

Bleached Kraft Pulps from Blends of Wood and Hemp. Part I. Demand for Alkali, Yield of Pulps, Their Fractional Composition and Fibre Properties

DOI: 10.5604/01.3001.0012.7514

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Abstract

In the paper, results of the kraft pulping of blends of birch or pine with hemp stalks or a hemp-woody core composed of 80% wood and 20% hemp fibrous raw materials are presented. The unbleached kraft pulps produced were then subjected to oxygen delignification and bleaching in order to obtain bleached kraft pulps. The research performed made it possible to compare the final yield and viscosity of the bleached kraft pulps from raw material blends with those of kraft pulps from birch and pine. The effect of replacing 1/5 of birch or pine with hemp fibrous raw materials on the content of individual fractions of fibres in the bleached pulps, the relative content of fines in them, the average length and width of fibres, its coarseness and the fibres' deformation indices was also determined. From the study it follows that better effects of replacing a part of wood in the process of production of bleached kraft pulps are achieved with hemp stalks. Blends of wood with this fibrous raw material give a higher final yield of pulps, lower content of fines and higher average fibre length than using blends of wood and a hemp woody-core.

Key words: birch, pine, wood/hemp blends, kraft pulping, oxygen delignification, bleaching, pulps properties, fibres properties.

Abbreviations

NPFRMs – non-woody plant fibrous raw materials

PFRMs – plant fibrous raw materials

HFRMs – hemp fibrous raw materials

TM – thermo-mechanical

CSS – cold caustic soda

APX – alkaline peroxide extrusion

Hs – hemp stalks

Hwc – hemp woody-core

B – birch

P – pine

Q – chelation process

O – oxygen delignification

D₀ – first stage of pulp's ECF bleaching process

E – extraction stage of pulp's ECF bleaching process

D₁ – second stage of pulp's ECF bleaching process

DP – degree of polymerisation

CED – copper ethylenediamine solution

Introduction

In 2010 about 9% of all primary pulps used in the world to produce papers and boards were manufactured from non-wood plant fibrous raw materials (NPFRMs). In Europe, the amount of primary pulps produced from NPFRMs was only about 390 tonnes, being much higher in countries with poor forest cover, for example in the countries of Asia (approx. 15.000 tonnes) [1]. The Euro-

pean pulp and paper industry is dominated by the production of pulps from wood and recovered paper. The large use of wood for the manufacture of pulps for the paper industry is due to its good processability in equipment installed in pulp mills, which guarantees an intended production volume of pulps, as well as optimal properties of pulps required for the production of paper and boards [2-5].

The consumption of wood for paper production increases with population growth and improvement in the standards of people's lives. Therefore the demand for plant fibrous raw materials (PFRMs) will continue to grow in the nearest future [6, 7]. One of the ways of obtaining an extra amount of wood is to increase the number of trees cut down in forests, which recently has not been welcome because of the low degree of forest cover in many countries of the world, as well as increased awareness of the important role of forests in the environment [5, 8, 9]. Insufficient supply of wood for the pulp and paper industry has led many times in history to an increase in its price, the need for import of this material and primary pulps, as well as restrictions on investment and development in the pulp and paper industry in many countries, e.g. Poland [10].

Taking into consideration the intensification of criticism of the consumption

of wood in the pulp and paper industry, we have undertaken a study concerning the suitability of industrial hemp and other crops (cultivated mainly for energy purposes), which grow well in the climate prevailing in Poland, for the production of papers and paperboards. So far we have examined the chemical composition of the various anatomical parts of hemp (stalks, woody-core, bast fibres), stalks and leaves of *Miscanthus × giganteus*, and straw of tall wheatgrass, tall fescue, and tall oatgrass, and compared the results of delignification of these NPFRMs and wood using kraft pulping methods with those of the delignification of birch and pine wood, the two main wood species used in the Polish pulp and paper industry [11-14].

From a review of literature, it appears that the processing of hemp fibrous raw materials (HFRMs) into cellulose-pulps was studied earlier. They concerned the processing of hemp raw materials (stalks, woody-core or bast) into such types of pulps like: soda [15-20], soda-oxygen [21], Organosolv [22, 23], TM and CCS [24], APX [25], enzymatic-mechanical [26], bisulphite [18, 21], neutral-sulfite [21] and kraft [11, 12, 18, 21, 27, 28]. The kinetics of the delignification of hemp stalks and hemp woody-core [29, 30] and properties of bleached pulps from hemp roots [31] were also studied. However, in these studied HFRMs were pro-

cessed alone or only in comparison with the wood species chosen.

In this study, identification of the effects of the common processing of wood and HFRMs was performed. In the case of Poland as well as in Europe, such an introduction of hemp to the paper industry should be taken into account because of the, thus far, relatively low availability of hemp, the better rhythmicity of the production of pulps which can be obtained with this way of introduction of a new NPFrm to the pulp and paper industry, as well as the use of hemp fibers in other types of industry, e.g. textile [32, 33].

The description of the results obtained was divided into two parts. The part herein presents the results of determination of the demand of chemicals for the pulping of blends of wood with HFRMs, a comparison of the yield of unbleached and bleached pulps from such blends with that of pulps from birch and pine, its fractional composition, as well as morphological properties of the fibres.

■ Experimental

Plant fibrous raw materials

In the study, air-dried stalks of industrial hemp (variety Białobrzskie) (denoted as Hs) and hemp woody-core (Hwc) were used. These HFRMs were purchased from the Institute of Natural Fibres and Herb Plants in Poznan. For comparison, birch (B) and pine (P) chips obtained from the pulp and paper plant – International Paper Kwidzyn were also used. Hemp stalks were cut before pulping into fragments of 3-6 mm. This operation was performed at the Institute of Natural Fibres and Herb Plants in Poznan in an unknown device. Hemp woody-core was used as obtained, i.e. in the form of cross-wise and longitudinally defragmented parts of 0.5-3.0 cm length. Such a form of this PFRM was probably the result of the decortication process of hemp stalks.

Kraft pulping process

50 g of oven-dried (o.d.) birch, pine, hemp stalks, and hemp woody-core were cooked in 300 cm³ autoclaves using various amounts (18-23%) of active alkali, which were calculated as NaOH on oven-dried (o.d.) fibrous raw material. The experiments were performed in a Santasalo-Sohlberg sectional digester. In all experiments, pulping liquor had a sulphidity of 25%. The liquid-to-raw mate-

rial ratio in all pulping experiments was 4:1. The heating-up time, digestion time, and temperature of pulping were for all samples the same and as follows: 90 min, 90 min, and 165 °C. Pulped PFRMs were pre-washed with water in a sieve, washed with water by diffusion within 24 hours in a bucket, disintegrated in a laboratory disintegrator, and screened in a Weverk screen in order to remove uncooked parts of PFRMs. Uncooked parts of PFRMs separated in the Weverk screen were then defiberised in a laboratory Bauer disc refiner and added to unbleached pulp. The pulp obtained was then dried and its kappa number determined. Yields of the pulps were determined through their centrifugation, determination of the dryness, and appropriate calculations.

Removal of metal ions (Q)

The chelation stage (Q stage) was performed before each delignification of the pulps with oxygen. The pulps' consistencies in this stage were 10% the time of pulp treatment – 60 min, and the temperature – 75 °C. At the beginning of the treatment, a 0.5% charge of EDTA (calculated on o.d. material) was applied to the pulp, and the pH of the pulp suspension was adjusted to 4.5 using diluted H₂SO₄. After 60 minutes of treatment, the pulps were discharged from polyethylene bags, filtered, washed with four aliquots of 1000 ml of deionised water, and stored in a plastic bag for further treatment.

Oxygen delignification (O)

120 grams of o.d. pulp was disintegrated in distilled water, filtered on filter synthetic textile, and then squeezed and placed in a polyethylene bag. Then 0.2% magnesium sulphate on o.d. pulp (in the form of a 1% solution) and sodium hydroxide dissolved in the amount of water needed to reach 8% pulp consistency were added sequentially to the bag. The amount of NaOH needed to properly delignify the pulp was determined by multiplying the kappa number of the pulp by a factor of 0.1. Then the pulp was mixed in a bag by kneading and quantitatively transferred to a Jayme reactor. The autoclave of the digester was closed and filled with oxygen to pressure of 0.5 MPa, the rotary mechanism of the digester was then switched on, and the content of the autoclave was heated up to 100 °C for 30 minutes. This temperature was maintained in the autoclave for a further 60 minutes. After the preset

time elapsed, the autoclave was degassed and emptied. Then the pulp was washed using the same method as described for the Q process.

Chlorine dioxide bleaching (D₀, E, and D₁ stages)

The bleaching of pulps was performed according to the bleaching sequence D₀ED₁, where the D₀ and D₁ stages were the chloride dioxide treatment of pulps, while the E stage is the pulps' alkaline extraction stage. 100 g of o.d. pulp was disintegrated in distilled water, filtered on a filter textile, squeezed by hand and then placed in a polyethylene bag. Then a chlorine dioxide solution was added to the pulp in the bag. The total amount of this chemical used in the bleaching of each pulp was calculated by multiplying its kappa number by a factor of 0.25. Sixty-five percent of the amount of ClO₂ calculated was added at the D₀ stage and the rest at the D₁ stage. After the addition of ClO₂ solution to the pulp in the bag, it was kneaded by hand. Bleaching experiments were conducted at 10% pulp consistency. No adjustment of the pH of the pulp suspension was made. After kneading, the pulp was placed in a water bath with a constant temperature of 75 °C. The bag was kneaded again after a further 5 and 30 minutes. After 60 and 90 minutes of bleaching in the D₀ and D₁ stages, respectively, the bags were opened. The pulp samples were then washed using the same method used in the Q stage process. After the D₀ stage, the pulps were treated with an alkali using an alkaline extraction process (E stage). Deionised water and sodium hydroxide (1.5% on o.d. pulp) were added to the bag containing the pulp in amounts to get 10% pulp consistency. After kneading, the bag was placed in a water bath and heated during 90 min at 75 °C. The content of the bag was kneaded after 10 and 30 minutes. After the treatment, the pulps were washed using the same method as described for the Q process.

Determination of properties of pulps and papers

Characterisation of pulps involved the following parameters: kappa number (PN-85/P50095/02 1985) [34], intrinsic viscosity (ISO 5351-1:1981, glass Übelode viscometer) [35], brightness R₄₅₇ (ISO 2470 1999, Elrepho 2000, DataColor, USA) [36], number of fibres in 1 g of pulps, fine content, and morphologi-

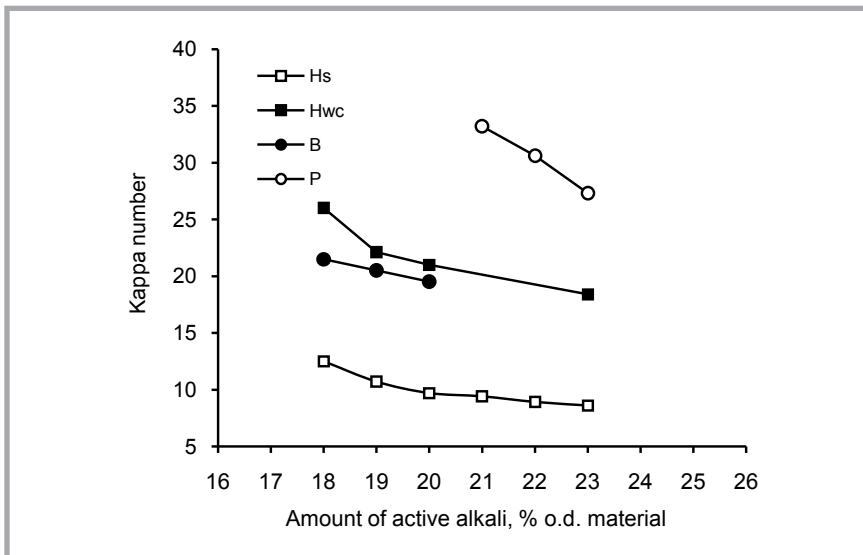


Figure 1. Kappa number of pulps obtained in the kraft pulping process of birch, pine, hemp stalks, and hemp woody-core.

cal properties (MorFi LB-01 apparatus, Techpap, France) [37].

Elaboration of results

Kraft pulping, oxygen delignification and bleaching experiments of the fibrous raw materials and pulp, respectively, were performed twice. Therefore the yields of pulps in these processes are the arithmetic averages of two procedures. The Kappa number, intrinsic viscosity and indices determined using MorFi apparatus were also determined twice, and hence their values presented are also the arithmetic averages of two results.

Results and discussion

Figure 1 shows the results of determination of the amount of alkali needed to obtain regular papermaking, kraft pulps (i.e. kraft pulps intended for bleaching) from birch or pine chips, as well as hemp stalks and hemp woody-core.

It is well known that softwood and hardwood must be pulped to a kappa number

of 30 and 20 units, respectively, in order to obtain pulps suitable for bleaching. As can be seen in **Figure 1**, in order to obtain such kraft pulps from birch or pine, one must use 19-20% and 22-23% of active alkali (as NaOH) on o.d. material, respectively, in this kind of pulping process. Using the amounts of alkali, hemp stalks are pulped to an 8-13 kappa number of pulps, while hemp woody-core is transformed into pulps with a kappa number of 19-23. Taking into account that wood was the dominating component of the blends' fibrous raw materials used in order to obtain wood/hemp pulps, kraft pulping experiments of B/Hs, B/Hwc and P/Hs, and P/Hwc blends were performed using 19% and 23% of active alkali on o.d. material, respectively.

The unbleached pulps prepared from B/Hs, B/Hwc and P/Hs, P/Hwc blends, and birch or pine wood were then subjected to oxygen delignification and ECF (Elemental Chlorine Free) bleaching according to the D₀ED₁ sequence. Results of the determination of the pulps' yields,

their kappa number and brightness after these processes are shown in **Table 1**.

As can be seen in **Table 1**, the replacement of 20% of birch or pine with hemp stalks (Hs) increased the yield of unbleached pulps by 2.1% and 2.8%, respectively, while replacement of 20% of birch or pine with hemp woody-core (Hw) decreased it by 1.4% or had little effect on this index, respectively. As for the kappa numbers of pulps from PFRM blends, one can state that, apart from B/Hwc pulp, the others from PFRMs blends had lower kappa numbers (higher degree of delignification) than those from birch and pine. Thus the replacement of 20% of birch and pine with hemp materials has a relatively small effect on the kappa number of unbleached pulps.

The similar kappa number of pulps from blends of materials and only birch and pine wood after pulping made it possible to perform oxygen delignification of these pulps in very similar conditions. As seen in **Table 1**, differences in the kappa numbers of pulps from wood/HFRM blends and only wood after oxygen delignification were also small, and thus the conditions of bleaching of oxygen delignified pulps were also similar.

After bleaching, the differences in yields between pulps observed after pulping were maintained (**Table 1**). The final yields of bleached B/Hs and P/Hs were higher from the final yield of bleached birch and pine pulp by 2.0 and 2.7%, respectively, while a replacement of 20% of birch or pine with hemp woody-core (Hw) decreased it by 1.8% or had little effect, respectively.

Apart from this, analysing the results of **Table 2**, one can also state that the processing of blends of birch with hemp stalks and hemp woody-core decreases the final brightness of unbleached pulps

Table 1. Results of kraft pulping of different PFRMs and oxygen delignification and bleaching of pulps obtained. $Y_{P,O,B}$ – yield of pulps in pulping, oxygen delignification and bleaching processes; Y_F – final field of bleached kraft pulps; κ – kappa number.

Process	Kraft pulping		Oxygen delignification (O)		Bleaching (D ₀ ED ₁)	After Sa, O and D ₀ ED ₁			
	Y _D , % o.d. PFRM	κ	Y _O , % o.d. pulp	κ	Y _B , % o.d. pulp.	Y _F , % o.d. PFRM	ΔY	R ₄₅₇ , %	ΔR ₄₅₇
B	52.0	20.1	96.5	9.0	97.5	48.9	–	88.6	–
B/Hs	54.1	19.3	96.8	9.0	97.1	50.9	+2.0%	88.1	-0.5%
B/Hwc	50.6	21.6	96.1	9.6	96.9	47.1	-1.8%	87.1	-1.5%
P	44.0	30.4	96.2	11.6	97.4	41.2	–	87.8	–
P/Hs	46.8	29.3	96.6	11.0	97.2	43.9	+2.7%	88.7	+0.9%
P/Hwc	44.2	29.9	96.3	10.5	96.8	41.2	0	87.3	-0.5%

Table 2. Intrinsic viscosity of bleached pulps from blends of wood and HFRMs in comparison with that of birch or pine pulp.

Kind of pulp	Intrinsic viscosity, cm ³ /g	Change, %	Kind of pulp	Intrinsic viscosity, cm ³ /g	Change, %
B+Hs	928 ± 20	-7.4	P+Hs	732 ± 16	+4.6
B+Hwc	980 ± 15	-2.2	P+Hwc	701 ± 10	+0.1
B	1002 ± 12	-	P	700 ± 23	-

by 0.5 and 1.5%, respectively, while the processing of blends of pine with hemp stalks and hemp woody-core increases and decreases this index of pulps by 0.9 and 0.5%, respectively.

A well-known phenomenon in the case of the common pulping of blends of different species of wood is deeper pulping of material with a lower lignin content, which may lead to a reduction in the degree of polymerisation (DP) of cellulose in pulps from such blends [38]. In order to determine the effect of the replacement of 20% of wood with HFRMs and common pulping of these PFRMs on the DP of cellulose of the pulps obtained, their intrinsic viscosity in a CED reagent was determined, which is still a commonly used indicator of the DP of cellulose in unbleached and bleached pulps in kraft pulp mills [39, 40]. Results of the deter-

mination of this index of pulps are presented in **Table 2**.

Form the data of **Table 2**, it can be seen that the replacement of birch with hemp stalks or hemp woody-core has a certain negative influence on the intrinsic viscosity of these pulps and a positive effect of this feature of pulps from pine/HFRMs blends; however, the changes observed are not high, i.e. only 2-7%. In the first case, a reduction in viscosity may result from a stronger reduction in the degree of polymerisation of cellulose of hemp bast fibres, because these fibres contain a small amount of lignin [11, 41] and in kraft pulping, the cellulose of these fibres becomes freed from the protective action of this lignin quite quickly. In the case of blends of pine and HFRMs, the final intrinsic viscosity was a little bit higher than that of pulp from pine, probably be-

cause the lower intrinsic viscosity of pine bleached pulp than that of bleached pulps from Hs and Hwc.

In the next part of the study, the effect of replacing of 1/5 of wood with hemp stalks or hemp woody-core on the fractional composition, number of fibres, average fibre dimensions as well as their damage indices are studied. In **Figure 1**, a comparison of the fractional composition of bleached pulps from wood/HFRM blends and birch or pine is presented.

From the data of **Figures 2.a and 2.b**, it can be seen that the replacement of 20% of birch with hemp stalks increases the content of short fibres with a length of 0.2-1.0 mm in the pulp, thus increasing its polydispersity. The results also indicate that the pulp from B/Hs blends also contains an additional fraction of fi-

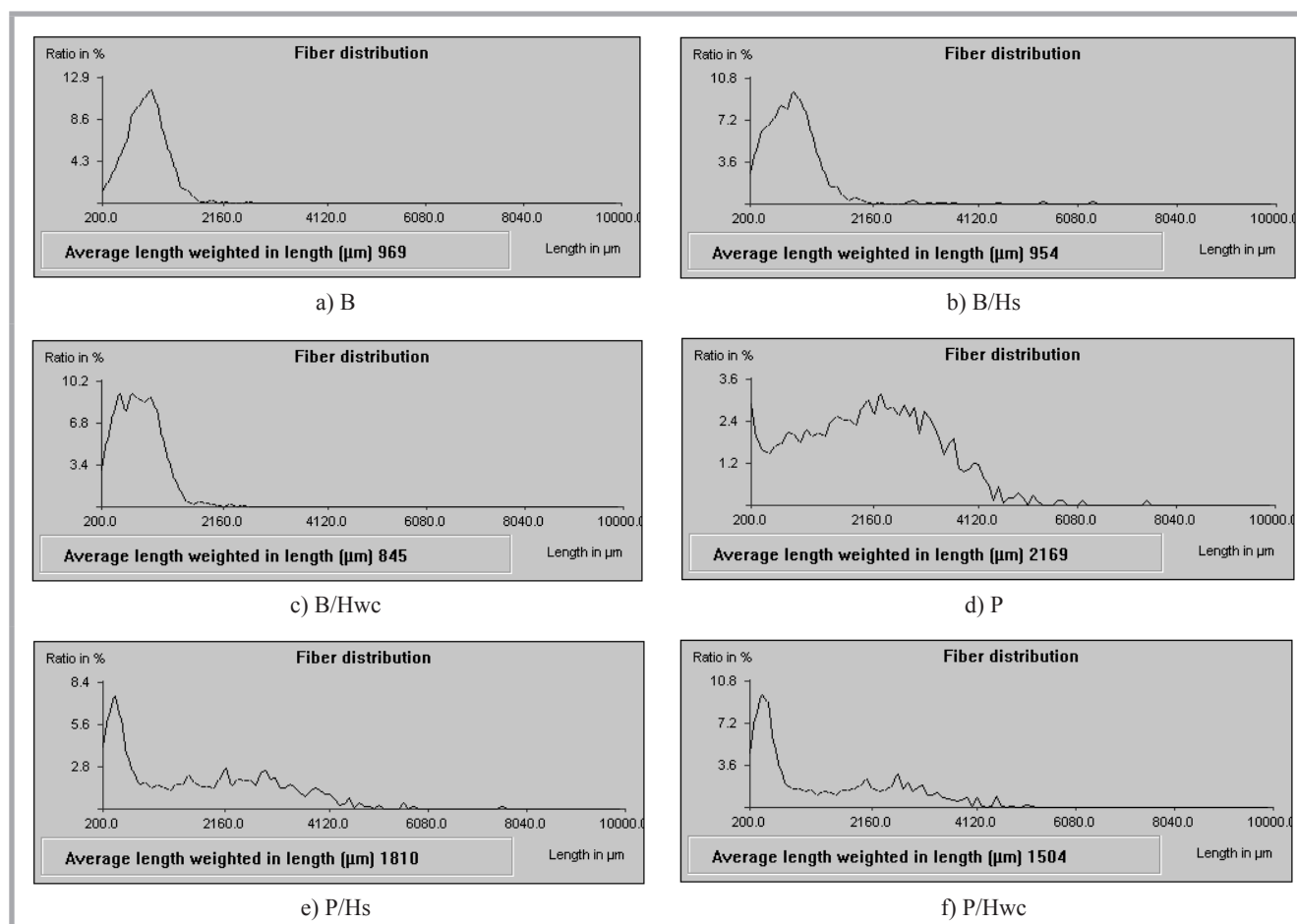


Figure 2. Compilation of fibre distributions of bleached kraft pulps from birch, pine, birch/HFRM, or pine/HFRM blends.

Table 3. Properties of pulps studied and their fibres. In brackets - percent change of these indices as a result of replacing 1/5 of wood with HFRMs.

Property	Pulp	B	B/Hs	B/Hwc	P	P/Hs	P/Hwc
Number of fibres in 1 g of pulp		9 346	10 854 (+15.1%)	12 925 (+38.3%)	4 018	5 904 +45.9%	8 801 (+116.4%)
Fines, % of area		5.53	6.20 (+20.3%)	9.32 (+48.1%)	3.74	5.14 (+27.4%)	5.26 (+50.1%)
Weighted average fibre length, mm		0.969	0.954 (-1.5%)	0.845 (-12.8%)	2.169	1.810 (-15.6%)	1.504 (-30.7%)
Average fibre width, μm ,		21.3	22.0 (+3.3%)	21.9 (+2.8%)	27.6	24.9 (-9.8%)	22.6 (-18.1%)
Coarseness, mg/m		0.1328	0.1265 (-4.7%)	0.1126 (-15.2%)	0.1988	0.1855 (-6.7%)	0.1483 (-25.4%)
Kinked fibres, %		49.9	40.1 (-19.6%)	37.8 (-24.2%)	52.5	38.0 (-27.6%)	38.8 (-26.1%)
Curl, %		9.9	8.7 (-12.1%)	8.0 (-19.2%)	14.0	9.3 (-33.6%)	8.8 (-37.1%)
Broken ends, %		20.42	20.21 (-1.0%)	20.14 (-1.4%)	33.48	27.91 (-16.6%)	22.08 (-34.1%)

bres with a length of 1.8-6.0 mm, which does not contain birch pulp. However, the amount of these fibres is small compared to the total amount in B/H_s pulp. It is for sure the fraction of shortened primary hemp and secondary hemp bast fibres. Both of these fibres are present in hemp bast [42-44]. It is reported that the original lengths of the bast fibres are in the range of 3 to 55 mm (on average 15-28 mm) [43, 45], while that of secondary bast fibres is only approx. 2 mm [15, 43].

The replacement of 1/5 of birch wood with hemp woody-core extends the length distribution of fibres yet further in the direction of short fibres than in the case of pulp from the B/H_s blend (**Figures 2.c and 2.b**). This trend should be recognised as negative, but on the other hand, one should take into account that such a trend must also occur in the case of mixing, for example, birch pulp with pulps from some species of eucalyptus or acacia, in the case of which the average length of fibres amounts to 0.63-0.78 mm [46-48].

The fibre length distribution of pine pulp is shown in **Figure 2.d**. a comparison of the data in this **Figure** and in **Figures 2.e and 2.f** shows that the replacement of 20% of pine wood with hemp stalks or hemp woody-core increases primarily the content of short fibres in the pulp from PFRM blends (length range: 0.2-0.8 mm), minimally influencing the content of fibres of 1 to 6 mm length.

The effects of replacing of 1/5 of birch or pine with hemp stalks and hemp woody-core were also examined for such pulp properties as the number of fibres in 1

gram, the content of fines, the average fibre length and width, coarseness, and fibre damage indices, such as curl, kink, and the number of broken fibres. These indices are presented in **Table 3**.

Data from **Table 3** indicate that the replacement of 20% birch wood or pine wood with hemp stalks or hemp woody-core increases the amount of fibre in 1 gram of pulps obtained from such PFRM blends. Such a replacement also increases the fines content in B/H_s, B/Hwc, P/Hs, and P/Hwc pulps in comparison with B and P pulps. The increase in the number of fibres in 1 gram of pulps from PFRM blends and fines is clearly higher for pulps from wood/hemp woody-core blends than for those from blends of wood and hemp stalks.

The most representative measure of the fibre length of pulps is considered to be the weighted average fibre length. a decrease in the average fibre length has a clear negative effect on all strength properties of pulps in the form of handsheets, as well as on their bulk [49-52]. As can be seen in **Table 3**, the reduction in the average fibre length is clearly smaller in the case of pulps made from wood/hemp stalks blend than in those from wood/hemp woody-core blends.

The average width of fibres, which affects their susceptibility to collapse, during consolidation of the paper web slightly increased in the case of pulp from the B/H_s blend, while for pulps from the P/Hwc blend it was markedly reduced. This is due to only the little higher average width of hemp fibres than that of birch fibres [53].

The average value of coarseness of fibres of pulps from B/HFRM and P/HFRM blends was markedly reduced in comparison with that of birch or pine pulps. This is due to the low coarseness of hemp woody-core fibres [53, 54]. This change should be conducive to achieving high tensile and bursting strengths of paper handsheets, while it should negatively impact its tear resistance and bulk [50, 55].

Pulp producers take a number of actions aimed at reducing the amount of damage to fibres in the processing of wood into cellulose-pulps, such as the cold blowing of pulped chips from a digester, a reduction in the number of defiberising devices, and pumping of the pulp in bleach plants [56]. Indices determining the degree of damage to the fibre of pulps from wood/hemp blends (curl, kink, broken ends) have lower values than in the case of pulps only from wood, which can be considered as a positive trend from the point of view paper properties.

Conclusions

- The final yield of bleached pulps from the common processing of birch or pine and hemp stalks is higher than that of the common processing of these species of wood with hemp woody-core.
- The production of bleached pulps from blends of birch or pine with hemp stalks or hemp woody-core does not have a significant negative effect on the intrinsic viscosity of bleached pulps from blends of these fibrous raw materials.
- Taking into account the final yield of bleached pulps from blends of wood with HFRMs and the reduction in the average fibre length, it follows that the replacement of part of birch or pine wood with hemp stalks is more favourable than that of wood with hemp woody-core.

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