

Sustainable Paper-based Packaging from Hemp Hurd Fiber: A Potential Material for Thermoformed Molded Fiber Packaging

Joe C. H. Law,^{a,b,#} Kelly R. Wade,^{a,*} Kate G. Parker,^a Anthony N. Mutukumira,^b and Michelle Sloane^a

Hemp hurd fiber, a low-value waste stream from the hemp industry, has potential downstream applications as an alternative to non-renewable plastics for single-use food service ware and packaging applications. Packaging paper substrates made from chemically pulped hemp hurd, mixed in varying ratios with bleached thermomechanical radiata pine pulp were developed and tested. Handsheets were characterized using several mechanical property tests including tensile strength, tearing resistance, burst strength, short-span compression, ring crush, together with Gurley air resistance, contact angle, and Cobb₆₀ tests. Generally, addition of hemp hurd fibers significantly improved handsheet mechanical properties. Hot-pressing of the handsheets so as to approximate molded fiber thermoforming further enhanced their performance, with pure hemp hurd handsheets having the highest mechanical properties and barrier performance. A prototype was successfully thermoformed from hemp fiber, demonstrating overall feasibility of this fibre source for molded fibre objects.

DOI: 10.15376/biores.19.1.1728-1743

Keywords: Industrial hemp hurd; Handsheet; Paper; Thermoforming; Mechanical properties

Contact information: a: Scion, Titokorangi Drive, Private Bag 3020, Rotorua 3046, New Zealand;

b: School of Food and Advanced Technology, Massey University, Auckland 0632, New Zealand;

#: This author subsequently had a legal change to their name;

* Corresponding author: Kelly.wade@scionresearch.com

INTRODUCTION

There is currently a global focus on reducing or eliminating single-use plastic items to minimize consumption of non-renewable resources and associated environmental impacts. Given their sustainable properties, fiber-based materials offer an appealing alternative to single-use plastic items. Wood is the primary feedstock utilized by the pulp and paper industry but is a scarce resource in some parts of the world (Amode and Jeetah 2021). Alternative biomasses, such as hemp hurd, have been considered for traditional pulp and paper applications. Use of such materials could help fulfill fiber shortfalls. Since these waste fiber streams are typically available in relatively small quantities, they are well suited to molded fiber processes, which can operate at small scale. Molded fiber manufacturing processes range from simple suction molded, air-dried objects such as egg trays to more complex thermoforming processes in which pulp is formed, then dried rapidly under high temperatures and pressures in smooth, heated molds. The latter approach produces stronger, more intricate, and attractive structures.

Industrial hemp (*Cannabis sativa* L.) is a natural fiber with a long history of cultivation with many applications including papermaking. Hemp is a sustainable, low

environmental impact crop that can be harvested after only four months of cultivation compared to forests, whose cycles can take decades. Hemp stalks have a layered structure, with bark surrounding an outer layer of bast fiber and an inner woody core, also known as hurd. Hemp bast is typically used as a raw material for papermaking (Ahmed *et al.* 2022), but hurd fiber, which typically contains cellulose (40 to 48%), hemicellulose (18 to 24%), and lignin (21 to 24%), is usually regarded as waste due to its inferior performance for papermaking (Bokhari *et al.* 2021). In addition to potential issues associated with separating the hurd and bast fibres, hemp hurd paper has been shown to have low tear strength, along with a low drainage rate, which limits the production rate (Danielewicz and Surma-Ślusarska 2017).

Employing hurd as a component of moulded fibre products, which have less stringent performance requirements and a more forgiving production process, may mitigate these factors. Blending hurd with other, longer fibres could improve mechanical performance, though there would still be a reduction in pulp freeness which would need to be accounted for during production. Another factor to consider is that thermoformed moulded fibre products are typically used for high-end applications such as electronics packaging, where smooth, detailed surfaces are required (Didone *et al.* 2017). The relatively small hemp hurd fibres would be expected to be favourable in this application.

This study involved hemp hurd and radiata pine as raw materials for producing fiber products. Molded fiber products were exemplified by handsheets, with a prototype cup product developed to serve as a proof of concept. Hemp hurd was pulped using the cold-soda process and blended with bleached-chemi-thermomechanical pulp (BCTMP) from radiata pine. The morphology of both pulps was determined along with the mechanical characteristics and barrier performance of handsheets. Selected handsheets were subjected to hot-pressing regimes to simulate molded fiber thermoforming processes. In addition, mixed pulp handsheets with varying pulp ratios, both control and hot-pressed, were examined to evaluate the bonding interactions of hemp hurd and pine within the product and to simulate the effects of thermoforming on the product.

EXPERIMENTAL

Preparation of Pulps

Hemp hurd pulp (HHP)

Hemp hurd was obtained from Hemp New Zealand™ Ltd., located in Tauranga, New Zealand. A soda pulping process was used with the addition of 16% sodium hydroxide (32 g NaOH) loading on the oven dry mass of hemp hurd (200 g). During pulping, cooking liquor (10 × dry fiber mass w/w) was added. The temperature was steadily increased from room temperature to 170 °C in the course of 90 min, then held for a further 90 min. The resulting pulp was defibered in a laboratory Bauer 8" refiner at approximately 2% consistency with a plate gap of 0.1 mm for four passes. Pulp was then screened using a Packer vibrating flat screen with a slot width of 0.025 mm to remove shive materials. The screened yield was 46.7% and rejects an additional 8.5%, to give a total yield of 55.2%. These conditions were chosen based on our past experience conducting pulping trials on hemp hurd and other similar, non-wood materials, and provided a reasonable starting point for such an investigation.

BCTMP pine pulp (BPP)

A dry bale of radiata pine BCTMP was obtained from Winstone Pulp International Limited in Ohakune, New Zealand, and used as received.

Blended pulps

Varying ratios of HHP and BPP (as shown in Table 1) were mixed and stirred in a stock beater at 0.3% consistency for 15 min at room temperature.

Handsheets Preparation

The preparation of handsheets (230 g/m², 159 mm diameter) was conducted according to TAPPI T205 sp-02 (2006). Hot-pressed samples were prepared by placing wet handsheets between two aluminium plates without spacers in order to ensure that the handsheet was subjected to the entirety of the force in a heated hydraulic press (G. Siempelkamp GmbH & Co. KG, Krefeld, Germany) for either 100 or 300 s at 150 to 200 °C using a pressing force of 50 to 250 kN. For calculation of pressure, the handsheet diameter of 159 mm was used. It was observed that this dimension did not change substantially following hot-pressing. All handsheets were conditioned at 23.0 ± 1.0 °C and 50 ± 2.0% relative humidity for 36 h before testing. At least 20 handsheets of each composition were produced to provide sufficient material for subsequent testing.

Table 1. The Mixing Ratios of HHP and BPP

	Mixing Ratio (%)		Code
	HHP	BPP	
Control (Not hot-pressed)	100	0	H
	0	100	P
	80	20	H:P (4:1)
	50	50	H:P (1:1)
	20	80	H:P (1:4)
	100	0	Hp-H
Hot-pressed	0	100	Hp-P

Determination of Handsheet Properties

Optical imaging of handsheets was performed using a Leica MZ12 stereomicroscope (model no. CH-9435; Heerbrugg Leica Microsystem Switzerland Ltd., Heerbrugg, Switzerland) at 3.2x magnification. Fiber properties were measured using a Fiber Quality Analyzer 360 (FQA-360, OpTest Equipment Inc, Hawkesbury, Canada). Acid-insoluble lignin and acid-soluble lignin content analyses were determined based on TAPPI T222 om-02 (2002) and TAPPI UM 250, respectively. Fiber carbohydrate analysis was based on the method of Wood Sugar Analysis by Anion Chromatography with modification (Pettersen 1991).

Mechanical sheet property tests, including tensile strength TAPPI T494 om-01 (2001), burst strength TAPPI T403 om-10 (2010), tearing strength TAPPI T414 om-98 (1998), Short-span Compression Test (SCT) TAPPI T826 om-08 (2008), and Ring Crush Test (RCT) TAPPI T822 om-02 (2002) were conducted using a tensile tester (Lorentzen and Wettre, Stockholm, Sweden), burst strength tester (Lorentzen and Wettre, Stockholm, Sweden), tearing strength tester (Lorentzen and Wettre, Stockholm, Sweden), compression testing machine (IDM Instruments, Hallam, Australia), and STFI compression strength tester (Lorentzen and Wettre, Stockholm, Sweden).

Gas and water barrier performance tests were conducted as follows: air resistance following AS/NZS 1301.420s:2006 (2006), water absorptiveness of sized papers using the Cobb test based on ISO 535:2014(E) (2014), and contact angle measurement were determined using a Gurley densometer (Lorentzen and Wettre, Stockholm, Sweden), a Cobb tester at Scion, and a contact angle goniometer (FTA 1000B Frame, First Ten Angstroms, Portsmouth, Virginia, USA), respectively.

Data Analysis

The t-test and one-way variance analyses were performed using Microsoft Office 365 Excel software (Microsoft Office, Microsoft Corporation, version: Office 365, Redmond, Washington, USA) at a significance level of $\alpha = 0.05$. All the error bars shown in this work represent the standard errors.

RESULTS AND DISCUSSION

Fiber Analysis

Fiber shape, chemical composition, fines content, and other pulp properties can influence the properties of the resulting paper. The average length-weighted (l_w) fiber length, fines content, and mean fiber width of HHP and BPP were obtained from the Fiber Quality Analyzer. The results in Table 2 show that the fiber length of HHP was approximately half that of BPP and their width also was narrower. Both pulps contained a similar percentage of fines. Shives were excluded from this analysis, although HHP still contained shives, which are visible in the images below. Shives from other batches of HHP prepared using the same process were 1.08 ± 0.01 mm in length and 232.7 ± 9 μ m in width. Shives were however retained in the pulp to better represent how hurd pulp would likely be utilised in an industrial operation where waste streams (such as shives) would ideally be minimised.

Table 2. Pulp Characteristics of HHP and BPP Pulps

Pulp	Fiber Length l_w (mm)	Fines l_w (%)	Mean Width (μ m)
HHP	0.595 ± 0.005	6.5 ± 0.0	32.2 ± 0.1
BPP	1.266 ± 0.015	7.4 ± 0.1	39.1 ± 0.6

Chemical composition of the pulps is shown in Table 3. The BPP, prepared *via* mechanical pulping, had a higher lignin content than HHP that was prepared from chemical pulping, a process that removes lignin. This was consistent with past experience with this BCTMP pulp, which was expected to have had only a small amount of its lignin removed. Generally, chemically pulped fiber is more flexible, collapsible, bonds well, and forms closely-packed networks compared to those from mechanical pulping which have poor fiber-fiber contact (uncollapsed). This is due to the stiffness caused by their higher lignin contents (Bajpai 2018). Cellulose content, the primary structural element of fibers and the most important component influencing end use properties, varied for each sample. HHP contained more cellulose than BPP, which under regular pressing conditions could allow for a greater degree of bonding between the cellulose fibers, resulting in improved sheet properties (Bajpai 2018). In addition to hydrogen bonding, the hemicelluloses, especially xylose, contribute to pulp quality and play a key role in promoting fiber strength (Pere *et*

al. 2019). HHP was shown to have triple the amount of xylosyl units in comparison to BPP, so this together with a greater percentage of cellulose and lower amounts of lignin shows it could play an important role in strengthening a sheet if combined with another, longer fibre. However, it is suggested that this will mainly be due to the small size and collapsibility of the hurd fibres enabling them to fill spaces between larger fibres

Table 3. Chemical Composition of HHP and BPP Pulps

Samples	% (w/w) Oven Dried Sample		
	Total Lignin	Cellulose	Hemicelluloses (% of xylosyl units)
HHP	20.63 ± 0.11	56.23 ± 0.23	19.15 ± 0.00 (18.76)
BPP	29.43 ± 0.12	41.73 ± 0.04	21.78 ± 0.06 (5.44)

Handsheet Morphology

Handsheets from both hemp hurd and pine pulps, and blends of the two were observed *via* optical microscopy to examine their fibrous networks. Photomicrographs (Fig. 1a-c) show an incremental increase in fiber density for the handsheets of P, H:P (1:1), and H, owing to the increasing proportion of chemically pulped hemp fibers incorporated into the mix (Naithani *et al.* 2019). For sample H:P (1:1), it can be seen that the density was greater than that of P; however, large voids are still apparent in the sheet structure. Other surface images clearly show the differences in sheet structure following hot-pressing (denoted as Hp-P and Hp-H; Fig. 1d-e). By comparison, those sheets not hot-pressed contained large voids (likely responsible for poorer barrier properties as presented later), which were not present in the Hp-H and Hp-P samples. It is likely the hot-pressing causes sufficient fiber deformation to close these voids, aided by promoting lignin flow. As the pressing temperature (150 °C) is higher than the glass-transition temperature (T_g) of lignin (Prasad *et al.* 2005), the lignin will inevitably soften and flow within the handsheet surfaces, making them more closed and tightly packed. As noted above, shives were retained in HHP and can be seen in the relevant images as broad, dark coloured objects surrounded by the light brown hurd pulp fibres or bleached pine fibres. Shive dimensions can be seen to be similar to the values noted above, around 230 µm wide and 1 mm long.

Hot-Pressing Conditions Optimization

To identify optimum hot-pressing conditions, the tensile strength of each sample was measured (Table 4) to assess the performance of hot-pressed samples subjected to varying pressing time, temperatures, and forces. Initial hot-pressing was performed at a force of 50 kN (corresponding to 2.5 MPa pressure on the handsheet) and a temperature of 150 °C for either 100 or 300 s. The tensile strength results showed a decrease from 16.89 ± 0.19 kN/m at 100 s to 14.02 ± 0.28 kN/m for 300 s, and statistical data indicated that the shorter pressing time was significantly ($p < 0.05$) more beneficial to sheet strength. Thermal degradation/oxidation of the fibers after the loss of equilibrium moisture associated with longer heating times is likely responsible for this observation, as described in the literature (Shahzad 2013). Having established that 100 s press time was preferable, the effect of differing press temperatures on tensile strength within the range 150 to 200 °C was considered. Values of tensile strength (which fell between 13.55 ± 0.37 and 14.24 ± 0.52 kN/m) were not significantly influenced ($p = 0.97$) by the temperature range considered here, so for energy efficiency, 150 °C was deemed most suitable.

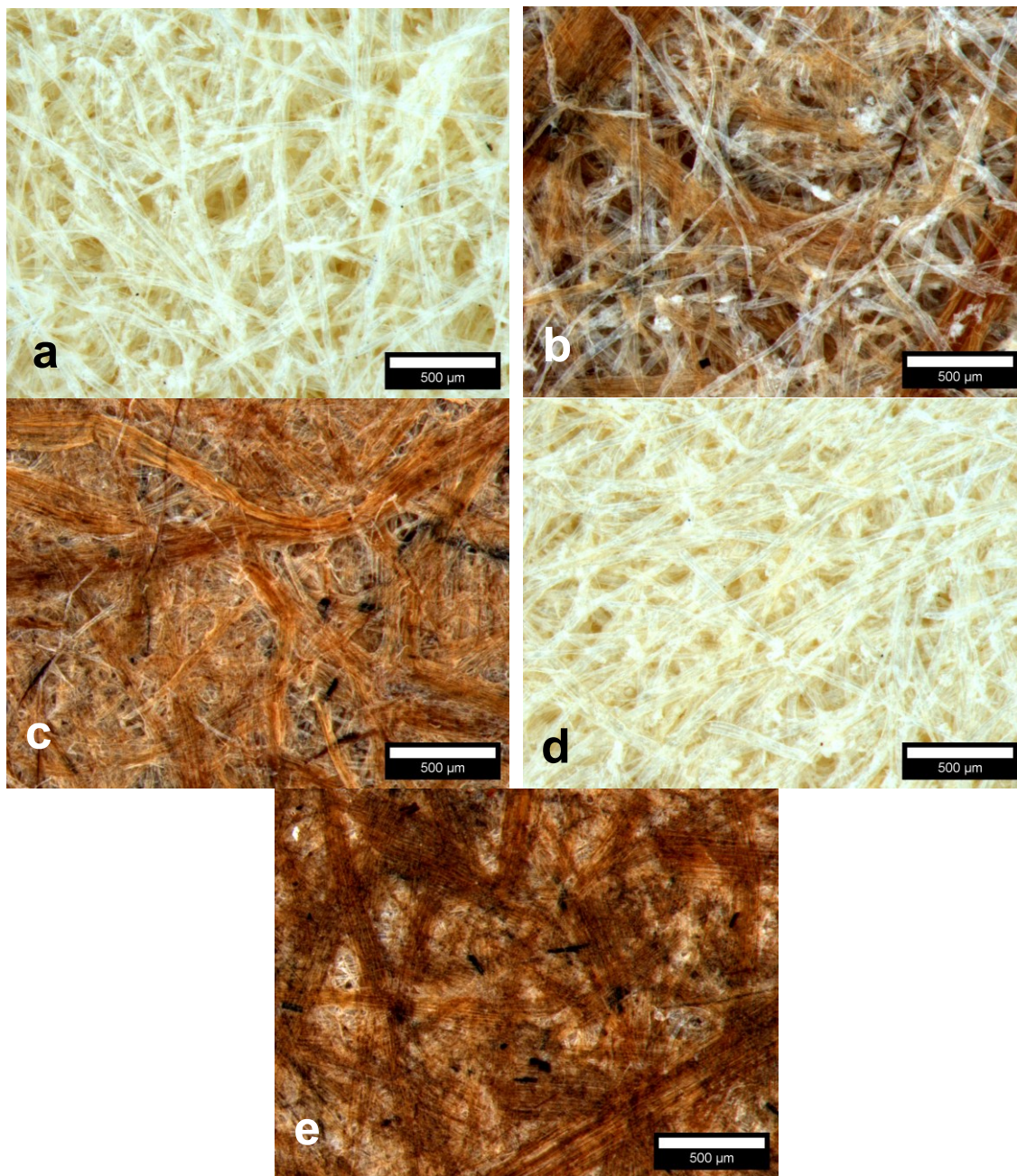


Fig. 1. The optical micrographs at 3.2x magnification of standard and hot-pressed handsheets. (a) P; (b) H:P (1:1); (c) H; (d) Hp-P; and (e) Hp-H

Lastly, the influence of press force from 50 to 250 kN (2.5 to 12.6 MPa pressure on the handsheet) on strength was assessed. An increase in force up to 150 kN (7.6 MPa on the handsheet) resulted in increased tensile strength (17.48 ± 0.40 kN/m) performance ($p < 0.05$). Beyond this point, pressure appeared to negatively influence the fiber network with surface sheet. Disruption was observed at 250 kN (12.6 MPa on the handsheet). Based on this preliminary testing, the optimum hot-pressing conditions used in subsequent parts of this investigation were 150 °C, 100 s, and 150 kN.

Table 4. Hot-Pressing Conditions Screening of Handsheets

Entry	Time (s)	Temperature (°C)	Press Force (kN)	Tensile Strength (kN/m)
1	100	-	-	16.89 ± 0.19
2	300	-	-	14.02 ± 0.28
3	100	150	-	13.55 ± 0.37
4	100	160	-	13.85 ± 0.71
5	100	170	-	13.53 ± 0.87
6	100	180	-	13.75 ± 0.78
7	100	190	-	14.24 ± 0.52
8	100	200	-	13.82 ± 0.34
9	100	150	50	12.61 ± 0.59
10	100	150	100	15.37 ± 0.17
11	100	150	150	17.48 ± 0.40
12	100	150	200	16.99 ± 0.27
13	100	150	250	-

Tensile Strength Index

The tensile strength of handsheets made of HPP and BPP, as well as blends of the two, are presented in Fig. 2 (compared *via* Tensile Index). Tensile strength increased proportionally with increasing hemp fiber content. Handsheets made from pure hemp fiber (H) were much stronger than any of the hemp: pine (H:P) blended samples. Papers from mechanical pulping tend to have lower strengths due to the remaining lignin, which weakens the bonding between fibers (Bajpai 2018). Although the pine fiber pulps were bleached, a high amount of residual lignin was still present within fibers after the pulping process. The residual lignin may be affecting tensile strength, but other factors may be involved.

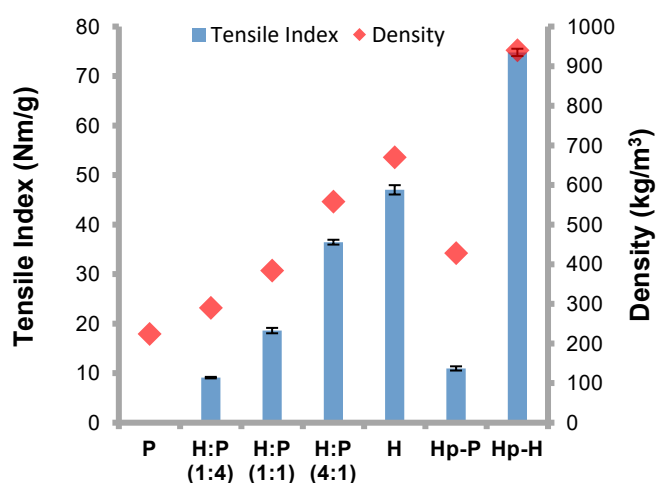


Fig. 2. Tensile index and handsheet densities of standard and hot-pressed handsheets. Error bars represent standard errors.

The strength properties of paper are influenced significantly by fiber length and fiber bonding. Typically, longer fibers contribute to improved tensile properties, but the degree of fiber bonding is considered the most crucial factor (Danielewicz and Surma-Ślusarska 2017). As expected, there was a strong correlation between sheet density and

tensile index observed in standard pressed sheets, which aligns with the findings reported by Joelsson *et al.* (2020). However, the tensile properties of handsheets made from BPP could not be analyzed due to their inherent weakness. The samples lacked sufficient fiber network bonding, resulting in an inability to withstand even pre-tensioning during testing. Long, flexible fibres such as softwood kraft would be expected to have far superior performance and should be considered for future investigations if high mechanical performance is required.

The tensile index of samples increased remarkably after hot-pressing, most likely due to enhanced densification arising from stronger fiber networks (Joelsson *et al.* 2022). The tensile index of Hp-H handsheets was the highest at 74.8 Nm/g, which is substantially greater than that of its standard counterpart (H). After hot-pressing, even the Hp-P handsheets registered a test value of 11.0 Nm/g tensile index, similar to the H:P (1:4) samples (9.1 Nm/g).

Burst Strength Index

Burst testing is used to determine the strength of paper products, such as paper bags, cardboard boxes, and packaging materials, to resist rupture or burst. Burst of paper products indicates the ability of the material to withstand the stresses and pressures that it may encounter during handling, shipping, and storage. As shown in Fig. 3, burst strength was found to be positively correlated with both tensile index and density, which is a well-known documented behavior (Wistara *et al.* 1999). Like tensile index, burst index improved proportionally with increasing hemp fiber ratios and upon hot-pressing. The Hp-H handsheets achieved the highest burst index at 2.60 kPa·m²/g, which was much stronger standard H. It is likely that burst strength is also influenced by the degree of fiber bonding due to the highly collapsed and closely packed nature of the fiber networks (Anjos *et al.* 2014). P had the lowest strength at 0.34 kPa·m²/g and hot-pressing had a minimal effect with just 0.02 kPa·m²/g difference between P and Hp-P handsheet samples. Again, this can be attributed to the pine fiber properties and the pulping process.

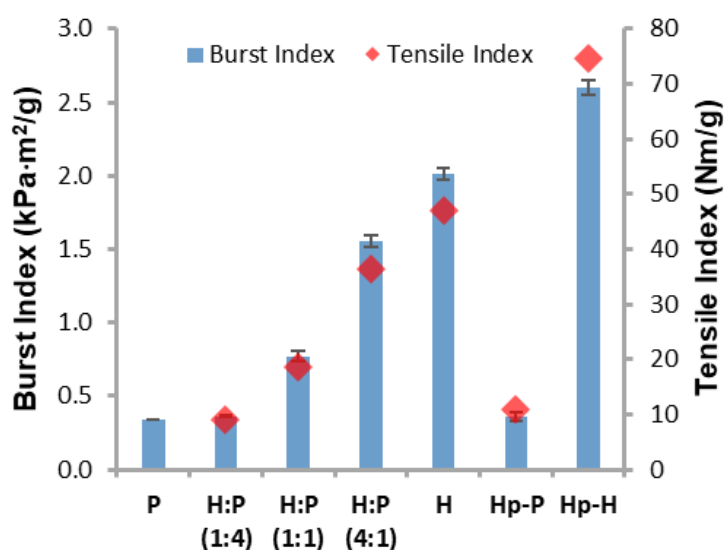


Fig. 3. Burst index of control and hot-pressed handsheets. Error bars represent standard errors.

Tearing Strength Index

Several factors such as fiber length and inter-fiber bonding influence tearing strength. Generally, there are two main contributions influencing tear strength: (1) the work for severing single fibers; and (2) the work for pulling out those unbroken fibers from the networks. It is well known that more energy (work) is required to pull out than to sever a fiber (Chang *et al.* 2012). Thus, shorter fibers are easier to pull out than longer fibers, resulting in lower tearing resistance. However, as fiber-fiber bonding increases within a handsheet, a fiber located in the pathway of a tear is more likely to be severed than pulled out. In addition, the stress at the apex of a tear is concentrated and is less readily shared with the rest of the structure due to the improved fiber bonding. As a result of these phenomena, the tearing strength typically shows an inverse correlation with tensile and burst strengths (Seth 1988).

Figure 4 shows average tearing strength for the different handsheets; adding hemp hurd to pine pulp was found beneficial to some degree. The relationship between tearing and tensile strengths is also indicated in the figure, which shows that tearing strength increased initially and then reduced with increasing tensile strength.

The tearing index of handsheets prepared from H:P (1:1) reached a peak of 7.18 mN·m²/g, consistent with contributions from both the longer fiber length from pine, and the increased fiber bonding from hemp hurd fibers due to increased surface area. The tear strength of standard hemp hurd handsheets was approximately double its hot-pressed counterpart (Hp-H). Fiber brittleness, an attribute also known to negatively affect tear, may contribute to these differences. Given the typical applications of thermoformed moulded fibre products, which typically need to contain or support objects, tear strength can likely be considered as less important than burst or tensile properties.

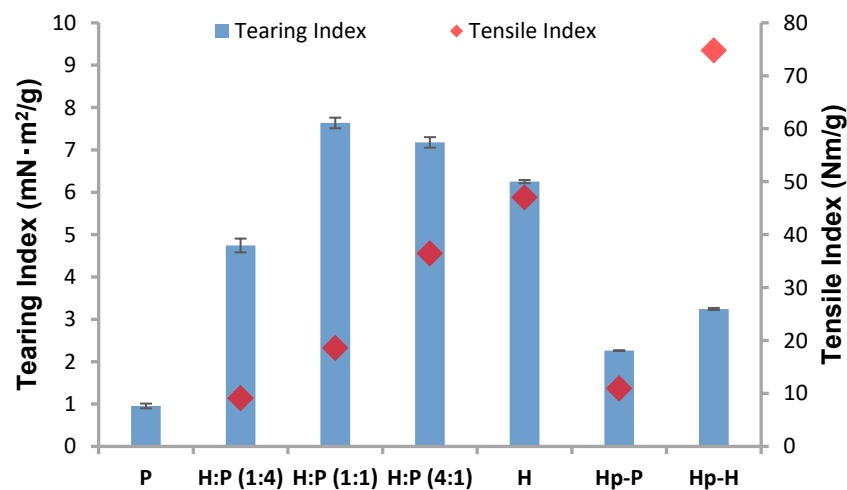


Fig. 4. Tearing index of control and hot-pressed handsheets. Error bars represent standard errors.

Short-span Compression Test (SCT) Index

Figure 5 shows SCT index results for handsheets made from hemp hurd and pine fibers. For control samples (*i.e.* not hot-pressed), SCT strength increased with increasing hemp pulp content; accordingly, pure hemp (H) had the highest SCT strength (27.27 Nm/g) and pure pine (P) the lowest (6.16 Nm/g). Hot-pressing had a positive effect on SCT, with Hp-H handsheets displaying a maximum SCT strength of 41.25 Nm/g. This was 34% stronger than that of H, while Hp-P was 45% higher than P. The improvement in SCT as

hemp hurd is added is likely caused by the increased density due to the enhanced fiber bonding. Another possible reason is the denser fiber networks and greater area for bonding due to hot-pressing, consistent with the literature (Joelsson *et al.* 2020).

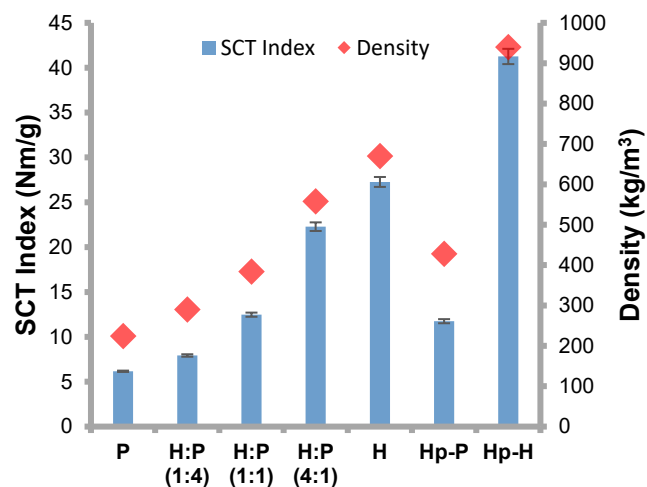


Fig. 5. SCT index of control and hot-pressed handsheets. Error bars represent standard errors.

Ring Crush Test (RCT) Index

RCT strength showed similar trends to SCT; as the content of hemp fiber increased, the RCT index of handsheets also increased (Fig. 6). Ring crush strength increased with sheet density, as with other bonded strength properties (Haslach 2000). As before, the density of handsheets increased after hot-pressing. Hp-H handsheets displayed the highest ring crush index at 28.08 Nm/g, which is stronger than that of the control. While still relatively low, the strength of hot-pressed pine handsheets was four times higher than that of the control pine handsheets. Once again, a likely reason is that the more densely packed fibers resulted in greater levels of fiber bonding. This is consistent with Parker *et al.* (2005), who showed that ring crush strength increased with increasing density of handsheets.

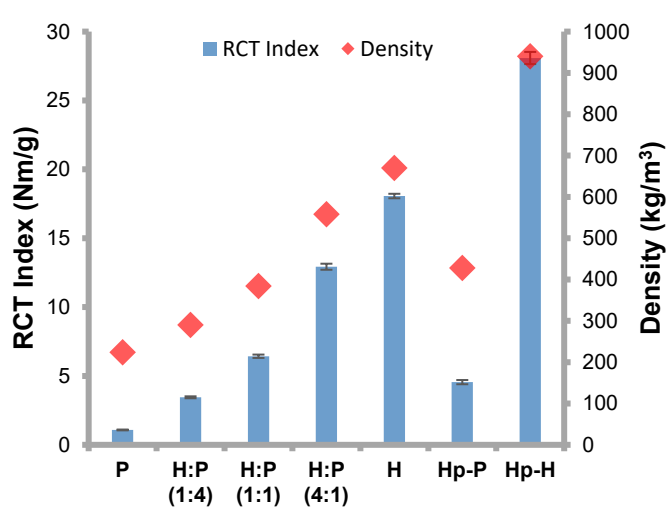


Fig. 6. RCT index of control and hot-pressed handsheets. Error bars represent standard errors.

Water Absorption

Cobb₆₀ test

The Cobb₆₀ test is commonly used to determine water resistance, with lower Cobb values indicating superior water resistance (Zhong *et al.* 2019). Figure 7 shows the Cobb₆₀ data for the handsheets and indicates that water resistance could be increased by consolidating the fibrous networks by increasing hemp fiber content and hot-pressing. The Cobb₆₀ value of pure pine handsheets was 565.2 g/m², and the Cobb₆₀ value of pure hemp handsheets was 335.8 g/m². Both were significantly reduced by 31% and 81% respectively, following hot-pressing (specimens Hp-P and Hp-H). One explanation for this observation would be the combination of high temperature and compression leading to increases in bonding between adjacent fibres and causing the lignin and extractives to move towards the surface, increasing hydrophobicity, as suggested previously (Joelsson *et al.* 2022). The impact of hot-pressing may also be attributed to the loss of internal cell wall porosity (hornification). Hornification potentially occurs when permanent crosslinks form between aggregated cellulose microfibrils during drying processes, resulting in the formation of bonded areas that resist swelling during rewetting; this occurrence is known to be more prevalent while drying at higher temperatures (Salmén and Stevanic 2018). Hornification is also affected by regular pressing processes in which fiber cell walls are exposed to compression and relaxation cycles, resulting in the loss of internal water because certain pores stay blocked after pressing, preventing water from re-entering the fibers when rewetted (Paulapuro 2001). From Table 2, it can be seen that the HHP and BPP had similar hemicellulose contents although with quite different percentages of xylosyl units, despite the HHP being produced by chemical pulping. Considering this, it is interesting to note the much larger reduction in water absorption ability of HHP than the BPP, despite the retention of hemicellulose, given that the presence of hemicellulose has been suggested to reduce susceptibility to hornification as summarised in earlier reviews (Hubbe 2014). While outside the scope of the present study, it is difficult to separate such effects from the effect of densification itself.

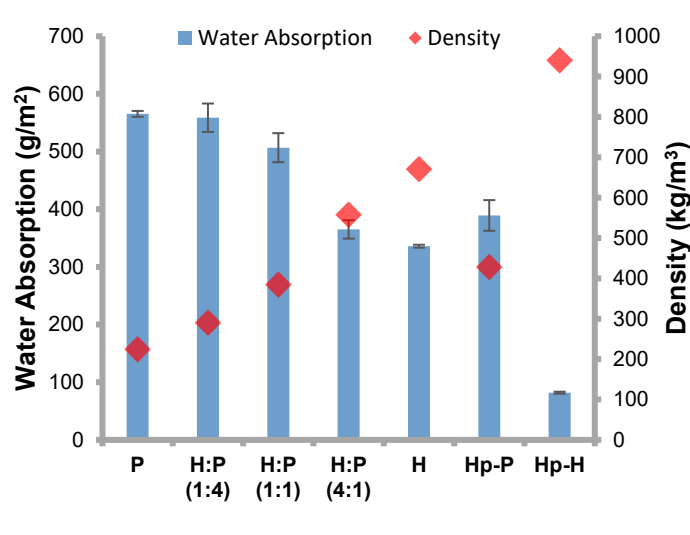


Fig. 7. The Cobb₆₀ value representing water absorption of control and hot-pressed handsheets. Error bars represent standard errors.

Contact angle (CA) measurement

In terms of the rate of water absorption, contact angle measurement provides information about the static and dynamic water absorption into paper surfaces as a function of time. Figure 8 depicts the contact angle of the handsheets at contact times 0.09 and 1.75 s. Initially, contact angles of 101.3°, 99.6°, and 101.2° were observed for samples of pine, hot-pressed pine, and the hemp-pine blend (1:1), respectively. In contrast, the initial contact angles for pure hemp hurd and hot-pressed hemp hurd ranged between 85 to 89°, which suggests they were relatively hydrophilic compared to pine. However, the water drop was absorbed more quickly into the surface of the pure pine handsheet resulting in a contact angle of 30.61° at 0.87 s, and it continued to decrease to 0° by 1.75 s. Consequently, pure pine proved to be strongly hydrophilic in contrast to the others over the short time frame examined.

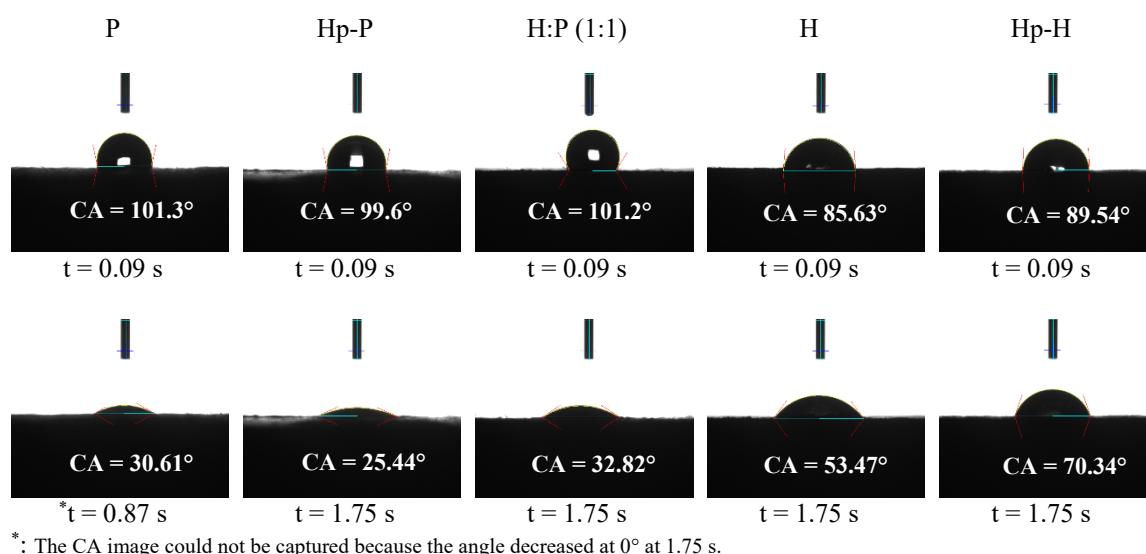


Fig. 8. Images of contact angles of water on the surfaces of control and hot-pressed handsheets

Figure 9 shows contact angle values as testing time was extended to 7.0 s. Hot-pressed samples were less absorbent for both hemp hurd and pine handsheets. After 1.75 s, the contact angle of Hp-H handsheets remained the highest out of the group (though still hydrophilic) and levelled off to around 65° after 7 s. Pure hemp hurd handsheets displayed the next best water absorption beyond 1.75 s, but in contrast to Hp-H its values sharply declined to 8° over the longer interval.

The greater long-term contact angle observed for the hemp sheets is likely attributable to their more closed fiber networks, accentuated by the melting of lignin coated on their surfaces as a result of hot-pressing as has been documented by others (Joelsson *et al.* 2022). Another likely contribution to this effect is the migration of hydrophobic monomers (*i.e.* extractives) to the surfaces during hot-pressing, essentially a “self-sizing” effect commonly seen in papermaking (Hubbe *et al.* 2007). This would be expected to provide good liquid hold-out when combined with small pores as present in the HHP samples.

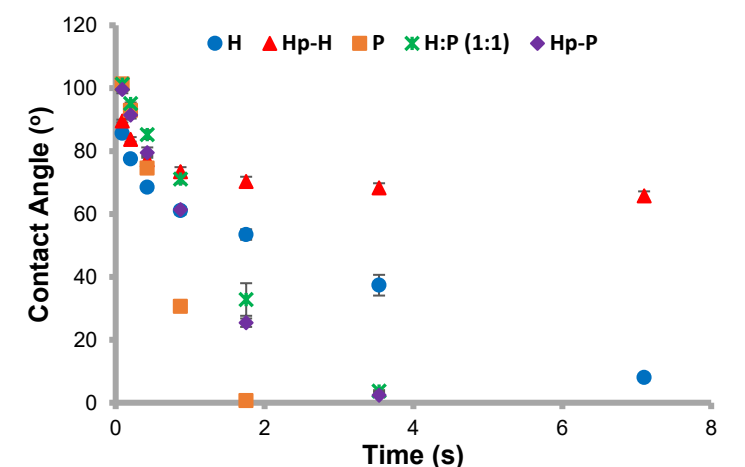


Fig. 9. Contact angle of water on the control and hot-pressed handsheets as a function of time. Error bars represent standard errors.

Air Resistance

The Gurley test was performed to determine the air resistance of sheets (Table 5). Pine handsheets had the lowest value at 0.36 s/100 mL (or porosity 16667 mL/min), but as more of the smaller hemp fiber was incorporated into the sheet mix, filling pore space, air resistance increased. Higher sheet densities due to both the more conformable hemp fibers and compaction from hot-pressing caused substantial rises in air resistance, with hot-pressed hemp handsheet values being the highest at 2462 s/100 mL.

Table 5. Air Resistance of Control and Hot-Pressed Handsheets

	P	H:P (1:1)	Hp-P	H	Hp-H
Gurley Seconds (s/100 mL)	0.36	5.07	1.86	100.64	2462
Porosity (mL/min)	16667	1184	3226	60	2.44
Density (kg/m ³)	224	384	428	670	940

Prototype Formation

A thermoformed molded cup prototype was produced from hemp hurd fiber to demonstrate its potential use as a substitute for single-use plastic packaging and food service ware. The process involved fabricating a mesh mold that was inserted into the agitated hemp fiber slurry and a vacuum was applied to the mesh until sufficient pulp was deposited on the mold. The newly formed shape was then ejected from the mesh using pressurized air and placed inside a previously cast outer mold. A hand-operated screw clamp was used to compress the mold tool together, which was then placed into a drying oven at 104 °C overnight, thus approximating a hot-pressing process. While this relatively low temperature, chosen in order not to damage the polymer molds, is not fully representative of the elevated temperatures present in thermoforming, this simple molding and drying process clearly showed that it is possible to produce a three-dimensional object with curved surfaces and sharp radius corners from HHP. For this reason, and due to the relatively small dimensions of the thermoformed example, it was not possible to carry out meaningful mechanical testing on the cups. This will be the subject of future investigations. The resultant product is shown in Fig. 10 along with a simple schematic of how this could be produced industrially.

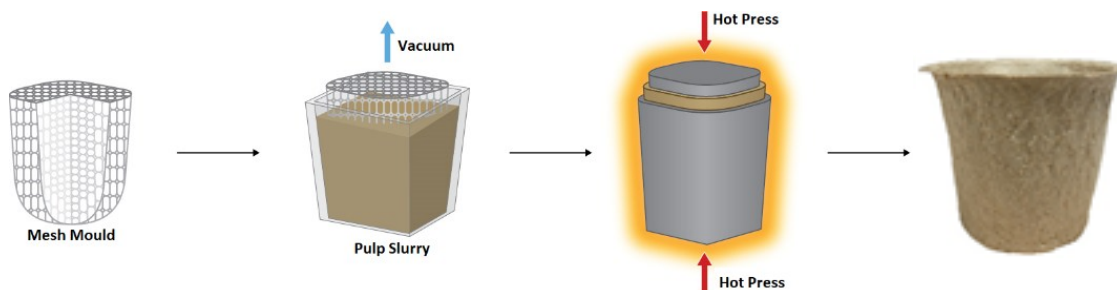


Fig. 10. A schematic of the thermoformed molded cup production process for hemp hurd fibers

CONCLUSIONS

1. Chemically processed hemp hurd fibers were used to produce sheets with superior mechanical properties and barrier performance, as compared to sheets made of mechanically processed pine fibers. The superiority was mainly due to the hemp fiber's ability to promote greater inter-fiber bonding and form denser sheet webs.
2. Increasing the proportion of hemp hurd fibers in the hemp-pine blend was beneficial based on the assessment of mechanical properties and barrier performance of control (not hot-pressed) pine and hemp fiber-based handsheets. This was attributed to the greater bonding potential of the more pliable hemp fibers.
3. Hot-pressing the samples under the optimized conditions of 150 °C and 150 kN for 100 s resulted in a significant improvement in their mechanical properties. This improvement was attributed to the more compacted fibrous networks. Additionally, the barrier properties of the samples were further enhanced due to the increased sheet density resulting from lignin flow and hornification effects under these conditions.
4. A prototype thermoformed molded fiber cup demonstrated that hemp hurd could be a viable replacement for single-use plastic products.

ACKNOWLEDGEMENTS

Funding for this work was provided by Ministry for Business, Innovation and Employment (NZ) via the Strategic Science Investment Fund, grant 79192. Technical support was provided by Donna Smith and Gavin Durbin. We are also grateful to Dr. Alan Dickson for assistance with microscopy and for providing valuable insights during the manuscript preparation, and to Dr. Karl Murton for providing valuable insights during the manuscript preparation.

REFERENCES CITED

- Ahmed, A. T. M. F., Islam, M. Z., Mahmud, M. S., Sarker, M. E., and Islam, M. R. (2022). "Hemp as a potential raw material toward a sustainable world: A review," *Heliyon* 8(1), article e08753. DOI: 10.1016/j.heliyon.2022.e08753

- Anjos, O., Santos, A. J., Simões, R., and Pereira, H. (2014). "Morphological, mechanical, and optical properties of cypress papers," *Holzforschung* 68(8), 867-874. DOI: 10.1515/hf-2013-0125
- AS/NZS 1301.420s:2006 (2006). "Methods of test for pulp and paper - Gurley air permeance of paper," Standards Australia, Sydney, Australia.
- Bajpai, P. (2018). *Biermann's Handbook of Pulp and Paper: Volume 1: Raw Material and Pulp Making*, Elsevier, Amsterdam, Netherlands.
- Bokhari, S. M. Q., Chi, K., and Catchmark, J. M. (2021). "Structural and physico-chemical characterization of industrial hemp hurd: Impacts of chemical pretreatments and mechanical refining," *Industrial Crops and Products* 171, article 113818. DOI: 10.1016/j.indcrop.2021.113818
- Chang, X. F., Olson, J. A., and Beatson, R. P. (2012). "A comparison between the effects of ozone and alkaline peroxide treatments on TMP properties and subsequent low consistency refining," *BioResources* 7(1), 99-111. DOI: 10.15376/biores.7.1.99-111
- Danielewicz, D. and Surma-Ślusarska, B. (2017). "Properties and fibre characterisation of bleached hemp, birch and pine pulps: a comparison," *Cellulose* 24(11), 5173-5186. DOI: 10.1007/s10570-017-1476-6
- Didone, M., Saxena, P., Meijer, E. B., Tosello, G., Bissacco, G., McAloone, T. C., Pigosso, D. C. A., and Howard, T. J. (2017). "Moulded pulp manufacturing: Overview and prospects for the process technology," *Packaging Technology and Science*, 30(6), 231-249. DOI: 10.1002/pts.2289
- Haslach, H. W. (2000). "The moisture and rate-dependent mechanical properties of paper: A Review," *Mechanics of Time-Dependent Materials* 4(3), 169-210. DOI: 10.1023/A:1009833415827
- Hubbe, M. A., Venditti, R. A., and Rojas, O. J. (2007). "What happens to cellulosic fibers during papermaking and recycling? A review," *BioResources* 2(4), 739-788.
- Hubbe, M. A. (2014). "Prospects for maintaining strength of paper and paperboard products while using less forest resources: A review," *BioResources* 9(1), 1634-1763. DOI: 10.15376/biores.9.1.1634-1763
- ISO 535:2014(E). (2014). "Paper and board – Determination of water absorptiveness – Cobb method," International Organization for Standardization, Geneva, Switzerland.
- Joelsson, T., Persson, E., Pettersson, G., Norgren, S., Svedberg, A., and Engstrand, P. (2022). "The impact of sulphonation and hot-pressing on low-energy high-temperature chemi-thermomechanical pulp," *Holzforschung* 76(5), 463-472. DOI: 10.1515/hf-2021-0109
- Joelsson, T., Pettersson, G., Norgren, S., Svedberg, A., Höglund, H., and Engstrand, P. (2020). "High strength paper from high yield pulps by means of hot-pressing," *Nordic Pulp & Paper Research Journal* 35(2), 195-204. DOI: 10.1515/npprj-2019-0087.
- Naithani, V., Tyagi, P., Jameel, H., Lucia, L., and Pal, L. (2019). "Ecofriendly and innovative processing of hemp hurds fibers for tissue and towel paper," *BioResources* 15(1), 706-720. DOI: 10.15376/biores.15.1.706-720
- Parker, I. H., Conn, A. B., and Jackson, T. (2005). "The effect of network variables on the ring crush strength of handsheets," *Appita Journal* 58(6), 448-454.
- Paulapuro, H. (2001). "Wet pressing - Present understanding and future challenges," in: *12th Fundamental Research Symposium* 639-678. DOI: 10.15376/frc.2001.1.639
- Pere, J., Pääkkönen, E., Ji, Y., and Retulainen, E. A. (2019). "Influence of the hemicellulose content on the fiber properties, strength, and formability of handsheets," *BioResources* 14(1), 251-263. DOI: 10.15376/biores.14.1.251-263

- Pettersen, R. C. (1991). "Wood sugar analysis by anion chromatography," *Journal of Wood Chemistry and Technology* 11(4), 495-501. DOI: 10.1080/02773819108051089
- Prasad, B. M., Sain, M. M., and Roy, D. N. (2005). "Properties of ball milled thermally treated hemp fibers in an inert atmosphere for potential composite reinforcement," *Journal of Materials Science* 40(16), 4271-4278. DOI: 10.1007/s10853-005-2799-5
- Salmén, L. and Stevanic, J. S. (2018). "Effect of drying conditions on cellulose microfibril aggregation and 'hornification'," *Cellulose* 25(11), 6333-6344. DOI:10.1007/s10570-018-2039-1
- Seth, R. (1988). "Fiber properties and tearing resistance," *Tappi Journal* 71(2), 103-107.
- Shahzad, A. (2013). "A study in physical and mechanical properties of hemp fibres," *Advances in Materials Science and Engineering* 2013, article 325085. DOI: 10.1155/2013/325085
- TAPPI T205 sp-02 (2006). "Forming handsheets for physical tests of pulp," TAPPI Press, Atlanta, GA, USA.
- TAPPI T222 om-02 (2002). "Acid-insoluble lignin in wood and pulp," TAPPI Press, Atlanta, GA, USA.
- TAPPI T403 om-10 (2010). "Bursting strength of paper," TAPPI Press, Atlanta, GA USA.
- TAPPI T414 om-98 (1998). "Internal tearing resistance of paper," TAPPI Press, Atlanta, GA, USA.
- TAPPI T494 om-01 (2001). "Tensile breaking properties of paper and paperboard," TAPPI Press, Atlanta, GA, USA.
- TAPPI T822 om-02 (2002). "Ring crush of paperboard (rigid support method)," TAPPI Press, Atlanta, GA USA.
- TAPPI T826 om-08 (2008). "Short span compressive strength of containerboard," TAPPI Press, Atlanta, GA, USA.
- TAPPI UM 250 (2006). "Acid-soluble lignin in wood and pulp," TAPPI Press, Atlanta, GA, USA.
- Wistara, N., Zhang, X., and Young, R. A. (1999). "Properties and treatments of pulps from recycled paper. Part II. Surface properties and crystallinity of fibers and fines," *Cellulose* 6(4), 325-348. DOI: 10.1023/A:1009255808215
- Zhong, L., Ding, Y., Zhang, B., Wang, Z., Li, C., Fu, X., and Huang, Q. (2019). "Effect of octenylsuccinylation of oxidized cassava starch on grease resistance and waterproofing of food wrapping paper," *Starch - Stärke* 71(7-8), article 1800284. DOI: 10.1002/star.201800284

Article submitted: October 24, 2023; Peer review completed: November 11, 2023;
Revised version received: January 18, 2024; Accepted: January 19, 2024; Published:
January 30, 2024.

DOI: 10.15376/biores.19.1.1728-1743