCC pulps without CNF have an ash retention ~85%, whereas these values decrease until 70 wt.% when a 3 wt.% of CNF was added (Figure 9).

### 3. Materials and Methods

#### 3.1. Materials

Recycled old newsprint (ONP) and old corrugated containers (OCC), manufactured by Holmen Paper Madrid (Madrid, Spain) and R  pina Paperivabrik AS (R  pina, Estonian), respectively, were used as raw materials to simulate broke streams to produce both the CNF and the recycled pulps in which CNF were added at different dosages. Table 1 presents the results of the morphological analysis of the ONP and OCC pulps, obtained using a Morfi analyzer V7.9.13E (Techpap, France).

A three-component retention and drainage system (C-PAM-B) was assessed as it is commonly used in the recycled paper industry. The doses used for the laboratory experiments are based on industrial recommendations [35]. The three-component system selected was a bentonite-based microparticle system that contains: 1.25 mg/g of cationic polyamine as coagulant (cationic charge density of 0.035 meq/g and high molecular weight); 0.75 mg/g of cationic polyacrylamide (PAM) with high molecular weight (cationic charge density of 3.66 meq/g) as flocculant; and 1.7 mg/g of hydrated bentonite clay, all of them supplied by BASF (Ludwigshafen, Germany).

#### 3.2. Methods

##### 3.2.1. CNF Production and Characterization

CNF produced from recycled ONP and OCC were obtained by TEMPO-mediated oxidation by using 2.5, 5, 10, and 15 mmol of NaClO/g of pulp. The reaction was performed at room temperature, maintaining pH at ~10, and using a NaOH solution at 0.5 M [36]. After the oxidation process, the pulp was cleaned through filtration steps using tap water to reach a neutral pH. Finally, CNF were homogenized in a PANDA PLUS 2000 laboratory homogenizer (GEA Niro Soavi, Italy) at 600 bar. The number of passes through the homogenizer was the required to obtain a gel suspension of CNF and depend on the oxidation grade (in the case of CNF with 2.5 and 5 mmol of NaClO, 15 passes were applied, for 10 mmol of NaClO the number of passes was 5 and, finally, 3 passes were applied for CNF with 15 mmol of NaClO).

To characterize the oxidized cellulose pulp the amount of carboxyl groups was measured as an indicator of the oxidation degree achieved after TEMPO-mediated oxidation by conductimetric titration according to Balea et al (2016) [15] and calculated based on the method development by Habibi et al. (2006) [37]. As for CNF characterization, nanofibrillation yield was measured in a diluted CNF suspension (0.1 wt.%) by centrifugation at 4500× g for 30 min. The nanofibrillated fraction is isolated in the supernatant from the nonfibrillated fraction deposited in the sediment. Transmittance of the CNF suspensions diluted at the same concentration as previously were measured between 400 and 800 nm of wavelength using a Cary 50Conc UV-visible spectrophotometer (Varian Australia Pty Ltd, Victoria, Australia). Cationic demand was measured by colloidal titration of the diluted suspension at 0.05 wt.%, with 0.001 N polyDADMAC, using a M  tek PCD04 particle charge detector (BTG Instruments GmbH, Herrsching, Germany). Finally, polymerization degree was calculated from the limiting viscosity number of CNF suspensions, using cupriethylenediamine as a solvent and determined by the international standard ISO5351/1, based on Mark-Houwink-Sakurada (MHS) equation and the studies of Marx-Figini (1978) [38] and Henriksson et al. (2008) [39].



### 3.2.2. Handsheet Preparation and Characterization

Recycled pulps (ONP and OCC) were prepared through disintegration of 20 g of dry recovered paper in 2000 mL of water using a Messmer pulp disintegrator (Mavis Engineering Ltd, London, UK). The recovered paper with the correspondent amount of CNF (1.0, 2.0, and 3.0 wt.%) was left to soak at least 24 h before disintegration to favor swelling. A three-component retention system was added to the pulp (1.25 mg/g of coagulant, 0.75 mg/g cationic polyacrylamide as flocculant, and 1.7 mg/g hydrated bentonite clay based on industrial recommendations). The pulp was used to prepare handsheets with basis weight of 80 g/m<sup>2</sup> for both recycled papers in a normalized handsheet former Rapid-Köthen (ISO 5269/2, DIN 54 358).

Mechanical properties were determined by measuring tensile strength index (kN·m/kg), tear strength (mN), porosity (μm/Pa·s), short-span compressive test (SCT) index (N·m/g), and bursting strength index (kPa·m<sup>2</sup>/g). Tensile strength was measured in a MTS Criterion Mode 43 from MTS Systems Corporation (Eden Prairie, MN, USA), following ISO 1924-3 (2014) standard. Tear strength was determined according to ISO 1974:2012 using a tearing resistance tester. Bendtsen porosity (μm/Pa·s) was measured with a Bendtsen Porosity Tester n° 8699 from Andersson & Sørensen (Copenhagen, Denmark) according to ISO 5636-3 (2013). To measure the cross directional short-span compressive strength a short span compression tester (Messmer Büchel, Veenendaal, The Netherlands) was used according to TAPPI T826 standard (2013). Finally, bursting strength was measured in a Messmer Büchel digital hydraulic board burst tester according to standard ISO 2759 (Veenendaal, The Netherlands).

### 3.2.3. Retention and Drainage Measurements

Drainage measurements of the pulp suspensions were carried out in a Müttek<sup>TM</sup> DFR-05 (DFR) from BTG Instruments (Säffle, Sweden), which provided the drainage curves of the pulp when it is drained by gravity through 150 mesh. Experiments were performed with 500 mL of pulp suspension at 0.5 wt.% consistency. First, the pulp suspension was placed in an agitation chamber and it was agitated at 300 rpm. After 30 s of initial stirring, the retention aids were added to the pulp in the DFR (coagulant was firstly added, then, at consecutive intervals of 30 s, cPAM and bentonite were also added). Finally, after a further 30 s of mixing, the stirring was stopped, and the filtration step began monitoring and recording the weight of the drained on real time. Solids retention was measured by gravimetric analysis of the total solids in the drained water at 105 °C, and ash retention was determined by incineration at 525 °C (ISO 1762, 2015).

## 4. Conclusions

In situ production of CNF, from recycled ONP and OCC that simulate broke streams of the paper machines, is feasible in terms of improving the final product quality. The potential increase of the strength properties depends on the CNF properties, which are linked to the CNF production, the CNF dosage, and the retention system used. Implementation of this strategy would reduce the costs and difficulties of CNF transportation and application, valorizing the waste streams containing cellulose and contributing to the sustainability and circular economy in the process. Furthermore CNF could be also sold in the local market for other applications to contribute to the economy of the process.

The use of 10 mmol of NaClO/g of pulp in TEMPO-mediated oxidation before the homogeneization is enough to improve mechanical properties of recycled ONP pulp with an increase of tensile index around 30% with a 3 wt.% of CNF and with slightly impact on tear index. These results are very similar to the application of 3 wt% CNC obtained from the same raw material and applied to the same newsprint pulp. A further increase in the oxidation conditions to produce CNF do not improve efficiently the mechanical properties.

On the other hand, the optimal conditions to prepare CNF from ONP were also applied to prepare CNF from OCC. In this case, tensile index increased above 60% with a 3 wt.% CNF, whereas tear, SCT, and bursting indexes raised ~15–20%. Finally, CNF from both cellulose sources had worse drainage,

but this effect was effectively counteracted with the optimization of the three-component retention system used.

**Author Contributions:** Conceptualization, C.N. and A.B. (Angeles Blanco); Formal Analysis, A.B. (Angeles Blanco), A.B. (Ana Balea), J.L.S.-S., C.N., and M.C.M.; Investigation, A.B. (Ana Balea), J.L.S.-S., and N.M.; Data Curation, A.B. (Ana Balea) and J.L.S.-S.; Writing—Original Draft Preparation, A.B. (Ana Balea), J.L.S.-S., A.B. (Angeles Blanco) and M.C.M.; Writing—Review and Editing, A.B. (Ana Balea), M.C.M. and N.M.; Supervision, A.B. (Angeles Blanco) and C.N.; Project Administration, C.N.; Funding Acquisition, A.B. (Angeles Blanco) and C.N.

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**Conflicts of Interest:** The authors declare no conflicts of interest.

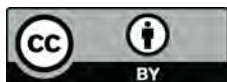
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**Sample Availability:** Not available.



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## Manufacturing fit-for-purpose paper packaging containers with controlled biodegradation rate by optimizing addition of natural fillers

Anna SANDAK, Jakub SANDAK and Izabela MODZELEWSKA

Natural fillers were utilized for manufacturing horticultural packaging products. Five types of pots produced from waste paper with wheat and rye bran additions were compared with commercially available containers. The aim was to examine the influence of soil type on the degradation rate and kinetics. Pots were degraded in three soil types: agriculture, forest and sandy soils and were monitored after 2, 4 and 8 weeks. NIR spectroscopy was used for non-destructive evaluation of the chemical composition of the investigated papers in addition to typically used standard methods. All tested configurations of papers might be used for manufacturing of plantable bio-containers that will slowly disintegrate during their use. The addition of cereal bran improves mechanical properties of the paper and extends the lifespan of pots. The rate and extent of decomposition depends mainly on the degradation time and type of soil. Paper pots in all tested configurations degraded most quickly in agricultural and forest soils, each stimulating growth of microorganisms responsible for the decomposition of paper. The obtained results allow selection of products with optimal composition for specific applications and to design the packaging containers degradation time in various in-field scenarios. The manufacturing approach proposed increases the positive footprint of packaging products by designing “eco-effective” solutions according to the Cradle to Cradle design framework.

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The Paper Industry Technical Association (PITA) is an independent organisation which operates for the general benefit of its members – both individual and corporate – dedicated to promoting and improving the technical and scientific knowledge of those working in the UK pulp and paper industry. Formed in 1960, it serves the Industry, both manufacturers and suppliers, by providing a forum for members to meet and network; it organises visits, conferences and training seminars that cover all aspects of papermaking science. It also publishes the prestigious journal *Paper Technology International* and the *PITA Annual Review*, both sent free to members, and a range of other technical publications which include conference proceedings and the acclaimed *Essential Guide to Aqueous Coating*.



# Manufacturing fit-for-purpose paper packaging containers with controlled biodegradation rate by optimizing addition of natural fillers

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**Abstract** Natural fillers were utilized for manufacturing horticultural packaging products. Five types of pots produced from waste paper with wheat and rye bran additions were compared with commercially available containers. The aim was to examine the influence of soil type on the degradation rate and kinetics. Pots were degraded in three soil types: agriculture, forest and sandy soils and were monitored after 2, 4 and 8 weeks. NIR spectroscopy was used for non-destructive evaluation of the chemical composition of the investigated papers in addition to typically

used standard methods. All tested configurations of papers might be used for manufacturing of plantable bio-containers that will slowly disintegrate during their use. The addition of cereal bran improves mechanical properties of the paper and extends the lifespan of pots. The rate and extent of decomposition depends mainly on the degradation time and type of soil. Paper pots in all tested configurations degraded most quickly in agricultural and forest soils, each stimulating growth of microorganisms responsible for the decomposition of paper. The obtained results allow selection of products with optimal composition for specific applications and to design the packaging containers degradation time in various in-field scenarios. The manufacturing approach proposed increases the positive footprint of packaging products by designing “eco-effective” solutions according to the Cradle to Cradle design framework.

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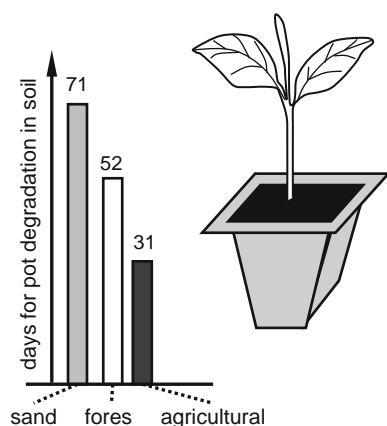
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## Graphical abstract



**Keywords** Waste paper · Paper pots · Cereal bran · Degradation rate · Soil · FT-NIR

## Introduction

Over 67 million tons of packaging wastes are generated annually in the EU, containing up to 30% of slowly degrading plastics. The Packaging and Packaging Waste Directive, which standardizes the production of packaging materials, waste management and the use of recycling, composting, and energy recovery by incineration regulates the bio-based packing sector (European Parliament and Council Directive 1994). There are clear targets regarding waste reduction in the EU. Seventy-five percent of packaging waste should be recycled by 2030 (Niero and Hauschild 2017). Re-utilization of waste paper is therefore important for reducing waste generation but also for saving wood resources (Kose et al. 2016). According to Chartered Institution of Waste Management, the reduction of waste quantity might save 72 billion € per year and create over 400,000 new jobs in Europe (Cheshire 2016).

The increased use of bio-based materials is essential in order to reduce the environmental impact of materials, including reuse and ultimately, disposal. It is expected that use of biodegradable materials will contribute to sustainability and reduction in the environmental impact associated with disposal costs (Song et al. 2009). Moreover, increased bio-based

material use is in line with Circular Economy (CE) objectives, which aims to maximize value at each point in a product's life by keeping products, components and materials at their highest utility at all times (Stahel 2016).

Currently, landfilling is the dominant method of packaging waste disposal, followed by recycling, incineration, and composting (Valdés et al. 2014). However, it is considered as “leakage” from circular system, meaning that valuable resources are wasted and lost from environmental and economical point of view (Cheshire 2016). The most favorable way for transformation of ligno-cellulosic wastes is by recycling. The feasibility of recycled fibers for the production of high value-added papers to be used for packaging purposes was recently reported by Tarrés et al. (2018). However, fibres recovered from paper wastes after re-pulping process have reduced mechanical properties. According to Wistara and Young (1999) tensile strength, bursting strength, and apparent density of the pulps decreases when recycling paper. After a maximum of 6–7 recycling cycles, fibres become too short for further processing. Consequently, additives (new fibres or fillers) are necessary to enrich recycled pulp and to minimize depreciation of its quality (Villanueva and Wenzel 2007). In some circumstances increased use of fillers leads to decrease paper strength (Balea et al. 2018). Moreover, physical recycling may be impractical in case of packaging materials contaminated with foods or other biological substances (Kale et al. 2007). Composting paper waste is considered as one of the less costly disposal routes, and is an option for recycling (López Alvarez et al. 2009). In this scenario, biodegradability becomes a desirable feature for several everyday products, including packaging. Although ultimate biodegradability in the natural environment is important, sustainable packaging products are required to biodegrade in a controlled and industrially acceptable way (Scott and Wiles 2001). However, the main factor that affects the formation and manufacturing of bio-based packaging is related to economic aspects.

Biodegradable pots, developed as an alternative for traditional petroleum derived plastic containers are environmentally friendly and frequently used for silvicultural and agricultural purposes. Such containers reduce overall costs, as seedlings with the biodegradable pots can be planted quickly while avoiding root disturbance or any interruption to plant growth.

The most commonly used disposable pots are made of peat or a mixture of peat with wood fibres. Such pots can be easily embedded into the soil with plants or converted into bio-gas (digested) after removing the plant. On the other hand, such containers are mechanically unstable and possess high permeability to water vapour. Salt deposition on pot walls is frequently observed and causing nutrient content to become unavailable. This may have a negative impact on plant production and therefore horticulturalists are not always confident to use pots made of peat (Treinyte et al. 2014). Moreover, some consumers avoid the use of peat because peat harvesting may be unsustainable and possibly contribute to global climate change (Mitsch et al. 2013). Containers produced from coconut fibres or bird feathers are interesting alternatives, as these are mechanically more resistant and retain moisture well. However, these cannot be embedded in soil with plants and can only be disposed or digested afterward (Treinyte et al. 2014). Recycled plastic geotextiles are other option recently introduced to the market. These are not easily biodegradable or compostable, but will slowly disintegrate when exposed to the soil. A common limitation of these products is their relatively high price, therefore continuous research regarding development of novel packaging products is ongoing.

Natural fibres and agricultural residues are becoming attractive fibre reinforcement solutions for biocomposites (Ochi 2011; Schettini et al. 2013; Nambuthiri et al. 2015; Tesfaye et al. 2017). Substances from plant waste materials (such as: cellulose, hemicellulose, starch, dextrin, and other carbohydrate polymers) are the most convenient solution as they solve two problems simultaneously: they contribute to efficient waste management and avoid or minimize the use of chemical additives as binders (Müller et al. 2007). A solution that fulfils both of these requirements is cereal bran. In typical flour production processes, cereal bran is devoid of nutrients, and is most often separated for disposal, leading to handling, storage and disposal costs. According to Formela et al. (2016) bran are interesting alternative for commercially available cellulosic fillers and could be successfully applied as a low-cost filler in polymer composites. Therefore, bran may be an ideal filler for the extruded paper or pulp containers used extensively in horticulture. It may also serve as an

inhibitor controlling the bio-degradation rate in various products (Sandak et al. 2011).

Evaluation of paper products decomposition in laboratory conditions with selected microorganisms was previously reported (Modzelewska et al. 2010; Jaszczur and Modzelewska 2011; Sandak et al. 2015). However, López Alvarez et al. (2009) have emphasized the necessity to establish biodegradation curves for different packaging products in landfill and/or composting end-of-life scenarios. Such experiments conducted in different soil types and climates are essential before adopting containers made with alternative materials. It is important to note that the same physical characteristics that promote degradation during composting could also contribute to premature degradation during production and transportation. Depending on their capacity to degrade at their end-of-life, alternative containers are usually classified as plantable, compostable or recyclable (Nambuthiri et al. 2015).

Various types of soil have different influence on degradation rates mainly due to variability in water-holding capacity (Rahman and Chattopadhyay 2007), available nitrogen, pH, presence of microorganisms and organic matter content (Nambuthiri et al. 2015). In soil with neutral or slightly acid pH (6.0–7.0), favourable microorganisms and minerals necessary for plant roots are present (Yeomans 1954). Such beneficial bacteria are not present in more acidic soils, leading to uncontrolled mould fungi growth. Moreover, most of minerals are insoluble in soils possessing low pH. Sandy soil is airy and highly permeable. It has rather low water storage capacity, is fast drying and easily loses nutrients due to leaching. In practical applications, it can be improved by adding organic matter or fertilizers. Soil present in coniferous forests contain litter compost composed primarily of pine or spruce needles, ericaceous understorey plant species and mosses (Hilli et al. 2010). The compost is highly acidic but has good leavening properties and can be used as an optimal peat substitute providing favourable conditions for plant growth (Drozd et al. 2002).

Recycled paper containers have been proven to have the comparable wet and dry vertical and lateral strength, similar to those of plastic containers, and showed no algal or fungal growth on the container wall (Nambuthiri et al. 2015). However, additives and fillers might influence the performance of packaging products.

The goal of this work is to increase the positive footprint of packaging products by designing “eco-effective” solutions according to the Cradle to Cradle design framework. Reutilization of two kinds of materials (waste paper and cereal bran) is proposed here in order to close the loop at the end of product life cycle. The aim was to design, manufacture and examine the degradation intensity of waste papers containing cereal bran with a special focus on the effect of the soil type. Both, paper sheets and paper pots were evaluated. It is expected that by understanding the degradation rate of the investigated products it will be possible to optimize paper pots manufacturing to assure sufficient mechanical resistance and the desired rate of degradation.

## Materials and methods

### Materials

Two forms of paper products were examined in this study including flat sheets of paper and paper pots. Five configurations were made for both products (Table 1). In case of paper sheets, 24 samples with dimension of  $15 \times 95$  mm, were cut from each configuration of paper. Fifty pots were manufactured from individual paper type.

#### *Paper sheets*

Paper sheets were produced in the laboratory at University of Life Sciences in Poznan on the Rapid-Köthen apparatus from recycled pulp type D (cardboard, paper, grey bags, corrugated board), weighing  $100 \pm 5$  g m<sup>-2</sup>. The pulp was milled on the laboratory mill PFI (till  $28 \pm 2^\circ$ SR value). The wetness was measured with a Schopper-Riegler device. The pulp

was defibred using a defibrator and formed into sheets of paper with a Rapid-Köthen device. Manufactured papers were made of pulp without and with 3% or 5% addition of wheat and rye bran. Both additives were by-product of the flour production and were provided by Gdańskie Młyny i Spichlerze Dr Cordesmeier Sp. z o.o. The fraction size of bran particles was below 0.4 mm. Details for the sample composition and labelling of investigated papers are summarized in Table 1.

#### *Paper pots*

Paper pots were manufactured in the laboratory from the same pulp as reference flat paper sheets and with identical additives configuration (Table 1). The custom machine for forming pots was developed at University of Life Sciences in Poznan (grant number N30900831). The pot was formed directly in the container from water pulp solution with 2.5% dry mass concentration. 600 cm<sup>3</sup> of the pulp solution was used for manufacturing of each pot. The perforated mold was used to deposit a layer of fibres with additives, where water was removed from the cast by a vacuum pump. The formed pots were dried for 12 h at 50 °C in a climatic chamber. The final size of the manufactured pot was  $6.0 \times 6.0 \times 5.5$  cm (width  $\times$  length  $\times$  height, respectively). Degradation and characterization were performed on commercially available products (pots manufactured with the addition of peat) in order to compare their overall performance with laboratory produced pots. Figure 1 presents images of pots manufactured in the laboratory and alternative commercially available products.

### Methods

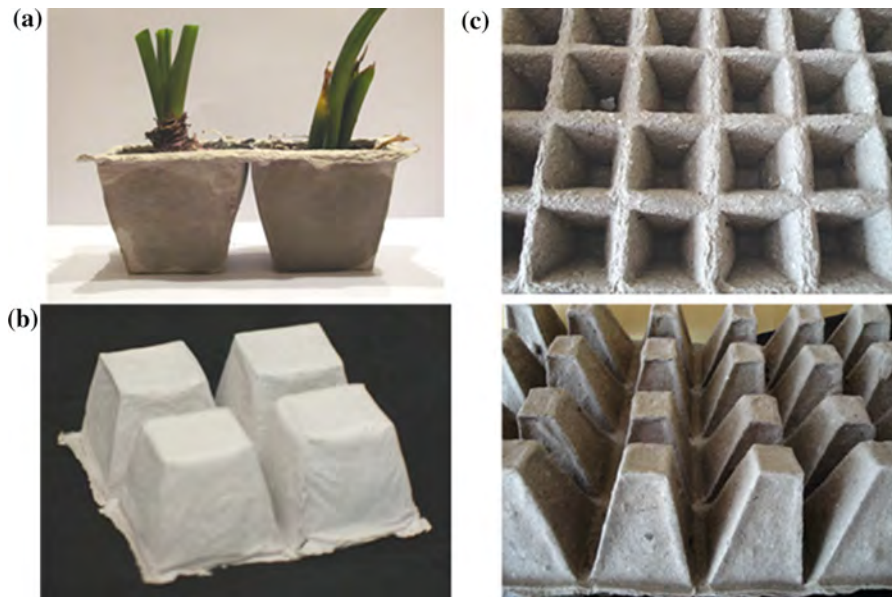
#### *Degradation in soil*

The substrate used in this experiment was a natural soil sample collected close to Poznan (Poland). The soil was sieved to less than 2-mm particle size before degradation tests, and obvious plant material, stones, or other inert materials were removed. The degradation process was conducted in conditioned laboratory in 20 °C and 65% RH. Three different types of soil (sandy, forest and agricultural) were selected as degradation environments for the investigated paper products. The pH of sandy soil was  $\sim 7.5$  to 8.0.

**Table 1** Summary of investigated samples composition

Sample code	Mass content of additives (%)	
	Wheat bran	Rye bran
WP	0	0
WP3W	3	0
WP5W	5	0
WP3R	0	3
WP5R	0	5





**Fig. 1** Plants growing in the bio-degradable paper pots (a), laboratory manufactured prototype pots made of waste pulp with addition of cereal bran (b), commercially available pots made of pulp with addition of peat (c)

Coniferous forest soil is usually enriched with cones and needles, but is rather poor in nutrients. It was the most acid soil studied and had pH of  $\sim 3.5$  to 4.0. Agricultural soil, which consisted of highly decomposed matter deeply mixed with mineral soil had pH of  $\sim 5.0$  and carbon to nitrogen ratio of  $\sim 10$ . The laboratory experiment was conducted in plastic boxes containing soils. Experimental samples (paper sheets) were fully covered by the soil. Paper pots were placed in the containers to the depth corresponding to  $\sim \frac{3}{4}$  of the pot height. All the samples were exposed to the soil for 2 months and the degradation progress was monitored at the 2nd, 4th and 8th week of the experiment. The moisture of the soil during the whole campaign was kept constant in the range of around 55%. The water capacity of the soil and the ambient temperature was continuously monitored during the tests in order to provide amount of water required to achieve an adequate level of humidity. Containers with samples after installation were kept in climatic chamber (20 °C, 60% RH). Once a month small amount of water was added to the constant weight of the containers.

#### *Evaluation of paper properties*

The following tests were performed before degradation in soil (according to corresponding standards) in order to characterize the produced paper sheets:

- Breaking length (PN-ISO 1924-1 [1998](#)) on the Schopper apparatus.
- Extensibility (PN-EN ISO 1924-2 [2009](#)) on the Schopper apparatus.
- Tearing resistance (PN-EN ISO 1974:2012P [2012](#)) on the Elmendorf apparatus.
- Burst test (PN-EN ISO 2758 [2005](#)) on the Mullen's apparatus.
- Air permeability according to Gurley (PN-ISO 11004 [1995](#)) on the Gurley apparatus.
- Rate of absorbency according to Cobb (PN-EN 20535 [1996](#)) on the Cobb apparatus.

In addition, crushing strength was tested on paper pots on the Zwick/Roell Z005 (Zwick Roell AG, Ulm, Germany) mechanical testing machine in compression mode according to TAPPI T804 standard ([2006](#)). All the samples were conditioned to  $7 \pm 1\%$  moisture content before characterization. It was impossible to perform all tests on some samples following degradation due to excessive disintegration.

#### *Polymerization degree*

The degree of cellulose polymerization (PD) for paper products was determined on samples before and after soil degradation. During the test, the intrinsic viscosity

was determined in cupri-ethylenediamine solution (CED) according to ISO 5351-1 (1981).

### Chemical analysis

The concentration of cellulose was determined according to the Seifert procedure by using acetylacetone-dioxane-hydrochloric acid (Browning 1967). The quantities of other wood components were determined according to the following standards:

- Hot water extractives (T207 cm-08 TAPPI 2008).
- 1% NaOH extractives (T212 om-07 TAPPI 2007).

The solubility in 1% H<sub>2</sub>SO<sub>4</sub> was performed following the same procedure as in 1% NaOH (T212 om-07 TAPPI 2007). All the chemical analyses were performed before and after degradation and were repeated three times and the maximum standard deviation of results was considered as the indicator of the measurement error.

### Statistical data analysis

A factorial one-way ANOVA was conducted to compare mean values of chemical components concentration and paper properties at significance level  $p < 0.05$ . In addition, Tukey contrasts simultaneous test for general linear hypothesis with multiple comparison of means was performed post hoc to identify groups of statistically similar samples. The analysis were performed in R software ([www.r-project.org](http://www.r-project.org)) by using multcomp module.

### Electron microscopy

A scanning electron microscope (SEM; S-3400 N, Hitachi High-Tech, Tokyo, Japan) in high vacuum

mode with a secondary electron detector was used for imaging samples. It was necessary to coat samples with a thin layer of gold in order to obtain quality SEM images.

### FT-NIR measurement

NIR measurements were made using a Vector 22-N spectrophotometer (Bruker Optics GmbH, Ettlingen, Germany). The measured spectral range was between 4000 and 12,000 cm<sup>-1</sup> (2500 nm and 833 nm) with a resolution of 8 cm<sup>-1</sup>. The spectral wavenumber interval was 3.85 cm<sup>-1</sup> with zero-filling = 2. An average of 32 consecutive measurements (internal scans) was acquired for each spectrum. The degradation stage of all papers was determined on three representative paper strips and measured on three spots, before and after 4 and 8 weeks of degradation in each soil. The resulting spectra (9 spectra) were averaged and stored in a data base for further analysis. All measurements were performed in an air-conditioned room (20 °C and 65% RH) in order to minimize the effects of the temperature and moisture variations.

### Spectral data analysis

The interpretation of spectra based on Schwanninger et al. (2011) and corresponding band assignments are summarized in Table 2. A narrow range, between 4000 and 6000 cm<sup>-1</sup>, was selected for spectra interpretation. Computation of the second derivative [21-point smoothing, Savitzki and Golay algorithm (1964)] was applied for spectra pre-processing. Spectra were also pre-processed with extended multiplicative scatter correction (EMSC) algorithm for investigation of the degradation rate. Principal component analysis (PCA) was used for data analysis. The commercially available OPUS 7.0 (Bruker Optics

**Table 2** Interpretation of the FT-NIR molecular vibrations (Schwanninger et al. 2011)

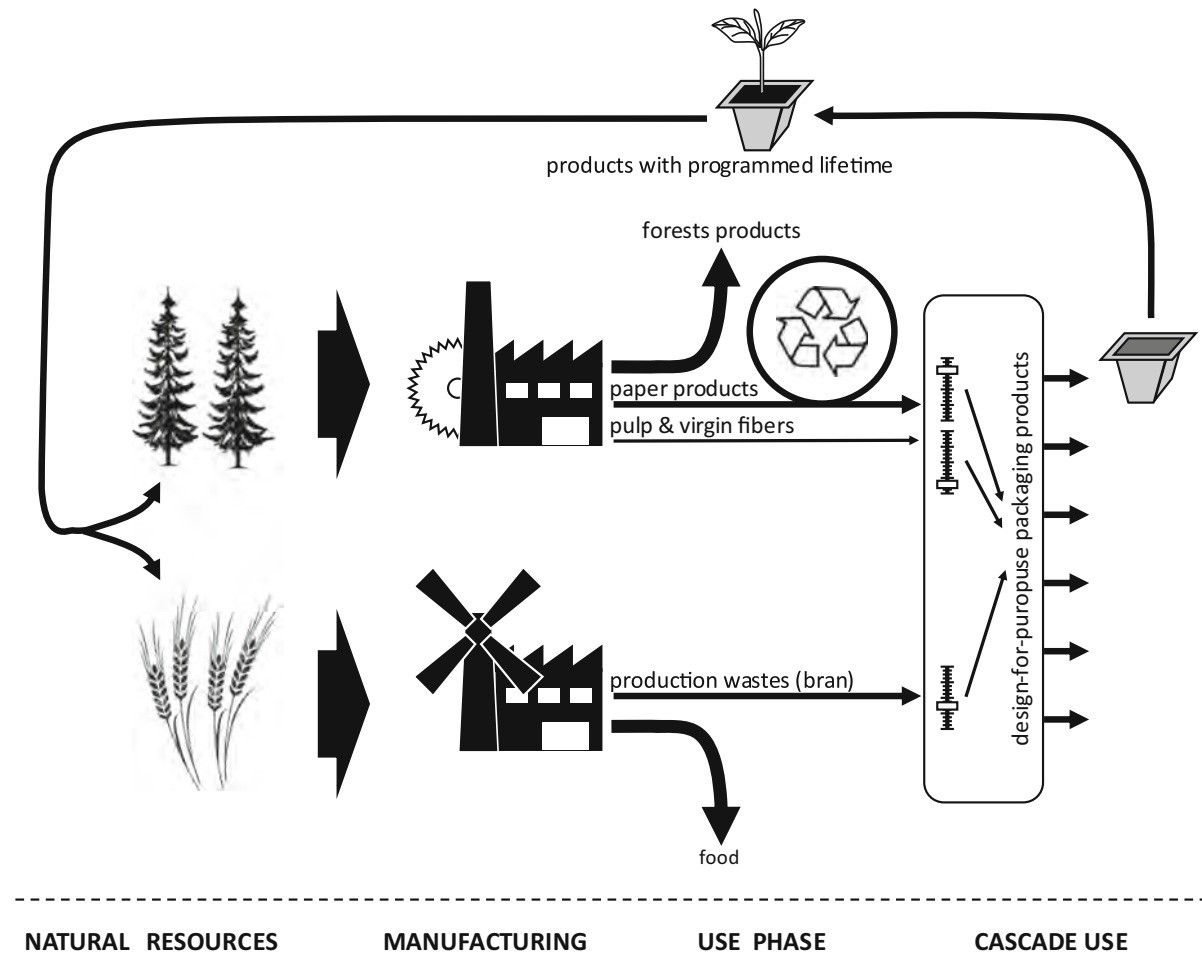
nr	Band assignment	Chemical component	Wavenumber (cm <sup>-1</sup> )
1	C–H def.	Holocellulose	4198
2	C–H def. + C–H str.	Cellulose	4280
3	C–H <sub>2</sub> def. + C–H <sub>2</sub> str.	Cellulose	4404
4	C–H def. + O–H str.	Cellulose	4620
5	C–H def. + O–H str.	Cellulose	4890
6	O–H def. + O–H str.	Water	5219
7	C–H def. + O–H str.	Semi-crystalline/crystalline cellulose	5464

GmBH) and Unscrambler® X (CAMO Software AS) software packages were used for spectra post-processing and data evaluation.

## Results and discussion

The overview of material flow representing manufacturing of design-for-purpose packaging products is presented in Fig. 2. The transition from open loop to closed loop systems is important in order to assure sustainable use of resources and the economic viability of modern bio-based industries. Production of packaging containers proposed here allows valorisation and utilization of waste generated during diverse industrial processes. This approach is in line with the industrial symbiosis concept, where studies regarding

flow of materials between industries leads to creation of opportunities to use “waste” from one industry as a raw material for another (Cheshire 2016). In this case fit-for-purpose paper packaging containers are manufactured by combining resources generated by pulp and paper with milling industries. Increasing the recycling and reuse rates of virgin wood fibres leads to increased availability of this resource for other use. However, any recycling round requires a certain amount of virgin paper input, usually from 20% to 95% (Villanueva and Wenzel 2007). Incorporation of cereal bran into packaging products allows minimizing virgin wood fibre inputs while maintaining (or improving) the required properties. The set of expected product characteristics can be optimized by adjusting proportion of recycled fibres, virgin fibres and bran (Fig. 2). Moreover, it solves a costly disposal



**Fig. 2** Scheme of materials flow for manufacturing materials of fit-for-purpose packaging containers

problem from industrial mills by providing a solution for cereal processing by-products use. Finally, control of the biodegradability rate of manufactured products allows predicting their use phase performance and duration of deterioration.

#### Effect of the bran additives on the properties of investigated paper products before degradation

The effect of additives on the chemical composition of manufactured paper products is summarized in Table 3. The effect was minor, but for all analysed components the differences between means were statistically significant (ANOVA,  $p < 0.05$ ). The most noticeable differences are in cellulose content (88.6–91.2%). Cellulose content in control samples was highest and with increased amount of bran (both types) diminished. It was a direct effect of dilution, as the amount of cellulose is low in bran when compare to the pulp and paper. The cellulose content in bran as reported in literature ranges between 5 and 13% (Kamal-Eldin et al. 2009; Chalamacharla et al. 2018). In fact, such a high variation in composition within both wheat and rye brans is influenced by the cereal species, provenance, batch and milling techniques applied for flour production. The chemical

composition of brans used in this study was researched by Modzelewska and Adamska (2006) and is summarized in Table 4.

The differences in extractives content augmented with the bran share increase.

It is an additional dilution effect of mixing components present in waste paper and bran. The bran itself contains several solvable substances that are contributing to the overall content of extractives. These, beside of cellulose include ash, dietary fibre, proteins, starch and diverse phytochemicals, among the others (Onipe et al. 2015). The relatively high amount of starch in cereal bran (estimated to be 15.8–18.9% of the dry mass) simplifies the coupling of cellulose fibres and paper forming as well as aids in filling micro-pores. However, as previously reported by the authors, the presence of starch in paper products may advance the degradation rate as it is a favourable breeding ground for bacteria and microfungi (Sandak et al. 2011).

NIR analysis of investigated papers with addition of the rye and wheat brans did not reveal any noteworthy differences between spectra due to presence of bran fillers (Sandak et al. 2011). Only slight variations were noticed for CH, CH<sub>2</sub> and OH functional groups assigned to cellulose and holocellulose.

**Table 3** Chemical composition of papers (% of dry mass) estimated before and after degradation in agricultural soil

Paper type	Before degradation					After degradation in agricultural soil				
	Cellulose*	Extractives			PD	Cellulose*	Extractives			PD
		H <sub>2</sub> O*	1% NaOH*	1% H <sub>2</sub> SO <sub>4</sub> *			H <sub>2</sub> O*	1% NaOH*	1% H <sub>2</sub> SO <sub>4</sub> *	
SD	0.1	0.1	0.1	0.1	–	0.1	0.1	0.1	0.1	–
WP	91.2 <sup>d</sup>	2.4 <sup>a</sup>	15.1 <sup>a</sup>	10.2 <sup>a</sup>	1102	83.5 <sup>d</sup>	2.8 <sup>a</sup>	16.0 <sup>b</sup>	13.8 <sup>b</sup>	949
WP3W	90.0 <sup>c</sup>	2.9 <sup>cd</sup>	15.5 <sup>b</sup>	10.5 <sup>b</sup>	1100	82.0 <sup>c</sup>	3.3 <sup>bc</sup>	15.8 <sup>ab</sup>	13.6 <sup>ab</sup>	954
WP5W	89.6 <sup>b</sup>	3.1 <sup>d</sup>	16.7 <sup>d</sup>	12.7 <sup>d</sup>	1097	76.9 <sup>b</sup>	3.5 <sup>c</sup>	15.7 <sup>a</sup>	13.5 <sup>a</sup>	950
WP3R	90.0 <sup>c</sup>	2.5 <sup>ab</sup>	16.5 <sup>cd</sup>	11.5 <sup>c</sup>	1101	77.1 <sup>b</sup>	3.2 <sup>b</sup>	15.9 <sup>ab</sup>	13.4 <sup>a</sup>	955
WP5R	88.6 <sup>a</sup>	2.7 <sup>bc</sup>	16.3 <sup>c</sup>	12.5 <sup>d</sup>	1097	76.2 <sup>a</sup>	3.4 <sup>bc</sup>	15.7 <sup>a</sup>	13.5 <sup>a</sup>	954

\*Differences significant at  $p < 0.05$ , letters in superscript correspond to the statistically similar groups determined with Tukey post hoc test

SD, maximum standard deviation of results; PD, polymerization degree

**Table 4** Chemical composition (% of dry mass) of rye and wheat bran

Bran type	Cellulose	Holocellulose	Starch	Lignin	Hot water extractives	Cold water extractives	Ash
Wheat	12.7	43.1	18.9	10.4	25.8	25.1	3.7
Rye	10.5	20.8	15.8	9.3	44.5	26.6	3.5



**Table 5** Selected properties of papers with added bran

Paper type	Breaking length* (km)	Extensibility* (%)	Tearing resistance* (mN)	Burst test* (kPa)	Air permeability* (s)	Absorbency* (g/m <sup>2</sup> )
WP	2.9 <sup>a</sup>	0.97 <sup>b</sup>	980 <sup>b</sup>	567 <sup>a</sup>	4.5 <sup>a</sup>	166 <sup>b</sup>
WP3W	3.4 <sup>b</sup>	0.87 <sup>a</sup>	879 <sup>a</sup>	678 <sup>b</sup>	4.8 <sup>ab</sup>	184 <sup>c</sup>
WP5W	3.4 <sup>b</sup>	0.93 <sup>b</sup>	880 <sup>a</sup>	689 <sup>b</sup>	5.0 <sup>ab</sup>	190 <sup>c</sup>
WP3R	3.8 <sup>c</sup>	0.97 <sup>b</sup>	960 <sup>b</sup>	683 <sup>b</sup>	6.1 <sup>c</sup>	123 <sup>a</sup>
WP5R	3.9 <sup>c</sup>	1.05 <sup>c</sup>	870 <sup>a</sup>	689 <sup>b</sup>	5.1 <sup>b</sup>	182 <sup>c</sup>

\*Differences significant at  $p < 0.05$ , letters in superscript correspond to the statistically similar groups determined with Tukey post hoc test

The influence of bran additives on selected paper properties is summarized in Table 5. The breaking length of papers with bran additives is about 25% higher than control samples. It can be explained by a positive effect of the filler increasing the number of connections between paper fibres and therefore the degree of bonding (Retulainen and Ebeling 1993). In this case the bran particles take part in the hydrogen bonds promoting consolidation of the paper structure. It was also noticed that the effect of mixing fibres with bran additions was not following the linear rule of mixture (Karlsson 2007). The breaking length increase was not significantly higher in 5% bran content compare to 3%. The breaking length was highest for paper products with rye bran.

The addition of bran seems to have rather casual influence on the paper extensibility, even if increased share of bran slightly augmented extensibility value. Conversely, tearing resistance diminished with increase of the bran content. It was expected as tearing is usually inversely correlated with the tensile strength and breaking length (Caufield and Gunderson 1988). The reduction of tearing can be explained by the fact that addition of bran particles affects fibre–fibre

bonding promoting pulling up of fibres out the network (Yu 2001).

The burst resistance index, frequently used to determine the quality of paper, does not depend on the kind, but rather on the amount of filler introduced. It is lowest for the paper without additives, and slightly increases with the added bran content. The trend corresponds to that expected as the burst resistance is as well correlated to the tensile strength (Caufield and Gunderson 1988).

Air permeability and absorbance increased in papers with additives. Both properties might be desirable when increased barrier properties against water are required. In the case of waste paper, adding bran leads to early disassembly of the structure of the paper while exposed to degradation; consequently, affecting the air permeability. This peculiarity has a beneficial effect for paper pots, where high air permeability improves the natural ventilation of the root system and stimulates plant growth (Nambuthiri et al. 2015; Akelah 2013).

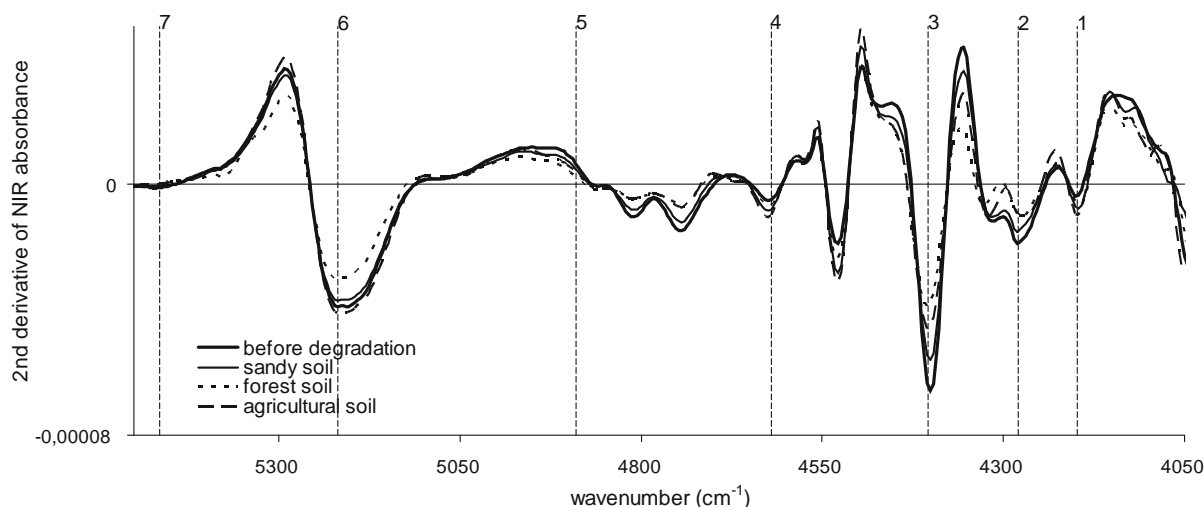
The results of mechanical tests for the compression strength of paper pots before degradation are presented in Table 6. The lowest crushing strength was

**Table 6** Results of the crushing strength of paper pots (kPa)

Pot type	Crushing strength* (kPa)	SD (kPa)	The crushing strength after 3 compression cycles* (kPa)	SD (kPa)
WP	414 <sup>c</sup>	12	388 <sup>d</sup>	11
WP3W	359 <sup>d</sup>	15	320 <sup>c</sup>	6
WP5W	339 <sup>c</sup>	5	300 <sup>a</sup>	7
WP3R	330 <sup>b</sup>	8	299 <sup>a</sup>	6
WP5R	357 <sup>d</sup>	14	315 <sup>b</sup>	6
Commercial pots	291 <sup>a</sup>	9	0 <sup>**</sup>	–

\*Differences significant at  $p < 0.05$ , letters in superscript correspond to the statistically similar groups determined with Tukey post hoc test

\*\*Experimental samples were damaged already after first compression cycle



**Fig. 3** Effect of soil type on the biodegradation of the recycled paper with addition of 5% wheat bran for 8 weeks

observed for commercial pots. Pots without additives (WP) possessed the highest mechanical resistance among all laboratory-manufactured products and any bran addition lowered the crushing strength. It was also found that paper pots with added peat were brittle, as did not retain any strength after first compression cycle. Conversely, pots manufactured from waste paper with added bran were elastic, maintaining integrity after several cycles.

#### Changes to paper due to biodegradation in soil

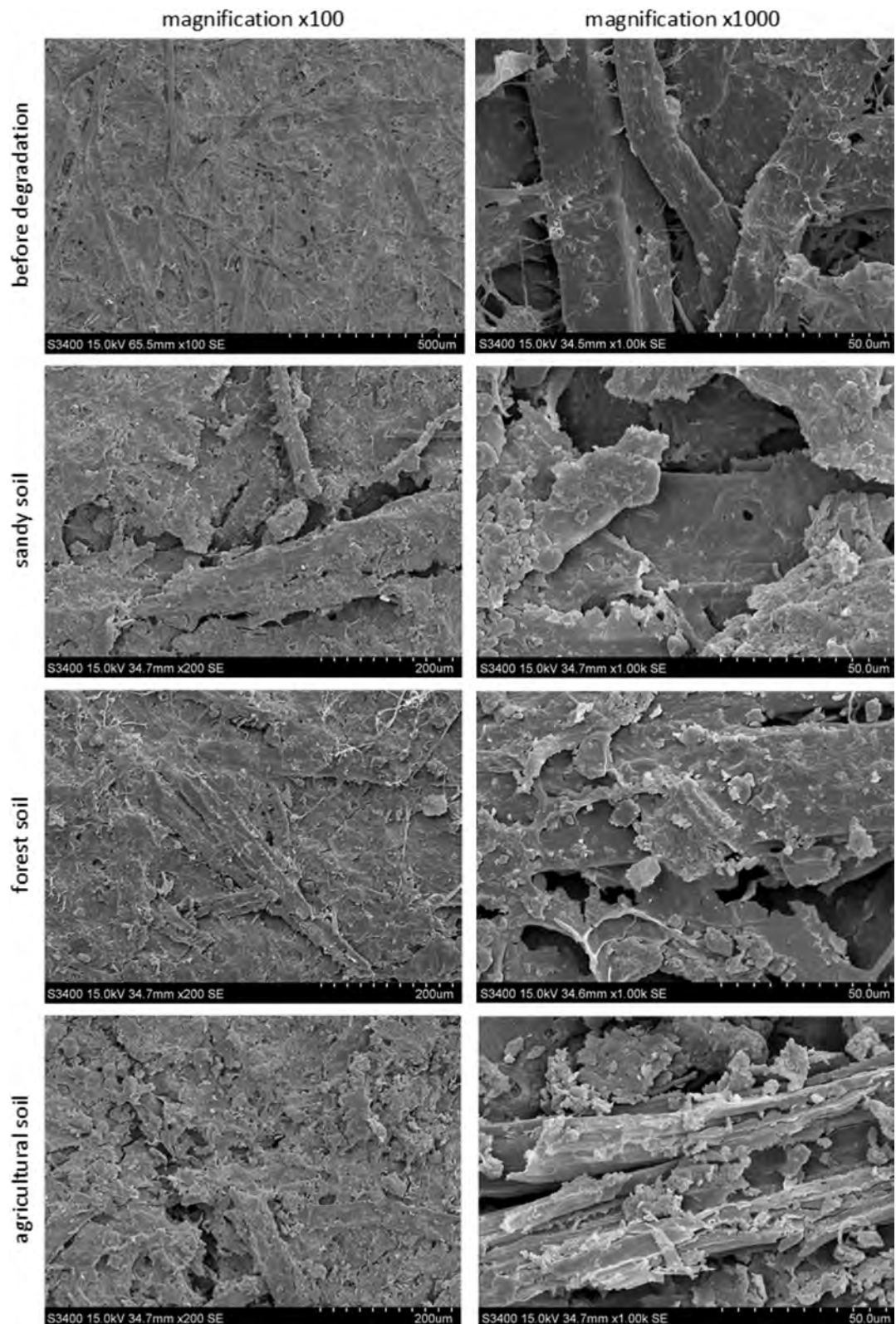
Degradation processes take place in the natural environment constantly and on a large scale (Pagga 1999). Biodegradation in soil is an important end-of-life option for bio-based materials used in agricultural applications. The rate of degradation can vary significantly, depending not only on the molecular structure of the material, but also on soil characteristics and conditions (temperature, water and oxygen availability which influence microbial activity) (Briassoulis et al. 2014). Biodegradation occurs in two steps. First, the polymers are fragmented into lower molecular mass by means of abiotic reactions (oxidation, photodegradation or hydrolysis) or biotic reactions (degradations by microorganisms). Then the polymer fragments are assimilated and mineralized by microorganisms (Vroman and Tighzert 2009).

The effect of soil type on the biodegradation of investigated papers was analysed by means of NIR spectroscopy. Figure 3 presents an example of

degradation progress, where the evolution of the spectra acquired after degrading sample WP5W (waste paper with addition of 5% wheat bran) in various soils is presented. All peaks mentioned in Table 2, with exception of region  $5464\text{ cm}^{-1}$  (7) were affected by the degradation process. However, the spectra of paper placed in a sandy soil seems to be most similar to the control samples. It demonstrates that the sandy soil containing the lowest organic content and persistent low humidity has the lowest impact on the speed of degradation, as in Mostafa et al. (2010). In contrast, the agricultural and forest soil accelerated the degradation speed.

Analyses of the chemical composition of the papers after degradation in agricultural soil for 8 weeks are summarized in Table 3. Cellulose changed considerably after degradation, including quality and quantity alterations, as observed also by Shogren (1999). The amount of cellulose decreased in all investigated cases, where major changes (over 12%) were observed for paper with rye bran and with 5% of wheat bran. Conversely, the cellulose polymerization degree PD drop was slightly higher for papers without any bran additives. The content of extractive components was higher after biodegradation, especially in the case of extraction in 1%  $\text{H}_2\text{SO}_4$ . It can be explained as a result of constitutive polymers degradation due to hydrolysis and biotic factors (Witkowska et al. 1989).

Microscopic analysis allows the assessment of changes in the micro structure of cellulose fibres as results of the degradation. Selected SEM microscopic



**Fig. 4** Microscopic pictures of the paper WP3W surface before and after biodegradation in sandy, forest and agricultural soils

images of the WP3W paper samples (waste paper with addition of 3% wheat bran) before and after degradation are shown in Fig. 4. The apparent presence of both bacteria and fungi are noticed on the degraded paper surface. It was particularly evident in the outer layer, where fungal hyphae and spores are clearly visible. Swelling of the cellulose fibres caused by breaking bonds stimulated the microorganism's growth and their further penetration toward paper bulk. It was especially noticeable in samples collected from agricultural soil, where the water reservoir was maintained at high level, and where degradation was more advanced.

The mechanical properties of paper were measured on the experimental samples before and after degradation. Table 7 presents the progression of the breaking length before testing, and after 2, 4, and 8 weeks of soil exposure for all investigated papers. The results obtained from samples before exposure confirm the positive impact of fillers on the mechanical resistance of paper products and corresponds to trends reported by other researchers (Nechita et al. 2010; Gonzalo et al. 2017). None of the samples were suitable for mechanical testing after 8 weeks of exposure due to excessive degradation. The highest degradation kinetics, as related to the breaking length loss, were observed on samples degraded in the agricultural soil. Mechanical properties had dropped significantly already after two weeks of exposure. According to Sridach et al. (2007) 50–80% of tensile strength is lost during the first week of burial process depending on the type of paper product and its composition. There are several thousands of paper types produced nowadays by the pulp and paper industries. The paper products differs due to composition, formula, additives, binders, fillers, retention agents, among the

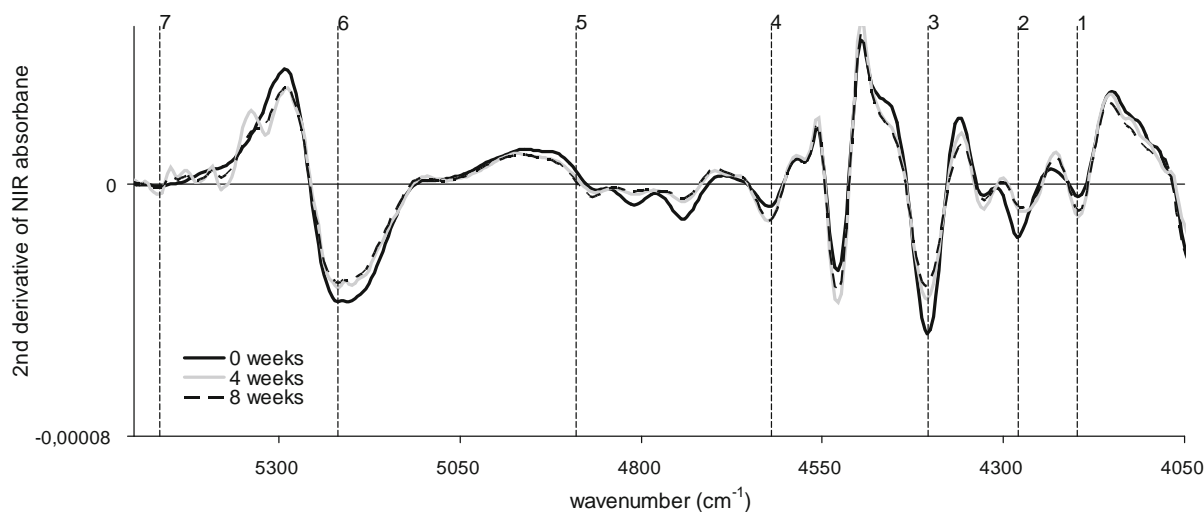
others. However, the highest impact has a variation within cellulose mass used for paper making that is produced with variety of pulping, bleaching, sizing, strengthening, drying and/or coating processes. The raw resource used in this research was of recycled paper origin. The composition of fibers was therefore highly anisotropic as well as each resource batch may be different than another. In addition, printing residuals with other impurities introduce important discrepancy to the paper products derived. It affects also the degradation processes of papers when exposed to soil, making universal determination of the detailed degradation mechanism rather difficult.

The effect of degradation duration on the NIR spectra of sample WP5W (waste paper with addition of 5% wheat bran) is shown in Fig. 5. Curves for most spectra bands differ from the control state (not exposed for degradation) after exposure for 4 weeks. Spectra are similar at 4 and 8 weeks exposure. This implies that partial decomposition of the examined paper products in forest soils had occurred by week four.

The chemical decomposition of different papers can be compared with principal components analysis (PCA) derived from NIR spectra. Figure 6 presents a PCA plot for three series of spectra corresponding to samples at different degradation stages of the tested papers in forest soil. Samples before degradation are grouped together in relatively small cluster. The highest dispersion of spectra was observed at 4 weeks' exposure, while tighter clustering was observed again after 8 weeks. The high degree of scatter observed at 4 weeks exposure, together with minor cluster overlapping signifies a high heterogeneity within all tested papers as well as varied degradation kinetics, being dependent on the paper type.

**Table 7** Breaking length (km) of investigated paper before and after soil degradation

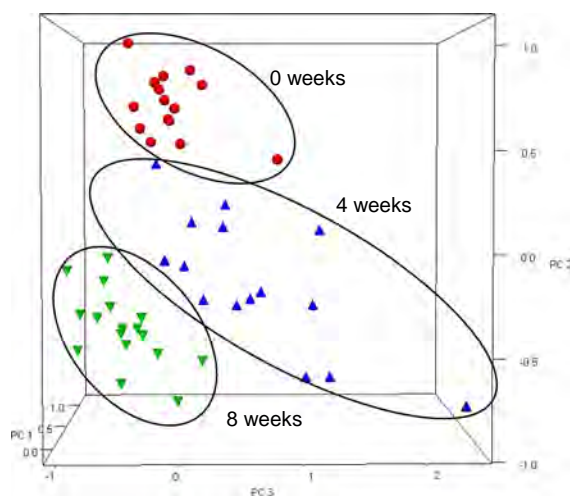
Soil type	Before	After								
		Forest			Sand			Agricultural		
Degradation time	0	2	4	8	2	4	8	2	4	8
WP	2.85	1.37	0.38	0	1.59	0	0	1.00	0.12	0
WP3W	3.38	1.37	0.40	0	1.50	0.38	0	0.60	0.45	0
WP5W	3.40	1.39	0.72	0	1.56	0.64	0	0.78	0.42	0
WP3R	3.81	1.42	0.62	0	1.62	0.50	0	0.77	0.30	0
WP5R	3.88	1.45	0.85	0	1.69	0.80	0	0.83	0.62	0



**Fig. 5** Effect of time on the biodegradation of the recycled paper with addition of 5% wheat bran in the forest soil

The vulnerability of the paper to degradation in soil was assessed by comparing the expected time to complete paper degradation. These degradation ratings for both sheets and pots, are summarized in Fig. 7 for all examined samples. Values were determined by visual estimation according to expert persons as the number of days until a terminal state would be reached. Disintegration of the paper sheet or decomposition > 75% of the paper pot was considered as a terminal state.

The lowest rate of degradation is expected when paper is exposed to sandy soil, where the degradation process is estimated to take between 60 and 70 days (depending on paper composition) (Fig. 7a). A higher degree susceptibility to degradation was observed for samples exposed to the forest soil, regardless of paper type; it was estimated that complete biodegradation occurred in 40–50 days for all paper types in forest soil. Agricultural soil was considered to be the most aggressive because complete paper destruction was expected after only 30 days (compared to approximately 60 days for sandy soil). It corresponds to the previous studies where Tumer et al. (2013) reported that changes related to the decomposition were most intense in organic soil, when compared to sandy soil. The presence of fillers reduced life of the paper products, despite products with 5% bran filler being observed to be more resistant to biodegradation than those with 3% bran filler.

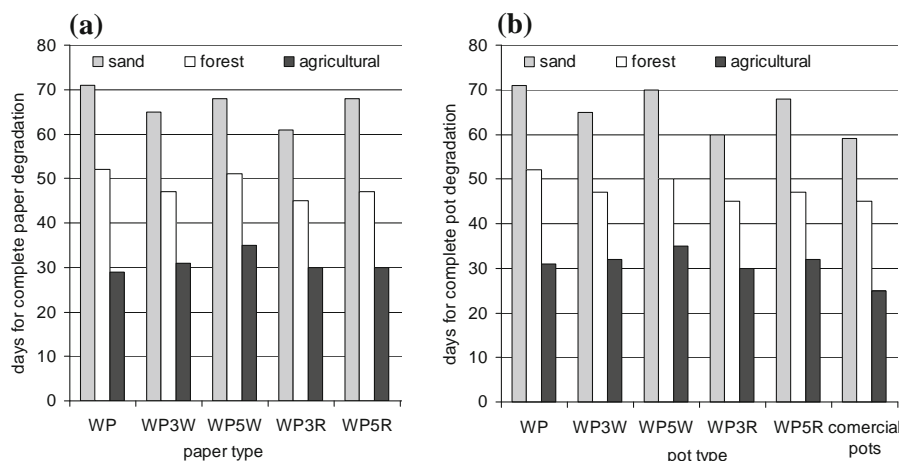


**Fig. 6** PCA analysis of NIR spectra of paper before degradation tests and decomposed in forest soil for 4 and 8 weeks. *Note* spectral range: 11,000–4150  $\text{cm}^{-1}$ , pre-processing: 2nd derivative + vector normalization

The estimated biodegradation rates of pots were similar to those of paper sheets (Fig. 7b). Commercial pots were found to decompose more rapidly than the products manufactured in the laboratory. This was due to the relatively large share of additives that increase water absorption and hasten decomposition. The laboratory-made pots with rye bran filler decomposed slightly faster than other samples. Therefore, it might be possible to adjust the degradation rate of waste paper by adding specific fillers and to deliver products most suitable for specific applications.



**Fig. 7** Visually estimated degradation rates for paper sheets (a) and pots (b), when exposed to different soils



## Conclusions

The cereal bran used in this experiment was characterized by a relatively high starch content. Starch promotes the binding of natural fibres during paper manufacturing and consequently improves the paper strength. Mechanical properties are particularly important in the production and utilization of paper pots manufactured from recycled fibres. The addition of cereal bran improved the mechanical properties of paper products tested in this study; paper pots with bran fillers were less susceptible to mechanical damage, when compare with commercial products. The biodegradation rate of pots changes with the quantity of added bran. Biodegradation in these pots is generally slower than in commercial pots containing peat, but faster than in pots without fillers.

It was concluded that the extent of decomposition does not extensively depend on the type of filling (rye or wheat cereal bran), but rather on the quantity of filler added, exposure time and soil type. Depending on the soil type, water holding capacity and pH, soil may stimulate or inhibit the growth of microorganisms responsible for the decomposition of paper products. Paper samples (both sheets and pots) in all tested configurations degraded most rapidly in agricultural and forest soils, while biodegradation proceeded slowly in the sand soil.

Analysis of NIR spectra revealed that the most advanced degradation occurred in agricultural soil. The organic content accelerated the degradation rate within all investigated papers. In contrast, sandy soil, which is low in organic matter, resulted in the lowest degradation rate and inhibited degradation processes.

All tested paper configurations could be suitable for manufacturing plantable bio-containers that will slowly disintegrate during their lifespan. Their use improves both the sustainability and public perception of the investigated products. However, in addition to environmental and economic aspects, the effect of alternative containers on plant growth and quality should be considered. Therefore, our future work will be related to calculation of environmental impact of manufactured paper packaging products. Proper use of the experimental results may help in selection of products with optimal composition for specific applications, including pots used in horticulture or for forest nurseries. Moreover, by proper fillers selection, packaging products with custom degradation rate best suited to certain crop cycle durations and adopted for specific types of soils may be designed.

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## Analysis of cellulose nanocrystals (CNCs) with flow cytometry

Anders STRAND, Lari VÄHÄSALO, Elias RETULAINEN and Anna SUNDBERG

Cellulose nanocrystals (CNCs) were prepared from kraft pulps of eucalyptus, birch, and softwood. The different kraft pulps were hydrolysed using strong sulfuric acid in order to obtain colloidally stable CNCs. The CNCs were studied using flow cytometry (FCM). The light scattering properties of the CNCs in side direction and forward direction were documented. The use of a selective staining agent in combination with FCM analysis enabled detection of the hydrophobic particle populations within the CNC suspensions. The hydrophobic particles were seen clearly in the CNCs from the eucalyptus and birch kraft pulps, but not in the CNCs from softwood pulp. These particles were shown to be linked with the content of lipophilic extractives, especially sterols, present in the kraft pulps. From this fact, it was concluded that FCM analyses offer useful information about the properties of CNCs in suspension. It was also concluded that the content of lipophilic extractives in different raw materials should be determined and considered prior to CNC production. Pre-extraction steps might be needed in order to produce high-quality CNCs from various raw materials, even from kraft pulps.

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# Analysis of cellulose nanocrystals (CNCs) with flow cytometry

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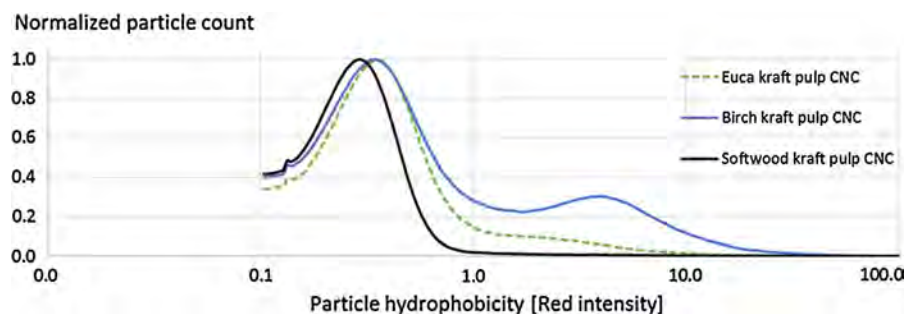
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## Graphical abstract



**Keywords** Cellulose · CNC · Flow cytometry · Light scattering · Colloids · Hydrophobicity · Lipophilic extractives

## Introduction

Cellulose nanocrystals (CNCs) have received a lot of attention in research due to their physical and chemical properties, renewability, sustainability, and usefulness in composite materials (Favier et al. 1995a, b; Ruiz et al. 2000; Habibi et al. 2010; Wang et al. 2012). CNCs are a strong contender for various future applications in composite materials, due to their low cost and abundancy. CNCs are produced by acid hydrolysis of the amorphous and para crystalline regions of cellulose fibers, while the crystalline regions are left intact as rod-like particles (Marchessault et al. 1961; Habibi et al. 2010). The crystallites of CNCs are, therefore, similar to the crystallites of the cellulose fiber raw materials. A wide array of different raw materials have been tested for CNC production and specific hydrolysis and separation protocols have been established for each of these (Beck-Candanedo et al. 2005; Habibi et al. 2010). The cellulose raw material is normally hydrolyzed by addition of strong acid, in combination with controlled reaction conditions, i.e. temperature, agitation, and time (Revol et al. 1994; Dong et al. 1998; Wang et al. 2014).

The length of the cellulose crystals after hydrolysis depends on the leveling-off degree of polymerization (LODP) of the starting material. It has been reported that milder hydrolysis conditions will eventually reach the LODP of the starting material with lower losses of material, but that the reaction times need to be severely

prolonged (Battista 1950; Håkansson and Ahlgren 2005). The LODP for bleached wood pulp has been reported as 140–200 units (Dong et al. 1998; Habibi et al. 2010). The surface charge of the CNCs will be very different depending on the choice of acid; sulfuric acid introduces sulfate half ester groups to the CNC, while e.g. hydrochloric acid does not (Araki et al. 1999; Beck-Candanedo et al. 2005). The introduction of the anionically charged groups on the surface of the CNCs will greatly increase its colloidal stability and even prevent sedimentation. It was reported that initial hydrolysis of the cellulose raw material by hydrochloric acid can be followed up by a separate sulfuric acid treatment to introduce sulfate half ester groups on the cellulose microcrystals (CMCs), and fine-tune the viscosity properties of the suspension (Araki et al. 1999).

CNC particles and suspensions have previously been characterized with a wide array of analytical methods, such as transmission electron microscopy (TEM), atomic force microscopy (AFM), photon correlation spectroscopy (PCS), x-ray diffraction (XRD), conductometric titration, elemental analysis, viscosity measurements, sedimentation measurements (Marchessault et al. 1961; Dong et al. 1998; Beck-Candanedo et al. 2005; Håkansson and Ahlgren 2005; Hirai et al. 2009; Chen et al. 2015). However, additional analysis techniques may still provide useful information, relevant to the ever-growing CNC community.

Flow cytometry (FCM) is a relatively new technique in the field of pulping and papermaking. The technique was adapted from medical science, where it is used mainly for the counting of cells (Shapiro 2003). FCM measures the light scattering intensity of particles in suspension in forward and side direction.

Furthermore, FCM can be used to measure the fluorescence of particles at different wavelengths, which is very useful when combined with addition of selective dyes to the samples. In the field of pulping and papermaking, FCM has so far been used to analyze colloidal wood pitch, bacteria, coated broke, precipitated oxalate, various filler particles, and polyelectrolyte complexes (Vähäsalo et al. 2003; Lindberg et al. 2004; Vähäsalo and Holmbom 2005; Strand et al. 2013; Häärä et al. 2014; Strand et al. 2018). With FCM techniques, it is possible to analyze particles in suspension directly without complicated pre-treatments. The strengths of the FCM technique are short analysis times, as well as insight into the behavior and interactions of particle populations in aqueous suspensions.

The aim of this study was to obtain new insights about CNCs in suspension by FCM analyses. Raw materials that are available in large quantities worldwide, i.e. different kraft pulps, were chosen for this CNC study. An additional aim was to determine if eucalyptus, birch, or softwood kraft pulp was most suited for continued CNC production.

## Materials and methods

### Materials

ECF-bleached, once-dried softwood kraft pulp was obtained from a Finnish pulp mill. The softwood kraft pulp was prepared from a mixture of Nordic pine and spruce. ECF-bleached, once-dried birch kraft pulp was obtained from a Finnish pulp mill. ECF-bleached, once-dried eucalyptus kraft pulp was obtained from a pulp mill in Uruguay. Concentrated, analytical reagent grade sulfuric acid was obtained from Fisher Scientific UK. The  $\text{H}_2\text{SO}_4$  was diluted with distilled water to 64 wt % and was allowed to cool down prior to use. Nile red was obtained from Tamro (Vantaa, Finland). The nile red was directly dissolved in methanol to a concentration of 10 ppm. A commercially prepared and available cationic starch (Raisabond 15) was obtained from Chemigate, Finland as a pre-boiled slurry. Sodium carboxymethyl cellulose,  $M_w$  250, degree of substitution 0.9, was obtained from Acros Organics. Polyelectrolyte complexes (PECs) were prepared from the cationic starch and CMCs at a polycation to polyanion ratio of 5.5:1 according to a

previously published procedure (Strand et al. 2018). The lipophilic extractives from thermomechanical pulp from Norway spruce were extracted with hexane. Colloidal wood pitch emulsions were prepared from the hexane extracts according to a previously published procedure (Sundberg et al. 1996b).

### Methods

#### *Analysis of carbohydrates*

Analysis of carbohydrates in the dry kraft pulps was performed through methanolysis, silylation, and gas chromatographic analysis (GC) (Sundberg et al. 1996a). Resorcinol was added as internal standard for quantification. The analysis of sugar units was conducted with a long column GC (HP-1, 25 m  $\times$  0.20 mm) with split injection, equipped with a flame ionization detector (FID). The methodology gave information mainly about the amount of non-crystalline carbohydrates in the fiber samples. The determined sugar unit contents of the different kraft pulps are presented in Table 1.

#### *Preparation of cellulose nanocrystals (CNCs)*

CNCs were prepared according to previously published methodology, with some modifications, and the concentration of acid during hydrolysis was chosen accordingly (Revol et al. 1994; Dong et al. 1998; Beck-Candanedo et al. 2005). Grinding of the different kraft pulps was performed with a laboratory-sized mill (IKA MF 10). The kraft pulp powders were freeze-dried for a few days. 5 o.d. g of the freeze-dried pulp was weighed into a glass bottle, and 250 mL of 64 wt %  $\text{H}_2\text{SO}_4$  was added to the pulp. The pulp suspension was agitated by magnetic stirring for 30 min at room temperature in order to homogenize the mixture. The temperature of the suspension did not increase, since the pulp was completely dry when the  $\text{H}_2\text{SO}_4$  was introduced. The glass bottle was sealed and placed in a 45 °C water bath for 25 min. The glass bottle was shaken manually continuously during this time, in order to further homogenize the mixture. The hydrolysis was stopped after 25 min by diluting the suspension 10  $\times$  with distilled water.

Secondary aggregation of particles took place slowly in the diluted suspension. The suspension was allowed to sediment over night at room temperature.

**Table 1** The determined amount of different sugar units in the three different kraft pulps after acid methanolysis (uncertainty  $\pm 5\%$ )

Polysaccharide film	Eucalyptus kraft (mg/g)	Birch kraft (mg/g)	Softwood kraft (mg/g)
Arabinose	0.2	0.6	7.8
Galactose	5.1	3.0	8.3
Glucose	102.5	94.0	120.2
Mannose	1.2	8.2	47.2
Rhamnose	0.6	0.3	0.1
Xylose	135.1	173.9	68.6
Galacturonic acid	0.2	0.3	1.1
Glucuronic acid	0.8	0.8	0.5
4-O-Me-glucuronic acid	7.6	7.4	2.5
Sum	253.2	288.6	256.3

The diluted suspension was concentrated by centrifugation at 3400 g for 15 min. The supernatants were removed using a suction flask. The bottom phases were collected, diluted with distilled water, and again centrifuged at 3400 g for 30 min. The supernatants were again removed in order to concentrate the suspension. The collected cellulose fraction was diluted to 1 L with distilled water for washing. The suspension was again centrifuged at 3400 g for 30 min, and the supernatant was removed. The washing procedure was repeated one additional time. The collected cellulose fraction was dialyzed against distilled water for 4–5 days in a dialysis membrane with 12–14 kDa cut-off (Medicell International, UK). The dialysis water was replaced daily until the pH of the dialysis water remained unchanged over the course of 1 day.

After dialysis, the CNC suspension was homogenized at 20,000 rpm using a Polytron PT3000 (GWB) for 5 min, and with ultrasonic impulses for  $2 \times 10$  min. The CNC suspension was allowed to sediment, in order let large residual fiber fragments sink to the bottom of the flask. Only the non-sedimenting particles in the supernatant was used for further analyses.

#### Flow cytometry (FCM)

Flow cytometry (FCM) was used to analyze the CNC suspensions, using a Partec CyFlow Blue apparatus, equipped with a blue Argon laser (488 nm). The light scattered by the particles were recorded in forward direction (FSC) and side direction (SSC,  $90^\circ$ ). The apparatus was also equipped with three different fluorescence channels, which recorded the scattering

intensity in the green (512–542 nm), orange (575–605 nm), and red (615–645 nm) spectra. Filtered distilled water was used as sheath fluid in the analyses. Nile red (Tamro, Vantaa, Finland) was used as a fluorescent dye to stain the particles prior to FCM analysis, as in previously published studies (Vähäsalo et al. 2003; Vähäsalo and Holmbom 2005; Strand et al. 2013, 2018). Nile red is an environment-sensitive fluorophore that exhibits a blue-shift proportional to the hydrophobicity of its environment (Greenspan and Fowler 1985). It was expected that the added nile red mainly stained the surface of the CNCs, since the solubility of nile red in water is negligible.

The CNC suspensions were diluted  $4 \times$  or  $15 \times$  with filtered distilled water prior to FCM analysis, depending on the concentration of detectable particles in the sample. 20  $\mu$ L of methanol solution of nile red (10 ppm) was added to stain the CNCs, and the fluorescence intensity in the red spectrum was used as an indicator of particle hydrophobicity. FCM analysis was used to count the number of particles in the sample, and group the particles together as populations based on their recorded light scattering properties. Particle populations were gated using Partec FloMax software, in order to calculate the average scattering of particles in side and forward directions, as well as particle hydrophobicity (Vähäsalo et al. 2003).

#### Analysis of extractives

Grinding of the different kraft pulps was performed with a laboratory-sized mill (IKA MF 10). The kraft pulp powders were freeze-dried for a few days. Roughly 4 o.d. g of the different kraft pulp powders were packed into stainless steel extraction cells

equipped with glass microfiber filters. Extractions were performed with a Dionex Corp. accelerated solvent extractor (ASE200) using a mixture of acetone:water:acetic acid (94:5:1 v/v). The samples were extracted consecutively three times for 5 min, at 100 °C and 13.8 MPa, and the extract from each extraction cycle was combined into one. The total volume of the extract was adjusted to exactly 50 mL at room temperature.

The concentration of extractives was determined by gas chromatography using both long and short column techniques (Örså and Holmbom 1994). Exactly 10 mL of the extracts were pipetted into test tubes. Exactly 2 mL of internal standard, containing 0.02 mg/mL of heneicosanoic acid (21:0), betulinol or cholesterol, cholesteryl heptadecanoate (Ch17), and 1,3-dipalmitoyl-2-oleyl glycerol (triglyceride standard) in MTBE, was added to the samples. The samples were dried at 40 °C under a stream of nitrogen gas. The samples were dried to complete dryness in a vacuum oven (40 °C). The dry samples were silylated by addition of a 1:4:1 mixture of pyridine, N,O-bis(trimethylsilyl) trifluoroacetamide (BSTFA), and trimethylchlorosilane (TMCS). The silylation reaction took place in a 70 °C oven for 45 min.

Analysis of different groups of extractives was conducted on a short column with a PerkinElmer Clarus 500 GC. The column was a HP-1, 7 m × 0.53 mm, with film thickness of 0.15 mm. Hydrogen was used as carrier gas (see e.g. Strand et al. 2011). The injection volume was 0.5 µL, on-column, and the detector used was a flame ionization detector (FID).

Analysis of individual components was conducted on a long column with a PerkinElmer AutosystemXL GC. The used column was a HP-1, 25 m, 0.2 mm, with film thickness of 0.11 mm (see e.g. Strand et al. 2011). Hydrogen was used as carrier gas. The injection volume was 1 µL, split 24:1, and the detector used was an FID.

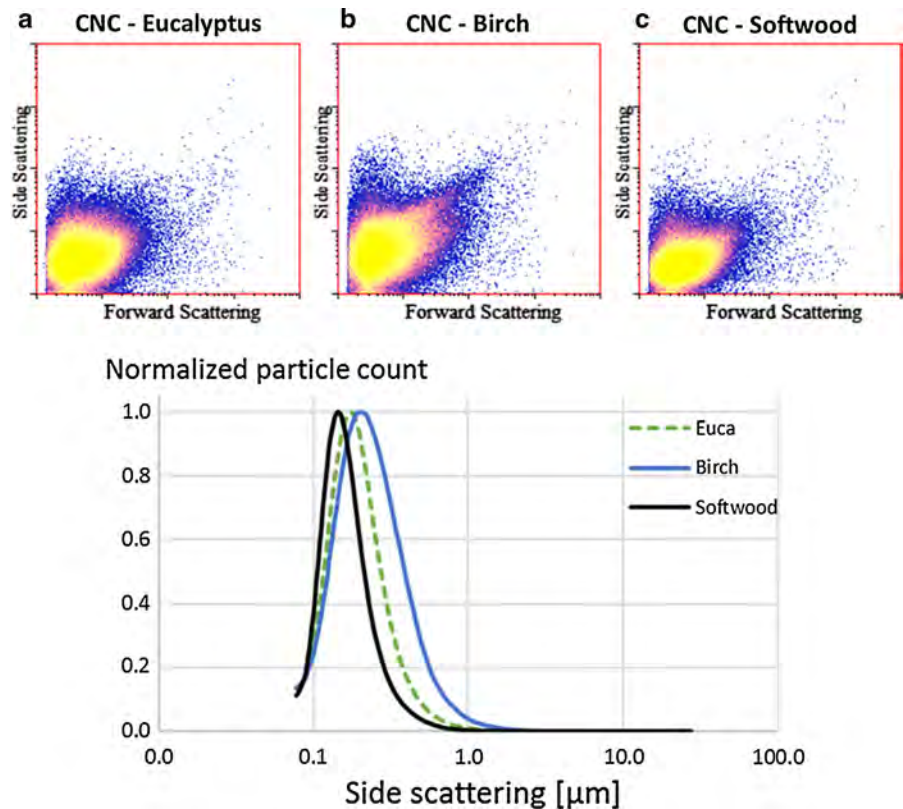
## Results and discussion

Previously, the weight loss upon hydrolysis prior to level-off degree of polymerization (LODP) has been used to assess the amount of disordered material in pulps of different origins (Håkansson and Ahlgren 2005). Another method to determine the amount of

disordered material in the starting pulps was applied in this study, i.e. acid methanolysis. It is known that the acid methanolysis procedure cannot hydrolyze crystalline cellulose, and it is commonly used to analyze the hemicelluloses and pectins in various lignocellulosic materials (Sundberg et al. 1996a). By performing acid methanolysis on these three kraft pulps, it was possible to get an approximation of the amount of amorphous and para-crystalline carbohydrate regions in the raw materials. The dominating sugar units detected in eucalyptus and birch kraft pulp were xylose and glucose (Table 1). The dominating sugar units detected in the softwood kraft pulp were glucose, xylose and mannose. A large part of the glucose was thought to originate from amorphous cellulose regions. By performing acid methanolysis on the kraft pulps, the content of easily hydrolysable carbohydrates was determined as roughly 25–29% by weight depending on raw material.

The reaction conditions used in this study were not able to cleave all fiber residue into CNCs, and a significant fraction of partly hydrolyzed fiber fragments remained in the suspension. It has been reported that insufficient hydrolysis will result in larger particles with lower surface charge (Dong et al. 1998; Beck-Candanedo et al. 2005). This type of large particles have previously been described as partially hydrolyzed cellulosic residue (Chen et al. 2015). The CNC suspensions were allowed to sediment prior to flow cytometry (FCM) analyses, in order to circumvent the presence of large particles. Only the non-sedimenting particles in the supernatants were used as representative CNC samples for the different kraft pulps. Flow cytometry of the CNCs prepared from the three different kraft pulps revealed clear particle populations (Fig. 1). The forward scattering (FSC) intensities of the CNCs were plotted against their side scattering (SSC) as logarithmic density plots. The graphs revealed single particle population for all of the three different CNCs as a function of their light-scattering ability. The shapes of the CNC populations were slightly different depending on the raw material used, which indicated that the choice of raw material influenced the resulting CNCs. Previously, FCM measurements of polyelectrolyte complexes (PECs) highlighted that the FSC and SSC offers quite different information about particles in suspension (Strand et al. 2018). It was theorized that the SSC gives information about the amount of dense and light scattering surfaces

**Fig. 1** a–c Logarithmic density plots of light scattering in the forward direction (FSC) versus the light scattering in the side direction (SSC) of CNCs made from the different kraft pulps. The size estimation ( $\mu\text{m}$ ) of the particles from side scattering are also plotted as normalized curves



in a particle, while the FSC offers information about the transparency of a particle, as well as the complexity of its external or internal texture. However, both the SSC and FSC are connected to particle size. Information about the structural density of particles in suspension can be obtained by comparing the size estimation from FSC and SSC (Strand et al. 2018). The SSC of particles, in combination with calibration curves, has previously been shown to satisfactorily estimate the particle size of a wide array of particles in suspension (Vähäsalo et al. 2003). The average values

of the different particle populations were calculated and documented into Table 2.

The determined average SSC of the different CNCs showed that birch kraft pulp resulted in slightly larger particles than eucalyptus kraft pulp, and significantly larger particles than the softwood kraft pulp. It is known that different raw materials will result in CNCs of different dimensions, depending on the LODP of the cellulose raw material (Marchessault et al. 1961; Håkansson and Ahlgren 2005). It has previously been reported that treatment of black spruce kraft pulp with

**Table 2** Determined and calculated properties of the three different CNCs by FCM analyses

CNC	Particle count (particles/ $\mu\text{L}$ )	SSC ( $\mu\text{m}$ )	FSC ( $\mu\text{m}$ )	Hydrophobicity (red intensity)	Structural density (FSC/SSC)
Eucalyptus	2963	0.21	0.34	0.75	1.62
Birch	2616	0.27	0.40	1.91	1.48
Softwood	12,113	0.17	0.31	0.39	1.82

SSC particle size calculated from light scattering in side direction, FSC particle size calculated from light scattering in forward direction, FSC/SSC measure of the structural density of particles



65%  $\text{H}_2\text{SO}_4$  gave CNCs with a length of 100–200 nm, and a width of about 5 nm (Revol et al. 1992, 1994). The measured side scattering of the particles by FCM are in the same size range as previously reported values of the lengths of CNCs made from cotton fibers (Dong et al. 1998). It has been reported that the length of CNCs from black spruce sulfite pulp and eucalyptus pulp were 147 nm and 141 nm respectively after treatment with 64%  $\text{H}_2\text{SO}_4$  at 45 °C for 25 min (Beck-Candanedo et al. 2005). The average length of CNCs produced from eucalyptus kraft pulp has been reported as between 130 and 281 nm, depending on the conditions during hydrolysis with  $\text{H}_2\text{SO}_4$  (Chen et al. 2015). It was reported that the length of CNCs produced from microcrystalline cellulose originating from Norway spruce was 200–400 nm, with a width of less than 10 nm (Bondeson et al. 2006). In the current study, the determined SSC values of the different CNCs ranged between 170 and 270 nm. This indicated that the size-estimation from SSC actually represented the length of the CNCs, while the width was far below the detection limit of the FCM apparatus. From the normalized side scattering-curves it was also seen that the size of the CNCs from the birch and eucalyptus kraft pulps resulted in slightly larger particles than the softwood CNCs.

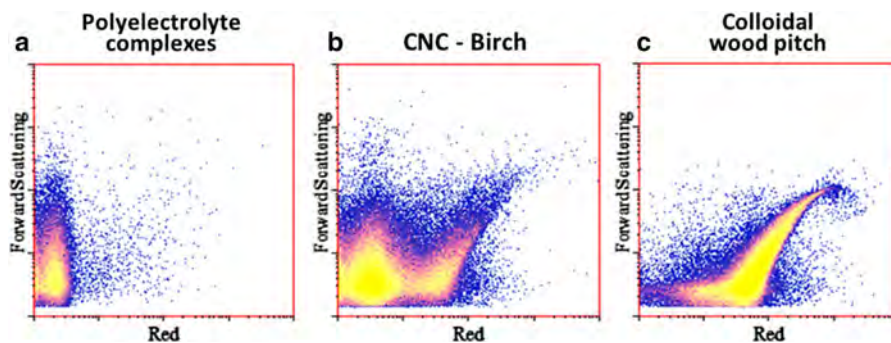
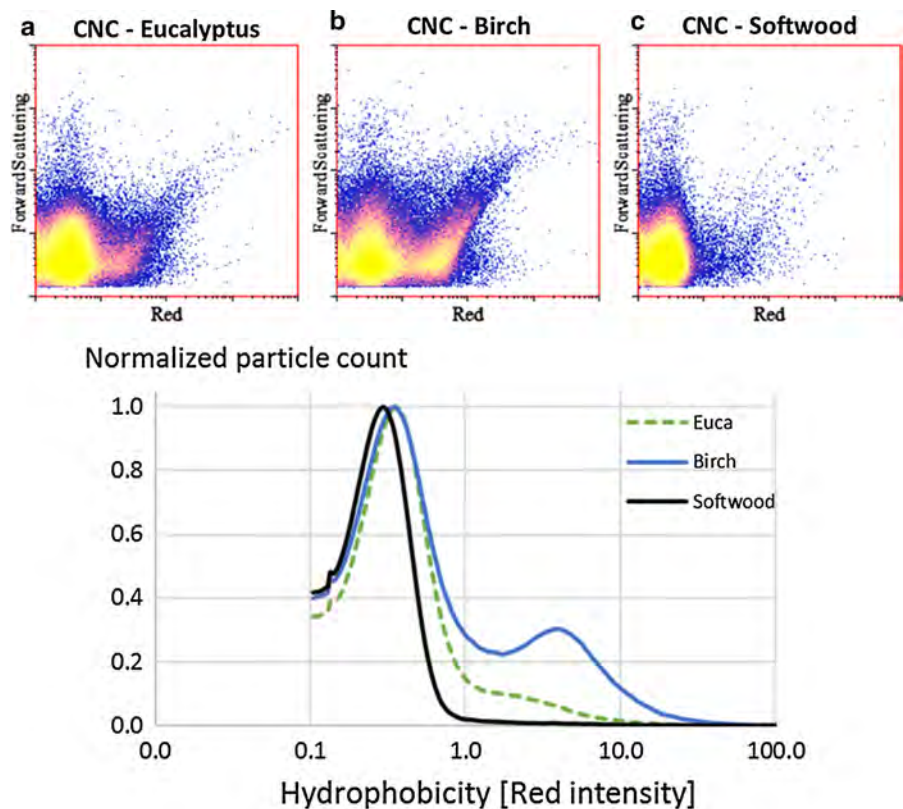
The hydrophobicity of the CNCs were measured by FCM analyses with the added hydrophobic staining agent Nile red. A small volume of Nile red dissolved in methanol was added to the CNCs prior to injection into the FCM apparatus. Nile red is an environment-sensitive fluorophore that exhibits a blue-shift proportional to the hydrophobicity of its environment (Greenspan and Fowler 1985). The fluorescence intensity from Nile red in the red spectrum has previously been used to investigate the hydrophobicity of various particles in combination with FCM analysis (Vähäsalo et al. 2003; Vähäsalo and Holmbom 2005; Strand et al. 2013, Strand et al. 2018). The Nile red is believed to mainly adsorb onto the surfaces of the particles, since its solubility in water is negligible and migration into the CNCs is unlikely (Greenspan and Fowler 1985; Jose and Burgess 2006). The FSC intensities of the CNCs were plotted against their fluorescence intensity in the red spectrum (red) as logarithmic density plots (Fig. 2). Plotting FSC versus particle hydrophobicity revealed that the CNCs in fact consisted of two different particle populations, which was not detectable when only the light-scattering

properties were plotted (Fig. 1). The FCM measurements showed a dominating particle population with low hydrophobicity, and a smaller population of particles with higher hydrophobicity in the eucalyptus CNCs and in the birch CNCs (Fig. 2). However, only traces of the hydrophobic population were seen in the CNCs from softwood.

The fluorescent intensity in the red spectrum of the CNCs prepared from birch kraft pulp was compared with previously encountered particle populations in FCM. As an example of very hydrophilic particles, polyelectrolyte complexes (PECs) from a commercial cationic starch mixed with carboxymethyl cellulose was chosen (Fig. 3). It is known that these PECs are very hydrophilic particles, since they consist of hydrated polysaccharide chains locked in contact by the attraction of oppositely charged groups (Strand et al. 2018). Calculations showed that the average red fluorescence intensity of this PEC population was 0.35. As an example of very hydrophobic particles, colloidal wood pitch droplets in suspension was chosen (Fig. 3). It is known that these particles are hydrophobic, since they consist of lipophilic extractives dispersed in water. The colloidal wood pitch has been reported to consist of a core of triglycerides and steryl esters, with an outer shell of surface active resin and fatty acids (Qin et al. 2003). Calculations showed that the average red fluorescence intensity of the colloidal wood pitch population was 8.33.

When reexamining the two particle populations in the CNCs from birch kraft pulp alongside the PEC population and the colloidal wood pitch population, it is clearly visible that there are traces of these two populations within the CNC sample (Fig. 3). By gating the populations separately, it was possible to calculate the average red fluorescence intensity of the hydrophilic CNC population and the more hydrophobic population. The hydrophilic part of the birch kraft CNC had an average hydrophobicity value quite close to that of hydrated polysaccharide surfaces, i.e. 0.41. The hydrophobic part of the birch kraft CNC had an average hydrophobicity closer to the average hydrophobicity of colloidal wood pitch, i.e. 5.75. The hydrophilic part of the CNC was slightly more hydrophobic than clean hydrated polysaccharide surfaces of the polyelectrolyte complexes (Fig. 3a). The hydrophobic part of the CNC was less hydrophobic than colloidal wood pitch on its own (Fig. 3c). This indicated that the two populations were in fact a

**Fig. 2** a–c Logarithmic density plots of fluorescence intensity in the red spectrum (red) versus the light scattering in the forward direction (FSC) of CNCs made from the different kraft pulps. The hydrophobicity estimation of the particles from their red intensity are also plotted as normalized curves



**Fig. 3** Logarithmic density plots of fluorescence intensity in the red spectrum (red) versus the light scattering in the forward direction (FSC) of **a** polyelectrolyte complexes consisting of

cationic starch and carboxymethyl cellulose, **b** CNCs made from birch kraft pulp, and **c** colloidal wood pitch

mixture of hydrophilic and hydrophobic components. The interactions between various hydrophilic filler particles and hydrophobic colloidal wood pitch has previously been studied in model systems by FCM analyses (Strand et al. 2013). It was shown that hydrophilic and hydrophobic populations in fact interacted with each other, and that they formed hybrid populations with intermediate hydrophobicity

values. It was also shown that the interactions were slightly diminished by steric stabilization when water-soluble hemicelluloses (galactoglucomannans) were introduced as a third component to the system. In the current study, combining the two hydrophobicity values of the sub-populations resulted in an average value of 1.91 for the birch kraft CNCs. Because of these FCM analyses of the CNCs, it was theorized that

the kraft pulps most likely contained lipophilic extractives that had survived the harsh acidic conditions of CNC production. Extraction of the three kraft pulps with an appropriate solvent was required in order to confirm this theory.

The three kraft pulps were extracted with a mixture of acetone, water, and acetic acid. The content of lipophilic wood pitch components in extracts were analyzed according to standard laboratory procedure (Örså and Holmbom 1994). It was seen that all of the kraft pulps contained lipophilic components to some extent (Table 3). The three pulps did not contain noticeable amounts of triglycerides or steryl esters, since these components had been hydrolyzed into fatty acids and sterols by the strong alkaline conditions during the kraft cook (Back 2000). The dominating components in the eucalyptus and birch kraft pulp were fatty acids and sterols, as expected (Ekman and Holmbom 2000). It is known that the water-solubilities of long-chained fatty acids are very limited even at alkaline pH, and can be severely decreased at high conductivity values (Sundberg et al. 2009; MacNeil et al. 2011). This fact makes it very difficult to completely remove fatty acids by alkaline washing, even after kraft pulping. Traces of resin acids were also noticed in the hardwood pulps, which may have been added to aid deresination during washing at the pulp mills. The dominating components in the softwood kraft pulp were fatty acids and resin acids (Table 3). The detectable content of lipophilic extractives in the three kraft pulps were quite low overall, only from 48 to 188 µg/g o.d. pulp. Still, these amounts were significant enough to be detectable by FCM analyses of the final CNCs. It is known that lipophilic extractives are not degraded by acidic conditions, which has previously been seen in analyses of extractives throughout an acidic sulfite pulping line (Sithole et al. 2010a, b). This meant that the fatty acids, resin acids, and sterols observed in the kraft pulps were able to withstand the harsh acidic

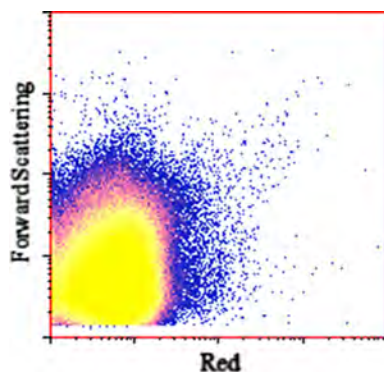
hydrolysis step of CNC production, and remained with the CNCs through the rest of the production steps.

When reexamining the visible hydrophobic particle populations in Fig. 2, it was clear that the highest content of hydrophobic particles was seen in the CNCs made from birch kraft pulp. There were significantly less hydrophobic particles in the eucalyptus CNC, and only a very low amount in the softwood CNC. It was expected that the content of lipophilic particles in the kraft pulps would correlate directly with the observed hydrophobicity of the CNC. However, eucalyptus kraft pulp had the highest content of lipophilic components, while birch kraft pulp contained slightly less (Table 3). In order to understand this, the hydrophobicity of the different groups of extractives need to be taken into account. The pH of the CNC suspensions after dialysis was roughly 5.5. It is known that a fraction of the fatty acids may have been deprotonated, formed soaps, and have been dissolved into the aqueous phase even at this moderate pH (Ström 2000; Sundberg et al. 2009). However, the large and bulky carbon skeletons of sterols, and the lack of carboxylic acid groups, indicate that these are very hydrophobic compounds (Sjöström 1981; Ekman and Holmbom 2000; Nisula 2018). Sterols have also been shown to be quite troublesome in deresination of sulfite pulp, unlike resin and fatty acids (Sithole et al. 2010a). When taking the surface-active nature, and pH-dependent water-solubility of fatty acids into account, it is clear that fatty acids need to be considered significantly less hydrophobic than sterols, at least at pH values higher than 3. The sterols most likely influenced the particle hydrophobicity to a higher extent than fatty acids. A more satisfactory correlation was found between the hydrophobic populations in the CNCs and the sterol content of the three different kraft pulps. The least hydrophobic particles were produced from the raw material that contained the lowest amount of lipophilic components, i.e. the softwood kraft pulp. Some of the birch kraft pulp was

**Table 3** Quantified amounts of extractives found in the acetone:water:acetic acid extracts of the different kraft pulps. The amounts are expressed as µg per o.d. g of pulp (uncertainty ± 5%)

Kraft pulp	Fatty acids and fatty alcohols (µg/g)	Resin acids (µg/g)	Sterols (µg/g)	Other* (µg/g)	Lipophilics (µg/g)
Eucalyptus	134	8	46	22	188
Birch	79	1	56	50	136
Softwood	38	10	0	26	48

*Other\** various identified degradation products. *Lipophilics* the sum of fatty acids, fatty alcohols, resin acids, and sterols



**Fig. 4** A logarithmic density plot of fluorescence intensity in the red spectrum (red) versus the light scattering in the forward direction (FSC) of CNC made from pre-extracted birch kraft pulp

ASE-extracted with acetone:water:acetic acid (94:5:1 v/v) prior to CNC production. After pre-extraction of the birch kraft pulp, no separate hydrophobic population was seen in the CNCs, which indicated that the hydrophobic particles most likely originated from lipophilic extractives in the starting material (Fig. 4).

These results indicate that the extractives content of different raw materials for CNC production needs to be determined and taken into account. Even low quantities of lipophilic extractives will alter the properties of CNC surfaces significantly, and lead to misinterpretation of data. The raw material containing the lowest amount of lipophilic components, especially sterols, is recommended for CNC production, unless a pre-extraction of is performed to remove these components.

## Conclusions

Cellulose nanocrystals (CNCs) were prepared from kraft pulps of eucalyptus, birch, and softwood. The different kraft pulps were hydrolyzed using strong sulfuric acid in order to obtain colloiddally stable CNCs. The CNCs were studied using flow cytometry (FCM). The light scattering properties of the CNCs in side direction and forward direction were documented, as well as the hydrophobicity of the CNCs.

Polysaccharide surfaces, such as cellulose, are known to be very hydrophilic in aqueous media. However, the presence of hydrophobic particles were seen clearly in the CNCs from the eucalyptus and birch

kraft pulps; i.e. the CNCs consisted of a hydrophilic and a hydrophobic particle population. It was shown that the hydrophilic particles in the CNCs had a similar hydrophobicity value as polyelectrolyte complexes consisting of pure hydrated polysaccharide chains. It was also shown that the hydrophobic particles in the CNC had a similar hydrophobicity value as colloidal wood pitch droplets. It was determined that the increased hydrophobicity of the CNCs was an effect of lipophilic extractives, especially sterols, present in the kraft pulps. These lipophilic components were able to withstand the harsh acidic hydrolysis step of CNC production, and remained with the CNCs through the rest of the production steps.

The use of a selective staining agent in combination with FCM analysis enabled detection of the hydrophobic particle populations within the CNC suspensions, even though the concentrations of lipophilic extractives in the raw materials were quite low. The difference in particle hydrophobicity was not detected from light scattering alone and would go completely undetected without the use of FCM. Furthermore, the FCM technique enables the user to gate certain particle populations and plot/calculate particle populations separate from each other. These facts indicate that FCM is a useful and powerful analysis technique also for CNCs. It should also be concluded that the content of lipophilic extractives in different raw materials need to be determined and taken into account prior to CNC production, if no pre-extraction step is employed.

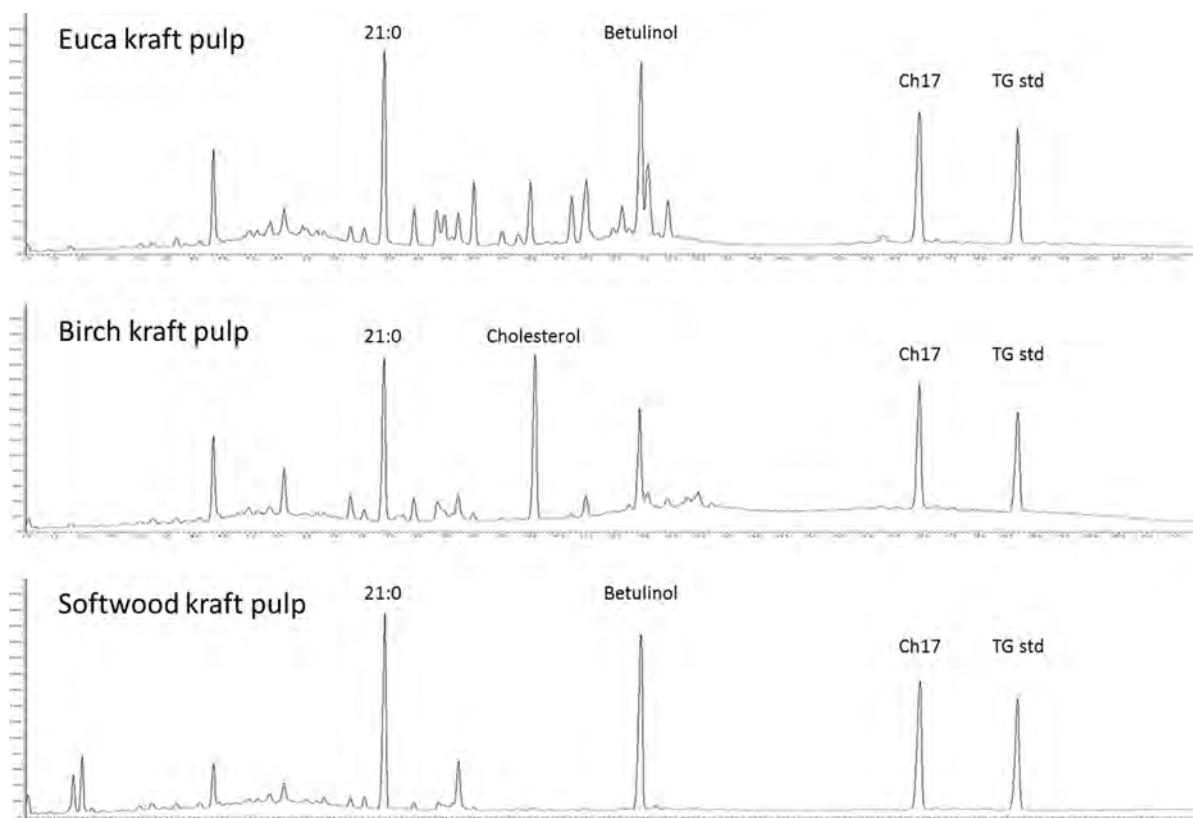
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## Appendix

See Fig. 5.





**Fig. 5** Short column GC chromatograms of the silylated extracts from the three different kraft pulps. The four internal standards used are marked in the chromatograms

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## Bio-based Polymers for Sustainable Packaging and Biobarriers: A Critical Review

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Barrier materials have an important role in various packaging applications, especially considering the requirements associated with protection and shelf life. Most barrier materials used in today's industry are either manufactured from oil resources or metals. Driven by the increase in environmental awareness, access to oil resources as well as legislation, new and environmentally benign alternatives are at the center stage of scientific and industrial interest. This article covers the use of wood-derived polymers and those produced from microorganisms, which display remarkable barrier properties. Wood-based products have received great attention for their air/oxygen resistance. As far as their properties, microorganism-derived biopolymers are comparable to conventional oil-based thermoplastics, but their cost may still be an issue. Both wood and microorganism-derived biopolymers are challenged when moisture, grease and oxygen resistance are simultaneously required. Hence, multilayer structures and composites are needed to fulfil the most demanding requirements of packaging materials. Here we offer a review of these topics together with a discussion of their prospects.

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**Keywords:** Biopolymer; Packaging; Barrier

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## INTRODUCTION TO BIOBARRIER MATERIALS

Bio-based and biodegradable packaging materials have gained increased global attention. Among the drivers towards more sustainable packaging materials, the following ones stand out: growing environmental awareness (Andersson 2008; Mousavioun *et al.* 2010; Hermann *et al.* 2011; Philp *et al.* 2013), waste management and landfilling (Andersson 2008; Hermann *et al.* 2011; Johansson *et al.* 2012; Philp *et al.* 2013; Khan *et al.* 2014), resource insufficiency (Andersson 2008; Wu *et al.* 2009; Chung *et al.* 2013), the accumulation of plastics in the ocean (Philp *et al.* 2013), waste legislations, producer and consumer accountability (Andersson 2008), the need to reduce energy consumption (Mousavioun *et al.* 2010), and marketing trends (Weber 2000; Khan *et al.* 2014). In fact, compared to those sourced from fossil carbon, the use of bio-based polymers represents a solution that can effectively benefit from the above pressures, mainly owing to their sustainability, biodegradability, biocompatibility, availability, and non-toxicity (Rastogi and Samyn 2015); in addition, they bring about a possible reduction in overall carbon footprint (Greene 2014).

While the packaging industry is focusing on creating lighter products, to reduce raw material use, transportation costs, and waste volumes (Johansson *et al.* 2012; Vartiainen *et al.* 2014), consumers and producers are focusing on recyclable,

environmentally-friendly, and non-fossil-based packaging solutions (Talja *et al.* 2011). As a component of packaging materials, paperboard provides the necessary mechanical strength. However, it needs to be combined with other materials to promote the required barrier performance (Andersson 2008; Rastogi and Samyn 2015). For instance, traditionally, paperboard packaging materials are coated with synthetic polymers that enhance their resistance to water, moisture, grease, oxygen, and odor (Talja *et al.* 2011). In this review, we use the term “biobarriers” to refer to the main components of the system if they are bio-based, either in their pure, blended, or composite forms. So far, fossil-derived synthetic polymers have been the preferred choice, owing to their beneficial properties and the relatively low price (Siracusa *et al.* 2015). In stark contrast to paperboard, however, most petrochemical-based polymers exhibit poor biodegradability and represent a challenge for their disposal and subsequent landfilling (Johansson *et al.* 2012). In 2015, Europeans generated 84.5 million tons of packaging waste, equivalent to 166.3 kg per inhabitant. The share of plastic packaging waste was 19%, resulting in 31.6 kg plastic packaging waste per inhabitant and 15.9 million tons in total. The share of other packaging materials were 41% paper and cardboard, 19% glass, 16% wood, and 5% metal (Eurostat 2015). The food product packaging sector represents a minor share of the total environmental impact of packed food units (Johansson *et al.* 2012; Grönman *et al.* 2013). The adoption of biopolymer alternatives to petroleum-based plastics potentially reduce carbon dioxide emissions by 30% to 70% (Lackner 2015).

The main objective of a package is to protect the product from the surrounding environment and to achieve this result in a sustainable manner (Gröndahl *et al.* 2004; Mikkonen and Tenkanen 2012). Packaging materials should provide mechanical, chemical, and biological protection (Khan *et al.* 2014). A suitable packaging should fulfill performance metrics and should be safe, enable long shelf life, reduce the loss of food (Khan *et al.* 2014; Sand 2016), and make the product more sustainable (Sand 2016). To fulfill these requirements, barrier materials should protect against oxygen, carbon dioxide, moisture (Gröndahl *et al.* 2004; Arora and Padua 2010; Johansson *et al.* 2012; Mikkonen and Tenkanen 2012), aromatic compounds (Arora and Padua 2010; Johansson *et al.* 2012; Mikkonen and Tenkanen 2012), water, micro-organisms (Mikkonen and Tenkanen 2012), and grease (Johansson *et al.* 2012). Some of the main functional properties of packaging materials are included in Fig. 1, relevant to the exposure to given agents. Generally, the most common challenges with biobarriers have been their low resistance to water, gases (Arora and Padua 2010), heat, and mechanical stress (Johansson *et al.* 2012), as well as their relatively high price (Song *et al.* 2009; Philp *et al.* 2013).

In this review, wood- and microbial-derived polymers are considered as biobarrier materials. Wood-based materials are prominent for their high potential and availability (Vaca-Garcia *et al.* 1998; Gandini 2008; Edlund *et al.* 2010). Fermentation-based biodegradable barrier materials, such as polylactide (PLA), poly(butylene succinate) (PBS), and poly(hydroalkanoates) (PHAs), are synthesized from bio-based sources and are available at industrial scales (Fortunati *et al.* 2012; Rabu *et al.* 2013; Bugnicourt *et al.* 2014; Rastogi and Samyn 2015; Siracusa *et al.* 2015). Other options for bio-based barrier materials, which are not considered in this review, are proteins (*e.g.*, whey, soy, gluten, collagen) and polysaccharides such as starch, alginate, and chitosan (Rastogi and Samyn 2015). While biopolymer processing is not covered in this review, we refer to the literature for discussion on the main technologies used to apply barrier coatings and films, including melt extrusion, dispersion, and solvent-based approaches, reported in

(Rastogi and Samyn 2015) and others. We start this review by introducing the terminology around the biobarrier field followed by a discussion on the main biobarriers, their properties, and future prospects.



**Fig. 1.** Some of the desirable properties of packaging materials (left) in contact with different elements (right) and to meet a number of requirements, including cost effectiveness

## TERMINOLOGY

Biobarrier terminology is important as far as the polymer composition and associated definitions. For example, not all biopolymers are produced from renewable biomass (Philp *et al.* 2013), not all bio-based polymers are biodegradable, and *vice versa* (Song *et al.* 2009). In this section, we briefly introduce the most relevant terminology.

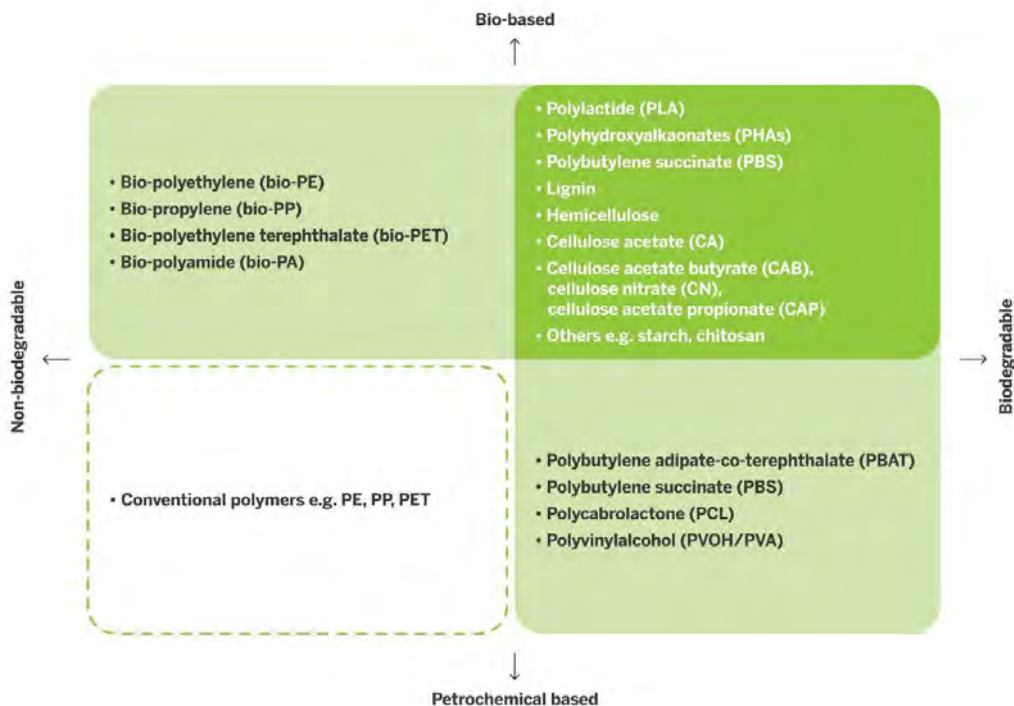
### Biopolymers

Along with the growing interest in biopolymers, their diversity, sources, and properties are ever expanding (Philp *et al.* 2013). Biopolymers can be generally considered to be (a) bio-based and biodegradable, (b) bio-based and non-biodegradable, and (c) petroleum-based and biodegradable (Fig. 2) (Philp *et al.* 2013; Lackner 2015; Rastogi and Samyn 2015). Traditional petroleum-based polymers are generally non-biodegradable, but there are some exceptions such as poly( $\epsilon$ -caprolactone) (PCL), polybutylene adipate terephthalate (PBAT), and poly(butylene succinate) (PBS) (Philp *et al.* 2013).

### Bio-based Polymers

Bio-based polymers originate either entirely or partially from renewable biomass resources (CEN 2017; European Commission 2017). Renewable biomass resources include plants, micro-organisms, and animals (Song *et al.* 2009). Bio-based polymers can either be directly derived from renewable biomass resources (*e.g.*, cellulose), produced by chemical synthesis where renewable monomers are used (*e.g.*, PLA), or produced by micro-organisms (*e.g.*, PHAs) (Weber 2000; Weber *et al.* 2002; Shalini and Singh 2009; Rastogi and Samyn 2015).





**Fig. 2.** Polymers and biopolymers shown according to their biodegradability and source (Puls *et al.* 2010; Xu and Guo 2010; Sabiha-Hanim and Siti-Norsafurah 2012; Philp *et al.* 2013; Lackner 2015; Yu *et al.* 2016)

The U.S. Department of Agriculture (USDA) has determined the minimum content of bio-based material for 109 different product categories (USDA 2017a). In the BIOPreferred program (USDA 2017b), the referenced products need to reach the specified minimum limit in order to be certified as bio-based (Greene 2014). Testing is required according to the ASTM D6866 standard (Greene 2014). The minimum relative content of bio-based material for disposable tableware made of or coated with plastic, for example, is 72% (USDA 2017a). Moreover, in addition to the BIOPreferred label, there are several other bio-based labels, such as OK biobased by Vincotte, which relates to the ASTM D6866 testing. In practice, the content of bio-based material can be determined by quantifying  $^{14}\text{C}$  content, according to ASTM D6866-05 (2005). This is because fossil-based carbon does not include radiocarbon ( $^{14}\text{C}$ ) due to its half-life of 5,730 years (Kijchavengkul and Auras 2008). There is no comparable ISO-standard test method for determining the content of bio-based materials currently available (Greene 2014).

## Biodegradability

There are numerous standards available for biodegradability (Müller 2005; Kale *et al.* 2007; Rudnik 2012; Philp *et al.* 2013) from international (ISO, ASTM, CEN) and national (*e.g.*, DIN) standardization bodies (Rudnik 2012). The European Committee for Standardization (CEN) has determined that biodegradable material degrades into biomass, carbon dioxide, and/or methane, and water under the influence of micro-organism activity (Müller 2005; Hermann *et al.* 2011). Likewise, other definitions are found, *e.g.*, in the European Parliament and Council Directive 94/62/EC; that source states that biodegradable packaging waste is required to undergo physical, chemical,

thermal, or biological decomposition, converting the material mainly into carbon dioxide, biomass, and water (Andersson 2008).

**Table 1.** Biodegradability Standards (Philp *et al.* 2013; Greene 2014)\*

Standard	Description	Final Disposal
AS 4736 -2006	Biodegradable plastics – biodegradable plastic suitable for composting and other microbial treatment	Compost
ASTM D5338 -98	Standard test method for determining aerobic biodegradation of plastic materials under controlled composting conditions	Compost
ASTM D6002 -96	Standard guide for assessing the compostability of environmentally degradable plastics	Compost
EN 13432 : 2000	Requirements for packaging recoverable through composting and biodegradation – test scheme and evaluation criteria for the final acceptance of packaging	Compost
ISO 14855: 1999	Determination of the ultimate aerobic biodegradability and disintegration of plastic materials under controlled composting conditions – method by analysis of evolved carbon dioxide	Compost
ASTM D5988 -03	Standard test method for determining aerobic biodegradation in soil of plastic materials or residual plastic material after composting	Soil
ISO 17556: 2003	Plastics – determination of the ultimate aerobic biodegradability in soil by measuring the oxygen demand in a respirometer or the amount of carbon dioxide evolved	Soil
ASTM D6691-01	Standard test method for determining aerobic biodegradation of plastic materials in the marine environment by a defined microbial consortium	Marine
ASTM D6692-01	Standard test method for determining biodegradability of radiolabeled polymeric plastic materials in seawater	Marine
ISO 15314:2004	Methods for marine exposure ISO 16221:2001 Water-quality – guidance for the determination of biodegradability in the marine environment	Marine
ISO 16221: 2001	Water quality — Guidance for determination of biodegradability in the marine environment	Marine
ISO 14851	Determination of the ultimate aerobic biodegradability of plastic materials in an aqueous medium - Method by measuring the oxygen demand in a closed respirometer	Marine*
ISO 14852	Determination of the ultimate aerobic biodegradability of plastic materials in an aqueous medium -- Method by analysis of evolved carbon dioxide	Marine*
ASTM D7081	Standard specification for non-floating biodegradable plastics in the marine environment	Marine*

Several publications are available on the topic of biodegradability standards (Müller 2005; Rudnik 2012; Philp *et al.* 2013). Among other issues, these publications describe the complexity of biodegradation process (Müller 2005; Rudnik 2012; Philp *et al.* 2013), including a comparison of different examination methods for polymer

biodegradability (Rudnik 2012). Biodegradation tests are recommended to select according to the material's expected application as well as its end-of-life, which can take place in different environments, such as compost, soil, and fresh or marine water (Philp *et al.* 2013). A few general standards related to biodegradation in compost, soil or a marine environment are listed in Table 1.

Oxo-biodegradability often involves the effect of additives, such as metal salts (*e.g.*, manganese, iron, cobalt, nickel) that are added to plastics (*e.g.*, PE) to expedite the otherwise very slow degradation. The outcome of oxo-biodegradation relates to the generation of non-visible, micro-sized plastic and metal particles (Philp *et al.* 2013). Testing of these materials is described in the ASTM D6954-18 (2018) standard. The use of oxo-degradable polymers is banned in Germany (Kosior *et al.* 2006).

A few standards for addressing marine degradation exist. In general, plastics that are degraded in marine environments biodegrade and disintegrate into seawater within a certain period without causing any impact on the surrounding marine organisms. In addition, these plastics are required to pass marine toxicity tests, have a very low heavy metal content, and should be industrially compostable (ASTM D6400-12 2012) (Greene 2014).

### Compostability

A compostable material is generally defined as a material that is biodegradable under aerobic conditions and converted into biomass, carbon dioxide, water, and inorganic compounds, in turn, without producing any toxic compounds and disintegrating during the fermentation phase (Mohanty *et al.* 2000; Kale *et al.* 2007; Philp *et al.* 2013). In addition, the compostable material, according to CEN norms, should not cause complications, neither to the composting process, nor to the compost itself (Weber 2000). Compostable polymers are considered as biodegradable, whereas biodegradable polymers are not necessarily compostable, which has higher demands, *e.g.*, of resulting biodegradation products (Weber 2000; Müller 2005; Kale *et al.* 2007), heavy metal content (European standard EN 13432 2000), disintegration within a certain time frame, and the requirement that they do not cause problems during the process of composting (Weber 2000). Philp *et al.* (2013) and Rudnik (2012) have presented multiple standards considering compostability in their publications. Polymers should be tested by relevant ISO, ASTM, and EN standards in order to meet the compostability criteria (Philp *et al.* 2013).

European standard EN 13432 for compostability of packaging is commonly used (Kosior *et al.* 2006; Philp *et al.* 2013). Test methods and certification refers to industrial composting (Kosior *et al.* 2006; Hermann *et al.* 2011). In industrial composting, the temperature ( $58 \pm 2$  °C), humidity, composting cycles (3 months thermophilic and 3 months maturation phase), and aeration conditions are carefully controlled. In contrast, home composting conditions vary widely. In the case of home composts, temperatures are lower and may vary considerably throughout the seasons (Kosior *et al.* 2006; Song *et al.* 2009). Moreover, there is no international standard for home compostability (Kosior *et al.* 2006; Endres and Siebert-Raths 2011; Hermann *et al.* 2011), and industrially compostable products (EN 13432) may not perform acceptably in home composts (Song *et al.* 2009; Endres and Siebert-Raths 2011). A certification for home compostable product is available from Vincotte, "OK Compost Home". This certification follows the EN 13432 testing standard, except that the process takes a longer period of time (365 days instead of 180 days) and requires lower temperatures (20 °C to 30 °C instead of 58

°C) (Song *et al.* 2009; Endres and Siebert-Raths 2011; Hermann *et al.* 2011). Industrially compostable certifications, such as Din-Geprüft industrial compostable and OK Compost are following EN 13432 testing (Kale *et al.* 2007; Song *et al.* 2009; Hermann *et al.* 2011). There are also other certifications, *e.g.*, “Compostable”, which are based on ASTM D6400-12 (2012) (Kale *et al.* 2007).

## Legislation

The safety of packages and packaging materials are secured by a variety of laws and regulations. General product safety directive 2001/95/EC (European Commission 2001), food contact material regulation EC No 1935/2004 (European Commission 2004), the regulation considering manufacturing of food contact materials EC No 2023/2006 (European Commission 2006) (GMP Regulation), and the positive list of substances for plastic packaging materials EC No 10/2011 (European Commission 2011) are a few of the guidelines which food contact materials are required to fulfill (Leminen *et al.* 2013).

In addition, there are regulations and recommendations used globally for food contact materials (Leminen *et al.* 2013). They include those from the U.S. Food and Drug Administration (FDA), the Bundesinstitut für Risikobewertung (BfR) recommendations (Andersson 2008; Leminen *et al.* 2013) and Chinese Standards, *e.g.*, Food-Contact Use Paper, Paperboard and Paper Products GB 4806.8-2016 (2016), and GB 9685-2016 (2016) National Food Safety Standard: Standard for the Use of Additives in Food Contact Materials. Other regulations and national laws need also to be considered (Leminen *et al.* 2013).

The European Commission (May 2018) has published a proposal for a directive of the European Council on the reduction of the impact of certain plastic products on the environment. The goal is to prevent and reduce marine litter from single-use plastics and plastic fishing gear. Some products are being banned from the market, *e.g.*, plastic cutlery, straws, and plates, whereas the use of plastic cups and food containers need to be reduced by the Member States. Among these actions, there are other rules that Member States and their producers must fulfill (European Commission 2018). One example of a national ordinance, which is a driving factor for increasing the use of bio-based polymers, is the French ordinance Décret n° 2016-1170 (2016). It requires all disposable tableware (*e.g.*, cups and plates) to be home-compostable and produced from 50% renewable materials by January 2020. Furthermore, the required renewable content will be raised to 60% by January 2025.

## Barrier Materials

In the European Union (EU) legislation for plastic materials, Regulation (EU) No. 10/2011, a functional barrier is defined as a layer that prevents substances from migrating from behind the barrier layer into the food.

To avoid confusion, this review considers the term “matter” to refer to both ideal and non-ideal gases, vapor, or liquid, whereas by “gas” we refer to gases, such as those in the atmosphere. Permeating matter displays two primary ways of transport through film material: either by transmitting through the entire thickness of a solid, homogeneous film, or by transmission through its defects, such as pores or pinholes. As porosity and pinholes resemble an unwanted heterogeneous film structure, this review will focus on the former case. Permeating matter undergoes three physical phenomena referred to as absorption, diffusion, and desorption (Paine and Paine 1992; Auvinen and Lahtinen 2008; Nair *et al.* 2014).

Several types of barrier properties are measured to determine the exposure of a package's contents to ambient conditions. The most common barrier measurements include gas transmission such as oxygen, liquid, and vapor transmission, such as water and water vapor, and oil transmission, followed in test conditions typically by castor oil. The properties of the permeating molecule, the properties of the film material, and ambient conditions all influence the barrier properties. For instance, the size and polarity of the molecule, the polarity and crystallinity of the film material, the temperature, and relative humidity conditions influence the amount and rate of permeating matter. In principle, transmission is non-existent when the permeant molecule and the film material are insoluble in each other, and *vice versa*. In this case, the cohesive energy is high between the permeating molecule and film material, whereas a high solubility, in turn, indicates low cohesive energy (Auvinen and Lahtinen 2008).

Inside a polymer film, the molecular motion of permeating gas appears in small scale compared to the free space to another stochastically bouncing molecules. However, in the large scale there is a clear trend of molecular flow, which tends to equalize the difference in chemical potential between the sides of the film material. Thereafter, the large-scale motion of gas molecules from one side of a film material to the opposite side follows Fick's first law of steady state diffusion, as indicated in Eqs. 1 (concentration difference between film edges) and 2 (pressure difference between film edges). In steady state conditions, the absorption of permeating matter into the film from one side is the same amount of matter as its desorption out of the film from the other side, thus resulting in steady diffusion of matter inside the film. Equation 2 is obtained when adding Eq. 3 (Henry's law of solubility) to Eq. 1. Finally, Eq. 4 is a common permeation equation related to the relation of the gas coefficients (Paine and Paine 1992; Auvinen and Lahtinen 2008).

$$J = \frac{D(c_1 - c_0)}{l} \quad (1) \quad J = \frac{P(p_1 - p_0)}{l} \quad (2) \quad c = S p \quad (3) \quad P = D S \quad (4)$$

In Eq. 4,  $J$  is the *steady state diffusion* [work/area], e.g., [J/cm<sup>2</sup>],  $P$  is the *permeation coefficient* [amount\*material thickness/area\*time\*pressure difference], e.g., [cm<sup>3</sup>cm/cm<sup>2</sup>s Pa],  $D$  is the *dissolution coefficient* [area<sup>2</sup>/time unit], e.g., [cm<sup>2</sup>/s],  $S$  is the *solubility coefficient*, e.g., [cm<sup>3</sup> (STP)/cm<sup>3</sup> polymer],  $l$  is the *film thickness*, e.g., [cm],  $c$  is the *concentration of dissolved gas*, e.g., mL/L or M,  $p$  is the *partial pressure of a gaseous solute*, e.g., Pa,  $p_1 - p_0$  is the *partial pressure difference between the permeating gas on one side and the other side of the film*, and  $c_1 - c_0$  is the *concentration difference between the permeating gas on one side and the other side of the film* (Piringer 2000a; Auvinen and Lahtinen 2008).

In steady state, given by Eqs. 1 to 4, the activation energy of diffusion is reached, thus allowing diffusion to occur. Equations 1 to 4 contain dimensionless coefficients, set by  $P$ ,  $D$ , and  $S$ . The diffusion coefficient is a measure of the speed by which molecules pass through a given area of the film material, while the solubility coefficient is a measure of the molecules that pass through given area. Basically, the coefficients as such resemble ideal gas behavior, whereas for non-ideal gases, vapors, and liquids, the coefficients are corrected by applying to Eq. 4 the Arrhenius equation of activation energy (Paine and Paine 1992; Auvinen and Lahtinen 2008). Moreover, Fick's second law of diffusion is related to unsteady state conditions. Before reaching steady state and constant diffusion, the permeating gas exhibits an unsteady state, accelerating diffusion



after reaching the state of its activation energy. The reader is referred to Piringer (2000a,b) and Paine (1992) for more information.

### Determining Barrier Properties

Methods such as water contact angle and the Cobb test (see standard ISO 535, 2014) are useful to assess the surface properties and wettability of the substrate onto which a barrier layer is applied. Most relevant, for quantitatively determining the extent of the four types of barrier properties, standardized and non-standardized methods are available. Relations such as Eqs. 1 to 4, are typically based on phenomena described by permeation or transmission and gravimetric analyses. Several authors, including (Tunç and Duman 2010; Rastogi and Samyn 2015) have used gravimetric techniques to evaluate the water solubility of films, Eq. 5,

$$\text{Water solubility (\%)} = 100 (W_1 - W_2) / W_1 \quad (5)$$

where  $W_1$  and  $W_2$  represent the initial and undissolved dry matter weights (g) of films, respectively.

For water vapor permeability, there are both standardized and non-standardized test methods. The water vapor transmission rate (WVTR) represents the mass of water per surface area unit of barrier film, which permeates through the surface within a predetermined time interval and pressure difference, within a constant temperature and constant relative humidity. A typical unit of WVTR is  $\text{g} \cdot \text{m}^2 \cdot \text{day}^{-1}$  (Dufresne 2012; Rastogi and Samyn 2015). Han *et al.* (2010) calculated WVTR by combining Henry's Law and Fick's first law by using Eq. 6,

$$\text{WVTR (g/m}^2 \cdot \text{24 h)} = w/A \quad (6)$$

where  $w$  represents the increase in weight (g) of the film in the measuring unit after 24 h and  $A$  is the area ( $\text{m}^2$ ) of the exposed film. The unit can be also expressed as  $\text{cc} \cdot \text{m}^{-2} \cdot \text{s}^{-1}$  (Siracusa *et al.* 2008).

Bilbao-Sainz *et al.* (2010) calculated water vapor permeability (WVP) by a "gravimetric modified cup method", following ASTM E96 (ASTM E96 / E96M-16 2016). They also defined adsorption desorption isotherms of water by using a dynamic vapor sorption analyzer (DVS).

Being the initiator of life on our planet, oxygen is the most bioactive and reactive gas of all atmospheric, gases. Therefore, it is essential to protect a package contents from oxygen to ensure its high quality and to promote a long-lasting shelf-life. For oxygen, there are a few methods of measurement. The most traditional one is the oxygen transmission rate (OTR) by using the MOCON instrument, according to ASTM D3985-17 (2017) standard. In this method, the barrier material is placed between two chambers at ambient atmospheric pressure in dry condition ( $\text{RH} < 1\%$ ). One chamber contains nitrogen and the other oxygen. The permeated oxygen is detected by a coulometric detector. The unit of OTR is given as  $\text{cc} \cdot \text{m}^{-2} \cdot \text{s}^{-1}$  (Siracusa *et al.* 2008). Once the oxygen transmission rate is determined, the transmission rates of other atmospheric gases can be predicted by knowing the relationship between each other. For example, for nitrogen, the transmission rate is one-third of that of oxygen, while carbon dioxide permeates six times as fast as oxygen (Paine and Paine 1992). However, an assigned quantity for carbon dioxide also exists, known as carbon dioxide transmission rate ( $\text{CO}_2\text{TR}$ ), expressed as  $\text{cc} \cdot \text{m}^{-2} \cdot \text{s}^{-1}$ . Similar to WVTR and OTR, one area of importance for  $\text{CO}_2\text{TR}$  is in food packaging (Siracusa *et al.* 2008).

Although more seldomly measured compared to oxygen/air, water, and water vapor permeability, the fat/oil/grease barrier performance is very important in the food packaging industry (Lavoine *et al.* 2012). This is due to an inherently high fat content in many food products (Leminen *et al.* 2015). Moreover, a few different methods of measurement for grease and oil exist. According to Auvinen and Lahtinen (2008), the amount of grease or oil penetration into a film material depends on both internal and external factors. The internal factors govern properties such as the relation of saturated fats to unsaturated fats and the average length of fat chains; the external factors govern the temperature and the relative humidity. Several options for the measurement of oil absorption are available: the COBB-Ungern method (SCAN-P 37:77 1976), the Kit-test (TAPPI Test Method T 559 cm-12 2012), and the ASTM F119-82 (2015) standard test are some of the most commonly applied methods.

Other types of phenomena that need to be minimized in order to protect the package content are worth mentioning. In fact, the barrier film itself is not necessarily inert to the package's contents. Instead, it may release molecular species to the contained product. These can include volatile organic compounds (VOCs), including aromatic compounds, flavors, and fragrances. In EC No 10/2011 of the European Union (EU) legislation for plastic materials, the unit of migration is mg/kg. Here, migration loosely refers to mass transport through the barrier film, which affects food safety and quality, and involves direct contact with the package content and the package layer (Auvinen and Lahtinen 2008). In addition, pathogens, including various microbes and bacteria, such as *Escherichia coli* and *Staphylococcus aureus*, can harm the package content. Finally, given frequencies of light radiation can deteriorate the package content. UV light can cause harm to the package content by inducing oxidative rancidity. Likewise, fat-containing products tend to deteriorate when exposed to sunlight. In commercial packaging materials, a coating of aluminum foil is often used for protection against UV light (Paine and Paine 1992; Kirwan 2005; Lavoine *et al.* 2012).

## WOOD BASED BARRIERS

Wood consists mainly of cellulose (40% to 50%), hemicellulose (25% to 35%), and lignin (18% to 35%) (Pettersen 1984). These materials can be also isolated from various agro-based feedstocks (Laine *et al.* 2013) and, in fact, cellulose, hemicellulose, and lignin are the most abundant plant-based natural polymers (Klemm *et al.* 2005; Antonsson *et al.* 2008; Albertsson *et al.* 2011). Interesting properties from the barrier point of view have been discussed in several studies. In this section cellulose derivatives, nanocellulose, lignin, and hemicellulose are considered.

### Cellulose Derivatives

Before adoption of petroleum-derived plastics, several commercially-produced cellulose plastics (cellulosics, cellulose chemicals, or cellulose derivatives) have been commonly used. From 1960s onwards, the superior performance and low cost of petroleum-derived plastics made them more attractive. Many large volume cellulose derivatives are still used today, in specific applications (Gilbert 2017). Cellulose derivatives are prepared from native cellulose, which is hydrophilic, yet water insoluble. Common cellulose derivatives are soluble in various industrial solvents, and this can be a great advantage for processing and for film development (Rastogi and Samyn 2015). The

swelling tendency of cellulose, because of its hydrophilic and hygroscopic nature, limits its film-forming capability while it requires an energy-consuming drying process. The motivation to produce cellulose derivatives is to respond to the limitations of native cellulose (Rastogi and Samyn 2015), while maintaining the bio-based origin and characteristic biodegradability. Cellulose derivatives include cellulose ethers and cellulose esters (Brydson 1999; Kuusipalo *et al.* 2008; Granström 2009). Their preparation can be done by heterogeneous or homogeneous modification (Granström 2009), the former of which generally maintains the fibrous structure while the latter applies to organic solvents for derivatizing the cellulose backbone. There are several variables that affect the properties of cellulose derivatives, out of which the degree of substitution (DS), and the chain length, described by the degree of polymerization (DP) of the anhydroglucose monomers, are quite relevant. The DS relates to the average number of hydroxyl groups substituted per anhydroglucose units. Thus, the higher the DS, the more complete the reaction (Reese 1957). There are several properties that are dependent on the DS. Whereas viscosity indicates the average DP, associated with mechanical and rheological properties, the softness, hardness, and moisture absorption of the material depend strongly on the DS (Gilbert 2017). Moreover, the DS affects the biodegradability of cellulose ethers; a higher DS means a larger number of ether linkages and therefore lower biodegradability (Andersson 2008). Another important parameter is the purity of the cellulose used as raw material, as indicated by the relative mass fraction of pure cellulose (alpha-cellulose) (Burton and Rasch 1931). The more amorphous and the less crystalline the structure, the higher the rate of diffusion, being specific for different reagents (Gilbert 2017). In heterogeneous modification, the DS is significantly dependent on the location of the anhydroglucose unit in the fibrous structure (Thielking and Schmidt 2006; Andersson 2008). For improved results in cellulose modification, both the position of the modified anhydroglucose unit in the chain and in the fibrous structure as well as the positions of the substituents around the anhydroglucose ring and along the chain should be controlled (Fox *et al.* 2011; Gilbert 2017). Uniform distribution of reaction during cellulose structural modification is required for targeting advanced end-use properties. Regioselectivity is facilitated by homogeneous modification and associated solvents, while today's heterogeneous modification relies on analytical methods and controlling regioselectivity (Fox *et al.* 2011). Cellulose derivatives have been reported to act as suitable matrix materials for cellulose nanocrystals, which in turn improve the water vapor barrier of cellulose derivatives, while acting together as a filler composite (Paunonen 2013).

### Cellulose Ethers

The preparation sequence of cellulose ethers consists of an alkalization and an etherification step. The purpose of alkalization is to activate the hydroxyl groups of the cellulose polysaccharides, which yields alkali cellulose. In the subsequent etherification, the alkali concentration of the preceding alkalization step defines the final amount of substituents that shall replace the hydroxyl groups in the backbone of alkali cellulose. Therefore, the alkaline concentration regulates the DS of the polymer (Brydson 1999; Thielking and Schmidt 2006). The cellulose ethers presented in the following section can all be used as barrier materials (Rastogi and Samyn 2015). Except for hydroxypropyl cellulose (HPC), cellulose ethers are non-thermoplastic, which means that they do not provide heat-sealable coatings, although they can be cast as films and are water- and ethanol-soluble. Moreover, they inherently display modest moisture barrier and slow

biodegradation, yet they possess a relatively resistance to oil and fat (Andersson 2008). For packaging purposes and compared to cellulose esters, cellulose ethers can be substituted regio-selectively since the substituents, the ethers, are located closer to the main group of the cellulose polysaccharide (Fox *et al.* 2011). However, while comparing with starch, Andersson (2008) concludes that cellulose ethers are expensive to produce in large-scale as coating materials due to their costly derivatization process associated with their recalcitrant crystalline structure.

#### *Methyl cellulose*

Methyl cellulose (MC) is a non-thermoplastic, water-soluble cellulose ether (Khan *et al.* 2010) with high oxygen barrier ability. As its hydrophilic nature would suggest, the water vapor barrier performance of MC is modest (Paunonen 2013). Liu *et al.* (2018) improved the water vapor barrier by grafting a coating of polyethylene-reinforced graphene oxide on a MC substrate. Lagarón and Fendler (2009) obtained a high water barrier by combining methyl cellulose with two types of fillers, either montmorillonite (MMT) or mica. A high water barrier by increasing MMT content was also found by Tunç and Duman (2010). MC is used as a viscosity modifier in fields such as food packaging and pharmaceutical industry, where it is applied as an edible film. In addition, MC is used as a thickener, emulsifier, and water-containing substance, and can be applied as a film or coating material (Paunonen 2013). It has been investigated for drug delivery, antimicrobial materials, and regenerative applications (Liu *et al.* 2018). A potential blend of polycaprolactone (PCL) and methyl cellulose (MC) was proposed by Khan *et al.* (2012), which exhibited low water vapor permeability.

#### *Carboxymethylated cellulose*

Carboxymethylated cellulose (CMC) is prepared by an alkali-catalyzed reaction aided by chloroacetic acid. The CMC is a hydrophilic, non-thermoplastic, water-soluble polymer which displays decent thermal gelatinization. The hydrophilic nature of CMC has been reported to increase along with its DS, which in turn is linked to alkali concentration in the alkalization step used for preparation. Thus, the DS is directly proportional with the WVP, although after a certain alkaline concentration, the DS and WVP reach their maximum and start to decrease. In industry, including papermaking (Paltakari 2009), CMC is used as viscosity modifier, thickener, water-retention agent and as a structural or adhesive component. Likewise, in the packaging field CMC is used for edible films (Paunonen 2013). Mazhari Mousavi *et al.* (2017) coated nanocellulose (e.g. nanofibrillated cellulose, NFC) with CMC as an additive for paperboard. As a result, the barrier properties were improved. The CMC allowed a higher NFC solids content while also reducing NFC flocculation and blocking pores. He *et al.* (2008) demonstrated the procedure of producing CMC from paper sludge as a more cost-effective method than conventional CMC production methods, which utilize cotton linters as precursor material.

#### *Hydroxypropyl cellulose*

Hydroxypropyl cellulose (HPC) is a thermoplastic polymer that provides a good water vapor barrier and satisfactory grease resistance (Thielking and Schmidt 2006). It displays a liquid crystalline behavior with cholesteric, nematic, and smectic phases, which affect both its barrier and mechanical properties, the latter of which are strongly dependent on the measured direction (Andersson 2008). Leminen *et al.* (2015) studied HPC-based dispersion barrier coatings on paperboard to improve its oil resistance. He

postulates that HPC is the only both edible and film-forming thermoplastic cellulose derivative, which makes it especially interesting for multi-component dispersion coatings. HPC formulates readily as gel because of its low DS and abundant hydroxyl groups that form strong hydrogen bonds. The film-forming properties of HPC are relatively good, yet the resulting oil barrier is sufficient only for fast food packaging. HPC can be used in coatings as a film-forming material, thickener, binder, and as a suspending agent (Andersson 2008). Johnson *et al.* (2008) mixed fillers of cellulose nanocrystals (CNC) and microfibrillated cellulose (MFC) into HPC matrix material.

#### *Hydroxypropyl methyl cellulose*

As with most cellulose ethers, hydroxypropyl methyl cellulose (HPMC) is non-thermoplastic, and therefore it cannot form heat-sealable coatings (Paunonen 2013). However, it has been commonly used as barrier material in coatings (Rastogi and Samyn 2015). It displays moderate resistance to fat and oil, yet one of its drawbacks is its poor mechanical film integrity (Bilbao-Sainz *et al.* 2011). Its moisture barrier properties have been reported to improve considerably when introducing fatty acids (Andersson 2008). Coma *et al.* (2001) improved the moisture barrier with fatty acids as well as reduced the resistance of HPMC films against the bacteria *Listeria innocua* and *Staphylococcus aureus*. The etherification step of HPMC consists of methylation, in which pure MC is produced by reacting alkali cellulose with methyl chloride, either in its liquid or gaseous forms. The grades produced in this preparation display a DS between 1.7 and 2.3. Among the several applications, the most popular use of HPMC in industry is as a protective colloid used in the production of vinyl chloride (Thielking and Schmidt 2006). HPMC films are reported for their potential applications in the food industry due to environmental appeals, low price, as well as their flexibility and transparency (Bilbao-Sainz *et al.* 2011). Mahadevajah *et al.* (2016) studied the mechanical and barrier properties of HPMC films by applying various combinations of plasticizers. Larsson *et al.* (2017) applied HPMC at different concentrations in films of cellulose nanofibrils and nanocrystals. However, increasing the content of HPMC did not improve water vapor barrier. Bilbao-Sainz *et al.* (2011) used HPMC as a film matrix for microcrystalline cellulose (MCC), in which MCC improved the moisture barrier compared to a pure HPMC film.

#### **Cellulose Esters**

From the advent of petroleum-based plastics around 1950 and until today, the cellulose esters are some of the most applied thermoplastics. In the 1990s there was a large interest in biodegradable cellulose esters (Gilbert 2017). High oxygen barrier properties while applying cellulose ester to fillers have been reported (Dou *et al.* 2013; Uddin *et al.* 2016), yet water vapor barrier properties are limited. Cellulose esters are produced by reacting an organic or inorganic acid substituent with the three hydroxyls of an anhydroglucose unit (Kuusipalo *et al.* 2008). The preparation sequence of cellulose esters is in principle the same as that of cellulose ethers, yet instead of alkalization and etherification it involves acidification and esterification. Similar to alkalization, the purpose of acidification is to activate the hydroxyl groups of the anhydroglucose units. The extent of activation depends on the acid concentration (Granström 2009). In the subsequent esterification step, the substituent is an acid that corresponds to the final cellulose ester (Brydson 1999).



### Cellulose acetate

In the preparation of CAs, acetic acid anhydride, together with a catalyst of zinc chloride or sulphuric acid, acts as a reagent to substitute the hydroxyls of the cellulose backbone in esterification (Kuusipalo *et al.* 2008). Found in applications in fields such as molding and extrusion (Gilbert 2017), cellulose acetate (CA) is currently the most commonly applied thermoplastic cellulose derivative. Likewise, many of the applications of CA and its forms are found in the food packaging industry, being used as rigid wrapping films (Paunonen 2013). Cellulose acetate can exist in several forms, some of the most common ones being cellulose acetate butyrate (CAB), cellulose triacetate (CTA), and cellulose acetate propionate (CAP). Out of these forms, CAB displays the lowest water absorption. However, the value is still high compared to its counterparts in today's industry, vinyls, such as polyvinyl chloride (PVC). Several biodegradable CAs have been introduced in the 90s. They were marketed as Bioceta® and Biocellat®, the products of the former including eco-friendly hair brushes, *etc.* Likewise, the blends of CA have been marketed under the name Biodegrade® by FKUR. They are food-contact approved and applicable for injection molding and extrusion (Gilbert 2017). Uddin *et al.* (2016) found promising results as far as high oxygen barrier and interfacial adhesion, while combining CA with graphene oxide in order to produce a CA-based oxygen barrier material for biodegradable packaging applications. Dou *et al.* (2013) reported a drastic improvement in oxygen barrier by combining CA and layered double hydroxide nanoplatelets (LDH), after which thermal annealing treatment was carried out. Moreover, there have been several attempts to apply CAs as antimicrobial films, as well as matrix material for nanocellulose fibers (NCF) motivated by the solubility of CAs to organic solvents (Paunonen 2013). Kabiri and Namazi (2014) reported a maximum decrease of 47% in WVP with 0.8% of graphene oxide (GO) on a matrix of nanocrystalline cellulose acetate, where CA was used to link graphene oxide fillers with cellulose nanocrystals. Likewise, Grunert and Winter (2002) combined trimethylsilylated cellulose nanocrystals from bacterial cellulose (BC) with CAB acting as matrix material.

### Cellophane

Cellophane exhibits potential barrier properties (Tome *et al.* 2011). Cellophane, regenerated cellulose in film form, is produced in the viscose process together with rayon fibers (Alén 2011; Paunonen 2013). Tome *et al.* (2011) studied the permeability of atmospheric gases, such as oxygen, and water vapor barrier properties of cellophane by esterifying with fatty acids. As a result, an improvement of 50% in water vapor barrier and 8% in oxygen barrier was reached. These studies encourage further investigation on the barrier properties of cellophane, which is biodegradable and fully suitable in food packaging. However, cellophane use has diminished given the emergence of several other alternatives for packaging. Environmental effects associated with carbon disulfide and other by-products of the viscose process are also important factors; however, cellophane itself is 100% biodegradable, a reason for its popularity as a food wrapping.

### Nanocellulose and Nano-lignocellulose

There are several main reasons for the barrier properties of films comprising micro- or nanofibrillar cellulose or nanocrystalline cellulose. The degree of crystallinity, the length-to-width ratio of fibrils, the surface polarity, and the internal cohesion of the fibrillar suspension all play a role (Lagaron *et al.* 2004; Dufresne 2012; Hubbe *et al.* 2017). The uptake of moisture from surroundings is a significant drawback of

nanocellulose, while facilitating other superior barrier properties (Lindström and Aulin 2014). As is typical with hydroxyl group-abundant biopolymers, nanocellulose exhibits low water-resistance and high water vapor permeability (Hubbe *et al.* 2017). Principally, crystallinity is beneficial in terms of barrier properties, because it is more difficult for molecules to penetrate the crystalline areas (Siró and Plackett 2010). As such, crystallinity is beneficial in terms of water resistance and water vapor barrier. Moreover, heat treatment improves wet strength, rendering the film denser, possibly due to the aggregation of adjacent cellulose chains, and less porous, which is beneficial in preventing leakage (Österberg *et al.* 2013; Hubbe *et al.* 2017). Sharma *et al.* (2014) showed that when heating films of nanofibrillated cellulose (NFC) for 3 h at 175 °C, the water vapor permeability was reduced by 50% (Nair *et al.* 2014), whereas Xia *et al.* (2018) reported a ten-fold decrease in WVTR while comparing 3 h post-treated TEMPO-oxidized nanofibrillated cellulose (TONFC) films to untreated TONFC films. Likewise, for a treated film OTR values of 0.007 and 0.584 mL.µm.kPa<sup>-1</sup>m<sup>-2</sup>day<sup>-1</sup> were measured at room temperature and relative humidity (RH) of 50% and 80%, respectively. The result for RH 80% is 100 times lower than most plastic films, such as PET or PVC. The results are in line with Österberg *et al.* (2013) who indicated OTR values that improved by hot-pressing NFC films. Feng *et al.* (2015) reported variations in the properties of bacterial cellulose (BC), depending on the drying method used. This was highlighted by a water-holding capacity obtained by freeze-drying half that obtained by oven-drying, with values of 6000% and 12,000%, respectively. Likewise, while comparing NFC with CNC, Peng *et al.* (2013) noticed differences in crystallinity and thermal stability from different drying methods. As a result, for NFC, spray-drying displayed the highest combination, in terms of thermal stability and crystallinity, whereas the conclusion was ambiguous for CNC. According to Xia *et al.* (2018), the barrier-enhancing effects of heat on NFC are explained by both increase in crystallinity and reduction in porosity. The increase in crystallinity by heat treatment was also found by Peng *et al.* (2013), who discussed various drying methods for NFC. The higher crystallinity leads to lower oxygen permeability, while water vapor permeability is reduced simply by increased material density.

The larger the length-to-width ratio and the surface area of fibrils, the higher the fibril entanglement and the longer the path for molecules need to travel through the barrier material (Dufresne 2012). The mechanical fibrillation is used to manufacture micro- or nano-fibrils, which influences the barrier properties by affecting fibril dimensions (Kangas 2014). The mechanical fibrillation step involves a few alternative disintegration methods, as presented in Fig. 3. The influence on barrier properties correlate with the reduction of length-to-width ratio and surface area of the fibrils during the mechanical fibrillation (Dufresne 2012; Kangas 2014). As expected, this reduction is greater as the number of steps or passes, is increased (Siró and Plackett 2010). However, mechanical fibrillation also tends to decrease crystallinity (Siró and Plackett 2010), which is a drawback when aiming for superior barrier properties.

#### *Nanocellulose and reinforced composites*

Nanocellulose possesses a high capacity for interacting with fillers when blended in a polymer matrix, leading to exceptional mechanical strength. These properties are enhanced by the high aspect ratio of nanofibers, as well as the inherent reactivity of cellulose. Given suitable structural conditions in the blend, one form of interaction of NFC with a filler is by generating a rigid percolation network. The percolation network

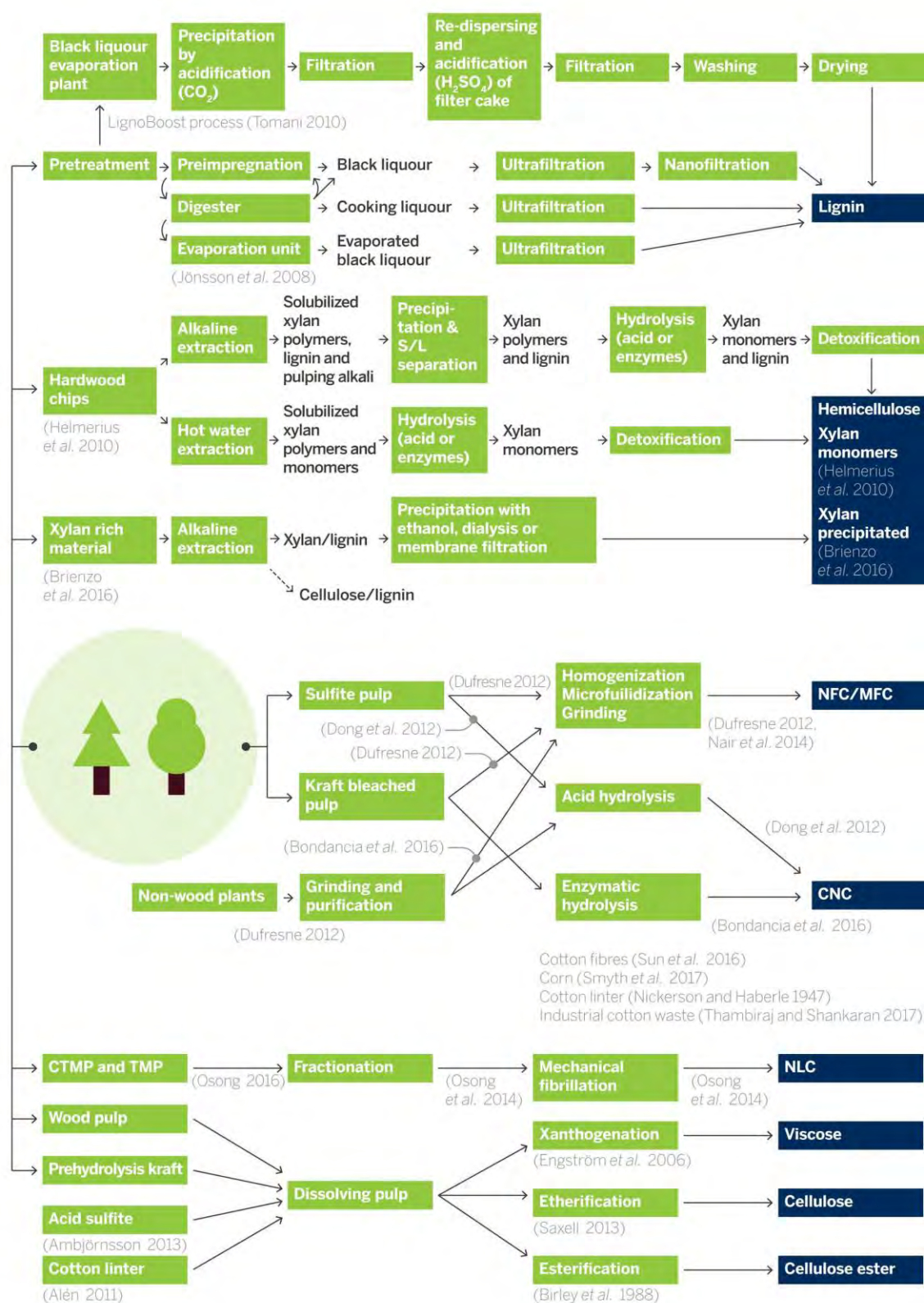
has been cited in many publications (Dufresne 2013). Moreover, superhydrophobic paper has been topical and with high potential demand (Rastogi and Samyn 2015). Arbatan *et al.* (2012) successfully coated NFC together with precipitated calcium carbonate (PCC) as two separate layers on filter paper while applying the dip-coating method. In addition, they applied alkylene ketene dimer to render the paper superhydrophobic.

#### *Nanocellulose/biopolymer blends*

Lindström and Aulin (2014) raised the issue of compatibility between a thermoplastic and non-thermoplastic material, which would need to be tackled prior to industrial-scale implementation. The difficulty of blending nanocellulose with thermoplastic, hydrophobic materials, such as PLA, has been discussed. Attempts to resolve this issue have so far involved the application of surfactants or emulsions. To date, the challenge has not yet been resolved. Nair *et al.* (2018) presented a solution in the form of nanocellulose fibrils with high lignin content (NCFHL), which were wet mixed with PLA. A strong compatibility was found between the fibrils and PLA. In addition, the NCFHL fibrils increased the mechanical, thermal, and water vapor barrier properties. Song *et al.* (2014) applied a blend of nanocellulose fibrils and polylactic acid (NCF/PLA) on paper by a cast-coating process. To disperse it conveniently in the hydrophobic PLA, NCF was rendered hydrophobic by grafting hydrophobic monomers *via* free radical polymerization. Consequently, the WVTR of paper was reduced. We note, however, that to render them hydrophobic or compatible with a hydrophobic matrices, the surface treatment of nanomaterials, such as nanocelluloses, which have a very high area per unit mass, imply a high treatment cost. Jonoobi *et al.* (2010) improved the processability of NFC while blending it with PLA. The improvement was due to the enhanced mechanical and thermal properties. The improvement in storage modulus of the blend was attributed to nanofibers restricting PLA segmental mobility. Espino-Pérez *et al.* (2013) obtained a satisfactory composite, despite some thermal stability issues, by blending cellulose nanocrystals (CNC) with PLA by grafting with n-octadecyl isocyanate. As a result, this hydrophobic, long-chain aliphatic molecule was noted to decrease WVTR, yet OTR remained unchanged.

#### *Nano-lignocellulose*

Nano-lignocellulose (NLC) or ligno-nanocellulose, is a form of nanocellulose produced from mechanical pulp, as displayed in Fig. 3. Mechanical pulp typically contains more lignin than chemical fibers. Therefore, the prepared nanocellulose also inherently contains lignin. Due to the presence of lignin, the mechanical properties of NLC are somewhat lower than those of chemical pulp nanocellulose (Osong *et al.* 2014). One benefit of using NLC instead of chemical pulp nanocellulose is the more economical production of mechanical pulp compared to that of chemical pulp (Osong *et al.* 2016). According to Spence *et al.* (2010), the presence of lignin increased water vapor permeability, due to increased porosity that compensates its hydrophobic nature. However, hot-pressing appears to increase material density and thereafter impart an improved oxygen barrier and surface hydrophobicity. Rojo *et al.* (2015) reported a reduction in oxygen permeability and surface wettability due to increase in material density as a consequence of hot-pressing of NLC films. Likewise, while adding lignin, the reduced wettability was found to correlate with a decrease in the dispersive component of NFC surface energy.



**Fig. 3.** Schematic illustration of some of the processes and precursors used to produce from wood biopolymers and materials that can be used as biobarriers (see references provided for more details).

Despite the significant growth in the number of publications related to nanocellulose fibrils during the latest decade, it is notable how little emphasis has been given on the upscaling of nanocellulose production and the associated economic aspects. Recently, extensive reviews on these challenges for both cellulose micro- and nanofibrils (De Assis *et al.* 2018) and CNC (de Assis *et al.* 2017) were published. Several technical challenges in the behavior of NFC should be resolved prior to implementing industrial production. Some products are already produced by applying NFC as raw material (De Assis *et al.* 2018). Accordingly, Lindström and Aulin (2014) articulated a few practical challenges that might limit the implementation of nanocellulose in packaging applications. One key challenge is to blend hydrophilic nanocellulose with hydrophobic matrices: until today, most advancements are related to demonstrations of surfactant and emulsion-based systems to improve nanocellulose dispersion in a matrix and achieve improved overall properties. Another key challenge relates to the prevention of unwanted hornification and shrinkage, an issue that has not been fully addressed to date. In terms of conventional pulp, the extent of hornification relates to the content of hemicelluloses (Östlund *et al.* 2010), which suggests that hemicelluloses influence the hornification of NFC. A third key challenge relates to the tendency of nanocellulose to absorb moisture from air, given its hygroscopic nature, which tends to compromise other advantageous properties. This issue can be addressed by applying nanocellulose in the form of dense, layered structures within which a high density of hydrogen bonding and the tight packing reduces related interactions. Likewise, combining nanocellulose with other materials, such as PLA or lignin can reduce its hygroscopic nature. A fourth key challenge relates to the drying of nanocellulose, as it typically exhibits high water-holding capacity and high swelling ability. In addition, one challenge relating to upscaling, also pointed out by Lindström and Aulin (2014) is the inherently high viscosity of NFC, which might affect pumping and transport (Hubbe *et al.* 2017; Kumar 2018). Furthermore, according to Kangas (2014), there are a few more issues related to the industrial implementation of nanocellulose. The possibilities of nanocellulose are indicated by the variety of potential applications, for example, to replace oil-based products in packaging, such as polyethylene and polypropylene (Piringer and Baner 2000), and their ability to add new functionalities, such as electroconductivity and printability (Guo 2017; Kumar 2018). A current drawback is the uncertainty in the costs and production scale. Despite these factors, the interest continues to grow at an accelerated pace (Kangas 2014). Some recent attempts to accelerate the use of nanocelluloses for the manufacture of continuous films include a laboratory-scale coating (Kumar 2018) and a pilot-scale SUTCO machine to produce surface-treated nanocellulose films (Peresin *et al.* 2012), to name only a few.

## Hemicelluloses

Hemicelluloses can be found in plant cell walls between cellulose microfibrils. Hemicelluloses have been separated from wood and various agro-based materials (Albertsson *et al.* 2011; Mikkonen and Tenkanen 2012; Laine *et al.* 2013). The content and the composition of hemicelluloses are dependent on the origin and location in the plant (Albertsson *et al.* 2011; Mikkonen and Tenkanen 2012). Depending on the species, wood contains 20 wt% to 30 wt% of hemicelluloses: hardwood contains slightly more hemicelluloses than softwood. The hemicelluloses in hardwood are mainly xylans, while a lesser amount consist of glucomannans. The main hemicelluloses in softwood, on the



other hand, are glucomannans, while a considerably lower amount consist of xylans (Jääskeläinen and Sundqvist 2007; Mikkonen and Tenkanen 2012).

In kraft pulping, hemicelluloses are isolated from hardwood chips and from hardwood black liquor (Talja *et al.* 2011). Hemicelluloses can also be isolated as a co-product from several production processes, such as the production of dissolving pulp, nanocrystalline cellulose (CNC), nanofibrillated cellulose (NFC), and sugar for biofuels (Mikkonen *et al.* 2015). Hemicelluloses could be recovered also by filtrating wastewater streams (Edlund *et al.* 2010). However, hemicelluloses are less-commonly used in industrial scale relative to starch and cellulose (Mikkonen and Tenkanen 2012). A couple of examples from hemicellulose isolation processes are presented in Fig. 3.

Hemicelluloses as such are hydrophilic. The hydrophilicity enables good resistance towards oil and grease (Mikkonen and Tenkanen 2012; Laine *et al.* 2013). Challenges in hemicellulose-based films involve their hygroscopicity and mechanical properties (Saadatmand *et al.* 2013; Chen *et al.* 2016). The high internal cohesion leads to the film-forming properties of xylan to be relatively poor (Talja *et al.* 2011; Vartiainen *et al.* 2014). Nevertheless, hemicelluloses are chemically modifiable due to their free hydroxyl groups. Plasticizers are typically used to improve the flexibility of hemicellulose-based films (Hansen and Plackett 2008; Laine *et al.* 2013; Chen *et al.* 2016). Suitable plasticizers for this purpose include xylitol, sorbitol, glycerol (Hansen and Plackett 2008; Vartiainen *et al.* 2014) and bio-based polymers such as carboxymethyl cellulose (CMC), lignin (Chen *et al.* 2016), and alginate (Vartiainen *et al.* 2014).

Hemicellulose-containing pulp has been a preferred conventional practice in the paper industry to provide a higher yield and to enhance mechanical properties. Hemicelluloses have also been utilized in the food industry, *e.g.* as a sweetener, thickener, and an emulsifier (Gröndahl *et al.* 2004). For example, xylan has been used industrially as a raw material for chemicals (Talja *et al.* 2011). Hemicelluloses have a great potential to be utilized also as bio-based barrier coatings: the raw material is easily available, and suitable application methods include air brush, bar, and curtain coaters (Gröndahl and Bindgård 2014). Due to the encouraging oxygen, grease, and taint resistance of hemicellulose-based films, dry food packaging could be a suitable application. Nevertheless, further improvements with respect to the mechanical properties are required in order to utilize hemicellulose-based barrier materials in application fields, such as food packaging. Mikkonen and Tenkanen (2012) have published a comprehensive article on the use of xylan and mannans in food packaging materials.

Researchers have reported promising oxygen barrier properties with hemicellulose-based films (Hansen and Plackett 2008; Edlund *et al.* 2010; Laine *et al.* 2013; Saadatmand *et al.* 2013). Laine *et al.* (2013) achieved a significantly lower oxygen permeability level by using xylan-based barrier material than with a coating of polyethylene terephthalate (PET). In addition, the same xylan-based barrier functioned as a mineral oil barrier. Acetylated galactoglucomannan (AcGGM) has a promising resistance level towards oxygen, yet it is sensitive to moisture. Hartman *et al.* (2006) produced benzyl-galactoglucomannan (BnGGM) films, which were less sensitive to high-moisture conditions than unmodified AcGGM films, consequently maintaining their oxygen barrier properties. Moreover, galactoglucomannan (GGM) has been shown to have a low resistance towards carbon dioxide, which is desirable in some packaging applications (Mikkonen and Tenkanen 2012). However, there is a need to improve GGM's tensile strength and elongation at break properties (Mikkonen *et al.* 2008). There have been studies about the permeability of aroma compounds into different

hemicellulose-based films including birch xylan, GGM and konjac glucomannan (KGM), with promising results being achieved (birch xylan coated paper < KGM < GGM) (Mikkonen and Tenkanen 2012).

#### *Hemicellulose/mineral composites*

Several studies have been conducted with different hemicellulose composites to obtain enhanced barrier properties (Johansson *et al.* 2012; Mikkonen and Tenkanen 2012). Talja *et al.* (2011) produced glycerol plasticized birch xylan/nanoclay (bentonite) composite coating, which was found to enhance WVTR and aroma barrier properties due to the addition of nanoclay. Nanoclays have the potential to improve barrier properties against gas, water vapor, and aromas due to their feature of making rambling diffusion paths for the molecules. Excellent oxygen barrier performance ( $< 0.18 \text{ cm}^3\mu\text{m}/\text{m}^2\text{dkPa}$ ) was achieved by using oat spelt arabinoxylan (AX) mixed with sorbitol and 50 wt% cellulose nanocrystals. Spruce galactoglucomannan (GGM) displays resistance towards oxygen, although the oxygen resistance did not improve with the addition of cellulose nanocrystals. The same phenomenon was noticed in water vapor properties. The moisture resistance of oat spelt AX films increased by adding cellulose nanocrystal, whereas the cellulose nanocrystal addition did not improve moisture resistance of GGM-based films. Nanofiller additions have also been studied in the perspective of enhancement of mechanical properties (Mikkonen and Tenkanen 2012).

#### *Hemicellulose/biopolymer blends*

The mechanical properties of hemicelluloses depend on the chemical structure, relative humidity, and possible additives, *e.g.*, plasticizers (Mikkonen and Tenkanen 2012). Many studies have investigated the mechanical properties of hemicellulose-based films and their improvement through the addition of another polymer (Mikkonen *et al.* 2008; Mikkonen and Tenkanen 2012), fillers, or by crosslinking.

Mikkonen *et al.* (2015) achieved improved oxygen and water vapor resistance of hydroxypropylated birch xylan (HPX) with an addition of an external plasticizer (sorbitol). Likewise, Gröndahl *et al.* (2004) reported high oxygen barrier properties with aspen glucuronoxylan with 35 wt% of sorbitol. Hemicellulose-based coatings, while blended with a crosslinking agent or a hydrophobizing agent, improved their resistance to oxygen, aroma, and grease in parallel to the improvement of their water vapor resistance. However, due to their hydrophilic nature, hemicellulose-based films are sensitive to changes in RH. An increase in the amount of plasticizer, such as glycerol or sorbitol, reduced the moisture uptake of GGM films at lower RH (Mikkonen and Tenkanen 2012). Crosslinked (5% citric acid) and plasticized HPX films achieved promising grease, mineral oil, oxygen, and water vapor barrier properties. The WVTR was comparable with that of commercial PLA films (Vartiainen *et al.* 2014). A light barrier is an important property in food packaging to maintain the quality of the packaged good. The GGM was found to act as an UV light barrier (Mikkonen and Tenkanen 2012). Improved tensile strength of GGM was achieved by blending with KGM (Mikkonen *et al.* 2008; Mikkonen and Tenkanen 2012).

#### **Lignins**

Lignin is an amorphous polyphenolic macromolecule with functional groups (Mousavioun *et al.* 2010; Gordobil *et al.* 2014), including hydroxyl phenylpropane

(Gullichsen 2000). The main precursors of lignin are coniferyl alcohol, sinapyl alcohol, and *p*-coumaryl alcohol (Alén 2000; Vanholme *et al.* 2010).

The origin and the type of processing of lignin influence its composition, which can vary considerably among different types of sources (Alén 2000; Domenek *et al.* 2013; Hult *et al.* 2013a; Gordobil *et al.* 2014). Lignins can be found in the cell walls of plants (Gullichsen 2000; Yu *et al.* 2016) and are classified as softwood, hardwood, and grass lignins (Alén 2000). The content of lignin in softwoods is normally higher than in hardwoods. For example, the lignin content of pine (*Pinus sylvestris*) is approximately 25% to 30% (dry weight), whereas in birch (*Betula pendula*) it is 20% to 25% (Jääskeläinen and Sundqvist 2007).

Major quantities of lignin can be obtained as a byproduct of pulp production (Pouteau *et al.* 2004; Gandini 2008), whereas another noteworthy source of lignin is the bioethanol industry (Yu *et al.* 2016). A common extraction method of lignin is precipitation from black liquor by acidification (Jönsson *et al.* 2008). A few examples from lignin production routes can be seen in Fig. 3. Currently, most of the technical lignin from the pulp industry is burnt as fuel in recovery boilers (Pouteau *et al.* 2004; Gandini 2008; Domenek *et al.* 2013; Hult *et al.* 2013a,b; Gordobil *et al.* 2014). However, there are several commercial and semi-commercial facilities to produce lignin. Stora Enso, one of the largest kraft lignin producer in the world, has a lignin production of 50,000 tons annually (Upton 2018). The great availability of this complex polymer is one of the main reasons why it is such an interesting raw material (Gandini 2008). In addition, lignin has a great potential for chemical modification into specialty products (Antonsson *et al.* 2008; Hult *et al.* 2013a,b).

Technical lignins are rather dark-colored, while the lignin in the wood is nearly colorless. In some applications, the dark color of lignin needs to be removed. Decolorization methods have been presented, *e.g.*, UV irradiation in a tetrahydrofuran (THF) solution and blocking of the free phenolic hydroxyl groups followed by oxidation (Wang *et al.* 2016).

The formation of films with technical lignins is a great challenge due to its brittleness and rigidity. However, the required thermoplastic properties can be improved by chemical functionalization (Hult *et al.* 2013a,b; Li *et al.*, 2018). Esterification of lignin improved its moisture and oxygen resistance. A piece of paper coated with two layers (3.9 g/m<sup>2</sup> each) of tall oil fatty acid (TOFA) esterified softwood lignin resulted in a 70% reduction in WVTR as well as improvements in OTR (Vartiainen *et al.* 2014). Lignin films and coatings have been fabricated, *e.g.*, with a bar coater (Hult *et al.* 2013a), and solution casting methods (Bhat *et al.* 2013; Shankar *et al.* 2015). Possible application fields for lignin have been presented to include emulsifiers, binding dispersing agents (Jönsson *et al.* 2008; Watkins *et al.* 2015), thermosets, paints, dyes (Watkins *et al.* 2015), wet strength additives (Jönsson *et al.* 2008), chelating agents for heavy metal removal (Toledano *et al.* 2010; Kaewtatip and Thongmee 2013), flame retardant (Kaewtatip and Thongmee 2013), and antioxidants (Kaewtatip and Thongmee 2013; Shankar *et al.* 2015). Lignin and its blends have been reported to provide both gas (Hult *et al.* 2013a,b) and UV light barrier properties, as well as to work as an antimicrobial barrier (Yu *et al.* 2016; Rai *et al.* 2017). Due to these features, lignin-based films have been considered suitable for food packaging applications (Shankar *et al.* 2015; Rai *et al.* 2017).

*Lignin/biopolymer blends*

In spite of native lignin being generally hydrophobic, its hydrophobic nature is not sufficient to provide paperboard with a hydrophobic barrier (Antonsson *et al.* 2008). However, the derivatives, blends, and composites of lignin have achieved promising results as far as water resistance (Antonsson *et al.* 2008; Spiridon *et al.* 2010; Bhat *et al.* 2013; Hult *et al.* 2013a,b). Furthermore, while examining acetylated lignin blended with PHB, lignin was found to lower the crystallinity of PHB and to enhance its thermal stability in TGA analysis by increasing  $T_{5\%}$  22 °C,  $T_{50\%}$  19 °C, and  $T_{\max}$  17 °C (Bertini *et al.* 2012). In addition, Mousavioun *et al.* (2010) studied PHB/bagasse soda lignin (up to 40 wt%) blends. Soda lignin enhanced the overall thermal stability of the PHB matrix. Kovalcik *et al.* (2015) made films from poly(3-hydroxybutyrate-co-3-hydroxyvalerate) (PHBV) together with lignin (up to 10 wt%). The addition of lignin enhanced the thermo-oxidation stability as well as oxygen and carbon dioxide resistance of the films. These films were recommended to be used as packaging materials (Kovalcik *et al.* 2015). Shankar *et al.* (2015) studied agar/lignin films where the lignin content was 1wt% to 10wt%. The authors noticed a positive difference in water vapor barrier, UV-light barrier, and mechanical properties compared to the neat agar films. They proposed food packaging film end-use for the agar/lignin films due to their UV-light barrier properties (Shankar *et al.* 2015). Antimicrobial activity against Gram (+)ve and Gram (-)ve bacteria of chitosan/lignin films has also been examined. Clear activity against the bacteria was detected and found suitable for active food packaging applications (Rai *et al.* 2017).

Antioxidants can be used in active packaging and as a product protector against oxygen and gas. In this context, owing to the polyphenolic structure of lignin, its antioxidant behavior has been examined (Gordobil *et al.* 2014; Ye *et al.* 2016). Domenek *et al.* (2013) studied antioxidant activity of two different alkali lignins blended with a PLA matrix. As a result, the oxygen barrier properties were improved while the mechanical properties decreased slightly (Domenek *et al.* 2013). Indeed, some PLA/lignin blends have resulted into decreased mechanical performance, including tensile strength and elongation. Furthermore, Chung *et al.* (2013) enhanced the UV barrier properties of PLA by blending in 10 wt% of synthesized lignin-g-PLA copolymers. Moreover, Gordobil *et al.* (2014) studied two types of lignins at different content blended together with PLA. In the study, commercial alkaline lignin and almond shell extracted lignin were acetylated and compared. Finally, the addition of both lignins enhanced thermal stability, yet the crystallinity of PLA remained unchanged. Both acetylated lignins seemed to inhibit the hydrolytic degradation of PLA. In terms of mechanical properties, the elongation at break was enhanced with the addition of lignins, yet the other mechanical properties did not improve. If the lignin was not acetylated, the mechanical properties decreased (Gordobil *et al.* 2014).

Lignin has been reported to improve thermal stability, tensile strength, and hydrophobicity in starch-lignin blends. However, it has also been reported to impair elongation and transparency (Spiridon *et al.* 2010; Miranda *et al.* 2015). Indeed, lignin has been used as a filler in thermoplastic starch (TPS) blends. It has been reported to enhance mechanical and thermal properties, as well as moisture resistance (Gordobil *et al.* 2014). In addition, lignin has been reported to enhance plasticity in starch/lignin blends, to reinforce cellulose and to work as a compatibilizer in cellulose/starch blends. Bhat *et al.* (2013) improved the properties of starch-based films through the addition of 1% to 5% lignin. The main improvement was in water and water vapor resistance and in the heat seal-ability of the film. The film was seen suitable for food packaging

applications. Wu *et al.* (2009) studied cellulose/starch/lignin films and found that the content of both lignin and cellulose influence the mechanical properties of the film.

Unmodified lignin decreased the mechanical properties when blended together with thermoplastics (Chung *et al.* 2013). More specifically, lignin addition into polyolefins decreased the elongation at break, while had no effect on tensile strength. Blends with more compatible polymers, *e.g.*, polyesters, can result in enhanced strength, stress at yield, and Young's modulus (Pouteau *et al.* 2004). Modifications, such as esterification, etherification, and graft polymerization, enhance several properties of lignin/thermoplastics –dispersions (Chung *et al.* 2013). Likewise, the usage of additives (*e.g.*, plasticizers) have been reported to improve the reinforcing effect of lignin (Pouteau *et al.* 2004).

## BARRIERS BASED ON MICROORGANISM-DERIVED BIOPOLYMERS

PLA, PBS, and PHAs are all biodegradable thermoplastic polyesters, and each of them can be produced from biomass-based raw materials by fermentation (Gorrasi *et al.* 2008; Xu and Guo 2010; Bhatia *et al.* 2012; Bugnicourt *et al.* 2014; Harmsen *et al.* 2014). These potential biopolymers ARE bio-based alternatives to petroleum-based thermoplastics (Bhatia *et al.* 2012; Bugnicourt *et al.* 2014; Rastogi and Samyn 2015). All of these biopolymers are found in industrial scale production (Rizzarelli and Carroccio 2009; Rabu *et al.* 2013; Bugnicourt *et al.* 2014). The production routes of these materials are presented in Fig. 4.

Different blends and composites have been studied both in order to improve mechanical, chemical, and thermal properties and to reduce the cost of these polymers (Dufresne *et al.* 2003; Lin *et al.* 2011; Bhatia *et al.* 2012; Gorrasi *et al.* 2014). In the following sections, PLA, PBS, and PHAs are introduced, together with their different blends and composites.

### Poly lactide

Poly lactide (PLA) is a broadly available aliphatic and thermoplastic biopolyester (Liu 2006; Rhim *et al.* 2009; Bugnicourt *et al.* 2014; Rastogi and Samyn 2015) that was commercialized in the early 90s (Tang *et al.* 2012; Rabu *et al.* 2013). PLA is obtained from lactic acid, which is a bacterial fermentation product of starch-rich products, *e.g.*, corn, sugarcane (Yu *et al.* 2006; Gorrasi *et al.* 2008; Rhim *et al.* 2009; Papageorgiou *et al.* 2010; Tang *et al.* 2012; Rabu *et al.* 2013; Reddy *et al.* 2013), sulphite liquors, agro-wastes (Rastogi and Samyn 2015), or food industry wastes (Andersson 2008). It has been reported that with 1.6 kg of sugars, 1.0 kg of PLA can be obtained (Reddy *et al.* 2013). Lactic acid (LA) is the building unit of PLA and it exists as L- and D-lactic acid enantiomers. The most common stereoisomers of PLA are poly(L-lactide) (PLLA), poly(D-lactide) (PDLA), and poly(DL-lactide) (PDLLA) (Farah *et al.* 2016). The PLA can be produced *via* lactic acid polycondensation or *via* lactide ring-opening polymerization (Rhim *et al.* 2009; Rabu *et al.* 2013). The principles for the production process are presented in Fig. 4. Commercial, high molecular weight PLA is produced *via* the ring-opening method (Andersson 2008; Papageorgiou *et al.* 2010).

PLA is biodegradable yet also compostable and recyclable (Andersson 2008; Picard *et al.* 2011; Golden and Handfield 2014). PLA has good mechanical (Yu *et al.* 2006; Arora and Padua 2010; Papageorgiou *et al.* 2010; Bhatia *et al.* 2012) and moisture



barrier properties (Weber *et al.* 2002; Liu 2006; Shalini and Singh 2009), great transparency and printability (Arrieta *et al.* 2014a), and it has shown promising results as a barrier of hydrophobic aroma compounds (Ducruet *et al.* 2011). PLA has great processability (Rhim *et al.* 2009; Arrieta *et al.* 2014a; Golden and Handfield 2014; Rastogi and Samyn 2015) with several techniques, for example by extrusion, thermoforming (Picard *et al.* 2011) and blow molding (Turalija *et al.* 2016); in addition to these applying techniques bar coating and solution casting have been used (Rastogi and Samyn 2015). Commercialized PLA is currently used for several packaging applications (Weber *et al.* 2002) and food service products (Andersson 2008; Fortunati *et al.* 2012; Rabu *et al.* 2013; Reddy *et al.* 2013).

Challenges in PLA utilization include its high brittleness (Harada *et al.* 2007; Andersson 2008; Kuusipalo *et al.* 2008; Rhim *et al.* 2009; Vroman and Tighzert 2009; Ducruet *et al.* 2011; Bhatia *et al.* 2012; Ojijo *et al.* 2012; Tang *et al.* 2012; Chung *et al.* 2013; Gordobil *et al.* 2014), relatively low thermal stability (Yu *et al.* 2006; Rhim *et al.* 2009; Vroman and Tighzert 2009; Fortunati *et al.* 2012; Tang *et al.* 2012; Chung *et al.* 2013; Gordobil *et al.* 2014), poor gas barrier properties (Andersson 2008; Rhim *et al.* 2009; Arora and Padua 2010; Picard *et al.* 2011; Johansson *et al.* 2012; Chung *et al.* 2013; Gordobil *et al.* 2014; Rastogi and Samyn 2015), low resistance against UV light (Chung *et al.* 2013) and, relatively high cost (Arora and Padua 2010; Papageorgiou *et al.* 2010; Bhatia *et al.* 2012; Chung *et al.* 2013; Golden and Handfield 2014; Gordobil *et al.* 2014; Gorrasi *et al.* 2014). In addition, the degradation rate of PLA is rather slow (Golden and Handfield 2014; Farah *et al.* 2016; Turalija *et al.* 2016) and reactive side-chain groups are desirable for this purpose (Golden and Handfield 2014; Farah *et al.* 2016).

In order to improve properties or to lower the price of the product, efforts have been made by blending and developing composites with other polymers and fillers (Bhatia *et al.* 2012) as well as other additives, such as thermal stabilizers and plasticizers (Gorrasi *et al.* 2014). For example, the brittleness of PLA has been addressed by plasticization, blending with tough polymers, and rubber toughening (Nampoothiri *et al.* 2010). Plasticization with 10-20 wt% ester-like plasticizers, such as poly(ethylene glycol, PEG), is effective but lowers the stiffness. When added up to 10 wt%, impact modifiers, such as ethylene-based copolymers, reduce the brittleness and maintain acceptable levels of stiffness. The main disadvantage has been their non-biodegradability, which have limited their use in large volumes. Biodegradable options for impact modifiers of PLA have been introduced, including poly( $\epsilon$ -caprolactone, PCL), poly(-butylene succinate, PBS) and poly(-R-3-hydroxybutyrate, PHB). The amount required to reach acceptable toughness has been reported to be 20 to 40wt% (Notta-Cuvier *et al.* 2014). The PLA based coatings are widely used in food-packaging where food safety is the major driver, for example, the migration of additives and toxicological properties of the blend need to be checked (Johansson *et al.* 2012).

#### *PLA/filler composites*

Many studies have involved layered silicates and reported to improve gas barrier properties by 50% to 60% (23 °C, RH 50%). However, for products with a high barrier demand, the improvement is not enough (Johansson *et al.* 2012). Several other filler options have been studied in addition to layered silicates, such as metal oxides (*e.g.*, TiO<sub>2</sub>, ZnO), carbon nanotubes, and metal nanoparticles (*e.g.*, Ag, Au) to achieve

improvements, for instance, in mechanical, thermal, gas barrier, antibacterial, and UV barrier properties (Papageorgiou *et al.* 2010; Gorrasi *et al.* 2014).

Several researchers have reported improvements in barrier and mechanical properties of biodegradable materials by 1.0 wt% to 5 wt%, by adding montmorillonite (MMT) (Sanchez-Garcia and Lagaron 2010). Picard *et al.* (2011) mixed PLA together with 4 wt% organically modified montmorillonite (OMMT), subsequently achieving enhance gas barrier properties. Ojijo *et al.* (2012) studied the blend of (PLA/PBSA)-organically modified clay composite. The ratio of polymers was kept constant at 70:30 (PLA/PBSA), while organically modified clay content was varied between zero wt% and 9 wt%. As a result, an improvement of 29% in elongation was obtained by a blend of PLA/PBSA/2 wt% organically modified clay compared to plain PLA. Moreover, other mechanical properties were also good in the blend, and reduction in thermal stability was only moderate (Ojijo *et al.* 2012). Rhim *et al.* (2009) investigated how different nanoclay types and concentrations in PLA films influence the mechanical, barrier, and antimicrobial properties of the film. The behavior varied among the types of nanoclays and concentrations. Promising improvement against water vapor (6% to 33% decrease) was noticed with organically modified clay. However, tensile strength and elongation decreased due to the filler addition (Rhim *et al.* 2009). While blending 5 wt% of organo-modified mica-based clay grade with PLA, Sanchez-Garcia and Lagaron (2010) achieved 54% lower water permeability, 55% lower oxygen permeability, a 75% decrease in UV light transmission (wavelength 250 nm), and a 32% reduction in visible light transmission (wavelength 650 nm) than with neat PLA. Gorassi *et al.* (2014) obtained upgraded water vapor barrier properties by mixing 6 wt% and 12 wt% of silane-treated halloysite nanotubes (HNT) with PLA matrix. Filler content was the main factor influencing the barrier properties. One filler option is the main byproduct, gypsum, from the PLA production. Gypsum is a calcium sulfate and can improve the mechanical properties of PLA. Furthermore, the filler size and content had the biggest influence on the water vapor resistance of a PLA/calcium sulfite hemihydrate biocomposite (Gorrasi *et al.* 2008). A good compatibility between PLA and modified kaolinite have been achieved, which resulted in improved oxygen resistance (50%) (Arora and Padua 2010). Due to the addition of nanoclays, PLA/nanoclay and PLA/PCL/nanoclay films have been reported to have improved oxygen and water vapor barrier properties (Andersson 2008).

Fortunati *et al.* (2012) achieved a 34% improvement in water vapor barrier properties with PLA matrix together with 1 wt% surfactant modified cellulose nanocrystals (s-CNC). In addition, they obtained good gas barrier properties and low overall migration levels in solvent casted PLA/s-CNC and PLA/unmodified cellulose nanocrystal (pristine, CNC) biocomposite films. Active food packaging materials from PLA, together with 1.0 wt% CNC and 1.0 wt% silver (Ag) nanoparticles, indicated a 46% reduction in the oxygen transmission rate. Similar improvement was obtained with a biocomposite of PLA/5wt% CNC/1wt% Ag. Fortunati *et al.* (2013) have reached acceptable levels in overall migration with PLA-based CNC/Ag-biocomposites, but they noticed that silver was easily released from them. By applying alcohols, Turalija *et al.* (2016) modified PLA surface properties to make it more hydrophilic and antimicrobial. Glycerol and polyethylene glycerol, chitosan, and silver nanoparticles were also considered. Enhancement in hydrophilicity with alcohols and chitosan decreased the water contact angle from 70° to 40-45°. Silver was applied on PLA-based films by plasma deposition. Turalija *et al.* (2016) noticed increased hydrophilicity and a noteworthy enhancement in the antimicrobial properties of PLA-based films with silver.

PLA/30 wt% flax fiber composites with different additives, *e.g.*, kraft lignin, have also been studied. Good impact strength was achieved with (NaOH-treated) flax fiber addition, further enhanced with < 3wt% addition of pine kraft lignin (however, higher addition levels weakened the mechanical properties of the composite) (Johansson *et al.* 2012).

#### *PLA/ biopolymer blends*

PLA's crystallinity was increased by mixing 25 wt% of poly(hydroxybutyrate) (PHB) and 5 wt% CNC. In addition, PHB enhanced the oxygen and water barrier properties, although it simultaneously impaired the transparency of PLA (Arrieta *et al.* 2014a).

PLA/starch blends have been a topic of study (Yu *et al.* 2006; Johansson *et al.* 2012; Tang *et al.* 2012). Starch is a renewable and biodegradable hydrophilic polymer, which has been a common material in bioplastics (Yu *et al.* 2006). In order to reduce price and to enhance biodegradability, the aim has been to blend PLA together with starch (Johansson *et al.* 2012; Tang *et al.* 2012). Several studies have been carried out with different starches and additives, such as native corn starch together with a plasticizer, corn starch *vs.* high-amylose corn starch and gelatinized starch with water/glycerol (Tang *et al.* 2012). About 30 wt% to 50 wt% starch has been blended with PLA, subsequently causing a reduction in mechanical properties, such as in tensile strength and strain at break (Johansson *et al.* 2012). The crystallization rate of PLA has been increased with talc (1 volume %) and with starch (1.0 to 40% volume) addition. However, there are challenges involving the hydrophobic nature of PLA and the hydrophilic nature of starch, thus causing weak cohesion to each other and resulting in poor properties without additives or compatibilizers (Yu *et al.* 2006).

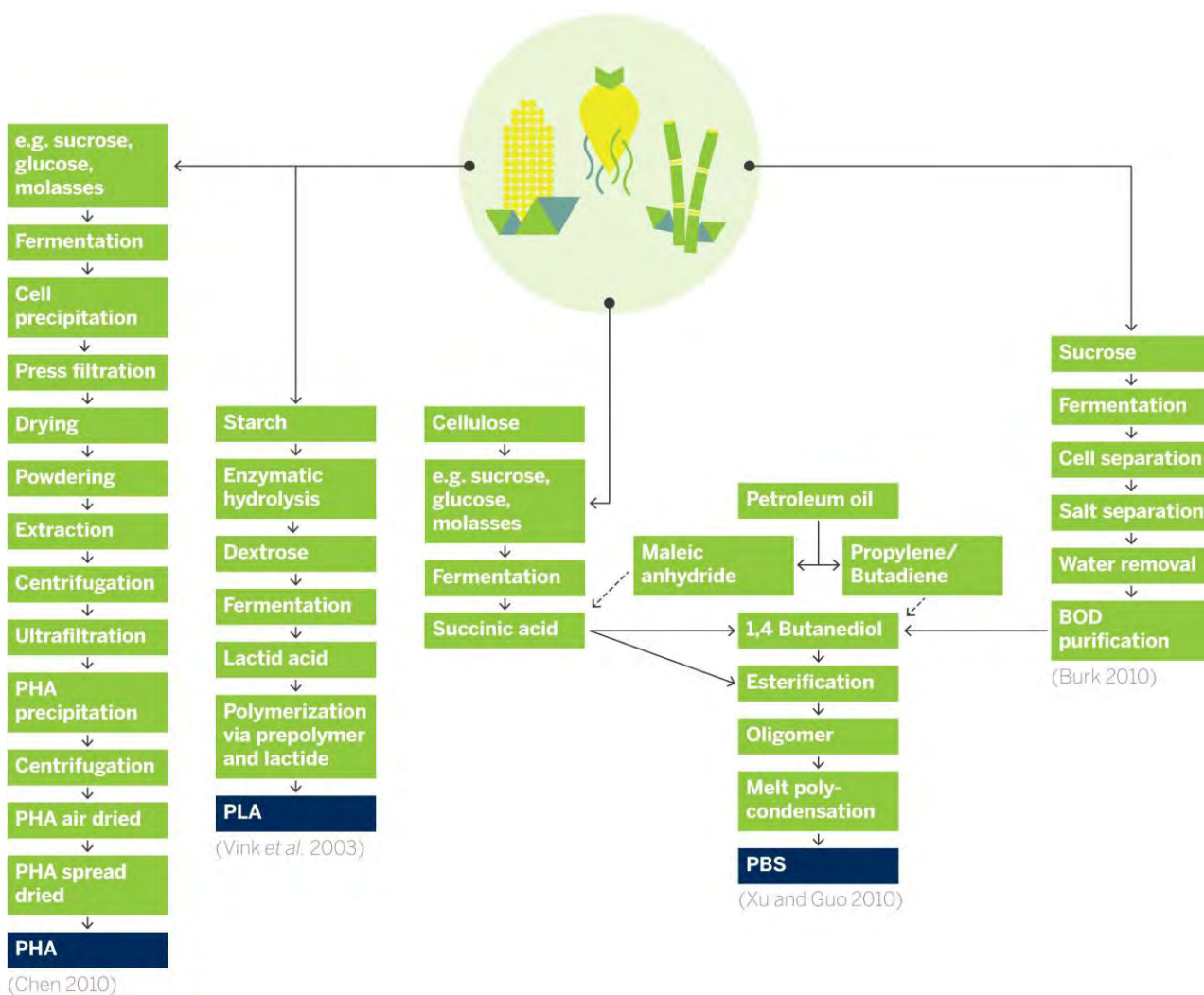
Blending of enantiomeric polymers was reported to enhance thermal properties. Blending poly(L-lactide acid) (PLLA) together with poly(D-lactide acid) (PDLA) improved thermal stability compared to PLLA or PDLA alone. They achieved a 50 °C higher melting temperature by making the blend of PLLA/PDLA (Yu *et al.* 2006). The L and D isomers also have an effect on the crystallinity and mechanical properties of the polymer. High crystallinity can be achieved with L-form and amorphousness with copolymers of D and L isomers (Andersson 2008).

PLA has been blended and copolymerized with biodegradable poly(caprolactone) (PCL) to decrease brittleness and improve the mechanical properties (Tang *et al.* 2012), such as impact strength. The PLA-based urethane blend was reported to increase impact strength when used as an additive. In comparison, PLA/ poly(butylene adipate-co-terephthalate) (PBAT) enhanced impact strength when the concentration on PBAT was 20 wt%. Harada *et al.* (2007) mixed PLA with PBS (90/10 wt%) by using a reactive processing agent, lysine triisocyanate (LTI). As a result, the impact strength was enhanced, from 18 kJ/m<sup>2</sup> to 50-70 kJ/m<sup>2</sup> at a LTI loading of 0.5 wt% (Harada *et al.* 2007).

#### **Poly(butylene succinate)**

Poly(butylene succinate) (PBS) is one of the most promising environmental-friendly aliphatic polyesters, which offers a great alternative for common polyolefins (Bhatia *et al.* 2012; Phua *et al.* 2012; Wang *et al.* 2013; Charlon *et al.* 2015). PBS was developed in Japan in the early 1990's by Showa Polymer (Vroman and Tighzert 2009; Phua *et al.* 2012). The PBS is commonly produced from succinic acid and 1,4-butanediol

(BDO) (Xu and Guo 2010; Bhatia *et al.* 2012). PBS can be manufactured fully or partially from renewable resources (Xu and Guo 2010), even though currently it is polymerized partially fossil based monomers in the industrial scale (Yim *et al.* 2011). The major steps for PBS production are included in Fig. 4. Renewable succinic acid is manufactured by bacterial fermentation. Possible raw materials include, for example starch, glucose, xylose (Xu and Guo 2010), or agricultural waste (McKinlay *et al.* 2007). By similar means, renewable BDO can be produced *via* the fermentation of sugars (Harmsen *et al.* 2014). Currently, the prevalent production method of BDO is still from oil-based feedstocks (Yim *et al.* 2011).



**Fig. 4.** Block diagram to illustrate the production of biobased barriers from biomass fermentation (see Burk 2010; Chen 2010; Xu and Guo 2010)

The PBS exhibits good mechanical strength, great processability with traditional polyolefin processing equipment (Wang *et al.* 2013), high elasticity and a reasonably high thermal and chemical resistance (Lin *et al.* 2011; Bhatia *et al.* 2012; Phua *et al.* 2012). In addition to biodegradability in soil and aquatic environments, PBS is also

compostable (Nam *et al.* 2011). Kanemura *et al.* (2012) observed that both the recycling of PBS is possible as well as the mechanical properties of PBS improve after reprocessing. A typical way to produce PBS film and coating is by melt extrusion casting (Wang *et al.* 2013).

It might occur that, for high barrier demand products, the soft PBS material is not effective enough (Lin *et al.* 2011; Phua *et al.* 2012; Charlon *et al.* 2015). Moreover, the gas barrier properties of PBS are barely sufficient for gas sensitive products (Lin *et al.* 2011; Phua *et al.* 2012; Zhou *et al.* 2016). Melt viscosity and a relatively high price are also limiting the use of PBS (Lin *et al.* 2011). Furthermore, PBS has been studied as component in composites or blends, for example, to improve gas barrier (Bhatia *et al.* 2012; Boonprasith *et al.* 2013; Charlon *et al.* 2015; Zhou *et al.* 2016), mechanical, and thermal properties (Lin *et al.* 2011).

#### *PBS/filler composites*

PLA/PBS/nanoclay is a good example of related composites, where a lower oxygen permeability level is obtained through the addition of the clay. PBS (20 wt%) was found to increase the water vapor permeability (WVP) of PLA (80 wt%). Thus, the addition of nanoclay offered an 18% improvement in WVP (Bhatia *et al.* 2012). PBS/thermoplastic starch (TPS) blend with added nanoclays decreased the OTR and WVTR values significantly (Boonprasith *et al.* 2013). The improvement of gas barrier by adding clays or nanoclays relates to the tortuous path the gas molecules need to travel through the barrier (Zhou *et al.* 2016).

Lin *et al.* (2011) blended 2 wt% and 5 wt% of cellulose nanocrystals (CNC) and starch nanocrystals (SN) separately with PBS. They improve the strength and elongation properties of the PBS matrices by both fillers. PBS/2 wt% CNC enhanced tensile strength by 11% and elongation by 17%, while PBS/5 wt% SN enhanced tensile strength by 8% and elongation by 28% (Lin *et al.* 2011). Someya *et al.* (2004) investigated different types of organo-modified montmorillonites blended with PBS. They found out that organo-modified montmorillonites promoted crystallization of PBS and therefore acted as nucleating agent. The addition of 3 wt% organo-modified montmorillonites resulted greater tensile and flexural modulus and weaker the tensile strength with most of the clay-types (Someya *et al.* 2004).

Moreover, PBS has been blended with agro-fillers, such as wood and rice husk flour, wheat straw, and bagasse. Kim *et al.* (2005) studied the PBS/agro-filler blends in filler concentrations of 10, 20, 30, and 40 wt%, by preparing a blend of PBS/rice husk flour and PBS/wood flour. As a conclusion, the mechanical properties deteriorated when filler content was increased, whereas the reduction of filler particle size caused the tensile strength properties to increase moderately. Moreover, the tensile strength of the wood flour filled blend was higher than in the rice husk flour filled blend. Liu *et al.* (2009) studied the biodegradability of PBS/jute fiber composites. The composites containing 10, 20, and 30 wt% of jute fibers degraded faster than the plain PBS film or the plain jute fiber. The most significant weight loss (62.5% in 180 days) in a compost soil burial test was performed by the PBS/10 wt% jute fiber composite (Liu *et al.* 2009). Nam *et al.* (2011) studied PBS/alkali-treated coir fiber composites with a coir fiber concentration of 10 wt% to 30 wt%. As a result, they found the highest improvement in mechanical properties with an alkali-treated coir fiber concentration of 25 wt%. In the particular composite, the tensile strength, tensile modulus, and the flexural modulus increased by 54%, 141.9%, and 97.4%, respectively, in contrast to plain PBS.



### *Poly(butylene succinate-co-adipate)*

Poly(butylene succinate-co-adipate) (PBSA) is a random copolymer of PBS. Due to its flexibility of polymer chains and lower crystallinity and, it is more sensitive to biodegradation than PBS (Ray *et al.* 2007a,b). Ray *et al.* (2005) blended PBSA together with (3, 6, and 9 wt%) organically-modified clay to improve the mechanical properties (stiffness, and elongation), thermal stability of PBSA. The same authors investigated a PBSA/ organically modified synthetic fluorine mica (OSFM) blend (Ray *et al.* (2007a). An improvement in mechanical properties, such as in elastic modulus, as well as in thermal stability was noted. Ray *et al.* (2007b) also studied the morphology of a blend of 5 wt% organo-modified montmorillonite in poly(propylene) PP/PBSA (80:20) matrix. After the clay addition, the blend displayed a co-continuous structure and a lower viscosity ratio of the blend matrices. Likewise, improvements were noticed in mechanical, thermal, and rheological properties. Chen and Yoon (2005) produced twice-functionalized organoclays (TFC) and blended them with PBSA. They reported to increase the linear storage modulus of the blend compared to the organoclay /PBSA blend. In addition, PBSA/TFC blends displayed an improved tensile modulus and strength at break.

### *PBS/biopolymer blends*

PBS has been blended with several bio-based polymers, such as cellulose, cellulose acetate, cellulose whiskers, starch, starch nanocrystal, chitosan, silk, plant- and red algae fibers, PLA, and PHAs (Lin *et al.* 2011). The PBSA/ starch blend (5 wt% to 30 wt%) has been investigated. In the study, it was found that starch addition did not considerably decrease mechanical properties, although the addition noticeably increased the degradation properties of the blend, starting from 5 wt% addition. Starch /PBS and starch/PBSA blends have been used in food packaging applications, for instance, as biodegradable biscuit trays or films (Tang *et al.* 2012).

### **Poly(hydroalkanoates)**

Poly(hydroalkanoates) (PHAs) are a diverse group of linear thermoplastic biopolyesters (Liu 2006; Thellen *et al.* 2008; Bugnicourt *et al.* 2014). PHAs are naturally synthesized *via* bacterial fermentation under physiological stress (Liu 2006; Misra *et al.* 2006; Esteban *et al.* 2008; Johansson *et al.* 2012; Bugnicourt *et al.* 2014; Rastogi and Samyn 2015). PHA is obtained from the bacteria by extraction, which is followed by drying and powder or resin formation (Kuusipalo *et al.* 2008). The PHAs function as bacterial carbon and energy storage and their concentration can be from a marginal amount up to more than 80% of their cell dry mass, depending on the bacteria (Valentin *et al.* 1999; Esteban *et al.* 2008; Koller 2014). Sugar and glucose are common fermentation raw materials for the industrial production of PHAs. In addition to carbohydrates, lipids, such as vegetable oil and glycerin, have also been considered (Bugnicourt *et al.* 2014). Different types of wastes and wastewaters have been used for the production of PHAs (Bugnicourt *et al.* 2014; Rastogi and Samyn 2015). Some main processes used for PHA production are shown in Fig. 4.

There are various monomer components enabling versatile properties and application fields of PHAs (Valentin *et al.* 1999; Liu 2006; Koller 2014). In addition to the structural variations, the fermentation process and its carbon source also affect the properties of PHAs (Liu 2006). The dominant and simplest polymers from the group of PHAs are poly( $\beta$ -hydroxybutyrate) (PHB) (Dubief *et al.* 1999; Liu 2006; Yu *et al.* 2006;

Tang *et al.* 2012; Arrieta *et al.* 2014a; Bugnicourt *et al.* 2014; Follain *et al.* 2014; Rastogi and Samyn 2015) and its copolymer poly(3-hydroxybutyrate-co-3-hydroxyvalerate) (PHBV) (Pardo-Ibáñez *et al.* 2014).

PHAs have been shown to be renewable and biodegradable under anaerobic and aerobic environments and are compostable biopolymers (Bugnicourt *et al.* 2014). PHA is a promising oxygen, water vapor, and UV-light barrier material (Bugnicourt *et al.* 2014; Rastogi and Samyn 2015). Compared to PLA, PHAs provide a better UV light barrier (Arrieta *et al.* 2014a). In addition, PHAs display better WVTR properties compared to other common extrudable biopolymers, such as PLA and PBS (Kuusipalo *et al.* 2008). Furthermore, there have been studies about the biodegradability of PHAs compared to other biopolymers, and this order was reported as PHB > PBS > PLA (Bugnicourt *et al.* 2014). PHAs display good film formability and coating ability (Tang *et al.* 2012). PHAs have been processed by several different techniques, such as extrusion, injection (Bugnicourt *et al.* 2014; Koller 2014) and compression molding (Rastogi and Samyn 2015), thermoforming (Koller 2014), solvent and spin casting (Thellen *et al.* 2008). PHBV-coated paperboards have been reported to handle creasing and to be heat sealable to itself and to paperboard within a temperature range of 190 °C to 230 °C (Andersson 2008). PHAs have been utilized as surface-sizing agents on paper with promising results on mechanical properties and the water resistance of the paper (Rastogi and Samyn 2015). PHB has also been used in other applications, *e.g.*, food and other types of packaging (Weber *et al.* 2002), in the manufacture of ropes, bank notes, and cars (Reddy *et al.* 2013) and in biomedical products (Misra *et al.* 2006).

Challenges of PHAs involve, for example, the production cost (Valentin *et al.* 1999; Weber *et al.* 2002; Dufresne *et al.* 2003; Liu 2006; Andersson 2008; Gandini 2008; Kuusipalo *et al.* 2008; Mousavioun *et al.* 2010; Tang *et al.* 2012; Bugnicourt *et al.* 2014), low acid and base resistance, poor thermal processability (Rastogi and Samyn 2015), and the fact that the raw materials that are currently used compete with food sources (Bugnicourt *et al.* 2014). The PHAs have weak thermal stability above the melting point (~175 °C), although this aspect can be controlled by using additives (Johansson *et al.* 2012). Due to PHAs' tendency to be brittle, they are often blended with additives or other polymers (Bugnicourt *et al.* 2014). Moreover, there is potential for improvement in terms of the gas barrier properties of PHAs (Andersson 2008; Tang *et al.* 2012). PHAs have been blended with other polymers and fillers to enhance their properties and to reduce the cost (Dufresne *et al.* 2003; Yu *et al.* 2006; Mousavioun *et al.* 2010).

#### *PHAs/filler composites*

Sanchez-Garcia and Lagaron (2010) investigated PHBV/organomodified clay composite. Compared to neat PHBV, its composite with 5 wt% clay resulted in a reduction of permeability levels to water (by 76%), to oxygen (by 32%), and to oil (limonene) (by 78%). The PHB/nanoclay composites have been studied but challenges still exist in the formation of the composite material due to PHB degradation behavior and instability. Improvements in mechanical and thermal properties have been achieved by combining nanoclay with PHB (Tang *et al.* 2012).

Pardo-Ibáñez *et al.* (2014) improved the barrier properties of PHBV by adding keratin fibers. The PHBV with 1.0 wt% of keratin fiber blend improved water, limonene, and oxygen barrier properties as well as elastic modulus compared to pure PHBV. Dufresne *et al.* (2003) applied cellulose flour (up to 70 wt%) as reinforcement filler into a PHBV polymer matrix in order to reduce its price while still maintaining its

biodegradability. They observed poor interaction between the filler and PHBV, poor mechanical properties, and an increased degree in crystallinity. Nevertheless, the mechanical properties were enhanced at higher temperatures (above glass–rubber transition of PHBV). Dubief *et al.* (1999) studied nanocomposites, where the matrix polymer was poly( $\beta$ -hydroxyoctanoate) (PHO) and nanofillers were starch microcrystals and cellulose whiskers. As a result, the addition of nanofillers enhanced the mechanical properties of the matrix polymer. The PHB has been used in plasticized PLA/CNC nanocomposite to increase the crystallinity of PLA. Consequently, the resulting composites were transparent and compostable (Arrieta *et al.* 2015).

#### *PHAs/biopolymer blends*

The mechanical and thermal properties as well as the processability of PHB were enhanced by low and high molecular weight plasticizers. By adding 20 wt % plasticizer (blend of dioctyl phthalate and di-2-ethylhexylphthalate), Erkske *et al.* (2006) achieved enhanced strength, elongation, and decreased brittleness. In addition, the melting temperature was lowered and, overall, the processing window was expanded considerably. The authors also added 20-60 wt% of starch to a PHB/ plasticizer blend. The elongation and strength properties of the composite decreased by increasing the starch content, whereas water vapor barrier increased (the optimal starch content 25 to 40 wt%).

PHB has been blended with starch-adipate and grafted starch-urethane derivatives, resulting in limited mechanical properties (Tang *et al.* 2012). In another study, PHB was blended with starch acetate (SA) to change the crystallization, *e.g.*, lower temperature and enthalpy of non-isothermal crystallization of PHB. PHB/SA blends were found to be immiscible (Zhang *et al.* 1997).

PHB was blended with cellulose propionate (CP), which resulted in higher ductility. The components of the PHB and CP blend were miscible. Likewise, a miscible blend of PHB and cellulose acetate butyrate (CAB) expanded the processability of the system by lowering the degree of crystallinity and the melting temperature. Additional studies have considered chitin and chitosan blended with PHB (Yu *et al.* 2006). Ikejima and Inoue (2000) compared PHB/chitin and PHB/chitosan blends and found an improvement in the biodegradability compared to neat chitosan and chitin. In addition, they observed that 25wt% PHB containing PHB/chitin blend degraded more rapidly than neat PHB or chitin as a result of decreased crystallinity of PHB.

A PHB/poly(hydroxybutyrate-co-hydroxyhexanoate) (PHBHHx) blend was found to strengthen the elongation at break significantly when the PHBHHx content in the blend was increased from 40% to 60% (Zhao *et al.* 2003). The PLA/PHB films were brittle and rigid without plasticizing with, for example, poly(ethylene glycol) (PEG). The PLA/PHB based films in the Arrieta *et al.* (2014b) study displayed a compostable character. Abdelwahab *et al.* (2012) studied PLA/PHB blends with a plasticizer (5 wt% and 7 wt%). The elongation at break increased by the addition of the plasticizer. Olkhov *et al.* (2003) investigated PHB/poly(vinyl alcohol) (PVA) blends, where the PHB content varied from 0% to 100%. The highest water vapor permeability was observed when the PHB content was 40 wt%.

## PROSPECTS

Barrier materials have an important role in different types of packaging applications, considering the protection from the environment and shelf life that they provide. Conversely, in some instances, the barrier material protects the environment from the product. Several barrier materials in today's industry are manufactured either from oil resources or metals. Driven by both the increase in environmental awareness, as well as the resource scarcity, novel and environmentally benign alternatives have been sought. This review covered wood and fermentation-based materials, which display interesting barrier properties and offer a potential for utilization in packaging.

Wood-based products have received great attention in a wide variety of fields. Hemicellulose-based barriers have been reported to display resistance against oil, grease, aroma and oxygen. Moreover, hydrophilic hemicelluloses offer promising barrier properties, and are easily modified. For polysaccharides streams, the main challenges include their mechanical properties, processability, and low water resistance. A relatively weak heat stability and narrow processing window may be challenging, and lower running speeds may be needed compared to oil based alternatives. Also, the adhesion may be challenging, depending on the substrate. Derivatives of cellulose render it soluble in various solvents that are widely used in industry. Their thermal properties and facile application on surfaces are great advantages. However, a more detailed discussion is needed, especially if focused on processing of eco-friendly barrier films at speeds relevant to industrial production.

A few essential variables are critical when considering cellulose derivatives and structure-property-process relationships for upscaling novel cellulose derivatives. These variables include the degree of substitution that is linked with water vapor permeability. The reaction conditions of cellulose derivatives regulate their DS, DP, and final properties, which include film-forming and barrier properties. There is interest for developing novel cellulose derivatives, due to a trend aiming for methods to replace petroleum-based packaging materials. Firstly, there have been suggestions for blends with other materials that compensate the poor WVP and enhance other barrier properties. Simultaneously, novel cellulosic solvents may bring promise, together with homogeneous modification methods, for increased regioselectivity leading to improved and more end-use-specific properties of the cellulose derivatives.

The interest towards lignin has been based on its availability and cost as well as its possibilities for chemical modification. Considering potential barrier applications, the properties of interest in lignin include its antioxidant behavior and UV-light resistance. However, challenges with technical lignins involve its poor film-forming properties and dark color.

There have been several publications covering heat treatment of nanocellulose. The crystallinity is important for oxygen permeability, whereas material density is decisive for water vapor permeability. In addition, the combination of hydrophobic, thermoformable matrices with nanocellulose is under study, and some promising solutions exist regarding compatibility, but also challenges, such as the low WVP of nanocellulose. The economic aspects and upscaling potential in nanocellulose production has been reported, and a few nanocellulose-containing end-use products are already commercialized. Nevertheless, several challenges remain to be resolved prior to large-scale industrial production, such as the hygroscopic tendency of nanocellulose to absorb moisture from its surroundings, swelling, and others.

PLA, PBS, and PHAs are commercial, biodegradable biopolymers, with characteristics that are comparable to conventional petroleum-based thermoplastics. The challenge in considering these polymers is their relatively high price, low production volumes, and the more challenging processability compared to that of conventional polymers, *e.g.*, polyolefins. In addition, the most common bio-based raw materials for PLA, PBS, and PHAs production compete with food. However, different waste-streams, wastewaters and agro-wastes, have been studied as an alternative feedstock. The PLA, PBS, and PHAs offer adequate mechanical and barrier properties for many applications, specially for packaging. Nevertheless, in order to enable a more extended use of these biopolymers in the packaging industry, the price level should be comparable to that of conventional polymers. This means the need for cheaper raw materials, more efficient production processes, thinner barrier layers, or the development of cost-efficient blends or composites. Another powerful accelerator could be a change in the legislation considering packaging materials, *e.g.*, similarly to European directive of single use plastics or French ordinance Décret n° 2016-1170 (2016).

For both wood-based or microorganism-based biobarriers, the incorporation of inorganic and mineral nano- and microparticles offer interesting prospects, for example, to develop antibacterial, thermal resistance and other properties (Hoseinnejad *et al.* 2018; Wang *et al.* 2018). For instance, high-barrier and fully biodegradable food packaging materials have been achieved by coating PLA with glycol chitosan-clay nanocomposite (Habel *et al.* 2018). Likewise, major advances have been made with compositions to achieve UV protection (Niu *et al.* 2018) as well as scavenging and releasing activities, all relevant to food and pharmaceutical packaging, to extent the shelf life, for diagnostic, identification and communication (quality tracking, brand protection, *etc.*). This topic has been reviewed recently (Janjarasskul and Suppakul 2018) and is a subject of current research. In all these contexts, safety and regulatory aspects need careful attention.

While this review did not discuss the topic in detail, an interesting solution to biodegradability, to improve the properties of packaging materials, and to enhance barrier effects, is the use of multicomponent polymers and blends. This includes the synthesis of systems via interpenetrating networks (Bai *et al.* 2015). For related purposes, various compatibilizers, including those that can be adopted during melt processing, have been discussed (Muthuraj *et al.* 2018). Along similar ideas, proteins have been proposed as compatibilizer and eco-friendly dispersant in composites comprising cellulose nanofibrils and PLA (Khakalo *et al.* 2018). Another aspect of interest is the possibility to make patterned biobarriers, which can be useful to engineer surfaces in advanced applications (Guo *et al.*, 2018). Finally, the so-called “solvent welding” is being researched to tune the surfaces of biobased films, expanding the scope of properties and offering a possibility to facilitate multilayered structures (Reyes *et al.* 2019)

## CONCLUSIONS

Packaging materials are selected by end use requirements and they can be combined to fulfill given target properties. No single bio-based material will satisfy all potential markets or applications. Existing petroleum-based barrier solutions comprise products that have been developed over the course of several decades. The use of bio-based materials is not as matured and needs further development. Growing interest in designing packaging concepts include multilayer structures. PLA, PBS, and PHBs are



becoming material alternatives for green, food packaging while wood-derived materials, including hemicelluloses and nanocelluloses are in the earlier stages of their consideration.

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## Effects of rotational velocity and hold time at folding posture on time-dependent release behavior of creased white-coated paperboard

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In this work, a folding experiment was performed to investigate the time-dependent creasing characteristics of white-coated paperboard of 0.3mm thickness. After folding up to the tracking angle of 90° under a specified rotational velocity, the creased part was held for a chosen short time (0~20s) and the time-dependent release behavior of folding angle was experimentally investigated for the elapsed release time of 10s. When using the paperboard scored with a specified indentation depth, both the hold time of folded posture of creased part and the rotational velocity of fixture were varied. The folding angle of the paperboard was measured by a CCD camera of digital microscope and the bending moment resistance was measured by a load cell of bending test apparatus in the folding experiment. Through the experiment, it was found that the time-dependent release angle consisted of the hold time based intercept part and the creep-recovery based gradient part as a logarithmic function of the elapsed release time. When varying the folding velocity against a fixed unfolding velocity, the unfolded released behavior was isolated by the hold time from the first half folding velocity. Seeing the drop rate of bending moment at the tracking position and the dependency of initial release angle on the rotational velocity, a transient state and quasi-stationary state of bending moment relaxation were revealed.

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# Effects of rotational velocity and hold time at folding posture on time-dependent release behavior of creased white-coated paperboard

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## Abstract

In this work, a folding experiment was performed to investigate the time-dependent creasing characteristics of white-coated paperboard of 0.3mm thickness. After folding up to the tracking angle of 90° under a specified rotational velocity, the creased part was hold for a chosen short time (0~20s) and the time-dependent release behavior of folding angle was experimentally investigated for the elapsed release time of 10s. When using the paperboard scored with a specified indentation depth, both the hold time of folded posture of creased part and the rotational velocity of fixture were varied. The folding angle of the paperboard was measured by a CCD camera of digital microscope and the bending moment resistance was measured by a load cell of bending test apparatus in the folding experiment. Through the experiment, it was found that the time-dependent release angle consisted of the hold time based intercept part and the creep-recovery based gradient part as a logarithmic function of the elapsed release time. When varying the folding velocity against a fixed unfolding velocity, the unfolded released behavior was isolated by the hold time from the first half folding velocity. Seeing the drop rate of bending moment at the tracking position and the dependency of initial release angle on the rotational velocity, a transient state and quasi-stationary state of bending moment relaxation were revealed.

**Keywords :** Folding, Shear, Bending moment, Creasing, Relaxation, Creep, Paperboard

## 1. Introduction

Coated paperboard is a fundamental raw material for various printed-decorated packaging and transport packaging industries due to its advantages such as high strength-to-weight ratio, high surface smoothness, printability, sustainability, recyclability (Kirwan, 2013). If any cracks occur on the outside of the folded parts of paperboard, which is used for making a cabinet, the mechanical strength of the cabinet is weakened and also the folded parts are inferior in decorative aspects. Actual creasing range was investigated based on the relationship between the crease depth and crease width by Hine (1959). Nagasawa et al. (2003; 2008; 2011) reported about the quasi-static folding stiffness with respect to the indentation depth of the creaser and also discussed about the crease deviation effect on the folding deformation characteristics. Also, several advanced results are reported for the de-lamination mechanism and bulging deformation considering anisotropic material properties (Beex, et al., 2009; Nygards, et al., 2009; Sudo, et al., 2005). However, they were mainly based on the quasi-static solid mechanics.

On the other hands, the time-dependent bending moment resistance acting on a hinge which is folded onto a scored line, is important in order to adjust the mechanical conditions of boxing stage performed by an automatic folder gluer. Various time-dependent problems on actual deformation phenomenon have not been sufficiently discussed in the past, although there are several reports of fiber creep and in-plane tensile relaxation of thin paper (Johanson et al., 1964; Sharon, et al., 2010). It is difficult to estimate various time-dependent responses from the quasi-static strength of a creased part, such as the maximum bending moment and the initial gradient of bending moment. Since such the transient deformation of the creased part subjected to a bending moment was not observed by any movie camera or load cells during the dynamic bending test, the time-dependent bending stiffness or its residual strain state could not be verified

directly while the quasi-static bending was examined (Nagasawa, et al., 2014). Recently, Nagasawa et al. investigated the relaxation characteristics of bending moment resistance of 0.3 mm thickness white-coated paperboard during a folding motion from an initial position up to various tracking angles, and through the bending test by the use of the bending moment measurement apparatus (CST-J-1, 2013), the relaxation of the bending moment resistance was investigated by an exponential coefficient of logarithmic function which was independent to the normalized indentation depth and the tracking angle (Nagasawa, et al., 2014; 2015). Furthermore, a creep-recovery (release) response of folded angle during returning back was also approximated by a logarithmic function of elapsed time. Seeing the effect of rotational velocity on the release angle when the holding (stopping) time was kept in 0s at the tracking position, the initial release angle decreased with the rotational velocity (Nagasawa, et al., 2016). Here, the initial release angle was determined at the time when the reaction force of folding becomes zero. However, in that report, the effect of holding (stopping) time at the tracking position on the released behavior was not discussed. Therefore, the combination effect of the rotational velocity during folding/unfolding process and the hold time at the tracking position on the release behavior was investigated in this work. Firstly, the time-dependent behavior of release angle was reviewed under the specified rotational velocity of fixture 0.2 rps (revolution per second) ( $1.26 \text{ rad}\cdot\text{s}^{-1}$ ) when varying the hold time, and some coefficients of logarithmic approximation of release angle was discussed. Secondly, the initial released angle (when the rotation force of folding becomes zero) was investigated when both the rotational velocity and the holding (stopping) time were varied in a certain range.

## 2. Experimental condition and method

### 2.1 Initial creasing of specimen for pre-processing

Figure 1 illustrates viewpoints of a paperboard. The prepared white-clay-coated paperboard (basis weight  $\rho=228\sim237 \text{ g}\cdot\text{m}^{-2}$ ) had a thickness of  $t=0.3$  ( $0.297\sim0.303$ ) mm. Table 1 shows the analysis result of fiber size and pulp combination ratio. Regarding the mechanical properties of the paperboard, the in-plane tensile test properties in the Machine Direction of paper making (MD, the principal axis direction with reference to the fiber grain direction), were shown in Table 2. The specimens were kept in a room which had a temperature of 296 K and a humidity of 50 %RH. The test pieces were prepared as 5 pieces of rectangle-shaped white-coated paperboard, which had a width of 15 mm, length of 60 mm, for each condition.

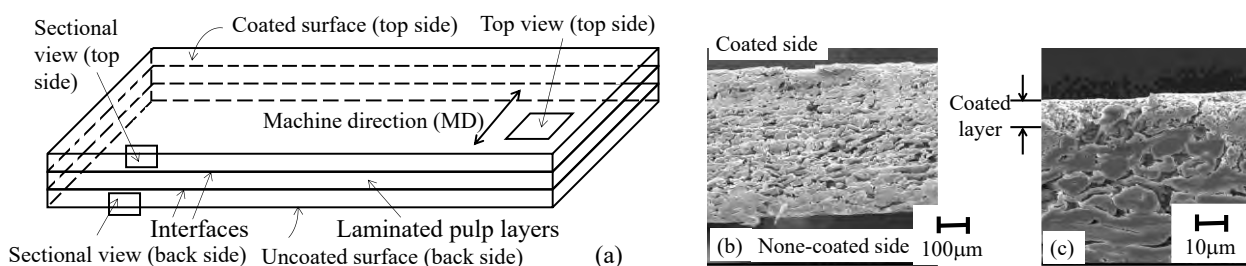


Figure 1 Outline of coated paperboard. (a) Schematic illustration. (b) SEM sectional view. (c) Top side SEM sectional view. The paperboard is composed of multiple plies and the coated top layer and the uncoated back layer are generally different (strong or tough) from the middle layer. When scoring the out-of-plane (top or back side) by the use of round-edge punching tool, the bonded interfaces of pulp layers apt to be de-laminated. This de-lamination is used for folding the paperboard without any breaking of outside layers.

Table 1 Size of fiber and pulp combination ratio of coated paperboard 230 (measured by Kajaani-FS300) L-BKP: Broad-leaved lumber (hard wood), bleaching kraft pulp; N-BKP: Needle-leaved lumber (soft wood), bleaching kraft pulp; N-TMP: Needle-leaved, thermal mechanical pulp; L(n): based on number of fibers in each fibrillation index class; L(l): based on length weighted number of fibers in each fibrillation index class; L(w): based on weight-weighted number of fibers in each fibrillation index class; CWT: Wall thickness of cell; Width: average width of fiber.

Unit	Pulp combination ratio /%			Projected length of fiber /mm			Size /μm		Section area /μm <sup>2</sup>
Item	L-BKP	N-BKP	N-TMP	L(n)	L(l)	L(w)	Width	CWT	CSA
Value	83.1	16.9	0.0	0.45	0.88	1.36	18.2	4.6	206.2

In order to make a smooth folding, a paperboard is usually scored by using a creaser at the pre-stage and make a localized delamination. (Kirwan, 2005). When the creased part is folded, the topside layers of fiber on the coated side are extended and require an adequate tensile strength and stretch, while the backside layers are compressed and bulged. In order to make a smart folding under forming the bulged zone, a scoring is processed before folding the paperboard.

Table 2 In-Plane tensile properties of white-coated paperboard in Machine direction (MD).

Tensile feed velocity was  $0.33 \text{ mm} \cdot \text{s}^{-1}$  (strain rate:  $0.00183 \text{ s}^{-1}$ ). Based on the procedure of JIS-P8113.

Ultimate Tensile Strength	$\sigma_B$ MPa	41.1 (40.2~42.7)
Breaking true strain	$\epsilon_B$ %	1.71 (1.62~1.81)
Young's modulus	$E$ GPa	5.72 (5.53~5.91)

The specimen was scored using a round-edge knife (a creaser) and rubber blocks as shown in Fig.2. Here, the creaser was set across MD of the specimen. Figure 3 shows a scoring state (crease forming) of a paperboard specimen using the creaser with a radius of  $r = 0.355 \text{ mm}$ , thickness of  $b = 0.71 \text{ mm}$ . When the creaser is indented to the paperboard, the expression:  $\tan \delta = (2d \cdot B^{-1}) = \gamma$  is the average shearing strain. This quantity  $\gamma$  is defined as the normalized indentation depth (Nagasawa et al., 2001). Also, using the paperboard thickness  $t$  and the thickness of creaser  $b$ , the groove width  $B$  was empirically chosen as  $2t + b = 1.3 \text{ mm}$ , the height of groove  $H$  was  $1.5 \text{ mm}$  and the indentation of creaser  $d$  was chosen as  $d < H$ . Regarding the time-dependent response of bending moment resistance (Nagasawa et al., 2014, 2015), the scored state of crease part was investigated as  $\gamma = 0.2 \sim 1.0$ , while the previous work (Nagasawa, et al., 2016) mainly discussed with the scored state of  $\gamma = 0.6$ . This was also chosen here owing that the indentation depth was empirically designed as  $\gamma = 0.4 \sim 0.6$  ( $d \approx t$ ). The scoring condition (using a rubber height of  $7 \text{ mm}$  by the hardness of 40(A) and a creaser height of  $5.6 \text{ mm}$ ) was empirically chosen from a commercial based production. All the specimens were formed without warpages. The feed velocity of creaser was chosen as  $V = 0.0167 \text{ mm} \cdot \text{s}^{-1}$  for scoring.

## 2.2 Folding and release of scored specimen

Figure 4 illustrates a conceptual mechanism of crease folding. When the paperboard is scored by a creasing knife, the intermediate layers are damaged as shown in Fig.4(a). Since the paperboard consisted of laminated plies, a certain extent of de-lamination damage is generated in the scored zone. When the paperboard is folded with this scored position, the damaged layers are further de-laminated and its inside (lower) layers are bulged as shown in Fig.4(b) (Hine, 1959; Nagasawa, 2004). The bending moment resistance of the creased zone appeared to consist of three mechanisms: (i) a tensile resistance of the outside (upper) layers, (ii) a compressive resistance of the inside (lower) layers, and (iii) a detaching (peeling) resistance of the middle layers (Nagasawa et al., 2011). The third item (detaching) affects only the transient folding resistance in the early stage, while the first (tensile of outside) and second (compression of bulged layers) items behave as the bending moment resistance in the full stage of folding test. Figure 5 shows a general view of bending test apparatus (CST-J-1, 2003). Figure 6 shows a conceptual illustration of the folding process and rotating method used in the bending test.

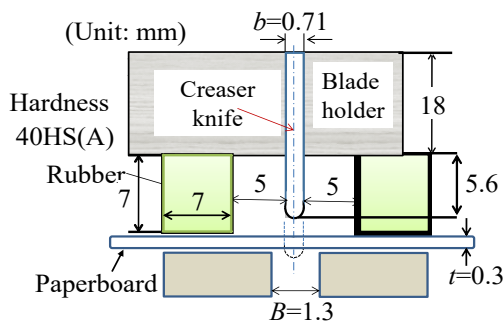


Figure 2 Layout of out-of-plane scoring by creaser

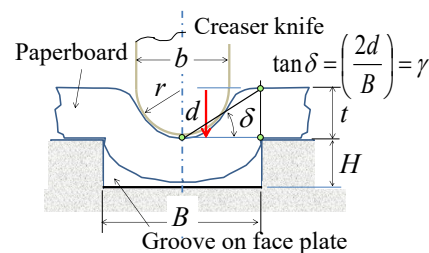


Figure 3 Schematic of scoring state and parameters

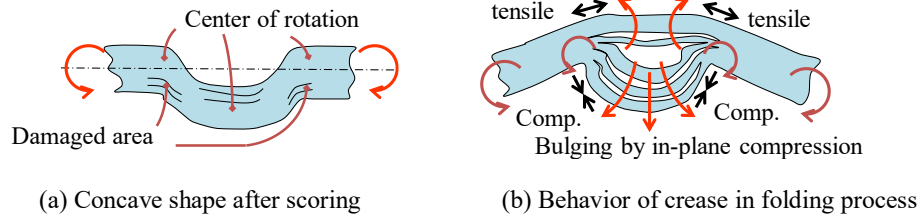


Figure 4 Model of crease mechanism. (a) Scoring by creaser knife makes damaged area in a laminated paperboard, and causes an offset with the center position of rotation. (b) Lateral in-plane compression on the inside layer buckles and makes the inside layer bulged. This moves the neutral plane of bending upwards and reduces the tensile stress in the outside layer. (Ref. Nagasawa, S. et al., 2004)

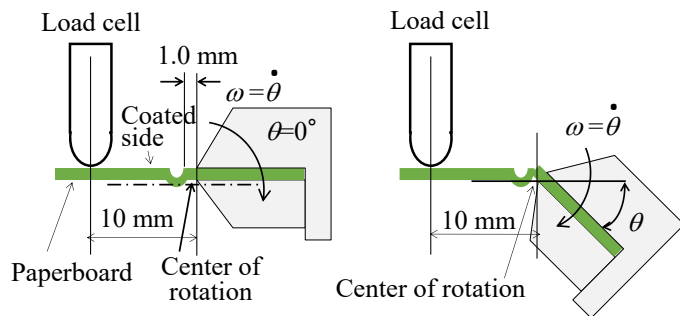
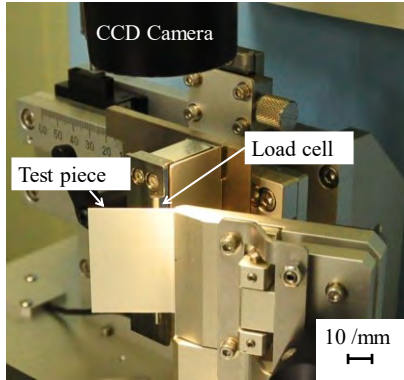


Figure 5 General view of bending test apparatus Figure 6 Conception of bending test (folding process)

In the folding process, the folding angle  $\theta$  was recorded by a CCD camera of digital microscope, while the bending moment  $M$  was measured by a load cell of the bending test apparatus. A scored paperboard was set up in the test apparatus and clamped by the fixture which rotated with the rotational velocity  $\omega$ . The clamping position was shown in Fig.6. The scored paperboard was bent from the original position  $\theta = 0^\circ$  up to the folding angle of  $\theta = 90^\circ$ . The folding process was completed at  $90^\circ$  (named as the tracking angle  $\theta = 90^\circ$ ) and its angle of fold state was paused for a specified duration defined as the elapsed hold (stopping) time  $t_{1ep}$ . This pause of  $\theta = 90^\circ$  was used for observing the relaxation of bending moment. After completing this relaxation process with the hold time  $t_{1ep}$ , the folded paperboard is sequentially released under returning back with the unfolding rotational velocity  $\omega'$  until the reaction force of the load cell becomes zero. This returning back duration was named as the unfolding or release process.

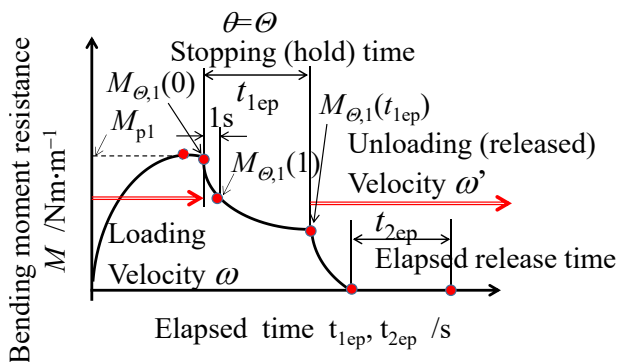


Figure 7 Conceptual bending moment response with elapsed time and measured parameters.

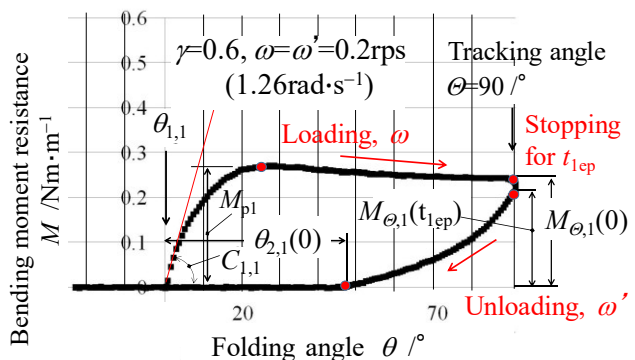


Figure 8 Analysis parameters on folding resistance diagram.

The conceptual relationship between the bending moment resistance (for the unit width)  $M$  and the elapsed time was illustrated in Fig.7. Figure 8 is an example of the first round's folding load response described as the relationship between the folding rotation angle  $\theta$  and bending moment resistance  $M$ . The first term maximum peak bending moment  $M_{p1}$  and the bending moment at the first round's tracking angle  $M_{\theta=90^\circ,1}(t_{1ep})$  were explained in the previous report

(Nagasawa et al., 2014, 2015). The first round's starting angle  $\theta_{1,1}$  was zero (in a flat attitude). In this work, only the first round was considered (the number of folding repetition was suffixed as “,1”). The early stage in which  $\theta$  was less than  $20^\circ$  was mainly characterized by the elastic bending stiffness without detaching and bulging of inside layers. Since the middle stage ( $20^\circ < \theta < 90^\circ$ ) had a certain stationary resistance (almost constant) under the specified rotation velocity  $\omega$ , the behavior of resistance appeared to be a sort of the creep response of Maxwell type two-element model (Betten, 2002).

This bending moment diagram was used here for confirming the hold time  $t_{1ep}=0\sim 20$ s and detecting the initial unfolded state  $t_{2ep}=0$ s. Here,  $t_{2ep}$  is the elapsed release time for observing the release angle  $\theta_{2,1}$ . The quantity  $\theta_{2,1}(0)$  is the initial release angle.

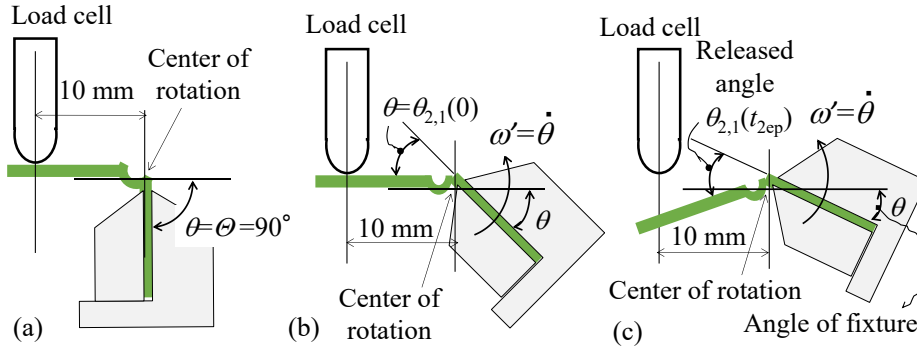


Figure 9 Schematics of unfolding and relationship between release angle and fixture angle (a) State of tracking angle of  $90^\circ$ ; (b) State of initial release angle ( $t_{2ep}=0$ ) when reaction force of load cell becomes zero; (c) In case of unfolded state when detaching the load cell (for  $t_{2ep}>0$ ).

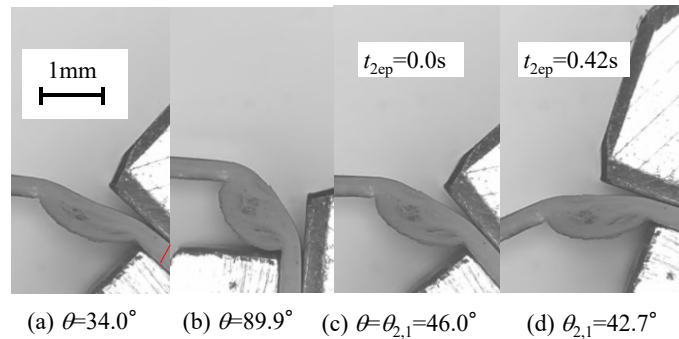


Figure 10 CCD camera photographs of side views of creased part during folding test ( $\gamma=0.6$ ,  $\omega=0.2$  rps). (Ref. Nagasawa, S. et al., 2016)

Figure 9 illustrates three states of unfolding process: (a) the state of tracking angle of  $\theta = 90^\circ$ , (b) the initial released state when defining  $t_{2ep}=0$ , and (c) the released state after detaching the load cell. Generally,  $\theta_{2,1}$  is not equal to the rotation angle of fixture  $\theta$  for  $t_{2ep}>0$ , while  $\theta_{2,1}$  is equal to  $\theta$  at  $t_{2ep}=0$  or before detaching the load cell. Figure 10 showed representative side views of real creased part during a folding test. In the folding process (a),(b), the bulged inside layers were compressed in the in-plane direction, while the height of bulged zone decreased with the released angle  $\theta_{2,1}$  in the unfolding process (c),(d). Namely, the released behavior of folded attitude appeared to be mainly caused by the released energy of compressed and bulged layers.

According to the preliminary experiments (Nagasawa, et al., 2014, 2015), the relationship between the bending moment at the tracking position  $M_{\theta=90^\circ,1}$  and the hold time  $t_{1ep}$  was linearly approximated with the logarithmic term  $\ln(t_{1ep})$  using Eq.(1),(2). Here, the intercept  $a_0$  was defined as  $M_{90,1}(1)$  (at  $t_{1ep}=1$ s),  $a_1$  was the gradient coefficient of Eq.(1), and the exponential coefficient  $p_1$  of relaxation was defined as the ratio of  $a_1/a_0$ . They describe the relaxation characteristics of bending moment accumulated at the tracking angle of  $90^\circ$ .

$$M_{90,1} = -a_1 \ln(t_{1ep}) + a_0 \quad (1)$$

$$M_{90,1}/a_0 = \bar{M}_{90,1} = 1 - p_1 \ln(t_{1ep}), \quad p_1 = a_1/a_0 \quad (2)$$

The logarithmic relaxation of bending moment at the holding of  $\theta=90^\circ$  was similar to the stress relaxation of the white-coated paperboard subjected to an uni-axial tensile displacement (Nagasawa et al., 2017). Namely, the value of



exponential coefficient  $p_1$  of bending resistance was almost equal to that of uni-axial tensile displacement. This means that the tensile relaxation of in-plane direction is a major factor in the bending resistance.

Regarding the relationship between the release angle  $\theta_{2,1}$  and the elapsed release time  $t_{2ep}$ , the linear approximation with the logarithmic term  $\ln(t_{2ep})$  was introduced (Nagasawa, et al., 2016) as Eq.(3),(4). Here, the intercept  $b_0$  was defined as  $\theta_{2,1}(1)$  (at  $t_{2ep}=1s$ ),  $b_1$  was the gradient coefficient of Eq.(3), and the exponential coefficient  $p_2$  was defined as the ratio of  $b_1/b_0$ . They describe the creep-recovery characteristics of folded angle.

$$\theta_{2,1} = -b_1 \ln(t_{2ep}) + b_0 \quad (3)$$

$$\theta_{2,1}/b_0 = \bar{\theta}_{2,1} = 1 - p_2 \ln(t_{2ep}), \quad p_2 = b_1/b_0 \quad (4)$$

Since the transient response seems to be caused by the relaxation and creep-recovery characteristics of paperboard during the folding process, the effect of fixture's rotational velocity  $\omega$  (folding) and  $\omega'$  (unfolding) on the release angle  $\theta_{2,1}(t_{2ep})$  was investigated for the range of  $t_{2ep}=0\sim 10s$  when  $\gamma=0.6$ .

The synchronized condition of  $\omega=\omega'$  was mainly investigated and its value was chosen as 0.02, 0.03, 0.05, 0.1, 0.2, 0.3 and 0.4 rps (0.13, 0.19, 0.31, 0.63, 1.26, 1.88, 2.51 rad·s<sup>-1</sup>). For the sake of comparison of asynchronous condition, when the returning back velocity was chosen as a constant of  $\omega'=0.2$  rps (1.26 rad·s<sup>-1</sup>), the folding velocity  $\omega$  was chosen as 0.02, 0.03, 0.05, 0.1, 0.2, 0.3 and 0.4 rps (0.13, 0.19, 0.31, 0.63, 1.26, 1.88, 2.51 rad·s<sup>-1</sup>).

### 3. Results and discussion

#### 3.1 Response of bending moment with rotational velocity

In the synchronized condition of  $\omega=\omega'=0.2$  rps (1.26 rad·s<sup>-1</sup>), Fig.8 illustrated a representative case of bending moment resistance  $M$  with the folding angle  $\theta$ . Since the intermediate stage ( $20^\circ < \theta < 90^\circ$ ) appeared to be a sort of creep response of Maxwell type two-element model in Fig.8, the values of  $M_{p1}$  and  $M_{90,1}(0)$  were expected to increase with  $\omega$ . When varying the velocity  $\omega(=\omega')$ , the maximum peak bending moment  $M_{p1}$ , and the relaxed bending moment at  $t_{1ep}=0s, 1s$  with  $\Theta=90^\circ$ :  $M_{90,1}(0), M_{90,1}(1)$  were measured and shown in Fig.11. Eq. (5), (6), (7) were derived as linear approximations with the logarithmic term  $\ln(\omega/0.2)$  from the experimental result.

$$M_{p1} = 0.013 \ln(\omega/0.2) + 0.244 \quad (5)$$

$$M_{90,1}(0) = 0.0061 \ln(\omega/0.2) + 0.215 \quad (6)$$

$$M_{90,1}(1) = a_0 = -0.004 \ln(\omega/0.2) + 0.175 \quad (7)$$

Seeing Eq.(5),(6), the bending moment under the folding process increased with the rotational velocity  $\omega(=\omega')$ . This tendency matched the prediction of Maxwell type relaxation response. However, the relaxed bending moment  $M_{90,1}(1)$  decreased with  $\omega(=\omega')$  from Eq. (7). This means that the dissipation energy or the bending moment drop increases with the rotational velocity  $\omega$  for a short duration  $t_{ep1} < 1s$ .

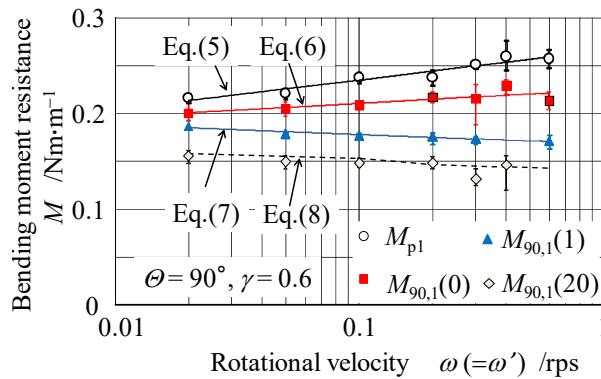


Figure 11 Dependency of bending moment on rotational velocity. The unfolding velocity  $\omega'$  was equal to the folding velocity  $\omega$ . As the representative quantities of bending moment resistance, the maximum peak  $M_{p1}$ , the relaxed three states at the tracking position  $M_{90,1}(0)$ ,  $M_{90,1}(1)$  and  $M_{90,1}(20)$  were plotted as the average (with the maximum and minimum bar).

Seeing the preliminary experiment (Nagasawa, et al., 2015, Fig.14), since the exponential coefficient of relaxation  $p_1$



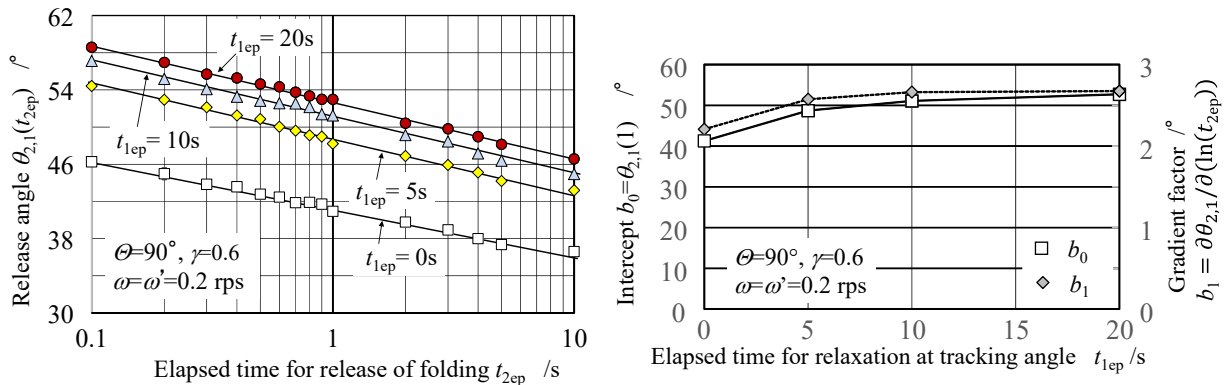
was expressed with  $\omega/0.2$  for  $0.2 < \omega/0.2 < 3$ , the relaxed bending moment at  $t_{1ep}=20$ s was estimated using Eq.(8). Here,  $a_0$  was calculated from Eq.(7), and  $p_1$  was estimated as 0.046 for  $\omega/0.2 < 0.5$  and 0.055~0.057 for  $0.5 < \omega/0.2 < 3$  from the preliminary experiment. The result of Eq.(8) was well similar to the current experiment as shown in Fig.11.

$$M_{90,1}(t_{1ep}) = a_0 (1 - p_1 \ln(t_{1ep})) \quad (8)$$

So far, it was revealed that the bending moment at the pre-stage of tracking position ( $\Theta=90^\circ$ ) was sufficiently accumulated as an elastic strain energy, namely the measured quantities  $M_{p1}$  and  $M_{90,1}(0)$  increased with the folding velocity  $\omega$ , whereas the quasi-stationary relaxation of the bending moment was performed when the hold time was kept in a certain duration. In such the relaxation state based on the hold time ( $t_{1ep} > 1$ s), the bending moment decreased slightly with  $\omega$ . Also, a drop rate of the bending moment in the early stage of holding process increased with  $\omega$ , owing that the difference of Eq.(6) and Eq.(7) derived the drop rate  $M_{90,1}(0) - M_{90,1}(1) = 0.01 \ln(\omega/0.2) + 0.04$ .

### 3.2 Dependency of release angle on hold time

Figure 12 (a) shows a relationship between the first round's release angle  $\theta_{2,1}$  and the elapsed release time  $t_{2ep}$  when choosing the hold time  $t_{1ep}$  as 0, 5, 10 and 20s under the synchronized condition  $\omega=\omega'=0.2$  rps ( $1.26 \text{ rad}\cdot\text{s}^{-1}$ ). Here, the value of  $\theta_{2,1}$  was plotted as the average of 5 samples. Seeing Figure 12 (a), it is found that the variation of  $\theta_{2,1}$  is decreased as a logarithmic form with  $t_{2ep}$ , and the intercept of  $\theta_{2,1}(1)$  tends to be increased with  $t_{1ep}$ . Hence, the release angle  $\theta_{2,1}$  was approximated with the term  $\ln(t_{2ep})$  using Eq.(3), (4).



(a) Relationship between release angle (average) and elapsed release time when varying hold time

(b) Approximation coefficients of folding angle of crease with respect to hold time

Figure 12 Response of release angle (average) with respect to elapsed release time when keeping rotational velocity  $\omega=\omega'=0.2$  rps ( $1.26 \text{ rad}\cdot\text{s}^{-1}$ ). The standard deviation of measured release angle was about  $0.7^\circ$ .

Figure 12 (b) arranges the dependency of those two coefficients  $b_1$ ,  $b_0$  on the hold time  $t_{1ep}$ . It is found that the intercept  $b_0$  is remarkably varied in a short time less than 5s, while it tends to be saturated or asymptotically increased for  $t_{1ep} > 5$ s. Since the exponent coefficient  $p_2$  was about 0.050~0.053 in stable, the time-delay characteristic (creep-recovery) of release angle appears to be independent to the hold time, when keeping  $\omega=\omega'=0.2$  rps ( $1.26 \text{ rad}\cdot\text{s}^{-1}$ ).

### 3.3 Dependency of initial release angle on rotational velocity

According to the preliminary experiment (Nagasawa et al., (2016), Fig.15), when  $\omega > 0.1$  rps ( $0.63 \text{ rad}\cdot\text{s}^{-1}$ ) and  $t_{1ep} = 0$ s, the exponential coefficient  $p_2$  was about 0.05 for  $t_{2ep} = 0.2 \sim 10$ s, namely  $p_2$  appeared to be insensitive for  $\omega > 0.1$  rps ( $0.63 \text{ rad}\cdot\text{s}^{-1}$ ). Since the expression of Eq.(4) is composed of two factors  $p_2$  and  $b_0$ , in order to discuss the effect of hold time on the release angle, the behavior of the intercept  $b_0 = \theta_{2,1}(1)$  seems to be necessary. In this work, the initial release angle  $\theta_{2,1}(0)$  was measured and analyzed instead of  $\theta_{2,1}(1)$ .

Figure 13 shows the initial release angle  $\theta_{2,1}(0)$  when varying the hold time  $t_{1ep} = 0 \sim 20$ s at the tracking angle  $\Theta=90^\circ$ . Here, the synchronized condition was considered:  $\omega=\omega' = 0.02 \sim 0.4$ rps ( $0.13 \sim 2.51 \text{ rad}\cdot\text{s}^{-1}$ ). The value  $\theta_{2,1}(0)$  was the average of 5 samples and the error bar shows the maximum and minimum value in the 5 samples for each rotational velocity. It is found that  $\theta_{2,1}(0)$  tends to be linearly varied with a logarithmic function of rotational velocity.

Therefore, Eq.(9) was introduced and the gradient coefficient  $\beta_1$  and the intercept  $\beta_0 = \theta_{2,1}(0)|_{\omega=0.2 \text{ rps}}$  were investigated

and arranged in Fig. 14.

$$\theta_{2,1}(0) = \beta_1 \ln(\omega / 0.2) + \beta_0, \quad \beta_0 = \theta_{2,1}(0)|_{\omega=0.2\text{ rps}} \quad (9)$$

Seeing Figure 13 and Fig.14, the gradient  $\beta_1$  was changed from the negative (decrease) to the positive (increase) in a short time holding:  $1s < t_{1ep} < 2s$ . It was asymptotically and slightly increased for  $t_{1ep} > 5s$ . This tendency seems to be caused by the relaxation of folding posture against the pre-stage accumulation of elastic bending energy. If the hold time is sufficiently long for reducing the residual bending stress, the effect of folding (first half) rotational velocity  $\omega$  seems to be isolated and then only the unfolding (second half as the returning back) velocity  $\omega'$  appears to affect the initial release angle.

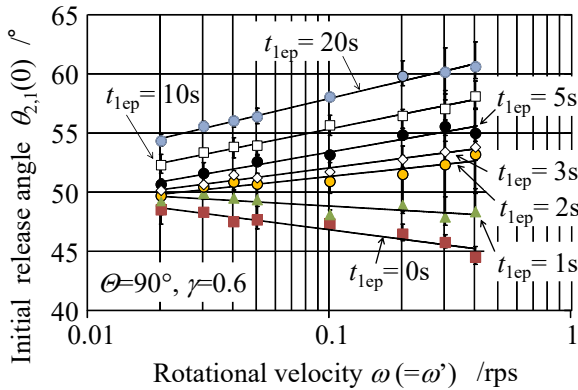


Figure 13 Relationship between initial released angle and synchronized rotational velocity by varying hold time.

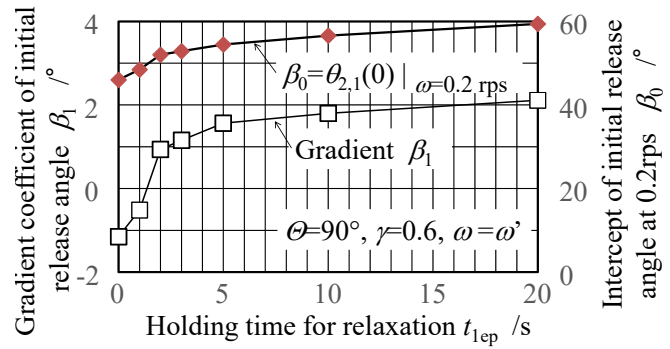


Figure 14 Gradient and intercept of initial released angle with respect to rotational velocity when varying hold time.

In order to verify this isolation effect, an additional experiment was carried out. Namely, when the unfolding velocity was kept in  $\omega' = 0.2 \text{ rps}$  ( $1.26 \text{ rad}\cdot\text{s}^{-1}$ ), the folding velocity  $\omega$  was chosen as 0.02, 0.2 and 0.4 rps ( $0.13, 1.26, 2.51 \text{ rad}\cdot\text{s}^{-1}$ ). This is the asynchronous condition. Figure 15 shows a ratio of the coefficients of Eq.(9):  $\beta_1/\beta_0$  with respect to the hold time  $t_{1ep} = 0 \sim 20s$ . Here, the synchronized condition ( $\omega = \omega'$ ) was derived from Fig.14. It is found that the coefficient ratio of asynchronous condition is sufficiently small-positive for  $t_{1ep} > 10s$ . The effect of first half rotational velocity  $\omega$  is fairly isolated by the hold time of 10~20s, but its effect is observed in a small extent.

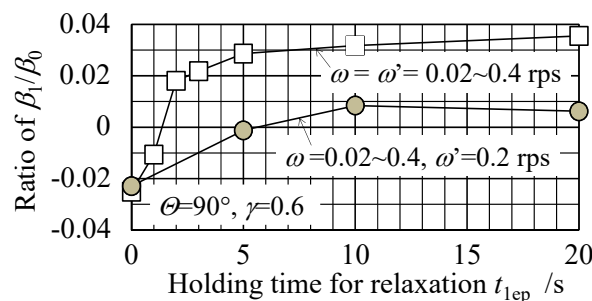


Figure 15 Comparison of ratio of coefficients  $\beta_1/\beta_0$  between synchronized and asynchronous condition

### 3.4 Dissipation of accumulated bending moment and variation of initial released angle

As mentioned in the section 9.1, in the synchronized condition, the time-dependent response of the bending moment at the tracking position ( $\Theta = 90^\circ$ ) was revealed as shown in Fig.11. When the hold time is kept in a certain duration, e.g.  $t_{1ep} > 1s$ , the energy dissipation of accumulated bending moment seems to be characterized with the pre-stage folding velocity  $\omega$ . As the result, when the release (returning back) process starts, the bending moment has a decreased level with respect to the pre-stage folding velocity  $\omega$ .

Seeing Fig.13 and Fig.14, the initial release angle  $\theta_{2,1}(0)$  increased with the velocity  $\omega$  (in the synchronized condition) when  $t_{1ep} > 2s$ . This is the same as that the spring back energy of folded posture decreases with  $\omega$  when the hold time is

longer than 2s. Comparing this tendency with the variance of  $M_{90,1}(t_{1ep})$ , it is revealed that the pre-stage and the early stage of the hold time (e.g.,  $t_{1ep} < 1$  or 2s) are extremely sensitive to the relaxation or dissipation of accumulated bending moment energy. From the results of Fig.13, 14, and also seeing Fig.12 (b), the quasi-stationary relaxation of holding motion is performed for  $t_{1ep} > 5s$ .

#### 4. Conclusions

One-loop bending characteristics of creased white-paperboard of 0.3mm thickness were investigated by varying the folding rotational velocity  $\omega$  and the unfolding (returning back) rotational velocity  $\omega'$  when the hold time  $t_{1ep} = 0 \sim 20s$  was considered at the tracking position of  $\Theta = 90^\circ$ , using a digital microscope camera. Through this work, the followings were found.

- 1) The release angle  $\theta_{2,1}(t_{2ep})$  was characterized with a logarithmic function of the elapsed release time  $t_{2ep}$  for the measured range of  $t_{2ep} = 0 \sim 10s$ , when keeping the folding and unfolding rotational velocities in a constant. The release angle  $\theta_{2,1}(t_{2ep})$  appears to be decomposed into the intercept part  $b_0$  which is determined by the hold time  $t_{1ep}$  at the tracking position and the gradient part  $p_2$  which is independently related to the release time  $t_{2ep}$ .
- 2) In the case of  $\omega = \omega' = 0.2$  rps ( $1.26 \text{ rad} \cdot \text{s}^{-1}$ ), the exponential coefficient  $p_2 = b_1/b_0$  of Eq.(4) was stably  $0.050 \sim 0.053$  for  $t_{1ep} = 0 \sim 20s$ , whereas the coefficients  $b_0, b_1$  of E.(3) were limitedly saturated for  $t_{1ep} > 5s$ .
- 3) When varying the folding and unfolding (released) rotational velocities under the synchronized condition:  $\omega = \omega'$  for  $0.1 \leq \omega/0.2 \leq 3$ , the initial release angle  $\theta_{2,1}(0)$  was characterized with a logarithmic term  $\ln(\omega/0.2)$  using the gradient coefficient  $\beta_1 = \partial \theta_{2,1}(0) / \partial (\ln(\omega/0.2))$  and the intercept  $\beta_0 = \theta_{2,1}(0)|_{\omega=0.2\text{rps}}$ .
- 4) When  $t_{1ep} < 2s$ , the coefficients  $\beta_1$  and  $\beta_0$  were remarkably varied. A change in the sign of  $\beta_1$  occurred at this duration. When  $t_{1ep} > 5s$ ,  $\beta_1$  and the ratio  $\beta_1/\beta_0$  were asymptotically and slightly increased with  $t_{1ep}$ .
- 5) Regarding the response of bending moment, a drop rate of the bending moment in the early stage ( $t_{1ep} = 0 \sim 1s$ ) of holding process remarkably increased with the term  $\ln(\omega/0.2)$  for  $0.1 \leq \omega/0.2 \leq 3$ , whereas the relaxation behavior settled down with the exponential coefficient  $p_1 = a_1/a_0$  of Eq.(2) for  $t_{1ep} > 1s$ . Comparing this early stage of bending moment with the item 4), it was revealed that the gradient coefficient  $\beta_1$  was under a transient state of the relaxation for  $t_{1ep} < 2s$ , while that was under a quasi-stationary state for  $t_{1ep} > 5s$ .
- 6) When varying the folding velocity  $\omega$  under keeping the unfolding velocity  $\omega' = 0.2$  rps ( $1.26 \text{ rad} \cdot \text{s}^{-1}$ ) (asynchronous condition), the gradient coefficient  $\beta_1$  and the ratio  $\beta_1/\beta_0$  were relatively smaller than that of synchronized condition. Also, the value of  $\beta_1/\beta_0$  was stably invariant for  $t_{1ep} > 10s$ .
- 7) Seeing 3), 4) and 6), the hold time at the tracking position makes the folded part in relaxed state and then the holding posture isolates the second half unfolding released behavior from the first half folding rotational velocity effect when the hold time is longer than 5s.

#### Nomenclature

- $d$  : indentation depth of creaser knife  
 $t$  : thickness of work sheet (paperboard)  
 $B$  : width of groove on counter face plate  
 $\gamma = 2d \cdot B^{-1}$  : normalized indentation depth, ( $\gamma = 0.6$  was chosen in this work)  
 $M$  : bending moment for the unit width against folding ( $\text{Nm} \cdot \text{m}^{-1}$ )  
 $\theta$  : folding angle of fixture ( $^\circ$ )  
 $\Theta$  : tracking (maximum) angle ( $^\circ$ ) ( $\Theta = 90^\circ$  was chosen in this work)  
 $n$  : number of folding repetitions ( $n=1$  was considered in this work)  
 $M_{\Theta(n)}$  : the  $n$ -th round bending moment at a tracking position  
 $\theta_{1,n}, \theta_{2,n}$  : the  $n$ -th round starting, release (angle) position ( $\theta_{1,1}$  was zero in this work)  
 $t_{1ep}$  : the  $n$ -th round hold time before returning back ( $t_{1ep}$  was varied from 0 up to 20s, in this work)  
 $t_{2ep}$  : the  $n$ -th round elapsed release time until the next folding ( $t_{2ep}$  was measured up to 10s in this work)  
 $\omega$  : rotational velocity of fixture for folding (rps, revolution per second) ( $= 2\pi \omega \text{ rad} \cdot \text{s}^{-1}$ )  
 $\omega'$  : rotational velocity of fixture for unfolding (released, returning back process) (rps) ( $= 2\pi \omega' \text{ rad} \cdot \text{s}^{-1}$ )  
 $a_1, a_0$  : relaxation coefficients derived from Eq. (1) Here,  $a_0$  is  $M_{\Theta,1}$  at  $t_{1ep}=1s$ .  
 $p_1 = a_1 \cdot a_0^{-1}$  : an exponential coefficient derived from Eq. (1), Eq.(2)  
 $b_1, b_0$  : release coefficients derived from Eq.(3) Here,  $b_0$  is the intercept as  $\theta_{2,1}$  at  $t_{2ep}=1s$ .

$p_2 = b_1 \cdot b_0^{-1}$ : an exponential coefficient derived from Eq.(3), Eq.(4)

$\theta_{2,1}(0)$ : initial release angle

$\beta_1, \beta_0$ : gradient coefficient and intercept derived from Eq.(9) Here,  $\beta_0 = \theta_{2,1}(0)|_{\omega=0.2\text{rps}}$

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## The Influence of Machining Parameters on Surface Roughness of MDF in Milling Operation

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This paper examined the effect of machining parameters on surface roughness of medium density fiberboard (MDF) machined using a computer numerical control (CNC) router. The machining parameters such as spindle speed, feed rate, depth of cut, and tool diameter were examined for milling. The experiments were conducted at two levels of spindle speeds, four levels of feed rates, two levels of tool diameters, and two levels of axial depths of cut. The surface roughness values of MDF grooved by CNC were measured with stylus-type equipment. Statistical methods were used to determine the effectiveness of the machining parameters on surface roughness. The influence of each milling parameter affecting surface roughness was analyzed using analysis of variance (ANOVA). The significant machining parameters affecting the surface roughness were the feed rate, spindle speed, and tool diameter ( $p < 0.05$ ). There was no significant influence of axial depth of cut on the surface roughness. The surface roughness decreased with increasing spindle speed and decreasing feed rate. The value of surface roughness increased with the increase of tool diameter.

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This paper examined the effect of machining parameters on surface roughness of medium density fiberboard (MDF) machined using a computer numerical control (CNC) router. The machining parameters such as spindle speed, feed rate, depth of cut, and tool diameter were examined for milling. The experiments were conducted at two levels of spindle speeds, four levels of feed rates, two levels of tool diameters, and two levels of axial depths of cut. The surface roughness values of MDF grooved by CNC were measured with stylus-type equipment. Statistical methods were used to determine the effectiveness of the machining parameters on surface roughness. The influence of each milling parameter affecting surface roughness was analyzed using analysis of variance (ANOVA). The significant machining parameters affecting the surface roughness were the feed rate, spindle speed, and tool diameter ( $p < 0.05$ ). There was no significant influence of axial depth of cut on the surface roughness. The surface roughness decreased with increasing spindle speed and decreasing feed rate. The value of surface roughness increased with the increase of tool diameter.

*Keywords:* Surface roughness; Spindle speed; Feed rate; Milling operation

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## INTRODUCTION

The surface quality of wood materials is an important criterion, especially for finishing and surface lamination applications and machining method (*i.e.*, milling, drilling, sanding) in the furniture industry. Moreover, it is necessary to know the machining parameters of optimum surface quality in order to use efficient and cost-effective use of advanced technology machines and to save on the process time. The quality characteristics of the machined surfaces are determined by the surface roughness parameters (Davis 1962). Several approaches have been proposed to determine surface roughness, and the first surface roughness measurements were conducted using sensory methods. Because sensory methods are very subjective, the contact (stylus type of profilometer) and non-contact (laser or ultrasonic systems) measurement methods have been proposed for measurement of surface roughness (Funck *et al.* 1992; Hızıroğlu 1996). In recent years, atomic force microscopy (AFM) and scanning electron microscopy (SEM) methods have been used to evaluate the surface properties of machined materials (Haq and Srivastava 2016). In addition, the optimization approaches such as genetic algorithm (GA), response surface method (RSM), and desirability function (DF) have been used to estimate optimum machining parameters, which make it possible to achieve a minimum surface roughness value (Hazir *et al.* 2018).



The surface quality of wood and wood-based materials depends on the parameters associated with material properties (*i.e.*, wood species, anatomical properties, moisture content, density), machining parameters (*i.e.*, spindle speed, cutting force, feed rate, axial depth of cut), and cutting tool properties (*i.e.*, tool wear, diameter, tool geometry) (Coelho *et al.* 2008; Magos 2008). In the milling of gypsum fiber composite, the effect of the processing parameters such as spindle speed, feed rate, and depth of cut on the cutting force has been optimized by RSM based on a mathematical model (Li *et al.* 2017). General standards and recommendations have been used due to the lack of a standardized method for determining the surface roughness of wood, medium density fiberboard (MDF), or particleboards. This has led to the use of a wide range of measuring instruments and parameters such as filters, filtering cut-off length, measuring length, and measuring resolution (Gurau and Irle 2017).

Previous studies on the surface roughness of wood material have focused on evaluating the effect of material properties such as wood species (Malkoçoğlu and Özdemir 2006), radial and tangential machining direction (Kılıç 2015, 2017), wood density, early wood and latewood ratio (Sadoh and Nakata 1987; Malkoçoğlu 2007; Zhong *et al.* 2013), and fiber direction (Mitchell and Lemaster 2002; Iskra and Tanaka 2005; Sütçü 2013). Moreover, the effects of computer numerical control (CNC) machining parameters such as spindle speed, feed rate, and cutting depth on the surface quality of wood have been monitored (Sütçü and Karagöz 2013; Koc *et al.* 2017; Sofuoglu 2017). CNC machines are widely used in the furniture industry for wood and MDF panels, especially for surface milling. MDF panels must have smooth surfaces for painting and finishing. The effects of machining parameters such as spindle speed, cutting speed, and feed rate on the surface quality of MDF machined with CNC have been investigated in many studies. Aguilera *et al.* (2000) reported that surface roughness of CNC machined MDF decreased with increasing material density. Davim *et al.* (2009) investigated the effect of processing parameters on surface roughness of MDF, which was grooved 5 mm depth of cut with CNC router, and stated that the surface roughness values decreased at high spindle speed and low feed rate. In another study, Gisip *et al.* (2009) indicted that the surface quality of MDF machined with the tool wear was decreased. Deus *et al.* (2015) also revealed that the surface roughness decreased with low depth of cut, high spindle speed, and low feed rate. Sütçü and Karagöz (2012) investigated the effect of machining parameters such as spindle speed, feed rate, stepover, and depth of cut on surface roughness of MDF pocket milling with CNC. They found that surface roughness value increased with increasing feed rate, stepover, and depth of cut and that material removal rate increased with high spindle speed at optimum surface roughness. In another study, the surface roughness of MDF panels has been shown to play an important role in the thin overlay applications such as thin melamine paper or polyvinyl chloride and other decorative overlays on the MDF surface (Aguilera 2000; Hizirolu and Kosonkorn 2006; Kılıç *et al.* 2009). At the same time, Li *et al.* (2018) investigated the surface color change of wood materials machined with a laser machine and found that the color change on the wood surfaces decreased with the increasing of feed speed and sweep width and decreasing of laser power.

As can be seen from the literature studies, the surface quality changes of different materials such as wood and wood based materials, MDF panels under different processing conditions (*i.e.*, milling, drilling, sanding, laser modification) were investigated by different experimental design methods. The differences of this study from other studies are different processing parameters with CNC router.

The efficiency and accuracy of machining in materials seem to be a very significant

criterion for the furniture industry. It is important to define the optimum surface machining parameters for the removal of additional sanding costs and labor in surfaces milled with CNC. Therefore, in this study, the effects of machining parameters on surface roughness of MDF panels machined with CNC milling have been examined. The grooving processing was performed on the MDF surfaces using varying processing parameters such as spindle speed, feed rate, depth of cut and tool diameter. The average roughness ( $R_a$ ) and mean peak to valley height ( $R_z$ ) of surface parameters were evaluated with a stylus type measurement device. The main and interaction effect of machining parameters on surface quality were analyzed by a statistical analysis program.

## EXPERIMENTAL

### Materials

MDF panels of 18 mm thickness (Kronospan, Kastamonu, Turkey) were used for surface milling. The density values ( $752 \text{ kg/m}^3$ ) and moisture content (7%) of the MDF panels were evaluated according to EN 323 (1993). MDF profile density value is an important factor affecting surface roughness. The density profiles of the MDF panels were obtained from the manufacturer to determine the effect of the depth of cut and density on the surface roughness. The density profile of MDF is shown in Fig. 1. The surface of the MDF was grooved with a CNC router that was used to process the aesthetic pattern, especially on the door surfaces. Two different CNC tools of 4 and 6 mm diameter were used for grooving. The rake angles of tools are  $7^\circ$ . The tool geometry is presented in Fig. 2a. A total of 36 experimental samples were grooved on MDF panels with a CNC router, as shown in Fig. 2b.

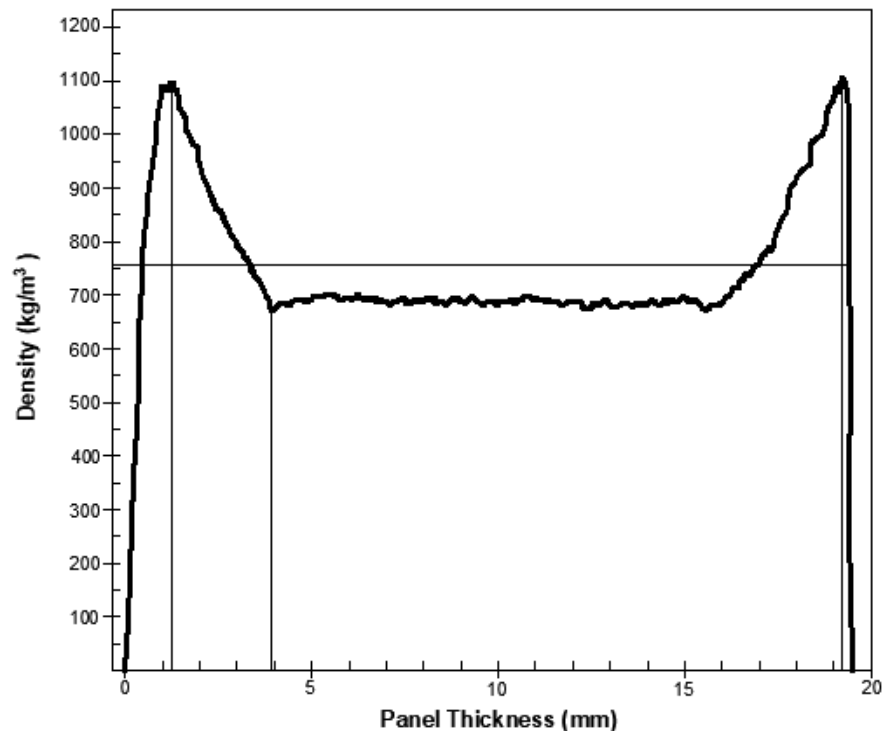


Fig. 1. Density profile of MDF panels

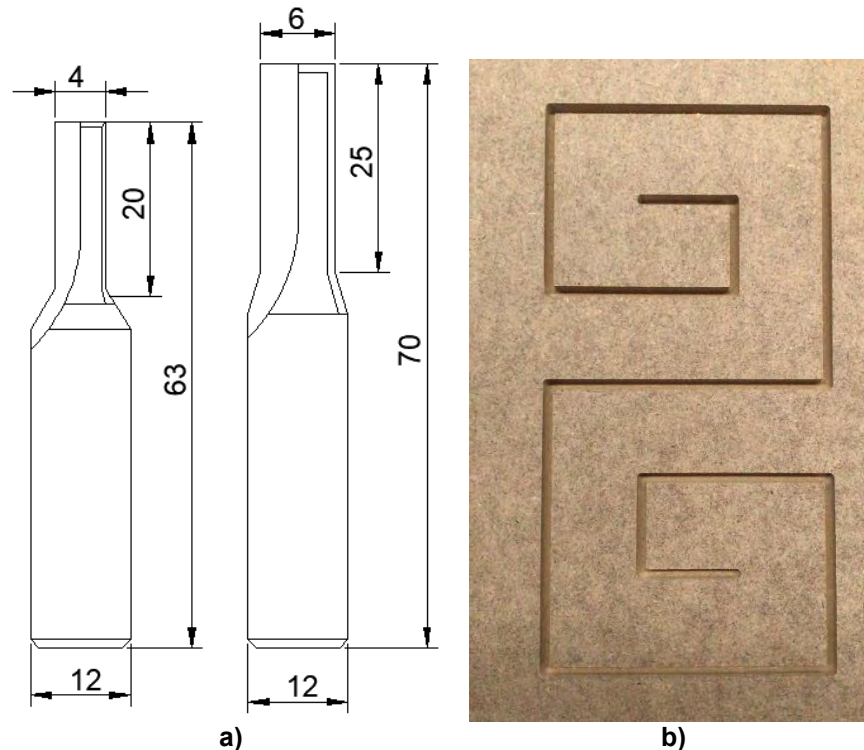


Fig. 2. a) The tool geometry and b) grooved MDF samples

## Methods

Milling parameters such as spindle speed, feed rate, tool diameter, and depth of cut were determined for MDF grooving milling tests, as shown in Table 1.

Table 1. Machining Parameters for MDF Grooving

Parameters	Unit	Level_1	Level_2	Level_3	Level_4
Spindle speed	rpm	18000	24000	-	-
Feed rate	mm/min	2500	5000	7500	10000
Depth of cut	mm	4	6	-	-
Tool Diameter	mm	4	6	-	-

Surface roughness was measured with a Handysurf E-35 measurement equipment (Tokyo, Japan) based on stylus technique (Fig. 3). Roughness measurement was performed from 10 different points on each samples. The average roughness ( $R_a$ ) and mean peak to valley height ( $R_z$ ) are considered roughness parameters and characterized by ISO 4287 (1997). The tip diameter was 5  $\mu\text{m}$ , and the measuring force was 4 mN. The surface roughness parameter was measured over a length of 1.25 mm and cut-off length of 0.25 mm. Experimental data of surface roughness was analyzed statistically using analysis of variance (ANOVA), which was performed at the confidence level of 95%. The parameters having a p-value less than 0.05 ( $p < 0.05$ ) were considered significant. The difference between the levels of the parameters with significant value ( $p < 0.05$ ) was determined by the t-test and Duncan test.



**Fig. 3.** The surface roughness measurement system

## RESULTS AND DISCUSSION

In this study, experiments were performed to determine the effects of milling parameters on surface roughness. Four different machining parameters (such as spindle speed, feed rate, tool diameter, dept. of cut) were selected for grooving on MDF with CNC router. A total of 32 experimental elements were produced and surface roughness was measured. Before the statistical analysis, a One-Sample Kolmogorov-Smirnov Test indicated that the surface roughness measurement data showed a normal distribution. The main and interaction effects of machining parameters on surface roughness values were analyzed with variance analysis (ANOVA) at a 95% confidence level. Table 2 and Table 3 present main and interaction effects of machining parameters on surface parameters  $R_a$  and  $R_z$ , respectively.

**Table 2.** Result of the ANOVA for  $R_a$

Source	DF	SS	MS	F-value	Sig.
Spindle Speed_A	1	5.118	5.118	7.815	0.006*
Feed Rate_B	3	15.674	5.225	7.978	0.000*
Depth of cut_C	1	0.228	0.228	0.349	0.555
Tool diameter_D	1	3.380	3.380	5.162	0.024*
A*B	3	0.432	0.144	0.220	0.883
A*C	1	0.018	0.018	0.027	0.869
A*D	1	0.660	0.660	1.007	0.316
B*C	3	4.705	1.568	2.395	0.069
B*D	3	2.138	0.713	1.088	0.354
C*D	1	0.284	0.284	0.433	0.511
A*B*C	3	1.462	0.487	0.744	0.527
A*B*D	3	1.907	0.636	0.971	0.407
A*C*D	1	1.492	1.492	2.278	0.132
B*C*D	3	3.466	1.155	1.764	0.154
A*B*C*D	3	1.548	0.516	0.788	0.501
Error	288	188.607	0.655		
Total	320	10600.009			
Corrected Total	319	231.121			

DF: Degrees of freedom, SS: Sum of squares, MS: Mean square, \*:p<0.05

There were significant effects of the spindle speed, feed rate, and tool diameter parameters on the  $R_a$  value and  $R_z$  at a confidence level of 95% (Tables 2 and 3). The depth of cut had no significant influence on surface roughness (*i.e.*,  $R_a$  and  $R_z$ ). There was no significant interaction between the model parameters (Tables 2 and 3).

**Table 3.** Result of the ANOVA for  $R_z$

Source	DF	SS	MS	F-value	Sig.
Spindle Speed_A	1	189.682	189.682	10.114	0.002*
Feed Rate_B	3	351.119	117.040	6.241	0.000*
Depth of cut_C	1	3.438	3.438	0.183	0.669
Tool diameter_D	1	120.651	120.651	6.433	0.012*
A*B	3	16.875	5.625	0.300	0.825
A*C	1	31.356	31.356	1.672	0.197
A*D	1	40.677	40.677	2.169	0.142
B*C	3	126.655	42.218	2.251	0.083
B*D	3	44.833	14.944	0.797	0.496
C*D	1	23.866	23.866	1.273	0.260
A*B*C	3	34.675	11.558	0.616	0.605
A*B*D	3	41.559	13.853	0.739	0.530
A*C*D	1	0.911	0.911	0.049	0.826
B*C*D	3	35.587	11.862	0.633	0.595
A*B*C*D	3	13.563	4.521	0.241	0.868
Error	288	5401.280	18.754		
Total	320	253768.290			
Corrected Total	319	6476.725			

DF: Degrees of freedom, SS: Sum of squares, MS: Mean square, \*:p<0.05

To examine the effects of levels of these machining parameters, t-tests were utilized to determine whether the differences associated with the two spindle speed factors (18000 and 24000 rpm) and two tool diameter factors (4 and 6 mm) were significant. The t-test results for the effect of spindle speed and tool diameter on the surface roughness are presented in Table 4. The effects of spindle speed and tool diameter levels on surface roughness were different. The surface roughness value ( $R_a = 5.82 \mu\text{m}$ ) at a spindle speed of 18000 rpm was higher than the surface roughness value ( $R_a = 5.565 \mu\text{m}$ ) at a spindle speed of 24000 rpm (Table 4). This result suggested that the surface roughness decreased with increasing spindle speed. The surface roughness values increased as the tool diameter increased. The surface roughness value ( $R_a = 5.795 \mu\text{m}$ ) at a tool diameter of 6 mm was higher than the surface roughness ( $R_a = 5.589 \mu\text{m}$ ) at a tool diameter of 4 mm (Table 4).

**Table 4.** Results of the *t*-test for Spindle Speed and Tool Diameter Level

Parameters	Level	N	Mean	SS	<i>t</i> _test	P
Spindle Speed	18000	160	5.818	0.838	0.627	0.008
	24000	160	5.565	0.848		
Tool Diameter	4	160	5.589	0.73401	11.689	0.031
	6	160	5.795	0.94528		

The effects of four different feed rate levels (2500, 5000, 7500, and 10000 mm/min) on surface roughness values were determined by the Duncan test, and the results are summarized in Table 5. There was no statistically significant difference between the effects of feed rate level 1 (2500 mm/min), level 2 (5000 mm/min), and level 3 (7500 mm/min)

on the surface roughness, while level 4 (10000mm/min) of feed rate was significantly different than the remaining levels (Table 5). The highest surface roughness value was obtained with feed rate of 10000 mm/min ( $R_a = 6,057 \mu\text{m}$ ), while the lowest surface roughness value was obtained with feed rate of 2500 mm/min ( $R_a = 5,486 \mu\text{m}$ ). The effects of the machining parameters on the mean surface roughness values are shown in Fig. 4. The surface roughness value increased with increasing feed rate and tool diameter and with decreasing spindle speed. The effect of the depth of cut parameter on the surface roughness value was negligible because of the same density in the profile area. Although the surface roughness decreased with increasing feed rate, the illustration shows that the average  $R_a$  value decreased slightly at 7500 mm/min of feed rate. This can be explained by the interaction effect of the tool diameter-feed rate. The interaction effects of machining parameters on the surface roughness are presented in Fig. 5. The interaction effect of spindle speed-feed rate, tool diameter-feed rate, and spindle speed-tool diameter feed influenced the surface roughness. However, the interaction effect of machining parameters was not significant. From the effect of dual interactions graph of spindle speed- feed rate, the surface roughness is found to be minimal at high spindle speed (24000 rpm) with low feed rate (2500 mm/min). In addition, the friction, machining force, and contact area between the cutting tool and the workpiece increases with the increased feed rate. Further, higher spindle speed means higher tooth passing frequencies and provides shorter plane area/reduction in chip thickness and hence the machining force and surface roughness decreases (Sarıkaya and Güllü 2014). The surface roughness is found to be minimal at high cutting speed with low feed rate. According to tool diameter- feed rate interaction, the high surface roughness occurred with an increase in tool diameter and feed rate. The reason for the high roughness can be explained with the increasing feed rate and tool diameter, causing vibration and a temperature rise between the work piece and the cutting tool (Suresh *et al.* 2012). The surface roughness values of the machined samples with 4 mm of tool diameter and 7500 mm/min of feed rate were observed, according to the processing conditions of 4 mm tool diameter and 5000 mm/min feed rate. However, with the increase in tool diameter (6 mm), the 5000 mm/min of feed rate provided a smoother surface quality than 7500 mm/min of feed rate. The effect of dual interaction graph of spindle speed-tool diameter shows that the surface roughness decreased with increasing spindle speed and decreasing tool diameter. Also, the best surface roughness was observed at a small tool diameter (4 mm) and a high spindle speed (24000 rpm). Compared to the surface roughness values with different spindle speeds (18000 rpm and 24000 rpm) at 4 and 6 mm of depth of cut, the lowest roughness value was provided at 2400 rpm of spindle speed and 4 mm of depth of cut.

**Table 5.** Results of the Duncan Test for Feed Rate Levels

Feed_rate (mm/min)	N	Mean	HG
2500	80	5.486	a
7500	80	5.550	a
5000	80	5.676	a
10000	80	6.057	b*

HG: Homogenous group \*: The highest surface roughness value



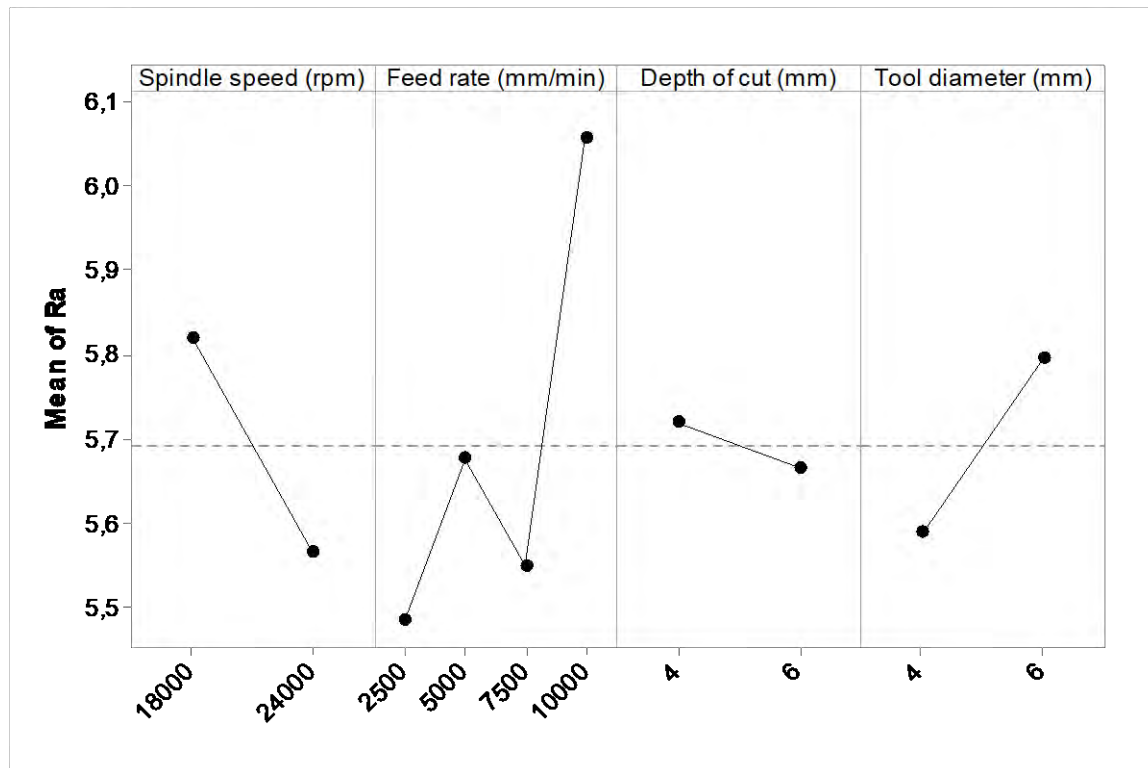


Fig. 4. Mean surface roughness values in machining parameters for  $R_a$

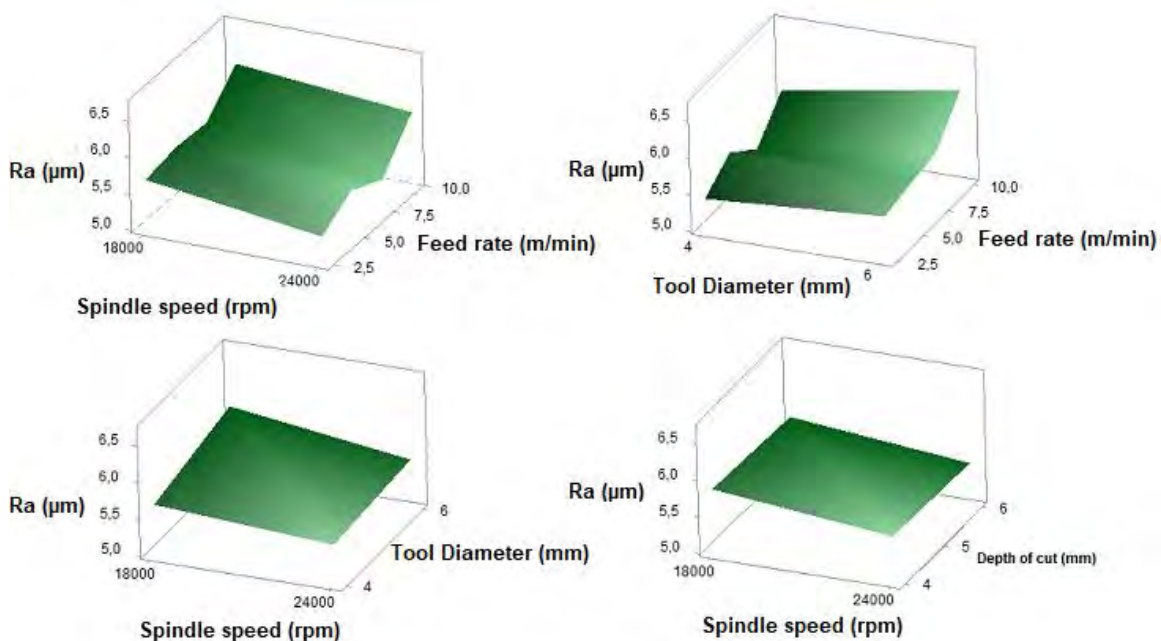


Fig. 5. The surface plots of  $R_a$  according to interactions effect of machining parameters

The main effects of factors (tool diameter, feed rate, spindle speed) on roughness were statistically significant, although interaction of factors had no effect on surface roughness. Spindle speed was a significant factor affecting surface roughness. The surface roughness decreased with increasing spindle speed. The spindle speed at 24000 rpm

provided the smallest surface roughness value. The feed rate at 10000 mm/min provided the highest surface roughness value, while feed rates of 2500 mm/min, 5000 mm/min, and 7500 mm/min provided similar results. The 4 mm tool diameter provided a lower surface roughness value than the 6 mm tool diameter. Depending on other machining factors, the roughness increased as the friction area between the material and tool increased with the increasing tool diameter. Previous studies indicated that the surface roughness decreases with increasing spindle and decreasing feed rate (Davim *et al.* 2009; Sütçü 2013; Sütçü and Karagöz 2013; Sofuoğlu 2015). The results of this study were consistent with the previous research.

Vertical density profile is one of the most important features that characterize the surface roughness properties of MDF in milling. The vertical density value decreases from the surface layer towards the core layer of MDF (Gupta *et al.* 2006). Therefore, the increasing the axial depth of the cut increases the roughness because the density of MDF layer decreases. Surface roughness increases with increasing axial depth of cut in MDF milling (Aguilera *et al.* 2000; Sütçü and Karagöz 2012; Sofuoğlu 2017). Deus *et al.* (2015) reported that surface roughness of MDF exhibits lower values with 1 mm depth of cut, higher spindle speeds, and lower feed rates. In this study, the statistical analysis indicated that there was no significant difference between axial depth cuts of 4 and 6 mm. Because the 4 and 6 mm depth of cut results in the same MDF profile density value, it had no effect on surface roughness (Fig. 1).

## CONCLUSIONS

1. Because depth of cut is same density zone in the MDF vertical density profile, the depth of cut is not an important factor affecting the surface roughness.
2. The increasing the tool diameter increases the cutting temperature, cutting force, and vibration intensity. In addition, tool wear occurs due to friction between the tool and the material. The value of surface roughness increased with the increase of tool diameter. The tool diameter of 6 mm achieved surface roughness higher than 4 mm tool diameter.
3. The value of surface roughness decreased with increasing spindle speed. Spindle speed at 24000 rpm were able to produce the best surface roughness in milling operation.
4. The value of surface roughness increased with the increase of feed rate. Feed rate at 10000 mm/min provided roughest surface from other feed rate levels (2500, 5000, 7500 rpm).

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## New PeCOD L50 COD Analyzer

The revolutionary PeCOD L50 analyzer provides accurate chemical oxygen demand (COD) results in 15 minutes, without the use of harmful chemicals such as dichromate and mercury. PeCOD L50 is the fastest available method for quantifying COD, providing real time data that allows operators to make timely decisions and generate savings on chemical and energy use.

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QCL supplies rapid analytical solutions for food and dairy testing laboratories and processing plants in the UK, enabling them to achieve better precision, performance and productivity. Established for more than 25 years, QCL is the exclusive distributor of several overseas manufacturers focusing in the areas of rapid diagnostic testing.

What follows is a case study using the PeCOD analyser.

The Paper Industry Technical Association (PITA) is an independent organisation which operates for the general benefit of its members – both individual and corporate – dedicated to promoting and improving the technical and scientific knowledge of those working in the UK pulp and paper industry. Formed in 1960, it serves the Industry, both manufacturers and suppliers, by providing a forum for members to meet and network; it organises visits, conferences and training seminars that cover all aspects of papermaking science. It also publishes the prestigious journal *Paper Technology International* and the *PITA Annual Review*, both sent free to members, and a range of other technical publications which include conference proceedings and the acclaimed *Essential Guide to Aqueous Coating*.



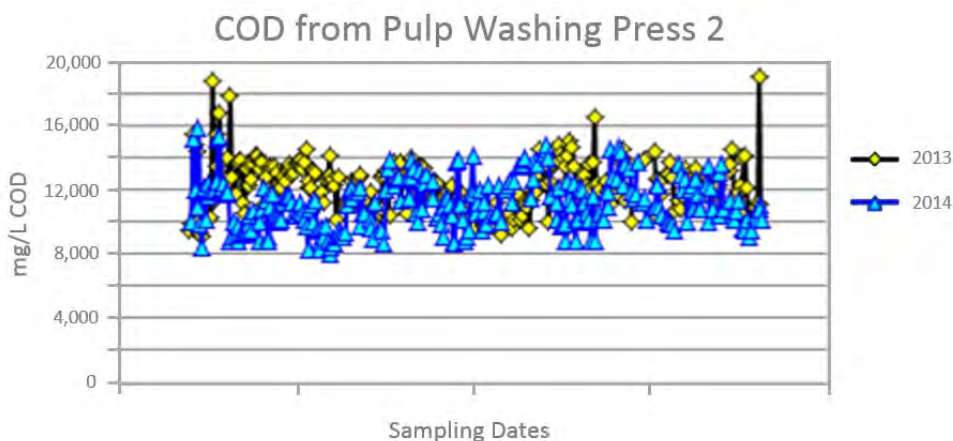
## PeCOD® Case Study #03

### Pulp and Paper

The pulp and paper industry utilizes wood as raw material to produce pulp, paper, card board, and other cellulose based products. Pulp and paper mills produce large volumes of wastewater and residual sludge which presents a number of challenges with regards to treatment and discharge. Contaminants in pulp and paper waste streams often include effluent solids, sediments, absorbable organic halides (AOX), chlorinated organic compounds, chemical oxygen demand (COD), and biochemical oxygen demand (BOD). In many cases, the delignification and wash pulp steps require excessive volumes of bleaching chemicals which contribute significantly to the higher COD contamination typical of this wastewater.



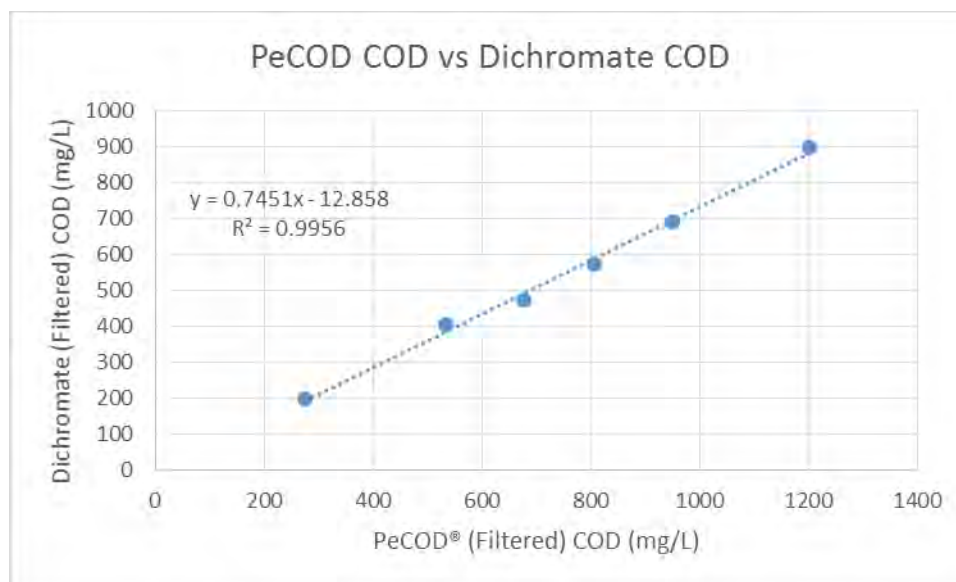
A Chilean multi-national paper company invested in a MANTECH PeCOD® COD Analyzer to improve the health and safety of operators and to significantly reduce analysis time. By continuously monitoring COD levels (less than 15 minutes), the mill was able to optimize the pulp process and reduce the consumption of bleaching chemicals. The decrease in chemical consumption resulted in a lower amount of organics to treat, and in turn, reduced treatment chemicals, energy and discharge fines. **Figure 1** demonstrates the reduction in COD levels between 2013 and 2014, since implementing the PeCOD® COD Analyzer. The pulp and paper mill saved greater than \$10,000 (USD) per day by optimizing treatment processes, reducing energy usage and decreasing chemical usage. Savings of 3 million US dollars per year yields a return on investment of just 3 days. The PeCOD® COD Analyzer has provided critical information to the operators while improving health and safety to the community and the environment.



**Figure 1:** A graph of PeCOD® COD results from the pulp washing step at a pulp and paper mill. Wastewater samples were collected and analyzed over a two-year period.

Another PeCOD project at a facility located in Quebec, Canada, focuses on improving the sustainability of the forestry industry by monitoring effluent wastewater produced by the pulp and paper industry. The traditional dichromate method has historically been used to monitor COD levels for this purpose. However, due to PeCOD's analysis speed and environmentally friendly nature, mills can optimize their processes and improve the health and safety of their staff in a safe and timely manner. Validation studies for the peCOD COD method are important for reporting purposes, as the traditional dichromate method is the industry standard for COD analysis.

The facility in Quebec is utilizing the PeCOD analyzer to test a range of effluent samples produced by an onsite mini-pilot plant, in addition to samples from established pulp and paper mills. **Figure 2** represents effluent wastewater samples measured for COD by both the peCOD and the dichromate method. A strong correlation is observed between peCOD COD and dichromate COD ( $R^2 = 0.9956$ ). This correlation indicates that the PeCOD technology is suitable for use in the pulp and paper industry as it is a reliable predictor of the regulated dichromate method for COD.



**Figure 2:** A linear regression of PeCOD® COD Analyzer results vs Dichromate COD results. A strong correlation is observed shown by an  $R^2$  value of 0.9956.

The PeCOD analyzer uses nanotechnology encompassing titanium dioxide, a powerful oxidizer with a higher oxidizing potential than dichromate. The results shown above support this fact, as peCOD COD measured slightly higher than dichromate for each of the samples. It is important to note that sample preparation for COD measurement by both peCOD and dichromate made use of a 35uM filter to remove any solid particulates from analysis and is thus a measure of soluble COD.

Both presented cases reduced COD analysis time significantly while validating the PeCOD with the traditional dichromate method. The first pulp and paper mill reported a decrease in response time by 95% and a decrease in cost for running the COD test by 66.4%. As a result, the PeCOD® COD Analyzer plays an important role in the pulp and paper industry by providing valuable financial savings to the mill, promoting process optimization, and supporting environmental efforts by establishing a green COD method; all of which contribute to forestry sustainability.

**Author:** Katelyn Wanka, B.Sc., M.BINF., Quality Control and Research Chemist at MANTECH



## Reducing energy use and greenhouse gas emissions

This article focuses on controlling bacterial growth in paper making with the addition of lime which contributes to less slime formation and decreases fatty acid production.

Conclusions will be drawn from measurements where the correlation between drying section temperatures and lime dosing has been proved. The resulting reductions in energy consumption and emissions will lend support to the outcomes identified.

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# Reducing energy use and greenhouse gas emissions

Dosing accuracy in the paper making industry

## Key conclusions

- Peristaltic chemical metering technology can help cut gas consumption, water usage and greenhouse gas emissions
- An important aspect of pulp production is to ensure neutral pH of pulp with the addition of lime
- Only by investing in the latest technologies can industry continue complying with strict and ever-tightening environmental regulations

## Executive summary

This article focuses on controlling bacterial growth in paper making with the addition of lime which contributes to less slime formation and decreases fatty acid production.

Conclusions will be drawn from measurements where the correlation between drying section temperatures and lime dosing has been proved. The resulting reductions in energy consumption and emissions will lend support to the outcomes identified.



Above: A Qdos 120 pump used for dosing lime slurry

### Introduction

Paper making uses large volumes of water, 90% of which is used for cooling and 10% as process water. Fibre is mixed with water at the stock preparation stage of the process in order to create pulp. This step is followed by forming and dewatering stages, where vacuum is applied to drain the water. Any remaining water in the product is evaporated in the drying section, most often by gas-heated ovens or steam-heated drying cylinders.

The paper drying section consumes most of the energy in the manufacturing process, and contributes to 68% of the total greenhouse gas emissions for the sector. However, increasing performance in the dewatering stage delivers the potential for less water to be heated and evaporated in the drying section.

To enhance dewatering, additives are used to increase drainage of the paper fibres. The calcium carbonate content

of the raw material also has a significant influence on dewatering, an area which seems underestimated by most process technologists.

### The problem

The reality is that the forestry industry contributes 5% to global greenhouse gas emissions and uses 2% of the world's energy industry-wide. As a result pulp and paper mills face strict and tightening regulations towards environmental issues. Apart from ambitious water reuse targets emissions need to be reduced significantly, which challenges the future profitability of many paper mills.

### The solution

The deployment of accurate chemical dosing technologies at paper mills can deliver a significant reduction in energy usage and greenhouse gas emissions. To evaluate the feasibility of this thinking, comprehensive trials took place at Huhtamaki OY, a Finnish multinational and

## Lime dosing accuracy has a significant impact on process performance and end product quality

global leader in moulded fibre technology for food and drink packaging. Full-scale industrial trials took place at the company's central research facility in the Netherlands.

The dry stock material used for these trials was a mixture of recycled paper fibres with grass added.

An important aspect of pulp production is to ensure neutral pH of the pulp by the addition of lime. Adding lime contributes to lower bacterial growth and decreases the presence of fatty acids in the pulp. This ►





*Above: Qdos pumps used for colourant dosing*

► translates into less slime in the system and lowers the odour of the end product.

More importantly, closed loop water systems, common in paper manufacturing, struggle with a high calcium load. Water evaporates at the drying section but the calcium remains in the system causing scale formation.

By adding the lime at the wet end, the calcium will be enclosed in the paper floc (substrate). This will lower the calcium load as the calcium leaves the manufacturing process via the end-product. This also enhances dewatering, resulting in lower drying section temperatures and lower gas consumption.

Lime is a challenge to work with, as it tends to settle, forming lumps in suspension. Diaphragm pumps are often used to add lime. However this can cause difficulties as ancillary foot valves

tend to get blocked by lime dispersion. Even after removing the foot valve and pressure-holding valve on the discharge side, diaphragm pumps do not deliver a constant flow. As the accuracy of the quantity of lime dosed has a significant impact on the process performance and end-product quality, it is critical to select the optimal dosing pump for the task.

For the trials at Huhtamaki's site, a Qdos 120 peristaltic chemical dosing pump from Watson-Marlow Fluid Technology Group was used to deliver the lime prior to dewatering the paper pulp.

## The technology

The selection of a chemical dosing pump often has a significant impact on the efficiency of a water treatment or manufacturing process. According to the specification sheets, most chemical dosing pumps deliver similar performance, although this test data is often based

## Dewatering was far more effective, resulting in an increase of 2.5% in dry solid content

on ideal circumstances, which are rarely the case in practice. In truth, process conditions vary, and end-users simply like to install a dosing pump at plug and play level into their process.

Qdos pumps deliver accurate flow, despite changing process conditions, and do not require additional ancillaries like inlet and pressure-holding valves. This lowers the cost of maintenance and process downtime due to clogged valves.

Huhtamaki recognised the issues with diaphragm pumps some years previously, and decided to participate in the development of Qdos peristaltic metering pumps for a number of paper pulp duties. Since the adoption of this new technology, various process enhancements have been made, achieving increased efficiency, improved product quality, safer maintenance and significant savings on chemical usage.

Driven by Huhtamaki's ongoing ambition to be the best moulded fibre packaging company worldwide, it decided to establish a project to bring down its greenhouse gas emissions to a minimum, and maximise the reuse of its process water.

The project set out to optimise the calcium load in their system and impact on the company's CO<sub>2</sub> emissions. By dosing lime with high accuracy, Huhtamaki succeeded in achieving optimal control over the pH. The trial proved that the dewatering stage was far more effective than it had been, resulting in an increase of 2.5% dry solid content at the entrance of the drying section. Moreover, less water had to be heated up to 100C and evaporated. ►

► There is a direct correlation between the dry solid content at the entrance to the oven and the required oven temperature. By accurately metering lime to the pulp, Huhtamaki succeeded in lowering the average oven temperature over the course of the trial.

### Summary

Due to the nature of the operation at Huhtamaki (24/7 for more than 350 days a year), this relatively modest decrease had a significant impact. A lowering in oven temperature of 15C correlates to a 3% reduction in gas consumption and, consequently, 3% reduction in greenhouse gas emissions. This equals a reduction of 18,000kg in carbon emissions per oven, per year, delivering a return on investment in just four weeks.

**Additional benefits in the addition of lime include: higher machine throughput (estimated at 5%) due to lower equipment pollution levels; less product shrinkage thanks to lower oven temperature; low product odour and lower odour emissions.**

Any future research or development on chemical dosing equipment should focus on ultra-low flow. The new array of nano-scale high-performance chemicals will make an entrance into the paper and pulp industry in the near future. With this in mind, high precision chemical metering will be a necessity as the industry strives for fully closed loop water systems and zero emissions.

# 3%



A lowering in oven temperature of 15C correlates to a 3% reduction in gas consumption and 3% reduction in greenhouse gas emissions.

A return on investment was delivered in just four weeks.

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## Guidance on Safe Operation of Steam Boilers

Ref: BG01 - A joint document by the Safety Assessment Federation and The Combustion Engineering Association produced originally in consultation with the Health & Safety Executive.

This document, Guidance on the Safe Operation of Steam Boilers (BG01) has been developed and written by the Combustion Engineering Association (CEA) and the Safety Assessment Federation (SAFed) in consultation with other stakeholders within the boiler industry to help designers, managers and operators of new and existing boiler systems make health and safety and environmental improvements in the industry.

This revision (Edition 2) incorporates up-to-date information and best practices relating to the operation of steam boiler plant; hot water boilers will now be covered in a separate document.

This publication should not be regarded as an authoritative interpretation of the law, nor a mandatory legal requirement. However, if the guidance provided is followed, it will normally be regarded as sufficient to comply with the relevant health and safety duties.

The Paper Industry Technical Association (PITA) is an independent organisation which operates for the general benefit of its members – both individual and corporate – dedicated to promoting and improving the technical and scientific knowledge of those working in the UK pulp and paper industry. Formed in 1960, it serves the Industry, both manufacturers and suppliers, by providing a forum for members to meet and network; it organises visits, conferences and training seminars that cover all aspects of papermaking science. It also publishes the prestigious journal *Paper Technology International* and the *PITA Annual Review*, both sent free to members, and a range of other technical publications which include conference proceedings and the acclaimed *Essential Guide to Aqueous Coating*.



# Guidance on Safe Operation of Steam Boilers

Ref: BG01



A joint document by the Safety Assessment Federation  
and The Combustion Engineering Association produced  
originally in consultation with the Health & Safety Executive.



**CEA**  
Combustion Engineering Association



Guidance on the Safe Operation of Steam Boilers (Ref: BG01)

A joint document by The Combustion Engineering Association  
and the Safety Assessment Federation  
originally produced in consultation with the Health & Safety Executive.

Edition 2 – March 2019

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### Foreword

This document, Guidance on the Safe Operation of Steam Boilers (BG01) has been developed and written by the Combustion Engineering Association (CEA) and the Safety Assessment Federation (SAFed) in consultation with other stakeholders within the boiler industry to help designers, managers and operators of new and existing boiler systems make health and safety and environmental improvements in the industry.

This revision (Edition 2) incorporates up-to-date information and best practices relating to the operation of steam boiler plant; hot water boilers will now be covered in a separate document.

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Cover image courtesy of Vital Energi.

### Acknowledgements

SAFed and CEA acknowledge the support of the Health and Safety Executive in producing the original guidance, this second edition recognises changes in legislation and guidance since the first edition and incorporates comments and suggestions from users arising from widespread use of this guidance document in industry.

The Safety Assessment Federation (SAFed) is a trade association which represents the independent engineering inspection and certification industry in the UK; their primary aim is to promote safety and reduce accidents in the workplace. SAFed supports corporate social responsibility through compliance with the law and adopting best industry practice.

The Combustion Engineering Association (CEA) is an educational charity which promotes the science of combustion engineering in the commercial and industrial sector. The CEA is concerned with industry good practice and the safe and efficient operation of combustion related plant and equipment.

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### In this document the following words convey specific meaning:

**Should:** Compliance with this clause is not essential where supported by risk assessment and/or design calculation.

**Shall:** Compliance with this clause is required in order to claim compliance with this document.

**Must:** Compliance with this clause is a legal requirement within the United Kingdom.

Unless otherwise stated, all pressures refer to gauge pressure.



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## 1 INTRODUCTION

Guidance on the Safe Operation of Steam Boilers (Ref: BG01) is a guidance document intended to assist the designers, managers, operators, maintenance personnel and Competent Persons (CP) of new and existing steam boiler systems in addressing the following issues:

- The safe and efficient use and operation of the boiler installation;
- Determining adequate supervision and maintenance requirements (levels and competence) that are consistent with the installed plant and its location;
- Reducing the likelihood of explosion or other dangers from events such as:
  - Loss of feed water or low water level;
  - Over-pressure;
  - Overheating e.g. due to excessive scale;
- Using efficient boiler operation to avoid excessive pressure or thermal cycles and load swings which can accelerate boiler fatigue or failure;
- Having the proper treatment and monitoring of the feed water and condensate to:
  - minimise corrosion and scale; and
  - avoid carry-over of water with the steam which in turn can cause water-hammer and other issues;
- Compliance with the various legal requirements, in particular that for periodic examination by a CP in accordance with a Written Scheme of Examination (WSE).

## 2 SCOPE

This document applies to all industrial & commercial steam boiler plant (normally shell boilers) operating at a working pressure up to 32 bar gauge, including vertical boilers, mobile steam boilers and waste heat boilers.

The following boilers are **specifically excluded** from the scope of this Guidance Document:

- Hot water boilers;
- Water tube boilers;
- Steam boilers with a capacity exceeding either:
  - 37 MW nett rated thermal input, or
  - 32 bar gauge working pressure;
- Domestic and commercial boilers with a capacity less than 70 kW;
- Electric immersion boilers, electrode boilers and steam coil heated boilers;
- Steam coil boilers (steam generators);
- Boilers used for transport.

However, just because these boilers are outside the scope of BG01, this does not mean that the regulations and general principles in this document should not be applied where suitable and applicable.

Note that this edition of BG01 excludes hot water boilers which will be the subject of a new guidance note BG02 – Guidance on the Safe Operation of Hot Water Boilers.

### 3 LEGISLATION

Boiler systems are required to comply with different legislation, including a number of health and safety and environmental regulations, which are aimed at ensuring that new and existing boiler systems are continually designed, installed, operated and maintained in a safe manner.

The principal sets of health and safety legislation that support the Health and Safety at Work etc. Act 1974 and apply to the use of boiler systems covered by this guidance are:

- The Management of Health & Safety at Work Regulations (MHSWR);
- The Pressure Equipment (Safety) Regulations (PER);
- The Pressure Systems Safety Regulations (PSSR);
- The Provision and Use of Work Equipment Regulations (PUWER); and
- The Dangerous Substances and Explosive Atmospheres Regulations (DSEAR).

With the exception of MHSWR and PER, all the regulations listed above are supported by Approved Codes of Practice (ACoP) and Guidance produced by the Health and Safety Executive (HSE), and available as free downloads from [www.hse.gov.uk](http://www.hse.gov.uk).

There are numerous sets of environmental legislation applicable to steam boilers, including the Clean Air Act, the Industrial Emissions Directive, and the Environmental Permitting Regulations (including the Medium Combustion Plant Directive). Relevant legislation is addressed in the following text.

Refer to Appendix 1 for a list of currently applicable legislation. It is the reader's responsibility to ensure that they refer to the latest available version of any legislation or guidance.

#### 3.1 The Management of Health and Safety at Work Regulations (MHSWR)

The Management of Health and Safety at Work Approved Code of Practice (ACoP – L21) has been withdrawn and is no longer available. For those looking for information on how to manage risks in their business, HSE has a suite of guidance that will be able to help. Each level of guidance on HSE's website offers appropriately targeted information, focussed on making compliance as straightforward as possible.

If you need basic information or are getting started in managing for health and safety, then the best place to look is *Health and safety made simple: The basics for your business* (INDG449). You should also consult: *Safe management of industrial steam and hot water boilers. A guide for owners, managers and supervisors of boilers, boiler houses and boiler plant* (INDG436).

MHSWR apply to every employer and self-employed person who carries out any work activity whether or not they own or use a pressure system (all future references to employers in this guidance should be read to include self-employed persons).

They impose a duty to manage all risks from any work activity, not only within the workplace itself, but also any risks to all persons (including any non-employees) who may be affected by the activity in question.

Regulation 3 requires the completion of a suitable and sufficient risk assessment of the work activity in order to properly identify and adequately manage any risks. This is of central importance. The risk assessment must identify sensible measures to control identified risks that may otherwise result in injury or danger.

Risk assessments for boiler systems are covered in more detail in the next section.

### 3.2 The Pressure Equipment (Safety) Regulations (PER)

PER applies to the design, manufacture and conformity assessment of pressure equipment and assemblies of pressure equipment with a maximum allowable pressure >0.5 bar.

All items of new and substantially modified pressure equipment (including steam raising plant) comes within the scope of PER and they must comply with its requirements before they may be supplied for use.

The Regulations do not apply to:

- Excluded pressure equipment and assemblies (specified in Schedule 1 to PER); or
- Pressure equipment and assemblies placed on the market before 29 November 1999; or
- Pressure equipment or assemblies placed on the market on or before 29 May 2002 if they comply with the safety provisions in force in the UK on 29 November 1999 and do not bear a CE marking (unless required by another European Community Directive or any indication of compliance with PED).

Schedule 2 of PER details the essential safety requirements (ESR) that qualifying vessels must satisfy. Additionally, there are details of how the different products are classified, the technical requirements that must be satisfied, and the conformity assessment procedures that must be followed.

To comply with the ESRs the manufacturer must either produce a technical file that addresses each ESR in turn, or manufacture the equipment using standards that have been listed in the EU's Official Journal which give a 'presumption of conformity' to specific ESRs.

The Department for Trade and Industry (DTI) produced a very useful guide: *PRODUCT STANDARDS Pressure Equipment – GUIDANCE NOTES ON THE UK REGULATIONS APRIL 2005 URN 05/1074*. This document can be found through: <https://www.gov.uk/guidance/pressure-equipment-manufacturers-and-their-responsibilities>.

There is an easy-to-use flow chart in the DTI guide (Annex C) showing how equipment should be classified depending on, for example, what it is designed to contain and the operating pressure. This includes the conformity assessment procedure to be followed before placing the equipment on the market.

### 3.3 Pressure Systems Safety Regulations (PSSR)

PSSR set out the main legislative requirements to ensure the continued safety of the pressure systems in use (which includes steam boilers). PSSR applies to two clearly defined categories of people (**duty holders**). These are the

- **'Owner'** – an employer or self-employed person who owns a pressure system. Where the employer who owns the system does not have a place of business in Great Britain, or an agent in Great Britain who would take responsibility, then the user (see below) will be responsible; and the
- **'User'** – the employer or self-employed person who has control of the operation of the pressure system.

The distinction between **'Owner'** and **'User'** can be important in certain circumstances in determining the duty holder responsible for ensuring compliance with certain regulations under PSSR. However, in general, owners carry more responsibility in relation to mobile systems (but see "Temporary Boiler Plant" below), while users have responsibilities in relation to installed systems. Shell boilers are considered to be 'installed systems' for the purposes of the regulations.

The user/owner of the boiler is responsible for complying with the following requirements of PSSR:

- Safe Operating Limits (SOL) have been set and are not adjusted without informing the Competent Person (CP) and manufacturer where appropriate;
- The system is never operated unless a current Written Scheme of Examination (WSE) is in place. Any requirements of this scheme e.g. a report of the last examination, must also be satisfied (Regulations 8 & 9);
- The items identified in the WSE must be examined by a CP in accordance with the requirements of the scheme;
- The results of all tests and examinations must be recorded by the CP (Reg 9) and retained by the user/owner for a suitable period (see Log Sheets, Appendix 4). A period of at least two years is recommended for retention of records of routine tests (see section 8);
- All repairs and modifications shall be carried out by people suitably competent in such work (Regulation 13, PSSR, ACoP Para 176). You must discuss and agree any changes with the "Competent Person" and include any changes within your written scheme of examination (WSE) (ACoP Para 116,117). The details of such work shall be retained for the life of the plant;
- The statutory technical documentation and other records must be kept and where required, be made available for examination.

All records may be kept on-site or at a designated central location but wherever they are kept, they must be secure and easily accessible, and records must be transferred when the ownership of a system changes (Regulation 14, PSSR).

The user must give operational employees adequate instruction so that the boiler can be operated safely (Reg 11 and para 145 ACoP). For a steam boiler these should include (paras 151 & 152) instructions covering:

- pre-firing and start-up instructions;
- feed water treatment; (see BG04)
- safe blowdown of the boiler (see BG03);
- precautions to be taken when emptying the boiler;
- precautions to ensure positive isolation and depressurisation of one boiler from a common header and blowdown system if internal access is required;
- precautions to be taken before carrying out maintenance operations;
- procedures to be followed in the event of a shortage of water, bursting of tubes or other event requiring the boiler to be shut down.

**Temporary Boiler Plant:** Companies who hire out steam boilers are usually hiring out a pressure system. Para 39 of the PSSR ACoP says that a steam boiler [fitted with skids] may be installed temporarily to maintain steam supply to the site during the replacement of an existing boiler, but such an installation should not be treated as a mobile system. So mobile steam boilers are not in fact mobile plant for the purposes of PSSR, and where a person supplies an installed system by way of lease or hire, and agrees in writing to be responsible for discharging the duties of the user, all the provisions of regulations 8(1) and (2), 9(1), 11(1), 12 and 14 must be followed (Reg3(5)) and the requirements of PSSR Schedule 2 must be followed.

CEA BG08 Guidance on Temporary Steam and Hot Water Boiler Plant contains detailed information regarding the safe use of temporary boiler plant.



### 3.4 Provision and Use of Work Equipment Regulations (PUWER)

Any employer who either provides equipment for use at work (including boiler systems) or has control over the way and manner in which equipment is used at work has a legal responsibility to comply with the relevant provisions of this regulation. An important, often overlooked, requirement under PUWER is that a maintenance logbook, when provided, must be kept up to date.

Under PUWER, all employees required to use equipment at work must be trained to do so (Reg 9). This will therefore extend to the competence assessment and training of operators and managers of boilers, all ancillary plant, and any feed water treatment plant used for the boilers.

Other parts of PUWER of relevance to boiler systems cover such topics as equipment suitability, maintenance, inspection, information & instructions, and control systems. This is not an exhaustive list.

### 3.5 The Construction (Design and Management) Regulations (CDM)

Although installing or replacing a steam boiler might not be a large enough project on its own to be notifiable under CDM, the principles of the regulations should still be followed, and if the steam boiler is part of a major installation the regulations will apply in full and must be considered at every stage of the project from conceptual design through installation to maintenance and ultimate demolition.

Clients must appoint a Principal Designer and a Principal Contractor to ensure that the CDM Regulations are properly followed.

### 3.6 The Dangerous Substances & Explosive Atmospheres Regulations (DSEAR).

A risk assessment under DSEAR must be undertaken. DSEAR applies to all boilers (not just gas fired) as incorrect combustion can lead to an explosive atmosphere in the boiler itself or indeed in a separate combustor or CHP engine exhaust.

The owner of the system may assist the manufacturer by providing information from an assessment of the probability of the presence and the likely persistence of a potentially explosive atmosphere in the proposed working environment.

Equipment supplied for use in a potentially explosive atmosphere must also satisfy the relevant requirements of the *Equipment and Protective Systems Intended for Use in Potentially Explosive Atmospheres Regulations*.

### 3.7 The Environmental Permitting Regulations

All combustion plants rated between 1 MW and 50 MW nett rated thermal input will be required to comply with the Medium Combustion Plant (MCP) Directive which has been transposed into UK legislation through changes to *The Environmental Permitting (England and Wales)(Amendment) Regulations*, *The Pollution Prevention and Control (Scotland) Amendment Regulations*, and *The Pollution Prevention and Control (Industrial Emissions) (Amendment) Regulations (Northern Ireland)*.

This legislation requires the registration of all new combustion plants put into first use after 19 December 2018 and the registration of existing combustion plant before 01/01/2024 for plants individually 5 MW and above, and 01/01/2029 for plants rated from 1 MW to <5 MW. Where more than one new plant is on a site the new plants will be aggregated to a single MCP.

From the date of first use (in the case of new plants after 20/12/2018) and from 01/01/2025 (for existing 5-50 MW plants) and 01/01/2030 (for existing 1-5 MW plants) the emissions from those combustion plants must not exceed specified emission limit values (ELV) for NO<sub>x</sub>, SO<sub>x</sub> and dust (total particulates), and these will be measured at specified intervals along with CO (no limits currently set for CO). Plants rated 20 MW and above will be measured annually, and plants below 20 MW will be measured every 3 years.

The Environment Agency (EA) in England and their equivalents in the devolved UK administrations will administer the new legislation and will consult with Local Authorities where there may be a combustion plant in or close to a Local Air Quality Management zone. This may mean tighter ELVs will be applied. Sites that currently have environmental permits for other activities will have any MCPs added to their permits at the due date.

The EA have produced detailed guidance on how these regulations will be applied.

## 4 LEGAL RESPONSIBILITIES

### 4.1 Risk assessments – for new and existing sites

Regulation 3 of MHSWR requires that a 'suitable and sufficient' risk assessment be carried out before the work activity commences. Its purpose is to determine whether any risks are present and, if they are not adequately managed, what further control measures are required. The significant findings of the risk assessment must be recorded where there are 5 or more employees.

The control measures must have the primary aim of eliminating the risks. Where elimination is not possible, the control measures must aim to reduce the risks to a level as low as is reasonably practical (ALARP). Regulation 4 and Schedule 1 of MHSWR sets out the principles of prevention.

The responsibility for the risk assessment lies with the employer although he may do this using input or assistance from various sources such as boiler manufacturers and control system experts, or have the entire risk assessment carried out on his behalf by someone competent to do so.

For a boiler, the risk assessment should consider issues such as:

- The likelihood and severity of injuries from:
  - Burns from hot water, steam, burners and flues;
  - Electric shock;
  - Fuel escape;
  - Fire;
  - Asphyxiation, and toxic effects from combustion products;
  - Falls from height;
  - Impact by a moving vehicle (particularly sites using solid or liquid fuels)
- The location of the boiler with respect to:
  - Numbers of persons likely to be affected;
  - Proximity to industrial premises/workers;
  - Proximity to the public especially vulnerable populations - such as in nurseries, schools, hospitals, care homes etc.;
  - The potential impact on neighbouring sites due to an incident;
- Capability of safety-related systems;
- Level of supervision;
- The positioning of alarms and the associated response times;
- The presence of other dangerous materials;
- The adequacy of boiler house ventilation and flue integrity;
- Environmental effects, e.g. noise, pollution;
- Effect of chemicals on workers, the environment and others, e.g. water treatment chemicals;
- Operational risks:
  - Mechanical or water damage to plant or equipment;
  - Water-side explosion due to catastrophic failure of the pressure envelope;
  - Combustion explosion caused by unspent fuel;
  - Failure of the water treatment equipment to deliver properly treated water to the boiler;
  - Speed of response to loss
  - of steam to process.

Since risk assessments must assess the existing control measures, they should also consider information regarding:

- Manning and supervision (see section 5.9 below);
- Type and reliability of controls and the integrity of safety-related systems;
- Additional controls for remote or unsupervised boiler operation.

Risk assessments must be reviewed periodically, after any accident or incident, and when there is a significant change e.g. a system variation, change in operating parameters or manning levels etc. The outcome of any reviews must be recorded.

As an example, an owner moving to a lower level of supervision of the boiler shall, as a first step, review the boiler design and the current risk assessment to take account of the planned change in manning levels. The results of the risk assessment will be used to determine any measures necessary to ensure that the boiler remains safe to use and to operate. Such measures may include:

- The proper formulation and correct application of all modifications and installations to ensure they have sufficient safety integrity to adequately mitigate the risk of a dangerous occurrence;
- Amendment of procedures where appropriate to ensure the plant continues to be operated safely;
- Ensuring all personnel on-site & off-site and in surrounding property remain safe.

#### 4.2 Written scheme of examination (WSE)

The requirement for a WSE is set out in Regulation 8 of PSSR. The user/owner is ultimately responsible for ensuring that the scope of the WSE covers all relevant parts of the boiler system, and they should select an organisation with sufficient knowledge and expertise on the systems in question to carry out the CP duties on that system.

The CP role and responsibilities are covered in the PSSR ACoP. A brief summary is provided in section 5.3 below.

The WSE must include the name of the CP who certified the scheme as suitable, the date of the certification, and the following information:

- All parts which require examination by the CP;
- Justification for excluding items from examination;
- All protective devices;
- The nature and frequency of the examinations required;
- Details of any preparatory work required by the user/owner in order for the examinations to be completed;
- Details of any requirements for the initial examination;
- Details of any repairs and modifications where the CP needs to be involved.

Where there is more than one WSE for a single pressure system, (e.g. one for the boiler house and another covering the site) or there are hired boilers brought to site, the respective responsibilities for each part of the pressure system must be clearly identified. The boundaries of each WSE must be adjacent to each other with no physical gaps.

### 4.3 Examinations in accordance with the WSE (Thorough Examinations)

Regulation 9 of PSSR requires that all pressure systems be periodically examined by a Competent Person (CP) in accordance with a WSE, itself being drawn up by a CP.

The user/owner is responsible for ensuring their boilers meet this requirement. Where the WSE specifies any preparatory work, they are also responsible for ensuring that this is completed before the examination.

As soon as possible following examination, the CP will prepare a report of examination for the user/owner. The report will also include, amongst other information, the following:

- Whether any repairs are required and the date by which they must be completed;
- The latest date by which the next examination must be carried out;
- Whether any modifications are required to the WSE.

Note that the CP may also specify the manner and procedures which these modifications should take. The CP may also specify the nature of the required modifications to the scheme.

If any of these issues are raised in the report of examination, the user/owner must:

- Ensure that the boiler is not used or supplied if the date set for any repairs or examinations passes without these being completed;
- Make the required modifications to the WSE and have it re-certified by a CP;
- Ensure the boiler is not used or supplied if the date set for the modifications to the WSE passes without these being implemented and certified by a CP.

### 4.4 Summary of responsibilities

The user/owner of a boiler system is ultimately responsible for ensuring the system complies with all the relevant Health & Safety legislation (not just those responsibilities mentioned above).

While third parties, e.g. maintenance contractors, can be used to assist in achieving compliance with these legal obligations, the overall and legal responsibility remains with the user/owner and cannot be contracted out although there is scope for certain duties to be transferred (as set out in a written agreement) between the owner and user.

Useful help and advice on ensuring boiler systems remain safe to operate can be obtained from a number of sources, such as the CP carrying out the periodic examination of the boiler, or from the equipment manufacturer.

## 5 PERSONNEL AND RESPONSIBILITIES

### 5.1 User/owner

These legal terms have earlier been defined in section 3. The distinction between these terms is important as it will determine the duty holder responsible for ensuring compliance with certain regulations under PSSR. Similarly the duties have been outlined in sections 3 and 4 above.

In general, the legal responsibilities of the user/owner cannot be transferred e.g. by an employer to an employee. In situations where more than one employer or self-employed person may have an interest in the operation of a plant, para 46 of the ACoP to the PSSR provides guidance as to who is the user. It may however be prudent to take legal advice on the matter in this type of situation as it must be clear to all parties who is responsible under the Regulations.

### 5.2 Competent Person (CP)

A Competent Person (CP) is defined in Regulation 2, PSSR as "a competent individual person (other than an employee) or a competent body of persons corporate or unincorporate and accordingly any reference in these Regulations to a CP performing a function includes a reference to him performing it through his employees."

From para 10 of the PSSR ACoP this term refers to the organisation employing the person who carries out these duties. Therefore, the legal duty to comply rests with a CP's employer, and not with an individual, unless that person is self-employed.

A CP is required to undertake two distinct functions under PSSR:

- To draw up, certify or review the written scheme of examination; and
- To carry out the examinations in accordance with the scheme and to produce a report after each examination.

These roles may be undertaken by the same or more than one organisation. The user/owner remains responsible for selecting a CP who possesses sufficient expertise in the particular system and is capable of carrying out the duties in a proper manner. A CP is also able to act in an advisory role and advise on other aspects of PSSR such as the scope of the written scheme and establishing the safe operating limits of pressure systems.

In addition to the above legally defined personnel, there are also a number of other personnel involved in the day to day safe operation of boilers. These are discussed below but it should be borne in mind, these may not be terms that have a legal definition.

### 5.3 Employers

Under the Health & Safety at Work etc Act 1974 (HSWA), employers have general duties, amongst other things, to provide safe places of work and adequate training for staff. This general duty on employers is also required under other legislation such as MHSWR and PUWER. **This legal responsibility cannot be transferred to employees or third parties.**



## 5.4 Employees managing the operation of boiler plant

Employers must appoint sufficient suitably trained and competent persons to be responsible for the safe management and operation of boiler systems. These supervisors or managers must be adequately trained to carry out all the duties they are expected to perform at each specific site. The authority of a person in a management position should be commensurate with the duties and responsibilities of that person.

The duties of boiler house managers may include but are not limited to:

- Ensuring compliance with relevant law (PSSR is specifically noted);
- Risk assessment and risk management;
- Ensuring that manning levels are sufficient;
- Ensuring that plant is maintained correctly;
- Oversight on boiler operators;
- Oversight on sub-contractors;
- Defining and maintaining competencies;
- Management of personnel;
- Record keeping.

## 5.5 Competent Boiler Operator

It is a legal requirement for the user/owner to appoint sufficient trained persons to be responsible for the daily safe operation of the boiler system. These boiler operators must be adequately trained to carry out all the duties they are expected to perform at each specific site. The training should enable the operators to recognise when the limits of their own expertise are reached and when to call for assistance.

The duties of the boiler operator should be determined as a logical outcome of a site specific risk assessment. These may include, but are not limited to:

- Shutdown of a boiler in an emergency or if it is unsafe;
- Implementing the boiler manufacturer's instructions, especially with regard to attendance when starting up from cold, and for all the other aspects of boiler operation, use, maintenance and cleaning etc.
- Carrying out all functional tests of limiters & controls where required, before the boiler is left unattended and at all specified frequencies and in the specified manner. Records of all these tests must be maintained;
- Carrying out the recommended water quality tests, routine water treatment, recording the results and making adjustments where necessary in accordance with established standards and guidance (BG04, BS 2486:1997, BS EN 12953-10 or the manufacturer's instructions). This should be in addition to any testing contracted out to a water treatment specialist; note that the user/owner remains responsible and the water treatment specialist contractor shall have specific and demonstrated expertise in the treatment of water for steam systems;
- Tests on ancillary equipment;
- Checking the burner and associated equipment;
- Responding to alarms and taking appropriate action;
- Identification of maintenance requirements and faults;
- Investigation of abnormal operating conditions;
- Appropriate supervision of contractors;
- Recording the results of checks and tests and boiler house visits.

## 5.6 Personnel monitoring boiler alarms from on-site and off-site locations

All such persons must possess sufficient training and information to take the appropriate action in the event of an alarm condition before calling for the assistance of a boiler operator. In some cases, this may involve the emergency shutdown of the system.

Persons whose function is to monitor alarms shall ensure that the boiler is safe in response to an alarm condition, or shut it down in response to a site emergency from a location deemed appropriate by a risk assessment.

Untrained persons and persons whose only function is to monitor alarms shall not enter a boiler house during an emergency unless there is a system or procedure in place to ensure that access is safe. Only trained persons should enter during an emergency and this entry process should include a dynamic risk assessment to ensure their personal safety.

Untrained persons and persons whose only function is to monitor alarms shall not reset a boiler following a lock out.

## 5.7 Maintenance personnel

All maintenance personnel must possess sufficient knowledge and training to be able to carry out their expected duties. Maintenance personnel must only carry out the maintenance work for which they have been trained and are deemed competent. Suitable training courses and maintenance services for maintenance personnel can usually be provided or recommended by manufacturers of boilers, burners, fittings or control equipment.

## 5.8 Sub-contractors

Sub-contractors are employed on many sites to perform specific specialist tasks or manage the day to day operation of the steam raising plant.

The contracting out party (normally the user/owner) shall ensure that the chosen sub-contractor is competent to perform the required tasks. Suitable and sufficient oversight should be exercised on sub-contractors to ensure that:

- legal requirements and legally imposed duties are met;
- works are undertaken in a safe manner;
- plant is left in a safe condition (whether usable or otherwise) during and after works;
- relevant tests and checks are performed on the plant before it is returned to service.

## 5.9 Manning and supervision of boiler houses

Manning and supervision levels in boiler houses shall be established as a result of a detailed boiler house technical risk assessment, firstly at the design stage and then revised later as the operation of the boiler house evolves. In simple terms, the more automation, measurement and control that is installed the lower the manning requirements might be, BUT this has to be taken in context with other issues such as the location of the boilers, the likelihood of water quality issues, the possibility of contaminated condensate, risks associated with a loss of steam to process and the risks associated with actually getting competent operators to the boilers in adverse weather, as just a few examples.

Furthermore, different operating scenarios may dictate different supervision levels for the same level of automation. A boiler needs to be fully manned whenever it is in a vulnerable state, such as during start up, but it may be assessed as safe for daily visits during production periods and safe to leave for the weekend when the site is out of production but alarms are still monitored locally.

Arrangement drawings in Appendix 3 are **not associated with any particular level of supervision** - they are provided to guide designers and users/owners of steam boiler plant in the direction of possible boiler control and measurement arrangements, and do not represent final solutions for any particular circumstance. A detailed risk assessment is the only way to establish the manning requirements for your plant.

For all levels of manning, shell boilers shall not be warmed through from cold, put on the range, or reset after a lockout without the competent boiler operator present to observe all limiters and alarms, and take the necessary actions.

Boiler plants which incorporate systems which significantly exceed the minimum requirements of the law and include the highest level of automation and monitoring may in certain circumstances still need to be fully manned, and this may be for reasons of steam security to process or other considerations.

Note the definitions used in this section and elsewhere:

**Competent Boiler Operator** - Someone appointed by their employer who has attended a training course with assessment, is familiar with the boiler system on site, and has sufficient knowledge & experience to operate the boiler and system safely.

**Suitably Trained and Instructed Person** – Someone who has been trained to respond to specific boiler house alarms by taking agreed actions which include contacting the duty Competent Boiler Operator.

**Check the boiler** - carry out all documented tests and inspections relating to the boiler and ancillary plant according to local procedures, recording all necessary readings and actions, and making reports of actions and interventions as appropriate.

### Local control and alarms

Where the risk assessment determines that the boilers cannot be left alone, a competent boiler operator shall be in the immediate vicinity of the boilers at all times whilst the boilers are operating. They shall be within earshot and sight of alarms at all times, and able to attend the boilers immediately.

This type of supervision is required when the boiler controls are extremely basic or the boiler is in a vulnerable state, e.g. on start-up or after an unexpected alarm. It is also commonly used when firing solid fuels or unusual liquid fuels, or if there is an unacceptably high risk with the location of the boilers.

## **Fail safe and alarm**

If the boilers can, and actually do, automatically shut down safely as a result of any malfunction or incident, a competent boiler operator shall be on site at all times whilst the boilers are operating; they shall be able to attend the boiler house within 5 minutes.

The operator shall be able to hear or see if the boiler is in alarm at all times. Electronic call devices may be used if accepted by risk assessment.

The boiler operator may have other duties at the site, but they will be present for warming through, starting and stopping the boiler, and shall have specific boiler operational duties such as testing alarms and water quality tests. The boilers shall be their first priority.

## **Automatic shut down on limiters with alarms**

Where the boilers automatically shut down safely as a result of any limiting device activating (low water for instance), or a malfunction or an incident, a competent boiler operator shall attend the boilers at least on a daily basis – they might not be based on site. Speed of response to alarms might be a critical part of the risk assessment. Auto TDS and bottom blowdown shall be installed on any boiler left unattended for 24hrs.

However, a trained person on site shall be able to respond to an alarm in the absence of a competent boiler operator to ensure that the boilers shut down, and be able to summon a competent boiler operator. The trained person may need do no more than respond to an alarm (which can be as simple as "If this red light comes on press this red button and contact the duty boiler operator") but they shall be on site at all times to carry out that action if it is required.

## **Remotely monitored fail safe with alarms**

If the boilers are monitored from a remote monitoring location all the time they are operating then a competent boiler operator shall attend the boilers at least on a daily basis or once in every 24 hours; they might not be based on site. Also, a suitably trained and instructed person at the remote monitoring location shall have the ability to respond to an alarm and summon a competent boiler operator.

The boilers must have advanced controls and monitoring, such as high integrity water level probes, flame detection units and pressure control and limiter. The boilers shall automatically shut down safely as a result of a limiting device activating (low water for instance), or a malfunction or an incident. Auto TDS and bottom blowdown shall be installed on any boiler left unattended for 24hrs.

All the main boiler operational data and alarms shall be visible or audible at the remote monitor at all times. This could be a manned control room, either on site or off site, or a contracted monitoring centre where the suitably trained and instructed person has the ability to confirm the boiler has shut down and can summon a competent boiler operator. In the event that the system monitoring the boiler status fails or loses its capability to communicate, the system shall sound an alarm.

This level of supervision will typically suit sites with multiple boiler houses where operations are centrally monitored, for example, or energy management contractors who operate many sites from one central location. Speed of response to alarms might be a critical part of the risk assessment.

## Remotely continuously monitored on limiters

Where automation on the boilers is such that the boilers self-monitor all operational parameters and have the proven ability to shut themselves down safely in the event of any limiting device activating or a malfunction or an incident, a competent boiler operator shall attend the boilers at least every 72 hours - they might not be based on site. Speed of response to alarms might be a critical part of the risk assessment.

The boilers must have advanced controls and monitoring, such as high integrity water level probes, flame detection units and pressure control and limiters. The boiler water, feed water and condensate return chemistry must be checked automatically at a periodicity identified through risk assessment but at least every 24 hours. Continuous monitoring of the water treatment plant, TDS levels and hot well shall be provided, along with any other checks and alarm systems identified in the risk assessment. Auto TDS and bottom blowdown shall be installed on any boiler left unattended for 24hrs. All of the above must send out an alarm condition if they go out of set parameters, and shut the boiler down safely.

The boiler must be continuously monitored all the time it is operating from an external monitoring location. This could be a manned control room, either on site or off site, or a contracted monitoring centre where the suitably trained and instructed person has the ability to confirm the boiler has shut down and can summon a competent boiler operator. In the event that the system monitoring the boiler status fails or loses its capability to communicate, the boiler shall automatically shut down and sound an alarm.

It is worth taking note that this level of automation and remote supervision is extremely rare, normally due to the boiler house technical risk assessment identifying unforeseen risks and also the high cost of the installation and maintenance of the monitoring equipment.

## 6 TRAINING

Employers must ensure that all personnel possess sufficient knowledge of the boiler systems on which they work to perform their duties properly. Every employer shall ensure that any of his employees who supervises or manages the use of work equipment has received adequate training for purposes of health and safety (PUWER Reg 9).

Any training shall form part of a structured scheme taking into account the particular types of boiler on site and the full range of maintenance tasks required for safe operation of the boiler. All training (including that for boiler systems) should be a structured on-going process which is updated to keep pace with developing technology, equipment and legislation. The level of competence required (and the corresponding training requirements) must be reviewed when a system is modified, e.g. increased automation or remote supervision. The training shall be delivered by personnel possessing the appropriate practical experience, assessment skills, and knowledge of the working environment.

The employer must ensure that all managers and operators and other relevant personnel are regularly assessed through work audits. Training must also be reassessed periodically. All training shall be validated by assessment (written and/or oral) and the results of the assessment recorded.

The Boiler Operation Accreditation Scheme (BOAS) is recognised by the Health and Safety Executive, the UK insurance industry, the Safety Assessment Federation (SAFed) and industry members through the Combustion Engineering Association. Training providers accredited under the Boiler Operation Accreditation Scheme (BOAS) are accredited to the industry standards.

### 6.1 Training courses

There are a number of courses available at various levels. It is recommended that operators and managers achieve the national industry standards for:

- Certified Industrial Boiler Operator (CertIBO) for operators; or
- Diploma in Boiler Plant Operation Management (DipBOM) for managers.

These qualifications form part of the Boiler Operation Accreditation Scheme (BOAS) which covers various types of boiler plant including shell boilers.

The level of training for operatives and managers should be tailored to the equipment an individual is expected to operate and the duties that are expected to be performed while operating that equipment, either normally or under exceptional circumstances.

Generic boiler system training courses can be used to provide basic information at varying levels. All training courses should involve site-specific elements. Courses should include the following topics:

- Boiler operation including start-up and shut-down;
- Boiler & burner controls and failure modes, taking account of fuels used;
- Feed water/boiler water analysis;
- Condensate drainage and water-hammer;
- Actions to be taken in an emergency, and the consequences of inappropriate action;
- Responsibilities of all parties involved and legal aspects;
- Site specific training plus documented written and oral examination on completion of the course.



For shell boiler systems operators and managers, Category 2 BOAS courses cover the following in more detail:

- Basic heat & heat transfer concepts
- Draught & combustion
- Feed water & boiler water analysis
- Control & instrumentation
- Safety & legal requirements
- Energy efficiency
- Environment
- Boilers & auxiliaries
- Operation
- Fuel concepts

BOAS courses cover these basic requirements for boiler operators and managers in general terms, but further training for specific activities is highly recommended. In particular, boiler house operators and managers should be encouraged to undertake enhanced training in steam boiler water testing (in accordance with BG04), industrial gas operations (I-GAS), manufacturer specific training for burners and combustion systems, and bespoke training for the operation and daily maintenance of any other plant items provided in their boiler house.

## 6.2 Training records

Employers must ensure that all relevant training and assessment records are maintained and kept securely, including details of content and results of assessments. Appropriate audit records must be maintained and kept securely. Such evidence of training may be required to be viewed by enforcing authorities.

## 7 DESIGN AND INSTALLATION

All new and substantially modified steam raising boilers must be designed to satisfy all relevant requirements of the Pressure Equipment (Safety) Regulations (PER).

When repairs or modifications, including changes to control systems or commissioning of a new system are undertaken, the risk assessments must be reviewed with a view to eliminating the risks or reducing them to a level as low as reasonably practicable (ALARP).

### 7.1 Design considerations

Many trades and professions are involved in the design, construction, operation and maintenance of a boiler system, so it is essential that all equipment, instrumentation and controls are designed and installed by suitably qualified and experienced personnel in accordance with the manufacturers' instructions.

The design shall be based on the results of a risk assessment and relevant information from the appropriate design standards which provide further detail on the construction of shell boilers and their equipment. Boiler system designs shall address the following safety issues as a minimum:

- Boiler house ventilation - ensure adequate air supply for combustion. Designs shall comply with IGEM UP/10, IGEM UP/16 and BS 6644 as appropriate;
- The source of the boiler feed water, its effective treatment, and means for efficient monitoring of the water treatment plant, all in accordance with BG04, BS 2486:1997, BS EN 12953-10 or the manufacturer's instructions;
- Electrical installation - designs to comply with BS 7671 IET Wiring Regulations. Note: Consideration should be given to the operating environment, ensuring that cable type, size, routing and connections will prevent erroneous operation & maintain the required integrity of the control system;
- Boilers shall fail-safe, i.e. ensure boilers enter a safe mode under automatic control without requiring manual intervention. They shall also have a control integrity appropriate to their mode of operation;
- Critical alarms relating to plant safety shall default to lock-out and require manual reset as defined by BS EN 12953-6;
- Interruption of the electrical supply to water level and firing control equipment shall cut off the boiler automatically. Restart shall only be possible if the normal requirements for start-up are met and the boiler system has been designed to do so.

Other considerations in boiler design include:

- Appropriate types of controls and safety-related systems;
- Site manning levels & competency;
- Testing and maintenance requirements;
- Normal, extreme and transient conditions including safe start-up and shut-down and management of boiler blowdown (see BG03);
- Emergency procedures;
- Access for operation and maintenance;
- Relevant aspects of the Construction Design and Management Regulations (CDM).

For guidance, three typical arrangements of steam boiler controls are outlined in the appendices. They are intended to be used in conjunction with the findings of the risk assessment, and represent suggested typical arrangements of certain steam boiler installations – they should not be used as design solutions or procurement specifications for new plant.

## 7.2 Control systems

Safe and efficient operation depends on the boiler remaining within its safe parameters during operation. A wide range of additional equipment that can be fitted to the boilers is available to help ensure this.

This equipment can have a monitoring role or a safety function where it acts in a predetermined manner to prevent a dangerous situation. For example, on an older installation, the “first low” water level alarm may prevent burner operation when the water level is low, but allows an automatic restart and resumption of operation once the water level has risen to a safe level. On the other hand, should the water level continue to fall, the “second low” water level alarm shuts the burner down completely and does not allow an automatic re-start. The burner may only be re-started manually once the cause of the low level event is established, the second low water level alarm is cleared, and the water level restored.

Control equipment includes the various level sensors, limiters, control devices, relief devices and gauges as well as the communication and alarm systems. The level of control and monitoring will depend on a variety of factors. In general, boilers with automatic control and remote monitoring systems will require more monitoring and control equipment than a locally manned boiler system.

New safety-related systems shall be designed, documented and applied according to the requirements of BS EN 61508 so that safety functions are determined, i.e. the Safety Integrity Level (SIL) of each safety function is specified and the measures used to achieve the specified SIL for each safety function are described. BS EN 50156, *Electrical Equipment for Furnaces and Ancillary Equipment* provides information on the application design and installation of electrical equipment.

Every employer shall ensure that, where appropriate, work equipment is provided with one or more readily accessible emergency stop control device (PUWER Reg 16).

### 7.2.1 Level sensing devices

These can be mounted through the boiler shell or in external chambers providing that the system has proven reliability and is inherently fail-safe. Detailed information can be found in BS EN 12953 Part 9, which specifies the following:

- External chambers must have, as a minimum, 20 mm diameter boiler shell connections at the steam and water level;
- Protection tubes (where fitted) must be designed:
  - With adequate venting to water and steam space;
  - To prevent steam bubbles causing undue disturbance to the water level;
  - To prevent sludge build-up;
  - With a minimum clearance of 14 mm from the probe;
- The two low water level limiters shall be mechanically and electrically independent so as to avoid “common cause” failures. Note that while the limiters should be independent of each other they do not have to be independent of other controls. i.e. a controlling probe can also act as a limiter provided it meets all of the other requirements of EN 12953-9.

Many existing boilers which were not designed and constructed to BS EN12953 will have a first low level cut-out and alarm (auto reset) and a low level limiter (lock-out). Risk assessment may demonstrate that this is not satisfactory for unmanned (remotely operated) boiler systems.

### 7.2.2 Combustion control devices

The system shall incorporate the following (as applicable):

- Ignition flame and main flame detection and safety systems;
- Forced draught and induced draught fan proving systems;
- Air and flue damper position proving systems;
- Flame detectors. High-integrity devices are required on all systems where the combustion system does not progress through a restart at least once per day;
- Systems to monitor the correct ratios of fuel and air;
- Interlocks where simultaneous fuel combustion is not permitted.

### 7.2.3 Pressure and temperature devices

Heat input must be controlled automatically (refer to BS EN 12953 Part 6) as follows:

- Steam boilers to be controlled by pressure controls;
- Limiting devices must be fitted to prevent excessive pressure or temperature. For new boilers they must be in accordance with BS EN 12953 Part 9.

Users/owners shall ensure that an adequate test regime for all pressure and temperature limiters is incorporated into the operating procedures for the boilers.

### 7.2.4 Water treatment plant

The system shall incorporate the following (as applicable for the manning level):

- Means for treating incoming water, such as base exchange or reverse osmosis;
- Measurement and control devices to confirm that water flow is maintained, softened water is provided within the correct parameters, brine tanks are kept full of salt, and backwash is routinely and correctly carried out;
- Devices to record hotwell temperatures and levels, and alarm on deviation;
- Devices for monitoring condensate quality and potential contamination;
- Means for safely collecting boiler water samples from appropriate locations (such as from inside the boiler, from the hotwell, in the condensate return line, and from the softener outlet);
- Means for delivering water treatment chemicals at appropriate points in the system with measurement and control devices to alarm if chemical dosing is low or out of specification, chemical stocks are low, or chemical dosing plant has failed (dosing pump faults, leaks, etc.);
- Equipment for on-site measurement and testing of boiler water parameters.

Increasing time between boiler house visits will increase the quantity and quality of the feed water and condensate monitoring and alarm equipment that is required.

### 7.2.5 Blowdown

*Blowdown Systems, Guidance for Industrial Steam Boilers* (Ref: BG03) is a guidance document intended to provide advice to designers, specifiers, manufacturers, installers and those responsible for the management and operation of steam plant as well as Competent Persons (CP). It is applicable to both new and existing installations of steam boilers and addresses the following issues:

- The safe discharge of blowdown from boilers;
- The safe use and operation of blowdown vessels;
- The safe use and operation of blowdown pits;
- Proper maintenance and inspection of blowdown vessels and pits including requirements for regular inspection.

Advice was previously provided by *Health and Safety Executive Guidance Note PM60 Steam boiler blowdown systems 2nd edition 1998* which has been withdrawn. This new, comprehensive guide deals with all aspects of steam boiler blowdown for industrial steam boilers and why it is necessary to carry out the function of “blowing down” the boiler.

It is aimed at the User/Owner, Engineer, Manager and Operator of the boiler plant to help them understand all aspects that affect the boilers and why blowing down is necessary, both from a practical operational performance view and for the legal requirements.

It covers who is responsible for the safe and efficient operation of steam boiler plant, and who is responsible for managing the safe operation of this type of equipment. Ultimately the responsibility lies with the user/owner as defined by the PSSR.

Where a high TDS alarm is fitted to the boiler system this shall be through a shell mounted probe and not a probe in the blowdown line.

### 7.2.6 Chimneys and flues

The safe handling of the products of combustion from steam boilers must be carefully considered. Poor combustion, and poorly constructed chimneys and flues, can give rise to life threatening accumulations of CO and other pollutants, and the emissions to atmosphere from combustion processes must be managed in accordance with environmental legislation such as the Clean Air Act and the Medium Combustion Plant Directive.

All new steam boiler installations will be notifiable under local planning requirements and larger installations (>1 MWth) will be subject to environmental permitting regulations and require a permit to operate. Chimneys will need to be designed to cope with the expected products of combustion under normal and abnormal operating conditions.

Structural requirements may require the advice of specialists in supporting the loads, providing safe access to work on the chimneys, and providing access platforms for emissions monitoring activities.

Where multiple fuels can be burned in a single furnace or multiple flues enter a single chimney there may be a need for interlocked dampers and interlocked fuel supplies to provide for safe operation under all possible combinations of firing. These should be rigorously tested at appropriate intervals.

### 7.3 Communications and alarms

The number and type of alarms will depend on a number of variables, and a review of the design and risk assessments must be undertaken to validate this decision. Boiler systems shall be designed such that boilers will always remain in a safe condition and will shut themselves down upon critical alarm, without manual intervention.

A lock-out condition requires that the boiler be attended and can only be reset locally. Some typical alarms are indicated in the three arrangements in Appendix 3.

Risk assessment is likely to indicate that there is benefit in also relaying alarms and providing an emergency shut-down facility at a remote location e.g. for boilers that are left unattended for a defined period of time.

Where the risk assessment shows that the existing alarms are inadequate for the proposed operation, new alarms will be required in order that boiler operators can take appropriate action. The following should be considered:

- The response time for personnel to investigate and rectify alarm conditions shall be considered as part of the design of the control system; where a competent boiler operator is unable to attend the boiler within a reasonable time, a remote shut-down and lockout facility shall be provided;
- Alarms shall be clearly audible and visible at a permanently manned location where persons who are trained to take the appropriate action can hear or see them;
- It shall be possible to ascertain the current status of the boiler from the remote location; this may be as simple as a green light to indicate a no-fault condition or as complex as full boiler telemetry. The level of information required at the remote location shall reflect the level of knowledge of individuals at that remote location; e.g. it is unlikely to be appropriate to provide full boiler telemetry in a gate-house or reception area while more detailed information could be of use to those in, say, an engineer's office;
- The integrity and testing of communication links between the boiler house and remote locations, and the action to be taken by the automated system on the loss of that communication shall be considered as part of the design of the control system. An "auto-dialler" is not considered a robust means of monitoring a boiler unless it is capable of checking the integrity of the communications system, or taking action in the event of a loss of communication, or incorporates a means of remotely determining the boiler status and remotely shutting it down.

### 7.4 Gas detection, fire detection and automatic fuel shut-off systems

Automatic fire detection and fuel shut-off is mandatory for all oil-fired plant. Burners shall include automatic shut-off valves on all fuel trains, and the control system shall close these valves when a fuel is not in use, and in the event of a fault condition. Dual or multi fuel systems shall include interlocks to prevent simultaneous use if the burner or boiler is not designed for this.

The need for gas detection and automatic fuel shut-off systems will be determined during the risk assessment; generally speaking, modern boiler houses are regularly attended and well-ventilated spaces, making it unlikely that an accidental release of natural gas of sufficient volume to create a flammable atmosphere will develop. Further information is available from IGEM/UP/16 and IGEM/SR/25.

Gas detection systems will be necessary where forced inlet and/or extract ventilation systems are employed, and where the gas is not sufficiently odourised (e.g. producer gas, or bio-gas) as leaks are likely to go unnoticed by boiler attendants. Similarly it may be necessary to consider CO and H<sub>2</sub>S detection in certain circumstances (e.g. where CHP engine exhaust ducting passes through a boiler house).



The positioning of gas detection systems is of vital importance to ensuring their correct operation; the use of certain gases such as LPG will require careful consideration. Always consult the equipment manufacturer on the correct placement of sensors.

It is recommended that emergency push buttons isolate all fuels and power to the burners using the fuel train safety shut-off valves rather than the fitting of an extra automatic isolating valve. In most control systems it is possible to achieve this remotely so causing the system to go to lockout.

## 7.5 Typical control arrangements

The notes and diagrams in Appendix 3 describe three different typical steam boiler arrangements that might apply to different installations. **They are not definitive drawings of actual installations and must not be interpreted as being compliant with any particular circumstance – a boiler house technical risk assessment will always be required for every boiler house. They should also not be used as procurement instructions for particular boiler installations.**

The levels of attendance and manual testing will depend on the boiler and equipment layouts. They must be considered in conjunction with the findings of the risk assessment and information on the type and level of manning that is intended to be employed. It is more important that the target levels of monitoring and supervision are met rather than having a boiler that matches the example in the diagram. Different operating scenarios may well dictate different levels of supervision for the same level of automation.

The relationship between automation levels and supervision levels is discussed in more detail in Section 5.

## 8 BOILER OPERATION

This section details the requirements for operating the boiler and the various regular checks and procedures that should be carried out on boiler systems.

Employers must ensure that site-specific risk assessments are carried out for each boiler and site to determine:

- the appropriate types of controls and limiters; and
- the particular site manning and supervision levels

to ensure that all risks remain as low as reasonably practicable. Additionally, all shell boilers must be examined and tested by the CP before first use (PSSR Reg 8 (3) c).

### 8.1 Boiler instructions

Boiler instructions shall as a minimum include the following:

- Instructions for the safe operation of steam boiler systems to comply with Regulation 11 of the Pressure Systems Safety Regulations, BS EN 12953-13, and the Pressure Equipment (Safety) Regulations;
- The recommended daily checks required including water treatment plant performance and water quality test results;
- How to warm through boiler systems starting from cold in a controlled manner, and add boilers to the range. Steam boilers shall be manned throughout the warming period and the water levels corrected to allow for expansion. The controls and limiters shall be tested prior to the boiler entering service;
- Information on the safe systems of work, including appropriate standards of isolation that should be implemented for any work on the boiler systems;
- How to protect off-line boilers against corrosion, freezing and sudden thermal shocks;
- The requirement to notify any significant planned change in boiler operating conditions (e.g. reduction in operating pressure or increase in cyclic operation) to the Competent Person prior to making such change, so that the Written Scheme of Examination can be reviewed and, if necessary, amended to reflect the new operating regime.

System re-starts following lock-out must only be made by a suitably experienced and competent boiler operator. Repeated attempts to re-start boiler plants must not be made except as part of a controlled fault identification process.

### 8.2 Recording of controls, limiters and water quality tests

Clear, written instructions describing how and when to carry out routine tests must be kept on-site and be followed by suitably trained and competent boiler operators. Where the boiler controls may be operated off-site, under IEC 61508, these instructions must also be available at the point of control and operated by a person competent to do so.

Routine testing of controls, limiters and water quality is essential to ensure continued safe, reliable and efficient operation. It can help prevent the following dangers:

- Low water level which can expose the furnace or fire tubes and lead to metal overheating & catastrophic boiler failure;
- High water level which can lead to priming of the boiler or carry-over of water, causing water-hammer, damage to valves and pipework as well as sudden steam leaks;

- Scale, excessive sludge deposits and dissolved solids which can quickly build up in a boiler through inadequate blowdown or water treatment regimes. These can cause boiler overheating or water carry-over which can ultimately cause boiler or system failure;
- Faulty combustion controls which can allow the uncontrolled presence of fuel, air and an ignition source, which can result in fires or explosions.

The tests and their frequency shall be based upon:

- Risk assessment of the plant and boiler system;
- Manufacturers' or modifiers' instructions; and
- The controls and manning levels.

A record of such tests shall be maintained to keep an audit trail of the boiler operation. Examples of daily and weekly boiler log sheet contents are given in Appendix 4.

Examples of the type of records and documents that shall be kept and made available for scrutiny include:

- Risk assessment;
- Boiler log book;
- Water treatment test records;
- Combustion analysis records;
- Manufacturer's records and instructions;
- Standard Operating Procedures;
- Emergency Procedures;
- Written Scheme of Examination (WSE);
- Examination reports;
- Record of periodic tests (e.g. Non Destructive Testing (NDT), Hydraulic test);
- Certificates of thorough examination;
- Records of servicing & modifications;
- Maintenance of controls;
- Training records for boiler operators, supervisors and managers;
- Audit reports for boiler operators.

The use of loose-leaf log books is not recommended. Paper logs shall be securely bound, while electronic logs must comply with the requirements of BS 10008:2014: *Evidential weight and legal admissibility of electronic information. Specification.*

Careful consideration of where logbooks are stored is required. While it is useful for information flow between operators to keep the current logbook in the boiler house, there is a risk that the log itself could be lost in the event of a catastrophic incident. For that reason, only the current log should be stored near the boiler. Verified copies and older logbooks should be stored away from the boiler house.

Logbook entries shall be reviewed regularly by a senior manager within the organisation; this may be a useful time to make appropriate copies for remote storage and prompt a review of the procedures and risk assessment.

### 8.3 Water level controls and limiters

The testing regime for water level controls needs to be specific to the type of equipment employed. As a minimum it shall verify the functionality of the water level controls and the associated alarms & limiters. This shall form part of the operating instructions for the boiler system.

The following need to be considered when drawing up instructions:

- The manufacturer's recommended test methods must be carried out as a minimum;
- Any departure from the test frequencies outlined in the arrangements must be supported by the risk assessment;
- Only a competent boiler operator shall carry out the tests;
- At no time during a test shall the water be lowered to the extent that it disappears from the gauge glass;
- If a boiler fails a functional test of the level limiting devices it must be shut down and not brought back into service until such time as the fault has been repaired and the level limiting devices successfully re-tested;
- Test results shall be logged (either electronically or manually) with boiler operator's name, date of test plus any corrective action taken;
- Corrective action following alarms shall always be taken by the competent boiler operator;
- After tests have been completed, ensure that the water level is restored and that all valves are in the correct operating position. The boiler shall not be left until it is operating correctly.

Further details of tests can be found in BS EN 12953 Part 6 Annex C. While the recommended tests are useful for all boilers, the recommended frequency is only appropriate to boilers designed to this standard; risk assessment may demonstrate that some tests should be carried out more frequently. As a minimum, the level limiters shall be proven by test on a weekly basis unless risk assessment demonstrates otherwise. It is unlikely that a lower frequency will be suitable for a boiler that does not possess systems for limiting the water level in accordance with the relevant parts of EN 12953.

It is strongly recommended that gauge glasses on steam boilers are always left open to the boiler during normal operation and the connections to the gauge glasses must be fitted with auto shut off devices for safety of boiler operatives.

### 8.4 Burners and combustion tests

Combustion equipment must comply with the relevant standards (see Appendix 1, References). Maintenance and testing by a qualified person in accordance with manufacturer's instructions is essential to ensure safe and efficient operation.

Manufacturer's instructions for the operation of burners shall contain such information as is required for a boiler operator to use and test the equipment supplied.

Access to burner controls and safety related devices which are to be tested by operators shall not be obstructed by fixed panels or otherwise obscured.

Combustion tests shall also be carried out as appropriate to the type of system in operation. Certain tests, such as visual flame examination or furnace inspection may not be possible or practicable on some designs of boiler, so use of an alternative test such as a CO, CO<sub>2</sub> or O<sub>2</sub> may be appropriate.

Relevant systems must comply with the requirements of the Medium Combustion Plant Directive which places limits on emissions of NO<sub>x</sub>, SO<sub>x</sub> and particulates for all plant with a net thermal input of 1 MW to 50 MW. Some plant may need additional abatement systems in order to meet the Emission Limit Values (ELVs) in which case the abatement system shall be maintained in accordance with the manufacturer's instructions.

All tests shall be recorded on the log sheet and allowable limit data must be readily available. Suitably qualified persons shall investigate any problems and take corrective action.

All manufacturers' tests shall be carried out at recommended frequencies with special attention to:

- Testing flame surveillance equipment operation & recording the results. Prove lock-out and manually reset (but see note below). In a process where the burner is firing continuously, a self-checking photocell shall be used;
- Testing correct operation of forced ventilation and its interlocks and/or ensure natural ventilation is to design standards and is unobstructed;
- On dual fuel installations, it is recommended that the changeover to the stand-by fuel should be tested monthly or as recommended by the burner manufacturer;
- Fuel leak and shut-off checks:
  - Gas - if a significant gas leak is suspected, the gas supply must be shut down immediately and be reported to the Responsible Person. Follow site procedures for any necessary evacuation of personnel and/or activation of audible hazard alarms;
  - Oil - visually inspect pipework, tanks, bunds and supply lines for leakage. Record and immediately report any leaks to maintenance personnel; bund alarms are recommended, particularly where sites are unattended for 72 hours.

**Note:** Some types of high integrity self-checking photocell need professional adjustment and setting, and the manufacturer's recommendations and timescales must be followed.

Should the Emission Limit Values of any environmental permit be exceeded, the user/owner must notify the relevant authorities as soon as possible. If the plant cannot be brought back within limits in a reasonable time, the plant must be taken offline.

Where shell boilers are fitted with new burners to cope with new or additional fuel types, the design, installation and commissioning of the new equipment must be carried out in accordance with all required legislation and guidance. As one example, if changing a heavy oil fired installation to gas firing, a full check of the ventilation requirements will be required and may involve modifications to the boiler house.

## 8.5 Solid fuel (coal and biomass) and alternative sources of heat

Whilst this BG01 guidance is primarily written for oil & gas, much of its contents are relevant for other sources of heat such as biomass and Combined Heat and Power (CHP). In this case, references to burners and fuel systems can be taken to mean the heat source and any associated fuel handling equipment.

Where a heat source cannot be completely removed quickly, for example in the case of a solid fuel fired boiler where fuel is already on the grate or in the case of a CHP where it is unsafe to regularly and repeatedly trip the engine, particular consideration shall be given to:

- The residual heat left in a boiler after a shut-down condition. The plant shall be designed so as to be able to accept this heat;

- The margin between normal working pressure and the safety valve pressure;
- The sinking-time of the boiler, i.e. the time during which the water level will sink from the lowest permissible water level to the highest point of the heated surfaces. This may involve consideration of an automatically closing valve on the steam outlet so as to prevent steam export;
- In some installations, there may be exceptional environmental or operational implications to testing of boiler controls. Testing regimes should be established to ensure that the controls and trips can be proven without tripping the plant except under controlled conditions as justified by a risk assessment.

## 8.6 Feed water and boiler water checks

A water treatment specialist shall undertake regular checks on the water treatment plant and test the feed water, boiler water and condensate quality. If scale is found in boilers, the water treatment system should be checked for correct operation and appropriate corrective action taken immediately.

In addition, a suitably trained and competent employee or the boiler operator shall make the following checks, usually on a daily basis unless suitable automatic testing/monitoring and a supporting risk assessment is in place, in which case a frequency of up to 72 hours might be acceptable:

- That the feed tank level is adequate and there are no contaminants;
- That the feed tank temperature is above the required level for the chemical water treatment dosing levels, specified by the water treatment specialist, for complete oxygen scavenging;
- That any chemical dosing metering device is functioning and there are adequate chemical stocks, both in the tanks and elsewhere on site;
- That in-house routine sample results are within their given parameters provided by the water treatment specialist and/or any recognised standard including BG04, BS 2486:1997, BS EN 12953-10 or the manufacturer's instructions, and take remedial action when and where necessary. In-house routine testing is expected to include at least the following:
  - oxygen scavenger reserve;
  - alkalinity tests;
  - pH;
  - hardness checks of softening plant, feed tank, and boiler;
  - total dissolved solids level within the boiler;
  - appropriate tests of the condensate;
  - that the temperature is above the required level for the chemical water treatment dosing levels, specified by the water treatment specialist, for complete oxygen scavenging;
  - other tests as determined by risk assessment.

For more detailed and specific guidance please see BG04, BS 2486:1997, BS EN 12953-10 or the manufacturer's instructions.

Unless risk assessment demonstrates otherwise the minimum frequency of checks on the feed and boiler water shall be the same as the minimum attendance requirement on the boiler when operating.

Special consideration shall be given to the water treatment requirements for reserve boilers and boilers that are to be left unused for any period.



## 9 MAINTENANCE, REPAIR AND MODIFICATION

### 9.1 Maintenance

Boiler systems must be properly maintained and in good repair, so as to prevent danger, and must take account of manufacturers' instructions in accordance with PSSR Regulation 12 and PUWER Regulation 5.

All maintenance requirements and activities shall be fully documented, including the frequency that maintenance should take place, and maintenance logs must be kept up to date.

### 9.2 Modification & repairs

Prior to any changes or modifications, a risk assessment should be undertaken, and the effects of any modifications, repairs or adjustments to the pressure equipment must be assessed by the CP to determine whether a review of the WSE will be required; this assessment shall take place prior to the work being undertaken. The WSE itself must be reviewed at appropriate intervals (PSSR Reg 8) and it is recommended it is reviewed by the CP at each examination (PSSR ACoP para 117).

Modifications and repairs to pressure systems must comply with PSSR Regulation 13. For significant repairs, the following points must be addressed:

- All alterations to the boiler must be documented and reports or records kept for the life of the boiler;
- Repairs and modifications may in and of themselves only address the symptom. The underlying causal factors which necessitated the repairs or modification must themselves also be addressed;
- Design of the repair must make reference to the original design code and other suitable guidance and achieve an equivalent standard;
- Materials must be suitable and closely match the properties of the original equipment;
- Workmanship must be in accordance with suitable standards including non-destructive examination where applicable;
- Significant repairs or modifications to boiler systems, changes in their operating pressure or changes in cyclic operation must be notified to the CP, the WSE reviewed and the system thoroughly examined prior to coming back into use;
- Any alterations to the original specification of either the boiler system or the boiler house will require consideration and approval by the manufacturer and CP/s before instigating;
- Steam and hot water leaks are dangerous and will waste energy. Identified leaks should be cordoned off and repaired as soon as practicable;
- It may be necessary to carry out modifications or repairs to the burner control and alarm systems. Significant modifications and repairs, where they affect integrity and/or safety of the system, its controls & software, shall be properly considered and the CP shall be kept fully informed of proposals.

Modifications to shell boiler installations may not directly affect the pressure envelope but could be just as significant. For example, MCP users/owners may find that the emissions limits in the MCPD are quite onerous for certain fuels and a change of fuel is proposed.

Designers and installers of new fuel systems and other modifications for shell boilers should ensure that all the necessary measures are taken to meet the legislative and standards requirements for the new equipment, and that comprehensive testing and commissioning of the installation by competent staff is undertaken and recorded.

### 9.3 Responsibility

The importance of adequate maintenance on boiler control and alarm systems cannot be over-emphasised. Responsibility can be divided between those who own and operate the boiler system and those who maintain it. As this can be different in each case it is imperative that the limits of responsibility of each organisation are clearly defined in writing and understood by all parties.

In particular, it is important that the following points are noted:

- The user/owner is responsible for ensuring that all persons working on or with a boiler are trained to do so, including directly employed staff, agency staff, and sub-contractors;
- Boiler operators must ensure that they hand over the boiler to maintenance personnel in a safe condition;
- On completion of maintenance, the checking of all controls, limiters and alarms shall be verified by the boiler operator in the presence of the maintenance personnel before the boiler is placed on line;
- If the maintenance is carried out at the same time as the boiler examination, the controls, limiters and alarms will also be verified by the CP.

## 10 PERIODIC EXAMINATION OF BOILERS

The boiler must be examined in accordance with a WSE which will specify the parts to be examined, the types of examination required and the intervals between them. Depending on the circumstances and degree of expertise available the WSE may be:

- Written and certified by an independent CP; or
- Written and certified by the in-house CP (so long as they are sufficiently independent from the operating function); or
- Written in house by staff with sufficient technical capability, but certified by an independent CP.

The overall examination consists of two parts, firstly with the boiler and its fittings stripped down (“out of service”) and then after it has been returned to operation (“in service” examination). The second part of the examination includes verifying the protective devices are functioning correctly and it must be performed as soon as reasonably practicable after the out of service examination. In any event, pre-checks on the functionality of controls and protective devices should have already been performed by the user/owner as soon as the boiler was returned to operation.

The protective devices that must be checked and/or tested include:

- Pressure gauge;
- Pressure controller;
- Safety relief valve; followed by
  - Pressure limit switch;
- Water level controls/limiters;
- Flame detection device.

The user/owner must ensure that any necessary preparatory work is completed so that the CP can carry out the examination safely. After the examination, the CP will issue a report of examination and all recommendations contained in the report shall be implemented.

Other devices or controls not classed as protective devices in PSSR but should still be checked and tested include:

- Fuel interruption lockout;
- Fuel proving systems;
- Control system power failure;
- Mains power failure;
- Critical alarms (including temperature alarms where fitted);
- TDS alarm.

SAFed Guidance *PSG06: Examination of Pressure Systems in Accordance with Written Scheme of Examination*, and *PSG 07: Guidelines – on the PSSR SI 2000 No. 128 – Working examination requirements in WSE's* provide further information.

## 11 ENERGY AND ENVIRONMENT

### 11.1 Energy management

Energy management of boilers is sensible to minimise operating costs & emissions, to facilitate safe operation and to prolong plant life. Expert advice should be sought before any change in the operating parameters of a boiler which may affect the safety, environmental impact and efficient operation. This may include the following:

- Metering to monitor boiler efficiency;
- Water treatment;
- Combustion analysis and burner adjustment to reduce energy wastage & emissions;
- Energy improvement devices such as economisers, variable speed drives, flue gas dampers, auto TDS control, combustion control etc.;
- Plant scheduling and boiler optimisation to maximise plant efficiency.

The ability to carry out measurement is recommended to demonstrate efficient operation and compliant emissions.

It should be noted that reducing steam pressure may not necessarily improve efficiency.

Certain large organisations (ones that employ at least 250 people, or have an annual turnover in excess of €50 million and a balance sheet in excess of €43 million) will also have to comply with the Energy Saving Opportunities Scheme (ESOS); most public sector bodies are excluded.

### 11.2 Environmental issues

All combustion plant has an impact on the environment through a combination of emissions to air, land and water.

Larger installations will already be covered by a permit issued by the Environment Agency, NRW, SEPA or NIHES. Individual combustion plants with a nett rated thermal input of between 1MW and 50MW will eventually all be covered by a permit issued under the Medium Combustion Plant Directive. This permit will detail the boiler's effect on the environment and list the permit conditions applied to the operator. It is illegal to operate the plant without a permit and outside these conditions, and all new medium combustion plants first fired after 19/12/2018 require a permit.

Smaller plants <1MWth will be regulated by local authorities under the Clean Air Act 1993 with the environmental agencies responsible for emissions to water courses. Local Authorities are principally concerned with the issues of nuisance, such as smoke and dust emissions, which will be regulated. However, operators still have a requirement to ensure that all products of combustion are adequately dispersed.

All solid and liquid waste products produced by a combustion plant must be removed by a licensed waste carrier.

Water discharged to drains must comply with water utility restrictions, and a discharge temperature of greater than 43°C is not allowed under the terms of the Water industry Act 1991.

Legislation and guidance can be downloaded from gov.uk, hse.gov.uk, or the CEA and SAFed web sites.

## APPENDIX 1 - REFERENCES

The following is a list of applicable documents current at the time of preparation of this publication. The following should be noted:

- This is an indicative, not comprehensive list. Users should ensure they are working with the latest information available.
- Free copies of all legislation are available from gov.uk.
- Legislation marked with an asterisk is supported by Approved Codes of Practice and Guidance (ACoP) published by the HSE.
- Legislation marked with a double asterisk is supported by more than a single ACoP.
- The Electricity at Work Regulations (EAW) 1989 are supported by a Memorandum of guidance published by the HSE.

1. Health and Safety at Work etc Act 1974.
2. Management of Health and Safety at Work Regulations (MHSWR) 1998  
SI 1999/3242.
3. Provision and Use of Work Equipment Regulations (PUWER) 1998\*  
SI 1998/2306.
4. Electricity At Work Regulations 1989 - SI 1989/635
5. Confined Spaces Regulations 1997\* - SI 1997/1713.
6. Control of Substances Hazardous to Health Regulations (COSHH) 2002\*  
SI 2002/2667.
7. Dangerous Substances and Explosive Atmosphere Regulations (DSEAR)\*\*  
SI 2002/2776.
8. Control of Noise at Work Regulations 2005 - SI 2005/1643.
9. Construction Design and Management Regulations (CDM) 2015\* - SI 2015/51.
10. Supply of Machinery (Safety) Regulations (SMSR) 2008 - SI 2008/1597.
11. Equipment and Protective Systems Intended for Use in Potentially Explosive Atmospheres Regulations 2016 - SI 2016/1107.
12. Pressure Equipment (Safety) Regulations (PER) SI 2016/1105.
13. Pressure System Safety Regulations (PSSR) 2000\* - SI 2000/128.
14. Work at Height Regulations 2005 SI 2005/735.
15. The Regulatory Reform (Fire Safety) Order 2005 – SI 2005/1541.
16. The Gas Safety (Installation and Use) (Amendment) Regulations (GSIUR) 2018 \*  
SI 1998/2451.
17. The Environmental Permitting (England and Wales)(Amendment) Regulations 2018  
SI2018/110 (MCPD).

18. L5 The Control of Substances Hazardous to Health Regulations 2002. Approved Code of Practice and guidance.
19. L22 Safe use of work equipment Provision and Use of Work Equipment Regulations 1998. Approved Code of Practice and guidance.
20. L101 Safe work in confined spaces. Confined Spaces Regulations 1997. Approved Code of Practice, Regulations and guidance.
21. L108 Controlling noise at work The Control of Noise at Work Regulations 2005 Guidance on Regulations.
22. L122 Safety of pressure systems. Pressure Systems Safety Regulations 2000. Approved Code of Practice.
23. L138 Dangerous Substances and Explosive Atmospheres Regulations 2002. Approved Code of Practice and guidance.
24. L153 Managing health and safety in construction. Construction (Design and Management) Regulations 2015. Guidance on Regulations.
25. HSG253: The safe isolation of plant and equipment.
26. Permit-to-work systems HSE INDG98 ISBN 0 7176 1331 3
27. HSE Pressure Systems website <http://www.hse.gov.uk/pressure-systems/index.htm>
28. Business Innovation and Skills Pressure Equipment Guidance Notes on the UK Regulations - PRODUCT STANDARDS Pressure Equipment – GUIDANCE NOTES ON THE UK REGULATIONS APRIL 2005 URN 05/1074.
29. BG02 Guidance on Safe Operation of Hot Water Boilers. (CEA)
30. BG03 Guidance on Steam Boiler Blowdown Systems. (CEA)
31. BG04 Guidance on Boiler Water Treatment. (CEA)
32. BG08 Guidance on Temporary Steam and Hot Water Boiler Plant (CEA)
33. BS 799: Part 4:1991 Specifications for atomising burners (other than monobloc type) together with associated equipment for single burner & multiburner installations.
34. BS 5410-2:2013 Code of practice for oil firing - Part 2: Installations over 45 kW output capacity for space heating, hot water and steam supply services.
35. BS 5925:1991 Code of practice for Ventilation principles and designing for natural ventilation.
36. BS 6644:2008 Specification for Installation of gas-fired hot water boilers of rated inputs between 70 kW (net) and 1.8 MW (net) (2nd and 3rd family gases).
37. BS 7671 Requirements for electrical installations. IET Wiring Regulations.
38. BS EN 298:1994 Automatic Gas burners Control systems for gas burners and gas burning appliances with or without fans.
39. BS EN 676:1997 Automatic Forced Draught Burners for Gaseous Fuels.

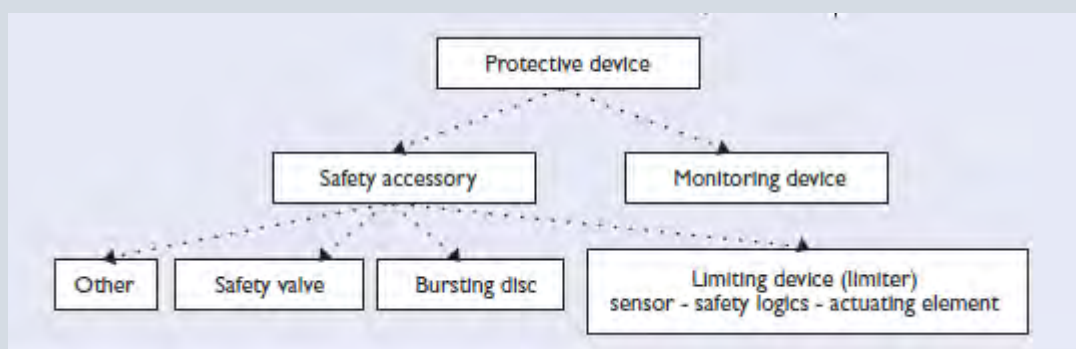


40. BS EN 746:1997 Part 2 safety requirements for Combustion and Fuel Handling Systems.
41. BS EN 12953 Shell Boilers.
42. EN 45510 Guide for procurement of power station equipment Part 3-2 Shell Boilers.
43. IEC 61508 Functional Safety of Electrical/Electronic/Programmable Electronic Safety-Related Systems.
44. Institution of Gas Engineers and Managers Utilisation Procedure IGE/UP/1A - Strength/tightness testing and direct purging (Small I&C) and IGEM/UP/1C - Strength/tightness testing and direct purging (Meters).
45. Institution of Gas Engineers and Managers Utilisation Procedure IGEM/UP/2 - Installation pipework.
46. Institution of Gas Engineers and Managers Utilisation Procedure IGEM/UP/10 Installation of gas appliances in industrial and commercial premises.
47. Institution of Gas Engineers and Managers IGEM/UP/12 Application of burners and controls to gas fired process plant.
48. Institution of Gas Engineers and Managers IGEM/UP/16 Design for Natural Gas installations on industrial and commercial premises with respect to hazardous area classification and preparation of risk assessments.
49. Institution of Gas Engineers and Managers IGEM/SR/25 - Hazardous area classification of Natural Gas installations.

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## APPENDIX 2 - DEFINITIONS

Boiler system	Boilers, ancillaries and all related items including pipework. Additionally may include: fuel supply, water treatment, feedtank, flue, ventilation, blow down equipment, vents, monitoring, limiters and control equipment etc.
Boiler operator	Someone who has attended a training course with assessment, is familiar with the boiler system on-site and has sufficient knowledge & experience to operate the boiler system safely.
Cold boiler or steam system	At atmospheric pressure and a temperature low enough to prevent harm to persons working on the equipment.
Competent Person (CP)	Competent Person as defined in The Pressure Systems Safety Regulations 2000 (PSSR). The individual or organisation that certifies the written scheme of examination and/or carries out the required examinations in accordance with the WSE.
Control	Devices used for maintaining the variable to be controlled (e.g. pressure, temperature, water level) at a specific value (set point).
Controlled blow down	Manually lowering the water level within the boiler in order to perform tests of level controls, having due regard to any discharge constraints. Discharge temperature to drain should not exceed the permissible limit of 43°C.
Cut-out	A monitoring device, which on reaching a fixed value (e.g. pressure, temperature, flow, water level) is used to interrupt the energy supply and does not require manual reset when conditions return to normal.
Diversity	The provision of more than one different means of performing the required function, e.g. other physical principles, or other ways of solving the same problem.
Fail-safe	A limiter or control device is fail-safe if it possesses the capability of defaulting to remain in a safe condition or transferring immediately to another safe condition in the event of certain faults occurring, e.g. loss of power supply.
High-integrity	Refers to a control, limiter or cut-out system where a fault condition does not lead to loss of safety function (fail-safe). Components are high-integrity when they are of fail-safe design so that a single fault in any related part does not lead to loss of safety function. This may be achieved by fault avoidance techniques, self-monitoring, redundancy, diversity or a combination of these methods.
Limiter	A device that, on reaching a fixed value, e.g. pressure, temperature, flow, water level, is used to interrupt and lock-out the energy supply. Note: A limiting device comprises: <ul style="list-style-type: none"> <li>• A measuring or detection function; and</li> <li>• An activation function for correction, or shutdown, or shutdown and lock-out, and which is used to carry out safety related functions as defined in the PED, on its own or as part of a safety (protective) system (e.g. sensors, limiters). If this is achieved by multi-channel systems, then all items or limiters for safety purposes are included within the safety (protective) system.</li> <li>• Protective devices and safety accessories according to Directive 97/23/EC (PED/PER) and (from PSSR) devices designed to protect the pressure system against system failure and devices designed to give warning that system failure might occur, including bursting discs.</li> </ul>



Lock-out	A safety shut-down condition of the limiter, such that a restart can only be accomplished by a manual reset of the limiter or by a manual reset of the safety logic and by no other means. This will be achieved by a competent operator taking account of the physical situation.
Maintenance personnel	Suitably trained persons who are responsible for undertaking maintenance on the plant.
Manned	A boiler operator is on-site during hours of boiler operation.
MAP	Maximum allowable pressure
Off-site monitoring	An off-site location with direct links to the boiler controls and alarms, where monitoring takes place. A competent boiler operator attends site to carry out checks and is available to attend site at all other times.
On-site	Physical presence on-site, not necessarily in the boiler house.
Owner	'Owner' in relation to a pressure system, means the employer or self-employed person that owns the pressure system or: if he does not have a place of business in Great Britain, his agent or: if there is no such agent; the user (Regulation 2, PSSR).
Redundancy	The provision of more than one device or system which, in the event of a fault, will still provide the necessary facilities.
Self-monitoring	Regular and automatic determination that all chosen components of a safety system are capable of functioning as required.
Shell boiler	In a shell boiler, hot gases pass through the furnace and tube banks, the heat from the hot gases transfer via convection through the tubes and conduction into the water within the boiler shell. Also known as fire tube boiler, shell and tube boiler, package steam boiler, smoke tube boiler.
SOL	Safe operating limit.
Steam generator	Steam is made in a coiled tube surrounded by products of combustion. No perceptible water level in the tube.
User	The user of a pressure system - the employer or self-employed person who has control of the operation of the pressure system
Water-hammer	Dynamic shock loading resulting from the accumulation of condensate in steam pipework.
WSE	Written Scheme of Examination.

## APPENDIX 3 – DIAGRAMS OF TYPICAL BOILER ARRANGEMENTS

Arrangement drawings in this Appendix are not associated with any particular level of supervision - they are provided to guide designers and users/owners of steam boiler plant in the direction of possible boiler control and measurement arrangements, and do not represent final solutions for any particular circumstance. **A detailed risk assessment is the only way to establish the manning requirements for your plant.**

Note the definitions used in this Appendix and elsewhere:

**Competent Boiler operator** - Someone appointed by their employer who has attended a training course with assessment, is familiar with the boiler and system on site and has sufficient knowledge & experience to operate the boiler system safely.

**Suitably Trained and Instructed Person** – Someone who has been trained to respond to specific boiler house alarms by taking agreed actions which include contacting the duty Competent Boiler Operator.

**Check the boiler** - carry out all documented tests and inspections relating to the boiler and ancillary plant according to local procedures, recording all necessary readings and actions, and making reports of actions and interventions as appropriate.

### Typical Arrangement 1

This shows the minimum equipment required for the lowest levels of automation. This level does not meet the requirements of boiler standard BS EN 12953.

With typical Arrangement 1 the following factors need to be considered:

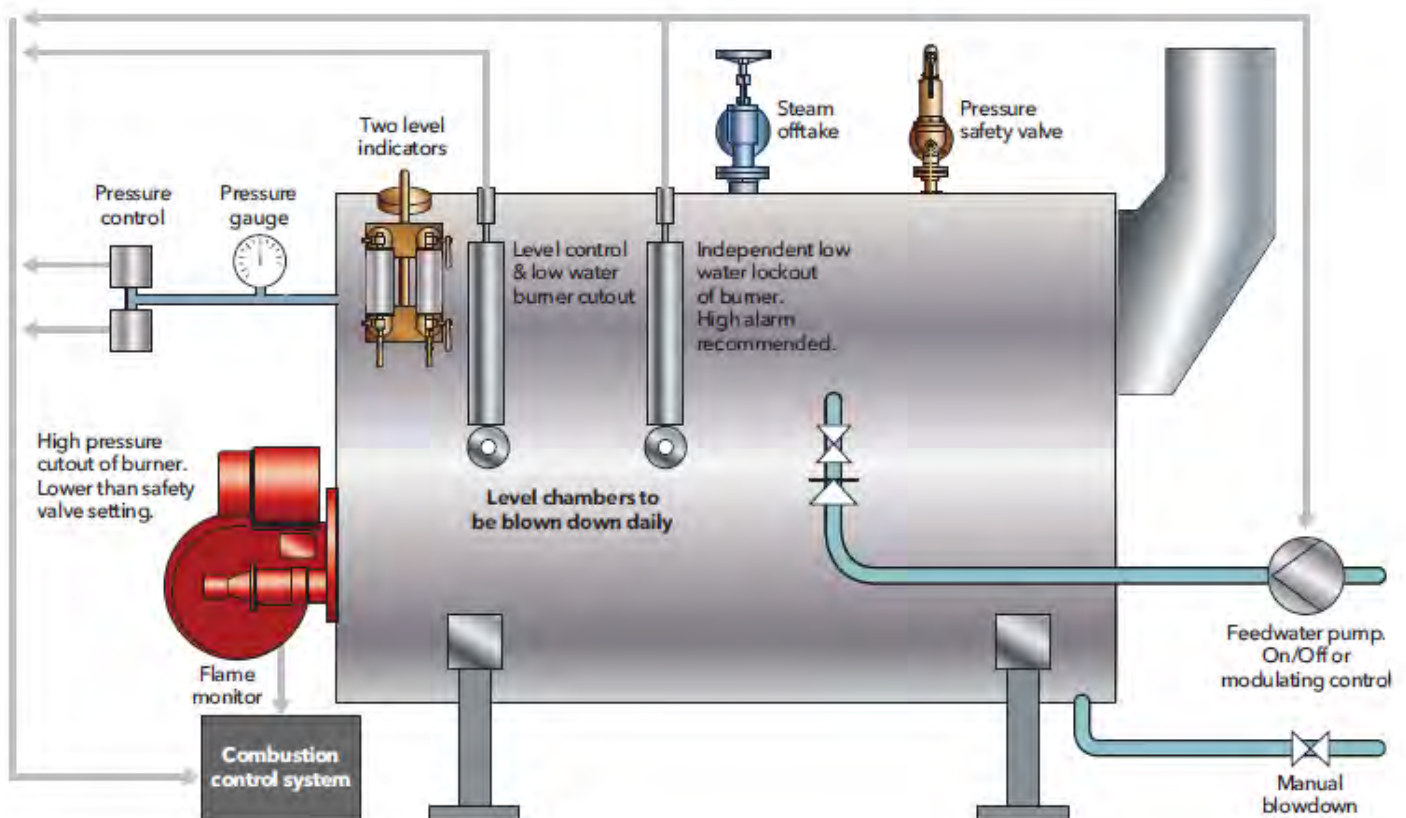
- **Attendance:** A competent boiler operator shall be on-site at all times that the boiler is operating and be able to respond immediately to an audible and/or visual alarm condition.
- **Equipment Integrity:** All control equipment shall be fail-safe.
- **Boiler house fire protection:** Fire detection should be provided. For oil fired installations, automatic fuel shut-off must be provided.
- **Minimum frequency of routine testing:**
  - Low water level devices **in external chambers:**
    - Daily checks: External chambers shall be manually blown down at least once per shift (or daily for continuous operation) and the low water cut-out and lock-out tested;
    - Weekly Checks: In addition, the low water level cut-out and lock-out shall be tested by lowering the boiler water level by evaporation and controlled blow down. Discharge temperature to drain must not exceed permissible limits.
  - Low water level devices **in internal protection tubes** in the boiler:
    - Daily Checks: The low water cut-out and lock-out shall be tested at least once per shift (or daily for continuous operation) by lowering the boiler water level or by an integrated test device, or at the beginning of each shift if a shift pattern is used;
    - Weekly Checks: In addition, the low water level cut-out and lock-out shall be tested by lowering the boiler water level by evaporation and controlled blow down. Discharge temperature to drain must not exceed permissible limits.

- **Level Indicators (gauge glasses):** Manually blown down at least once per shift (or once per day for continuous operation).
- **Boiler and feed water:** Monitoring of the water treatment plant parameters, hotwell temperature and water sample test results.

It is strongly recommended that gauge glasses on steam boilers are always left open to the boiler during normal operation and the connections to the gauge glasses shall be fitted with auto shut off devices for safety of boiler operatives.

The levels of attendance and manual testing will depend on the boiler equipment layout. They should be considered in conjunction with the findings of the risk assessment and information on the type and level of manning that is intended to be employed. It is more important that the target levels of monitoring and supervision are met rather than having a boiler that matches the example in the diagram.

Typical equipment for the boiler:





## Typical Arrangement 2

This shows the minimum equipment required for a boiler with critical alarms monitored on-site by a remote panel located in a manned area such as a gatehouse. New installations shall be manufactured to BS EN 12953, which requires additional limiters to be fitted.

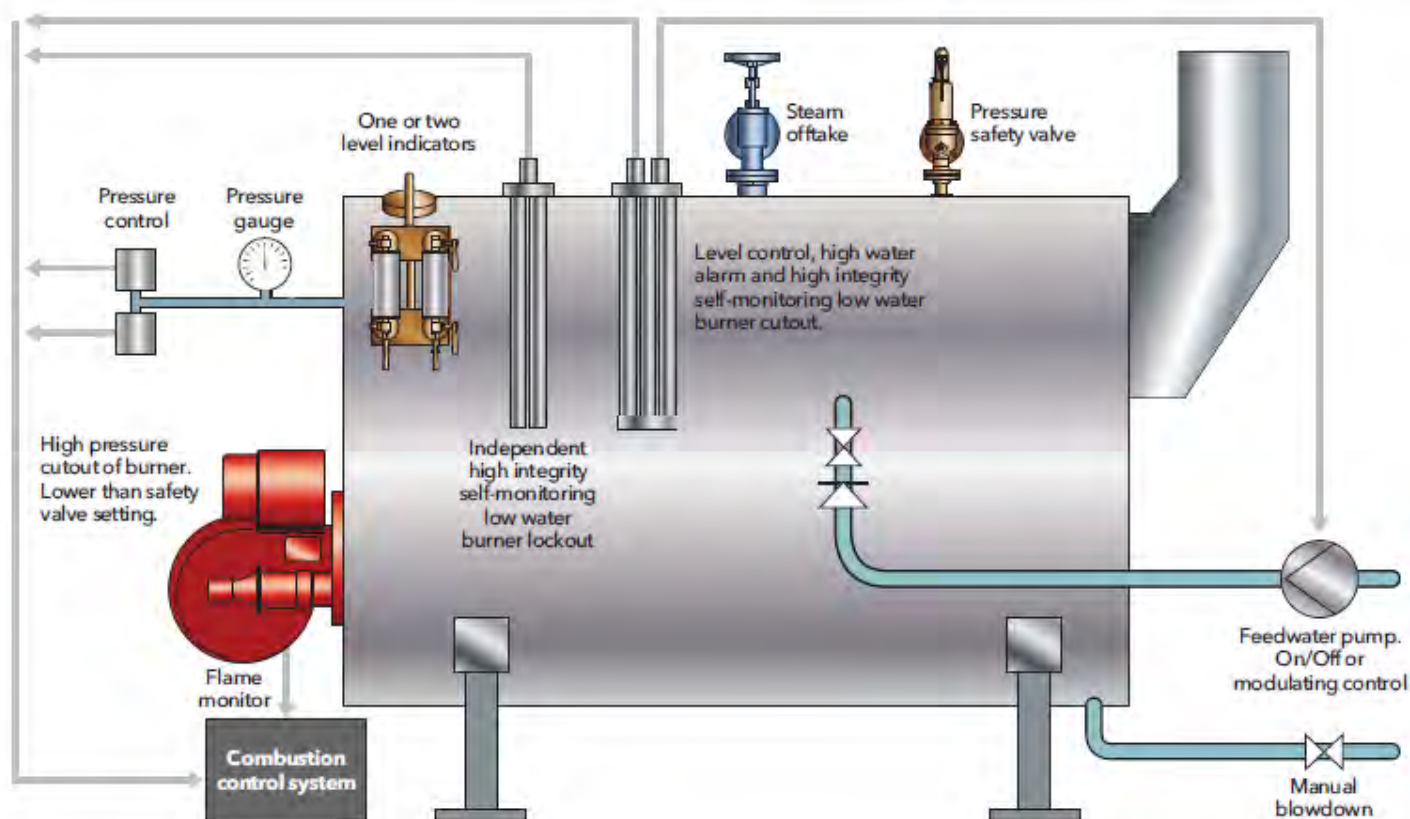
With typical Arrangement 2 the following factors need to be considered:

- **Attendance:** A suitably trained and instructed person must be on-site at all times that the boiler is operating and must be capable of responding to an alarm condition. As a minimum, that person must ensure that the boiler has shut down and notify the boiler operator of the alarm condition.
- The competent boiler operator must check the boiler at least once every day.
- **Equipment Integrity:** Low water level devices and an excess pressure device of the high integrity type must be fitted. All control equipment must be fail-safe. Some types of high integrity self-checking photocell need professional adjustment and setting, and the manufacturer's recommendations and timescales must be followed.
- **Boiler house fire protection:** Fire detection shall be provided. For oil fired installations, automatic fuel shut-off must be provided. For gas firing, gas detection and alarm shall also be considered.
- **Minimum frequency of routine testing:**
  - Low water level devices fitted directly to the boiler:
    - Weekly: The low water level cut-out and lock-out shall be tested by lowering the boiler water level by evaporation and controlled blow down. Discharge temperature to drain must not exceed permissible limits.
  - Low water level devices in external chambers fitted with automatic blow down facilities:
    - Daily: External chambers shall be automatically blown down at intervals typically of at least every six hours.
    - Weekly: In addition to the daily test, the low water level cut-out and lock-out shall be tested by lowering the boiler water level by evaporation and controlled blow down. Discharge temperature to drain must not exceed permissible limits.
- **Level Indicators (gauge glasses):** Manually blown down at least once per day.
- **Boiler and feed water:** Monitoring of the water treatment plant parameters, hotwell temperature and water sample test results.

It is strongly recommended that gauge glasses on steam boilers are always left open to the boiler during normal operation and the connections to the gauge glasses shall be fitted with auto shut off devices for safety of boiler operatives.

The levels of attendance and manual testing will depend on the boiler equipment layout. They should be considered in conjunction with the findings of the risk assessment and information on the type and level of manning that is intended to be employed. It is more important that the target levels of monitoring and supervision are met rather than having a boiler that matches the example in the diagram.

Typical equipment for the boiler:



### Typical Arrangement 3

This shows the minimum equipment requirements for the lowest degree of supervision, where no boiler operators could be on-site for up to 72 hours and with status monitoring and boiler safety shutdown by a remote location/telemetry system.

New installations shall be manufactured to BS EN 12953, which requires additional limiters to be fitted.

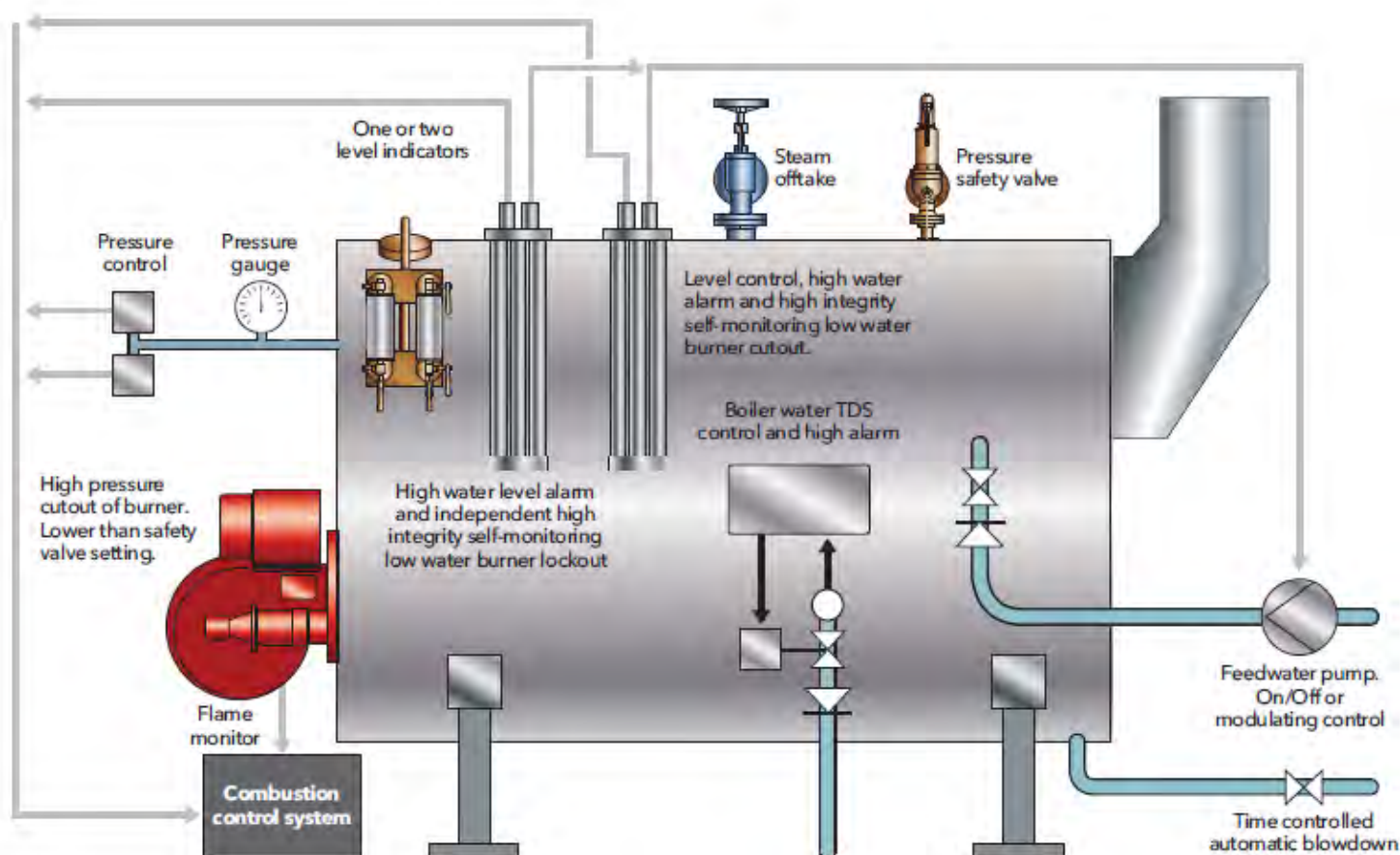
With typical Arrangement 3 the following factors need to be considered:

- **Attendance:** The site must be visited and checked by a boiler operator at least every day. Visits may be extended to 72 hours where additional monitoring is included such as that shown on the diagrams below. In any case, risk assessment may determine a more frequent visit is needed for reasons other than the level of automation. Boiler status is monitored from either an on-site or off-site location.
- **Equipment integrity:** This arrangement is the highest level of automation requiring the greatest degree of confidence in the boiler controls and equipment. Low water level devices and an excess pressure device must be high-integrity. Combustion control system shall be high-integrity. Auto TDS and bottom blowdown shall be installed on any boiler left unattended for 24hrs. All control equipment must be fail-safe. Some types of high integrity self-checking photocell need professional adjustment and setting, and the manufacturer's recommendations and timescales must be followed.
- **Boiler house fire protection:** Fire detection, fire alarm and automatic shut-off of fuel oil systems must be provided. For gas firing, gas detection and alarm shall also be considered.
- **Minimum frequency of routine testing:**
  - Low water level devices fitted directly to the boiler:
    - Weekly: The low water level cut-out and lock-out shall be tested by lowering the boiler water level by evaporation and controlled blow down. Discharge temperature to drain must not exceed permissible limits.
  - Low water level devices in external chambers fitted with automatic blow down facilities:
    - Daily: External chambers shall be automatically blown down at intervals typically at least every six hours. Discharge temperature to drain must not exceed permissible limits.
    - Weekly: In addition, the low water level cut-out and lock-out shall be tested by lowering the boiler water level by evaporation and controlled blow down. Discharge temperature to drain must not exceed permissible limits.
- **Level indicators (gauge glasses):** At least once every three days, manually blown down.
- **Water treatment:**
  - At least every three days, testing and recording of the feed water plant parameters, hotwell temperature and water sample results including pH, turbidity or other measurements of boiler water;
  - Water treatment plant and chemical dosing plant shall have flow monitoring for feedwater and chemical dosing integrity, and level monitoring for salt and chemical stocks;
  - Condensate monitoring and feedwater/dosing plant shall alarm if parameters are not met.

It is strongly recommended that gauge glasses on steam boilers are always left open to the boiler during normal operation and the connections to the gauge glasses shall be fitted with auto shut off devices for safety of boiler operatives.

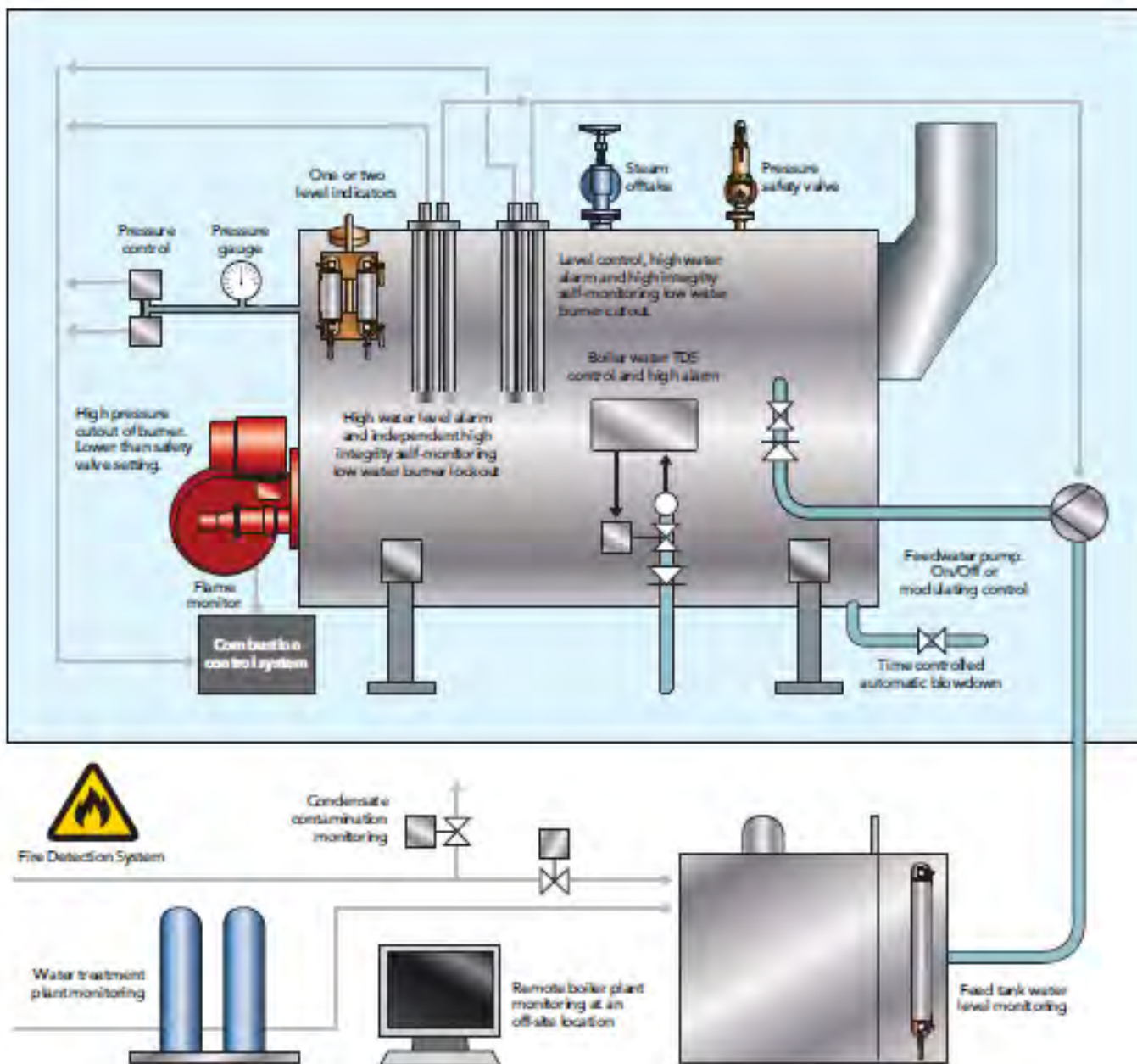
The levels of attendance and manual testing will depend on the boiler equipment layout. They should be considered in conjunction with the findings of the risk assessment and information on the type and level of manning that is intended to be employed. It is more important that the target levels of monitoring and supervision are met rather than having a boiler that matches the example in the diagrams.

Typical equipment for the boiler:



Note – for a TDS alarm to be effective, the probe must be directly mounted on the boiler shell, and not located in the blowdown line.

Typical additional equipment in the boiler house - 72 hours unattended is only possible with the following additional controls:



Where this additional equipment is not provided this decision must be supported by a risk assessment and other control measures may be required.

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## APPENDIX 4 – TYPICAL LOG SHEET EXAMPLES

The boiler logs possess two functions:

- They should be formulated as the logical outcome of a risk assessment and as such the checks contained within constitute a risk assessment checklist.
- They are also a record of the activities that occur within a boiler house and as such all visits, work, actions and interventions which may affect the operation of the boiler should be recorded in as much detail as necessary for safe and efficient operation.

The examples that follow are suggestions for the types of records that need to be kept for typical boiler houses – **every boiler house is different and will need its own log sheet.**

For arrangements 1 and 2, the example log book has a front cover sheet with general information, a daily log (2 sheets) and a weekly log (2 sheets).

For arrangement 3, the suggested list of items to be logged comes from BS EN 12953 part 6.

## Recommended checks and tests schedule for shell boilers to Arrangements 1 or 2.

### Log book front sheet

Boilerhouse log book for the boilers at \_\_\_\_\_

Date started: \_\_\_\_\_

Date closed: \_\_\_\_\_

Site name and address: \_\_\_\_\_

\_\_\_\_\_

### Important notes

- All tests and records shall be completed and recorded by a competent boiler operator.
- Every visit to the boiler house and the name of every visitor shall be recorded.
- Visits by third parties who work on the boilers or associated plant shall be recorded in this log book and include a brief note of the work undertaken and the reference numbers of their job sheets.
- Keep all water treatment checks and records for a minimum of 2 years.
- On re-starting a boiler following maintenance or a breakdown, a full set of tests must be carried out and recorded prior to putting the boiler back on line.
- This log book contains 31 sets of daily check sheets followed by 5 sets of weekly check sheets.
- This log book shall be kept in a safe, secure location and shall be retained for a minimum of 2 years (INDG436).
- All annual inspection reports stay with the boiler for life

## DAY 1 (2, 3, 4 ....)

Print Name:	Date:		Time:	
Boiler	ONE	TWO	THREE	
Status	Online / Offline / Off	Online / Offline / Off	Online / Offline / Off	
Is water showing in LH glass?	Yes / No	Yes / No	Yes / No	
Is water showing in RH glass?	Yes / No	Yes / No	Yes / No	
<b>If either glass indicates no level, shut down, isolate and report</b>				
LH Sight glass blow-down	Pass / Fail	Pass / Fail	Pass / Fail	
RH Sight glass blow-down	Pass / Fail	Pass / Fail	Pass / Fail	
Do both glasses blow down in the same way? (Comment if required)	Yes / No	Yes / No	Yes / No	
Do the glasses show the same level? (Comment if required)	Yes / No	Yes / No	Yes / No	
<b>If neither glass blows down correctly, shut down, isolate and report.</b>				
1 <sup>st</sup> low water electronic function test	Pass / Fail	Pass / Fail	Pass / Fail	
2 <sup>nd</sup> low water electronic function test	Pass / Fail	Pass / Fail	Pass / Fail	
<b>If either of these tests fail, the boiler must be shut down, isolated and the incident reported.</b>				
Boiler pressure reading (gauge)				bar g
Steam main pressure				bar g
Burner firing rate				%
Exhaust temperature A				°C
Ambient temperature B				°C
A – B = C				°C
<b>If there is a significant change at C from previous tests, shut down, isolate and report</b>				
Flame inspection (describe if abnormal)	Normal / Abnormal	Normal / Abnormal	Normal / Abnormal	
<b>If a part of the furnace is glowing orange / yellow, shut down, isolate and report</b>				
Feed pH	8.5-9.5			pH
Boiler water pH level	10.5-12.0			pH
Boiler alkalinity	320 -1,200			ppm
Boiler water sulphites	30-70			ppm
TDS PPM test result	< 3,500			ppm
TDS PPM readout	< 3,500			ppm
TDS recalibrated?	Yes / No	Yes / No	Yes / No	
<b>Duty pump</b>	One - Two	One - Two	One - Two	

Boiler	ONE	TWO	THREE	
Gas meter reading				m <sup>3</sup>
Water meter reading				m <sup>3</sup>
Steam meter reading				kg/h
Hotwell		One	Two	
Temperature				°C
Hotwell level				litre
Salt bin water level		Adequate / Inadequate		
Is salt visible in salt bin?		Yes / No		
Number of bags of salt on site				
Duty softener operating		One	Two	
Water test after the softener	2 max			ppm
Water meter reading at softener				m <sup>3</sup>
Water daily consumption (softener)				m <sup>3</sup>

**Comments / faults / incidents**

**Signature:**

**WEEK 1 (2, 3, 4 ....)**

Date			Time			
Print Name						
Result of evaporation test	Circle as required					
	Boiler One		Boiler Two		Boiler Three	
Gauge glasses blown down	Yes	No	Yes	No	Yes	No
1 <sup>st</sup> low audible alarm	Sounded	Failed	Sounded	Failed	Sounded	Failed
1 <sup>st</sup> low visual alarm	Observed	Failed	Observed	Failed	Observed	Failed
Burner shutdown at 1 <sup>st</sup> low	Yes	Failed	Yes	Failed	Yes	Failed
2 <sup>nd</sup> low audible alarm	Sounded	Failed	Sounded	Failed	Sounded	Failed
2 <sup>nd</sup> low visual alarm	Observed	Failed	Observed	Failed	Observed	Failed
Boiler lock out at 2 <sup>nd</sup> low	Yes	Failed	Yes	Failed	Yes	Failed
Remote alarm (audible) Security Lodge	Sounded	Failed	Sounded	Failed	Sounded	Failed
Remote alarm (visual) Security Lodge	Observed	Failed	Observed	Failed	Observed	Failed
Is water visible in gauge glasses at lockout?	Yes	Failed	Yes	Failed	Yes	Failed
Pump on after 1 <sup>st</sup> low recovered	Yes	Failed	Yes	Failed	Yes	Failed
Burner restart check	Normal	Failed	Normal	Failed	Normal	Failed
Flame Inspection at Low Fire Hold	Normal	Abnormal	Normal	Abnormal	Normal	Abnormal
Pump stop and start	Normal	Abnormal	Normal	Abnormal	Normal	Abnormal
Flame failure device test	Passed	Failed	Passed	Failed	Passed	Failed
Manual blow down performed	Performed	Not performed	Performed	Not performed	Performed	Not performed
Blow down OK	Confirmed	Not confirmed	Confirmed	Not confirmed	Confirmed	Not confirmed
<b>IN THE EVENT OF ANY FAILURE OF EITHER THE EVAPORATION TEST OR THE FLAME FAILURE DEVICE, THE BOILER IS TO BE SHUT DOWN AND ISOLATED. THE FAILURE MUST BE REPORTED AND RECORDED IMMEDIATELY.</b>						

Chemicals	Stock	Usage	
Chemical Level –			litre
Chemical Level –			litre
Chemical Level –			litre
Boilerhouse general condition	Adequate	Inadequate	

Boiler run on oil	ONE	TWO	THREE
Start litres			
Finish litres			
Usage (start litres - finish litres)			
Propane ignition cylinders	Bottle 1	Bottle 2	
	Online / standby / empty	Online / standby / empty	
Oil tank level	Tank 1	Tank 2	
	litres	litres	

**Comments / faults / incidents**

**Signature:**



### Recommended checks and tests schedule for shell boilers to Arrangement 3.

(C) Observation of abnormal noises, smells or other noticeable factors.

(T) Checking and/or testing the functional behaviour of equipment parts, including observation.

Checks and testing	3 days	1 month	3 months	6 months	12 months	Remarks
Safeguards against excessive pressure (safety valves)	C				T	See NOTES 1 and 2 below
Water level indication	T					Compared with limiters and controls
Drain and blow-down devices	T					
Valves	C			T		As per manufacturer's instruction manual
Feed water control	C			T		
Low water protection	C	T				Functional check by lowering the water level to the switching points
Steam pressure and temperature indication	C					Compared with limiters and controls
Pressure limitation	C	T				Functional check by increasing the pressure to the switching points
Temperature limitation	C	T				
Devices for water quality protection	C	T (1)		T (2)		(1) Comparison of the measured values with the reliable samples (2) Performed by a suitably qualified and competent person
Protective systems	C			T (3)		(3) Electrical and mechanical testing performed by a suitably qualified and competent person
Pressure parts (pipes, inspection openings, flanges, gaskets, joints...)		C				
Pressure controller and temperature controller	C			T		
Feed water supply	C		T			
Water quality	T					See BG04
Heat supply	C				T (5)	(5) Performed by a suitably qualified and competent person as per manufacturer's instruction manual but not less than once a year

NOTE 1 Additional function tests and observation can be required either by National Rules, third parties or the manufacturer.

NOTE 2 Deviations of periods or tests are possible with agreement of third parties if safety level will not be reduced.

NOTE 3 Consideration should be given to functional testing of additional devices outside the boiler.  
From BS EN 12953 – 6 2011

### Recommended checks and tests schedule for shell boilers NOT to Arrangement 3.

(C) Observation of abnormal noises, smells or other noticeable factors.

(T) Checking and/or testing the functional behaviour of equipment parts, including observation.

Checks and testing	daily	weekly	1 months	6 months	12 months	Remarks
Safeguards against excessive pressure (safety valves)	C				T	See NOTES 1 and 2 below
Water level indication	T					Compared with limiters and controls
Drain and blow-down devices	T					
Valves	C			T		As per manufacturer's instruction manual
Feed water control	C			T		
Low water protection	C	T				Functional check by lowering the water level to the switching points
Steam pressure and temperature indication	C					Compared with limiters and controls
Pressure limitation	C	T				Functional check by increasing the pressure to the switching points
Temperature limitation	C	T				
Devices for water quality protection	C	T (1)		T (2)		(1) Comparison of the measured values with the reliable samples (2) Performed by a suitably qualified and competent person
Protective systems	C			T (3)		(3) Electrical and mechanical testing performed by a suitably qualified and competent person
Pressure parts (pipes, inspection openings, flanges, gaskets, joints...)		C				
Pressure controller and temperature controller	C			T		
Feed water supply	C		T			
Water quality	T					See BG04
Heat supply	C			T (5)		(5) Performed by a suitably qualified and competent person as per manufacturer's instruction manual but not less than once every 6 months.

NOTE 1 Additional function tests and observation can be required either by National Rules, third parties or the manufacturer.

NOTE 2 Deviations of periods or tests are possible with agreement of third parties if safety level will not be reduced.

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## KEY CHANGES FROM EDITION 1 TO EDITION 2

- Errors corrected and text revised throughout. All sections reviewed and expanded.
- Title changed to 'steam boilers' – hot water boilers all now covered in a new document, BG02 Guidance on safe operation of hot water boilers.
- Scope clarified – does not include steam generators as well as other listed types.
- MHSWR updated – ACoP no longer available.
- PSSR updated: includes note on temporary boilers.
- PUWER guidance expanded.
- CDM regs added.
- DSEAR added.
- Environmental Permitting Regs added.
- New section on Technical Boiler House Risk Assessment.
- New descriptions of Competent Boiler Operators and personnel monitoring boilers.
- New section on sub-contractors.
- New section on manning and supervision levels and detailed descriptions of manning levels.
- Typical arrangement drawings removed to Appendices – not to be used as design drawings or procurement instructions.
- Arrangement 4 removed.
- New section on design and installation of water treatment plant.
- New section on blowdown
- New section on chimneys and flues.
- App 1 References list updated.
- App 2 Definitions list updated
- App 3 Diagrams of typical boiler arrangements revised and updated
- App 4 Log Sheet examples – new examples used.

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#### The Combustion Engineering Association

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#### Guidance on the Safe Operation of Steam Boilers

This document will be formally reviewed periodically, although amendments and revisions may be made more frequently as required.

Users of this document should ensure they are working to the latest edition of this document and the related legislation and guidance.

Ref: BG01 Edition 2 - Published March 2019 - © all rights reserved





## Summarising Skills

A summary can be defined as an overview of a longer piece of text. The key aim of a summary is to condense a piece of text without losing its most fundamental information or ideas. A summary should present a concise representation of main points, ideas, concepts, facts or text in your own words.

The process of summarising requires important reading, thinking and writing skills which are fundamental to success at any course level. This skill allows you to exhibit and present your understanding and interpretation of major concepts and ideas in your topic or subject rather than commit plagiarism by copying passages from books, article, web sites or journals.

<https://studyskillscitycollege.wordpress.com>

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Summarising is an important skill which is often used when researching, gathering or presenting information. Many assignments you produce will draw upon important ideas, writings and research of experts in your field. No doubt, you will include the work of various writers in your own assignments to support claims or provide evidence of your own writing; however, incorporation of such works must be done carefully in order to avoid plagiarism.

A summary should present a concise representation of main points, ideas, concepts, facts or text in your own words.

A summary – or précis – is a shorter version of a longer piece of writing. The summary captures all the most important parts of the original, but expresses them in a [much] shorter space.

Summarizing exercises are usually set to test your understanding of the original, and your ability to re-state its main purpose.

Summarizing is also a useful skill when gathering information or doing research.

The summary should be expressed – as far as possible – in your own words. It's not enough to merely copy out parts of the original.

### **How to summarise your text:**

1. Read through the original text to understand its overall meaning.
2. Capture the main points of the text by highlighting them and ignoring any unnecessary facts, descriptions, opinions or examples which do not affect the core message.
3. Note down important details.
4. Without the original text, re-write your notes in your own words by linking together the key points using full sentences or paragraphs as appropriate.
5. If the original text is very long it may be useful to use headings or sub-headings.
6. It is important to re-read your summary to make sure that you have not lost the overall point of the original information.

[Depending upon the reason for summarising a piece of text, it may be necessary to reference the original source to prevent claims of plagiarism. Indeed, referencing sources is good practice anyway.]



## Skim Reading & Scan Reading

Skimming and scanning are reading techniques that use rapid eye movement and keywords to move quickly through text for slightly different purposes. Skimming is reading rapidly in order to get a general overview of the material. Scanning is reading rapidly in order to find specific facts. While skimming tells you what general information is within a section, scanning helps you locate a particular fact. Skimming is like snorkelling, and scanning is more like pearl diving.

Use skimming in previewing (reading before you read), reviewing (reading after you read), determining the main idea from a long selection you don't wish to read, or when trying to find source material for a research paper.

Use scanning in research to find particular facts, to study fact-heavy topics, and to answer questions requiring factual support.

[www.butte.edu/](http://www.butte.edu/)

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### **Skimming to save time**

Skimming can save you hours of laborious reading. However, it is not always the most appropriate way to read. It is very useful as a preview to a more detailed reading or when reviewing a selection heavy in content. But when you skim, you may miss important points or overlook the finer shadings of meaning, for which rapid reading or perhaps even study reading may be necessary.

Use skimming to overview your textbook chapters or to review for a test. Use skimming to decide if you need to read something at all, for example during the preliminary research for a paper. Skimming can tell you enough about the general idea and tone of the material, as well as its gross similarity or difference from other sources, to know if you need to read it at all.

To skim, prepare yourself to move rapidly through the pages. You will not read every word; you will pay special attention to typographical cues-headings, boldface and italic type, indenting, bulleted and numbered lists. You will be alert for key words and phrases, the names of people and places, dates, nouns, and unfamiliar words. In general follow these steps:

1. Read the table of contents or chapter overview to learn the main divisions of ideas.
2. Glance through the main headings in each chapter just to see a word or two. Read the headings of charts and tables.
3. Read the entire introductory paragraph and then the first and last sentence only of each following paragraph. For each paragraph, read only the first few words of each sentence or to locate the main idea.
4. Stop and quickly read the sentences containing keywords indicated in boldface or italics.
5. When you think you have found something significant, stop to read the entire sentence to make sure. Then go on the same way. Resist the temptation to stop to read details you don't need.
6. Read chapter summaries when provided.

If you cannot complete all the steps above, compromise: read only the chapter overviews and summaries, for example, or the summaries and all the boldfaced keywords. When you skim, you take a calculated risk that you may miss something. For instance, the main ideas of paragraphs are not always found in the first or last sentences (although in many textbooks they are). Ideas you miss you may pick up in a chapter overview or summary.

Good skimmers do not skim everything at the same rate or give equal attention to everything. While skimming is always faster than your normal reading speed, you should slow down in the following situations:

1. When you skim introductory and concluding paragraphs
2. When you skim topic sentences
3. When you find an unfamiliar word
4. When the material is very complicated



### **Scanning for research and study**

Scanning, too, uses keywords and organizational cues. But while the goal of skimming is a bird's-eye view of the material, the goal of scanning is to locate and swoop down on particular facts.

Facts may be buried within long text passages that have relatively little else to do with your topic or claim. Skim this material first to decide if it is likely to contain the facts you need. Don't forget to scan tables of contents, summaries, indexes, headings, and typographical cues. To make sense of lists and tables, skim them first to understand how they are organized: alphabetical, chronological, or most-to-least, for example. If after skimming you decide the material will be useful, go ahead and scan:

1. Know what you're looking for. Decide on a few key words or phrases—search terms, if you will. You will be a flesh-and-blood search engine.
2. Look for only one keyword at a time. If you use multiple keywords, do multiple scans.
3. Let your eyes float rapidly down the page until you find the word or phrase you want.
4. When your eye catches one of your keywords, read the surrounding material carefully.

### **Scanning to answer questions**

If you are scanning for facts to answer a specific question, one step is already done for you: the question itself supplies the keywords. Follow these steps:

1. Read each question completely before starting to scan. Choose your keywords from the question itself.
2. Look for answers to only one question at a time. Scan separately for each question.
3. When you locate a keyword, read the surrounding text carefully to see if it is relevant.
4. Re-read the question to determine if the answer you found answers this question.

Scanning is a technique that requires concentration and can be surprisingly tiring. You may have to practice at not allowing your attention to wander. Choose a time and place that you know works for you and dive in.





## Note Taking Skills

Effective note-taking is an important transferable skill, a skill that can be applied in all aspects of life, socially, at work and during study.

Note-taking is a powerful aid to communication, a way of summarising and retaining the key points from what you've heard and understood.

There are different approaches to note taking, depending on the type of communication you're engaged in. This article covers effective note-taking for verbal exchanges – that is, summarising what has been said, in face-to-face conversations, over the phone and in group situations – like in meetings or when attending a lecture.

[www.skillsyouneed.com](http://www.skillsyouneed.com)

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## **What is Note-Taking?**

Note-taking is, simply, a way of concisely recording important information so that you can recall it later.

Regardless of how good you think your memory is – you will need to take notes in certain situations to remind yourself what was said. It is a mistake to think, when going to a meeting or attending a lecture or some other important talk, that you will remember the details of what has been said – you won't. You may well remember the overall topic of the discussion, even some very specific details, but you won't remember everything.

It is important to recognise that taking notes should not distract you from listening intently to what the speaker is saying. Effective note-taking involves listening whilst jotting down key points that will be important later: in a business meeting this may include action points that you have agreed to attend to; in a lecture this may include new vocabulary or theories that you can investigate further later.

Before you can take effective notes you need to be somewhat organised. It may seem obvious but you need to remember to take some appropriate note-taking equipment with you to meetings, lectures etc. The nature of the 'appropriate' note-taking equipment will depend partly on you and partly on the circumstances. The simplest low-tech way of taking notes is to use a pen (or series of different coloured pens) and a pad of paper. Bring plenty of paper and at least one spare pen or pencil.

Some people prefer to take notes on a laptop, tablet, smartphone or some other device – this is fine as long as you are very comfortable with the technology – so that they can concentrate fully on their notes – not on the actual process of writing them. If you are using some form of computer to take notes it is usually a good idea to turn off any messaging services first – otherwise you are likely to be distracted by new emails, text messages or the like.

When you arrive at the meeting or lecture try to sit so that you can clearly see and hear the main speaker.

## **General Note-Taking Guidelines:**

1. **Before you start taking any notes be clear about why you are attending the talk or meeting.** What are you hoping to learn or gain from it? Think of your notes as a guide to your learning and development after the event. Your notes form part of a working document that you'll return to and add to later.
2. **Think about whether or not a point is noteworthy before you write it down – do not take notes for the sake of taking notes.** Otherwise you'll end up with lots of irrelevant points, which will distract you from the important things. You probably only really need to make notes on things that are new to you.
3. **Do not write down everything that is said, word-for-word, that would be transcribing, which is an altogether different skill.** Concentrate on the key points, remain alert and attentive and listen to what is being said.
4. **Write in your own style and use your own words,** you don't need to worry too much about spelling, grammar, punctuation or neatness as long as you can read your notes later and they make sense to you. Your personal note-writing system will evolve and improve with practice.
5. **Try to use short concise points,** single words or phrases or short sentences, use bullet or numbered lists if necessary. If you are using a pen and paper then it is easy to add linking lines to join ideas and concepts.
6. Write down in full, key information that can't be shortened: names, contact details, dates, URL's, references, book titles, formulas etc.
7. Use abbreviations to help you – just note what they mean!
8. **Use underlining, indentation, circle words or phrases, use highlighter pens** – whatever system works for you to emphasise the most important points and add some structure to your notes.
9. **Use some sort of shorthand system that you will understand later** – develop this system as you become more skilled at note-taking.
10. **Don't panic if you miss something.** You can usually ask the speaker to repeat a point or ask a colleague or peer after the event. Note down that you have missed something to remind you to do this.



Once the event has finished:

1. **As soon as possible, after the event, you should review and, where necessary, rework your notes.** Fill in any gaps, adding content and further research to your notes. If your notes are handwritten you may want to type them into a computer. The more you interact with your notes the more you will remember and ultimately learn.
2. **If possible share and/or compare your notes with a colleague or peer.** Discuss your understandings and fill in any gaps together.

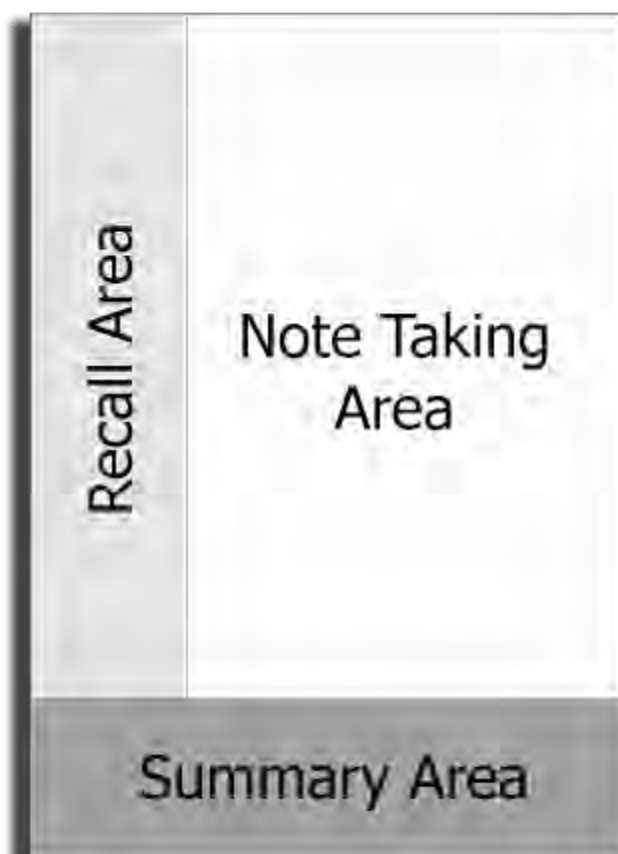
### **The Cornell Method**

The Cornell Method of note-taking is highly effective, see if it works for you.

1. Divide your sheet of paper, as the diagram, so you have a wide left margin (the recall area) and a deep (summary area) at the bottom. Leaving the rest of the sheet for the notes you take while attending the class or meeting.
2. Write notes in the 'note taking area'. After the event fill in any gaps in your notes, try to leave some white space between points. For each major point or idea covered in your notes write a 'cue word' or 'keyword' in the recall area of your sheet.

For example: If your notes were about 'note taking methods' and you had a section describing the Cornell Method then you would probably write 'Cornell' or 'Cornell Method' in your recall area aligned with the specific notes.

3. Use the summary area to write a brief summary of what your sheet contains – it may be useful to colour code this area. The summary will help you to find relevant notes later when you need to review them – this is especially useful for students when revising for exams or writing an assignment.



The Cornell Method of note-taking can be used as a powerful aid to recalling information.

Test your memory and knowledge by putting another sheet of paper over the 'note taking area' so just the 'recall area' is visible. Use the phrases in your recall area as your cue and recite as much information about each point as you can remember – check what you have remembered with your main notes. You will quickly find where the gaps in your knowledge are.



## Use the SBI Feedback Model to Understand Intent

When somebody disappoints you, fails to deliver what you expected, or lets you down in some way, what do you do? If you're like most people, you make assumptions that are usually not positive:

That guy is not a team player...he's lazy...doesn't care...just doesn't get it.

And then you take action:

I'll just find a workaround...get somebody else to do the work...rethink responsibilities...talk about him to someone else...initiate discipline.

We often don't even realize that we create stories about people in our heads, especially when they disappoint us. This happens all the time. We see a behaviour and assume we know why the other person acted a certain way, and react based on those assumptions without checking their accuracy.

Many difficulties can be avoided by having a clarifying discussion. Though people usually intend to do the right thing, sometimes something gets scrambled or misinterpreted along the way, and the impact is far from what they intended.

But the only way to know what someone intended is to ask them – and the only way to let a person know their impact is to tell them. These important conversations rarely happen, though, and we move through our days in a tangle of misperceptions and actions based on incorrect assumptions.

So, how do you have conversations to find out why a person chose to behave a certain way? We recommend closing the gap between intent and impact by using SBI to explore intent.

<https://www.ccl.org>

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### What Does SBI-I Stand For?

CCL's widely-recognized model for delivering feedback, the Situation-Behaviour-Impact Model (SBI), is proven to reduce the anxiety of delivering feedback and also reduce the defensiveness of the recipient. SBI is simple and direct: You capture and clarify the Situation, describe the specific Behaviours observed, and explain the Impact that the person's behaviour had on you.



### How Can You Use SBI-I to Give Feedback?

SBI can be used for giving both positive and negative feedback, as in these examples:

**1. Situation.** Describe the specific situation in which the behaviour occurred. Avoid generalities, such as "Last week," as that can lead to confusion.

Example: "This morning at the 11 a.m. team meeting..."

**2. Behaviour.** Describe the actual, observable behaviour. Keep to the facts. Don't insert opinions or judgments.

Example: "You interrupted me while I was telling the team about the monthly budget," instead of "You were rude."

**3. Impact.** Describe the results of the behaviour. Because you're describing exactly what happened and explaining your true feelings – not passing judgment – the listener is more likely to absorb what you're saying. If the effect was positive, words like "happy" or "proud" help underscore the success of the behaviour. If the effect of the behaviour was negative and needs to stop, you can use words such as "troubled" or "worried."

Example: "I was impressed when you addressed that issue without being asked" or "I felt frustrated when you interrupted me because it broke my train of thought."

This success of SBI is enhanced when the feedback, which is one-way, is accompanied by an inquiry about intent, which makes the conversation two-way:



**4. Intent.** Ask about the person's original intentions. This enables you to close the gap between impact and intent.

Example: "What were you hoping to accomplish with that?" or "What was going on for you?"

Inquiring about intent prevents us from veering off in the wrong direction based on faulty assumptions.

Extending the SBI feedback model to SBI-I allows the conversation to address what's behind a person's actions. This not only clarifies things, but it also builds trust and understanding. Simple solutions usually follow.

Inquiring about intent is also where good coaching starts.

When you inquire about intention, motivation, or what is behind the action, you are essentially in a coaching conversation – one that can make a positive difference well before a performance review or disciplinary conversation.





## DRIVING ISSUES

From fuel efficiency and load stability to UK driving laws, car and van drivers have a lot to remember whenever they get on the road. Below are six recent posts received in the PITA e-post bag with tips and information, mainly for UK drivers, though some of it is applicable universally.

- |  |        |
|--|--------|
| 1. 10 Tips to Increase Fuel Efficiency           | Page 2 |
| 2. Seven tips to keep in mind when loading a van | Page 3 |
| 3. Vehicle defects cause 32 fatal crashes a year | Page 4 |
| 4. Van Drivers Must Follow HGV Rules             | Page 5 |
| 5. Ten Strangest UK Parking Laws                 | Page 6 |
| 6. Eight Tips to Protect Tools in Vans           | Page 7 |

[www.LeaseVan.co.uk](http://www.LeaseVan.co.uk)

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## **10 TIPS TO INCREASE FUEL EFFICIENCY**

With fuel prices slowly climbing, vehicle owners are being advised of the ways they could drive more economically to help increase their fuel efficiency. With the average cost of a litre of unleaded fuel being just over £1.27, Brits are being reminded how they can change their driving to help keep their money in their pockets – particularly those who rely on vehicles for work.

Tim Alcock from LeaseVan.co.uk said: “Paying for fuel is another costly expense those with vehicles need to pay for, and if they’re reliant on the car or van for business it can soon add up. There are ways to help keep petrol costs as low as possible, mainly by being a bit more aware of driving habit and adapting to increase fuel efficiency. Most cars and vans now have built-in systems measuring mpg, which is a great way to look and see whilst you’re driving how your fuel is being affected. Drivers can consume almost 15% less fuel by taking note of what their fuel consumption display is telling them.”

These are LeaseVan.co.uk ‘s ten top tips to increase fuel efficiency:

### **1. Save on weight**

The more weight in the vehicle, the more fuel you’re going to use. If you know you won’t need a certain set of tools during the day, leave them at home.

### **2. Maintenance**

Regular upkeep and maintenance of the vehicle means that all the systems will be working to their optimum standards, meaning they’re as efficient as possible. Checking tyre pressures regularly can improve efficiency. Under-inflated tyres can cause you to use up to two per cent more fuel.

### **3. Accelerate gently**

The harder the acceleration, the more fuel consumed. The smoother the drive, in terms of acceleration and breaking, the less work the vehicle is going to do meaning less fuel will be used.

### **4. Be kind to your gears**

Staying in one gear for too long can slash your efficiency. Many vehicles are now fitted with gear shift indicators, which makes it easier to know which gear you should be in and when to change up or down.

### **5. Maintain a steady speed**

Your speed to drop when you travel uphill, then speed as you roll downhill can also have a positive impact.

### **6. Keep rolling**

It can seem inevitable to stop-start when in traffic, but slowly rolling along at a constant, low speed can help increase the efficiency of your driving. Try slowing extra early when approaching traffic lights or a queue and you might not have to stop completely.

### **7. Use your air-con wisely**

At low speeds air-con can have a dramatic effect on reducing efficiency, instead try opening windows to get air through the vehicle. However, at higher speeds such as on a motorway, the effect of air con isn’t that noticeable.

### **8. Stick to the limit**

The faster you’re travelling, the more fuel you’re using. Driving at 70mph uses up to 9% more than at 60mph, and up to 15% more than at 50mph!

### **9. Stay aerodynamic**

Wind resistance increases fuel consumption. Keeping windows shut at high speeds, removing roof racks and ensuring everything fits inside the vehicle can help save money on fuel.

### **10. Drive a manual**

According to the AA, automatics can use 10% to 15% more fuel than manuals.



## **SEVEN TIPS TO KEEP IN MIND WHEN LOADING A VAN**

New van drivers have been offered seven tips on how to properly load their vehicles without breaking any rules or regulations.

Vehicle leasing experts at LeaseVan.co.uk have revealed advice on the best loading and unloading techniques to help maximise space and reduce the risk of damaged goods.

Common sense goes a long way, but transporting goods safely by van requires a little more thought than simply throwing everything in and hoping for the best.

According to the Road Traffic Act, a person could actually be guilty of an offence if they use a motor vehicle on the road when the weight, position, or distribution of its load could be a risk or danger to someone.

Tim Alcock from LeaseVan.co.uk commented: "Loading goods and transporting them safely shouldn't have to feel like a game of Tetris or Jenga. Not only can a proper loading and unloading technique help you maximise space and reduce the risk of damaging goods during a move, but it's also a legal requirement."

### **1. Load large items first**

Loading bulky items first will allow you to fit all your smaller items around them later, helping you make the most efficient use of your space. As large items are also likely the heaviest items, it's also a good way to prevent damaging fragile items by crushing them. Heavy items should also be distributed evenly across the floor surface of your van, ideally across areas that are most structurally sound.

### **2. Distribute the load evenly**

Always load items in rows from the back to the front of the van space and utilise all space under and inside items to help restrict the movement of small items. You must be careful not to stack your load too high though, and do not exceed your van's load capacity – it's set for a reason and is the driver's responsibility to make sure it's road legal at all times.

### **3. Create a barrier**

Even if your load is secured, there's always a risk that items might move about on the road. In case they do, you need to protect yourself from being hit by any items that could come flying your way when you're driving. Mesh, netting or a sturdy piece of plywood can be used to prevent this.

### **4. Secure your goods**

Cargo can slide around during the journey, which can be a safety concern when it comes to unloading if items have piled up against the doors, or are stacked precariously. Protect fragile and delicate items with moving blankets. Use anchoring points within the van and lashing/tensioning straps to tie items down.

### **5. Flag it**

Although it's generally advised that you should avoid having anything protruding from the back of your vehicle, if a load must hang off the back of your van, ensure it's carefully secured and made visible by fixing high-vis materials to the most protruding points. Likewise, if you're carrying a particularly heavy or fragile load that requires you to drive more slowly, it can be helpful to indicate this to other drivers.

### **6. Control your speed and braking**

Regardless of the load you're carrying, driving a van full of goods is different from driving your average family car because if you accelerate too quickly or brake too harshly, items can move about inside. Van drivers must also be aware of the impact a heavy load has on steering, as a heavy load can have the momentum to turn your vehicle more or less than usual.

### **7. Unloading**

Make sure you unload your van in an area free from traffic or other obstructions so you can take your time. Choose a flat surface, or place brake chocks under the wheels to secure the vehicle if on an incline. Make sure you move items carefully so as not to disturb others than might fall and break. Always make sure to wear gloves too.



## **VEHICLE DEFECTS CAUSE 32 FATAL CRASHES A YEAR**

Drivers have been warned to ensure their vehicles are road worthy after the shocking number of UK road accidents caused by defects has been highlighted.

Motoring experts from LeaseVan.co.uk have reminded drivers just how many crashes in Britain can be attributed to faulty cars or vans and urged vehicle owners to regularly check the quality and condition of parts including tyres, brakes, lights and mirrors.

The latest Department for Transport statistics show that in 2017, a staggering 1,539 road accidents in the UK counted vehicle defects as a contributory factor – including 359 serious and 32 fatal.

Defective brakes were the most common problem and specifically cited in 570 of the most recent year's figures, whilst defective, illegal or under inflated tyres caused 472 crashes, including 115 serious collisions and 14 deadly ones.

Faulty tyres can cause a car or van to lose grip, especially on challenging road surfaces, whilst underperforming brakes can reduce a driver's ability to react promptly to unpredictable situations when behind the wheel.

Tradespeople in particular should bear in mind the maximum load their van can safely carry or tow, as an overloaded or poorly loaded vehicle or trailer was a significant factor in 119 UK road accidents over the twelve-month period, of which four proved fatal and another 29 were deemed serious.

Other vehicle defects that have led to a large number of serious and sometimes deadly crashes include faulty lights or indicators, sub-standard steering or suspension and flawed or missing mirrors.

They were a reported contributory factor in over 400 UK road accidents during 2017.

Such faults also lead to many near misses every day, with a motorist's ability to see properly at night and what's behind them both crucial to road safety.

Highly dangerous cars and vans that can't be steered properly or are too heavy should also be removed from the road until the problem is rectified.

It is recommended that vehicle owners in any doubt about how to assess quality and condition of any potentially defective parts, seek specialist advice as soon as possible.

All motorists must keep their MOT up to date, which legally must be every year if the vehicle is over three years old.

A regular service could be considered too, particularly for commercial or older vehicles and those that cover a high mileage.

Tim Alcock from LeaseVan.co.uk said: "Road safety should always be every vehicle owner's number one concern. It's always better for a journey to be delayed or not undertaken at all, than to travel on the road while risking a predictable and easily avoidable crash that could prove serious or even deadly. Vehicle defects are responsible for a serious collision on UK roads nearly every day, causing two to three deaths per month. But even one is one too many.

"There is nothing the government can do – all the required legislation is in place and it's impossible to enforce all the vehicles on the road all of the time. Drivers themselves must take responsibility and be sensible road users. Specialist assistance should be sought immediately if there is any doubt over the condition of your vehicle or if you're not sure how to check thoroughly themselves. Travelling with even the smallest suspicion that your vehicle could have faulty brakes, defective tyres, flawed lights, missing mirrors or substandard steering or suspension is beyond reckless."



## **VAN DRIVERS MUST FOLLOW HGV RULES**

Tradespeople who use work vans to get to and from jobs could be hit with a fine of up to £300 if they exceed the UK daily driving hours limit, experts have warned.

Motoring specialists from LeaseVan.co.uk have highlighted the law which states that drivers operating a van for commercial purposes must observe the same working hour restrictions and rest period requirements as professional HGV drivers.

Drivers operating a work van for more than four hours per day are not permitted to be behind the wheel for more than ten hours and aren't allowed to be on duty for more than 11 hours on any day in which they drive.

Working drivers can only reach the ten-hour limit twice per week. For other days that week, they are then restricted to nine hours – or 56 hours in a week and 90 hours in any fortnight.

Van drivers on the roads for business purposes must also get at least 11 hours rest daily, take breaks totalling at least 45 minutes after a maximum of four and a half hours of driving and take an unbroken rest period of 45 hours weekly.

Skipping breaks and exceeding these limits could see van drivers hit with a fine of up to £300.

It could also prove dangerous for both the driver and other road users, with tiredness and fatigue among the leading causes of road accidents and being potentially as deadly as drink or drug driving.

Some tradesmen and women don't currently track their time on the roads between jobs, but all have now been urged to keep an eye on their van driving hours.

Though vans don't require a tachograph to be fitted like HGVs, written records should still be kept.

The limits are suspended for the duration of emergencies, where the driver needs to take immediate preventative action to avoid danger to someone or an animal's life or health, or serious damage to property.

Restrictions are also relaxed when a major interruption to essential public service must be avoided – examples include gas, water and electricity supply, drainage, telecommunications or post, and roads, railways, ports and airports.

Drivers of military, police and fire brigade vehicles are also exempt from the UK driving hours rules, as well as those who never drive on public roads, whilst separate rules apply to Northern Ireland.

Tim Alcock from LeaseVan.co.uk said: "Some van drivers might not realise that if they're using their vehicle for business more than four hours daily, they have to follow the same rules as lorry drivers. When travelling from job to job or making a long delivery, it could be quite easy for a van driver to rack up a few hours behind the wheel in a day. But exceeding the applicable legal limit or not getting enough rest in could see van drivers hit hard in the pocket and facing a much-increased risk of crashing. In short, you should never drive for business for more than ten hours a day and you shouldn't work for more than eleven hours a day in which you drive.

"Van drivers also need to take at least eleven hours off between shifts on the road, though this can be reduced to nine on occasion. They should take at least 45 minutes in breaks if they drive for four and a half hours and take consecutive days off at least once a fortnight too.

"It's also important to keep a written record of driving hours if you expect to exceed four hours behind the wheel in a day, even though a tachograph isn't required on light goods vehicles. It's essential that all drivers remember, though, that driving when tired or fatigued can cause accidents and prove as dangerous as operating a vehicle while intoxicated."



## **TEN STRANGEST UK PARKING LAWS**

Drivers who want to steer clear of hefty fines have been urged to get to grips with ten of the strangest British parking laws. Researchers from LeaseVan.co.uk have revealed some of the lesser known parking laws that apply in the UK and advised vehicle owners to take note or risk being caught out by authorities. Offences that could land motorists in hot water include saving a space, using the pavement and parking badly.

Tim Alcock of LeaseVan.co.uk said: "Vehicle owners across the country could be breaking the law simply by parking their vehicles, and not even realise it. It's easy to use common sense and we've all got used to the wide array of rules that can apply in private car parks and where permits are required. But there are many more obscure rules that drivers could still fall foul of when parking on a public road. To help Brits avoid getting caught out by the relevant authorities, we've highlighted ten of the strangest parking laws in the UK and urge all motorists to take note."

Here is the LeaseVan.co.uk list of the ten strangest parking laws in the UK:

### **1. Don't save a space**

Using wheelie bins, cones or other objects to reserve a parking space near your house or workspace could see drivers fined as it may be seen as causing a dangerous obstruction on the road.

### **2. Leave the lights on**

Vehicles parked on a road where the speed limit is more than 30mph, facing away from the traffic or outside of a designated parking area should technically have their side lights left on overnight, to help prevent a nasty collision.

### **3. Check all mirrors**

Drivers and passengers need to check all their mirrors before exiting a parked vehicle, as it's always the occupants' responsibility to make sure that opening doors won't impede passing pedestrians, cyclists or vehicles.

### **4. Avoid the pavement**

Though it's commonplace on tight residential streets across the country, parking on the pavement has been outlawed by default in London since the 1970s unless permission is otherwise granted. For the rest of the UK, the practice isn't allowed where it might cause an obstruction.

### **5. Hug the kerb**

Bad drivers who essentially abandon their vehicle in the middle of the road by parking more than 50cm away from the kerb could be faced with an immediate on the spot fine.

### **6. Give junctions a wide berth**

It doesn't matter if it's the only space close to your house or work – parking within 10m of a junction is prohibited, to maintain road safety.

### **7. Keep it clean**

If a motorist allows their vehicle to build up too much dirt while it's parked they're breaking the law, as having an unreadable number plate is illegal.

### **8. Don't use cycle lanes**

Parking over a designated cycle lane that's painted on the carriageway isn't allowed – it would, technically, be blocking a lane – so drivers should look elsewhere for a space.

### **9. No beeping**

Pulling up outside a friend, relative or colleague's address and announcing your arrival with a beep is not permitted, as any use of a vehicle's horn while it's stationary is against the law. This also applies to taxis and delivery drivers.

### **10. Only load big items**

Vehicle owners can only use double yellow lines for loading if the goods being dropped off or collected are of sufficient size, weight or difficulty. Grabbing a quick coffee or lunch time meal deal doesn't count.





## **EIGHT TIPS TO PROTECT TOOLS IN VANS**

Tradespeople have been urged to fit their vans with CCTV and a safe to protect valuable tools and equipment from pesky thieves.

Van experts from LeaseVan.co.uk have revealed eight security measures tradespeople can take against criminals in their work vehicles, if forced to leave tools or equipment in vans while working or overnight.

From deadlocks and CCTV to deliberately parking badly and dirty vehicles, the specialist advice could help deter thefts and catch those responsible when a crime is committed.

Tim Alcock from LeaseVan.co.uk said: "Wherever possible, it's advisable to move expensive tools and equipment indoors for secure storage. But sometimes location and circumstances mean tradespeople have no other option than to leave items in their vans while they're parked up on a job or overnight. While of course thieves remain responsible when items are stole, wise tradesmen and women should do whatever they can to avoid becoming a target or victim of crime. To help deter criminals and make a theft less likely to occur or be successful, we've identified some precautionary steps van owners could take."

Here is the LeaseVan.co.uk list of security measures tradesmen and women can take when storing tools or equipment in their commercial vehicles:

### **1. Intruder alarms**

Many Brits nowadays find standard vehicle alarms annoying and will be tempted to ignore them, so it's worth considering installing a separate intruder alarm to discourage possible thieves.

Guard valuable good by fitting a coded device that will trigger when your van is accessed by someone who doesn't have permission.

Some alarms available online can be controlled remotely, synced with your mobile phone and even include a tracker.

### **2. Tool vault boxes**

To give valuable tools and equipment an added layer of security, purchase a van vault box or safe to store them in.

Requiring a key or combination to open, the range of high security options work in a similar way to a personal home safe, but often with tailored compartments for specific tools too.

### **3. Deadlocks**

Deadlocks can give vans enhanced defences against thieves by adding an extra and usually stronger locking point to cab and rear doors.

They are operated independently to a vehicle's standard, factory fitted locks, which allows the driver to control when each door is unlocked individually.

### **4. CCTV**

Most businesses will protect their premises with CCTV cameras that can help both deter and capture potential criminals, so why not their commercial vehicles too?

Small, high quality devices can now be brought relatively cheaply online and one should be installed on both the inside and outside of vans, either permanently or temporarily while parked.

Remember to display a clear warning sign too – this could make a thief turn away before the cameras are even called in to action.

### **5. Permanent tool marking**

Invisible anti-theft marking on your tools and equipment that requires ultra-violet light make them much easier to trace and their ownership indisputable, should they be stolen.

Tradespeople could also choose to use clear and obvious tool markings, such as carving or laser-burning a name or logo into handles, to put off thieves before items are taken.





## **6. Dyes**

Dye tags like those used in clothing retail are the best way to catch thieves literally red handed, as a burst of ink triggered by opening or moving a tool box without permission leaves little doubt who the criminal is and could deter them from trying to make off with your goods.

Concealed packs of dye, used by many banks within stacks of notes, could also be hidden within expensive equipment. They're armed by magnets and triggered by radio waves on a timer.

## **7. Inaccessible parking**

When there are tools and equipment in your commercial vehicle, reverse it into a parking space where the rear doors are practically touching a wall or other obstacle.

If a potential thief can't easily access your van and its contents, they're more likely to be discouraged and move along.

## **8. Simple dissuasion**

A basic sticker on the outside of a van declaring that no tools are stored in the vehicle may be enough to convince a criminal to try elsewhere. Even if it's not true, it's cheap, quick and worth a go.

Keeping the outside of your commercial vehicle in an unusually dirty condition when equipment must be stored inside could also put off thieves, by making the van seem to the outside world like it's less valuable and unlikely to contain important items.



## Sugar

It is now recommended that our intake of free sugars should not exceed 5% of total energy intake each day. But there is lots of confusion around this, with many of us not knowing how much this is and how to reduce our consumption.

In this article, we answer some common questions to help reduce the question mark surrounding sugar intake.

[www.thehealthyemployee.co.uk/](http://www.thehealthyemployee.co.uk/)



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### **What are free sugars?**

Many people think of sugar as something they add to their recipes or drinks in the form of table sugar. However, free sugars are all sugars added to food and drink including those in food production. This includes everything from crisps, biscuits, cakes, soft drinks, ready meals and sauces. These sugars should be consumed less often and in small amounts.

### **What are natural sugars?**

Sugar in dairy foods such as milk and yoghurt, fruit and vegetables in all forms, i.e. canned, frozen, fresh and dried, all contain natural sugars and don't count towards your free sugar intake. These foods form an important percentage of a healthy and balanced diet.

### **Why should I reduce my free sugar intake?**

Having a higher intake of free sugars means you will likely be consuming more calories overall, which is likely to lead to weight gain and eventually obesity. You are also at higher risk of developing tooth decay and type 2 diabetes.

### **Why do fruit and vegetables contain natural sugars but fruit and vegetable juices and smoothies contain free sugars?**

The UK government have separated these two types of sugars. Whole fruits and vegetables contain sugars that remain inside their cells, whereas juices, smoothies, purees and pastes contain sugars that are released from their cell structure during production. It has been suggested that sugars that have been released from their cell structure are more easily consumed in greater quantities. For example, you would likely be able to drink a whole smoothie much more quickly and easily than you would consume the equal quantity in whole fruit and vegetables that it took to make it. This could lead to overconsumption in calories and sugar. Therefore, it is recommended not to consume more than 150ml of fruit and vegetable smoothies each day, with this counting at one of your 5-A-Day.

### **Which foods contain the most free sugars?**

The main sources are both foods and drinks that contain added sugar. This includes soft drinks, cakes, biscuits, sweets and desserts.

### **How do you know how much free sugar is in the foods I buy?**

By law, packaging must include the total sugars on their nutrition label. Total sugars are a combination of free sugars and naturally occurring sugars.

Working out the free sugar quantity is tricky due to that fact that we are given the total sugar value. Looking at the ingredients is helpful as sugars added to the product must be included on the list. Free sugar can appear on the ingredients list as 'sugar, honey, brown sugar, maple syrup, molasses, treacle, nectars, maltose, corn syrup, fruit juice concentrate, isoglucose and crystalline sucrose'. Ingredients are listed in descending order of weight; the lower down the list the added sugar comes, the less that has been added. If there is no sign of any, all the better.

### **Are there lots of hidden sugars in the foods I buy?**

Savoury convenience foods can have sugar added to them, such as sauces condiments, ready meals and soups. The government has implemented a strategy to reduce calories of such foods by 20% by 2024, resulting in a reduced level of added sugar. However, sugar isn't 'hidden' in food, by law everything contained within the food product must be displayed within the ingredients. Take a few seconds to check over the ingredients list before purchasing.

### **How can I decrease my free sugar intake?**

Eating a diet of whole foods with limited processed products will enable you to significantly reduce your sugar intake. Eat a diet of fibre-rich starchy carbohydrates, fresh fruit and vegetables, foods high in protein such as lean meat, fish, eggs and low-fat dairy products. Swap sugar laden drinks with unsweetened alternatives such as water, low-fat milk, no added sugar squash and fruit teas. Bear in mind that alcoholic drinks currently contribute to around 10% of adults' free sugar intakes. Therefore decrease your intake by alternating between alcoholic beverages and glasses of water.

### **How to go sugar free with the family**

Things are usually easier to stick with if there are others around you going through the same experience. We also often worry about the health of our family, and want to instil the healthiest lifestyle choices upon them where possible.



Both of the above points make a great argument for cutting down on sugar as a family. Getting through the cravings together will help to strengthen willpower and resilience, seeing you through to the other side, where sugar isn't all that powerful and divisive!

The World Health Organization warn that we shouldn't be consuming more than 5% of our daily calories from added sugars, yet according to statistics from the National Diet & Nutrition Survey, adults aged 19-64 are consuming on average 12.1% of their daily calorie intake from added sugars.

Cutting down your sugar intake will not only decrease your risk of developing a vast range of chronic disease, but it will also leave you with greater energy, increased performance, reduced hunger and decreased cravings. So how can you do this in a way that will work for the whole family?

### **1. Don't reward with sugary treats**

This is something that most of us have probably experienced as a child, be it from a parent or grandparent. Occasional treats will do little damage, but instigating regular sweet treats will reinforce a link between achievement and sugar. Instead, arrange a day out or experience for whole family to enjoy together.

### **2. Aim to eat from scratch**

It's almost impossible to estimate the amount of sugar you are consuming if you eat food from packets. Sugar can be camouflaged using a range of terms such as glucose, sucrose, maltose and fructose – just to name a few! Aim to make food from scratch wherever possible.

### **3. Short cut dinner time**

It is no secret that there are just not enough hours in the day. We all do our best to make evening meals from scratch but occasionally buckle under the pressure of time constraints and opt for something a little less healthy. But a healthy and balanced meal needn't take hours, there is nothing wrong with using pre-chopped vegetables, or boiling frozen veg. A healthy and delicious stir fry can be made using pre-bagged stir fry vegetables and frozen prawns with a splash of soy sauce, this would only take a matter of minutes and not a takeaway menu in sight!

### **4. Producing for the masses**

No one understands the importance of cooking in large quantities than someone who has many mouths to feed. Cooking large batches of meals such as bolognaise, stews and curries. Split all of the leftovers into portions sizes before popping in the freezer for whenever you need them. Once again, this will stop the temptation of any unhealthy convenience foods making an appearance when a frozen home-cooked meal is one simple defrost away.

### **5. Be cautious of fruit juices**

Only one cup (150ml) of fruit juice counts towards 1 of your 5-A-Day. Any more than this quantity will not count and simply contribute excessively to your sugar intake. Try to stick to squash with no added sugar or fruit flavoured water. You could also make your fruit juice stretch a little further by watering it down with some fizzy water.

### **6. Keep snacks handy**

Becoming hungry in between meals calls for a snack that will give you the boost you need and sustain your energy levels until the next meal. Crisps, chocolate and cake will see a spike in blood sugar and a surge of energy, followed briskly by a swift plummet in both, leaving mood and energy on the floor. Snacks without the sugar surge can be quick and require no effort such as a piece of fruit, or some vegetable sticks with hummus. Alternatively you could make things a little more tempting by creating healthy energy bars that include ingredients such as nut butters, seeds, oats and dried fruit.

### **7. Make breakfast count**

Starting the day off well from the off will often dictate the day ahead. So make it a good one by avoiding cereals with high levels of sugar. Instead opt for a low sugar option such as, porridge topped with fresh berries and plain nuts, Greek yoghurt with fresh fruit or poached eggs with mushrooms and tomato. Filling up on protein will keep you full until lunchtime and help to keep any cravings at bay.

High sugar intake means we become accustomed to the taste, but by making some simple changes, the transition isn't as difficult as it may first appear.



## Movember, Men's Health Awareness Month

Movember is a global charity, but what do they stand for? Read on to find out.

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### Tackling...

- Prostate cancer
- Testicular cancer
- Mental health
- Suicide prevention

How can you get involved in Movember? [Movember in the workplace.](#)

On average, men die 6 years earlier than women, largely through preventable causes. Movember is a global charity is committed to increasing awareness, support, funding and education around men's health. Ultimately enabling men to live happier, healthier and longer lives.

### Suicide

Globally, every minute, a man dies by suicide. In the UK, 75% of suicides are men.

### What can we do?

- Talk
- Ask
- Listen
- Encourage action
- Check in

### [The importance of employee mental health.](#)

*'70% of men say their friends can rely on them for support, but only 48% say that they rely on their friends. In other words: we're here for our mates, but worried about asking for help ourselves. Reaching out is crucial.'*

[Papyrus](#) Aims to reduce stigma associated with suicide and increase awareness of young suicide through phone, SMS, and email.

[Calm](#) Seeks to prevent male suicide offering support to men in the UK, of any age, who are down or in crisis via their helpline and website.

[Mind](#) Information and support to make sure no-one has to face a mental health problem alone.

### Cancer

- Prostate cancer, [know the facts](#).

Prostate cancer rates will double over the next 15 years. Currently 1 in 8 men will be diagnosed in their lifetime.

- Testicular cancer, [know the facts](#).

Testicular cancer has doubled in the last 50 years. Those most at risk are aged 25-49.

### [Processed foods are driving up cancer risk.](#)

### 5 things men need to know to improve their health

1. **Spend time with people who make you feel good**
2. **Talk, more**
3. **Know the numbers** – Age 50, talk to your doctor about prostate cancer. If you're black, or have a father or brother with prostate cancer, talk to your doctor when you're 45. Know your numbers, know your risk.
4. **Check yourself** – Get to know what's normal for your testicles. Check regularly and speak to your GP if you notice a change.
5. **Move, more**

We offer a corporate nutrition workshop that specialises in men's health – [Mens health workshop](#) for more information.

[Get in touch](#) to discover how we can assist your organisation and improve workplace health.





## Understanding Stress Management

Understanding stress management and the impact this could be having on your health and wellbeing is a huge factor when it comes to the quality of your day-to-day life. Feeling stressed? Are you accepting it or doing something about it? Could you be happier by making a few lifestyle changes?

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### **Can stress be positive?**

- Stress can sometimes be positive
- Increase alertness and help you perform better in certain situations
- Excessive or prolonged stress can contribute to illness

### **What can prolonged stress lead to?**

- Lower immunity levels
- Digestive and intestinal difficulties
- Depression and anxiety

### **What happens to my body when I experience stress?**

- Sleeping problems
- Sweating
- Appetite changes
- Headaches
- Muscle tension
- Pain
- Nausea
- Indigestion
- Dizziness
- Heart palpitations
- Increased blood pressure
- In the long-term, increased risk of heart attacks and stroke

### **What are the behavioural and emotional effects of stress?**

- Anxiety
- Irritability
- Low self-esteem
- Becoming withdrawn, indecisive and tearful
- Constant worry and racing thoughts
- Irrational behaviour
- Being verbally or physically aggressive

### **What causes stress?**

- Work, money matters and relationships with partners, children or other family members
- Major upheavals and life events – divorce, unemployment, moving house and bereavement
- Minor irritations – feeling undervalued at work or arguing with a family member
- Sometimes, there are no obvious causes

### **Relationships and stress:**

- Your partner, parents, child, friend or colleague, can increase your stress levels

### **Work-Life Balance and Stress:**

- 1% of the UK working population works 45 hours+ per week
- Neglecting aspects of your life because of work may increase stress
- In 2008, mental health accounted for 442,000 cases of work-related illnesses



### **Money and stress:**

- StepChange Debt Charity found an increased demand of 56% for debt advice and support from 2012-2014
- 42% of those seeking debt help had been prescribed medication by their GP to help them cope
- Chronic stress and debt can result in depression and anxiety, and has been highlighted as a factor linked to suicidal thoughts and attempts

### **Smoking, drinking, drug use and stress:**

- Alcohol may make existing mental health problems worse. It is important to know the recommended limits and drink responsibly.
- Prescription drugs which may have been prescribed for very good reasons, can also cause mental and physical health problems if used for long periods of time.
- Street drugs, such as cannabis and ecstasy, are usually taken for recreational purposes. Problems start as your body gets used to repeated use of the drug, leading to increased doses to maintain the same effect.

### **How can you help yourself?**

1. Realise when stress is causing you a problem
  - Try to make the connection between feeling tired or ill and the pressures you are faced with
  - Look out for physical warnings such as tense muscles, over-tiredness, headaches or migraines
2. Identify the cause
  - Sort the possible reasons for your stress into three categories: Those with a practical solution, those that will get better in time, those you can't do anything about
  - Try to release the worry of those in the second and third groups
3. Review your lifestyle
  - Are you taking on too much?
  - Are there things you are doing which could be handed over to someone else?
  - Can you do things in a more leisurely way?
  - Do you have an understanding of stress management?
  - To act on the answer to these questions, you may need to prioritise things you are trying to achieve and reorganise your life
  - This will help to release pressure than can come from trying to do everything at once

### **Remember to seek health and support when you need it:**

- It is okay to ask for professional help
- It is important to know that you can get help as soon as possible, and that you deserve to get better
- The first person to approach is your family doctor, they can give advice about treatment, and may refer you to another local professional
- Other charities that could offer assistance include, Anxiety UK, Citizens Advice or StepChange

Our Health and Wellbeing Roadshows are a snippet of our full catalogue of Workplace Wellbeing services and events and are constructed of many healthy lifestyle zones, including stress management, enabling employees to connect the dots when it comes to living a happy and healthy life.



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*'70% of men say their friends can rely on them for support, but only 48% say that they rely on their friends. In other words: we're here for our mates, but worried about asking for help ourselves. Reaching out is crucial.'*

[Papyrus](#) Aims to reduce stigma associated with suicide and increase awareness of young suicide through phone, SMS, and email.

[Calm](#) Seeks to prevent male suicide offering support to men in the UK, of any age, who are down or in crisis via their helpline and website.

[Mind](#) Information and support to make sure no-one has to face a mental health problem alone.

### **Cancer**

- Prostate cancer, [know the facts](#).

Prostate cancer rates will double over the next 15 years. Currently 1 in 8 men will be diagnosed in their lifetime.

- Testicular cancer, [know the facts](#).

Testicular cancer has doubled in the last 50 years. Those most at risk are aged 25-49.

### **[Processed foods are driving up cancer risk.](#)**

### **5 things men need to know to improve their health**

1. **Spend time with people who make you feel good**
2. **Talk, more**
3. **Know the numbers** – Age 50, talk to your doctor about prostate cancer. If you're black, or have a father or brother with prostate cancer, talk to your doctor when you're 45. Know your numbers, know your risk.
4. **Check yourself** – Get to know what's normal for your testicles. Check regularly and speak to your GP if you notice a change.
5. **Move, more**

We offer a corporate nutrition workshop that specialises in men's health – [Mens health workshop](#) for more information.

[Get in touch](#) to discover how we can assist your organisation and improve workplace health.



## Products & Services

### PITA CORPORATE SUPPLIER MEMBERS

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### OTHER SUPPLIERS

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The Paper Industry Technical Association (PITA) is an independent organisation which operates for the general benefit of its members – both individual and corporate – dedicated to promoting and improving the technical and scientific knowledge of those working in the UK pulp and paper industry. Formed in 1960, it serves the Industry, both manufacturers and suppliers, by providing a forum for members to meet and network; it organises visits, conferences and training seminars that cover all aspects of papermaking science. It also publishes the prestigious journal *Paper Technology International* and the *PITA Annual Review*, both sent free to members, and a range of other technical publications which include conference proceedings and the acclaimed *Essential Guide to Aqueous Coating*.





## **ABB ADDS NEW FEATURES TO ITS WEB IMAGING SYSTEM**

Full Sheet Formation Analysis and Real-Time Wrinkle Count will help pulp and paper makers achieve optimal results.

Technology leader ABB launched two new features as part of its Web Imaging System, designed to help manufacturers deliver on-specification paper as part of ABB's commitment to providing end-to-end integrated solutions for the pulp and paper industry.

For fine paper and paperboard manufacturers seeking to optimize printability of their end product, reduce rejects and achieve consistent output quality, Full Sheet Formation Analysis provides conclusive, highly visualized measurement of paper uniformity for the full web. Aiming to redefine the standard of measurement accuracy, this patent-pending new feature offers deeper and more precise analysis of formation, helping papermakers better optimize established paper properties to achieve higher quality goals.

While traditional measurement techniques only look at a small portion of the web and may not reveal the floc sizes or shapes, ABB's full sheet solution leverages proprietary methods, processing parallelism, and the flexibility of the FPGA (field-programmable gate array) based smart camera platform to reveal and classify paper formation floc sizes and shapes. This greatly improves the ability to determine the uniformity of paper formation, while removing the possibility of misleading results.

For linerboard and liquid packaging manufacturers that require accurate detection and analysis of wrinkle formation, Real-Time Wrinkle Count provides online measurement and analysis of all paper web-based products. The new feature is the only system available for pulp and paper manufacturers that identifies wrinkles and aggregates the data to easily reveal problems across the web in real time, giving papermakers a competitive edge on maintaining quality.

The feature captures up to 1.4 million wrinkle defects per camera without overloading the system and sends alerts when KPIs are outside of user-defined thresholds. It then aligns defect maps to sample machine or cross direction location. It provides highly consistent results, enables precise laboratory correlation, allows for better management of the process, and reduces the amount of rejects.

"The launch of these two powerful new features demonstrates our continued investment in our Web Imaging System and reinforces our commitment to helping papermakers achieve the highest quality in their product output," said Stephen Mitchell, Product Manager, Web Imaging at ABB. "We are determined to push the industry forward with the development of new measurement techniques that use the most advanced technology to make online quality control easier to achieve."

Real-Time Wrinkle Count and Full Sheet Formation Analysis are part of the ABB Ability™ Quality Management System integrated solution suite consisting of products, services and applications for quality control. ABB's web inspection system, with formation analysis and/or wrinkle count features, will deliver actionable information to ABB's QCS (Quality Control System) and DCS (Distributed Control System) systems, based on industry-leading ABB Ability™ System 800xA, enabling process alarms and quicker corrective actions to quality issues.

<https://new.abb.com/news/detail/36344/abb-adds-new-features-to-its-innovative-web-imaging-system-to-help-paper-makers-enhance-quality>



## **ABB LAUNCHES WINDER PERFORMANCE OPTIMIZATION SOLUTION**

ABB's patented solution optimizes winder performance for improved availability, product quality and asset life.

Technology leader in digital industries ABB introduces Winder Performance Optimization, a digital solution for papermakers seeking to maximize asset performance, convert to different paper grades and/or increase machine speed, without replacing existing winders. The new solution works by benchmarking winder performance, implementing improvements, monitoring to sustain performance and—uniquely—further optimizing productivity by applying online calculations that continuously adjust winder acceleration and deceleration targets. This optimization aligns capacity to demands and can improve productivity significantly; recent implementations have provided 8 percent improvement.

The benefits of optimizing existing winders are many. The solution is cost-effective and easy to implement – requiring limited or no shutdown of the winder. Bottlenecks caused by larger, faster machines in the production process can be reduced and winder capacity maximized without compromising quality. KPIs are continuously monitored and analyzed to enable preventive maintenance and increased uptime. In regenerative mode, the solution feeds energy back into the system while decelerating winder drives, enabling mills to be more energy efficient.

“We understand the importance for papermakers to ensure their existing winders are able to handle demands of different grades, various roll orders with high quality and productivity improvement needs,” said Shankar Singh, global product manager, ABB Digital Solutions. “Our goal with this product is to help mills get improved productivity out of their existing winders, without the need to invest heavily in new equipment. With several existing installations around the world, we are pleased that papermakers are already realizing the benefits of improved winder performance and productivity by utilizing our patented solution’s unique ability to continuously adjust winder speed.”

Further advantages of ABB Winder Performance Optimization include the ability to monitor and improve roll set quality, and improved utilization of machine reel capacity. Local and remote dashboards enable instant, straightforward data visualization, with advanced analytics and daily analysis of performance, control (speed, load share, tension) and roll set performance carried out by ABB experts. Customers also benefit from configurable alerts to highlight performance falling outside site-specific thresholds.

ABB Winder Performance Optimization is part of ABB Ability™ Performance Optimization solution suite, whose offerings focus on maximizing equipment and process performance to ensure efficient operations. The Ability™ platform enables structured monitoring and analysis of KPIs and drives system performance to make sure the winder productivity is maximized.

For further information on performance optimization for pulp and paper, please visit: <https://new.abb.com/pulp-paper/abb-in-pulp-and-paper/collaborative-operations/winder-performance-optimization>



## **ABB'S NEW DODGE® SAFETY MOUNT SPHERICAL ROLLER BEARINGS**

Dodge® Safety Mount spherical roller bearings with patented built-in mounting system substantially cut installation time and improve safety and productivity

ABB has launched Dodge® Safety Mount spherical roller bearings with a built-in patented locking mechanism that reduces installation time by up to 75 percent compared to traditional products. Best suited for bulk material and air handling applications, the new bearing mounts by tightening fasteners instead of using a hammer and other tools. The new system also allows for simple installation and removal from the same side of the bearing, which means only one person is needed for the task.

“The new mounting system replaces the blows of a hammer with the tightening of fasteners,” says Jim Madsen, Dodge Mounted Roller Bearing Product Manager, ABB. “It also makes it faster to install large bearings, but more importantly, it makes it safer for the installer.”

Safety Mount bearings feature a triple-lip contact seal and corrosion-resistant flinger sealing system which prevents contamination from entering the product during installation and operation. A labyrinth seal option is available for high-speed and high-temperature applications.

Dodge Safety Mount spherical roller bearings are suitable for use with the ABB Ability™ Smart Sensor for mounted bearings, an easy-to-use, wireless sensor that monitors the health of bearings.

Dodge Safety Mount bearings combine the advantages of the Dodge Imperial family of bearings: factory sealed and greased, shaft ready out of the box, with no feeler gauges required. They are offered in split cap and single piece housing options in standard SAF, metric SN, Type E, and Imperial housing dimensions in sizes 4-15/16” to 7” (115 mm to 170 mm).

<https://new.abb.com/news/detail/26053/abb-dodge-safety-mount-spherical-roller-bearings>





## **NEW ANALYTICAL TRANSMITTER (AWT210)**

Analytical transmitter with plug-and-play technology offers cost-effective and future-proof solution for industrial pH and conductivity measurement.

ABB has taken an innovative approach to transmitter design with its new AWT210 single-channel two-wire transmitter for measurement and control of pH, redox (ORP) and conductivity in hazardous and non-hazardous industrial applications. The modular design reduces process downtime and overall operations expenditure while improving safety and boosting performance by optimizing plant control and availability.

The low power high-performance transmitter is built around single sensor interchangeable modular plug-and-play technology. This means that a major benefit of the AWT210's modular design is its ability to handle future sensor technologies such as ABB's digital EZLink sensors. By simply upgrading the transmitter with the necessary module, operators can avoid the cost and time of purchasing and installing new transmitter units.

Operational simplicity is at the heart of the AWT210. Based on ABB's common intuitive HMI, users can easily navigate the device. Easy setup menus provide step-by-step guidance and routine calibration tasks can be initiated at a touch of a button.

The AWT210 pH and conductivity systems ensure optimal safe performance in the harshest of applications. This combined with greater user flexibility and environmental compliance make them ideal for the oil and gas, pulp and paper, metals and mining and chemical and petrochemical industries.

The highly flexible transmitter design enables the same unit to be used with pH, redox (ORP) and ion-selective sensors as well as two- and four-electrode and toroidal conductivity sensors. Factory-calibrated modules for the different sensor types can be quickly fitted and exchanged when required via the transmitter's hinged door, enabling fast and easy upgrading and maintenance in the field.

This same approach applies when integrating the AWT210 into different control systems, equipped with a range of exchangeable modules available for 4-20mA with HART®, FOUNDATION Fieldbus® and Profibus PA® communications protocols.

With its range of wall, pipe and panel mounting options and IP66 rated enclosure, the AWT210 can be installed almost anywhere in an industrial process with both intrinsically safe and non-incendive versions for hazardous area installation certified by USFMc and ATEX/IECEx.

To prevent unauthorized modifications to calibration and configuration settings, multi-level security access ensures that users can only perform tasks within their specific profiles, with a choice of read-only, calibrate and advanced security access levels.



<https://new.abb.com/products/measurement-products/analytical/continuous-water-analysis/transmitters/awt210-2-wire-transmitter>



## **ABB ABILITY™ CONDITION MONITORING SMART SYSTEM**

Transcontinental Advanced Coatings extends successful pilot of ABB's smart sensor monitoring and predictive maintenance solution as it aims for zero downtime.

Industrial coatings producer Transcontinental Advanced Coatings has successfully piloted ABB Ability™ Condition Monitoring on critical equipment at its UK facility. The manufacturer used the pilot project to enhance the existing predictive maintenance regime on a critical oxidizer process that cleans the air before it's released to the atmosphere.

ABB Ability™ Smart Sensors – fitted to the motors and mounted bearings that operate on the oxidizer's two fans – collect and analyze data to monitor performance. The system alarms if pre-defined limits for parameters such as temperature and vibration are exceeded. This enables preventive maintenance to be carried out before problems occur.

Transcontinental Advanced Coatings – based in Wrexham, North Wales, UK, and Matthews, North Carolina, United States – makes precision coated papers, films and specialty substrates for digital imaging, electronics, medical and optical technologies. Based on the success of the pilot project, the company will extend the remote condition monitoring solution to an entire production line in its Wrexham facility.

“Our overall goal is zero downtime. ABB Ability™ Condition Monitoring ensures we can identify equipment issues early on – before they happen – and take action to prevent breakdowns from occurring,” says Dr. Keith Vidamour, Engineering Manager for the Transcontinental Advanced Coatings North Wales plant. “Conducting maintenance as needed rather than on a fixed schedule will help us improve reliability and process control.”

Previously, the company relied on monthly manual monitoring of the fans' motors and bearings using thermal imaging, oil sampling, and vibration analysis. These tests were only a snapshot of the condition of the process rather than a continuous real-time picture. The results also relied on an individual engineer's interpretation.

“We are keen to move to a more objective, more data-based condition monitoring regime,” says Dr. Vidamour. “We are now monitoring additional parameters and have access to far more objective information than ever.”



The second phase of the project will use multiple smart sensors to remotely monitor the motors and bearings throughout a process, together with up to four Bluetooth gateways connecting with ABB's secure server. Ultimately, the company will roll out the solution across its global platform.

“Transcontinental Advanced Coatings wanted to prove that the condition of critical machinery could be remotely monitored,” says Derek Robinson, ABB's Key Account Manager for High- and Low-Voltage Motors. “The successful trial proved they could significantly improve overall maintenance efficiency. The company's decision to continue to add additional ABB Ability™ Smart Sensor technology will give it a real competitive edge.”

<https://new.abb.com/motors-generators/service/advanced-services/smart-sensor>





## **ANDRITZ LAUNCHES MASTERDRY™ DRYER FABRIC PORTFOLIO**

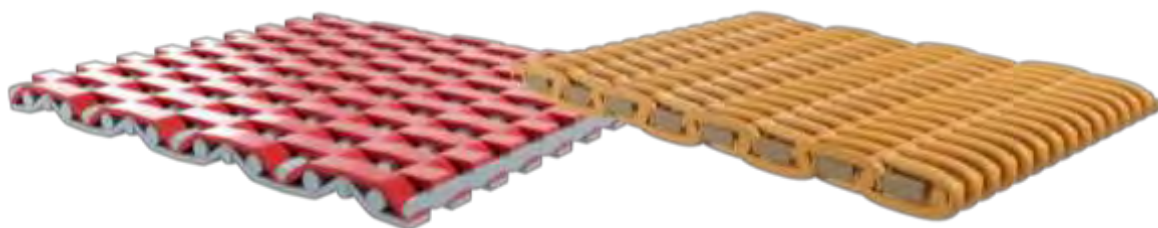
International technology Group ANDRITZ presents its new “MasterDry™” dryer fabric portfolio based on premium-performance dryer fabric technology.

MasterDry includes best-in-class woven and spiral fabrics specifically designed for the broadest range of applications, from the fastest low-caliper, single-tier positions, for high-temperature positions, and for those positions requiring extreme contamination resistance. It includes specially engineered woven or spiral dryer fabrics to cover an entire paper machine producing paperboard/packaging and graphical papers or also pulp machines, thus providing customers with a very comprehensive suite of high-performance dryer fabric options.

“We will continue to focus on R&D to launch new innovative products and to invest in our plants to become the premier global supplier of the highest quality dryer fabric technology with shortest delivery times,” says Bill Butterfield, Executive Vice President and Chief Technology Officer at ANDRITZ Fabrics and Rolls.

With the new MasterDry portfolio of dryer fabrics, ANDRITZ once again confirms its position as one of the global market leaders for the supply of innovative fabrics, press felts, and roll technology solutions.

For more information, please visit [www.andritz.com/fabrics-and-rolls](http://www.andritz.com/fabrics-and-rolls).



MasterDry offers a complete portfolio of high-quality woven and spiral dryer fabric technology for every application in paperboard/packaging, graphical, and pulp machines.



## **ANDRITZ LAUNCHES STRATAPRESS™ SX SINGLE-SEAM PRESS FELT**

International technology Group ANDRITZ has launched its new-generation “StrataPress™ SX” felt with patented, integrated single-seam technology.

StrataPress SX is specifically engineered for the most demanding positions in paperboard/packaging, graphical, and pulp machines. Its patented, integrated seam ensures strength and durability while delivering rapid and safe installation.

It is specially designed with a game-changing combination of materials, base fabric structures and unique batt concepts that deliver the highest sheet quality, faster machine speeds, reduced energy consumption, and extended life potential.

“With StrataPress SX, for the first time in our industry, customers can now enjoy the convenience and safety of seam felts along with the superior machine performance previously only available from premium-class endless press felts. And with its unique compressibility and nip dampening characteristics, StrataPress SX provides that superior dewatering performance even in the hardest press nip applications, and with reduced operating costs,” says Bill Butterfield, Executive Vice President and Chief Technology Officer at ANDRITZ Fabrics and Rolls.

### **STRATAPRESS SX – THREE PRODUCT CLASSES**

As StrataPress SX technology is built utilizing three distinct warp systems, there is an exact product specification specially engineered to maximize machine performance for graphical, board and packaging, and pulp grades.

StrataPress SX is also available with ANDRITZ’s exclusive “QS” Quick Saturation technology. Hydrophilic components embedded in the base fabric structure ensure optimized water management to provide improved start-up behaviour, enhanced profiles and better NIP dewatering over the entire service lifetime.

With the new StrataPress SX technology, ANDRITZ once again confirms its position as one of the global market leaders for the supply of innovative fabrics, press felts, and roll technology solutions.

For more information, please visit [www.andritz.com/fabrics-and-rolls](http://www.andritz.com/fabrics-and-rolls).



StrataPress SX is specifically engineered for the most demanding positions in paperboard/packaging, graphical, and pulp machines.





## **VALMET DEVELOPS DIGITAL PLATFORM FOR FIELD SERVICES OPS**

Valmet is investing in a new digital platform to support, streamline and develop its strategically important field services. The global implementation of the new platform will be completed by spring 2020, and it will replace multiple current platforms.

Valmet's vision is to become the global champion in serving its customers. One step toward the vision is to further improve field services as an important part of Valmet's customer service, support and local presence. Valmet Field Services offering consists of maintenance and process supporting services, annual shutdowns, and remote assistance.

"The new platform will further improve Valmet's capability to provide world-class services to our customers by providing workforce transparency and connecting our on-site field service professionals with Valmet Performance Centers through remote connections. This makes delivering desired services at customers' sites faster and more accurate. It also allows us to better predict and advise services and develop shared maintenance and service roadmaps for our customers' equipment and processes," says Anders Öhrblad, Director, Field Services Growth, Valmet.

### **The investment supports Valmet's strategy to become a frontrunner in field services**

The investment is part of Valmet's strategic decision to develop its field services through a dedicated program. The focus is on new field services offerings, improved service capacity and availability, developing competencies, and harmonization of processes. Today, Valmet has more than 1,100 field services professionals, an increase of 11 percent in the past year.

The platform will be delivered by ServiceMax, a leading provider of Field Service Execution Software.

"We're honored to support Valmet's vision for field service excellence and the strategic importance of service for the company's asset-intensive customers," says Neil Barua, CEO of ServiceMax. "Our partnership with Valmet will help not only deliver a world-class industrial field service program, but also provides Valmet's technicians with the tools to better predict service requirements. The partnership will also help in developing shared maintenance and service roadmaps around customers' industrial equipment and processes for the long-term."

<https://www.valmet.com/media/news/press-releases/2019/valmet-develops-its-field-services-operations--invests-in-a-new-digital-platform/>



## **VALMET INTRODUCES A NEW USER INTERFACE FOR THE DNA SYSTEM**

Valmet is taking process automation further by introducing a new web-based user interface for its Valmet DNA automation system. Leveraging modern web technologies, the innovative Valmet DNA User Interface (DNA UI) extends the use of automation system beyond the traditional control room.

The introduction of the DNA UI is a part of the continuous renewal of Valmet DNA automation system, which is used in pulp, paper, energy and other process industries around the world.

### **Relevant information based on the needs of different user groups.**

Intuitive user experience and serving the needs of various user groups has been the main focus in the development of the new Valmet DNA User Interface.

The system adapts shown information based on the needs of various users and user groups. Relevant information is delivered in visual, well-structured, easy-to-understand dashboards, process and sub-process views, which allows the users of the automation system control the process better than ever before.

“It is essential for us to understand how our customers consume information, so we can help different user groups to process information faster. In the new DNA User Interface, we have structured and visualized data in new ways to make the workflow more intuitive – which in turn helps to make faster conclusions based on the data,” explains Jukka Ylijoki, Vice President, R&D, Automation business line, Valmet.

### **Control room no longer sets boundaries for work**

Traditionally, system information and people using it have been tied in the control room. Built with latest web technologies, Valmet DNA User Interface comes with a secure web-based access that enables the mill or plant teams to access relevant information whenever they need it, regardless of their location.

“From logistics and laboratory to the boardroom, the entire site community needs specific information about the process. With Valmet DNA User Interface, users no longer need to stay in the control room to be on top of the situation,” Ylijoki continues.

### **Increased awareness leads to increased efficiency**

With over 40 years of heritage in digital industrial automation, Valmet wants to make sure that industrial plants are safe and efficient to operate and that they meet the set quality and production targets.

“For the users, the new Valmet DNA UI will mean a complete feeling of awareness. They will always have the situation in hand and can easily manage information content to support their decisions. This leads to efficiency and helps to achieve savings throughout the process,” Ylijoki says.

Valmet DNA User Interface is the first web-based user interface on the market that can be delivered also as an upgrade to an existing Valmet DNA automation system. This means that in the future the existing customers can unlock the benefits the web-based user interface offers via upgrade rather than a full system renewal.

<https://www.valmet.com/automation/control-systems/valmet-dna/user-interface/ui/>



## **VALMET'S NEW INLINE OPTICAL TOTAL CONSISTENCY & ASH SENSOR**

Valmet has launched a new Valmet Optical Consistency Measurement (Valmet OC2R), which has been developed for recycled pulp manufacturing applications, as well as general stock preparation applications in paper, tissue, and board.

Valmet OC2R shares the total consistency measurement method utilized in the original Valmet Optical Consistency transmitter with the added capability of ash content available as a second measured value. Particularly in low-consistency measurements, Valmet's optical transmitters are frequently the only possible measurement technique to provide reliable results. Valmet OC2R offers easy and low-cost installation with a measuring probe that makes insertion and removal possible without requiring special tools or a process stop.

For mechanical and chemical pulp, as well as for recycled fiber furnishes, Valmet OC2R is an ideal choice for control applications, from re-pulping and screening all the way to the machine chest.

"The addition of the ash measurement to total consistency opens new possibilities for recipe management in pulp blending, better broke handling, and reducing machine chest furnish variability. In recycled fiber processing, the benefits of the two measurements include better end product quality with improved strength and optical properties," says Heikki Föhr, Product Manager, Valmet Optical Consistency Measurement, Automation business line, Valmet.

### **Technical information about Valmet Optical Consistency Measurement (Valmet OC2R)**

Valmet OC2R joins the Valmet Optical Consistency Measurement (Valmet OC) family of transmitters, each optimized for use with eucalyptus pulp, recycled fiber and chemical pulps in addition to mechanical furnishes. Installed in an area of turbulent flow, the temperature and vibration resistant probes are self-cleaning even in the demanding environment of recycled fiber processing. Sharing the same basic components and modules optimized for each application, they have gained a deserved reputation for accuracy, reliability, and ease of installation.

<https://www.valmet.com/valmetoc>





## **VALMET'S NEW ROLL COVER MATERIAL**

Valmet is the first company in the world to offer roll covers based on biomaterials and recycled materials for board, paper, tissue and pulp making. These new composite covers for press, guide and calender rolls are now available for the first customer deliveries.

In new composite roll covers Valmet uses bio-based resin and hardener in the polymer matrix. The reinforcing fiber and filler are originating from recycled consumer plastic and glass. Depending on cover type, the content of recycled or bio-based raw materials is 75-96 percent. New materials are being tested continuously, and the target is to reach 100 percent as soon as possible.

Valmet has set strict criteria for the bio-based raw material in the covers. Only renewable materials derived from non-food chain plants or plant parts are used, in order to prevent the effect to global food production. Cultivation and harvesting of plants must not endanger the growth of natural forests either.

"In best cases, bio-based materials can be produced from plant parts that would otherwise be waste. For example, lignin, carbon black made of lignin and nanocellulose can be utilized as reinforcing fillers in roll covers," explains Jani Turunen, R&D Manager for Polymeric Roll Covers, Valmet. "Our customers do not have to compromise in product performance either, as the results have shown that in some applications the performance is even above the traditional offering."

### **A more sustainable future with a new generation of roll cover materials**

Roll covers used in paper, board, tissue and pulp making need periodical renewal and thus consume tons of materials. A major part of the raw materials of traditional roll covers has been manufactured from fossil-based materials, which has been refined and processed from crude oil.

Valmet's R&D work focuses strongly on enhancing raw material and energy efficiency and promoting the use of renewable raw materials. Valmet is continuously investigating the possibilities to replace fossil-based materials with renewable or recyclable materials. For example, recycling and more sustainable use of ceramic and metallic roll covers is under active research.

"Our continuous investing in R&D of sustainable solutions for our customers helps us increase the occupational safety of our employees, replace fossil-based raw materials with renewable ones, re-use and recycle materials and save energy. This is our contribution to the global challenge we face regarding our planet's future. And by doing this, we are helping our customers do their part as well," says Jukka T Heikkinen, Senior Manager, Roll R&D, Valmet.

"In best cases, bio-based materials can be produced from plant parts that would otherwise be waste. For example, lignin, carbon black made of lignin and nanocellulose can be utilized as reinforcing fillers in roll covers," explains Jani Turunen, R&D Manager for Polymeric Roll Covers, Valmet.

<https://www.valmet.com/media/articles/services/recycled-and-renewable-raw-materials-in-future-roll-covers/>



## **VALMET'S ULTRA-HIGH DEFINITION OPTICS TO FIBER ANALYSIS**

Utilizing more than 35 years of experience in laboratory and online fibre measurements, Valmet has launched a completely redesigned optics module. The ultra-high definition optics can measure more fibres with much higher resolution in order to expand application areas and improve measurement precision.

Since its launch in 2013, the Valmet Fiber Image Analyzer (Valmet FS5) has made precise standardized fiber morphology measurements possible without special training, sample preparation or laboratory facilities. Now, the new optics feature a wider measurement cell to allow improved shive particle measurement, a larger image area to measure more and longer fibres and an ultra-high definition camera providing faster fibre analysis and increased sharpness for better fibril and small particle detection. The new optical module is also available as an upgrade to existing Valmet FS5 analyzers.



"Our Research & Development team did it once again! The improvements in the optics allow our customers to see smaller particles, identify particles like parenchyma cells, and to be able to measure longer fibers or larger shives particles than before," says Tommi Niskanen, the Product Manager for Valmet FS5.

Valmet FS5 is intended for use in regular mill testing while also meeting the requirements for research laboratories. The results are ISO standard compliant and fully traceable and provide a better understanding of the paper making potential of the pulp, helping papermakers to better manage the process to get targeted end product quality.

The upkeeping requirements for Valmet FS5 are minimal and each unit is calibrated with an accredited calibration tool at Valmet's production center in Kajaani, Finland.

"Valmet FS5 platform has been validated by over one hundred happy customers globally. The possibilities with the new optics have created positive feedback from both our old and new customers," says Tuomo Kälkäjä, Director for Valmet's analyzers, measurements and performance solutions.

### **Ultra HD technology detects smaller particles**

The Valmet Fiber Image Analyzer enables pulp and paper mills to unlock the true value potential of their raw material for optimum end product quality. With the new compact and user-friendly unit, customers can benefit from a wider range of available measurements combined with improved usability and reliability resulting in savings in terms of both time and resources.

The analyzer requires no special facilities other than water, drain and electricity. It is transportable for use on the machine floor as well as shift laboratory, when and where needed for fast and accurate pulp property analysis. Easily programmed for customized analysis and reporting, the FS5 is provided with basic measurements of fiber dimensions (length & width), fines, coarseness and curl, with two optional measurement packages that add blend ratio, external fibrillation, kink and cell wall thickness as well as neural network classification of vessel cells, shives, parenchyma cells, fibre entanglements, and more.

Read more: [www.valmet.com/fs5](http://www.valmet.com/fs5)



# CHEMICALS





## **BASF LAUNCHES NEW DEFOAMER**

BASF launches new defoamer that complies with major food contact regulations for adhesives, paper coating applications and functional packaging

The new new defoamer – Foamaster® WO 2360 – is compliant with China's food contact regulation GB 9685-2016 for adhesives and major food contact regulations such as Code of Federal Regulation (CFR) Title 21, Food Contact Materials Regulation (EC), Bundesinstitut für Risikobewertung (BfR) and Swiss Ordinance on Food Contact Materials for packaging.

China's National Food Safety Standard GB 9685-2016 governs the use of additives in food contact materials and their products. It was introduced by China's National Health and Family Planning Commission (NHFPC), which came into force in October 2017.

The new white oil-based defoamer demonstrates excellent and persistent defoaming efficiency, excellent compatibility across acrylic PSA systems and good oil-slick resistance during storage for adhesive formulations. When applied in paper coatings, Foamaster® WO 2360 also exhibits excellent defoaming efficiency coupled by good anti-foaming and fast foam break time.

The versatility of Foamaster® WO 2360 makes it widely applicable for paper coating formulations, barrier coatings, paper and paperboard applications that all require contact with food. This, in turn, removes the need for customers to carry multiple defoamers in its inventory.

For more information, please visit [www.basf.com/additives](http://www.basf.com/additives)

## **BASF HAS RECENTLY LAUNCHES A HIGH MOLECULAR WEIGHT DISPERSING AGENT**

BASF has recently launched a new high molecular weight dispersing agent: Dispex® Ultra PX 4290, which features excellent performance in pigment stabilization, outstanding color strength and brilliant viscosity reduction.

Dispex® Ultra PX 4290 offers the benefits of higher pigment loadings while maintaining exceptional flow characteristics. Other highlights also include improved gloss, anti-flooding behaviour and excellent flocculation stability.

Dispex® Ultra PX 4290 was developed in response to a surging market demand for a dispersing agent, which enables broad applicability in both inorganic and organic pigments and fillers used in water-based automotive OEM and refinish coatings, industrial coatings and wood coatings. Its application can be further extended to printing inks and adhesives as well.

For more information, please visit [www.basf.com/additives](http://www.basf.com/additives)



## **EXXONMOBIL LAUNCHES LANDMARK GEAR AND CIRCULATING OIL**

Breakthrough synthetic lubricant offers class-leading performance benefits to the pulp and paper industry

- Mobil SHC™ Elite performs at temperatures up to 150°C in intermittent service
- Advanced gear and circulating oils can extend oil drain intervals by up to 12 times<sup>1</sup>, reducing maintenance costs and enhancing safety
- Offers up to a 3.6% energy efficiency benefit versus mineral oils<sup>2</sup>

ExxonMobil is introducing Mobil SHC™ Elite, a breakthrough synthetic gear and bearing circulating oil for pulp and paper mills. The lubricant's advanced formulation can help protect equipment operating at extreme temperatures, while increasing energy efficiency and extending oil drain intervals.

Extensive testing shows that Mobil SHC™ Elite can deliver double the oil life<sup>3</sup> of traditional synthetic products and up to 12 times<sup>1</sup> the oil life of mineral products. It can also help protect equipment operating at temperatures up to 150°C in intermittent service making it ideally suited for a range of paper industry applications, such as helping to protect dryer and calendar drives, as well as kiln and trunnion bearings.

The advanced synthetic lubricant is specifically formulated to deliver excellent wear protection and oxidation resistance without any of the compatibility challenges often associated with glycol-based products used in high temperature applications.

In addition, Mobil SHC™ Elite has also demonstrated an enhanced torque ratio, enabling it to deliver a 3.6%<sup>2</sup> energy efficiency improvement when compared with conventional mineral oils.

“Protecting heavily loaded paper manufacturing equipment from high in-service temperatures can be a major challenge for mill operators,” said Emre Noyan, industrial marketing manager at ExxonMobil. “Mobil SHC™ Elite’s extended oil life, wide temperature range performance and energy efficiency improvement can help operators increase uptime and cut costs – giving them a competitive edge.”

Mobil SHC™ Elite has already received approval for its proven performance from leading gearbox manufacturers including Siemens, whose FLENDER gear units depend on effective lubrication to ensure reliability in intense operating environments.

For more information about Mobil SHC™ Elite or Mobil SHC™ products, please visit [www.mobil.com/industrial](http://www.mobil.com/industrial).

- [1] Actual results can vary depending upon the type of equipment used and its maintenance, operating conditions and environment, and any prior lubricant used.
- [2] Energy efficiency relates solely to the performance of Mobil SHC™ Elite when compared to conventional (mineral) reference oils of the same viscosity grade in gear applications. The technology used allows up to 3.6% efficiency compared to the reference when tested in a worm gearbox under controlled conditions. Efficiency improvements will vary based on operating conditions and application.
- [3] Up to 2x oil life as demonstrated in numerous bench and rig tests. Oil life will vary based on application and operating conditions



## **VPCI®-386 WATER-BASED ANTI-CORROSION COATING**

It can be challenging to balance corrosion protection and worker safety in manufacturing, since many powerful corrosion inhibitors are hazardous chemicals. However, Cortec® Corporation has repeatedly demonstrated the potential for replacing many traditional hazardous materials with more environmentally responsible, user-friendly alternatives such as Cortec's VpCI®-386 water-based coating.

Just last year, Cortec® was able to help a refrigerator coil manufacturer replace a solvent-based coating with a water-based coating for increased worker safety and better corrosion protection. The manufacturer was using the solvent-based paint to coat new refrigerator coils. This exposed workers to a strong solvent smell and associated hazards. To make matters worse, the paint did not provide the long-term level of corrosion protection desired. Cortec® was able to introduce VpCI®-386 to the manufacturer as a water-based corrosion inhibiting alternative, helping them reduce their solvent VOCs and providing the desired corrosion protection.

VpCI®-386 is a fast-drying water-based acrylic one-coat system topcoat that can be applied direct to metal for protection in harsh, outdoor, unsheltered applications. The complex mixture of corrosion inhibitors offers protection that competes with most paints and zinc-rich primers. VpCI®-386 has excellent UV resistance. It is also weldable and can be used to keep surfaces corrosion-free prior to welding. VpCI®-386 comes as a clear coat for minimal change to the surface appearance, or it can be matched to most custom colors.

Learn more about VpCI®-386 at: <https://www.corteccoatings.com/product/vpci-386/>



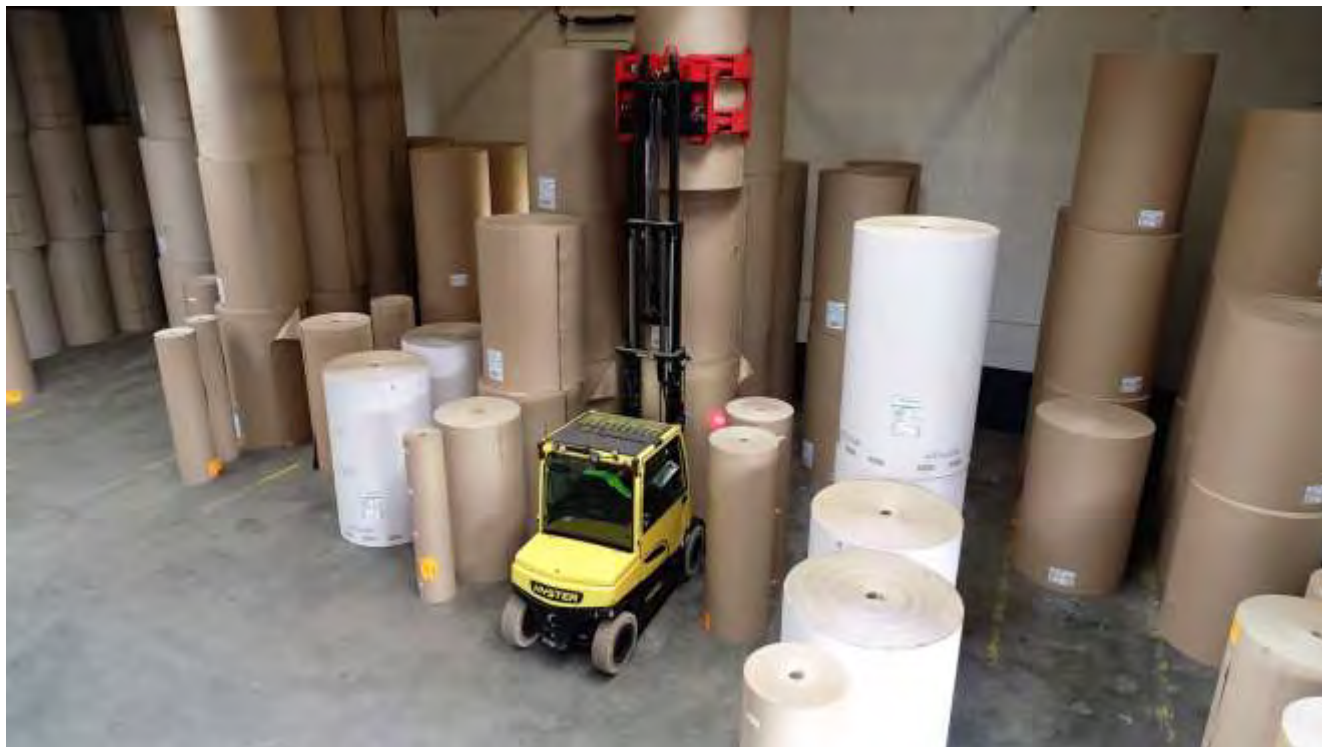


# **MATERIALS HANDLING**



## **HYSTER LIFT TRUCK SOLUTION SIMPLIFIES LOADING OF PAPER REELS**

To simplify the process of loading trailers with paper reels in line with industry best practice recommendations, Hyster Europe has specially optimised the J5.5XN6 electric lift truck.



“When transporting paper reels by road, it is typical industry best practice to load and position the reels down the centre line of the lorry trailer in single file to help evenly distribute weight and improve stability,” says Josie Burrell, Industry Manager for Hyster Europe.

“This would ordinarily require a 7-tonne capacity lift truck with an attachment spacer to reach over the trailer bed and handle the reel,” she continues. “However, the Hyster® J5.5XN6 lift truck with tilting carriage option and a reel clamp attachment makes all of this possible with a 5.5-tonne capacity lift truck instead.”

The electric Hyster® J5.5XN6 lift truck design extends capability in arduous paper reel handling applications more effectively than simply increasing the lift capacity. This is combined with a hydraulic tilting carriage which increases reach by 250mm at lorry bed height, ideal for efficiently loading reels onto a trailer in the correct position.

To help reduce costly potential damage to paper reels, with the truck mast on full forward tilt, the Hyster® tilting carriage option truck brings the paper reel clamp back to a horizontal level. This, combined with the stability of the extended load centre, helps the paper reels to be placed down flat, reducing damage to the edge of the reel.

A chain slack prevention valve also helps avoid wrapper damage. When the clamped load is stacked and the mast lowering function is operated, this feature prevents the mast chain from losing tension. Therefore, when the clamp attachment arms are opened, they do not slip and damage the roll wrapper.





“The right choice of attachment is key for both efficiency and damage avoidance in paper reel handling operations,” says Josie. “However, there are a number of additional options and upgrades that may help too.”

This includes environment protection features, such as a belly pan, tilt cylinder boots and a sealed drive axle to prevent the ingress of dust. Also available are camera solutions to support operator accuracy, truck-to-truck, truck-to-object and truck-to-pedestrian detection systems and Hyster® Tracker telematics for fleet management.

The agile electric Hyster® J5.5XN6 lift truck also offers space saving benefits, with a compact design and zero turn radius (ZTR) axle making it easy to manoeuvre in both the warehouse and the loading bay, and tough enough to withstand all weathers when used outdoors.

“Organisations in the paper supply chain are often keen to reduce their carbon footprint,” says Josie. “This electric lift truck offers zero emissions and a low energy performance mode option, providing an environmentally friendly solution but with no compromise on performance.”

Like the rest of the Hyster® range of lift trucks and warehouse equipment, the Hyster® J5.5XN6 forklift offers a low cost of ownership, with long service intervals, maintenance free AC traction and hoist systems and easy serviceability. Paper applications worldwide receive service and support from local Hyster distribution partners.



[www.hyster.eu](http://www.hyster.eu)





## **ANALOGUE TOOLS VITAL IN A SMART FACTORY FUTURE**

With a smart future edging closer and closer, manufacturing stands to be one of the most heavily affected industries. While some may associate this change with disruption, a leading UK-based materials handling equipment provider says that analogue tools and human input will remain key elements in both the creation and the long term success of a smart factory future.

Midland Pallet Trucks, a West Midlands-based provider of hand pallet trucks, lift platforms, moving skates, and a wide range of other materials handling equipment, believes that fears of complete upheaval are unfounded. The firm says that analogue tools and human knowledge will both play a critical role in the digital shift, with manual processes and decision making, alongside operation of digitally-enhanced machinery, being supported through the use of analogue tools.

The company cites two primary reasons why the use of analogue tools will remain a 'go to' method for operational success. Firstly, the human element must always be factored into plans for a smart factories; the ability for skilled workers to manually operate equipment for optimal results in particularly complex moves, for example.

Secondly, a smart factory future will not be an instantaneous change. The notable skills gap which currently exists means that a smart factory future is a long term change, with a significant 'hand over' period where analogue and digital concepts will be used in unison to fully develop a safe, effective, efficient and smart environment.

"We often hear that a smart future is on the way, but what many don't realise is that the essential building blocks of industry 4.0 are already widely available," says Phil Chesworth, Managing Director of Midland Pallet Trucks. "Of course, many operational processes can and indeed will be automated eventually using the latest connected technologies, but materials handling, and many other complex areas of warehouse management, will largely continue to be supported through analogue.

"It's not just about supporting the skilled human decision makers on-site, but also about cost. Quite simply, growing businesses don't want to be paying out for IoT-enabled handling equipment when analogue gets the job done."

Smart factories are on the rise around the world, with traditional machinery beginning to be replaced with connected, Internet of Things-enabled solutions which automate many of the more predictable processes within the workplace. However, smart factories are rarely anticipated to be fully automated environments, and are instead considered to be collaborative spaces which succeed through the combined use of both digital and analogue tools, along with skilled human activity.

To find out more about the wide range of analogue equipment products available from Midland Pallet Trucks, visit <https://www.midlandpallettrucks.com/>



## **MIDLAND PALLET TRUCKS CHAMPIONS MOVING SKATES RANGE**

Midland Pallet Trucks, which holds one of the largest collections of high quality moving skates available to UK customers, is championing its extensive range of the warehouse staple to increase awareness of its unique advantages. As demand for moving skates continues to grow, Midland Pallet Trucks has announced a commitment to promoting the benefits of the equipment across a variety of industries in a bid to improve workplace productivity and safety in Britain.

Moving skates ensure the safe and secure movement and transport of heavy loads, traditionally within warehousing, manufacturing and logistics environments. The skates, which often come in sets of 2 or 4, can be strategically positioned to move pallets, machinery, and other heavy load items. While there are many different types of moving skate available, they typically come with durable nylon, polyurethane, or steel wheels to suit various loads and workplace conditions, and are operated through a simple, manual push/pull system to offer complete control over direction.

“While we know that our customers couldn’t live without moving skates, we feel that many industries simply don’t think to look at equipment such as this as a problem solver” says Midland Pallet Trucks Managing Director Phil Chesworth. “We believe that skates are somewhat of the unsung heroes of the warehouse as they’re frequently overlooked and yet offer a range of benefits that could bring tremendous value.

“Our aim is to continue expanding and promoting our vast range of moving skates, which is already one of the largest ranges in the UK, to ensure we’re always able to provide both new and existing customers with the quality equipment they need to operate safely within the workplace... whatever type of workplace that may be!”

Midland Pallet Trucks currently stocks a number of different types of moving skate, including fixed skates, turntable-style skates and steerable skates. These come in a variety of sizes, and with a load capacity of up to 60 tons. Much of the equipment is imported from Europe directly from the manufacturer, ensuring that all skates and complete skate sets are of the highest quality and bring true value to many sectors.

Midland Pallet Trucks hopes that in championing its line of moving skates, businesses in many industries will see that there are many benefits to this sort of equipment. While moving skates are typically associated with heavy loads, they can also be used to move lighter loads contained within confined spaces where traditional forklifts would struggle due to restrictions stemming from reduced turning circles. Similarly, limited load trolley jacks could easily be replaced with higher capacity moving skates.

In addition to moving skates, Midland Pallet Trucks is also a leading supplier of a wide range of other equipment, including hand pallet trucks and lift platforms.

To find out more about Midland Pallet Trucks’ extensive range of moving skates, visit <https://www.midlandpallettrucks.com/product-category/skates/>



## **MIDLAND PALLET TRUCKS LAUNCHES SEMI-ELECTRIC AERIAL WORK PLATFORM**

Midland Pallet Trucks has announced that the revolutionary SESP3.9 semi-electric aerial work platform is now available to order directly online.

Providing a more versatile alternative to standard mobile towers, the introduction of this innovative piece of equipment holds the key to improving workplace safety across a variety of settings and environments, including hospitals, schools, warehouses and shopping centres where safety is paramount.

The four scissor SESP3.9 semi electric scissor aerial work platform extends to a height of 3900mm, with a 33 second lifting time, and 30 second descent. Despite offering a safe working height of 19 feet and 4 inches, the model is remarkably compact, designed to effortlessly fit through standard doorways with a 1300mm length and 740mm width. Constructed with safety in mind, the aerial platform features 2 fixed wheels and 2 lockable swivel wheels, making it easy to manoeuvre in warehouses and other busy environments while still ensuring complete stability when in use.

In addition to aerial work platforms, Midland Pallet Trucks also stocks a wide range of other high quality manual and electric materials handling equipment including high lift pallet trucks and lift platforms.

“Mobile towers have traditionally been the go-to equipment for working at height,” says Midland Pallet Trucks Managing Director Phil Chesworth, “but some models are known to be notoriously difficult to use, which heightens risk within the workplace.

“We’ve been looking to expand our range of aerial work platforms and lifts for some time now, and the versatility of the SESP3.9, which can be adjusted at the touch of a button, makes this particular option an excellent addition to our growing collection.”

This is not the first SESP aerial work platform model to be offered by West Midlands-based Midland Pallet Trucks. It stocks the SESP3.0 mobile aerial work platform; a smaller, three scissor alternative with a maximum platform height of 3000mm for an average 5 metre safe working height.

The new addition is designed to bring all the benefits of the SESP3.0, such as efficient lifting and descent time, and an impressive 300kg lift capacity, to a larger model, allowing businesses to work safely at greater heights of up to 5.9 metres.

Aerial work platforms are designed to create safe, temporary access to areas that are otherwise inaccessible due to their height. The platforms are typically used in maintenance, construction, and warehousing, but can be used across many sectors.

To find out more, visit: <https://www.midlandpallettrucks.com>



## **COMPACT, RELIABLE AIR CONVEYORS WITH NO MOVING PARTS**

Air Conveyors, also known as air hopper fillers, are a convenient way of moving powdered or granulated products around a factory and a new range introduced by The Spray People Group (SPG) is a good example of these types of product. Manufactured by Canadian company Nexflow and sold under The Air Nozzle People sub-division of SPG, these compact conveyors offer important advantages over conventional conveyors, such as no moving parts, high reliability and easily controllable flow.

The conveyors work by converting a compressed air stream into a strong moving air current with a partial vacuum at one end and a positive air flow at the other. This allows light product to be moved through a tube system with no moving parts and no external power source other than the compressed air feed line.

They are available in a range of materials including hygienic 316 stainless steel for food grade applications and in several different sizes to fit pipework from 3/8" up to 5". Also, an XS-PC option is available as a clog resistant conveyor which is suitable for moving materials that may be prone to clogging conventional conveyors.

Typical applications for air conveyors occur in food and general manufacturing such as hopper loading, fibre tensioning, material conveying, waste / trim removal, chip removal, part transfer and filing operations. ENDS

More details at: <https://www.airnozzle.co.uk/products/air-operated-conveyors/>





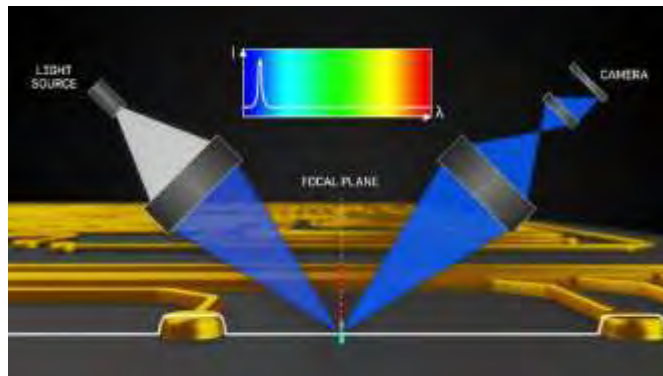
# **SENSORS / TESTING / ANALYSIS**



## **AMETEK SURFACE VISION SMARTVIEW® COLOR FUNCTIONALITY PROVIDES ADDITIONAL DEFECT DETECTION CAPABILITIES**

AMETEK Surface Vision, a world leader in automated online surface inspection solutions, now offers manufacturing industries a precise and accurate method of detecting key surface defects of interest, utilizing a new color classification option available in Surface Vision's industry-leading SmartView® defect inspection system.

The most advanced surface inspection platform available, SmartView combines powerful software, linescan camera technology, high-intensity lighting and industry-leading engineering for trusted automatic defect detection. Color classification extends SmartView's abilities by enabling the recognition of common contaminants in the production process, particularly blood, oil spots, water spots and dirt: substances that, even in minute traces, can compromise the quality of food or medical grade materials.



SmartView's color functionality works by identifying, cross-checking and classifying a specific defect through a color feature set integrated into SmartView's comprehensive defect libraries. By classifying and distinguishing each defect, users are supported in making production decisions that maintain high product quality and improve productivity. For example, as SmartView is able to distinguish water drops from oil spots, users can improve winder operation efficiency, as the ability to dry water drops later in the process means the paper does not need to be stopped at the winder.



Users get the immediate benefits of SmartView's SmartLearn functionality, which provides an advanced classification solution that classifies easily distinguishable defects, with real-time results at any production speed. Unlike systems that require the user to set the parameters manually by building a manual classification table, SmartLearn's patented automatic multi-step classification technology enables much quicker start-ups.

SmartView's color option is applicable in manufacturing light coated papers; food package grade paper; fine and light coated paper used in magazine printing and office copying; coated board for high grade packaging; and nonwoven materials such as medical cloth.

SmartView's color functionality is fully compatible with the most recent iterations of the software.

[www.ameteksurfacevision.com](http://www.ameteksurfacevision.com)





## **BTG LAUNCHES NEW CHARGE MEASUREMENT SENSOR (SPC-5500)**

BTG launches its new online charge instrument called Single Point Charge SPC-5500. It is considerably smaller, easier to install and easier to handle than previous products. Its single point modular design allows for greater flexibility and it comprises BTG's expert knowledge in charge measurement.

The SPC-5500 is suitable for wet-end applications in all papermaking processes such as in tissue, packaging and board or specialty paper. It is used to control key process stages like anionic trash levels in incoming thick stock streams, especially for closed loop control of fixatives. Furthermore, the effectiveness of functional additives dosed along the process can be evaluated through charge measurement.

The SPC-5500 continuously measures the charge of colloidal dissolved substances in process streams. Cationic demand or anionic demand is detected by automatic titration of the sample with an oppositely charged poly-electrolyte solution. Each measurement cycle follows a cleaning which is a basis for correct results.

The BTG Group is an international provider of integrated, highly specialized process solutions for the global pulp and paper industry. BTG is committed to help its customers achieve significant, sustainable gains in business performance.

<https://www.btg.com/news-and-events/btg-launches-the-next-generation-of-charge-measurement-with-the-single-point-charge-spc-5500/>





## **DATACOLOR - KEYS TO RELIABLE DIGITAL COLOR COMMUNICATION**

The challenge that we all face regardless of our business is how to get the right product to the customer at the right time when they are ready to purchase. This is especially true in the apparel business. Consumer preference changes quickly and failure to have products that fit the current trend can mean more than just a short-term loss of business.

The adoption of digital colour communication has facilitated a significant reduction in colour development cycle time, but it has also introduced challenges of its own. Failure to control the process by which the digital colour data is created will lead to:

- The exchange of incorrect information
- Misunderstanding about product quality
- Delays in the development cycle

To ensure confidence in the quality of the digital colour data that is being exchanged, the sources of variation in the measurement process must be identified and controlled.

### **Basics of Digital Colour Communication**

Multiple processes are required for successful colour development from the time that a designer selects a colour until the time that it ends up on a showroom floor. In order to have products for a particular season, the development process must start months in advance. The challenge is how to ensure that the right colour is communicated from concept to consumer.

### **Identify the Standard**

The first step in the colour specification process is to identify a standard to use for colour matching. The original source for inspiration may be a swatch of fabric or a colour created on a monitor, and this target must be communicated accurately to the mills providing the final product.

Experience bears out, however, that physical standards are often a source of misunderstanding between designers and manufacturers and can lead to delays if they're not selected carefully. While finding a good standard solves many of the problems associated with getting a good shade match, they do nothing to improve upon the colour development process as a whole. Physical standards must still be distributed and the target colour maintained throughout the development process in order to achieve the desired colour in the market.

### **Communicate the Digital Representation**

The only way to guarantee the colour integrity of the standard and to also decrease development time is to communicate the digital representation of the selected standard. What does a digital standard look like? It is simply a set of numbers called spectral reflectance values that describe how the object interacts with light. Whether we are beginning the colour process with a physical standard or a CAD design, the colour can be defined numerically in this way. Numerous computer programs have been developed that interpret these numbers and provide basic descriptions of colour and colour difference.



### **Proceed with Development**

Once the physical colour standards have been selected, they can be measured on a spectrophotometer and instantly e-mailed to suppliers who can then begin their development process. Suppliers will typically use dye formulation software to quickly produce dye recipes that have been optimized to control issues such as:

- Metamerism
- Repeatability
- Coordination with other components

### **Provide Samples to Retail Customer**

Suppliers then communicate samples to the retail customer in either physical form or in digital form. The advantage of a digital process is that samples can be quickly compared to target standards using quality control software as a filtering tool to eliminate unacceptable samples before they are given final review.

This is critical as the volume of samples typically received in any given period is substantial. Those samples that pass the numeric filtering process can then be evaluated visually using calibrated monitor technology. The samples are evaluated individually against a standard or previewed in various light sources to determine if metamerism is a problem. Prior to final approval the samples may be evaluated with other components to assess colour coordination and consistency.

Depending on the volume of development work required, the savings in time and expense realized by a retailer who employs electronic communication of standards and colour submissions can be substantial. It must be noted, however, that not all materials and colours are suitable for digital color communication and will continue to be processed using existing manual methods. Care must be taken at the beginning of the process to identify these types of materials to avoid any misinterpretation of the success of the digital colour program.

### **Ensuring Digital Colour Reliability**

The key to the success of the digital colour communication process is the reliability of the digital data being communicated during each step of the development cycle. Digital data can be deemed reliable when a different person can get the same results when the physical sample is remeasured at a different location on a different instrument. This is possible by careful control of the instrument, the measurement technique, the environment in which the samples are measured, and the methods employed by the person evaluating the data. Problems in any of these areas can introduce errors that manifest themselves as the wrong colour at the end of the development process.

What is your company doing to streamline colour development? Do you have any questions about how you can improve the process? Send us a note at [marketing@datacolor.com](mailto:marketing@datacolor.com) and let us know.

To download the free ebook ***Fundamentals of Color*** click [here](#).



## **LMI TECHNOLOGIES ACQUIRES FINNISH OPTICAL METROLOGY COMPANY FOCALSPEC**

LMI Technologies (LMI), the global leader in 3D inline scanning and inspection, is pleased to announce the acquisition of FocalSpec, an innovative optical metrology company based in Finland that designs and manufactures patented Line Confocal Imaging (LCI) products. LMI's parent company - The TKH Group - will acquire 100% of the shares of FocalSpec. The company will be integrated into the LMI group of companies and the LCI products will continue to be sold under the FocalSpec product brand.

FocalSpec LCI sensors leverage a unique optical method based on lateral chromatic confocal scanning that is mounted off-axis to simultaneously capture 3D topography (surface geometry), 2D intensity (surface contrast), and 3D tomography (scanning multiple layers beneath a transparent surface) at sampling speeds up to 27M data points per second and a measurement repeatability of 70nm. No other vision solution on the market combines this level of speed, precision, and multi-modal, multi-layer imaging capability.

The acquisition of FocalSpec expands LMI Technologies' smart sensor portfolio of laser profilers and structured light snapshots with patented confocal technologies. Together, our scanning and inspection solutions lead the industry in solving challenging applications across a variety of markets such as consumer electronics (CE), battery, pharma, semiconductor and medical.

"Line confocal sensors offer a leap in technological performance for scanning opaque, transparent and curved materials, such as hybrid glass assemblies common in cell phone manufacturing. By combining this game changing optical approach with our proven Gocator inspection software and volume manufacturing know-how, customers will be able to solve challenging inline metrology applications at a price/performance and ease of use never seen in the market today." said Terry Arden, CEO, LMI Technologies.

Sauli Törmälä, Chairman of FocalSpec: "The addition of LCI technology to the 3D product portfolio of LMI Technologies builds a highly complementary set of solutions for metrology applications in critical assembly processes. Along with their leading inspection software, we believe FocalSpec and LMI will be a power house of metrology in the years to come and look forward to joining forces."

For more information on FocalSpec LCI products, please visit [www.focalspec.com](http://www.focalspec.com). Additional information will be communicated on the LMI website at [www.lmi3D.com](http://www.lmi3D.com) in the months to come upon the successful integration of FocalSpec with the LMI group.



## **NEW CMR SENSOR FIRES IMPROVED BURNER CONTROL AND QUALITY**

The use of 1MW/unit gas burners in paper production can now be improved with the application of the new NIRIS (Near Infrared Intelligent Sensor) Natural Gas sensor from CMR Group.

The sensor, which reduces consumption levels of natural gas-powered burners used for heating, furnaces and other industrial purposes through the real time measurement of fuel quality, is directly connected to the gas feeder pipeline, boosting combustion control and providing enhanced quality control and monitoring capabilities.

The sensor is built around smart infrared hardware and data treatment software and features a CAN bus (controller area network) communications facility which enables the system to be upgradeable without dismantling the sensor for improved performance and retro applications.

Natural gas consumption levels can be further reduced by using the sensor to fine tune and calibrate burners. Other benefits include lower fuel analysis costs, correct burner performance, stable combustion, and the overall alleviation of time consuming and costly damage to components brought on by inferior or low-grade gas fuels.

CMR Group designs, manufactures and commissions automation, control system and turnkey project solutions for global industrial and renewable energy sectors, alongside specialist instrumentation for high power diesel or gas engines. More at [www.cmr-group.com](http://www.cmr-group.com)







## **TURBIDITY MEASUREMENT MADE EASY**

The new EXspect 231 NIR absorption sensor from EXNER is not only compact and robust, but also can be used in a multitude of processes and, at the same time, is easy to use.

With the EXspect 231 absorption sensor, EXNER brings a measuring device to market which can be reliably used to determine turbidity in a multitude of processes. The new type is based on the tried and tested EXspect 230 and EXspect 250 sensors. The robust stainless steel housing of the sensor has a hygienic design and is executed in protection class IP69. The measurement itself is done in the near infrared range (NIR) with a wavelength of 850 nm, which allows colour-neutral measurement. The LED light source and the detector are protected by a durable sapphire window. Cleaning by means of CIP / SIP processes is possible. For the calibration and verification of sensors in the field NIST-traceable reference filters are available.

Thanks to its hygienic and simultaneously robust design, the EXspect 231 sensor can be used for a wide variety of applications not only in the food industry, but also in the processing industry, as well as in environmental technology. Applications like the monitoring and control of separators, the determination of yeast concentrations in breweries, concentration measurement in the headbox in paper production, and the monitoring of purification processes in dairies, for example, are belonging to the areas of application.

The sensor is available in a variety of configurations and can thus be optimally adapted to suit the customer's requirements. The new EXspect 231 type replaces the previous sensor types 230 and 250 of the EXspect range.

<https://www.pressebox.de/pressemitteilung/exner-process-equipment-gmbh/Turbidity-measurement-made-easy/boxid/973299>







# MISCELLANEOUS



## **MICROBES FOR NATURAL WASTEWATER SOLUTIONS**

Bionetix® International helps solve wastewater imbalances and odour problems around the world by supplying specially targeted microorganisms and nutrients to degrade waste with greater efficiency. Bionetix® International's full line of natural wastewater treatments ranges from solutions for starting new operations, to meeting wastewater discharge limits, to making biogas plants more productive.



### **Wastewater Startup Solutions**

Starting a new wastewater operation requires “seeding” it with a healthy microbial population that can handle a heavy influx of contaminants. These beneficial microorganisms play a critical role in biodegradation by breaking down waste and essentially using it as food. If the waste outbalances the microbial population, treatment plant operators will have trouble keeping up with the overload. A shock dose of microorganisms and nutrients such as those in BCP50™ gives an important boost to the system, so it can handle the first sudden influx of waste.



For the last several years, Bionetix® International has been supplying BCP50™ to a local college in Quebec to seed the “pilot plant” students use to study wastewater treatment. The college successfully uses BCP50™ to create a new batch of sludge (or MLSS) every year to run the miniature aerobic tank for educational purposes. BCP50™ was recommended because of its use in municipal wastewater plants. It is an excellent choice for “seeding” new operations.

### **Meeting Wastewater Discharge Limits**

Wastewater contaminant levels are often measured by BOD (biological oxygen demand) and COD (chemical oxygen demand). Industrial or municipal wastewater operations must meet certain BOD and COD

limits before discharging effluent. Bioaugmentation is an excellent strategy for reducing contaminants in order to meet discharge limits and avoid extra fees. For greater effectiveness, Bionetix® doses specialized microbial and nutrient blends according to waste type. For example:

- BCP22™ targets high grease content
- BCP55™ targets high starch content
- BCP655™ targets high levels of ammonia, nitrate, and nitrite (together with organic waste)
- BCP57™ targets high cellulose content
- BCP80™ targets manure waste



In Spain, the wastewater lagoon effluent at a pig farm was marked by high levels of ammonium. A laboratory test conducted on effluent samples found that bioaugmentation with A55L™ successfully reduced ammonium levels, while BCP50™ reduced COD levels. In Mexico, a trial was done at an automotive factory using BCP50™ to maintain an 81% reduction in COD levels in the test tank. BCP50™ outperformed the global automaker's standards and was approved for use in factories around the world.

### **Increasing Biogas, Reducing Odor**

Bioaugmentation also has many side benefits related to waste treatment. In 2018, Bionetix® was able to help two biogas plants in Japan deal with inefficiency and bad odor problems. The plants dosed BCP12™ and STIMULUS™ at both locations, in addition to BCP80™ at the manure biogas plant and BCP57™ at the plant with high herbage content. The odor problem was resolved, methane production increased from 40% to the industrial norm of 60%, and power production rose from 100 kW/generator to reach full 300 kW/generator capacity.

These are just a few examples of what Bionetix® has accomplished with biological treatments that release powerful natural mechanisms. By tailoring the right microorganisms to the job, Bionetix® is able to maximize the efficiency of wastewater treatment systems and keep operations in healthy balance for a variety of applications around the globe.

Learn more about Bionetix® biological solutions for municipal wastewater treatment here:  
<http://www.bionetix-international.com/products/municipalwaste.html>



## **FLUIDMASTER DEVELOPS A HIGHLY EFFICIENT FLUSHING PROCESS**

Kipp Umwelttechnik had previously focused largely on the mechanical cleaning of heat transfer systems with different processes like the JetMaster, SpeedMaster and TubeMaster.

The FluidMaster system closes a gap in the field of closed, non-removable heat transfer systems. Kipp Umwelttechnik can now offer devices and systems for the cleaning of all heat transfer systems. Together with its sister company mycon, Kipp Umwelttechnik focuses on automation or partial automation as far as possible and economical. FluidMaster is also equipped with a PLC. The suitable cleaning program can be easily selected and started.

The system has several sensors that indicate the development of pressure and volume flow as well as the temperature and pH value of the cleaning medium. FluidMaster is constantly monitored for tightness of all connections within the system and switches itself off automatically in the event of an unexpected flow (e.g. leakage).

All recorded values are constantly recorded and stored per second or minute, depending on the setting. The course of the cleaning is thus completely visible and can be documented at any time during or after the cleaning in any desired form. FluidMaster can therefore also remain in operation for longer periods without personnel monitoring onsite.

FluidMaster works with relatively high flow velocities. For large-volume heat transfer systems, the mycon SpeedMaster system can also be used as a combination device. SpeedMaster increases the flow rate of the cleaning medium and can also introduce a second cleaning medium at the same time if required.

For cleaning smaller heat transfer systems, the Mini-FluidMaster is also available as a lightweight cleaning device.

<https://www.pressebox.de/pressemitteilung/kipp-umwelttechnik-gmbh/Kipp-Umwelttechnik-GmbH-uses-new-FluidMaster-cleaning-system-from-sister-company-mycon-GmbH/boxid/973778>







## **DUSTCONTROL LAUNCHES NEW CENTRALISED VACUUM SYSTEM**

Those in the food and drink processing, packaging and pharmaceutical industries can now benefit from the advanced range, which includes additional components to offer a fully integrated centralised vacuum system when fixed and fitted to the firm's complimenting equipment.

This means that the Good For Food range has been extended to now specifically include flap valves, suction brushes, flat nozzles, hose connectors, full stainless tubing system, joints, pre-separator, automatic shutter valves, and stainless steel filter units.

Manufactured specifically for the food industry, the equipment offers a unique combination of properties. For one, it is antistatic and ESD certified, which means it can be used in ATEX Zone 22, where dust explosions can occur.



The brushes and nozzles are also approved for food surface contact and the system for food transport, being both FDA compliant and the European equivalent.

The brush and accessories colour coding, covering five handle colours and two easily interchangeable brush colours, allows for different applications in the factory, different areas or even for different days of the week depending on need.

In addition, their material composition makes them detectable via metal detector as well as being autoclavable up to 121°C, allowing for high-pressure saturated steam cleaning.

James Miller, Managing Director of Dustcontrol UK, said: "Our Good For Food range has been developed specifically for the needs of the food industry. The range is now far more than merely brushes, with a wealth of research and development going into the design, so we're very excited to see the range evolve into a complete system."

The firm's Good For Food range is designed to offer a source extraction system that can be fully integrated into the production process for recycling, or used as a centralised vacuum cleaning system. Ultimately, it provides a flexible system where all parts are approved for food contact.

James concluded: "Through our unique suction brushes and a complete source extraction system with all materials approved for food contact, we're continuing to help those in the food industry extend their improvement of safety and hygiene standards."

Dustcontrol with its UK subsidiary based in Milton Keynes, has over 45 years of experience in designing and installing complete source extraction systems globally to fit client requirements in the food industry. They are experts in capturing dust at its source - both where and when it's created.

For further information on Dustcontrol UK's Good For Food brushes and centralised vacuum system, please call 01327 858001, or visit <http://dustcontrolfood.com/>.

Alternatively, for further information on Dustcontrol UK, visit [www.dustcontroluk.co.uk](http://www.dustcontroluk.co.uk).



## Installations

*The following pages contain a summary of the various installations and orders from around the world of papermaking, wood panel and saw mills, and bio-power generation, received between the end April 2019 and the start of November 2019.*

The Paper Industry Technical Association (PITA) is an independent organisation which operates for the general benefit of its members – both individual and corporate – dedicated to promoting and improving the technical and scientific knowledge of those working in the UK pulp and paper industry. Formed in 1960, it serves the Industry, both manufacturers and suppliers, by providing a forum for members to meet and network; it organises visits, conferences and training seminars that cover all aspects of papermaking science. It also publishes the prestigious journal *Paper Technology International* and the *PITA Annual Review*, both sent free to members, and a range of other technical publications which include conference proceedings and the acclaimed *Essential Guide to Aqueous Coating*.





COMPANY, SITE	SUPPLIER	ORDER DESCRIPTION	START-UP
Ajin P&P Dalseong Mill Korea	Valmet	A forming section rebuild introducing a novel Sleeve Roll technology for PM3 <a href="#">Valmet - Ajin P&amp;P</a>	2020
APP OKI Pulp and Paper Mill Indonesia	Valmet	Evaporation plant with integrated ash crystallization <a href="#">Valmet - OKI P&amp;P</a>	
APP Rudong Jiangsu Mill China	PMP	Eighteen tissue machines (new mill) <a href="#">Valmet - APP Rudong</a>	Mid-2020
Arauco ("MAPA" project) Chile	Andritz	Long-term maintenance and service contract <a href="#">Andritz - Aruaco MAPA</a>	Sept 2019
Arctic Paper Munkedal Mill Sweden	Adven AB	Construct and operate 30MW boiler power plant <a href="#">Adven - Arctic Paper</a>	2021
Bashida Group Heze Baishida Wood China	Valmet	Defibrator system (MDF mill) <a href="#">Valmet - Bashida Group</a>	Spring 2020
Bashida Group Jiangsu Ronghui Wood China	Valmet	Defibrator system (MDF mill) <a href="#">Valmet - Bashida Group</a>	Spring 2020
Berneck S.A. Lages Brazil	Andritz	Pressurized refining and evaporation system for their third MDF production line <a href="#">Andritz - Berneck SA</a>	end 2020
BS Energy Braunschweig Germany	Valmet	Biomass-fired boiler and a flue gas treatment plant to this combined heat and power plant <a href="#">Valmet - BS Energy</a>	early 2022
Cartiera Confalone Montoro Avellino Italy	Toscotec	Turnkey supply of a new energy-efficient tissue line. <a href="#">Toscotec - Cartiera Confalone</a>	Q4 2020
Cartiera Marchigiana Montelupone Italy	Toscotec	Dryer section rebuild (PM1) <a href="#">Toscotec - Cartiera Marchigiana</a>	H2 2019
Cartiere del Polesine Adria Mill Italy	Andritz	Rebuild of existing OCC line <a href="#">Andritz - Cartiere del Polesine</a>	Q1 2020
Cartones Ponderosa San Juan del Río Querétaro Mexico	Valmet	Quality Management System for PM1 <a href="#">Valmet - Cartones Ponderosa</a>	
Century Pulp & Paper Lalkuan District Nainital India	Mineral Technologies	Satellite PCC plant <a href="#">Mineral Technologies - Century Pulp &amp; Paper</a>	Q2 2020



COMPANY, SITE	SUPPLIER	ORDER DESCRIPTION	START-UP
Convertidor de Papel, S..A de C.V. Mexico	PMP	Tissue production line <a href="#">PMP - Convertidor</a>	Mid 2020
Crecia-Kasuga Co., Ltd Fuji Mill Japan	Valmet	Tissue production line (new mill, delivery in collaboration with Kawanoe Zoki) <a href="#">Valmet - Crecia Kasuga</a>	
Delkeskamp Nortrup Mill Germany	Valmet	DNA Automation System and IQ Quality Control System (BM1) <a href="#">Valmet - Delkeskamp</a>	
Dezhou Taiding New Material Science and Technology Co., Ltd. Dezhou Shandong China	Andritz	P-RC APMP (Pre-Conditioning Refiner Chemical Alkaline Peroxide Mechanical Pulp) fiberline with a capacity of 400 t/d <a href="#">Andritz - Dezhou</a>	Q3 2020
Faderco Warak Setif Algeria	Valmet	Tissue line, including rewinder and automation <a href="#">Valmet - Faderco</a>	June 2020
Foshan Huahan Sanitary Material Co. Ltd. China	A.Celli	Two printing machines for a diaper process line <a href="#">ACelli - Foshan</a>	Dec 2019
Gondi Group Monterrey Mexico	Pöyry	Detailed engineering services agreement for containerboard machine <a href="#">ÅF Pöyry - Gondi Group</a>	Mid 2019
Graphic Packaging International Kalamazoo Mill Michigan USA	Valmet	To supply a coated board machine to produce coated recycled board (CRB) grades (white line chip board, WLC grades) <a href="#">Valmet - Graphic Packaging</a>	H2 2022
Gulf Paper Manufacturing Mina Abdullah Paper Mill Kuwait	Toscotec	Rebuild PM1 dryer section <a href="#">Toscotec - Gulf Paper</a>	Q4 2019
Gulf Ply Dammam Mill Saudi Arabia	ABB	A mill-wide distributed control system (DCS) and quality control system (QCS) for the new paper mill <a href="#">ABB - Gulf Ply</a>	Oct 2019
Hamburger Rieger GmbH Papierfabrik Spremberg Germany	Andritz	Three paper machine approach flow systems as well as broke screening for the new greenfield production line PM2 <a href="#">Andritz - Hamburger</a>	Mid-2020
Helen Ltd Helsinki Finland	Valmet	An optimisation solution for district heat production and network in Helsinki, Finland <a href="#">Valmet - Helen Ltd</a>	Spring 2021.



COMPANY, SITE	SUPPLIER	ORDER DESCRIPTION	START-UP
Hitachi Zosen Corporation Tokushima Japan	Andritz	Fluidised bed boiler with flue gas cleaning for biomass power plant <a href="#">Andritz - Hitachi Zosen</a>	Early 2023
Ilim Group Ust-Ilimsk Mill Russia	Andritz	Recovery boiler <a href="#">Andritz - Ilim Group</a>	2021
Ilim Group Ust-Ilimsk Mill Siberia	Voith	Kraftliner machine <a href="#">Voith - Ilim Group</a>	2021
Ishikari Shinko New Energy Hatsuden Godo Kaisha Ishikari city Hokkaido Japan	Andritz	Circulating fluidised bed biomass boiler <a href="#">Andritz - Ishikari Shinko</a>	2022
ITC Bhadrachalam Pulp Mill India	Valmet	New recovery boiler and board machine rebuild (PM7) <a href="#">Valmet - ITC</a>	Late 2021
JK Paper Limited Fort Songadh Mill India	Valmet	A board machine to produce FBB, solid bleached board (SBS) and cup and barrier board grades; includes coating stations. <a href="#">Valmet - JK Paper</a>	End 2020
JSC "Yarpaper" Yaroslavl Mill Russia	Toscotec	Rebuild of PM1 (which produces test liner and fluting) <a href="#">Valmet - Yarpaper</a>	Q3 2020
Klabin ("PUMA II" project) Ortigueira Paraná Brazil	Valmet	Kraftliner machine (PM27), a new fibreline, a new continuous cooker and a pulp dryer rebuild <a href="#">Valmet - Klabin</a>	Q2 2021
Klabin ("PUMA II" project) Ortigueira Paraná Brazil	Andritz	Major pulp production technologies and key process equipment for a new pulp mill <a href="#">Andritz - Klabin</a>	Q2 2021
Kokkola Energy Oy Kokkola Industrial Park Finland	Valmet	Flue gas condensing and heat recovery equipment <a href="#">Valmet - Kokkola Energy</a>	End 2020
Kraft of Asia Paperboard & Packaging Phu My Vietnam	Valmet	Three headboxes and a press section <a href="#">Valmet - Kraft of Asia</a>	H2 2020
Maashan Huawang New Material Technology Co., Ltd Hangzhou City plant Zhejiang province China	A.Celli	Paper rewinder <a href="#">ACelli - Maashan Huawang</a>	Jan 2020
Metsä Board Joutseno Mill Finland	Valmet	TwinRoll dewatering press type VPE 1245 for bleached chemi-thermomechanical pulp production <a href="#">Valmet - Metsa Board</a>	Oct 2020



COMPANY, SITE	SUPPLIER	ORDER DESCRIPTION	START-UP
Metsä Board Oyj Husum Mill Sweden	ÅF Pöyry	Pre-engineering services for recovery boiler rebuild <a href="#">ÅF Pöyry - Metsa Board</a>	Q4 2019
Metsä Fibre Kemi Mill Finland	ÅF Pöyry	Pre-engineering assignment for new bioproduct mill <a href="#">ÅF Pöyry - Metsa Fibre</a>	H1 2020
Metsä Fibre Rauma Mill Finland	ÅF Pöyry	Pre-engineering assignment for a pine sawmill <a href="#">ÅF Pöyry - Metsa Fibre</a>	H1 2020
MG TEC Group Dej Romania	A.Celli	Two rewinders <a href="#">ACrlli - MG TEC</a>	
MG TEC Industry Dej Romania	Andritz	Two tissue production lines <a href="#">Andritz - MG TEC</a>	2020 (TM1) 2022 (TM2)
Palm Paper Aalen-Neukochen Mill Germany	Valmet	Containerboard line (PM5) <a href="#">Valmet - Palm</a>	2021
Papel San Francisco Mexico	Valmet	Tissue line <a href="#">Valmet - Papel San Francisco</a>	Q3 2020
Papelera Guipuzcoana de Zicuñaga S.A. Hernani Guipuzcoa Spain	BTG	Single point Kappa and consistency transmitters (for cellulose plant modernisation project) <a href="#">BTG - Papelera Guipuzcoana</a>	
Papierfabrik Palm Aalen-Neukochen Mill Aalen Germany	Voith	Stock preparation unit <a href="#">Voith - Palm</a>	
Papier- und Kartonfabrik Varel Germany	Voith	Rebuild PM4 with 'Papermaking 4.0' technologies <a href="#">Valmet - Papier und Kartonfabrik Varel</a>	
PRAJ Industries India	Valmet	Biomass pretreatment system for bio-refinery project <a href="#">Valmet - PRAJ</a>	2020
Saint-Gobain ADFORS Litomyšl facility Czech Republic	Andritz	Wetlaid line for the production of glass fibre mats <a href="#">Andritz - Saint-Gobain</a>	Mid-2021.
Sappi ("Project Vulindlela") Saiccor Mill South Africa	ABB	Automation, electrification and instrumentation <a href="#">ABB - Sappi</a>	Winter 2020
Shan Don Sun Honghe Paper Industry Co., Ltd China	A.Celli	Paper winder <a href="#">ACelli - Shan Don Sun</a>	July 2019
Shandong Sun Paper China	Tieto	Software - TIPS Production Planning and Trim Solutions implementation for Sun Paper's 7 mills and 14 paper machine lines <a href="#">Tieto Shandong Sun</a>	



COMPANY, SITE	SUPPLIER	ORDER DESCRIPTION	START-UP
Shandong Sun Paper Industry Joint Stock Co., Ltd. China	Valmet	Boiler diagnostics system <a href="#">Valmet - Shandong Sun Paper</a>	Q4 2019
Slovak Hygienic Paper Group Paloma Slovenia	Toscotec	Tissue production line <a href="#">Toscotec - Paloma</a>	June 2020
Smurfit Kappa Sangüesa Mill Spain	Veolia	Concentration and evaporation system <a href="#">Veolia - Smurfit Kappa</a>	
Stora Enso Oulu Mill Finland	Andritz	Rebuild of pulp fibreline and drying machine <a href="#">Andritz - Stora Enso Oulu</a>	End 2020
Stora Enso Oulu Mill Finland	Valmet	Grade conversion for PM7 (coated to packaging) <a href="#">Valmet - Stora Enso Oulu</a>	End 2020
Stora Enso Skoghall Mill Sweden	Valmet	Green liquor clarifier <a href="#">Valmet - Stora Enso Skoghall</a>	Spring 2020
Suzhou Taison Paper Jiangxi Mill China	Valmet	Automation and quality management systems <a href="#">Valmet - Suzhou Taison</a>	Q3 2019
Uni Viridas Babina Greda Croatia	Valmet	Ten-year operation and maintenance agreement (biomass power plant) <a href="#">Valmet - Uni Viridas</a>	
WestRock Três Barras Mill Brazil	Voith	Modernisation of PM3 and PM4 <a href="#">Voith - Westrock</a>	
Whakatane Mill Limited New Zealand	Valmet	A multiyear agreement for continuous performance service for Whakatane PM3 board line <a href="#">Valmet - Whakatane</a>	
Xuan Mai Paper Co. Ltd. Ho Chi Minh City Vietnam	Andritz	Tissue production line <a href="#">Andritz - Xuan Mai Paper</a>	Q2 2020
Xuzhou Zhongxing Paper (subsidiary Jianping Group) China	Valmet	DNA automation system and consistency measurements <a href="#">Valmet - Xuzhou Zhongxing</a>	Q4 2019
Zhejiang Jingxing Paper Joint Stock Co., Ltd Pinghu Zhejiang China	PMP	Hydraulic headbox, shoe press and metered size press for PM10 <a href="#">PMP - Zhejiang Jingxing</a>	Q1 2020



## Research Articles

*Most journals and magazines devoted to the paper industry contain a mixture of news, features and some technical articles. However, very few contain research items, and even fewer of these are peer-reviewed.*

*This listing contains the most recent articles from the five main journals that publish original research:*

- APPITA JOURNAL
- IPPTA JOURNAL
- J-FOR
- NORDIC PULP & PAPER RESEARCH JOURNAL
- TAPPI JOURNAL

The Paper Industry Technical Association (PITA) is an independent organisation which operates for the general benefit of its members – both individual and corporate – dedicated to promoting and improving the technical and scientific knowledge of those working in the UK pulp and paper industry. Formed in 1960, it serves the Industry, both manufacturers and suppliers, by providing a forum for members to meet and network; it organises visits, conferences and training seminars that cover all aspects of papermaking science. It also publishes the prestigious journal *Paper Technology International* and the *PITA Annual Review*, both sent free to members, and a range of other technical publications which include conference proceedings and the acclaimed *Essential Guide to Aqueous Coating*.





**APPITA JOURNAL, Vol.72, No.2, April-June 2019**

1. The Value of Wood Extractives
2. Nanocellulose, Starch and Paper Strength
3. Colloidal stability of Pinus radiata wood extractives. Part 2: competing interactions between wood extractives and process variables determined from regression modelling
4. Study on catalytic ozonation degradation of guaiacol with cupric oxides loaded on activated carbon

**APPITA JOURNAL, Vol.72, No.3, July-September 2019**

1. The age of fibre
2. To Abbreviate or Not
3. Wood extractives recovery from flotation of thermo-mechanical pulp process water
4. Evaluating the lignin content in the fibre line of a birch kraft pulp mill with a TDS sensor
5. Suitability of banana stem pulp as replacement of softwood pulp for making superior grade unbleached paper from agro residue pulp

**IPPTA JOURNAL, Vol.30, No.4, October-December 2018**

1. An Innovative & Green approach for Synthesis of optical brightening agents and their applications in Paper Industry
2. An Innovative Strategy and Pilot Scale Study to Achieve ZSD (Zero Solid Waste Dumping) at Genus Paper 7 Board Ltd applying Extrusion Technology to Convert Plastics and Rice Husk Ash into Valuable Commodity
3. Blending of Melia Dibia with Eucalyptus Tereticornis and Casuarina for Evaluation of Pulp and Paper Making Potential
4. Burning Issue – Earning Solution
5. Controlling of Odour Issues in Packaging Industries
6. Converting a Mill from Writing & Printing to Packaging Grades – a Case Study
7. Improvement in strength properties of packaging paperboard using biopolymer chitosan following a green approach
8. Innovative solutions for sustainable paper packaging
9. Low Carbon Technology Roadmap for Paper Sector
10. Paper Odour Problem – Maxim's Innovative Solutions
11. Productivity Improvements in Packaging Board by Overcoming bottlenecks and Technology upgradation – a case study by M/s.JK Paper Unit: CPM
12. Sustainable Coating solutions for Paper & Paper board

**IPPTA JOURNAL, Vol.31, No.1, January-March 2019**

1. Advancement & Digitalization in Cooling Water Treatment to have Informed Decision making & Avoiding Microbiological fouling to improve the asset life and Quality in reverse Osmosis Membranes
2. Artificial Intelligence for Wet-end optimization
3. Controlling Microbiological Growth and Foul Odors in Pulp & Paper Industry
4. Development of More Sustainable Barrier Technology for Packaging
5. FibreLean MFC – Cost Saving through Innovative Product
6. Handling Mineral Deposits in Pulp Bleaching & Recovery
7. Innovative Solution to Address Cracking Problem in Kraft Paper
8. Production, Brightness and Environmental Dynamics in modern Pulp making
9. Recent Trends & Industry Challenges in Coated Paper & Board market
10. Strength, odour, and hygiene – in the face of increased re-use of fibre and water



11. Wastewater and waste disposal management solution through innovative solutions like sludge pre-Treatment, TSS control by IoT and Polymer mixing Technology to improve dryness of Sludge
12. Water based coating solutions: a sustainable alternative to plastic

**J-FOR, Vol.7, No.5, 2018**

1. Surface Addition of Micro-nano Fibrillated Cellulose on TMP Paper by Wet-on-wet Curtain Coating on a Pilot Paper Machine
2. Process Parameter Optimization for Multi Fuel Fired Lime Mud Reburning Kiln Operation by Taguchi Method
3. Short-column Ion Exchange for Precipitator Dust Treatment: A Summary of Experience in Chloride Removal and an Introduction to Potassium Removal Capability
4. Paper Quality Control
5. Experiments on the Vertical Velocity Distribution of Black Liquor Spray in the Furnace

**NORDIC PULP & PAPER RESEARCH JOURNAL, Vol.34 No.2, June 2019**

1. Bleaching: Pre-bleaching of kraft acacia pulp
2. Paper technology: Effect of chemical additives on softness components of hygiene paper
3. Paper technology: Length-based hydrodynamic fractionation of highly networked fibers in a mini-channel
4. Recycling: Nano-lignocellulose from recycled fibres in coatings from aqueous and ethanolic media: effect of residual lignin on wetting and offset printing quality
5. Recycling: The influence of laccase/histidine system on the properties of OCC pulp fibers, and of handsheets made thereof
6. Recycling: Recovery of recycled paper in the removal of the textile dye basic yellow 28: characterization and adsorption studies
7. Lignin: Lignin-based adhesive crosslinked by furfuryl alcohol–glyoxal and epoxy resins

**NORDIC PULP & PAPER RESEARCH JOURNAL, Vol.34 No.3, September 2019**

1. Biorefinery: The influence of bio-fibers from different pulping processes on the pulp-poly-lactic acid composites (PPCs) properties from sugarcane bagasse
2. Chemical pulping: Deeper insight into the morphological features of sunflower stalk as Biorefining criteria for sustainable production
3. Chemical pulping: Optimization of the microstructure of carbonized lime mud by sodium polyacrylate
4. Bleaching: Improvement in selectivity of ozone bleaching using DTPA as carbohydrate protector for wheat straw pulp
5. Bleaching: Fiber structures and properties of eucalyptus kraft pulp via different bleaching methods
6. Paper technology: On the modeling of tensile index from larger data sets
7. Paper chemistry: The correlation between the water retention values of fibers by the centrifugation method and maximum content of fiber bonding water by the headspace GC method
8. Paper chemistry: Preparation of carboxymethyl cellulose from tea stalk and its use as a paper-strengthening agent
9. Paper chemistry: Evaluation of the adhesion performance of latex-starch mixtures to calcium carbonate surfaces



10. Paper chemistry: Determination of low molecular weight chlorinated organic compounds in polyamideaniline epichlorohydrin solution
11. Coating: Use of spherical silica particles to improve the barrier performance of coated paper
12. Coating: Mixing of oxidized starch and polyvinyl alcohol for surface sizing of paper
13. Environmental impact: Experiment on pretreatment of waste water from bamboo heat treatment by combination of iron-carbon micro-electrolysis and Fenton method
14. Recycling: Circular action treatment (CAT): a new strategy for mechanical treatment of old corrugated container III
15. Papermaking: Numerical investigation of upstream cylinder flow and characterization of forming fabrics

#### **TAPPI JOURNAL, April 2019**

1. Guest Editorial: Addressing nanocellulose commercialization needs: R&D collaboration is vital
2. Characterization of the redispersibility of cellulose nanocrystals by particle size analysis using dynamic light scattering
3. From biorefineries to bioproducts: conversion of pretreated pulp from biorefining streams to lignocellulose nanofibers
4. Priorities for development of standard test methods to support the commercialization of cellulose nanomaterials
5. Nanocellulose in Japan: An industrial perspective

#### **TAPPI JOURNAL, May 2019**

1. Editorial: Nanocellulose: What's next?
2. Research needs for nanocellulose commercialization and applications
3. Sources, collection, and handling of noncondensable gases in modern kraft pulp mills
4. Nanocellulose: Market perspectives
5. A novel approach for determining the reactivity of dissolving pulp based on the COD method
6. Prehydrolysis kraft pulping of jute cutting and caddis mixture for rayon production
7. Effects of preincubation on the gelatinization of cassava and corn starch suspensions containing sodium hydroxide as a main component of corrugating adhesives

#### **TAPPI JOURNAL, June 2019**

1. Editorial: TAPPI Journal reinstates the "Technical Brief" short-form research paper
2. Fundamental molecular characterization and comparison of the O, D0, and E stage effluents from hardwood pulp bleaching
3. The sticky behavior of pulp and paper mill biosludge during drying
4. The evolution of reel statistical methods
5. Fabrication of cross-linked starch-based nanofibrous mat with optimized diameter
6. Large data set analysis to determine refiner plate total cost of ownership

#### **TAPPI JOURNAL, July 2019**

1. Editorial: Let's talk tissue
2. Kraft pulp bleaching with a P-stage catalyzed by both bicarbonate and TAED
3. Measurement of the dynamics of fluid sorption for tissue papers
4. Enhancement of processability, surface, and mechanical properties of paper based on rice straw pulp using biopolymers for packaging applications



5. Citrus-based hydrocolloids: A water retention aid and rheology modifier for paper coatings

**TAPPI JOURNAL, August 2019**

1. Editorial: Foam forming: Technology of many opportunities
2. Upscaling of foam forming technology for pilot scale
3. Polyvinyl alcohol as foaming agent in foam formed paper
4. Real-time monitoring of bubble size distribution in a foam forming process
5. Progress in foam forming technology

**TAPPI JOURNAL, September 2019**

1. Editorial: A preview of PEERS 2019
2. Rheological characteristics of platy kaolin
3. Critical parameters for tall oil separation I: The importance of ration of fatty acids to rosin acids
4. Flow characteristics of drag-reducing natural bamboo fiber suspensions with minimal environmental load
5. A new technique for the measurement of show-through mottle of fine paper



## Technical Abstracts

*The general peer-reviewed scientific and engineering press consists of several thousand journals, conference proceedings and books published annually. In among the multitude of articles, presentations and chapters is a small but select number of items that relate to papermaking, environmental and waste processing, packaging, moulded pulp and wood panel manufacture. The abstracts contained in this report show the most recently published items likely to prove of interest to our readership, arranged as follows:*

Page 2	Coating
Page 3	Energy / Environment
Page 5	Enzymes Moulded Pulp
Page 6	Nano-Science
Page 8	Novel Products
Page 9	Packaging Technology
Page 10	Papermaking
Page 12	Pulp / Pulping
Page 14	Recycling Testing
Page 16	Waste Treatment
Page 17	Wood Panel

The Paper Industry Technical Association (PITA) is an independent organisation which operates for the general benefit of its members – both individual and corporate – dedicated to promoting and improving the technical and scientific knowledge of those working in the UK pulp and paper industry. Formed in 1960, it serves the Industry, both manufacturers and suppliers, by providing a forum for members to meet and network; it organises visits, conferences and training seminars that cover all aspects of papermaking science. It also publishes the prestigious journal *Paper Technology International* and the *PITA Annual Review*, both sent free to members, and a range of other technical publications which include conference proceedings and the acclaimed *Essential Guide to Aqueous Coating*.



## COATING

**Application of Some Polymer latexes in Preventing Paper Documents Forgery, *Egyptian Journal of Chemistry*, 62 (1), 2019, pp.1-14.** The main objective of this research is to improve ink printability of papers via their modification with various types of water-based polymer latexes during paper manufacturing process. In order to achieve this target, various types of styrene-acrylate polymer latexes were used for modification of different types of papers. Styrene-acrylate latexes were prepared via emulsion polymerization of styrene with various acrylate monomers using emulsion polymerization technique. The effect of different emulsifying agents on the properties of the prepared emulsions was studied. The obtained results showed a significant improvement in degree of gloss and ink density on the polymer-coated papers which positively affect the ink ability, and ink holding properties of the modified paper. In addition, ink gloss and ink density of the coated papers improved with increasing the concentration of modifying latexes containing Texapon®P as emulsifier. These promising results open the way to use polymer latexes in treatment of documented value which can be used in stabilization of disappearing ink on paper leading to prevent forgery.

**Water vapour and grease resistance properties of paper coating based starch-bentonite clay Khairuddin et al, *Journal of Physics: Conference Series*, 1153, conference 1.** In the present study, the water vapour and grease resistance properties of paper coating based starch – bentonite clay has been investigated. The clay concentration was 0%, 10%, 15%, 23%, 30%, 40% and 50% w/w. The composites were prepared by solution casting and coated on the surface of the paper, and analysed using Payne cup method (water vapour transmitter rate-WVTR), grease resistance test and X-Ray diffraction (XRD). The WVTR results showed that the addition of clay improved water vapour barrier properties and the most optimal improvement was obtained at clay concentration in the range of 10 - 23 wt %. However, the opposite trend was observed for grease resistance properties which showed that the grease resistance decreased with increasing clay concentration. XRD results showed that starch entered clay gallery and formed intercalated bilayer structure both at clay concentration of 10 and 23 wt % wt. The clay/starch composite (23 wt%) was more ordered and higher amount of starch chains entered gallery than those of clay/starch (10 wt%) supporting better water barrier properties at high clay concentration.

**Effect of coating with novel bio nanocomposites of cationic starch/cellulose nanocrystals on the fundamental properties of the packaging paper, Khashayar Vaezi et al, *Polymer Testing*, 80, 2019, online.** In this research, an environmentally friendly and biodegradable cationic starch (CS)/nanocrystalline cellulose (NCC) nanocomposite coating was produced and it was applied to improve the mechanical, barrier and physical properties of packaging kraft paper via surface coating. The mechanical and barrier properties of the paper were affected by the CS/NCC nanocomposite coating and it was the subject of investigation. CS/NCC nanocomposites were prepared by the solvent method. Structural and morphological properties of the nanocomposite were studied by FESEM and TEM observation. Results showed that the increased NCC nanoparticles loading, increased the tensile strength, oil resistance and air resistance of the coated paper. Also, the water absorption of the coated paper decreased 50% at 5 wt% NCC concentration. In our study it was concluded that the optimized amount for the NCC nanoparticles (among, 3 wt%, 5 wt%, and 7 wt%), is 5 wt%. The CS/NCC nanocomposite coatings are then proposed as a favorable booster which intensifies the mechanical and barrier properties of the paper in the food packaging application.





**Influence of Nano-silica on Inkjet Paper Coating, Huanmei Wang et al, *Advances in Graphic Communication, Printing and Packaging*, Springer Verlag, pp.689-696.** The influence of nano-silica with different particle sizes on inkjet paper coating was investigated. Laboratory self-made silica sol with particle size of 16 and 100 nm was employed as the pigment, and Polyvinyl Alcohol (PVA) was used as the binder. In order to study how nano-silica influences the properties of the inkjet paper coating, four groups of coating were prepared by variation of the dosage for two types of silica sol particle sizes. The viscosity of coating, the microstructure, physical properties, inkjet printing quality and dynamic permeability were characterized. The results showed that 16 nm silica sol could increase the viscosity of coating and reduce coating liquidity. Meanwhile, it did not contribute to the improvement of the physical properties and permeability of the coated paper. When the ratio between 16 and 100 nm silica sol was 30:70, the coated paper exhibited the best glossiness, smoothness and the solid density. In addition, it was found that the microstructure of the coating demonstrated good correlation with the performance of the coated paper.

**Characterization of the Interface Between Coating and Fibrous Layers of Paper, H. Aslannejad et al, *Transport in Porous Media*, 127 (1), pp.143–155.** Coated paper is an example of a multi-layer porous medium, involving a coating layer along the two surfaces of the paper and a fibrous layer in the interior of the paper. The interface between these two media needs to be characterized in order to develop relevant modeling tools. After careful cutting of the paper, a cross section was imaged using focused ion beam scanning electron microscopy. The resulting image was analyzed to characterize the coating layer and its transition to the fibrous layer. Such image analysis showed that the coating layer thickness is highly variable, with a significant fraction of it being thinner than a minimum thickness required to keep ink from invading into the fibrous layer. The overall structure of the coating and fibrous layers observed in this analysis provide insights into how the system should be modeled, with the resulting conclusion pointing to a specific kind of multi-scale modeling approach.

## **ENERGY / ENVIRONMENT**

**Exploring external and internal pressures on the environmental behavior of paper enterprises in China: A qualitative study, Zhengxia He et al, *Business Strategy and the Environment*, 28 (6), pp.951-969.** As one of the typical high-polluting and high-energy-consuming industries in China, the paper industry's environmental behavior has become the focus of a range of stakeholders, policy makers, and the whole society because the industry's business activities are a main source of environmental pollution and contribute to massive energy consumption. This study used a qualitative approach to examine the relative importance of external and internal pressures (EP and IP) in driving the environmental behavior of paper enterprises in China. Based on grounded theory, this study aimed to examine the EP and IP on the environmental behavior of paper enterprises to create a comprehensive theoretical model based on grounded theory code analysis. It was found that government pressure, economic pressure, social pressure, and IP have direct and significant positive effects on the corporate environmental behavior (CEB) of paper enterprises in China. Furthermore, government, economic, and social pressures have indirect and significant positive effects on CEB through other pressures. Finally, the paper concludes with a discussion of these four pressures and provides policy implications.



**Industrial verification of energy saving for the single-tier cylinder based paper drying process, Xiaobin Chen et al, *Energy*, 170, pp.261-272.** The paper drying process has the highest level of energy consumption in the pulp and paper production process. Analysis and optimization of the energy system during the paper drying process is critical for improving the energy efficiency of the entire paper mill. In the existing model for the paper drying process, the solution requires accurate boundary conditions such as the air temperature and humidity of the pocket area and the cylinder surface temperature, which are very difficult to obtain in the papermaking process. This can result in significant deviations between the model solution and the actual production process. This paper focuses on the single-tier dryer cylinder-based paper drying process that has been widely used with high-speed papermaking machines in recent years. A mathematical model is proposed based on real-time data. The verification via industrial production demonstrates that the proposed model is reliable for the paper drying process. Based on the simulation results, two optimization operations have been proposed. The energy consumption decreases from 1.51 t steam/t paper to 1.44 t steam/t paper, 4.6% of the steam and  $1.26 \times 10^6$  RMB can be saved for a medium-scale paper mill with the annual production capacity of  $10^5$  t paper.

**Life cycle analyses of alternative fibers for paper, Alice Favero et al, *Journal of Advanced Manufacturing and Processing*, 1 (3), e10023.** This study provides a quantitative and qualitative review of life cycle analyses of alternative fibers for paper production. Alternative fibers include both virgin fibers from rapidly renewable sources (hemp, flax, *Arundo donax*, bamboo, kenaf) and agricultural residues (wheat straw and bagasse). A comparison is made with conventional wood fibers, including northern and southern softwood and eucalyptus, and with recycled fiber. The evaluation characterizes the major environmental impacts of alternative fibers that have been identified in previous studies. The assessment of the literature indicates that a substantial portion of the environmental impacts of paper products is associated with the pulping and paper-making processes across all fiber types. Alternative fibers may have somewhat different pulping impacts, although the differences are generally not large in the overall impacts of the life cycle.

**Resource value flow analysis of paper-making enterprises: A Chinese case study, Zhen Li et al, *Journal of Cleaner Production*, 213, pp.577-587.** Papermaking enterprises are currently under both environmental pressure and economic pressure for sustainable development in China. Thus, the efficiency, effectiveness, and benefits of resource utilization need to be improved. High-consumption and high-pollution companies should manufacture paper using sustainable methods. This study highlights a resource value flow analysis from the circular economy perspective, developing an extension of material flow cost accounting and modifying it by accounting for environmental damage as well as economic benefits. With reference to the Plan-Do-Check-Act cycle, this specific case study was conducted to verify the comprehensive utility of resource value flow analysis by establishing decision-making prioritization according to the dualistic diagnosis of "internal resource loss–external environmental damage costs." In general, applying a resource value flow analysis can both reduce resource consumption and minimize environmental damage, enhancing the sustainable development of a process industry with limited resources.



## **ENZYMES**

**Cellulases Production and Application of Cellulases and Accessory Enzymes in Pulp and Paper Industry: A Review, Muhammad Imran et al, *PSM Biological Research*, 4 (1), 2019.** Cellulases are produced from a variety of microorganisms; include both bacteria and fungi. Solid-state fermentation, liquid-state fermentation, and fed-batch fermentation techniques are utilized for the manufacture of cellulases. Cellulases have vast applications in almost every industry and are extensively used in fabric manufacturing. Enzymes have been used in the pulp for juice making, food processing, paper manufacturing, and pharmaceutical applications. The process of recycling in the paper industry had great importance and its value increased day by day in writing and printing papers. Many manufacturing services in the paper industry were integrated. Paper mills start with wood chipping at first, followed by pulping, bleaching, papermaking, and recycling of the past consumer products. Reduction of energy by cellulase enzyme and chemicals has been used to improve the quality of the paper and help to decrease the environmental influence of pulp production. In the near future, the need for cellulases will be strongly recommended for the commercial production of biofuels and bioenergy.

## **MOULDED PULP**

**Production of Unitary Moulded Pulp Products Using Rapid-Köthen Apparatus, Monika Sikora & Dariusz Danielewicz, *BioResources*, 14 (4), pp.9781-9785.** This study shows that it is possible to manufacture moulded products from fibrous pulp at the laboratory scale using the Rapid-Köthen apparatus (found in almost every paper laboratory) and a special sieve form set. This process includes the design of elements of the mould forming sets by special software, production of these elements using a numerically controlled tool machine, the assembly of the sieve form, its installation in the Rapid-Köthen apparatus, and the forming and drying of the pulp product.

**On the drying process of molded pulp products: Experiments and numerical modelling, Didone, Mattia et al, *Drying Technology (an International Journal)*, 2019, online.** Molded pulp products are experiencing increased importance due to their environment-friendly characteristics with regards to both production and disposal. In this study, the thermoforming process for the production of molded pulp products was examined by means of two experimental campaigns, with the scope of distinguishing between the water removal due to only compression (better known as cold pressing) and the water removal under additional thermal conditions. The underlying multiphysics of the processes were identified by comparison with similar manufacturing processes found in available literature, and then implemented using a 1-dimensional finite element model in COMSOL software. This fast, simple model was able to predict the water removal for a specific set of process conditions and the results were supported by corresponding experimental observations. Within a process time of up to 15 s, temperatures ranging from 80 °C to 120 °C, and a final dryness between 40% and 65%, the simulations gave accurate estimates, with a difference in prediction down to 0.3%. The paper thus documents the first combined experimental-simulation study for thermoforming of molded pulp products.

**Moulded pulp products manufacturing with thermoforming, Mattia Didone & Guido Tosello, *Packaging Science & Technology*, 32 (1), pp.7-22.** Over the past years, eco-friendly packaging solutions such as moulded pulp have resonated with a growing number of consumers. Among all of them, the thermoformed products make use of the most recent manufacturing approach that produces high-quality, thin-walled items. However, it remains an underresearched area, and the development of an efficient and precise manufacturing process is fundamental in order to increase the implementation of sustainable packaging.



With the purpose of setting a step towards in the standardization of design and testing practices of eco-friendly packaging, this work focused on the characterization of the thermoforming process of moulded pulp products and their characteristics. Three different analyses were carried out for this purpose, covering the dewatering efficiency of the process, a quantification of the moulding geometrical accuracy, and an analysis of the internal microstructure of the parts. Experimental results and statistical analysis show that the dewatering efficiency is mainly governed by the mould's temperature while the duration of the contact time is not influential. In the second investigation, the geometrical accuracy of the mouldability of microfeatures was assessed. The process appeared to be dependently related to the pulp type employed. Finally, the internal microstructure was documented using X-ray computed tomography. The analysis shows an increase in the internal void fraction linked with an increase in the mould's temperature. The role of the water change of phase in the thermoforming process is also discussed by reference to the work conducted on impulse drying.

### **NANO-SCIENCE**

**Cellulose nanofiber (CNF) as a versatile filler for the preparation of bamboo pulp based tissue paper handsheets, Min Guan et al, *Cellulose*, 26 (4), pp.2613–2624.**

Tissue paper that is prepared from bamboo has a very promising future in the world, especially in China, thanks to the various merits of bamboo fibers. However, the water absorption behavior and mechanical properties of bamboo pulp based tissue paper need to be improved due to the inherent drawbacks of bamboo fiber, such as high stiffness, weak interaction between bamboo fibers etc. Hence, cellulose nanofibers (CNFs) were combined with bamboo fibers before the tissue paper-making process, to improve the water absorption behavior and mechanical properties of tissue paper. The hypotheses are that: (1) CNFs themselves possess large specific surface area and abundant hydroxyl groups as well, thus enhancing the hydrophilicity of tissue paper; and (2) the added CNFs can form 3D structures in tissue paper, thus providing abundant pores with uniform small size, which would facilitate the capillary effect for water absorption; and (3) more hydrogen bonds will be formed between CNF and bamboo fibers, thus improving the strength properties of tissue paper, thanks to the excellent mechanical and physical properties of CNF. The results from water absorption and tensile strength tests of bamboo handsheets indicated that the addition of CNFs can increase the water absorption capacity from 6.6 to 8.7 g/g when the CNF dosage was 10 wt% (based on the dried pulp). The water retention value of prepared bamboo fibers increased from 163 to 190% at the same CNF dosage, the tensile index increased from 18.5 to 24.5 N m/g as well. The results from the bulk and pore size analyses, FTIR, as well as SEM images of tissue paper also evidenced the conclusions above.

**Carboxymethylated cellulose nanofibrils in papermaking: influence on filler retention and paper properties, Ana F. Lourenço et al, *Cellulose*, 26 (5), pp.3489–3502.**

The papermaking industry competitiveness has been exponentially increasing. In order to improve the paper properties, processes have to be optimized in such a way that new horizons, such as the synthesis of new materials, are in sight. The present paper deals with the production of cellulose nanofibrils (CNF) from bleached *Eucalyptus* kraft pulp by carboxymethylation and TEMPO-mediated oxidation, followed by high pressure homogenisation. The main purpose of the work was to increase the filler retention and mechanical strength of printing and writing paper grades. Mineral fillers are of utmost importance in papermaking and therefore a thorough study of the CNF influence in filler-containing handsheets is mandatory. In this sense, flocculation studies revealed the extraordinary ability of CNF to flocculate calcium carbonate, which was translated into high





filler retentions in the paper matrix. Moreover, the interactions between bleached pulp, CNF, mineral fillers and common paper additives, such as cationic starch, alkenyl succinic anhydride and cationic polyacrylamide, were investigated. The results allowed concluding that, depending on the materials applied, CNF are able to promote an adequate bonding between fibres and filler aggregates, reducing the requirements for the additives. The addition of carboxymethylated or TEMPO-oxidised CNF to the fibrous matrix led to handsheets with better structural, mechanical and optical properties than those of reference handsheets (without CNF and with additives).

**Bacterial nanocellulose in papermaking, Matej Skočaj, *Cellulose*, 26 (11), pp.6477–6488.** Bacterial nanocellulose (BNC) is a unique natural nanomaterial that shares very few similarities with other natural or industrially produced nanomaterials. BNC can be produced by a variety of bacteria, as a survival aid in different ecological niches. BNC is traditionally produced by static or shaking culture methods, and the 'mother vinegar', or biofilm, is a typical example of this product after static vinegar fermentation. BNC has great potential in biomedicine, and recent studies have also demonstrated its use in the papermaking industry. It has nanoscale fiber size and large numbers of free hydroxyl groups, which ensure high inter-fiber hydrogen bonding. Thus, BNC has great potential as a reinforcing material, and is especially applicable for recycled paper and for paper made of nonwoody cellulose fiber. As well as enhancing the strength and durability of paper, modified BNC shows great potential for production of fire resistant and specialized papers. However, the biotechnological aspects of BNC need to be improved to minimize the cost of its production, and to thus make this process economically feasible.

**Isolation of lignocellulose nanofiber from recycled old corrugated container and its interaction with cationic starch–nanosilica combination to make paperboard, Seyed Mehdi Yousefhashemi et al, *Cellulose*, 26 (12), pp.7207–7221.** In recent years, many studies have been carried out on the use of cellulose nanofiber (CNF) produced from virgin fiber as a strengthening agent for improving the physical and mechanical properties of paper, while the use of CNF isolated from bleached virgin fiber is not necessary or reasonable for many recycled/impure products. In this due, novel lignocellulose nanofiber (LCNF) was produced from inexpensive recycled old corrugated container pulp by the ultra-fine grinding technique. The diameter of the resulted LCNF was in the range of 10–80 nm, while the cellulose crystallinity index and crystallite size reduced during the process to 49% and 4 nm, respectively. Regarding the chemical composition of LCNF, no significant change was observed in comparison to OCC fiber. But, an obstacle for the application of nanofibers, especially for paperboards, is dewatering problem. Accordingly, it was tried to evaluate the potential of cationic starch–anionic nanosilica combination as a drainage/retention aid to compensate for the negative effects of applying nanofibers in the pulp suspension, meanwhile the combination enhances the gains of LCNF application. The evaluation of pulp freeness showed that the addition of 3% nanofibers reduced dewatering ability about 100 ml CSF (around 33% loss). But, interaction of the nanosilica–starch system with the furnish containing LCNF not only compensated for the freeness reduction, but also caused a 32% or 57% increase in tensile index, in comparison to sample containing LCNF or control pulp respectively. Moreover, the addition of starch–nanosilica system with LCNF to pulp suspension, improved the retention of fine materials. Also, LCNF caused a reduction in thickness, bulk and bending resistance index of paperboard, while employment of the starch–nanosilica combination somehow off-set these negative effects. In addition, as a result of the cationic starch–anionic nanosilica system, the tear index was improved.



**Processing nanocellulose to bulk materials: a review, Qianqian Wang et al, *Cellulose*, 26 (13–14), pp.7585–7617.** Various types of nanocellulose have been isolated from the cellulosic feedstock. It was expected that nanocellulose could be used to replace fossil-based plastic in certain areas because it is biodegradable, biocompatible, environment-friendly, and has outstanding performance. Unlike conventional plastic processing, nanocellulose is generally isolated and processed in aqueous environments. Therefore, dewatering and drying are essential unit operations for nanocellulose processing. Different drying methods for colloidal nanocellulose suspension mediated different self-assembly behaviors and thus resulted in different nanocellulose morphology and physical properties. The most utilized techniques for nanocellulose processing, such as spinning, vacuum/pressurized filtration, solvent casting and roll to roll casting, coating and roll to roll coating, and additive manufacturing are investigated. Process parameters such as temperature, pH, ion species, concentration, and external electrical field, affect the orientation and assembly behavior of nanocellulose, which in turn influence the properties of the prepared materials. Therefore, the method for assembling nanocellulose into bulk materials in a controlled way is vital for the properties of the fabricated nanocellulose composites. Here, some of the recent advances in the processing of nanocellulose for bulk materials are reviewed.

**Nanocellulose Applications in Papermaking, Carlos Salas et al, *Production of Materials from Sustainable Biomass Resources*, pp.61-96. (Part of the *Biofuels and Biorefineries* book series, BIOBIO, volume 9).** Research on the utilization of biomass feedstocks has evolved rapidly in the past decades. Key developments include the production of materials with a more sustainable footprint than those derived from petrochemicals. Among associated materials, nanocelluloses have been produced from different sources and routes, such as high shear fibrillation and hydrolysis (chemical or enzymatic) or their combinations. The unique properties of nanocelluloses have sparked a myriad of uses including those related to the fields of oil and gas, adhesion, film formation, coating, packaging, food and composite processing. High end uses include the development of advanced lightweight materials, biosensors and energy harvesting systems; however, central to this review are uses closer to the source itself, namely fiber processing and, in particular, papermaking. In this chapter, the literature in these latter applications is discussed with emphasis on the use of nanocellulose to achieve favorable strength and barrier properties as well as in coating and paper sheet-forming.

## **NOVEL PRODUCTS**

**Lignin as a Wood-Inspired Binder Enabled Strong, Water Stable, and Biodegradable Paper for Plastic Replacement, Bo Jiang et al, *Advanced Functional Materials*, Wiley, online.** Plastic waste has been increasingly transferred from land into the ocean and has accumulated within the food chain, causing a great threat to the environment and human health, indicating that fabricating an eco-friendly and biodegradable replacement is urgent. Paper made of cellulose is attractive in terms of its favorable biodegradability, resource abundance, large manufacturing scale, and low material cost, but is usually hindered by its inferior stability against water and poor mechanical strength for plastic replacement. Here, inspired by the reinforcement principle of cellulose and lignin in natural wood, a strong and hydrostable cellulosic material is developed by integrating lignin into the cellulose. Lignin as a reinforced matrix is incorporated to the cellulose fiber scaffold by successive infiltration and mechanical hot-pressing treatments. The resulting lignin-cellulose composite exhibits an outstanding isotropic tensile strength of 200 MPa, which is significantly higher than that of conventional cellulose paper (40 MPa) and some commercial petroleum-based plastics. Additionally, the composite demonstrates a superior





wet strength of 50 MPa. Adding lignin also improves the thermostability and UV-blocking performance of cellulose paper. The demonstrated lignin-cellulose composite is biodegradable and eco-friendly with both components from natural wood, which represents a promising alternative that can potentially replace the nonbiodegradable plastics.

**Poly(lactic acid) composites reinforced with kraft pulp fibres: Production by a papermaking process and characterisation, Sónia Sousa et al, *Composites Part A: Applied Science and Manufacturing*, 121, pp.273-282.** Four different pulp fibres, representing short and long cellulose fibres and chemically modified fibres, were tested as reinforcements for poly(lactic acid) composites. A simple papermaking method was used to form the composite sheets, which were further compression moulded. The effects of morphological, chemical, and mechanical characteristics of kraft pulp fibres and their contents on the PLA composite characteristics were investigated. The incorporation of kraft pulp fibres provided composites with 16–24% and 36–39% higher tensile strength and Young's modulus values, respectively, than those of neat PLA. Unbleached pulp fibres, which had higher intrinsic strength than that of the corresponding bleached pulp, provided composites with higher tensile properties. In the tested range, the effect of the cellulose fibre length was not significant. Compared to those of neat PLA, barrier properties for water vapour and oxygen were slightly superior for composites incorporating up to 30% (wt) of kraft pulp.

#### **PACKAGING TECHNOLOGY**

**Measurement of thermal conductivity of paper and corrugated fibreboard with prediction of thermal performance for design applications, E. M. Gray-Stuart et al, *Cellulose*, 26 (9), pp.5695–5705.** Understanding heat transfer in corrugated fibreboard is important to the design of more effective packaging for industries which involve the freezing and chilling of food. In this work the thermal conductivity of papers which compose corrugated fibreboard were measured and used to validate finite element models of heat transfer in fibreboard. The results showed paper to be highly anisotropic, with thermal conductivity in the machine and cross machine directions being almost an order of magnitude larger than in the thickness direction. The finite element models showed good agreement with experimental results and demonstrated that the majority of heat transfer in corrugated fibreboard is through the fluted medium. Based on the finite element models, simple models for the prediction of the thermal performance of corrugated board were evaluated and shown to be very effective in reproducing the results of the more complex finite element methods. These simple methods can be used to perform corrugated fibreboard design calculations, and the models with and without radiation can be used to provide estimates of the lower and upper bounds of the thermal resistance for a given board design.

**An overview of paper and paper based food packaging materials: health safety and environmental concerns, Gaurav Kr Deshwal et al, *Journal of Food Science and Technology*, 56 (10), pp.4391–4403.** Pulp and paper industry is one of the major sector in every country of the globe contributing not only to Gross Domestic Product but surprisingly to environmental pollution and health hazards also. Paper and paperboard based material is the one of the earliest and largest used packaging form for food products like milk and milk based products, beverages, dry powders, confectionary, bakery products etc. owing to its eco-friendly hallmark. Various toxic chemicals like printing inks, phthalates, surfactants, bleaching agents, hydrocarbons etc. are incorporated in the paper during its development process which leaches into the food chain during paper production, food consumption and recycling through water discharges. Recycling is considered the best



option for replenishing the loss to environment but paper can be recycled maximum six to seven times and paper industry waste is very diverse in nature and composition. Various paper disposal methods like incineration, landfilling, pyrolysis and composting are available but their process optimization becomes a barrier. This review article aims at discussing in detail the use of paper and paper based packaging materials for food applications and painting a wide picture of various health and environmental issues related to the usage of paper and paper based packaging material in food industry. A brief comparison of the environmental aspects of paper production, recycling and its disposal options (incineration and land filling) had also been discussed.

**Pla-zno nanocomposite paper for antimicrobial packaging application, *Journal Polimesin*. 17 (2), pp.1-6.** Many food packages (plastic wrappers) today cannot be broken down by the environment; therefore, it is necessary to add natural substances that can make the food package decompose and be resistant to contamination with bacteria. Development of biodegradable polymers from renewable sources is highly desirable for food preservation and packaging, provided they can be effective as plastics or paper that are currently used in packaging, protecting food against microbial contamination, physical damage and chemical reactions (eg oxidation). Poly lactic acid (PLA) is one of the natural polymers produced by several bacteria that grow in crops rich in carbohydrates (such as sugar beets, corn and others). This research aims to insert ZnO nanoparticles and chitosan into a plastic layer of PLA (poly lactic acid) which can improve the antibacterial properties of the resulting packaging. The method used in making PLA-ZnO-chitosan nanocomposite is the precipitation method and the heating method. PLA-ZnO nanocomposites were obtained by varying ZnO nanoparticles 0.5% by weight, 2% by weight, and 3.5% by weight. The results obtained in SEM images show that nanoparticles are homogeneously distributed on the plastic surface. Antimicrobial tests show nanocomposites work effectively in deactivating *E. coli* and *S. aureus*. where it was found that *E. coli* was more susceptible to this type of nanocomposite, where there was a reduction of 3.4 logs to 3.5% ZnO loading in the PLA layer.

## **PAPERMAKING**

**Highly Filled Papers, on their Manufacturing, Processing, and Applications, Benjamin Dermeik et al, *Advanced Engineering Materials*, 21 (6), 1900180.** Since more than a decade ago, the research on highly filled papers, as well as paper-derived inorganic materials, has greatly intensified. As presented in this review, highly filled papers as preforms allow for the design of porous or dense, multilayered, and geometrically complex structures. These paper-derived ceramic- or metal-based materials are generated by the heat-treatment of highly filled papers. Paper-derived materials are potential materials of choice for applications in transportation, energy-generation, environmental conservation, support structures, medical uses, and electronic components. Due to the adjustability of the filler content and the good machinability of highly filled papers, paper-derived sheets or multilayers may include intricate structures and tailored gradients in phase structure or porosity. Paper-derived multilayers also may contain cast ceramic tapes or other functionalized layers, as presented in some examples. Computer-aided manufacturing processes for paper-derived materials can be supplemented by prediction models for the sintering shrinkage in order to identify optimal post-processing steps, stacking orders and orientations for highly filled paper layers within multilayer green bodies. The accuracy of established component-level sintering models can be significantly increased by microstructure models of the highly filled paper.



**Enzymatic nanocellulose in papermaking – The key role as filler flocculant and strengthening agent, Ana F.Lourenço et al, *Carbohydrate Polymers*, 224, 115200.** Nanocelluloses have been increasingly used in composites since their reduced size, high aspect ratio and stiffness confer great strength to the materials. In papermaking, it has been proved that harsh and expensive chemical pre-treatments to generate nanofibrils, such as TEMPO-mediated oxidation, are not the most favourable and therefore the use of cellulose microfibrils (CMF) have gained extra attention, especially those produced with the aid of enzymatic hydrolysis. In the present work, strategies to improve filler flocculation and the papermaking properties, by using enzymatic CMF, are provided. The CMF degree of polymerization was found to be directly related to precipitated calcium carbonate flocculation, leading to higher retentions in the fibre matrix. Besides, the paper dry and wet strengths were much improved, allowing in return the production of high-filler loaded handsheets with reduced requirements for common paper additives.

**The mechanism of alkyl ketene dimer (AKD) sizing on cellulose model films studied by sum frequency generation vibrational spectroscopy, Lei Li et al, *Cellulose*, 26 (5), pp.3415–3435.** Sum frequency generation vibrational spectroscopy (SFS) was employed to study the alkyl ketene dimer (AKD) sizing mechanism employed in the papermaking industry for hydrophobization of cellulose. The AKD was spun coat onto model cellulose films, which resulted in  $\approx 2.6$  nm thick AKD layers. The chain orientation of AKD molecules during the sizing process was measured at different temperatures. It was demonstrated that the chain orientation and conformation of AKD do not correlate with observed changes in sizing. The distribution of AKD molecules on model cellulose surfaces as a function of time and temperature was imaged via fluorescence microscopy to complement SFS measurements. It was concluded that the distribution of AKD plays a major role in the sizing effect.

**Environment-friendly packaging material: banana fiber/cowdung composite paperboard, M. Vishnuvarthanan et al, *Environmental Chemistry Letters*, 17 (3), pp.1429–1434.** Wood is the main raw material for paper production, which in turn contributes to the decrease of forest resources. There is therefore a need of finding alternate sources for the production of paper. Here we prepared paperboard from the cowdung and banana fibers by chemical pulping. Banana fibers provide cellulose fibers, and lignin is removed. Pectin was added finely to the suspended pulp for binding fibers. Hydrogen peroxide was added to improve pulp brightness. The paper was tested for physical and mechanical properties. Results show that incorporation of cowdung increased the tensile and burst strength from 1 to 5 MPa and 10 to 50 kPa. The porosity was also decreased from 5 to 1 mL/min. The efficient water absorption (COBB) value was obtained for 50% of cowdung. The barrier properties such as oxygen transmission rate and water vapor transmission rate were gradually decreased to 1000 cc/m<sup>2</sup> day atm and 5 g/m<sup>2</sup> day. The antimicrobial properties of the prepared paperboard were tested against the *Escherichia coli* and *Staphylococcus aureus*, and it showed efficient activity against both the microorganisms.

**Investigation of the effect of para-amino benzoic acid (PABA) added starch-coated chemicals on the printability properties of paper, Arif Ozcan, *Journal of Applied Biomaterials & Functional Materials*, Jan-March 2019, pp.1-5.** Paper is the most important material of the printing industry and is being improved due to the increasing needs of industry. The most important process to improve the optical and physical properties of paper is the surface coating. Paper has a smoother and opaquer surface with surface coating. In addition, brightness, whiteness, and yellowness values are improved



with surface coating. Ultraviolet (UV) light in sunlight causes changes in the structure of the paper and coating chemicals and accordingly causes yellowing. Para-amino benzoic acid (PABA), due to its chemical structure, is a UV-blocking agent used in sunscreen creams.

**CO<sub>2</sub> capture and preparation of spindle-like CaCO<sub>3</sub> crystals for papermaking using calcium carbide residue waste via an atomizing approach, Liang Ma et al, *Korean Journal of Chemical Engineering*, 36 (9), pp.1432–1440.** Spindle-like CaCO<sub>3</sub> crystals with controllable sizes for papermaking were successfully prepared using CO<sub>2</sub> (8% CO<sub>2</sub>/N<sub>2</sub> mixture gas) and calcium carbide residue (CCR) waste, a by-product of acetylene gas and polyvinyl chloride production, as the raw materials by an atomization method at room temperature. The influences of solution concentration, reaction temperature, and gas/liquid flow rate ratios on the properties of the CaCO<sub>3</sub> crystal were systematically investigated, and a possible atomization mechanism was proposed. The size of the as-prepared CaCO<sub>3</sub> crystal with pure calcite phase was turned from 4.71×4.02 μm to 1.82×1.12 μm by adjusting the reaction conditions. The application of the as-prepared CaCO<sub>3</sub> crystals from CCR waste as a filler for papermaking was explored. The R475 blue light whiteness of paper was increased from 77.3 to 80.6 with 11.4% CaCO<sub>3</sub> crystals.

**Nanocellulose in the Paper Making, Elaine Cristina Lengowski et al, *Sustainable Polymer Composites and Nanocomposites*, Springer, pp.1027-1066.** In recent times, nanotechnology, which has been one of the main novelties to be developed in the 21st century, has been applied to many sectors, particularly to various industrial sectors including forest-based industry. An output of this is the development of nanomaterials of which nanocelluloses have been studied as high technology biopolymers for application in various materials through the development of films and as reinforcement in papers. With this background, the main objective of this Chapter is to present the use of nanocellulose in the paper making. Accordingly, the Chapter presents characteristics of the most used wood in the world for pulp and paper production, main methods of obtaining cellulose in nature, process of bleaching of pulp, paper making, processes to obtain different types of nanocellulose (microfibrillar nanofiber and cellulose nanocrystals), applications of nanocellulose in the paper making through coating and films as well as by nanocellulose-reinforced pulp and the resulting effects of the use of nanocellulose in paper production. These include increased tensile and burst strengths, weight loss, improved barrier properties for oils, oxygen and moisture, better printing surface, etc. In the end, marketing aspects, possible future opportunities and finally concluding remarks are given. These briefly mention the use of nanocelluloses in papermaking presenting interesting possibilities, which offer improvements in cost-benefit, energy efficiency and biocompatibility, in addition to generating new products with uses are not available today.

## **PULP / PULPING**

**A structural fibrillation parameter from small angle X-ray scattering to quantify pulp refining, Jia Mao et al, *Cellulose*, 26 (7), pp.4265–4277.** Pulp fibrillation results from refining and is of prime importance for papermaking. Yet a structural parameter reflecting the extent of fibrillation remains elusive. In this work, we demonstrate that in refined pulps, the interfibrillar distance at water saturated state ( $L_s$ ), as derived from the interference factor from small angle X-ray scattering, structurally reflects fibrillation degree. Interestingly, the minimal  $L$  obtained at low water content is close to the crystal thickness derived from wide angle X-ray scattering. For a series of refined pulp samples, significant regressions are established between  $L_s$  and equilibrium moisture content, transmittance ( $T\%$ ), surface energy components ( $\gamma_{LW}$ ,  $\gamma_{AB}$ ), and the normalized crystallinity index ( $CrI_n$ ).





These regressions establish  $L_s$  as a unique structural parameter for quantifying the fibrillation degree and derived properties of refined pulps without the need of a multi-parameter and time-consuming analyses.

**Impact of modifying conventional chlorine dioxide stage to hot chlorine dioxide during rice straw pulp bleaching on pulp, paper and effluent characteristics, Daljeet Kaur et al, *Cellulose*, 26 (12), pp.7469–7482.** Paper industry being a mature mega-scale industry is focusing on process oriented technical modifications to reduce dependency on wood fibers and resource utilization. In this research study, conventional chlorine dioxide ( $D_0$ ) bleaching method was modified to hot chlorine dioxide ( $D_{HT}$ ) with an aim of reducing the effluent load with special concern to chlorolignin compounds. These compounds were measured by gas chromatograph equipped with ECD detector. The chlorophenols, chlorocatechols, chloroguaiacols, chlorovanillins, chlorosyringols and bromophenols were reduced by 9%, 50%, 34%, 47%, 17% and 31%, respectively under  $D_{HT}$  based sequence at same dose of chemicals. The general environmental parameters i.e. COD, BOD, TS, colour, lignin and AOX also got reduced in  $D_{HT}$  based sequence. The modification of  $D_0$  to  $D_{HT}$  was found to be effective as effluent quality was enhanced without compromising the optical and strength properties of cellulosic paper even at low dose of chlorine dioxide.

**Dewatering parameters in a screw press and their influence on the screw press outputs, Bouchaib El idrissi et al, *Chemical Engineering Research and Design*, 152, pp.300-308.** A Thune SP23 screw press dewatering parameters were studied. The dewatering efficiency was affected more by the rotational speed and the pulp properties. The counter-pressure affects dewatering near the discharge end, and it was observed to influence the outlet consistency and filtrate flow rate of Kraft, which has much longer fibres and fewer fines compared to TMP and BCTMP. The feed stock freeness and consistency are very important variables to consider in the screw press performance. The freeness reflects the degree of drainage, which is an important parameter to consider when optimising the screw press, while the feed consistency is a parameter of the fibre-fibre contact degree. The pulp properties, especially the fines content and fibre flexibility are also two very important parameters that affect the screw press performance. This study was to provide an insight of the screw press performance and to show the complex effect of the operational parameters on the dewatering characteristics. Using three different pulps, Kraft and TMP softwood fibres and a BCTMP hardwood fibres, we have shown that the fines content and fibre properties are two dominant properties that should be highly considered when operating a screw press.

**Anatomical and chemical properties of wood and their practical implications in pulp and paper production: a review, J.T.B. Riki et al, *Journal of Research in Forestry, Wildlife and Environment*, 11 (3), pp.358-368.** Wood is a highly variable and complex material that has different chemical, physical and anatomical properties that influence its commercial value. This review therefore, explains the wide variability between anatomical and chemical properties of wood and their practical implication in pulp and paper production. In papermaking, fibres are the cell elements that impart strength to the paper sheet. The function of the vessel element is to conduct water and dissolved minerals from the roots to the higher parts of the plant. Generally, lignocellulose materials from wood and non-wood plant consist of lignin, hemicelluloses, extractive and some inorganic matter. Information on the chemical composition is important in deciding the techno-commercial suitability, pulping method and paper strength of a particular wood material.



## **RECYCLING**

**Macroscopic and microscopic properties of fibers after enzymatic deinking of mixed office waste paper, Jinran Wang et al, *Cellulose*, 2019, online.** The deinking of mixed office waste paper by cellulase, M/Las (modification of laccase aspartic acid system), and C-M/Las (cellulase synergistic modification of laccase aspartic acid system) was investigated. The fiber morphology parameters, hydrogen bond patterns, cellulose crystallinity, and fiber microstructure were observed via fiber quality analyzer (FQA), Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and scanning electron microscopy (SEM). The results showed that the effective residual ink concentration of enzyme deinked pulp decreased compared with blank pulp (pulp without enzymatic treatment). The deinking efficiencies of C-M/Las, cellulase, and M/Las were 18.17%, 14.01%, and 12.31%, respectively. FQA analysis indicated that the fiber length and curl index decreased, while the fiber width slightly increased. FTIR analysis revealed that after cellulase, M/Las, and C-M/Las treatment, the content of intermolecular hydrogen bonds increased by 14.65%, 13.37%, and 19.80%, respectively. This indicates that there is a synergistic effect between cellulase and M/Las. XRD showed that the cellulose crystallinity decreased after enzymatic deinking. SEM micrographs of fibers treated with enzymes revealed that the fiber surface became rough and more fibrils appeared. The water retention value was increased after enzymatic deinking.

## **TESTING**

**Quantitative non-destructive analysis of paper fillers using ATR-FT-IR spectroscopy with PLS method, Signe Vahur et al, *Analytical and Bioanalytical Chemistry*, 411 (20), pp.5127–5138.** A quantitative non-destructive express method of determining fillers—kaolin and chalk—in paper was created using attenuated total reflectance Fourier transform infrared (ATR-FT-IR) spectroscopy in the mid-IR and far-IR region ( $3800\text{--}245\text{ cm}^{-1}$ ) combined with partial least squares (PLS) data analysis. Altogether, 30 two-component (cellulose pulp + kaolin and cellulose pulp + chalk) reference paper samples with known different filler concentrations and one reference paper sample without any fillers were prepared for calibration and validation. The reference values of filler concentrations in the prepared papers were determined by gravimetric analysis via dry ashing (for establishing accurate concentrations of fillers in paper) and ATR-FT-IR microspectroscopy (for evaluating homogeneity of the papers). Two-component (cellulose pulp + kaolin or cellulose pulp + chalk) PLS models were created with papers of different cellulose types and containing different amounts of fillers. The best model had root mean square errors of prediction (RMSEP) for determining the kaolin or chalk content in the two-component papers of 2.0 and 2.1 g/100 g, respectively. The performance indices were 90.4% and 92.9%, respectively. As a demonstration of practical applicability of the method, different papers from books, journals, etc. were analysed. It was concluded that the developed quantitative method is suitable for non-destructive express analysis of kaolin or chalk in paper.

**Moisture adsorption in palletised corrugated fibreboard cartons under shipping conditions: A CFD modelling approach, T.M.Berry et al, *Food and Bioprocess Technology*, 114, pp.43-59.** Corrugated fibreboard packages (cartons) must support considerable mechanical loads during long term transport of fresh produce in refrigerated freight containers (RFCs). Fresh produce are transported under high relative humidity to reduce fruit moisture loss and preserve quality. However, these conditions can progressively reduce carton mechanical strength over time as a result of mechano-sorptive creep. Little is known regarding the actual moisture dynamics in stacked cartons in RFCs, which is important for mechanical strength assessments. To this end, a portion of a fully





loaded RFC was investigated using a computational fluid dynamics (CFD) model, with respect to moisture transport in the air and the corrugated fibreboard. Simulations included the effects of loading, defrost cycles, fruit respiration and transpiration. Results showed relatively low moisture content gradients in fibreboards through the stacked cartons under optimal shipping conditions. However, the initial activation of the RFC considerably accelerated the development of moisture content gradients in the cartons. Additionally, the most significant factor influencing spatial moisture gradients through the cartons was heat conduction from outside through the container wall.

**A technique to quantify morphological damage of the flute profile in the midplane of corrugated fibreboard, Mohamad Aiman Jamsari et al, *Packaging Technology & Science*, 32 (5), pp.213-226.** This research presents a technique to quantify morphological damage to flutes in corrugated fibreboard (CFB). The method involves laser cutting thin samples and analysing digital images of the flute profiles. The surface profiles of creased CFB before and after laser cutting were measured using fringe projection and showed that the sample preparation does not significantly affect the flute profile. After imaging the laser cut samples, skeleton analysis was used to derive a digitised profile of the flute shape. To characterise the level of damage to the flute profile, a similarity factor (SF) was introduced to quantify the relative difference between test sample and reference flute profiles. Validation of this analysis technique was done by generating known images of flute profile with variations that include distortions that could occur to CFB. These images were then fed into the skeleton analysis, and the results were compared with the original profile. This comparison showed good agreement between the initial and skeleton-analysed flutes. A demonstration of the skeleton analysis on purposefully damaged actual CFB flute profiles shows that the SF reduces as the level of crushing increases, showing that the technique could be used to enumerate morphological damage to CFB during manufacture, conversion, and use.

**Experimental and numerical performance of corrugated fibreboard at different orientations under four-point bending test, Mohamad Aiman Jamsari et al, *Packaging Technology and Science*, 32 (11), pp.555-565.** This paper presents experimental work, finite element (FE) model, and analytical solution for predicting the four-point bending on C-flute corrugated fibreboard (CFB) when oriented at different angles. The angles of the CFB samples used in this research study were 0° (cross-machine direction) and 30°, 45°, 60°, and 90° (machine direction). The CFB was assumed as an orthotropic shell element in the FE model and was validated by comparing the bending stiffness, maximum bending force, and failure formation from the experimental test. It was found in the experiment that the 90° sample had the highest bending stiffness with the lowest maximum bending force while the 0° sample had the opposite. An interesting finding was that the 30° and 45° samples improve the bending stiffness than does 0° without significantly affecting the maximum bending force. Both the FE model and analytical solution predicted the bending stiffness trend of the board from 0° to 90° with good agreement compared with experimental results. The maximum bending force in the FE model showed reasonable agreement with the experimental findings. The failure regions on the samples showed similar patterns in both experiments and the FE model. The accurate response in the FE model justify that it is a good tool to predict the bending behaviour of CFB.



## WASTE TREATMENT

**The effect of dried paper-mill sludge on cement hydration, Jurgita Malaiškienė et al, *Journal of Thermal Analysis and Calorimetry*, 2019, online.** In the paper, the impact of paper-mill sludge dried at 75 °C (PS) on cement hydration is being analysed. The used specimens were made of CEM I 42.5 R cement and the added PS replaced 0%, 5%, 10% and 15% by mass of cement. The results of calorimetric measurements of the mixtures and the compressive strength were analysed ( $W/(C + PS) = 0.35$ ). SEM and XRD tests were carried out after 3, 14, 28 and 56 days of curing  $W/(C + PS) = 1$ . PS was found to extend the induction period of the cement hydration and delay the time of the secondary heat release effect, especially in specimens with the highest content of the PS. The total heat release (after 96 h) measurement results showed that the highest total heat evolved from specimens without the PS. The total liberated heat gradually increased with the increase in the PS content in the cement paste mixture. In the specimens with 15% of PS, the total heat release decreased by 20%, as compared to the control specimen without the additive. XRD test results revealed that the standard minerals listed below were formed in cement stone: ettringite, calcite, portlandite, and calcium aluminium silicate hydrate. SEM tests revealed significant changes in cement stone microstructure caused by the increase in the PS in cement paste. The compressive strength of specimens with PS waste was found to be considerably lower in the initial stage of hardening; however, when samples hardening time is increased up to 56 days, the difference between the values of compressive strength for specimens 5% of PS and the control specimens decreases.

**Technical Feasibility of Zero Waste for Paper and Plastic Wastes, Deepak K. Sharma et al, *Waste and Biomass Valorization*, 10 (5), pp.1355–1363.** Complete material recovery followed by recycling of paper and plastic waste streams is crucial for the success or failure of achieving zero waste targets. The highest recovery of material from paper and plastic waste streams reported in the U.S. and Europe are 85 and 73% respectively. However, this means there is still a remaining 15 and 27% of paper and plastic waste which is not recycled or reused indicating it is not possible to completely recycle all paper and plastic. Investigating the limitations that impede recovery and recycle identifies other avenues for engineering a zero-waste process. This study discusses the effects of various properties of paper and plastics on their quality and recycling rates. Furthermore, we present a thorough analysis of the estimated recovery for paper and plastic wastes in processing facilities. The results show that the recovery rate obtained from a traditional material recovery facility (MRF) is lower than that obtained from a combined MRF and a modern mixed-waste processing facility (MWPF). However, the MWPF is still being commercialized, posing a practical limitation in recycling operation. This paper focuses on delineating major technical issues underlying the limited recycle of paper and plastic as well as the limitations to collection systems. For example, in the U.S. the state-of-the-art recycling equipment used for paper had a stagnant recovery rate (~46,000 tonnes) from 2008 to 2013 of 66.4% although the amount of paper available for recycle is near 70,000 tonnes. Plastic recycling is a similar case where nearly 79% can be technically recovered due to problems associated with specific property requirements for final recycled products. Importantly, these limitations are independent of the actual market available for the recycled material.



## **WOOD PANEL**

**Gypsum-Based Boards Made from Mixtures of Waste Cellulosic Sources: Part 1. Physical and Mechanical Properties, Halil Şahin & İlkan Demir, *European Journal of Science and Technology*, Issue 16, 2019, pp.567-576.** It was realized that post-consumer waste paper, old corrugated container (OCC) and secondary fiber addition (cellulosic additives) to gypsum in panel structure negative impact on Thickness Swelling (TS) values in water. However, highest TS values of 23.32% (A6) in A-type, 12.76 (B6) in B-type and 7.79% (C6) in C-type experimental boards found at similar proportions (50:50 w/w) of gypsum and cellulosic additives while the lowest with control sample that was only 1.88%. Moreover, the boards produced by secondary fiber/gypsum mixture (C type boards) under similar ratios (w/w) were found to higher IB strength than others. The highest IB strength value of 0.60 N/mm<sup>2</sup> found for C3 board while the ratio of the secondary fiber in the mixture to be more than 20% negative effects on IB values to a certain extent. The addition of all three cellulosic sources to the gypsum structure increases the bending strength properties some level. At 10% (A2: 6.59 N/mm<sup>2</sup>) and 50% (A6: 6.44 N/mm<sup>2</sup>) proportion levels, A-type boards show higher bending strengths than the B- and C-type boards. In all manufacturing conditions and board types, the natural weathered boards have always shown lower hardness properties than counterpart control samples.

**Thermomechanical surface instability at the origin of surface fissure patterns on heated circular MDF samples, Andrea Ferrantelli et al, *Fire & Materials*, 43 (6), pp.707-716.** When a flat sample of medium density fibreboard (MDF) is exposed to radiant heat in an inert atmosphere, primary crack patterns suddenly start to appear over the entire surface before pyrolysis and any charring occurs. Contrary to common belief that crack formation is due to drying and shrinkage, it was demonstrated for square samples that this results from thermomechanical instability. In the present paper, new experimental data are presented for circular samples of the same MDF material. The sample was exposed to radiant heating at 20 or 50 kW/m<sup>2</sup>, and completely different crack patterns with independent eigenmodes were observed at the two heat fluxes. We show that the two patterns can be reproduced with a full 3-D thermomechanical surface instability model of a hot layer adhered to an elastic colder foundation in an axisymmetric domain. Analytical and numerical solutions of a simplified 2-D formulation of the same problem provide excellent qualitative agreement between observed and calculated patterns. Previous data for square samples, together with the results reported in the present paper for circular samples, confirm the validity of the model for qualitative predictions and indicate that further refinements can be made to improve its quantitative predictive capability.

**Tailoring of oxidized starch's adhesion using crosslinker and adhesion promotor for the recycling of fiberboards, Muhammad Adly Rahandi Lubis et al, *Journal of Applied Polymer Science*, 136 (38), article 47966.** The growing interest in recycling waste medium density fiberboards (MDFs) is driving the development of new adhesives that provide sufficient adhesion, and allow disintegration of the waste MDFs. Described in here is the preparation of adhesives based on oxidized starch (OS) in combination with blocked-polymeric 4-4 diphenylmethane diisocyanate (B-pMDI) as a crosslinker and poly(vinyl alcohol) (PVA) as an adhesion promotor for the recycling of waste MDFs. The –COOH groups of OS were reacted with –NCO groups of B-pMDI to form amide linkages, and the –CHO groups were reacted with –OH groups of PVA through hydrogen bonding. Further, when applied as an adhesive, the OS formed ester linkages with –OH of MDF fibers. As the results, MDF bonded with 1% B-pMDI/15% PVA/OS adhesive had an internal bonding strength of 0.13 MPa, 0.01 mg L<sup>-1</sup> of formaldehyde emission (FE), and



12% of degree of fiber disintegration. These results demonstrate that the B-pMDI/PVA/OS adhesive is a possible alternative to the current urea–formaldehyde resins for the recycling of waste MDFs into recycled MDFs without FE.

**A Modified Glue for Producing Particle Boards and Boards Based on Waste of Annual Plants, S. A. Ugryumov et al, *Polymer Science, Series D*, 12 (3), pp.251–253.**

This paper proposes a water-soluble phenol-formaldehyde resin modified by an alkyd oligomer for reducing the surface tension of an adhesive composition and improving its distribution over wood particles or particles of annual plants during the production of compressed board materials for construction purposes. It was found that the physical and mechanical characteristics of boards increase when produced with the use of the modified glue obtained with a small change in its production technology.

**A Technology for Production of Fiberboards Based on Cotton Stalks, V. E. Tsvetkov et al, *Polymer Science, Series D*, 12 (3), pp.328–330.** This paper considers a technique for producing raw materials and grinding of fiber from cotton stalks, as well as its properties. The properties of medium-density boards obtained in the laboratory using the dry process under various technological conditions are investigated. The results of comparative tests of wood fiber and cotton fiber boards are given.



## Forthcoming Events

### DITP (Slovenia)

PAGES 3-4

20th & 21st November 2019

[ditp@icp-lj.si](mailto:ditp@icp-lj.si)

[DITP 2019](#)

### TECHNOLOGIE KRING (Apeldoorn, Netherlands)

PAGES 5-6

20th & 21st November 2019

[www.technologiekring.nl](http://www.technologiekring.nl)

### PITA 'Wet End Chemistry' Course (Darwen)

27th & 28th November 2019

[info@pita.co.uk](mailto:info@pita.co.uk)

[Wet End Chemistry Flyer - November 2019.pdf](#)

### PAPEREX (New Delhi, India)

PAGES 7-8

3rd to 6th December 2019

<http://india.paperex-expo.com/Conference>

### PAPER ONE SHOW (Dubai)

13th to 15th January 2020

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4th & 5th February 2020

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[Modern Papermaking March 2020.pdf](#)

### PITA 'Introduction to Wet End Chemistry' Course (Bury)

31st March & 1st April 2020

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[Wet End Chemistry Flyer March 2020.pdf](#)

### SPECIALTY PAPERS EUROPE (Vienna, Austria)

20th to 22nd April 2020

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**PITA 'Energy Optimisation' Course (Bury)**

6th & 7th October 2020

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**PITA 'Modern Papermaking' Course (Bury)**

3rd & 4th November 2020

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**DITP – 46 ANNUAL SYMPOSIUM, 20 & 21 NOVEMBER 2019**

**20.11.2019 15.00–16.45 “DIGITALISATION CONTINUES”**

THE FUTURE OF PAPER INDUSTRY AND DIGITAL TRANSFORMATION – Stephane Duchatelle, Robert Mihalyi; ABB AG (AT)

READY FOR THE FUTURE OF PAPERMAKING WITH DUOSHAKE DIGITAL GENERATION – Benjamin Kitze; Voith Paper Rolls GmbH&Co KG (AT)

PAPER PACKAGING TESTING FOR THE GLOBALIZED MARKET – Igor Karlovits, Urška Kavčič, Gregor Lavrič; Pulp and Paper Institute (SI)

LUBRICATION GOES DIGITAL – Hans-Georg Weber; SKF Lubrication Systems Germany GmbH (DE)

**21.11.2019 9.00–10.45 “MATERIALS FOR CIRCULAR BIOECONOMY”**

PROPERTIES OF CELLULOSE FIBERS OBTAINED FROM CORN STOVER AND WHEAT STRAW – Jan Hočevar<sup>1</sup>, Janja Zule<sup>2</sup>, Primož Titan<sup>3</sup>, Jernej Iskra<sup>1,3</sup>; <sup>1</sup>University of Ljubljana, Faculty of Chemistry and Chemical Technology, <sup>2</sup>Pulp and Paper Institute, <sup>3</sup>RGa research genetics and agrochemistry, Ltd (SI)

GRAPHIC PRODUCTS MADE FROM JAPANESE KNOTWEED – Diana Gregor Svetec, Gregor Franken, Klementina Možina; University of Ljubljana, Faculty of Natural Sciences and Engineering (SI)

THE EFFECT OF NANOCELLULOSE ADDITION ON THE PROPERTIES OF WATER-BASED POLYVINYL ACETATE ADHESIVE – Barbara Šumiga<sup>1</sup>, Tea Kapun<sup>1</sup>, Boštjan Šumiga<sup>2</sup>, Matjaž Kunaver<sup>3</sup>; <sup>1</sup>Pulp and Paper Institute, <sup>2</sup>University of Ljubljana, Faculty of Natural Sciences and Engineering, <sup>3</sup>National Institute of Chemistry (SI)

EVALUATION OF THE ENVIRONMENTAL FOOTPRINT OF SUSTAINABLE, RENEWABLE MATERIAL: AN EXAMPLE OF NANOFIBRILLATED CELLULOSE – Janez Turk<sup>1</sup>, Katja Malovrh Rebec<sup>1</sup>, Primož Oven<sup>2</sup>, Ida Poljanšek<sup>2</sup>, Anja Lešek<sup>1</sup>, Davor Kvočka<sup>1</sup>; <sup>1</sup>Slovenian National Building and Civil Engineering Institute, <sup>2</sup>University of Ljubljana, Biotechnical Faculty (SI)

**POSTERS 10.45–11.30 (Coffee break + poster presentations)**

ANTIMICROBIAL ACTIVITY OF TWO-LAYER COATING WITH OIL, GREASE BARRIER AND INCORPORATED MICROCAPSULES – Daša Medvešček<sup>1</sup>, Mateja Zajc<sup>1</sup>, Aleš Palatinus<sup>2</sup>, Jelena Vasiljević<sup>3</sup>, Barbara Golja<sup>3</sup>, Boštjan Šumiga<sup>3</sup>; <sup>1</sup>Pulp and Paper Institute, <sup>2</sup>Papirnica Vevče d.o.o., <sup>3</sup>University of Ljubljana, Faculty of Natural Sciences and Engineering (SI)

DEVELOPMENT AND DESIGN OF AN INTERACTIVE STUDY MATERIAL WITH THE CONTENT OF PRINTING PROCESSES – Simona Perovšek, Deja Muck, Helena Gabrijelčič Tomc; University of Ljubljana, Faculty of Natural Sciences and Engineering (SI)



**21.11.2019 11.30–13.15 “INNOVATIONS IN FIBER TREATMENT”**

IMPROVED STRENGTH DEVELOPMENT OF UNBLEACHED SOFTWOOD KRAFT PULP IN LAB REFINING THROUGH PRIMARY FINES ADDITION – Daniel Mandlez, Lukas Jagiello, Rene Eckhart, Wolfgang Bauer; Graz University of Technology, Institute of Paper, Pulp and Fiber Technology (AT)

INTELLIGENT REFINING TO CONTRIBUTE MINIMIZED OPERATING COSTS, OPTIMAL QUALITY AND INCREASED STABILITY – SOLUTIONS AND RESULTS – Juha-Pekka Juhtanen; Valmet GesmbH (AT)

LATEST ANDRITZ TECHNOLOGIES IN SCREENING AND DEWATERING – Thomas Schiffer; Andritz AG (AT)

EXPERT SYSTEM APPROACH FOR OPTIMIZATION OF FIBRE PRODUCTION PROCESSES – Balazs David<sup>1,2</sup>, David B. DeVallance<sup>1,2</sup>, Miklos Kresz<sup>1,2</sup>, Leatitia Marrot<sup>1,2</sup>, Anna Sandak<sup>1,2</sup>, Jakub Sandak<sup>1,2</sup>, Bojan Borin<sup>3</sup>, Ema Fabjan<sup>3</sup>, Janja Zule<sup>3</sup>; <sup>1</sup>Innorennew CoE, <sup>2</sup>University of Primorska, <sup>3</sup>Pulp and Paper Institute (SI)

**21.11.2019 14.30–16.15 “EFFICIENT RESOURCE MANAGEMENT IN PRODUCTION”**  
OPERATIONAL COST REDUCTION IN A PAPER MILL – Michael Kremsner; Flowtec Industrietechnik GmbH (AT)

HEAT RECOVERY SYSTEM INCREASED EFFICIENCY FOR STARCH PLANTS – Stefan Divjak; GAW technologies GmbH (AT)

MODERN PREPARATION AND CLEANING OF RECYCLED FIBERS – Nikolaj Orasche; KWI International Environmental Treatment GmbH (AT)

THE USE OF RECYCLED MATERIALS FROM PAPER PRODUCTION FOR GEOTECHNICAL STRUCTURE: THE EU PROJECT PAPERCHAIN – Karmen Fifer Bizjak<sup>1</sup>, Justina Šepetavc<sup>2</sup>, Ana Mladenovič<sup>1</sup>, Barbara Likar<sup>1</sup>, Stanislav Lenart<sup>1</sup>; <sup>1</sup>Slovenian National Building and Civil Engineering Institute, <sup>2</sup>Vipap Videm Krško d.d. (SI)

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**INTERNATIONAL SEMINAR NOVEMBER 2019 TECHNOLOGIEKRING**

**'The paper-/board machine and paper-/board products'**

**Van der Valk Hotel Apeldoorn-De Cantharel, Van Golsteinlaan 20 7339 GT Apeldoorn  
November 20th (Wednesday afternoon) | November 21st (Thursday morning)**

**20.11.2019**

13.00 Welcome and opening by Jan Pille, Royal VNP. Young Talent and Paperschool

13.10 Setting the theme 'The paper-/board machine and paper-/board products' by the chairman of the day, Ronny Wurdiger, Deputy Technical Director/ Maintenance Manager, Mayr-Melnhof Eerbeek

**Masterclass: The theme of the June seminar was related to raw materials and stock preparations. Some topics will now presented, as a follow-up, because of actual relevance. There is attention to simulation of processes, classifying of recovered papers and fiber testing to get more understanding of the fiber potential for both virgin as well as recovered stock. The role of water and the stability of the watersystems is key in modern papermaking. A biocide free bacteiral control concept will be presented as contribution to realise this.**

13.30 Simulation Aided Process Control replaces the Papermaker's Little Black Book by Peter Fisera, CF ProcSim GmbH

13.50 Future classifying recovered paper per volume share by Peter Flieher, T.CON GmbH & Co. KG

14.20 L&W Fibertester Plus including Crill by Ad de Brouwer, ABB

14.40 Biocide-free Organic Deposit control with functional Bacteria by Pieter Steen-beek, Servophil AG

**Masterclass: Paper machine developments & Press section.**

**Papermachine developments have special attention in this seminar. The focus this time is more on the press section, both machinerie as well as felt concepts. Both measurements (sensing) to control performance and dry cleaning devices will be presented.**

15.30 Update and developments pressing by Peter Moedl and Jannes Koops, Voith

16.15 ProTect by Dominik Mai, Voith and Press felts measure instruments by Ad de Brouwer, ABB

16.45 Aoki dry cleaning of sieves by Uwe Sonntag, Petax

**Masterclass; Profile control.**

**In this block special attention is related to quality profiles measurements and matching with several converting challenges at customers side. After a starter with a masterclass presentation, papermakers will exchange related specific experiences in workshop setting. The workshop itself is in Dutch to improve knowledge / experience transfer.**



17.25 Quality measurements and controls by Antti Kunnas, Valmet Automation Business Line

17.45 Workshop Profiling in relation to process– and customer problems

18.30 Networking at the technology market

19:15 Informal dinner in first class networking atmosphere and official ceremony Technologie Kring Award November 2019

### **21.11.2019**

8:45 Opening by the chairman of the morning session, Leon Joore, Natural Fiber Application Center

#### **Masterclass: Paper Machine developments part 2.**

**In this block several topics will be presented in the area of new and/or advanced technologies like data analysis, virtual reality as service challenge, sensing & testmethods.**

9:00 Improved mill performance via advanced data analytics and advanced process controls by Hannu Latti, Valmet Technologies

9:20 Augmented reality for service, training and production by Andreas Zehnpfund, ABB

9:40 Smart Process Management – Power of Data by Annti Pirneskoski, Kemira

10:00 IGT Pick Test ISO 3783: Analysis method by Ferry Zuider-wijk, IGT

#### **Masterclass: Wet-end & Coating developments.**

**In this block there is attention to wet-end- and dry-end technologies like coating recipes to improve paper properties, like barriers etc., ending up in modern fit for products with added value.**

10:50 FiberLean MFC - Cost Saving through innovative Product by Karlheinz Hurst, Omya

11:10 New barrier products and their recyclability by Markus Blomquist, FP-pigments

11:30 Perfect contour for best functions by Maick Nielsen and Erich Kollmar, TSE Coating

11:55 Waterborne coating formulations for paper applications to substitute PE-laminates by Jan-Pieter Luyten, Topchim N.V. (Solenis)

12:15 The plastic crisis, Barrier coatings are added value, not added cost by Warwick Hudson, Bim Kemi

12:35 End of seminar and take away lunch



**PAPEREX 3rd to 6th December 2019, Pragati Maidan, New Delhi, India**

**CONFERENCE PROGRAMME**

**Day - 1 (Tuesday, 3rd December 2019) Technical Session I Paper Industry in the Coming Decade: Prospects(11.30 am to 1.30 pm)**

1. Prospects of Indian Paper Industry in the Coming decade. Dr. B.P. Thapliyal, Director; Zarka Afroz, Project Assistant; Arun Kumar, Sr. Res. Fellow; Dr. Kawaljeet Singh, Sci. E II, Central Pulp and Paper Research Institute, Saharanpur, Uttar Pradesh, India.
2. Indian Paper Industry – Key trends and challenges. Vikanshu Bhargava, Senior Manager; Ernst & Young LLP- India, New Delhi, India.
3. Paper Industry in the 2020's. Vijay Gupta, Managing Director, Founder Investments Squared Pte. Ltd., Singapore.
4. World Recovered Paper Markets. Bill Moore, President, Moore & Associates, USA.
5. Current status and challenges of the Japanese Paper Industry. Kunitaka Toyofuku; President; Toyofuku Paper Business Plan, Japan.

**DAY - 2 (Wednesday, 4th December 2019) Technical Session II: Industrial Internet and Analytical Software in Decision-making (9.30 am to 10.30 am)**

6. Assisted decision making through advanced data analytics and remote expert support. Jari Almi, Director, Industrial Internet, Valmet, Tampere, Finland and Samuli Lehtonen, Director, Operations Performance Optimization, Industrial Internet, Valmet, Tampere, Finland.
7. Field proven Mill Optimization of process performance via cutting edge digital IIoT Technologies. Ankur Mehrotra, Regional Manager – South East Asia, ANDRITZ Process Optimization, Chennai, India.

**Technical Session III: Innovations and New Developments (11.00 am to 1.30 pm)**

8. Why Asian Pulp Producers are investing in Ozone Bleaching. Alexis Métais, Xylem, Germany and Brendan van Wyk; Business Development Manager Pulp & Paper; Xylem Water Solutions South Africa (Pty) Ltd, South Africa.
9. Advanced New Technology Energy Efficient Pumping Systems for Paper Plants. Prabhakaran Iyer, Director India; Vinod Narkhede, Senior Manager, Advance Pumping solutions; Venkatesh R., Sen. Eng.; Rod Wheel Technologies Private Limited, Chennai, India.
10. A status review of utilization of proven Advance Process Control technology in Pulp & Paper Industry. Adam Melton, Global Sales Director, Process Solutions, BTG Group, USA and Akhlesh Mathur, ASPAC Fiber Segment & SEA Sales Director, BTG Group, Singapore.
11. Paper pigmentation using a metered size press. Andrew Findlay, Janet Preston, Eli Gaskin and Matthew Duggan; Imerys Minerals Ltd., U.K.
12. Lighter paper and board materials through recent advances in the application of dry strength technology. Dr. Jan-Luiken Hemmes, Head of global Business Development Strength Additives, Jonas Konn, Vladimir Grigoriev, Mikko Virtanen, Kemira Chemicals GmbH, Frankfurt, Germany.

**Technical Session IV: Wood & Agro-based Papermaking (2.30pm to 5.00pm)**

13. High Power Recovery Boilers in India. Pekka Rikkinen, Director, Recovery Boilers and NCG, P&E Recovery, Valmet, Finland and Kari Haaga, Product Manager, P&E Recovery Boilers and NCG Boiler Solutions, Valmet, Finland.



14. Potential growth challenges of Wood-based Paper Mills in India in the Coming Decade. P.K.Suri, Executive Vice President (Works), G.S.Patnaik, D.K.Tripathy and R.S.Patnaik, JK Paper Ltd., Unit: JK Paper Mills, Jaykaypur, Dist: Rayagada, Odisha, India.
15. Bagasse-based paper-making in the coming decade. Ganesh Bhadti, Vice President (Technical), Seshasayee Paper and Boards Limited, Unit Erode; Tamilnadu, India.
16. Solution to Raw Material Crisis in Wood Based Pulp and Paper Mills: Casuarina Clonal Technology. Dr. P. Sankaralingam and Dr. S. Subramanian; Tamilnadu Newsprint and Papers Limited, Kagithapuram, Karur, Tamilnadu, India.
17. Improvement in Pulpwood Qualities through Focused Plantation Research & Development. N. K. Khanna, O. P. Shukla, Chief General Manager (RM), S. L. Narkhede and S.K.S. Chauhan; Plantation Division - Clonal Production and R&D complex; JK Paper Limited, Unit- CPM, Fort Songadh, Gujarat, India.

**Day - 3 (Thursday, 5th December 2019) Technical Session V: Nano Materials (9.30 am to 11.00 am)**

18. Impact of Synthetic Nano Materials on Economy, Energy and Environment in Paper and Board Industry. Dr. Vijay Mathur, Chairman and Chief Technical Officer, Pacific Nano Products, USA.
19. Microfibrillated cellulose: An innovative guide for the future bioproducts. Dr. Seema Saini, Research Engineer; Jalila Bachiri, Technical Centre Manager and Alain Lascar, Director Technology & Innovations; Kadant Lamort, Vitry-le- François, France.
20. Innovative instruments that supports the MFC production and industrialization. Didier Rech, TECHPAP SAS, Grenoble, France.

**Technical Session VI: Packaging and Board-making (11.30pm to 1.30pm)**

21. Tailor-made Stretchable Papers and Deriving Composite Materials in the Packaging Industry in the Coming Decade. Mrs. Marion Sterner, Head of R&D Gruppo X di X Gruppo srl, Italy
22. Innovative cellulosic materials for Packaging application. D. Guérin, P. Martinez, C. L'ocre, F. Vercelli; Director of Customers Relations; Centre Technique du Papier, Grenoble, France.
23. Finishing aspects of paperboard - cracking at the fold, varnish demand and laser marking. Janet Preston, Tony Hiorns, Michael Wasser and Andy Findlay; Imerys Minerals Ltd., U.K.

**Technical Session VII: Minerals and Printing (2.30 pm to 3.30 pm)**

24. Innovations in PCC Applications to Support Sustainability. Amit Dholakiya, Yashika Saxena, Ishan Dholakiya, Sidharth Weley, Lotta Hirvikoski, Specialty Minerals Inc (SMI)- Business Segment of Minerals Technologies Inc (MTI), USA.
25. Investigative Study on Paper Coating Binders and its Effect on Print Products. Kiran P. Prayagi; pmIndia-Graphic Arts Technology & Education; Consultant – EOC Tailor Made Polymers India (P) Ltd, Mumbai, India.

<http://india.paperex-expo.com/Home>





## **Premier of ZELLCHEMING-Conference held prior to the ZELLCHEMING-Expo**

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Just before the start of the ZELLCHEMING-Expo, taking place from 24 - 25 June 2020 in Frankfurt am Main, the ZELLCHEMING e.V. will be holding a conference in Darmstadt for the first time. The new event complements the existing ZELLCHEMING event offering a science-focused platform.

The ZELLCHEMING Conference will be held from 21 – 24 June 2020 in Darmstadt with the title "Cellulose-based Materials – from Science to Technology". The association ZELLCHEMING e.V. is cooperating with its technical committees and the Technical University of Darmstadt to provide an international knowledge platform, which will bring together researchers from science and industry interested in topics related to polysaccharides, paper-based materials and bioeconomics.

The scientific program, created by invited speakers, ranges from polysaccharide chemistry and paper chemistry to paper physics and paper technology.

The proximity to Frankfurt am Main makes it possible for the attendees to visit the ZELLCHEMING-Expo trade fair following the conference. On 24 June 2020, a coach will link the two locations so that participants can combine scientific knowledge with all the benefits of the exhibition, such as networking and gathering information on the latest trends and products.

Further information on the conference can be found at [www.zellcheming.de](http://www.zellcheming.de).

### **Key players already registered for the ZELLCHEMING-Expo**

Exhibitors along the entire paper value chain have already registered for the ZELLCHEMING-Expo, including ABB Automation GmbH, Andritz AG, Biomontan Produktions und Handels GmbH, fipptec, Kühne+Vogel GmbH, Servophil AG, Valmet and Wöllner GmbH.

### **The "Road of Maintenance" will take place once again**

After the successful premiere in 2019, the "Road of Maintenance" will be held again next year. Providers of maintenance services will have the opportunity to present their products and services to users cost-efficiently and with maximum attention.

Further information about the exhibition is available at [www.zellcheming-expo.de](http://www.zellcheming-expo.de).