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Rahul Sharma, Fabiano Ximenes and Gil Garnier*

Australian wheat and hardwood fibers for advanced packaging materials

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Abstract: Alternative crop fibers have shown great potential for paper applications, especially packaging. We demonstrate Australian wheat straw processing using a Regmed MD-3000 disc refiner to produce mechanical pulp fibers and assessment by making 60, 120 and 300 g/m^2 handsheets. Wheat fibers and spotted gum fibers were then enzymatically sized (hydrophobized) by esterification to reduce the surface spread of water by 51 % and 36 %, respectively. Coffee pods (300 g/m^2 equivalent) were manufactured using a thermoformer to demonstrate the versatility of mechanical wheat straw pulp fibers as a sustainable resource for food packaging application.

Keywords: wheat straw; spotted gum; disc refining; coffee pods; enzymatic esterification

1 Introduction

Plastics are extensively used in packaging today and are considered environmentally toxic, creating an important load of contaminant accumulating in our soil and marine environment (González-Fernández et al. 2021; Mitrano et al. 2021). Non-woody biomass like wheat straw represents a valuable yet untapped resource for the production of low-cost, renewable, paper based packaging materials that has not been utilised to its full potential for papermaking (Tadesse et al. 2022; Worku et al. 2023). In New South Wales, Australia alone, on average 5.6 million tonnes of wheat straw are generated per year (Department of Primary Industries 2024). Some companies including Red Leaf (Canada), Essity (Sweden) and Trident Group (India), have elected straw fibers to manufacture products. This has led the shift towards non-woody crops available in large supply for biodegradable and green manufacturing. Woody feedstocks are also a valuable resource – Spotted gum (*Corymbia maculata*) is a native woody species managed for timber production in New South Wales and Queensland (Leggate et al. 2021; Lu et al. 2024) and which can also be grown on a short rotation basis for biomass production. The use of residues from spotted gum harvest and processing operations for paper packaging applications has not yet been fully explored.

One way to produce sustainable and low-cost paper making pulp is through mechanical refining. This process avoids using hazardous chemicals for biomass breakdown and digestion thereby reducing by-product wastes and cost. Previous work in pulping of wheat straw conducted by Leponiemi et al. (Leponiemi et al. 2010) explored hot water treatment and alkaline peroxide bleaching. This resulted in 10 % of wheat straw dissolving in hot water. A shortcoming of the mechanical refining that was outlined was that some fraction of the original wheat straw is found in the pulp fiber fraction. This was a detriment to the mechanical properties of the paper resulting in a Tensile Index of only 4.6 N m/g. This was further improved by mixing with 80 % addition softwood pulp long fibers to 34.9 N m/g. The addition of a wet strengthening agent like cationic starch was also explored at 1–2 | wt%. This improved the Tensile Index to 47.5–55 N m/g. In our work, we selected not to use wet-end additives to optimise sustainability and minimise processing costs.

In this study, we explored the mechanical pulping of wheat straw and spotted gum and their paper properties at 120 gsm. Lipases belong to the family of serine hydrolases and can catalyse several reactions depending on the environment, by employing a ping pong bi bi mechanism (Paiva et al. 2000; Reis et al. 2009). Enzymatic modification to improve hydrophobicity of cellulose using lipases has been investigated previously by Sharma et al. (Sharma et al. 2023b) employing transesterification between cellulose primary alcohols and a long chain alkyl ester. With 60 gsm

***Corresponding author: Gil Garnier**, Department of Chemical and Biological Engineering, Monash University, Clayton, Australia; and Department of Chemical and Biological Engineering, Bioresource Processing Research Institute of Australia, Monash University, Melbourne, VIC 3800, Australia, E-mail: gil.garnier@monash.edu
Rahul Sharma, Department of Chemical and Biological Engineering, Bioresource Processing Research Institute of Australia, Monash University, Melbourne, VIC 3800, Australia
Fabiano Ximenes, Forest Science, New South Wales Department of Primary Industries, Locked Bag 5123, Parramatta, NSW 2150, Australia

sheets, we explored the use of sustainable low cost, enzymatic functionalisation as internal hydrophobization with methyl myristate as ester donor to introduce surface nonpolar interactions. The novel use of wheat straw pulp to make sustainable coffee pods was also demonstrated.

2 Materials

An Australian wheat straw (Triticum spp.) bale was purchased from a local supplier. Spotted gum chips were sourced from a sawmill on the north coast of NSW (Pentarch). Never-dried unrefined Bleached Eucalypt globulus Kraft fibers (BEK) were received from Australian Paper, Maryvale at a solid's concentration of 10 wt%. In this study, the BEK fibers were only utilised for making composite handsheets for hydrophobicity assessment. Lipolase 100 L enzyme solution and Methyl myristate was purchased from Sigma.

3 Methods

Wheat straw and spotted gum compositional analysis was conducted in accordance with NREL Laboratory Analytical Procedures TP-510-42619, TP-510-42620 and TP-510-42622, as shown previously (Ang et al. 2020).

The wheat straw was shredded dry using a Ryobi mulcher (AC 2400 W, 45 mm cut capacity) to reduce straw length. The straw and spotted gum chips were soaked in 20 L hot water at 60 °C in a Lamort disintegrator overnight to soften separately. The biomass was well disintegrated for 1 h the next day. The solids were separated from the solution and collected in a separate bucket. A Regmed MD-3000 disc refiner was used for mechanical pulping of softened biomass set with FBD-16 brass plates (cutting speed 13.42 km/s). Using Leponiemi et al.'s (Leponiemi et al. 2010) study as a guide, optimisation was performed on wheat straw refining with different plate gaps (0.25 mm–10 mm). The best results were achieved with 10 mm plate gap as lower plate gaps resulted in fibers that were too short and therefore lacked strength to produce a hand sheet. This was also optimised to reduce any blocking in the refiner. Initially, 20 L water was run in the refiner and then small amounts of biomass was added in a 10 min batch run to ensure smooth running without creating blockages. The specific energy for each batch of run was calculated to be 2.56 kWh/ton. This is on the low side of the refining spectrum as Leponiemi et al. (Leponiemi et al. 2010) performed wheat straw refining at a 0.5 mm plate gap in a VTT Wing Refiner. The refined material was collected and then fractionated to remove the fibers from unpulped biomass material. The yield of pulp was approximately 50 % with 50 % coarse unpulped material rejected to run again in the refiner. The unpulped biomass was run through the refiner again later to avoid using fresh wheat straw stock. The diluted fiber fraction was then concentrated using a British Automatic Handsheet maker on a 150-size mesh (104 μm pore size). The batch refining runs were repeated until sufficient biomass pulp was collected.

Lipase surface transesterification of wheat straw fibers and spotted gum was performed as per Sharma et al. (Sharma et al. 2023b). Basically, biomass were filtered in a Buchner funnel till 30 wt% solids and then esterified with methyl myristate in equal weight basis with at 16 mg lipase protein/g dry fiber loading for biocatalysis independently.

ATR-FTIR was performed on washed and dried wheat straw and spotted gum fibers using an Agilent technologies Cary 630 with a diamond attenuated total reflectance. The resolution of the instrument was set at 4 cm^{-1} and eight scans were performed in the range of 4,000–500 cm^{-1}. For water barrier assessment, a solution of Brilliant Blue G (1 % w/v) was prepared in Milli Q water and five independent 20 μL drops were dropped on the surface of the 60 gsm handsheets containing unmodified and modified wheat straw or spotted gum blended at 10 % w/v with Bleached Eucalypt kraft fibers (90 %).

For sheet making, 1.2 g, 2.4 g and 6 g oven dry fiber equivalent were used to make 60, 120 and 300 gsm sheets as shown previously (Sharma et al. 2022). For each different grammage of sheets prepared, either 1.2 g, 2.4 g or 6 g fiber equivalent was mixed with 2 L water for disintegration in a 3 L Mavis standard Disintegrator. Then the solution was poured in an Automatic British Handsheet maker for sheet formation by settling on a 150-size mesh (104 μm pore size). Post-settling, the sheets were then collected and layered on steel plates for pressing twice. Spotted gum and wheat straw were added only at 10 wt% in 60 gsm sheets for assessment of surface hydrophobicity improvement in a BEK fibers basesheet (90 wt%). All sheets were conditioned for 24 h conditioning at 23 °C and 50 % relative humidity as per TAPPI T402 (TAPPI (2001b), 2008).

Water vapor transmission rate (WVTR) experiments were run as described by Lu et al. (Lu et al. 2016).

Tensile testing was performed on 120 gsm wheat straw sheets as per TAPPI T494 (TAPPI 2006) on at least five replicates. Water vapor transmission was performed in 120 and 300 gsm sheets as per ASTM standard E 96 (ASTM 2019) on three replicates. Fiber dimensional analysis was conducted using a MorFI Neo. Microscopy images were taken using a Nikon Eclipse N*i*-E optical microscope at two different dilutions.

A separate batch of 160 g oven dry equivalent in a 20 L suspension of wheat straw pulp fibers was prepared to produce coffee pods in a thermoformer. Thermoforming was performed with a pilot plant system from pulp suspension at 0.8 wt% consistency.

4 Results and discussion

The chemical composition of wheat straw and spotted gum was measured in accordance with NREL methods (Sluiter et al. 2004). Table 1 shows the chemical composition of wheat straw and spotted gum in terms of its individual biopolymers. The cellulose, hemicellulose and lignin content measured are close to the range expected (Isikgor and Becer 2015) but varies slightly due to growth conditions and maturity of the cell wall (Barakat et al. 2013). Ash and extractives are similar to the range expected from wheat straw (Biricik et al. 1999) with some variation due to similar variations. The chemical composition of spotted gum is also similar to a that reported in a previous study by Magalhães et al. (Magalhães et al. 2012), with lignin content slightly higher and holocellulose content slightly lower than those in Brazil.

To analyse the wheat straw, we performed a sugar analysis on the cellulose and hemicellulose fractions as shown in Table 2. Individual sugars present in the structure of cellulose and hemicellulose show high amounts of glucose and xylose (40.26 % and 16.20 %), as expected, while a low amount of arabinose and galactose (Barakat et al. 2013). This provides normalising data that native wheat straw from Australia is similar in composition to others grown around the world and would therefore yield similar results post processing.

Table 1: Chemical composition of wheat straw and spotted gum measured using NREL method. Average content % is shown against each component.

Chemical composition	Wheat straw	Spotted gum
Cellulose	40.3	39.9
Hemicellulose	20.5	11.1
Lignin	22.1	43.2
Extractives	8.1	5.66
Ash	9.0	0.19

Table 2: Compositional analysis of cellulose and hemicellulose in wheat straw (± Standard deviation).

Sugar	Average Content %
Cellobiose	1.37 ± 0.11
Glucuronic Acid	0.13 ± 0.05
D (+) Glucose	40.26 ± 0.74
D (+) Xylose	16.20 ± 0.8
D (+) Galactose	1.56 ± 0.03
D (+) Arabinose	2.78 ± 0.06

The length weighted fiber length and width distributions of the wheat straw pulp fibers are displayed in Figure 1. It is evident that mechanical refining is detrimental to the length of the fibers even at maximum disc gap which is the least energy intensive. Over 25 % of the fibers are in the bin 275–378 µm and the distribution drops sharply as the length increases. A similar pattern is observed in the width of the fibers. Almost 18 % of the fibers are in the width of 12–19 µm and the distribution declines till 67 µm. This is similar to the findings of Ang et al. (Ang et al. 2019) when bleached eucalyptus kraft fibers were refined for fiber dimension analysis. The unrefined (referred as BEK0K) were in the bins (12–19 µm) and there was a steady reduction in width as the fibers were further refined. As a contrast, unrefined BEK fibers were reported to be 1,220 µm in length. The refined wheat straw fibers shown here show a sharp decline in distribution of long fibers. Maximising fiber-fiber contact is essential for high strength in refined fibers and for that fibrillation to maximise surface area is necessary. There is some fibrillations present in the fibers as seen in Figure 2 microscopy images but there is an impediment by the coarse material to strengthen the overall structure in handsheets.

Handsheets were then made from wheat straw using the standard technique. The destructive and non-destructive properties of 120 grammage wheat straw sheets made post-disc refining are highlighted in Table 3. The water vapor transmission rates of 246 g/m^2. day is higher in comparison to bleached hardwood MFC (200 g/m^2. day) that were measured by Spence et al. (Spence et al. 2010) but much lower than the MFC produced from unbleached hardwoods which were in the range of 300–460 g/m^2. day. Since the fiber length is mostly in the range of 275–378 µm, there is very little fiber-fiber overlap allowing large pore sizes on the sheet and therefore higher levels of water transmission through the surface. Another factor is that the wheat straw fibers were not bleached before analysis. The improved water vapor barrier of bleached hardwood MFC is due to the abundance of alcohol groups available on the surface when lignin is not inhibitory. This allows better hydrogen bonding between fibers and therefore a smaller pore size to reduce water vapor transmission through the surface. Despite being unbleached, the wheat straw WVTR is close to this value which is encouraging when refined at minimal energy in a disc refiner.

Tensile Index of the wheat straw paper was measured to be 15.8 N m/g, quite low compared to Leponiemi et al. (Leponiemi et al. 2010) reporting 63.4–77.2 N m/g and Hou et al.

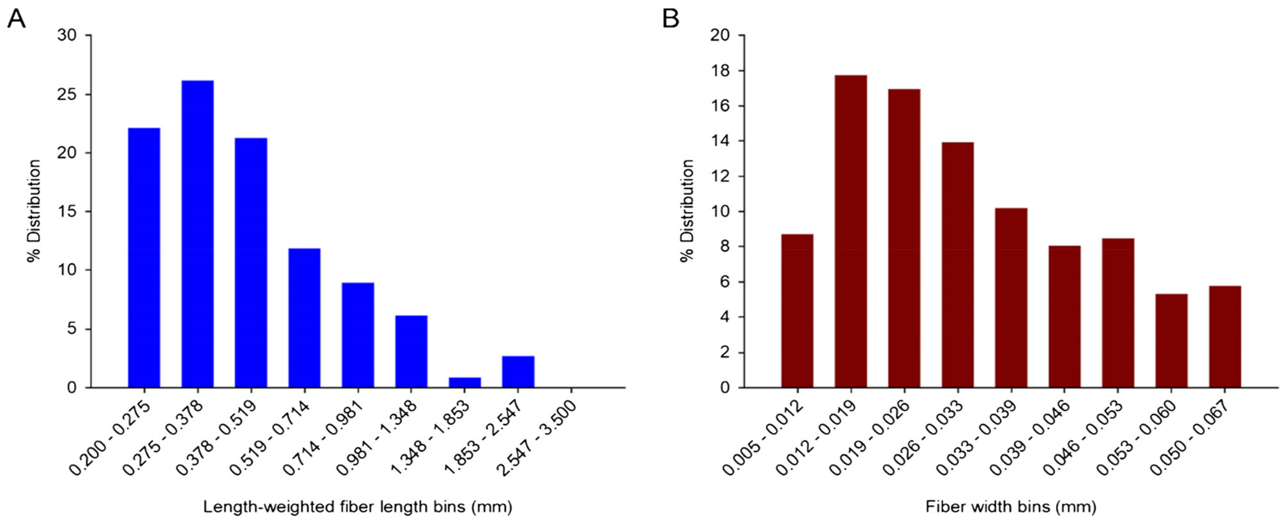

Figure 1: Wheat straw pulp fiber size after mechanical disc refining A) Length-weighted length B) width (right) distributions from MorFI neo analysis.

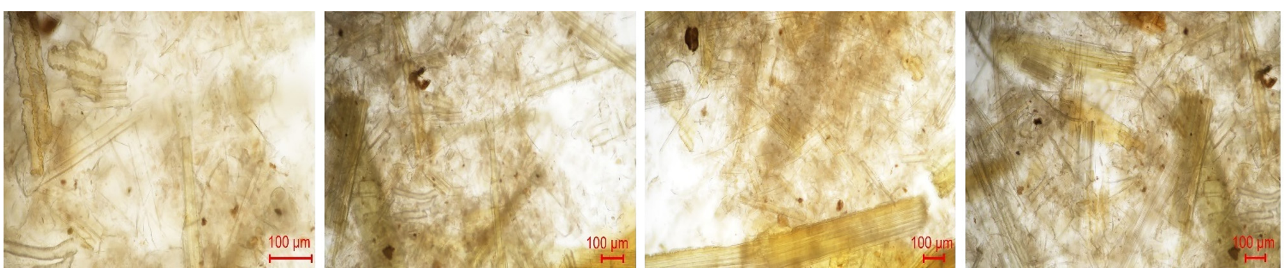

Figure 2: Optical microscopy images (100 μm scale) of disc refined wheat straw.

Table 3: Properties of wheat straw 120 gsm handsheet (± Standard deviation).

Handsheet thickness (μm)	293 ± 9.0
Handsheet density (kg/m^3)	363 ± 11
Water vapor transmission rate g/m^2.day)	246 ± 0.38
Young's modulus (MPa)	894 ± 24
Tensile stress at max force (MPa)	5.74 ± 0.26
Tensile index (N.m/g)	15.8 ± 0.72
Maximum force (N)	25.2 ± 1.14
Tensile strain at maximum force (%)	0.9 ± 0.11

(Hou et al. 2011) with 60–65 N m/g for wheat straw refining. However, our results are similar to those reported by Ang et al. (Ang et al. 2019) for the analysis of unrefined bleached eucalyptus fibers. Tozluoğlu et al. (Tozluoğlu et al. 2015) also reported Tensile Index for wheat straw paper in the range of 40–65 N m/g. Other studies also produced paper sheets of higher strength; however, they generally rely on chemical treatment of some type before mechanical treatment which seems to improve fiber bonding. Similarly, the other mechanical properties measured, are also shown to be quite low. A reason for this is the amount of unpulped straw coarse material that could not be removed from the fractionation process. These can be clearly seen in the microscopy images in Figure 2. The presence of those coarse unpulped straw particles show that these can pass through the fractionating and create an obstacle to fiber-fiber bonding. Additional refining of the fractionated materials which include fibers and coarse straw was not performed to preserve the fiber length. The density of the 120-gsm sheet is 363 kg/m^3 which is quite low and indicative of the high water vapor transmissions through the surface. Sharma et al.'s (Sharma et al. 2023a) work on 120 gsm sheets suggest density should be approximately 500 kg/m^3. This further highlight how the coarse material is acting to lower the density of the sheet on the same grammage basis. This could make the material an effective thermal and electrical insulator by creating an air trap.

Surface modification of cellulosic fibers can be valuable in creating desirable functionalities like hydrophobicity. Here, we performed an enzymatic internal sizing of the refined wheat straw and spotted gum. Spotted gum (*C. maculata*) is a hardwood species that is native to New

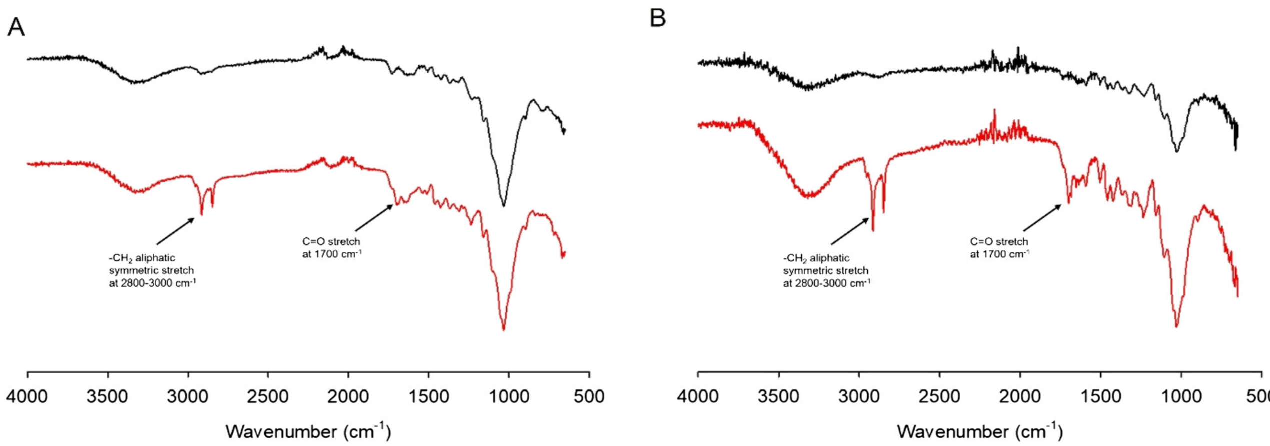

Figure 3: Fourier transform infrared spectroscopic analysis of wheat straw fibers. Legend for spectra: A) Native wheat straw fibers (black), lipase esterified wheat straw fibers (red). B) Native spotted gum (black), lipase esterified spotted gum fibers (red).

South Wales in Australia. Spotted gum mechanical pulp fibers were chosen for a comparison against wheat straw fibers for the lipase transesterification reaction with methyl myristate. We reacted wheat straw fibers with methyl myristate, a long chain aliphatic ester for trans-esterification of the primary alcohols on the surface of the fibers as displayed previously (Sharma et al. 2023b) using lipase as a catalyst. Post-washing to remove any unreacted constituents, a portion of the fibers was dried, and measurements were conducted using FTIR as shown in Figure 3 A and B. The chemical modifications are evident in the modified wheat straw fibers on the red spectra as there is a new carbonyl stretch at 1700 cm^{-1} and the aliphatic symmetric stretch between 2,800 and 3,000 cm^{-1} confirming successful functionalisation on both types of biomass. Unmodified and modified wheat straw and spotted gum were then added at 10 wt% in 60 gsm handsheets (1.2 g dry equivalent). In Figure 4 A-D, we show the effects of decorating the surface of wheat straw with non-polar ester groups. A water-soluble dye, Brilliant Blue was used to make a 1 % solution in water and 20 µL drops were deposited added on the surface of the 60 gsm sheets. There is a significant 51 % reduction in the spread of water drop as shown in Figure 4A and B whereas in Figure 4C and D there is a 36 % reduction on the surface by addition of hydrophobic wheat straw and spotted gum respectively. On the other hand, there is no significant difference in the water vapor transmission rates (WVTR) of these sheets. BEK sheets display a 381 g/m^2. day rate whereas the addition of modified wheat straw shows a display of only 376 g/m^2. day. The measurement of WVTR on these sheets is further evidence that the hydrophobicity on the surface is entirely due to interaction between repulsion between the polar groups of water and non-polar groups of the grafted ester.

Coffee pods were thermoformed as a demonstration of a packaging material and can be seen in Figure 5. The WVTR of this wheat straw coffee pod was measured to be 229 g/m^2. day which is quite high. However, the process shows fibers are sufficient to hold the pod together and none of the usual polymer additive for retention and wet/dry strength were used. In comparison to other woody pulps studied by Spence et al. (Spence et al. 2010), homogenised fibers are well within the range of our mechanically refined wheat straw. Bleached softwood and hardwood were reported to range from 200 to 240 g/m^2. day while unbleached softwood and hardwood were reported to be in the range of 220–460 g/m^2. day. The wheat straw is very similar to the water vapor transmission rates of bleached woody pulps in this respect. For packaging products, a WVTR below 100 g/m^2. day is more desirable as studied by Nair et al. (Nair et al. 2018). Further improvements could easily be made via addition of a polymer coating to reduce the WVTR further.

5 Conclusions

In this work, we displayed the potential of wheat straw as a valuable processing material for packaging. Disc refining was conducted without any chemical addition using enzyme sizing, highlighting a fully sustainable pulping method. The coarse material was shown as detrimental to the structure of handsheets, but the fibers produced have sufficient length and width to produce stable handsheets with a density of 363 kg/m^3 and tensile index of 15.8 N m/g. Enzymatic surface esterification was performed to show a water spread reduction of 51 % and 36 % on the surface of 60 gsm sheets with 10 wt% addition of wheat straw and spotted gum, respectively, in bleached Eucalyptus kraft

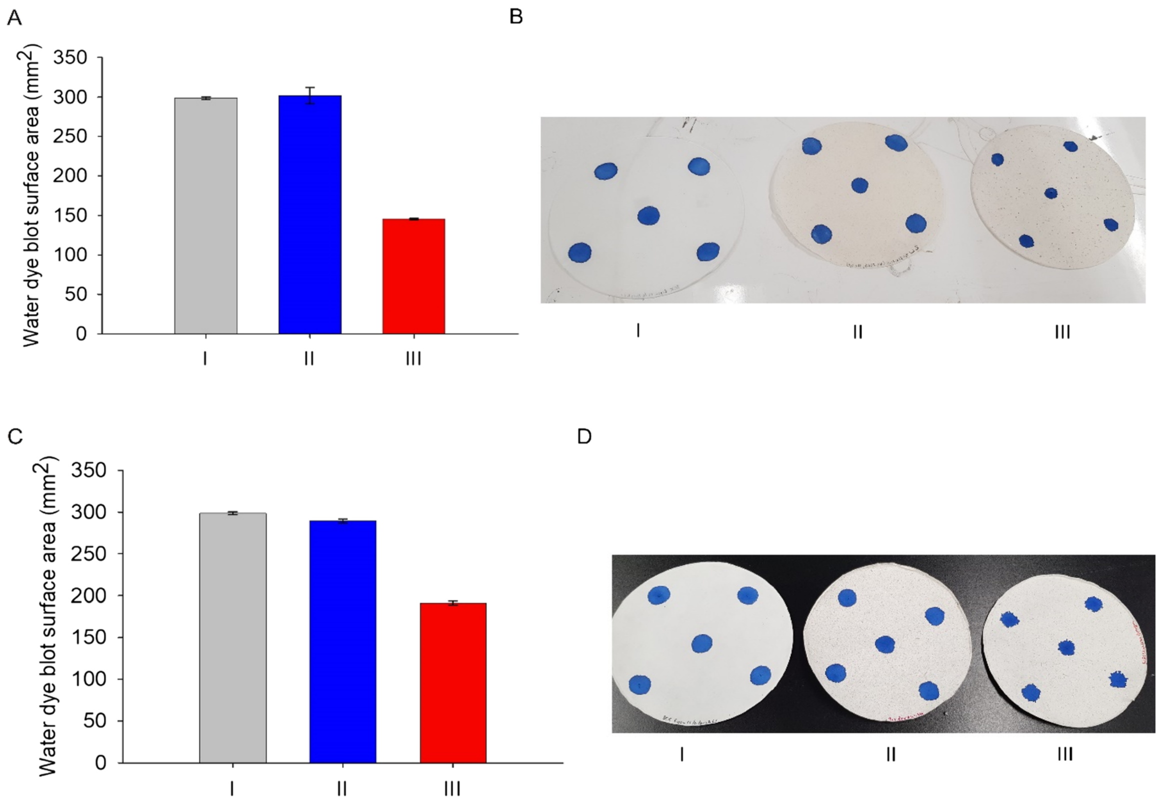

Figure 4: Assessment of surface water resistance of 60 gsm handsheets. A) Area of the spread of water droplets deposited onto handsheets containing wheat straw. B) Image of paper samples. Legend I: Native BEK fibers (grey), II: BEK containing 10 % w/w unmodified wheat straw fibers III: BEK containing 10 % w/w esterified wheat straw fibers (red). C) Area of the spread of water droplets deposited onto handsheets containing spotted gum fibers. B) Image of paper samples. Legend I: Native BEK fibers (grey), II: BEK containing 10 % w/w unmodified spotted gum fibers III: BEK containing 10 % w/w esterified spotted gum fibers (red).

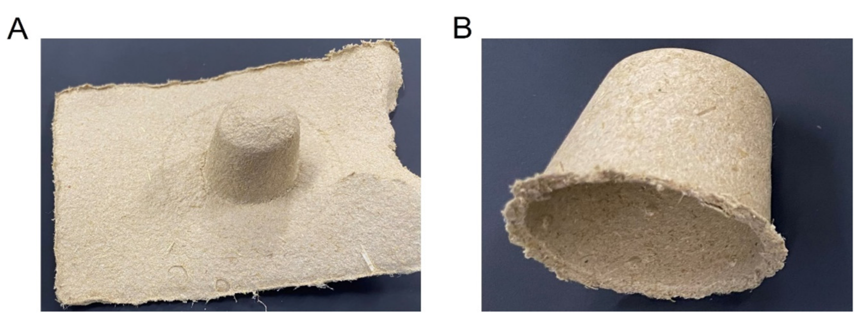

Figure 5: A) Thermoformed coffee pods made from wheat straw (300 gsm); B) single pod from sheet.

fibers. This is where enzymes provide a valuable alternative to more environmentally hazardous chemicals for functionalization and imparting hydrophobicity to wheat straw. Coffee pods were manufactured in a thermos-former to demonstrate how wheat straw can be utilised in an actual and finely engineered emerging product. As wheat straw is grown on all the habitable continents, it offers an attractive commodity for sustainable processing. In Australia, Spotted gum provides a strategic resource for use in papermaking as it is an important species managed for timber production, with large volumes of under-utilised harvest and processing residues. Due to the presence of unpulped coarse material in the fiber suspension, the water retention value (WRV) was not measured to avoid collusion. Mechanical processing of wheat straw biomass requires further improvement and optimization to meet the pulp stream industry standards where WRV is critical for manufacturing bio-based products. We have demonstrated that wheat straw is an excellent option for replacing both wood pulps and additives in wood pulp products. Further research in their mechanical pulping and assessment in pulp and paper technology will open new pathways for decarbonising the economy and provide an alternative to plastic packaging materials.

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