Drewno 2022, Vol. 65, No. 209
DOI: 10.12841/wood.1644-3985.400.02

Dilek YAYLALI, Canan URAZ, Esat GÜMÜŞKAYA

EVALUATING THE PERFORMANCE OF HEMP BAST FIBRES IN THE PRODUCTION OF PACKAGING PAPER USING DIFFERENT WASTEPAPER BLENDS

The predicted scarcity of wood-based fibre supplies has necessitated the search for alternative sources including non-woods by paper manufacturers. As a raw material for pulping, hemp bast fibre has some potential. To provide a guide for the pulp and paper industries, the suitability of hemp bast fibres for the pulp and paper industries was evaluated in this study, and blending trials with wastepaper pulp were conducted. In the hemp bast fibre preparation, the length of the fibres was first reduced to 4 mm. Subsequently, kraft pulping was applied to them. Along with the preparation of wastepaper pulps, hemp bast fibres and wastepaper handsheets were prepared according to a blending plan. Finally, the handsheets were subjected to paper strength tests. The properties of raw, cooked and beaten hemp bast fibres were investigated by characterisation methods such as optical microscopy, X-ray diffraction and Fourier-transform infrared spectroscopy. Additionally, carbohydrate components, solubility and ash content tests were run on raw hemp bast fibres. As a result of the experiments and analyses, the targeted results were achieved. Following the resolution of the fibrillation problem, it was determined that hemp bast fibre pulp can be used in adequate blends of wastepaper pulp when the strength values of the end product are desired to be increased (including CMT, CCT, RCT, SCT, stiffness and tear resistance) or decreased (air resistance).

Keywords: Hemp bast fibre, wastepaper, pulp, packaging paper

Dilek YAYLALI (*dilek.chemeng@gmail.com*), Canan URAZ✉ (*canan.uraz@ege.edu.tr*), Ege University, Faculty of Engineering, Department of Chemical Engineering, İzmir, Turkey; Esat GÜMÜŞKAYA (*gkaya@ktu.edu.tr*), Karadeniz Technical University, Faculty of Forestry, Department of Pulp and Paper, Trabzon, Turkey

Introduction

Paper has a huge impact on the social and economic advancement of countries in terms of education, communication and industrialisation. The demand for wood in the pulp and paper industries is increasing globally as a result of population and economic growth [Bowyer 2001]. About 40% of the goods produced by the pulp and paper industries come directly from wood-based sources [Danielewicz and Surma-Ślusarska 2017; Dutt et al. 2008]. Research into the wood economy has long sought to improve the yield of fibrous semi-manufactured products. It is widely acknowledged that these methods will not be able to solve the anticipated problem of a future wood raw material scarcity for paper production, and it is necessary to modify the proposed area of research. In addition to the expected scarcity of the raw materials needed to produce pulp and paper, the increasing environmental consciousness of society has prompted paper manufacturers to search for alternative sources of virgin cellulose fibres including those derived from non-wood or agricultural sources. These factors led researchers in the 21st century to focus on non-wood fibre materials and recyclable paper wastes as major sources. Currently, 89% of the world's paper production is based on wood sources, and just 11% of the world's pulp production originates from non-wood sources [Malachowska et al. 2015]. Straw is currently the most commonly used non-wood fibre, although abaca, reeds, cotton, cotton linters, hemp, sisal, kenaf, sugar cane bagasse and bamboo are also major non-wood fibres for the pulp and paper industries [Plazonic et al. 2016; Ashori 2006]. Regardless of the origin of the fibre (wood, non-wood, agricultural residues, industrial residues, or wild plants), it is crucial to determine whether specific fibres contribute significantly to the strength and quality of the paper [Plazonic et al. 2016]. One of the major problems in the world today is the increasing volume of waste generated together with insufficient collection and recycling methods. Wastepaper recycling is important because it helps to preserve forest resources over the long run while reducing the continuous deposition of wastes into the environment. While the potential benefits of recycling may appear positive, the performance of the end-product may not seem to benefit as much from the use of recovered raw materials owing to the degradation in quality, compared to virgin materials. The use of industrial hemp (*Cannabis sativa*) for the production of pulp and paper is a trending issue since it has demanded advantageous properties such as strength, high fibre content and low lignin content [Malachowska et al. 2015]. Researchers have primarily focused on resolving the major limitations of hemp usage, including the low tendency of its fibres for internal fibrillation, low hemicellulose percentage and high cost of hemp pulp. Pulps made from hemp bast fibres must be subjected to beating after the pulping process to shorten and fibrillate the fibres. In this experiment, the hemp bast fibres were shortened to 4 mm just before pulping, and the effect of size reduction and beating with a Hollander-type beater

on the fibrillation of the fibres was assessed. Additionally, the drawbacks of using wastepaper during pulp preparation and the benefits of using hemp bast fibres pulp were demonstrated through pulp blending trials (hemp pulp and wastepaper pulp). These studies can contribute to the paper production industry, particularly in our country where it is based on the usage of wastepaper. The blending study results can be used to meet market demands for paper mills in terms of paper grade and required quality by suggesting that the degradation in recovered material quality may be compensated by blending with virgin material or that low cost may be obtained by blending the virgin source with the recovered source. This study is aimed at providing information to wood-based or wastepaper-based mills that lack wood-based pulp but are located in areas with non-wood fibre sources that are readily available. It also aims at demonstrating the feasibility of incorporating a non-wood pulping line to meet fibre demands.

Materials and methods

Commercial-grade hemp bast fibres were obtained from a hardware store. Wastepaper with the CEPI grade number '1.04.00' was randomly selected from the wastepaper stockpile of 'Selkasan Kağıt ve Paketleme Malzemeleri A.Ş.-Turkey'. Glycerine with a purity of 86.5% used during microscope imaging was supplied from Frank P-TI GmbH, Germany. Tap water was used as a fibre--waster suspension preparation aid and diluent. In this study, the preparation of hemp bast fibre pulps was divided into three steps (size reduction, kraft pulping and beating). Subsequently, hemp bast fibre and wastepaper handsheets were prepared by increasing the hemp bast fibre ratio from 0% to 100% and decreasing the ratio of wastepaper by 20%, along with the preparation of wastepaper pulps. Additionally, certain analyses were performed to determine the properties of cooked and beaten hemp bast fibres (fibre morphology, crystallinity and chemical changes). Finally, paper strength tests were run on the handsheets prepared according to the blending plan. According to the fibre classification results of Tutuş et al. [2016], hemp bast fibre pulps are among the long-fibred pulps. The size of the hemp bast fibres must be reduced before processes to prevent clumping. A fine cutting mill (NETZSCH, model type: CS-Z) was used to size-reduce the hemp bast fibres to 4 mm (processed twice). The size-reduced hemp bast fibres are denoted as 'raw hemp bast fibres' from this point forward. In this study, two batches of hemp bast fibre pulps were produced under the same conditions. The batches were cooked (pulping) in a batch digester, which rotates four times in one minute and has temperature control capabilities. For each batch, 500 g of oven-dry raw hemp bast fibres were charged into the digester. The conditions selected for kraft pulping are given in Table 1.

Table 1. Kraft pulping conditions

Cooking parameters	Unit	Value
Temperature	°C	170
Pressure	bar	10
Time required to reach max temp.	min	80
Pulping time	min	90
Total pulping time	min	170
Na_2S	%	5
NaOH	%	15.5
Liquor/raw material		5/1

The digested materials were washed with hot water to remove the dissolved substances and black liquor. After the removal of residual chemicals, the uncooked bundles of fibre were separated from the pulp and disintegrated using a lab-type disintegrator on a lab-type screen with a 0.15 mm slot width. The sieved parts were dried to a dry matter percentage of 20–25%, mixed, and then placed in polyethylene bags. The bag vent was left open for 24 h to achieve moisture equilibrium. The moisture content was determined using the TAPPI T 210 standard [TAPPI T 210:2003]. The dewatered pulp was stored in a refrigerator for further studies. The pulp yield for each batch was determined using the oven-dry weight of the hemp bast fibres that were initially charged into the digester. The pulp yields were calculated using the following equation [Ruraltech 2020]:

$$Yield = \frac{w_1}{w_2} x100 \quad (1)$$

where w_1 is the oven-dry weight of the hemp bast fibre pulp and w_2 is the oven--dry weight of the hemp bast fibres initially loaded into the digester.

Beating

The strength of a handsheet significantly depends on the strength and bonding capacity of the fibres. The cooked hemp bast fibres were beaten using a Hollander beater (L&W, model type: 3-1) for fibrillation. A suspension of tap water and cooked hemp bast fibre (pulp) with a consistency of 1.76% was prepared and used as the stock suspension for the blending studies. The prepared slurry was disintegrated in the raceway of the beater for 30 min before the beating motion was initiated by decreasing the distance between the beater wheel and plate. The beating degree of the pulp was determined using the Schopper Riegler-type beating degree tester (L&W, model: 2-4) and the TAPPI T 227 standard [TAPPI T 227:1999]. The beating degree was calculated as follows:

$$\text{Beating degree (°SR)} = \frac{(1000 - Vwater)}{10}, \quad (2)$$

where ‘Vwater’ is the volume of water drained in a measuring cylinder. After beating for 37 min, a 20°SR beating degree of hemp bast fibre pulp was achieved.

Preparation of wastepaper pulp

For each blending, the required amount of wastepaper was weighted and torn from a 3-ply corrugated cardboard-type box into pieces measuring about 3 cm × 3 cm. To easily separate the box piece layers, the box pieces were then soaked for 5 min in a 1 L beaker filled with tap water. The paper layers were separated and kept in the same beaker for an additional 10 min to fully absorb the water. Finally, the wastepaper was disintegrated using a 3-blade rotor-type pulp disintegrator (L&W, model:5-1) at 1150 rpm for 20 min to obtain wastepaper pulp.

Preparation of handsheets

To prepare each blending stock suspension, the required amounts of beaten hemp bast fibre pulp (20°SR) and prepared wastepaper pulp were diluted with tap water. The pulp was subsequently disintegrated using a 2-blade rotor-type pulp disintegrator (Frank PT-I) at 1250 rpm for 20 min. Handsheets were made on a sheet former (Rapid-Köthen type) according to the TAPPI T-205 sp-02 standard [TAPPI T 205:2002].

Optical microscopy (OM) measurements

To examine the morphology of the cooked and beaten hemp bast fibres (length, diameter and wall thickness) as well as manually quantify the size of the raw material, trinocular-type OM (SOIF, model: BK5000, software: MSHOT MicroShot v1.2 Image Processing System) was employed. A glycerine and water solution of 65% was prepared and used as a medium to distribute the overlapping fibres evenly on the slide. The diameter, wall thickness and length of the raw fibres were measured manually from the images taken. The repetition number was 75 for each size measurement type [Dutt et al. 2008].

Carbohydrate component, solubility and ash content tests of hemp bast fibre

Carbohydrate components, solubility and ash content tests of hemp bast fibre as a raw material were conducted by the department of forest industry engineering at Karadeniz Teknik University. Table 2 lists common test types and standard operating methods. Hemp bast fibres were sampled and prepared for chemical component analysis using the TAPPI T257 cm-08:2012 test method [TAPPI T 257:2012]. The moisture content for hemp bast fibres was determined based on the TAPPI T210 cm-03:2003 test method [TAPPI T210:2003]. Wise’s Chloride

method was used to determine the holocellulose content of the hemp bast fibres, and Kürschner–Hoffner's approach was used to determine the cellulose content [Browning 1967]. The TAPPI T 222 om-11:2011 test method was t determine the amount of acid-soluble lignin in the hem bast fibres [TAPPI T 222:2011]. The hemp bast fibre ash content was determined using the TAPPI T 211 om-93:1993 test method [TAPPI T 211:1993]. The solubility of the hemp bast fibres was tested using the following TAPPI test methods: TAPPI T 204 cm-07:2007 [TAPPI T 204:2007] for solvent extractives of hemp bast fibres, TAPPI T 207 cm-08:2008 [TAPPI T 207:2008] for water solubility of hemp bast fibres and TAPPI T 212 om-12:2012 [TAPPI T 212:2012] for 1% sodium hydroxide solubility of hemp bast fibres.

Table 2. The applied standard test methods for determination of carbohydrate components, solubility and ash content of hemp bast fibre

Carbohydrate components tests	Applied standard
Holocellulose	Wise's chlorite method
Cellulose	Kurschner-Hoffner method
Lignin	TAPPI T-211 om-88
Solubility and ash content tests	**Applied standard**
Hot water solubility	TAPPI T-207 om-88
NaOH %1 solubility	TAPPI T-212 om-88
Alcohol-benzene solubility	TAPPI T-204 om-88
Ash content	TAPPI T-207 om-88

X-ray diffraction (XRD) measurements

The degree of crystallinity is determined by the crystallinity index (CrI), which is useful for grading the crystallinity of various cellulose types and observing the effects of physical and chemical treatments on crystallinity [Gümüşkaya and Usta 2006]. The crystallisation behaviours of raw, beaten and cooked hemp bast fibres were analysed by XRD patterns, and the degree of crystallinity was calculated using the patterns. The patterns were captured using a Panalytical Empyrean XRD system with a Cu Kα radiation source running at 45 kV voltage and 40 mA current. The patterns were obtained in the 2θ = 3–44° range. The CrI of the samples were calculated using the following equation, which was proposed by Segal et al. [1959] for native cellulose:

$$\mathrm{CrI}(\%) = \frac{\mathrm{I002-Iamorph}}{\mathrm{I002}} x100, \quad (3)$$

where I_{002} represents crystalline and amorphous regions of cellulose (max intensity at 2θ = 22,7°) and I_{amorph} represents the merely amorphous phase (intensity of the diffraction at 2θ = 14,8°). All experiments were repeated twice.

Fourier-transform infrared (FTIR) spectroscopy analysis

The functional groups of the raw, beaten and cooked hemp bast fibres were characterised by FTIR analysis using an FTIR spectrometer (PerkinElmer FTIR Spectrum 65). According to Zhbankov [1966], a layer of parallel, closely packed fibres was used for the investigation of the fibrous material, and 32 scans with a resolution of 0.5 cm^{-1} and absorption between 4000 and 600 cm^{-1} were taken for each sample.

Physical strength tests

Handsheets were conditioned for 24 h in an atmosphere (23 ± 1 °C, 65% relative humidity) following TAPPI T 402 om-88 [TAPPI T 402: 2008]. The average mass per unit area of the conditioned sheets was determined and recorded for the index calculations. Physical strength tests were run following the TAPPI T sp-220 standard [TAPPI T 220: 2001]. The physical strength tests were done in Selkasan Kağıt's laboratory (L&W testers). Thereafter, all of the test results were indexed using the conditioned mass per unit area of the test sheets. Finally, the indexed results were reported as the average of 6 replicates.

Results and discussion

Carbohydrate components, solubility and ash content

Carbohydrate components, solubility and ash content test results of the raw material hemp bast fibre are shown in Table 3. Similar carbohydrate content and solubility results were obtained in the study of Gümüşkaya and Usta [2006]. Hemp bast fibres were found to contain more holocellulose and cellulose than wood and several non-wood sources while having less lignin. The ash content of the hemp bast fibres was found to be 1.5% by Danielewicz and Surma-Ślusarska [2017] and 2.17% by Tutuş et al. [2016].

Table 3. Carbohydrate and ash content test results of hemp bast fibres

Test type	Content (%)
Holocellulose	87.7
Cellulose	74.8
Lignin	9.1
Ash content	3.5

These values are less than the ash content in this study (3.5%). These differences can be attributed to the different types of hemp bast fibre sources used. Tutuş et al. [2016] reported that the ash content of wood-based fibre sources ranges between 0.2% to 0.7%, and the ash content ranges between 2% and 7% for non-wood-based sources. Compared to wood-based sources, hemp bast fibres have high ash contents, although it is not as high as some non-wood fibre sources such as sunflower (7%–10%) and straw (4.9%) fibres [Gümüşkaya 2002]. The solubility test results of the hemp bast fibres (in this study) and some other fibre sources are presented in Table 4.

Table 4. Solubility test results of hemp bast fibers and some other fibre sources

	Solubility (%)		
Test type	Alcohol-benzene	Hot Water	%1 NaOH
Hemp bast fibers	3.8	9.3	24.1
Wheat straw	5.3	10.5	40.1
Hardwoods	2.3-13.5	1.1-13.8	8-19
Softwoods	0.4-4.7	0.6-12.8	11-22

The cell wall contents of non-wood sources have an impact on the solubility of hemp bast fibres, as do their morphological and structural properties [Gümüşkaya 2002]. Table 4 shows that hemp bast fibres are more soluble than hardwood and softwood fibre sources, but they are less soluble than wheat straw fibres. These aspects allow us to conclude that hemp bast fibre is an appropriate fibre source for the pulp and paper industry.

Kraft pulping and beating

Figure 1 depicts the colour change of the hemp bast fibres after different treatments. The colour of the hemp bast fibres (size-reduced raw material) was yellowish-brown before pulping. After pulping, the colour became lighter (cream). After beating, the whiteness increased.

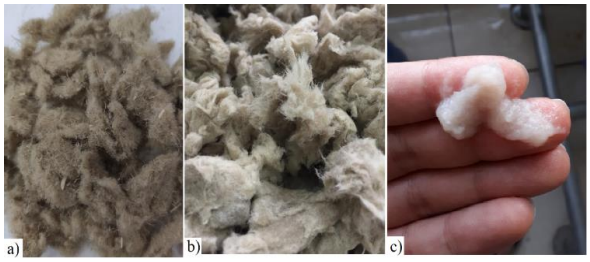

Fig. 1. The appearance of hemp bast fibres a) after size reduction, b) after pulping and c) after beating

According to Chieng et al. [2017], the degradation in cellulose, hemicellulose, and lignin after certain treatments is relevant to the change in fibres, and degradation is also associated with weight loss and changes in the chemical compositions of fibre. The yield of a given raw material is a very important factor that influences the profitability of processing the fibre source. The calculated pulping yields for the two batches were 70.4% and 71.2%. The average value was 70.8%, which is a high yield value for cooking. According to Cheremisinoff and Rosenfeld [2010], the yields between 40% and 55% ranged as low. According to the study results of Danielewicz and Surma-Ślusarska [2010], hemp bast fibre kraft pulp has a higher yield than commonly used woods. In their study, they evaluated the kraft pulping yields of hemp bast fibres, birch and pine and found that the yields were 73% (Kappa no: 20), 52% (Kappa no: 20) and 44% (Kappa no: 30), respectively. The high yield might be due to the low lignin and hemicellulose content of hemp bast fibres, most of which are dissolved during cooking. If hemp bast fibres contained a high amount of these substances, the yield would have been naturally lower. Additionally, the high amount of crystalline and highly stable crystalline structure of cellulose in the raw material has a negative impact on the accessibility and swelling of the cellulose fibres, which in turn reduces the rate at which other substances dissolve and the amount of cellulose that is lost during cooking [Gümüşkaya et al. 2007]. As a result, the yield increases.

Fibre fibrillation significantly affects the strength of the end product. The properties of cellulosic fibres are greatly optimised by beating or refining to remove primary fibre walls. This allows the fibres to become hydrated before swelling, increasing their flexibility and bonding power [Kumar Agrawal et al., 2015]. According to Danielewicz and Surma-Ślusarska [2017], the freeness value of pulp exceeded 23°SR after beating for 45 min. Therefore, to determine the mechanical treatment that the pulp had been subjected to, the beating degree was measured repeatedly until the targeted 20–25°SR was reached (hemp bast fibre pulp consistency: 1.76%). As with their investigation, it took 37 min to achieve the 20°SR. This was due to the highly stable crystalline structure and low hemicellulose content of hemp bast fibres [Gümüşkaya et al., 2007]. These properties caused a decrease in swelling capacity, which lowered the external fibrillation of the hemp bast fibres. This phenomenon leads to high energy consumption for beating since it takes too much processing time to reach the desired fibrillation degree.

OM analysis

The results of 75 measurements of size-reduced raw hemp bast fibres were analysed, and histograms were tabulated in terms of diameter, wall thickness and length. The master thesis of Yaylali [2020] contains the histograms for length,

diameter and wall thickness. In this study, a targeted raw hemp bast fibre size of 4 mm on average was attained using a fine cutting mill. The lengths of the hemp bast fibres ranged from 2.2 to 8.6 mm with an average length of 4.9 mm. The average value deviated by 22.5% from the targeted average value. The highest percentage (24%) of fibre lengths was within the 5 mm range. The length distribution was primarily found in the 5–6 mm range. These results and the average value indicate that despite processing the fibres twice, the targeted average length of hemp bast fibres was achieved using the fine cutting mill. For further studies, other size reduction methods can be considered for better precision. The diameters of the hemp bast fibres ranged from 11 to 39 μm with an average diameter of 23 μm. The highest percentage (24%) of the fibre diameter was in the 26 μm range.

The cell wall thickness of the hemp bast fibres ranged from 3.2 to 9.2 μm, with an average wall thickness of 6 μm, and the highest percentage (24%) of fibre wall thickness was in the 5 μm–7 μm range. The average cell wall thickness determined in this study was 10.4% lower than that in the results of Dutt et al. [2005] and 9.1% higher than that in the results of Danielewicz and Surma-Ślusarska [2017]. These differences in diameter and cell wall thickness of hemp bast fibres could be due to the OM measurement methods or the nature of the raw materials. The results indicate that there may be some variations in hemp bast fibre cell wall thickness amongst different cultivars of hemp. The cultivation conditions, harvesting time, subgenre difference, etc. also affect this phenomenon. The method of producing pulp from hemp stalks is also effective in this difference.

Figure 2 presents OM images of hemp bast fibres before and after treatments as well as the images of hemp bast fibres in (a) raw, (b) cooked and (c) beaten forms.

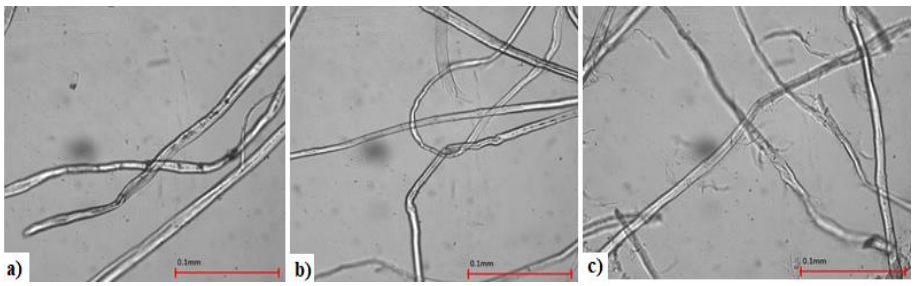

Fig. 2. Optical microscope images of (a) raw, (b) cooked and (c) beaten hemp bast fibres

These figures show that cooking has no effect on fibre fibrillation, but it is apparent that beating has some fibrillation effect on the cooked hemp bast fibres. Since the low degree of external fibrillation after beating, it seems to be the main problem for the wide usage of hemp bast fibres.

XRD analysis

XRD measurements were performed to investigate the change in crystallinity of the hemp bast fibres after different treatments. Figure 3 shows the XRD patterns of raw, cooked and beaten hemp bast fibres.

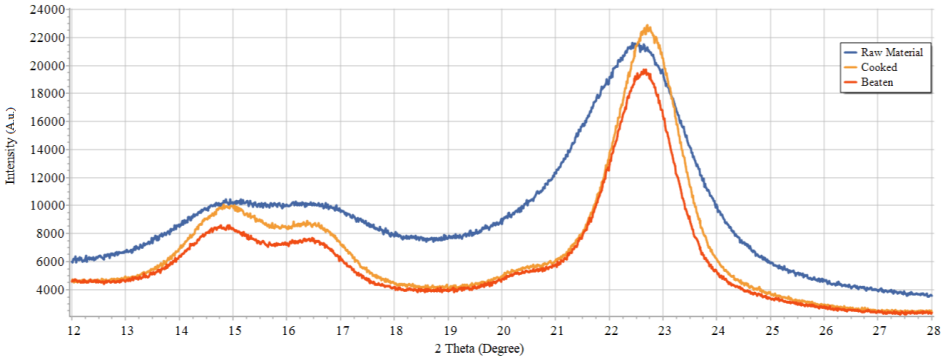

Fig. 3. X-ray diffraction patterns of raw, cooked and beaten hemp bast fibres

According to Fig. 3., the XRD patterns of the hemp bast fibres (raw, cooked, beaten) exhibited identical peaks at three different planes, including 2θ = 14.8° (plane 101), 2θ = 16.5° (plane 101) and 2θ = 22.7° (plane 002). Peaks at 14.8° and 16.5° were more intense for cooked and beaten hemp bast fibres when compared to raw material. During cooking, hemicellulose and lignin were dissolved, leaving just the crystalline components. Since the amorphous parts are effectively removed by this phenomenon, the peaks were sharper. The degree of crystallinity of raw, cooked and beaten hemp bast fibres were 51%, 56% and 57%, respectively (according to Segal's method). The results show that the degree of crystallinity of hemp bast fibres increased after cooking. Gümüşkaya and Usta [2006] also stated that the degree of crystallinity of hemp bast fibre increases when the amorphous parts of cellulose are chemically degraded. The crystallinity of cooked hemp bast fibres increased by 1% after beating. The number of amorphous regions increased after beating the cooked hemp bast fibres, resulting in a decrease in the degree of crystallinity [Eroğlu and Usta 2004]. This unexpected increase in the degree of crystallinity indicated that the applied beating did not affect the fibrillation of the fibres, as also indicated by the OM results of this study.

FTIR analysis

The raw material contained hemicellulose and lignin, and since pulping is meant to remove lignin and hemicellulose, the interpretation of the FTIR analysis results

was useful for examining the changes in functional groups. Figure 4 shows that the raw, cooked and beaten hemp bast fibres had peaks at about 3338 cm^{-1}, indicating the presence of cellulose polymeric OH stretches [Menon et al. 2018].

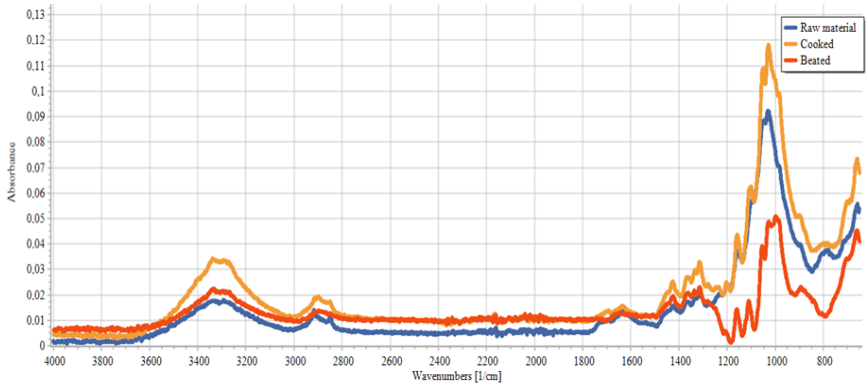

Fig. 4. FTIR spectral plot of raw, cooked and beaten hemp bast fibres

The peaks at 2900 cm^{-1} were due to stretching vibrations of the C-H functional group. Also, the absorbance peaks between 1500 and 900 cm^{-1} indicate the C-O-C functional group (1050 cm^{-1}, 1028 cm^{-1} and 984 cm^{-1}). These functional groups do not conflict with the base structure of the cellulose molecule [Munajad et al. 2018]. When cooked, the intensity of the peak at wavenumber 3338 cm^{-1} increases due to an increase in OH concentration as the alkaline weakened the hydrogen bonding in cellulosic hydroxyl groups [Chieng et al. 2017]. After beating, the peak intensity of the fibre was lower than that of the cooked fibres because of the increase in hydrogen bonding. The peak, which only appears in the raw material spectrum at 1732 cm^{-1}, corresponded to the C=O stretching of carbonyl functional groups from hemicellulose and lignin fractions. Cooking causes the removal of carboxylic groups, which may be traces of fatty acids on the fibre surface, and causes the C=O stretching to vanish [Chieng et al. 2017; Munajad et al. 2018]. The raw material spectrum at 1232 cm^{-1}, which corresponds to the syringyl ring and C–O stretching of lignin and xylan, declined in intensity before decreasing, demonstrating that lignin and a small amount of hemicellulose were removed from the raw material after treatment [Chieng et al. 2017; Zhbankov 1966]. At 900 cm^{-1} in the spectrum, the peak of beaten fibre was sharper because the cellulose was ground up. Therefore, the band around 900 cm^{-1} is only referred to as ‘amorphous’ because its intensity increased [Zhbankov 1966].

Wastepaper and handsheet preparation

In this study, wastepaper pulp and hemp bast fibre pulp (20°SR) were used to prepare handsheets. The wastepaper grade number 1.04.00 was chosen because it

is one of the lowest quality and abundant wastepaper grades. This choice supports this study's aim of compensating for the diminishing strength properties of wastepaper, which is typically observed when using lower-grade wastepaper. During the wastepaper pulp preparation step, some impurities/contaminants were observed on the wastepaper. Since the wastepaper was in a corrugated box, there were rusty staples and adhered box sealing tapes. The box was also mouldy and contained some nits inside of the corrugated part. To obtain real-condition results, the wastepaper pieces were torn from these contaminated parts of the box by way of sampling. After the wastepaper ply-pieces had been soaked in water for 10 min, it was observed that the corrugated medium plies did not absorb water as much as the liner plies, as shown in Fig. 5.

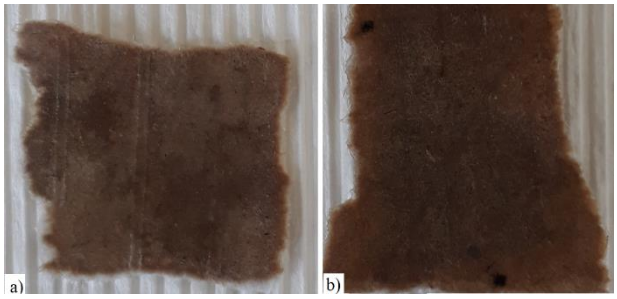

Fig. 5. The water absorption difference between a) corrugated medium ply and b) face ply

Sizing chemicals are used to reduce the water permeability of paper. This corrugated medium might have been highly sized, so the rewetting for 10 min was insufficient for this paper ply compared to face paper.

Figure 6 depicts the physical characteristics of handsheets with different hemp bast fibre percentages. There are some brown speckles on the handsheets shown in Fig. 6, particularly on the handsheets with 40%, 60% and 80% hemp bast fibre. These speckles are the paper clumps that were not disintegrated during wastepaper preparation and handsheet formation steps. The disintegration problem of the wastepaper was due to the low water absorption of the corrugated medium plies that did not dissociate completely during disintegration. In addition, few white speckles were smaller than the aforementioned brown speckles. These white speckles must have originated from the white top face ply of the corrugated wastepaper. The white top face ply differs from the brown ply in nature of the fibre (the white fibres are bleached) and may also contain fillers such as calcium carbonate [Eroğlu and Usta 2004]. These differences could be the cause of this slight disintegration problem of the white top face ply of the wastepaper corrugated box. The handsheet with 0% hemp bast fibre (100% wastepaper) had the same colour as wastepaper pulp. Since the hemp bast fibre pulp has white

colour, the colour of the resultant handsheet lightened as the hemp bast fibre percentage increased (from 0% to 100%).

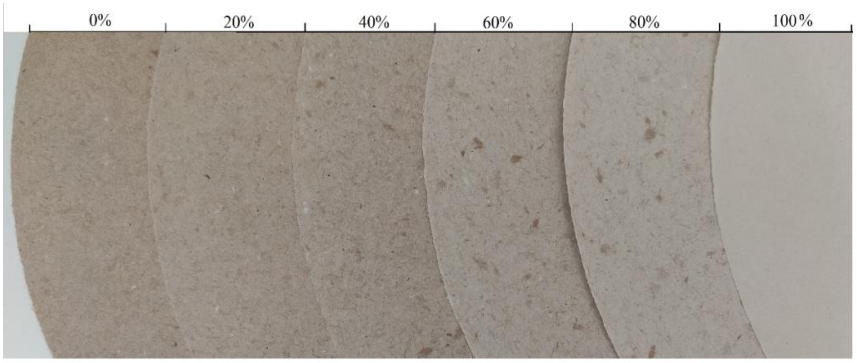

Fig. 6. The physical appearance of the hand sheets

Physical strength tests

In this study, physical strength tests were performed on the handsheets produced from wastepaper pulp and hemp bast fibre pulp (20°SR). The effect of the hemp ratio of the blend on the CMT, CCT, RCT, SCT and stiffness values of the papers were investigated, and the graphical results are included in the thesis of Yaylali [2020]. As shown in Fig. 7, the CMT, CCT, RCT SCT and stiffness indexes increase as the hemp bast fibre pulp ratio in the paper increases. The subsequent positive increase in the resultant compressive and bending strengths of the paper was due to the high stiffness of hemp bast fibres and the drawbacks of wastepaper. Hemp bast fibres are similar to pine in length and have thick cell walls. These properties increase the stiffness of hemp bast fibres. Recycling wastepaper results in flexibility decline, hornification, decreased fibre length and increased fines content [Kumar et al. 2020].

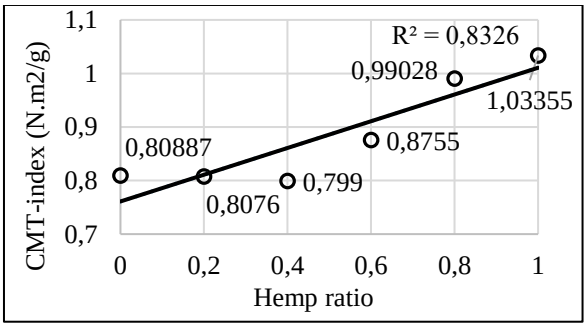

Fig. 7. The effect of hemp ratio of blend on the CMT value of paper

When paper made entirely from wastepaper (0% hemp) was compared with paper made with 100% hemp, the increases in paper indexes were as follows: 27.8% for CMT, 26% for CCT, 27.2% for RCT, 23.5% for stiffness and 6.2% for SCT. These increases are not significant because it was expected that blending with hemp bast fibres would considerably increase the compression strength values of the paper. Numerous analyses have shown that the main cause of this low increase is a fibrillation issue with the hemp bast fibres. The tear resistance index normally increases as the hemp bast fibre ratio in the paper increases [Yaylali 2020]. Danielewicz and Surma-Ślusarska [2017] suggested the same phenomenon with this study on adding hemp bast fibre pulp in small amounts to pulp with low tear strength, such as wastepaper pulp, to improve this property. The increase for paper made from 100% hemp compared to that of 0% hemp was 10.8% and 15.4% in the cases of paper made from 40% and 60% hemp, respectively. In contrast, the paper made of 80% hemp had a 24.6% higher tear resistance index than paper made of 0% hemp. This low and imbalanced increase in tear resistance index could be due to the low external fibrillation of the hemp bast fibres after beating. Numerous authors have noted that the hemicellulose content and consequently the degree of external fibrillation of fibres increase the tensile strength of paper [Gümüşkaya et al. 2007; Danielewicz and Surma-Ślusarska 2017]. During pulping and mechanical refining, hemicelluloses are absorbed into fibre surfaces, where they may facilitate inter-fibre bonding. Owing to their non-crystalline hydrophilic nature, they may also contribute to pulp swelling and facilitate sheet formation [Kalia et al. 2011; Kumar Agrawal et al. 2015]. Hemp bast fibres exhibit low external fibrillation due to their highly stable crystalline structure and low hemicellulose content, which aids swelling. In this study, these results are consistent with the drawbacks of hemp bast fibres. Breaking length and stretch indexes decrease generally with an increase in the hemp bast fibre ratio of the paper [Yaylali 2020]. The decrease in breaking length and stretch index of paper was 19.6% and 24%, respectively, for the 100%-hemp and 0%-hemp papers. With an increase in hemp bast fibre ratio, the 'low degree of external fibrillation of hemp fibres' phenomenon also generally causes a decline in the bursting strength index of paper [Yaylali 2020]. In the case of the 100% hemp sample (compared to the 0% hemp sample), there was a 22.6% fall in the paper bursting strength index. This was due to the inter-fibre bonding decreasing as the hemp bast fibre ratio increased. The air resistance index (passage time of 100 ml in air through the paper per g/m^2) generally decreases as the ratio of hemp bast fibres in the paper increases [Yaylali 2020]. Compared to the 0% hemp, the decrease in the air resistance index of the paper was 68.9% for the 100% hemp sample. Since hemp bast fibre pulps have low specific gravity and high bulk with open and looser fibre structures, papers made from hemp bast fibres have high air permeability and low air resistance [Ashok Kumar et al. 2017; Dutt et al. 2009]. In addition, wastepaper contains fines that are higher than those in refined

virgin pulp due to cycles of reuse. These fines block inter-fibre vacancies, and the hornification of the fibres causes the internal pore structure to collapse [Kumar et al. 2020]. The low extent of fibrillation of the hemp bast fibres after beating is also beneficial in this decrease since there are large pores between the fibres as they are not fibrillated at the desired level, allowing free flow of air to pass through the paper.

Conclusions

The conclusions for each examination topic are as follows:

- Hemp bast fibre is an appropriate fibre source for the pulping and papermaking industries in terms of holocellulose, cellulose, lignin content, ash and solubility.
- Hemp bast fibres are initially yellowish-brown in colour, but they become lighter (cream) after cooking and even lighter after beating.
- When compared to wood-based sources, the yield of kraft cooking is determined to be 70.8%, which is a high yield value.
- The low hemicellulose content and highly stable crystalline structure of hemp bast fibres reduce external fibrillation. Since it takes too long to process to reach the desired degree of fibrillation, it consumes a lot of energy to beat.
- With the use of a fine cutting mill, the desired average length of hemp bast fibres was successfully obtained. Alternative size reduction methods can be considered for better precision. The diameter and cell wall thickness of the hemp bast fibres used in this work were consistent with those in other studies. Cooking did not affect the fibrillation of the fibres; however, applied beating had some fibrillation effect on the cooked hemp bast fibres, but it was less than that of wood sources.
- According to XRD results, the degree of crystallinity of hemp bast fibres increases after cooking owing to degradation in the amorphous domains of cellulose. The unexpected increase in crystallinity indicates that the applied beating did not affect the fibrillation of the fibres, as supported by the OM results.
- FTIR results confirm the removal of lignin and small amounts of hemicellulose from the raw material after treatment.
- During the preparation of wastepaper pulp, some impurities/contaminants were observed. There were some apparent brown and white speckles on the handsheets made from wastepaper pulp and hemp bast fibre pulp, especially on the handsheets with 40%, 60% and 80% hemp bast fibre, due to a disintegration problem with wastepaper. This problem was due to low water absorption of the corrugated medium and white top face plies

that were not dissociated completely during the disintegration phase. These observations confirm the problematic nature of using wastepaper as a fibre source.

- The colour of the handsheet made of 0% hemp bast fibre was similar to that of wastepaper pulp. Additionally, since the hemp bast fibre pulp has a white colour, increasing the hemp bast fibre ratio lightens the colour of the resultant handsheet.
- According to the physical strength test results, CMT, CCT, RCT, SCT, stiffness and tear resistance increase as hemp bast fibre ratio in the paper increases, while breaking length, stretching percentage, bursting strength and air resistance decrease.

Overall, it was concluded that in addition to the size of the fibres, the carbohydrate components and the crystalline structure of the hemp bast fibre have an impact on the beating degree and fibrillation, which in turn affects the strength properties. Furthermore, it was determined that some strength properties of the paper increase as others decrease when the hemp bast fibre ratio is increased. The results can be a guide for the pulp and paper industries by showing that the hemp bast fibre pulp can be used in proper blends of wastepaper pulp when it is desired to increase the end product's (paper) strength values (such as CMT, CCT, RCT, SCT, stiffness or tear resistance) or decrease them (e.g. air resistance) after resolving the fibrillation problem. The conducted experiments and their results lay the groundwork for process optimisation and implementation under industrial conditions in the future.

References

Ashok Kumar T.N.V., Subash Kumar J. and Vijay Kumar J. [2017]: Fabrication and investigation of mechanical properties hemp fibre reinforced polyester composites. Science and Technology 2 [3]

Ashori A. [2006]: Nonwood fibres – A potential source of raw material in papermaking. Polymer-Plastics Technology and Engineering 45

Bowyer J.L. [2001]: Industrial hemp as a papermaking raw material in Minesota: technical, economic and environmental considerations] access date 17 June 2019 https://www.votehemp.com/wp-content/uploads/2018/09/hemp.pdf

Browning B.L. [1967]: Methods of Wood Chemistry. Vol. II. John Wiley and Sons Inc. New York

Cheremisinoff N.P. and Rosenfeld P.E. [2010]: Handbook of Pollution Prevention and Cleaner Production, Elsevier Inc.

Chieng B.W., Lee S.H., Ibrahim N.A., Then Y.Y. and Loo Y.Y. [2017]: Isolation and characterization of cellulose nanocrystals from oil palm mesocarp fibre. Polymers 9 [355]

Danielewicz D. and Surma-Ślusarska B. [2010]: Processing of industrial hemp into papermaking pulps intended for bleaching. Fibres and Textiles in Eastern Europe 18-6 [83]

Danielewicz D. and Surma-Ślusarska B. [2017]: Properties and fibre characterisation of bleached hemp, birch and pine pulps: A comparison. Cellulose 24

Dutt D., Upadhyaya J.S., Malik R.S. and Tyagi C.H. [2005]: Studies on the pulp and papermaking characteristics of some Indian non-woody fibrous raw materials. Cellulose Chemistry and Technology 39 [115]

Dutt D., Upadhyaya J.S., Tyagi C.H., Kumar A. and Lal M. [2008]: Studies on *Ipomea carnea* and *Cannabis sativa* as an alternative pulp blend for softwood: An optimization of kraft delignification process. Industrial Crops and Products 28

Dutt D., Tyagi C.H., Agnihotri S., Kumar A. and Siddhartha A. [2009]: Alkoxygen and alkoxygen-AQ delignification of *Ipomea carnea* and *Cannabis sativa* Indian Journal of Chemical Technology 16

Malachowska E., Przybysz P., Dubowik M., Kucner M., Buzala K. [2015]: Comparison of papermaking potential of wood and hemp cellulose pulps. Annals of Warsaw University of Life Sciences-SGGW 91

Eroğlu H. and Usta M. [2004]: Kağıt ve Karton Üretim Teknolojisi Esen Ofset Matbaacılık Trabzon, 1

Gümüşkaya E. [2002]: Kendir (*Cannabis sativa* L.) soymuk liflerinden asidik ve alkali ortamlarda üretilen kağıt hamurlarının kimyasal ve kristal yapı özellikleri (Chemical and crystalline structure properties of hemp (*Cannabis sativa* L.) bast fibres pulp obtained with alkaline and acidic processes. Doctor of Philosophy Thesis, Karadeniz Teknik University

Gümüşkaya E. and Usta M. [2006]: Dependence of chemical and crystalline structure of alkali sulfite pulp on pulping temperature and time. Carbohydrate Polymers 65

Gümüşkaya E., Usta M. and Balaban M. [2007]: Carbohydrate components and crystalline structure of organosolv hemp bast fibres pulp. Bioresource Technology 98

Kalia S. (ed.) [2011]: Cellulose fibers: bio- and nano-polymer composites. Springer, Berlin, pp 123–146. doi:10.1007/978-3-642-17370-7

Kumar Agrawal N., Agarwal A. and Mukherjee S. [2015]: Chemistry of raw materials for electrical grade paper: An overview. International Journal of Engineering Technology, Management and Applied Sciences 3 [2]

Kumar V., Pathak P. and Bhardwaj N.K. [2020]: Waste paper: an underutilized but promising source for nanocellulose mining. Waste Management 102

Menon A., Akhil T. and Prasad R.K. [2018]: Spectral characterization and surface morphology of delignification of kraft pulp with carbamide peroxide. Journal of Environmental Chemical Engineering 6

Munajad A., Subroto C. and Suwarno [2018]: Fourier-transform infrared (FTIR) spectroscopy analysis of transformer paper in mineral oil-paper composite insulation under accelerated thermal aging. Energies 11 [364]

Plazonic I., Barbaric-Mikocevic Z., Bates I. and Malnar L. [2016]: Chemical stability of prints made of hemp fibre based papers. Acta Graphica 27 [3]

Ruraltech [Pulp and Paper] access date 31 March 2020 http://www.ruraltech.org/projects/conversions/briggs_conversions/briggs_ch08/chapter08_combined.pdf

Segal L., Creely J.J., Martin Jr. A.E. and Conrad C.M. [1959]: An Empirical Method for Estimating the Degree of Crystallinity of Native Cellulose Using the X-Ray Diffractometer. Textile Research Journal 29: 786-794. doi: http://dx.doi.org/10.1177/004051755902901003

Tutuş A., Karataş B. and Çiçekler M. [2016]: Pulp and paper production from hemp by modified kraft method. 1st International Mediterranean Science and Engineering Congress Adana

Yaylali D. [2020]: Evaluation of the performance of hemp bast fibres in the production of packaging paper with different waste paper blends, Master Thesis, Ege University

Zhbankov R. G. [1966]: Infrared spectra of cellulose and its derivatives. Springer, Minsk

List of standards

TAPPI T 204 cm-07:2007 Solvent Extractives of Wood and Pulp
TAPPI T 205:2002 Forming handsheets for physical tests of pulp
TAPPI T 207:2008 Water solubility of wood and pulp
TAPPI T 210:2003 Sampling and testing wood pulp shipments for moisture
TAPPI T 211 om-93:1993 Ash in Wood, Pulp, Paper and Paperboard: Combustion at 525 Degrees Celsius
TAPPI T 212 om-12:2012 One Percent Sodium Hydroxide Solubility of Wood and Pulp
TAPPI T 220:2001 Physical testing of pulp handsheets
TAPPI T 222 om-11:2011 Acid-Insoluble Lignin in Wood and Pulp
TAPPI T 227:1999 Freeness of pulp (Canadian standard method)
TAPPI T 257 sp-12:2012 Sampling and Preparing Wood for Analysis
TAPPI T 402:2008 Standard conditioning and testing atmospheres for paper

Acknowledgements

The authors acknowledge the technical support from Selkasan Kağıt ve Pak. Malz. San. ve Tic. A.Ş. and the financial support from Ege University Scientific Research Fund under project no. FYL-2019-21080. We are grateful to Ege University Planning and Monitoring Coordination of Organizational Development and Directorate of Library and Documentation for their support in editing and proofreading service of this study.

Submission date: 27.08.2021

Online publication date: 31.07.2022