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UTILIZATION OF BY-PRODUCT FROM THE STARCH PRODUCTION OF PAPER FOR PACKAGING MATERIALS

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Abstract: Several alternative non-wood fibers for papermaking are currently known. Some of them even deserve application on a larger industrial scale after longer-term studies. This also includes selected by-products from starch processing or distillery refuse that arise in food or biofuel industry. The present paper presents the various methods of further processing of by-products from starch production with regard to machine capabilities in the pulp and paper industry. Hydro-mechanical and chemical treatment methods for paper production are discussed.

Keywords: Chemical treatment, corn kernel fiber, hydro-mechanical treatment, physical properties of sheets

1 Introduction

Green composite materials based on naturally occurring reinforcement such as jute, sisal, flax, hemp, and kenaf; and matrix materials like biopolymers or bio resins which have been derived from non-wood materials, starch, and vegetable oils are nowadays gaining popularity for the various industrial applications [1]. Non-wood natural fibers are biological materials based on three main sources:

a) agricultural production,

b) food industry,

c) production residue of crops processed for the primary uses [1, 2].

The non-wood agricultural residues have been drawing attention in recent decades as an alternative raw material for paper production since usage of fibers derived from wood is linked with environmental problems such as deforestation, greenhouse emissions and global warming [3]. Europe produces about 310 million tonnes of agricultural residues, 74 % of which are utilized in bio-energy and composting [4]. The agricultural residues such as straw from barley, wheat [5-9], oat [10] or rapeseed [11] have been studied as an alternative source of fibres for pulping and paper production. In addition to being used as a feedstock for paper production, agricultural residues can be used as a reinforcement material for paper production from secondary fibers [12].

Compared to wood, the non-wood plants are characterized by wide range of physicochemical properties based on stronger effect of plant species and local factors like soil and temperature [13]. They feature low bulk density, high fibre content, and short fiber length. Non-wood plants fibres also have higher hemicellulose and silica content than wood [14]. The high silica content in straw fibers causes operational problems in pulp machines' efficacy and mills' chemical recovery [15]. Non-wood plants have lower lignin content compared to woody plants and their cooking or bleaching requires reduced energy and amounts of chemicals [14, 16-20]. Lower lignin content of agricultural residues is also beneficial for pulp bleaching since the toxicity of

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effluent generated during its bleaching process is considered to be less harmful compared to softwoods and hardwoods [21]. Besides physicochemical properties of fiber feedstock, their morphological features must be taken into account. These characteristics directly affect paper formation from the wet end of paper machine and the mechanical and optical properties of produced paper [22]. Fiber length and its average distribution are very important fiber features. They have major impact on the paper strength, paper sheet formation and drainage [23]. Paper strength also depends on the lignin and cellulose content of raw plant materials; pulp mechanical strength and especially tensile strength is directly proportional to cellulose content [17, 24], whereas lignin is an undesirable polymer and its removal during pulping requires high amounts of energy and chemicals.

Corn fibre is a by-product of wet-milling in corn starch extraction process. It is released after previous steeping of the corn grain in water. Endogenous proteases, along with sulfur dioxide, contribute to starch release. Some waxy maize species – having 0 % starch amylose – are grown on contract for the wet milling industry [25]. In this process corn grains are softened to facilitate separation of starch, gluten, fiber and germ [26]. Corn fiber comes from pericarp which is another by-product derived from corn starch production [27]. However, corn fiber also contains cell wall material from the endosperm, which is not contained in corn bran [28]. The chemical analysis of corn fiber performed by Pažitný et al. [29] shows that it contains 60.10 % of holocellulose (of which 19.90 % is cellulose) and 15.38 % of total lignin. This proves that corn fiber could be incorporated into paper furnish in a certain proportion to replace wood-based fibers to a certain extent. Our workgroup made progress and the first experiments with successful paper production were carried out with corn fiber substrates. We even made progress and the pilot experiments with successful paper production with potential industrial scale were carried out, while we also approached industrial legal protection [30].

This paper discusses the possibilities of paper production based on softwood kraft pulp and corn fiber also with respect to the capabilities of papermaking machines. The physical and mechanical properties of the prepared papers are analyzed.

2 Material

Air-dried corn fiber sample based on corn kernel's casing (CF) was obtained from Tate & Lyle Boleraz, Ltd. (Slovak Republic). The original substance had dry matter content 41.6 %. Unbleached kraft pulp (KP) based on softwood with Kappa number 26 was obtained from Mondi Štětí JSC (Czech Republic). The pulp was separately washed prior to beating to remove residual lignin until the effluent was clear. Sodium hydroxide NaOH, sulphuric acid H_2SO_4 and magnesium sulfate heptahydrate $MgSO_4.7H_2O$ were obtained from Slavus Ltd. (Slovak Republic). Diethylenetriaminepentaacetic acid (DTPA) was obtained from E. Merck KG (Germany). Hydrogen peroxide H_2O_2 was obtained from Mondi Štětí JSC (Czech Republic). Content of H_2O_2 in aqueous solution was determined iodometrically with 0.1 N sodium thiosulfate $Na_2S_2O_3$ at 459 grams per litre. The water solution of chlorine dioxide ClO_2 with concentration 8 g ClO_2 per litre of solution was obtained from Mondi Štětí JSC (Czech Republic).

3 Methods

International standards such as ISO 1762, T 264 cm-07, T 222 om-98, Tappi UM 250, Wise's method and ISO 692 were used for analysis of corn fiber and kraft pulp. The determination of process and non-process chemical elements in the production of bleached pulps was carried out according to the procedure PP-LCHS-19 combined with Tappi T 266. The element content was determined by flame atomization AAS by the novAA 800D instrument. Suspension M13 prepared for beating process consisted of 40 % CF and 60 % KP. The raw material ratio is

specified relative to the dry mass weight. Suspension was beaten in laboratory Valley holander according to ISO 5264-1 at concentration of 2 % w/w. The suspension was beaten for 60 minutes. The samples for determination of beating degree values according to Schopper-Riegler (°SR) and water retention values (WRV) were taken at the time of 0, 20 ,40 and 60 minutes. The Schopper-Riegler (°SR) values of the pulp samples were measured according to the STN ISO 5267-1. The water retention value (WRV) determines the ability of fibers to retain water. The water retention value was determined according to SCAN-C 62:00 standard, with each beating time of pulp sample undergoing four measurements. The WRV determination by SCAN-C 62:00 standard is directly related to the ISO 23714:2007. The WRV is expressed as the ratio of the weight of water retained after centrifugation, conducted under specified conditions, by a wet pulp sample (m_{wet}) to the weight of the same sample after oven drying at 105°C (m_{od}) according to Eq. (1) which is an equation of the form:

$$WRV\ (\%) = \frac{m_{wet} - m_{od}}{m_{od}}\ x\ 100 \quad (1)$$

The bleaching of pulp suspensions was performed in five stages. All bleaching stages were carried out at 10 % w/w concentration. The whole bleaching process consisted of these stages performed by known procedures [31]:

I. Acidification of pulp suspension based on CF and KP,

II. Chelation process (Q stage),

III. Peroxide bleaching with added alkali (OP stage),

IV. Chlorine dioxide bleaching in D stage,

V. Peroxide bleaching in alkaline environment.

From selected samples of mixed pulp were prepared the samples of paper sheets using standard procedure in Rapid-Köthen sheet former (Frank-PTI, Germany) according to the ISO 5269-2. Mixed pulp suspensions adjusted to consistency of 0.5 % as paper stock were processed in the wet part of papermaking system by thickening in a suction cylinder with standard cross-section, into which such an amount of water was poured that the resulting basis weight of paper sheet was 60 $g.m^{-2}$. The thickened paper stock was then transferred to felt or paper substitute and, with auxiliary paper placed on the other side of the thickened paper stock. The final paper sheets were dried in the drying part of papermaking system at temperature of 85 °C.

Selected paper sheets were characterized with physical and mechanical properties. Density was determined according to the ISO 534 based on thickness and basis weight. Brightness of prepared sheets was measured by an apparatus ELREPHO (Lorentzen & Wettre, Elrepho 950 ERIC®, Sweden) at diffuse blue reflectance at 457 nm and illuminant C, according to the ISO 2469 and ISO 2470. Ultimate tensile load, tensile index, tensile energy absorption, tensile strength and relative elongation were determined with Instron instrument (Instron Corporation, USA) according to the ISO 1924. Air permeance Gurley was used as a standard method for determination air resistance of paper sheets according to the ISO 5636-5. Air resistance of paper sheets was determined using Gurley densometer (Lorentzen & Wettre, Sweden). Zero-span tensile strength was determined using Z-Span 1000 Tester (Pulmac Inc., Canada) according to TAPPI T-531. All measurements of mechanical tests were performed on conditioned samples (23 ± 1 °C, 50 ± 2% RH). The strength, Gurley air permeability and zero-span tensile strength values reported in results are the mean at least 10 measurements (10 strips with standard dimensions – width of 15 mm and length of 100 mm). The ISO brightness and burst strength values are the mean of 5 measurements.

4 Results and discussion

Table 1 collects information about individual components and chemical composition of corn fiber and softwood kraft pulp.

Table 1. Chemical composition of corn fiber and softwood kraft pulp [29]

Component	Percentage of analyzed components (% w/w o.d.)		Method
	Corn fiber	Kraft pulp	
Ash	1.67	0.25	ISO 1762
Extractives	2.91	2.70	ISO 624
*Klason lignin	6.84	27.17	Tappi T 222 om-98
Acid soluble lignin (ASL)	8.54	0.19	Tappi UM 250
Total lignin	15.38	27.36	-
Holocellulose	60.10	69.69	Wise
Cellulose	19.90	41.90	ISO 692

*Klason lignin content was corrected for the ash content.

Holocellulose is natural mixture composed of cellulose and hemicelluloses. The holocellulose content in industrially processed corn fiber (60.10 %) is comparable to that in kraft pulp based on softwood (69.69 %). The cellulose proportion in corn fiber (19.90 %) is half that of kraft pulp (41.90 %). The total lignin in kraft pulp (27.36 %) is approximately half that of corn fiber (15.38 %). Corn fiber contains ash consisting of inorganic compounds, at a rate almost seven times higher than that of kraft pulp. The extractives content is slightly higher in case of corn fiber (2.91 %) compared to kraft pulp (2.70 %). Non-wood raw materials such as corn fiber with a low Klason lignin content have a beneficial effect on papermaking machines because they reduce energy consumption during processing and minimize machine wear. The chemical composition of fibers, particularly the ratio of cellulose to hemicelluloses and lignin, further determines the behavior of pulps. Fibers with higher hemicellulose content tend to re-swell more easily and maintain better bonding, whereas lignin-rich fibers display reduced bonding potential but greater resistance to mechanical stress [32]. However, lignin-rich fibers cause greater wear on papermaking machines for these reasons. On the other hand, non-wood fibers including corn fiber can be used to produce special cellulose-based products such as lignin-containing cellulose nanofibrils (LCNFs) and other products with tailorable properties, helping to push this valuable resource towards new applications or improvement in current applications in terms of fibrillation processes [33].

The next step was to analyze elements, especially metals, as these, within their salts, contribute to encrustation and fouling of bleaching equipments and papermaking machines. Metals, especially Ca^{2+} and Mg^{2+} salts are released from process water and fillers in the papermaking process, or their content in the process is affected by the type lignocellulosic materials. The formation of calcium carbonate proceeds according to the chemical equation – Eq. (2):

$$Ca(HCO_3)_2 \rightarrow CaCO_3\,(s) + H_2O\,(l) + CO_2\,(g) \quad (2)$$

Table 2 shows characterization and content of selected metals (metal cations) and silicon dioxide content including insoluble residue.

The proportion of metals or metal cations was comparable between CF and KP within some analyses, with significantly abundant metal being potassium (0.59 % in CF and 0.33 % in KP). Higher content in case of CF probably comes from fertilizers with a high content of potassium salts added to the soil. Sodium was the most abundant metal at all (approximately 1.15 %)

within KP, however, CF contained 10 times less of sodium compared to KP. The content of silicon dioxide together with the insoluble residue at the level of 0.05 % in CF was higher than in KP with 0.02 %. The proportion of metals or metal ions was 1.8 times lower in case of CF compared to KP based on softwood, which predetermines the given substrate for paper production, since it is not necessary to use larger loads of chelating agents in any of the process stages of paper production. The encrustation with some of mentioned metals and components formed by those chemical elements can occur in screen part and press part of paper machine performing critical roles in forming and dewatering the wet paper web. The problems caused by precipitated solids based on the metals can include non-uniform distributions of solids within the paper web during water removal, mechanisms by which water removal rates can deviate from the predictions of some established models of dewatering, and mechanisms of rewetting of the paper web after vacuum dewatering and after wet-pressing. [34].

Table 2. The proportion of metals and silicon in corn fiber and softwood kraft pulp

Analyzed chemical element	Percentage of analyzed chemical element (% w/w o.d.)	
	Corn fiber	Kraft pulp
Potassium	0.5900	0.3269
Calcium	0.0320	0.1571
Magnesium	0.1200	0.0245
Iron	< 0.0100	< 0.0100
Silicon	0.0530	0.0187
Sodium	0.1200	1.1536
Zinc	< 0.0010	< 0.0010
Copper	< 0.0010	< 0.0010

The hydro-mechanical properties of beaten pulp composed of CF and KP in a ratio of 40:60 can be seen in figure 1.

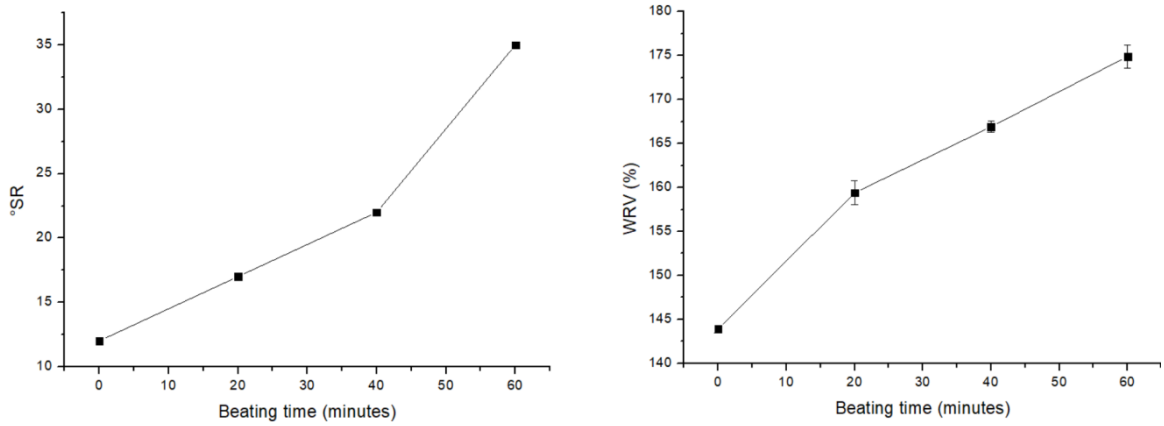


Fig. 1 Time-dependence of °SR (left) and WRV (right) for hydro-mechanically treated samples of CF and KP in a ratio of 40:60 (M13 series)

The effect of pulp beating on the properties of papermaking suspensions is significant. During the beating, fibres undergo separation, shortening, external and internal fibrillation, delamination, and an expansion of their surface area. This promotes water penetration into the fibres and enhances their swelling [35]. Figure 1 (left) shows rising trend of °SR beating degree values with increasing beating time. It is caused by enhanced fibrillation of fibers as a result of pulp suspension beating. This causes higher ability of pulp fibers to retain water, which can be supported by observing a growing trend of WRV with beating time (figure 1, right). The final beating degree value for the suspension M13 composed of CF and KP in a ratio of 40:60 was 35 °SR.

In our experiments with prepared pulp suspension M13, we used bleaching procedures designed by us including chelation with DTPA as a chelating agent, as it forms complex with biological ligands into a new, non-toxic complex between the metal ion and chelator and to excrete this complex from the bleaching system or living body [31, 36, 37]. Physical characterization such as basis weight, strength, barrier and optical properties of handsheets formed from M13-based pulp suspensions composed of CF and KP in an initial ratio of 40:60 is shown in Table 3.

Table 3. Physical properties – strength, optical and barrier properties of paper handsheets from pulp M13 composed of CF and KP in a ratio of 40:60

Physical property	Unit	Beaten sample	Beaten suspensions after chemical treatment and bleaching			
		M13	M13-Q	M13-OP	M13-D	M13-K
Beating degree	°SR	35				
Basis weight	g/m^2	65.1	60.9	61.3	60.7	57.8
Tensile strength	kN/m	3.6	2.9	3.9	3.7	4.0
Tensile index	Nm/g	55.1	47.5	64.1	60.9	68.9
Tensile energy absorption	J/m^2	55.3	69.9	119.4	98.3	116.1
Breaking length	km	5.617	4.841	6.534	6.218	7.025
Relative elongation	%	1.9	2.6	3.6	3.1	3.5
Burst strength	kPa	159.6	141.8	223.0	226.8	214.8
Air resistance (Gurley)	s / 100 ml	18.54	15.66	84.68	89.44	91.34
Zero-span tensile strength	N/cm	59.7±5.1	61.8±2.3	75.7±2.7	83.2±2.6	78.3±2.7
ISO brightness	%	25.31±0.46	26.23±0.50	47.52±0.34	45.22±0.62	70.43±0.39

Tensile strength including zero-span tensile strength is a crucial factor for products that serve in conditions of strength durability, load bearing, and conjunctions [38-40]. The zero-span tensile strength shows an increasing trend with the number of bleaching stages except for the last bleaching stage, where a slight drop from the previous trend can be seen. A rising trend is evident for ISO brightness, where a slight offset from the rising trend is observed in the D-stage. Combination of corn fiber with recycled waste paperboard provided lower ISO brightness values in our previous paper [31]. However, the largest brightness achieved after the last stage of bleaching and washing (suspension M13-K) was also significantly higher compared to our previous paper when we used distillery refuse itself based on corn which is similar material from the perspective of plant-based components and morphology of original fibers [41]. The growth of the relative elongation can be seen up to the third bleaching stage, then a minor decline of its value is noted, afterwards it again rises. Gurley air resistance values show an

upward trend apart from the value from second stage, which indicates an improvement in resistance to air penetration. The breaking length also shows the tendency to rise. Due to these barrier properties the corn fiber can be utilized in papermaking and paper substrates can be considered not only in production of packaging materials based on non-wood papers but also in many utility paper grades such as tag paper, kraft paper, index paper, utility industrial paper or other paper grades.

CONCLUSION

In this paper we aimed to find ways to utilize fibrous processing residues from corn starch production by addition them into the papermaking suspension consisting of corn fiber (CF) and kraft pulp (KP) based on softwood designated as M13. The suspension M13 was composed of CF and KP in a ratio of 40:60. The main goal was to produce the paper based significantly on non-wood substrate which has suitable strength, optical and barrier properties for production of packaging material.

The holocellulose content in industrially processed corn fiber (60.10 %) is comparable to that in kraft pulp based on softwood (69.69 %). Due to relatively high holocellulose content and low lignin content (15.38 %) corn fiber is chemically suitable for paper slurry production and papermaking. The chemical analysis was utilized in setting of mechanical and chemical processing of the suspension and revealed its suitability for production of paper with respect to the capabilities of papermaking machines. The water suspension was prepared by beating the fibrous mixture of CF and KP in laboratory Valley holander. The suspension was subsequently chemically treated in a multistage bleaching process to remove residual lignin and increase the pulp brightness. After every bleaching step, suspension samples were taken for further handsheet formation.

To evaluate the effect of the composition of the papermaking stock and the effect of its bleaching on the properties of the paper handsheets, their mechanical, barrier and optical properties were determined. The results show that with increasing number of applied bleaching stages, ISO brightness values grow for designed type of M13 suspension and bleaching process. The handsheets prepared from M13 suspension after final bleaching stage and washing, exhibit good physical properties and the combination of CP and KP is suitable for production of packaging materials based on non-wood papers but also in many utility paper grades such as tag paper, kraft paper, index paper and utility industrial paper.

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