Wheat Straw as Base Paper for Barrier Coating

Mohammed Ahmed,* Anke Lind, Sven Sängerlaub, and Emanuele Martorana *

A smooth and dense surface of the base paper is advantageous when the goal is to apply a liquid coating as a barrier layer. For such a base paper, non-wood fibers derived from wheat straw could be an alternative to wood fibers. In this research paper, wheat straw pulp was refined with different beating levels (up to 600 revolutions) followed by different calendering pressure loads (up to 50 N/mm) to test its influence on mechanical and surface properties. Alkyl ketene dimer (AKD) was used as sizing agent with concentrations up to 0.2 wt% followed by a mineral-based precoating to test its influence on the smoothness. Eucalyptus pulp was chosen as a benchmark. After beating, the initial Schopper-Riegler degrees of 28 °SR increased to 56 °SR. Beating also increased the tensile index from 24 to 49 Nm/g, the burst index from 1.2 to 2.8 kPa·m²/g, and the tear index decreased from 3.3 to 2.8 mN·m^2/g. Calendering reduced the initial roughness of 370 mL/min to 30 mL/min. When precoated and calendered again, the value was lowered to 15 mL/min. In summary, wheat straw paper is a relevant alternative to wood-derived base paper to produce barrier papers. Compared to eucalyptus, wheat straw paper showed better smoothness and much lower air permeability indicating excellent suitability for barrier coating.

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INTRODUCTION

A new trend in the pulp industry is the use of non-wood plants (Ferdous *et al.* 2021; Lexa *et al*. 2023) such as wheat straw (Nasser *et al*. 2015; Plazonic *et al.* 2016; Jia *et al.* 2018). Wheat straw is an available resource (Liu *et al*. 2018; Markevičiūtė and Varžinskas 2022). The utilization of non-wood fibers marks a notable trend in the industry (BRI 2023), supported by the various research and examination of the wheat straw fiber composition and capability for contribution to the pulp and paper production (Guo *et al*. 2009; Bhutto *et al*. 2010; Hart 2020). Wheat straw contains 30% to 45% cellulose, 20% to 30% hemicellulose, 15% to 20% lignin, and several minor organic compounds. Around 68 wt% of the straw (only internodes) is suitable for pulping to achieve sufficient physical and optical properties (Fang and Shen 2018).

Wheat Straw

In comparison with wood fibers (Table 1), wheat straw has a holocellulose content "… approximately equal to that of hardwood" (Singh *et al*. 2011) and less lignin (Chen and Liu 2014; Zhang *et al*. 2022). The fiber length and the cellulose ratio are similar to eucalyptus (Clarke *et al*. 2008; Vivian *et al*. 2017) and comparable to mature spruce

(Lundqvist *et al*. 2005; Tutus *et al*. 2010; Čabalová *et al*. 2021). Therefore, wheat straw is considered a short fiber with comparable characteristics to those of eucalyptus, which is a reason for its relatively high smoothness and relatively uniform sheet formation (Xu *et al.* 2006).

Table 1. Characteristics of Fibers from Different Plants

Property	Wheat Straw [a]	Eucalyptus [b]	Spruce [c]
Average Fiber Length (mm)	1.2(±0.1)	1.0(±0.1)	2.6(±0.4)
Average Fiber Width (um)	13.6(±1.7)	16.6(±2.7)	32.5(±2.5)
Wall Thickness (um)	4.0(±0.1)	3.9(±0.9)	2.7(±0.3)
Holocellulose (wt%)	72.2(±0.1)	70.3(±0.2)	74.5(±0.2)
Lignin (wt%)	21.1(±0.1)	27.1(±0.2)	25.2(±0.3)

[a] obtained from (Singh *et al.* 2011)
[b] obtained from (Vivian *et al.* 2017)
[c] obtained from (Lundqvist *et al.* 2005; Tutus *et al.* 2010)

Properties of Base Paper with Relevance for Barrier Paper

The base paper absorptivity influences the barrier performance of a coated paper (Shen *et al*. 2021). Referring to the Lucas-Washburn equation, the water absorption within paper is determined by a capillary transport mechanism as described by Eq. 1,

$$h^2 = \frac{r \cdot \Upsilon \cdot cos\Theta}{2 \cdot \eta} \cdot t \qquad (1)$$

where h is the distance (m) travelled, r is the capillarity radius (m), Υ is the liquid surface tension (N/m), Θ is the contact angle between the capillary wall and the liquid, η is the liquid viscosity, and t is the time (s) for sorption. The sorption is directly proportional to the square root of time (Washburn 1921). After calendering, the capillarity radius r is reduced; therefore less wet coating material penetrates a paper before it dries. Additionally, further mechanisms could contribute to water penetration into the substrate including liquid movement through the fibers, surface diffusion into the pores, and diffusion transport of vapor in the pores.

Air permeability is considered one of the critical parameters of barrier papers (Kjellgren and Engström 2005); hence, Darcy's law could display the correlation between the air flow through a sheet with specific thickness and the pressure difference applied by Eq. 2,

$$q = K \cdot \frac{\Delta p}{\eta \cdot Z} \qquad (2)$$

where q is air flow per unit area (m/s), K is the permeability factor (m^2), Δp is the pressure drop, η is the air viscosity (Pa·s), and Z is the sheet thickness (m). Because the permeability factor is affected by the pore volume, it could be estimated using the Kozeny-Carman equation as depicted in Eq. 3,

$$K = \frac{\varepsilon^3}{\kappa \cdot (1-\varepsilon)^2 \cdot S^2} \qquad (3)$$

where ε is the pore volume fraction, S the effective surface area per unit particle volume (m^2), and κ the Kozeny constant (≈ 5). This empirical relationship describes the influence of the pore sizes on the permeability performance through porous structures (Kjellgren

2005). The lower the pore volume ε, the smaller the permeability factor K, and the lower the air flow per unit area q. The pore volume is reduced by calendering.

Paper porosity influences the barrier and resistance properties, and the correlated factors affecting this are the porous structure, fiber swelling, wetting time, and the contact angle between the substrate surface and the liquid. Enhancing such an attribute is possible by refining the pulp to a certain beating level or by a final surface treatment by calendering lowering the porosity in the substrate (Sönmez and Özden 2018). Practically, the pores could be longer than the sheet thickness as the pores are randomly formed in many irregular shapes with twists. Thus, this could be described as tortuosity as shown in Eq. 4,

$$L = T \cdot Z \tag{4}$$

where T is the tortuosity, L the total length (m), and Z the sheet thickness (Purcell 1949). Additionally, low porosity contributes to obtaining a good barrier when a barrier coating is applied and stays on the substrate surface with or without limited penetration (Kimpimäki 1998). Furthermore, porosity influences the moisture diffusion as it penetrates through the porous structure as water vapor and through cell wall as condensed moisture (Defrenne *et al.* 2009).

Intention of the Study

Base papers for barrier applications should be smooth with a low porosity (Todorova *et al.* 2022). In this research paper, wheat straw pulp was tested as an alternative to wood fibers. The authors' hypotheses were: 1) due to its short fiber length, a smooth surface and low air permeability could be achieved making this type of non-wood fiber a prospecting nominee for barrier paper; 2) calendering reduces air permeability, and surface roughness; 3) precoating, and calendering further reduce surface roughness; and 4) properties, including mechanical properties, are similar to commonly used *Eucalyptus* pulp, which has short fibers.

EXPERIMENTAL

Materials

Commercial wheat straw pulp (J. Rettenmaier & Söhne Gmbh+Co. KG, Rosenberg, Germany) in the form of white flakes was used. The properties provided by the manufacturer were alpha cellulose content (TAPPI T203cm-99) of 77.5%, brightness (R457) of 75%, moisture content of 12%, oxide ash (850 °C, 4 h) content of 2.5% (max), pH-value (10% suspension) of 5 to 8, and bulk density (DIN EN ISO 60) of 130 g/L. For comparison, *Eucalyptus* pulp was used with properties provided by the Munich University for Applied Sciences database, which were of fiber length (Lc(l)ISO) of 0.865 mm, fiber width of 12 µm, fine particle content (Fines A) -with length and width under 0.2 mm- of 13.52%, and (Fines B) -with length over 0.2 mm and width under 10 µm- of 69.85%. All coating components were commercially available (Covercarb, Omya International AG, Oftringen Switzerland; Capim, Imerys S.A., Paris, France).

Fiber Morphology

According to DIN EN ISO 16065-2 (2014), samples were obtained from each beating level batch with the same total disintegration revolutions for each batch. The measurements were operated by the Valmet Fiber Image Analyzer (Valmet Corporation, Espoo, Finland).

Pulp Preparation

According to DIN EN ISO 5263-1 (2004), the pulp was soaked in water for 24 h prior and then transferred to the disintegrator (FRANK-PTI GmbH, Birkenau, Germany) for 30k revolutions. The pulp was thickened after the disintegration using a sheet former, reaching an approximate consistency of 27 wt%. Then it was diluted to a consistency of 10 wt% of oven dry fibers. According to DIN EN ISO 5264-2 (2011), the pulp was refined using a PFI mill S40110 (Frank-PTI, Birkenau, Germany) to 4 different beating levels (0, 200, 400, and 600 revolutions) running with 30 g oven dry with a consistency of 10 wt%.

Preparation of Handsheets

According to DIN EN ISO 5269-2 (2005), the handsheets were made using a sheet former (Rapid-Köthen S95854, Frank-PTI, Birkenau, Germany). The required basis weight of a single handsheet is approximately 60(±1.1) g/m^2. To achieve it, the required suspension was calculated where the stock consistency in the mix chest was approximately 0.4 wt% and the handsheet diameter would be 20 cm. Then, they were placed in a climate room with a temperature of 23 °C and a relative humidity of 50% according to DIN EN ISO 554 (1976).

Sizing

The sizing agent (FennoSize™ KD 544M, AKD, Kemira, Helsinki, Finland) was used to modify the hydrophobicity of the base paper had a solid content of 23.5 wt%, and the dosage would be varied to 0 wt%, 0.1 wt%, 0.15 wt%, and 0.2 wt% per oven dry pulp accordingly, and the conditions of making handsheet were the same in terms of single handsheet weight and drying temperature. The pulp suspension was mixed for 10 seconds after AKD additions, followed by immediate drainage of the suspension. For AKD curing, the handsheets were heated in the oven at 90 °C for 15 min then they were conditioned in the climate room for 1-2 days.

Calendering

The handsheets were calendered using a laboratory calender CA5/300 (Sumet Technologies GmbH & Co. KG, Denklingen, Germany) with a roll temperature at 23 °C, and a fixed speed of 10 m/min, but with variation of nip load to 30 and 50 N/mm, accordingly.

Precoating

The precoating layer role was to provide a better smoothness to limit the barrier coating weight applied on it, and to achieve a lower z-penetration of the coating, and to prevent it from migration into the base paper. The recipe that was used is displayed in Table 2. A precoating layer of 7.7 g/m^2 was applied by a Mayer rod coater (Gockel GmbH, Hamm-Rhynern, Germany) at a speed of 3 m/min.

Table 2. Precoating Formula

Product Type	Product Name	Parts
Pigment #1	Covercarb 75- GCC	70
Pigment #2	Capim NP- Clay	30
Binder	Styrene Acrylic Latex (SA)	8
Co-binder	Starch	5

Experimental Plan

To find the optimal conditions of the base paper, the strength, and the surface properties had been investigated first to identify the optimal refining and calendering settings (Fig. 1). Then it was compared with the eucalyptus pulp at the same refining and calendering settings. Later, the sizing dosages had been varied to find sufficient dosage of AKD, and these dosages had been applied to the optimal refining setting without calendaring (Fig. 2). Furthermore, the precoating had been applied on the final decided settings regarding the refining, sizing, and calendering.

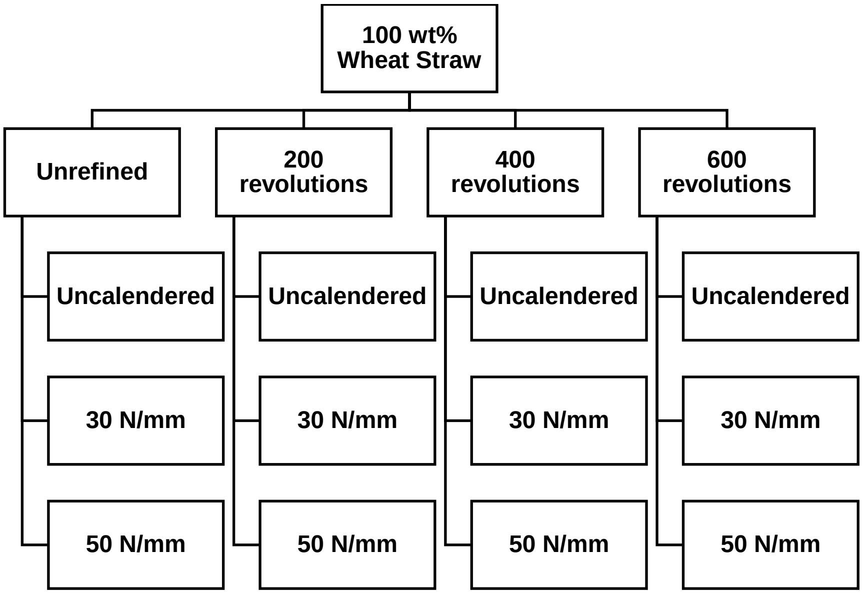

Fig. 1. Sample conditions of refining and calendering

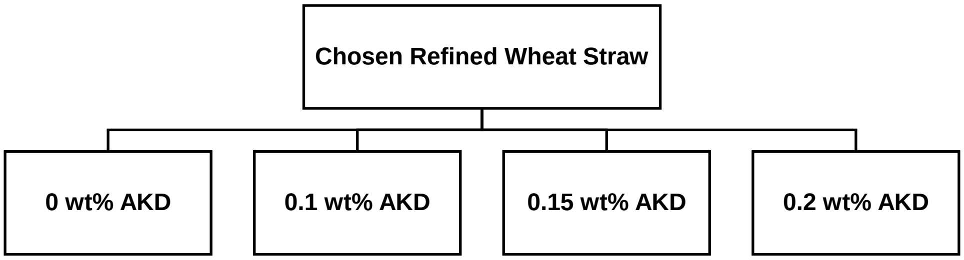

Fig. 2. Sample conditions of sizing dosage

Testing

The handsheets were sampled (DIN EN ISO 5270:2012) and then tested for paper thickness using a thickness gauge (LDAL-03, Lehmann AG Mess+Regeltechnik CH-2502 Biel, Switzerland) as per DIN EN ISO 534 (2011). Air permeability was tested using a Bendtsen tester (S624000, Frank-PTI, Birkenau, Germany) using the Bendtsen method as per DIN EN ISO 5636-3 (2013). The smoothness was measured by a Bendtsen tester (S624000, Frank-PTI, Birkenau, Germany) *via* the Bendtsen method, as per the DIN EN ISO 8791-2 (2013) standard. The tensile strength was evaluated by a universal testing machine (Zmart.Pro, ZwickRoell GmbH & Co.KG, Ulm, Germany) as per DIN EN ISO 1924-2 (2008). The burst strength was evaluated with a burst tester (S18534, Frank-PTI, Birkenau, Germany) as per DIN EN ISO 2758 (2014). The tear resistance was tested according to Elmendorf method (Digi-Tear ME-1653D, Messmer Instruments Limited, Gravesend, England) as per DIN EN ISO 1974 (2012). The water absorbency was accessed using the Cobb method *via* a Cobb tester (SFT 03t, IGT, Almere, Netherlands) and following the standard DIN EN ISO 535 (2014). The water absorption property was measured by the Cobb 60 test. The water used in these two measurements was deionized water to avoid any ions interfering with the sizing agents.

RESULTS AND DISCUSSION

Fiber Morphology

The fibers had an initial average ISO length-weighted value of 0.741 mm, then these lengths slightly declined along with increasing beating to 0.726 mm, 0.706 mm, and 0.696 mm (Table 3). The fibrillation values declined, which were supposed to increase along with the beating degree as the fibers would get externally more fibrillated. The authors' assumptions were that the fibers were "saturated" by the low refining beating degree, which caused a damage in the cell wall and partially delaminating of secondary wall. This may have led to a reduction in fiber width. The fibers' increased flexibility and changes to its morphological structure could have been influenced by the fibrillation.

Table 3. ISO Fiber Analysis by FS5 Displaying the Length-weighted Fiber Length (Lc(l)), Curl, Fiber Width and Fibrillation

PFI Beating Degree (rev.)	Lc(l)ISO (mm)	Curl (%)	Width (µm)	Fibrillation (%)
0	0.741	17.21	13.3	2.23
200	0.726	15.03	12.7	2.04
400	0.706	13.87	12.7	2.06
600	0.696	13.14	12.6	2.01

Schopper-Riegler

In Fig. 3, the Schopper-Riegler values for beating levels of 0, 200, 400, and 600 revolutions (minor) were 27.9, 43.8, 48.8, and 56.4 °SR, respectively.

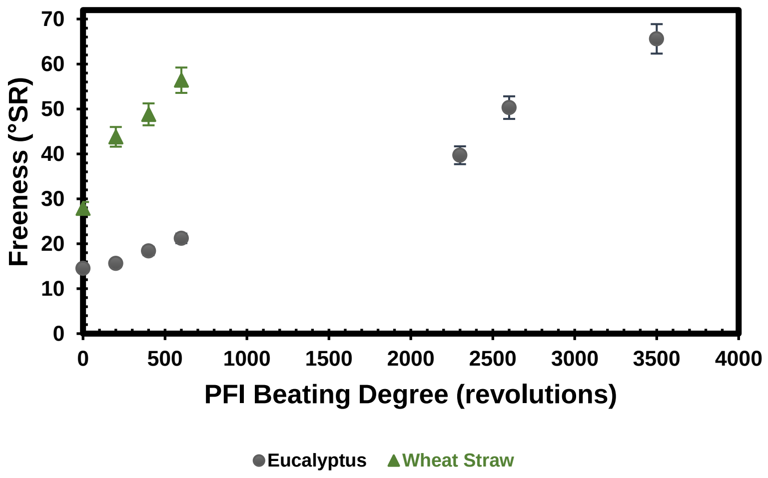

Fig. 3. Schopper-Riegler *vs.* PFI refining comparison between wheat straw and *Eucalyptus*

The initial recorded value of 27.9 °SR was considered high. Though the refining operating window was narrow with incrimination steps of only 200 revolutions, the final value reached 56.4 °SR. An explanation for this rapid change could be the morphological and ultrastructural properties (*e.g.*, thinner fiber wall) as well as the creation of fines during refining. In comparison, initial values of the *Eucalyptus* pulp were 14.5 °SR, and this rose to 50.3 °SR at a PFI beating degree of 2600 revolutions. Therefore, the refined *Eucalyptus* at 50.3 °SR will be compared later in the paper with the refined wheat straw at 48.8 °SR concerning the mechanical strength and the surface properties.

Roughness and Air Permeability (Bendtsen Method)

The surface properties were compared for the influence of the calendering line pressure, as it was assumed it would have more impact on the results than the refining. The roughness and the air permeability values of paper made of uncalendered pulp with 0, 200, 400, 600 revolutions (compared with Fig. 3) dropped (Fig. 4, Fig. 5). The roughness and air permeability values of calendered paper were further reduced; however, this effect was more intense for the roughness values. Refining and calendering contributed to the barrier concept in the base paper by significantly reducing the roughness and air permeability.

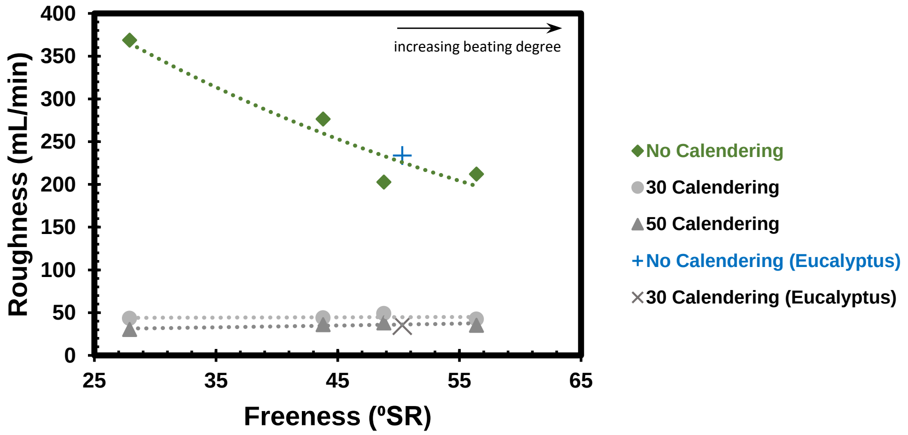

Fig. 4. Bendtsen values of roughness of wheat straw compared to *Eucalyptus* at 50.3 °SR

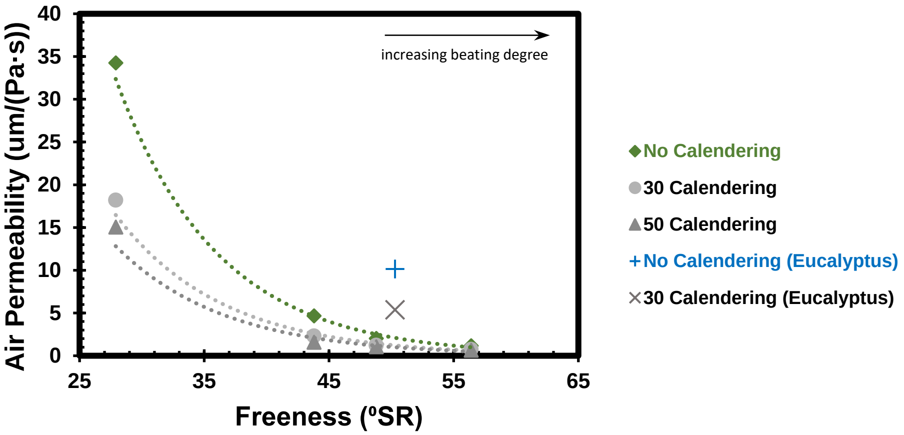

Fig. 5. Bendtsen values of air permeability of wheat straw compared to *Eucalyptus* at approximately 50 °SR

In comparison at similar freeness (approx. 50 °SR), the roughness of the refined uncalendered wheat straw paper recorded 203 mL/min, and for eucalyptus the value was 234 mL/min. While applying the same calender pressure load of 30 N/mm, the refined calendered wheat straw recorded 48.9 mL/min, and for eucalyptus the value was 35.2 mL/min. Moreover, the air permeability values of the refined uncalendered, and calendered wheat straw paper were dramatically lower than that of *Eucalyptus* paper with an approximate ratio of 1:5, respectively (with calendering wheat straw 1.21 *vs*. 5.37 µm/(Pa·s) for *Eucalyptus*).

Mechanical Properties

The mechanical properties degressively increased by refining (Table 4, Fig. 6, Fig. 7, compare with freeness in Fig. 3). However, the tear strength and tear index decreased along with refining (Fig. 8), resembling those results discussed by Singh *et al*. (2011) and Guo *et al*. (2009). An obvious explanation is the creation of fines during refining. These short fibers reduce the paper porosity; however the paper density is increased, as they create a more compact structure with better fiber-to-fiber bonding and formation. The refined, and therefore denser paper had a higher tensile strength, but the shorter fibers reduced the tear strength. Calendering tended to slightly reduce the tensile, burst, and tear strength (Fig. 6, Fig. 7, and Fig. 8). An explanation could be the dislocations of fibers created by the compression exerted by calendering.

The paper made of *Eucalyptus* pulp had similar values for the tensile and burst strength over the freeness compared to paper made of wheat straw. The tear strength was slightly higher what is an indication of less fines (Singh 1996).

Table 4. Mechanical Properties as a Function of the Beating Level (Refining) and Calendering Pressure

Beating Level (revolutions)	Calendering Pressure (N/mm)	Tensile Index (Nm/g)	Burst Index (kPa·m²/g)	Tear Index (mN·m²/g)
0	0	23.5	1.23	3.33
	30	23.4	1.26	3.19
	50	24.3	1.25	2.74
200	0	42.3	2.29	3.05
	30	42.1	2.28	2.94
	50	41.1	2.33	2.68
400	0	48.6	2.59	2.96
	30	46.3	2.62	2.64
	50	43.9	2.66	2.53
600	0	49.3	2.81	2.79
	30	48.1	2.85	2.67
	50	47.8	2.81	2.56
2600 (Eucalyptus)	0	43.9	2.50	4.02
	30	43.5	2.64	4.25

In comparison, the wheat straw pulp showed slightly higher tensile and burst strength indices, but it showed around 40% lower tear strength index compared to eucalyptus.

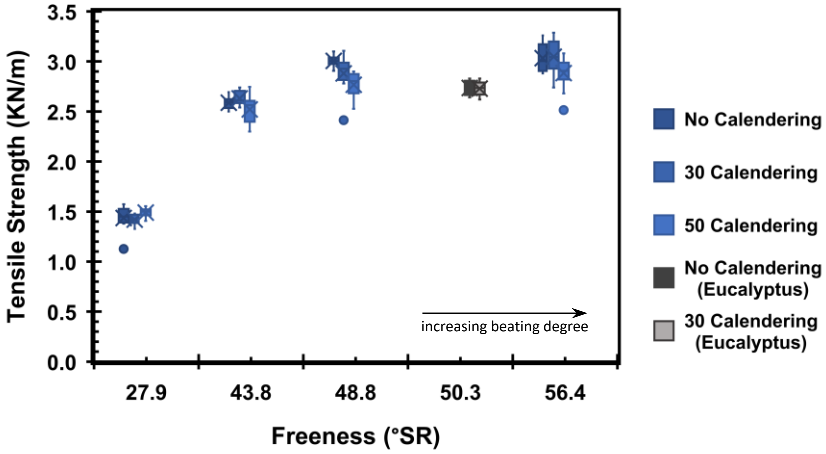

Fig. 6. Tensile strength development compared to *Eucalyptus* at 50.3 ºSR

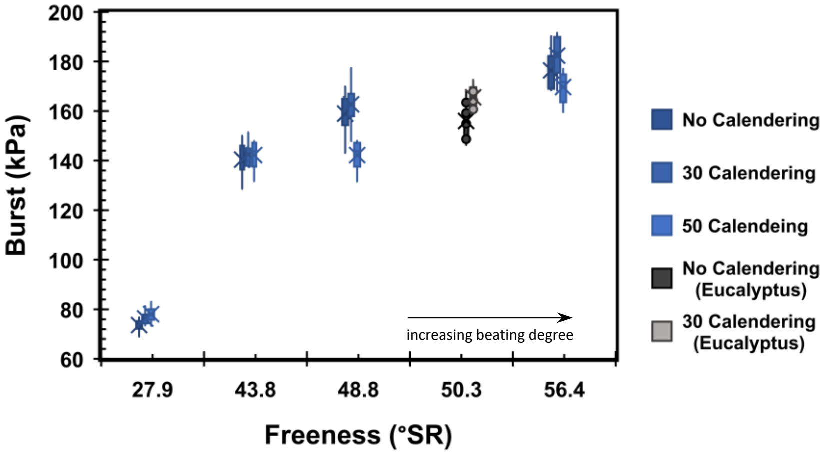

Fig. 7. Burst strength development compared to *Eucalyptus* at 50.3 °SR

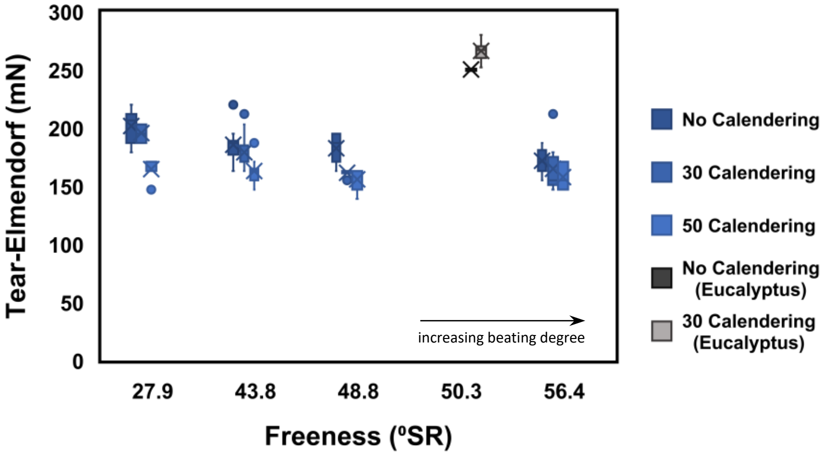

Fig. 8. Tear strength (Elmendorf method) development compared to *Eucalyptus* at 50.3 °SR

Summarizing the previous results, the static strength properties (tensile and burst) increased along with the refining possibly influenced by the growing presence of fines created by refining as it contributes to bonding leading to higher tensile strength (Watson and Garner 1997). However, the tear strength values declined because the tear strength obviously depended on the individual fiber length. Moreover, there was the development in the surface properties that increasing the beating level created smoother and denser handsheets (paper samples) with, explaining itself from theory, a higher tortuosity and lower z-penetration.

In Fig. 9, the operating window of the wheat straw refining could be clearly noticed between 47 ºSR and 50 ºSR (refining with 400 revolutions at wheat straw) to obtain the optimal results while overcoming the tear strength drop as a compromise.

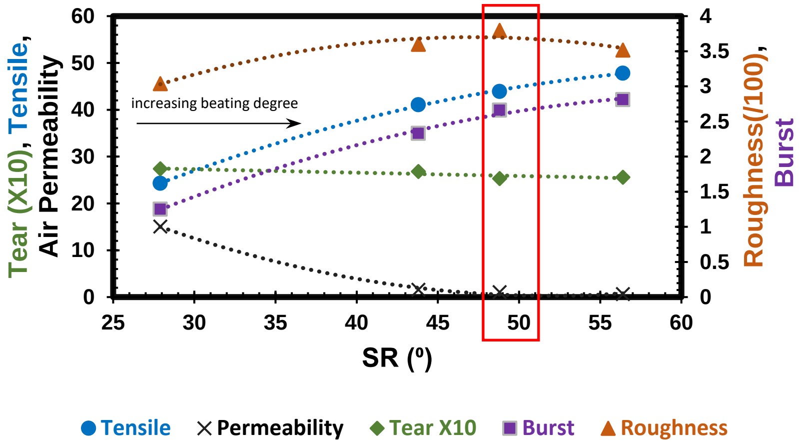

Fig. 9. Strength and surface properties of calendered handsheets (50 N/mm) over the freeness values, proposed operation window is depicted as red rectangle.

Influence of Sizing on Water Absorption (Cobb-values)

A conclusion from previous results of this study is that optimal refining conditions could be chosen to achieve acceptable tensile, burst, permeability, and roughness properties. Considering the decline in the tear strength, as a compromise, it is obvious that a refining with 400 revolutions, equivalent to 45 ºSR to 50 ºSR, and a calendering pressure of 30 N/mm would be a suitable adjustment to continue with coating of the wheat straw base paper.

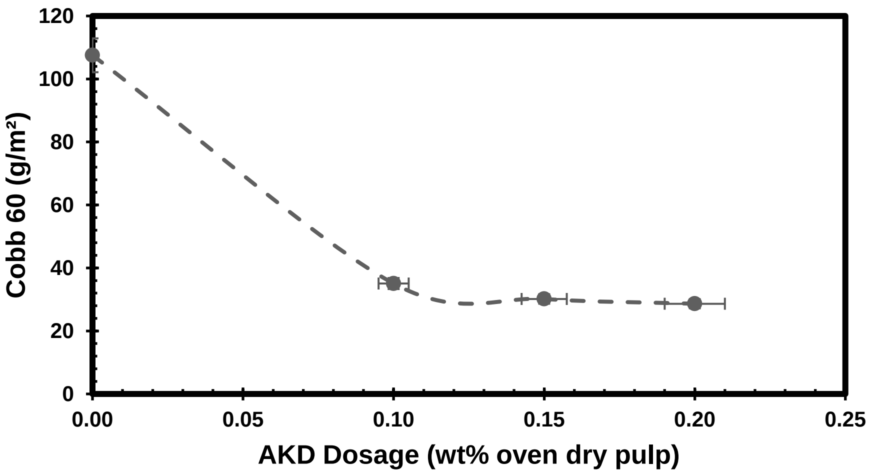

Fig. 10. Hydrophobicity performance against AKD dosage increase

Before coating, this paper was treated with a sizing agent (AKD) to reduce water absorption measured by the Cobb method. The Cobb 60-value of paper (refining with 400 revolutions, calendering with 30 N/mm) without sizing agent was 107.5 g/m^2. After sizing with increasing AKD concentrations, the Cobb-values degressively reduced to Cobb 60 values of 35.1, 30.2, and 28.6 g/m^2 by 0.10 wt%, 0.15 wt%, and 0.20 wt% dosages, respectively (Fig. 10).

Because the Cobb 60-values approached asymptotically to approximately 29 g/m², increasing the sizing agent dosage to more than 0.15 wt% to 0.20 wt% did not further reduce Cobb 60-values noticeably.

Precoating

For the coating, the refined handsheet made of wheat straw, with 400 revolutions and sized by 0.10 wt% AKD dosage was taken (Fig. 11). Conditions used were: a) uncalendered, and uncoated resulting in values for the air permeability of 2.0 µm/(Pa·s), and the roughness of 202.7 mL/min; b) pre-calendared (30 N/mm, the sheets were calendered before the precoating), and uncoated sheets resulting in values for the air permeability of 1.2 µm/(Pa·s), and the roughness of 48.9 mL/min; c) pre-calendered (30 N/mm) then coated resulting in values for the air permeability of 0.6 µm/(Pa·s), and the roughness of 41.3 mL/min; and d) pre-calendered (30 N/mm) then coated and calendered (30 N/mm) after the coating resulting in values for the air permeability of 0.5 µm/(Pa·s), and the roughness of 15.2 mL/min. The paper air permeability and roughness noticeably dropped after the application of calendaring whether it was pre-calendering or calendering after the precoating. The combination of pre-calendering, precoating, and additional calendering achieved the lowest values for roughness and air permeability in this study.

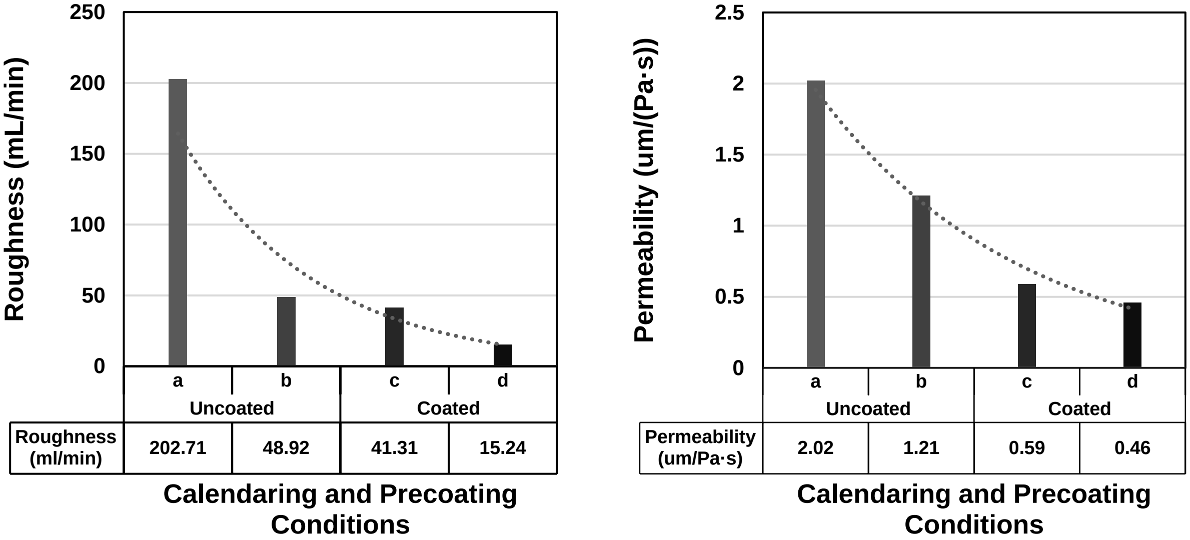

Fig. 11. Roughness and air permeability (Bendtsen values) against precoating and calendering times at 30 N/mm; a: uncalendered; b: pre-calendered; c: pre-calendered then coated; and d: pre-calendered then coated then calendered again

CONCLUSIONS

1. Wheat straw pulp exhibited an adequate performance, offering high smoothness, low air permeability, good web formation, and comparable tensile and burst strengths compared to *Eucalyptus*. Therefore, it is suitable as a base paper on which to apply a barrier coating.
2. The Schopper-Riegler degrees (ºSR) value sharply increased with a slight increase of the beating level (refining). Therefore, such treated fiber material could cause difficulties in the paper machine dewatering as well as fines retention from the white water. The tear strength of paper made of refined wheat straw pulp was lower than of paper made of *Eucalyptus* fibers.

3. The proposed refining window for optimal strength and surface properties would be in range of 47 to 50 ºSR.
4. The surface properties of the wheat straw fibers remarkably responded to calendering, as it significantly dropped the air permeability to roughly half of the values, and it improved the sheet smoothness by approximately 80% at different beating degrees by mere calendering pressure of 30 N/mm.
5. The results showed that the use of a moderate amount of alkylketene dimer (AKD) (0.10 to 0.15 wt%) was sufficient for these non-wood fibers to reach an optimal internal sizing value.
6. The substrate of wheat straw fibers showed an excellent performance in coating up-take for uniform coat-weight due to constant strength and controlled/even porosity.

In summary, it can be concluded that the wheat straw appeared to be a very good alternative solution to produce barrier base paper, as it resembles the properties of short wood-based fibers (*Eucalyptus*). Thus, it could be used in various applications of flexible packaging.

FUTURE WORK

The result of this study showed that tear strength of paper made of wheat straw pulp is lower than of paper made of *Eucalyptus* pulp. A modification worth examining in further studies would be mixing with longer fibers to overcome the tear strength drop while maintaining most of the barrier performance such as low air permeability.

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