High-Yield Pulp from *Brassica napus* to Manufacture Packaging Paper

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The stalks that are left on the field after harvesting rapeseed crops could be used to make packaging grade paper. This work evaluates the suitability of mechanical and thermomechanical pulps from rapeseed stalks for papermaking, with a view to alleviating the limitations of recycled fluting. Their performance was compared to that of commercial fluting (recycled fluting) of the same basis weight, 100 g/m^2, and to that of virgin pulps from pine wood. The thermomechanical pulp was refined to improve key mechanical properties. Its drainability was found to be very low, even before refining, and its breaking length after beating to 1200 PFI revolutions, 4 km, surpassed that of sheets of recycled fluting that were obtained under similar conditions. These findings support the hypothesis that high-yield pulps from rapeseed stalks are a strong choice of virgin fibres to produce fluting and, generally speaking, packaging paper.

Keywords: Rapeseed; Mechanical pulping; Thermomechanical pulping; Fluting; SEM; Refining

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INTRODUCTION

Worldwide demand for packaging paper, including fluting paper, is clearly increasing and is expected to rise in the coming years (Visiongain 2015). Paper is a bio-based and biodegradable product, and it clearly fits within the framework of the bioeconomy, as described by the European Commission, which comprises those parts of the economy that use renewable biological resources from land and sea (European Commission 2016). Regretfully, paper production requires large amounts of these renewable but finite resources, specifically cellulosic fibres. Thus, the papermaking industry must implement several strategies such as viewing recycling as a central part of its activities and/or increasing the yield of unit operations in pulp and paper processes (Hubbe 2014).

Fluting is mainly produced by recycling paper, but this is subject to limitations (Miranda and Blanco 2010). Many cycles of use diminish the quality of the material and generate larger amounts of sludge. Fibres suffer from shortening, debris, and hornification. Hornification reduces the fibre's ability to swell, the bonding capacity and, thus, the resulting mechanical properties of paper (Hubbe 2014). The content of impurities in the finished pulps increases, while deinkability, *i.e.*, the ease of deinking, decreases (Levlin *et al.* 2010). Products from recycled fibres often have a lower density, which may be an advantage for printing paper grades (Hubbe *et al.* 2007), but likely not for packaging paper.

As a consequence, the process may require the addition of fresh fibres and dry-strength agents (Rundlöf *et al*. 2000).

Since paper recycling is a limited source of fibres, manufacturers still need to use virgin raw materials, mainly wood. However, wood prices, despite having decreased since their peak in 2011, still constitute the majority of the production costs of paper and board (Wood Resources International 2015). Some of the largest paper producers, such as China and the U.S.A., have relative scarcity of this resource.

A third way, then, lies in virgin fibres from agricultural waste. In European paper industries, production of pulp from non-wood raw materials increased from 187 kt in 2014 to 230 kt in 2015 (25.5%), while the production of conventional woodpulp decreased slightly, from 36,364 to 36,035 kt (CEPI 2016). Currently, most European countries are wood importers. Some farm residuals, such as cereal straw and bagasse, are already widely used for the manufacturing of paper and board (Vargas *et al.* 2015; Yue *et al.* 2016), but researchers are exploring different materials that may prove equally useful (Moral *et al.* 2016). The use of agricultural residues may lessen environmental concerns, as no fresh resources are used and the residue is valorised instead of being burned *in situ* or dumped. In this sense, the use of agricultural waste as raw material for the production of packaging paper not only presents technical and economic advantages, but also promotes the circular economy of natural resources. In order to make a significant contribution to the environment, besides using alternative raw materials, pulping processes should be designed to obtain the highest operation yield possible. This implies generating less residues and alleviates consumption of fibrous resources.

Rapeseed (*Brassica napus*) is a crop plant with green foliage, branched stems, and yellow flowers. The oil from the seed is edible, and it can be used to produce biodiesel, which explains why thousands of hectares of land in the last few years have been dedicated to new rapeseed crops in Germany, Poland, Canada, and India, among other countries. The European Union is the largest producer, accounting for approximately one third of world production (European Commission 2014). After harvesting the seeds, up to 7 tonnes of stems per hectare remain in the field, and the main stem cannot be used for animal feeding (Housseinpour *et al.* 2010).

The morphology and the potential of rapeseed stalks for the manufacturing of printing paper have been evaluated by several authors. Tofanica *et al.* (2011) found rapeseed stalk fibres to be from 0.71 to 1.99 mm long, in the high range, and from 9.10 to 19.60 μm wide. González *et al.* (2013) and Aguado *et al.* (2015) tested soda pulps and organosolv pulps from rapeseed stalks, concluding that they were suitable for papermaking. Nonetheless, to the best of our knowledge, no one has addressed the potential of this material for paper packaging through high-yield pulping.

The present paper evaluates the suitability of mechanical and thermomechanical pulps as raw materials for the production of fluting, aiming to suggest an economically and environmentally feasible process to complement recycling. However, laboratory sheet formers give isotropic sheets, in which fibres are randomly oriented, so they cannot compete with sheets made in a paper mill. To overcome this hindrance, comparisons are made with sheets obtained under similar conditions from commercial fluting. Rapeseed pulps are also compared to virgin pulps from pine wood, which is the main constituent of kraftliners and is generally found in the rest of packaging grade papers (Adamopoulos 2006). The morphology of the fibres and some key mechanical properties are analysed and discussed.

EXPERIMENTAL

Chemical Analysis

Rapeseed stalks were harvested in Castilla y León (Spain) and dried to a moisture content lower than 15%. Some samples were milled and screened, choosing the fractions in the range of 0.15 to 0.25 mm, following the TAPPI standard T224 wd-77 (TAPPI 2016).

After measuring gravimetrically the remaining moisture, the amount of extractives in 3 g of biomass was determined using a Soxhlet extractor with 200 mL of ethanol/toluene (1:2, v/v) for 6 h. Then, the biomass was filtered and washed with ethanol until the filtrate was colourless. Another sample of the same mass was submitted to combustion at 525 ºC to calculate the ash content according to the TAPPI standard T211 om-12. Holocellulose was quantified by drying and weighing the solid that remained after a reaction involving 3 g of dry biomass, 100 mL of distilled water, 1.5 g of sodium chlorite, and 1.5 mL of acetic acid. This reaction took place at a constant temperature of 70 ºC for 6 h (Wise *et al.* 1946). Klason lignin was determined by hydrolysing carbohydrates with 72% H_2SO_4 (w/w) at room temperature, then diluting to 4%, boiling at 120 ºC for 60 min, filtering and weighting the retained fraction (T222 om-11). All analyses were performed twice, reporting the average value plus/minus two times the standard deviation.

Pulping

Raw material was fractionated to a size lower than 5 mm. Some chips were subjected to mechanical pulping (MP) in a Sprout-Waldron mill after being soaked in water for 24 h. The resulting pulp was centrifuged to remove water and filtered over a nylon screen.

The other chips were used to produce thermomechanical pulp (TMP) by means of a stainless steel batch reactor. This reactor, filled with water, was preheated to 80 ºC before adding the raw material. The water-to-solid ratio was 6. The temperature was increased to 160 ºC and held at that value for 10 min by a PID controller. The solid was separated by filtration, washed with cold water, and beaten in the Sprout-Waldron unit. Figure 1 shows a schematic diagram of the experimental procedure.

For comparison purposes, these procedures were repeated with chips of maritime pine (*Pinus pinaster*) wood to obtain mechanical and thermomechanical pulps.

Disintegrating and Refining

Samples of mechanical and thermomechanical pulps were diluted to a consistency of 1.5% and stirred at 3000 rpm for 10 min, by means of a pulp disintegrator from Lorentzen & Wettre.

Industrial fluting with a nominal basis weight of 100 g/m^2, produced mainly from recycled paper, was provided by SAICA (Spain). It was submitted to mechanical tests as received. At the same time, a fraction of it was soaked in water for 24 h, then disintegrated at 3000 rpm for 10 min.

The thermomechanical pulp from rapeseed stalks was processed in a PFI mill to various levels of refining, including 400, 800, and 1200 PFI revolutions. Part of the pulp obtained by disintegrating the fluting sheets was beaten to 1000 PFI revolutions.

The degree of refining of all pulps was quantified by the change in drainability. This effect was estimated by duplicate tests with a Schopper-Riegler tester that meets the ISO standard 5267-1 (ISO TC/6 2003), and measures the amount of water that was discharged from a pulp suspension.

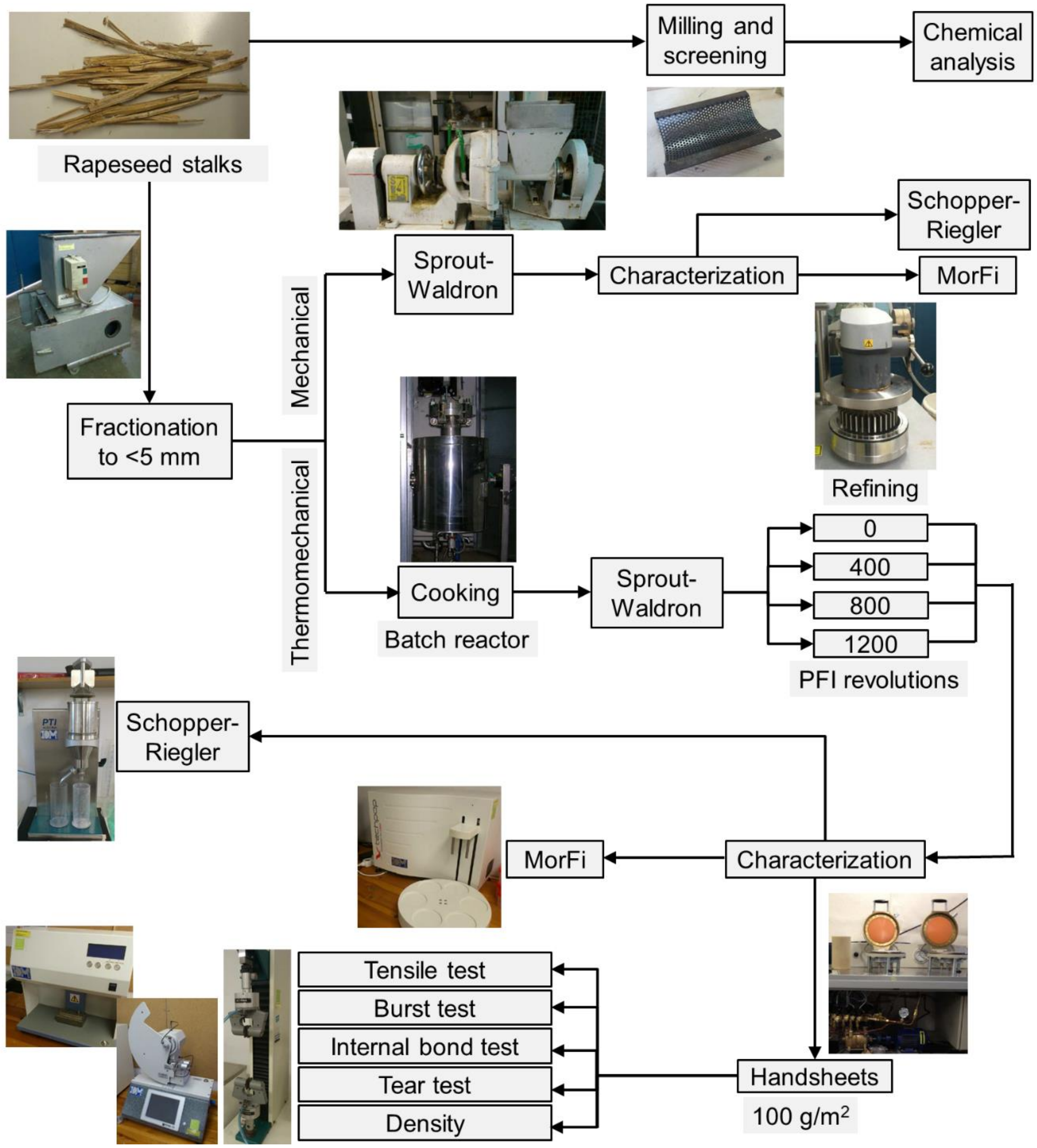

Fig. 1. Experimental procedure regarding the mechanical and thermomechanical pulp

Characterization of Pulps

Sheet formation

Up to 10 handsheets with a grammage of 100 g/m^2 were made from each of the pulps. The ISO standard 5269-2 was followed, including the use of standard plates, standard couch weights, standard stirrer, and a system to recycle water. Sheets were dried while pressed between standard rings, at 23 ºC and at a relative humidity of approximately 50%, for 24 h.

Testing

Scott's bonding strength was determined according to TAPPI standard T569 om-14, preparing the samples with a pressure of 5 to 8 bar and using an internal bond tester from IDM Instruments (Australia).

The tensile test for the breaking length was performed by means of an INSTROM dynamometer (Pennsylvania, USA), cutting a 180-mm-long and 15-mm-wide strip from each of the sheets (ISO 1924). The burst index was measured with a digital hydraulic tester from IDM Instruments, slowly increasing pressure until burst (ISO 2758). Properties are reported as the mean plus/minus two standard deviations.

The thickness in each case was measured by stacking five sheets of the same pulp and using a motor-driven micrometer. Then, bulk was calculated as the ratio of average thickness to grammage (T220 sp-01). The reciprocal of bulk is the apparent density.

A 50 mL pycnometer for liquids and solids was used to determine the real density of paper. A known mass of dry fibres was soaked for 24 h and placed in the pycnometer, which was then filled with distilled water. Its weight was compared to that of 50 mL of distilled water. Porosity was calculated from the ratio between apparent density and real density.

Morphology

In each case, 2 g of mechanical or thermomechanical pulp was diluted in 600 mL of distilled water. The sample was then placed in a MorFi device (Morfi V7.9.13.E, Techpap, France) until it counted 5000 images. Out of all parameters that are reported by this device, the fibre length, the fibre width, and the percentage of fines were selected for reporting. The slenderness ratio expresses the relation between fibre length and fibre width. The proportion of fines in pulps is quantified as the percentage in length (Moral *et al.* 2010).

Images were taken with a scanning electron microscope (SEM) from JEOL, model JM-6400. Each sample was put on a cylindrical slide and dried at 45 ºC and 200 mbar for 24 h. Then, it was coated with gold and visualized with a magnification of 100 and 1000 times.

RESULTS AND DISCUSSION

Chemical Composition of Rapeseed Stalks

The chemical composition of the harvested stalks is presented in Table 2, along with the composition of other potential and actual sources of fibres for papermaking. The amount of ethanol-toluene extractives (this work) is assumed to be similar, or at least comparable, to that of ethanol-benzene extractives. This percentage, 8.5%, is in the high range.

Rapeseed stalks were found to contain more lignin than some other apparently similar materials, such as sunflower stalks and sorghum stalks, yet less than conventional raw materials for papermaking, such as pine wood and eucalyptus wood. Holocellulose in this biomass accounted for the 68.4% of the raw material, akin to that of wood. Overall, the composition of rapeseed stalks was more similar to that of woody agricultural residues than to that of annual plants, regularly known as common and specialty nonwoods, respectively.

Table 1. Composition of Rapeseed Stalks, Compared to Some Other Sources of Fibres

Fibre source	Holocellulose (%)	Klason lignin (%)	Ethanol-benzene extractives (%)	Ashes (%)	Reference
Rapeseed stalks	68.4 ± 3.1	18.3 ± 1.9	8.5 ± 1.1*	4.8 ± 0.8	Present work
Sunflower stalks	71.8	13.4	4.07	7.9	López *et al*. (2005)
Vine stems	65.4	28.1	11.3	3.9	Mansouri *et al*. (2012)
Olive tree pruning	61.5	19.7	10.4	1.4	Requejo *et al*. (2012)
Sorghum stalks	71.7	13.4	6.3	4.8	Jiménez *et al.* (1993)
Rice straw	60.7	21.9	0.56	9.2	Rodríguez *et al.* (2008)
Pinus pinaster wood	69.6	26.2	2.6	0.5	Alonso (1976)
Eucalyptus globulus wood	68.1	25.0	3.1*	0.8	Ligero *et al*. (2008)

*Ethanol-toluene extractives.

Characterisation of Pulps and Fibres

Mechanical pulping is the process that results in the least waste. As shown in Table 2, both mechanical pulps were obtained with a yield close to 100%. Although the treatment was the same, rapeseed MP achieved a higher Schopper-Riegler degree than pine MP. The percentage of fines (< 100 μm) was 46.7%, similar to the proportion of fines found in wood pulp. Nonetheless, values of average diameter and length of rapeseed MP fibres were lower than those of pine MP fibres. The average breaking length of both mechanical pulps, that from pine wood and that from rapeseed stalks, was much alike. However, this value was too low to achieve enough strength, and it is not likely to be greatly improved by refining, which would make the fibres even shorter.

The results found for rapeseed MP provided encouragement to increase the severity of the treatment TMP from rapeseed stalks. The pulp was produced with a yield of 90%, which was lower than that of pine TMP, given the dissolution of a part of the extractives in hot water. A drainability of 65 °SR, which is very high for unrefined pups, was obtained. Fibres were found to be more slender than those of rapeseed MP, as their slenderness ratio was found to be close to that of pine wood fibres. The percentage of fines was as high as 60%, which explains the low drainability. Moreover, it explains the good performance of rapeseed TMP sheets in the tensile test, their breaking length being noticeably higher than the value for pine TMP sheets. Comparatively, González *et al.* (2013) reported some results from a chemithermomechanical pulp from rapeseed stalks with a considerably lower content of fines and, consequently, a lower Schopper-Riegler degree (34 °SR). This negatively affected the breaking length of the paper sheets (2609 m). As reported by Page (1969), the proportion of fines is one of the most relevant factors influencing paper strength, along with fibre slenderness and fibre-to-fibre bonds.

Figure 3 shows the microscopy images of rapeseed MP and rapeseed TMP. The mechanical pulp was found to contain more non-fibrous elements and thick fibre bundles, bound together by lignin (Fig. 2a). The surface of a randomly selected fibre (Fig. 2b) was smooth. Fibres were more isolated after thermomechanical pulping (Fig. 2c), which eases papermaking. As can be seen in Fig. 2d, external fibrillation was more noticeable in rapeseed TMP, as many microfibrils protruded from the surface of the fibres.

When pulps are mechanically refined, the bonding area, and the content of fines and fibrillation (both external and internal) increase, leading to denser papers and stronger interfibre bonds (Page 1969; Axelsson 2009). The effect of refining on rapeseed TMP is presented in Fig. 3.

Fibres were shortened (Fig. 3a), which may be detrimental for some mechanical properties, but the percentage of fines reached 69% after 1200 PFI revolutions (Fig. 3c). As for the average fibre width (Fig. 3b), its values do not follow a clear trend. Fibre diameter can either decrease due friction between the fibre and the rotor bars of the PFI mill, or increase due to swelling.

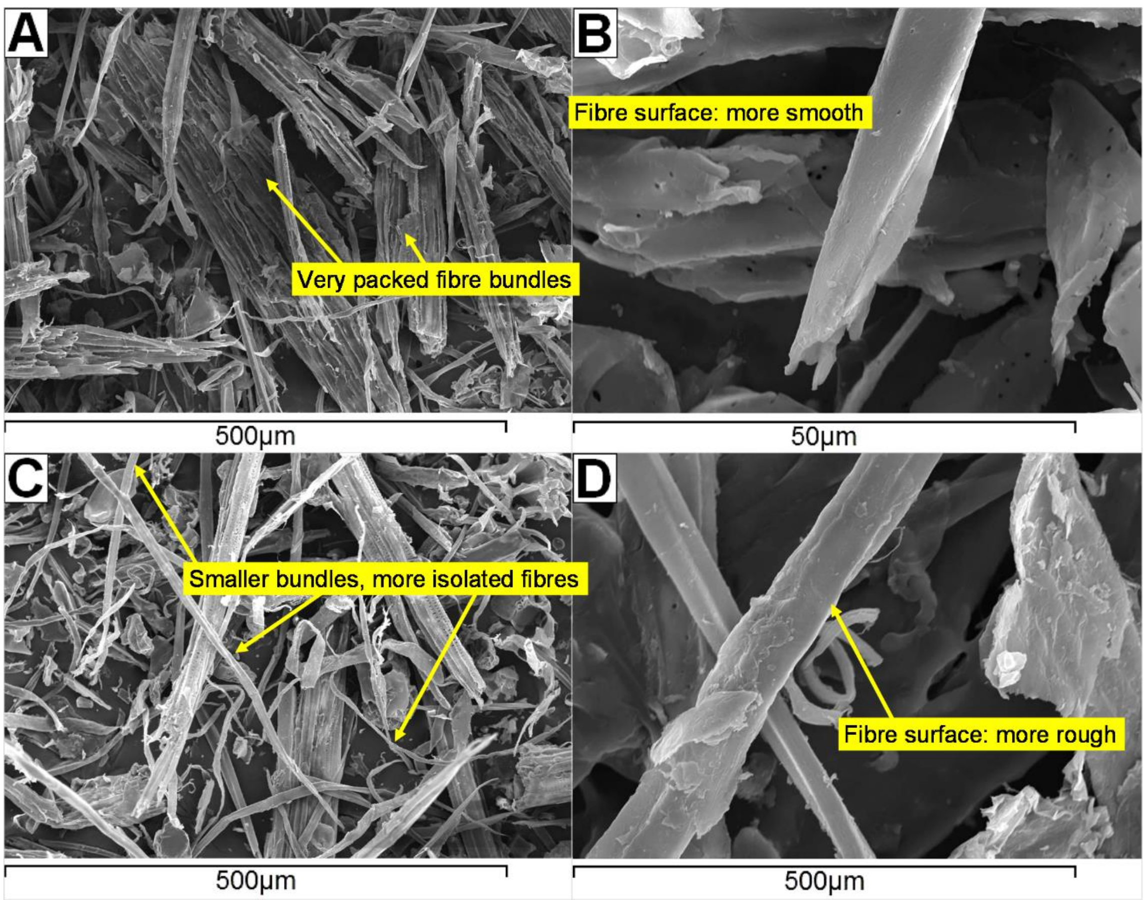

Fig. 2. SEM images of the mechanical pulp from rapeseed stalks with a magnification of 100 (a) and 1000 (b), and of the thermomechanical pulp with a magnification of 100 (c) and 1000 (b)

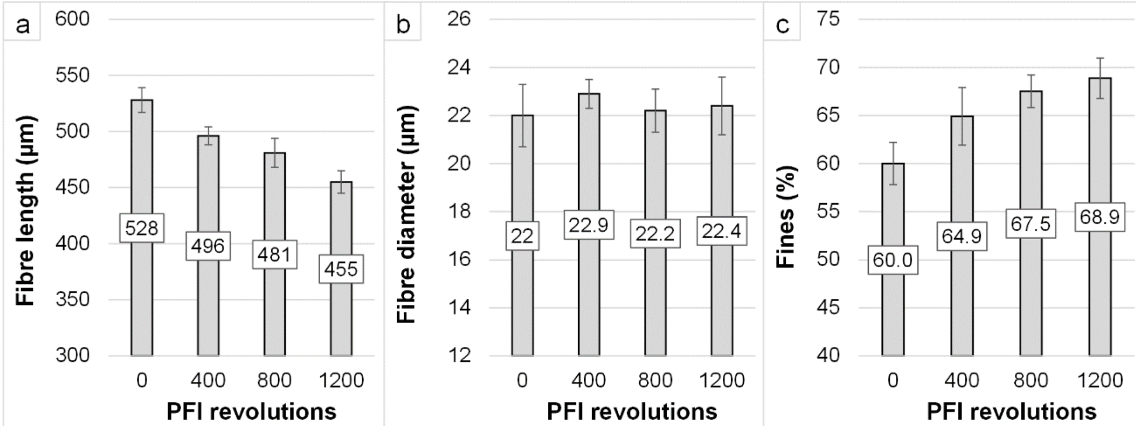

Fig. 3. Effect of refining on the average fibre length (a), on the average fibre width (b) and on the percentage of fines (c) in the thermomechanical pulp

Table 2. Properties of High-Yield Pulps from Rapeseed Stalks, Compared to those of High-Yield Pulps from Pine Wood

	MP		TMP	
Pulp	Rapeseed	Pine	Rapeseed	Pine
Yield (%)	99	98	90	95
Length weighted in length (µm)	400 ± 11	643 ± 9	528 ± 11	629 ± 16
Diameter (µm)	22.1 ± 0.3	27.0 ± 0.6	22.0 ± 1.3	27.0 ± 0.9
Aspect ratio (L/d)	18.1	23.8	24.0	23.3
Fines (%)	46.7 ± 2.1	40.5 ± 1.6	60.0 ± 2.2	49.6 ± 3.0
Schopper-Riegler degree (ºSR)	42	34	65	43
Breaking length (m)	1009 ± 21	1190 ± 19	3300 ± 149	1853 ± 96

MP: mechanical pulp. TMP: thermomechanical pulp.

Mechanical Properties of Rapeseed TMP and Recycled Fluting Papers

Industrial fluting was characterized with the purpose of establishing some physico-mechanical requirements to be achieved through the use of rapeseed stalks pulps. Table 3 shows the mechanical characterization of the recycled fluting sheets as received, tested in the longitudinal direction of the web (or machine direction) and in the transverse or cross-machine direction. It also presents key characteristics of the sheets made at laboratory, following the same procedure as for rapeseed pulps.

As can be seen from Table 3, the most notorious difference between the longitudinal direction and the transverse direction is reflected in breaking length, where the orientation of fibres remarkably influenced the resulting paper strength. However, the internal bond and the tear index presented their highest values when papers were tested in the cross direction. The number of bonds per volume unit is higher in the transverse direction. Likewise, the fissure propagates better when paper is tested in the machine direction, leading to slightly less tear resistance. To overcome the loss of paper strength caused by hornification, papermakers usually refine the recycled pulp, leading to higher values of key mechanical properties. Regarding the sheets made with a laboratory sheet former, in which fibres were randomly oriented, their performance in tensile tests and burst tests were improved by refining to 1000 PFI revolutions (Table 3). Tear index was slightly decreased, while both density and porosity remained almost constant. Internal bond was not measured due to equipment limitations. The Schopper-Riegler degree was high, as expected for this kind of paper from secondary fibres.

Table 3. Properties of Anisotropic and Isotropic Sheets from Recycled Fluting

	Anisotropic sheets (commercial, as received)		Isotropic sheets (formed at laboratory)	
Property	Longitudinal direction	Transverse direction	0 PFI	1000 PFI
Breaking length (m)	6335 ± 201	2387 ± 99	3710 ± 142	4092 ± 173
Internal bond (J/m^2)	250 ± 10	266 ± 10	228.8 ± 9.9	-
Tear Index	5.5 ± 0.1	6.4 ± 0.3	8.2 ± 0.3	7.8 ± 0.3
Bulk (cm^3/g)	1.4 ± 0.2		1.56 ± 0.31	1.56 ± 0.12
Porosity (%)	54 ± 4		57.53 ± 6.88	57.40 ± 2.75
Burst Index (kPa·m^2/g)	2.18 ± 0.28		2.15 ± 0.29	2.55 ± 0.13
Schopper-Riegler degree (ºSR)	46		46	51

It is worth mentioning that, like recycling itself, refining of recycled fibres is also subject to limitations and cannot replace the addition of fresh fibres. 1000 PFI revolutions only caused a small increase in the breaking length, from 3.7 km to 4.1 km (Table 3). This is due to the fact that those fibres had already been refined, and thus little improvement was possible. In contrast, refining of virgin fibres achieved a higher relative gain of paper strength at the cost of less energy. Even more energy can be saved by using non-wood raw materials (Aguado *et al.* 2015; Gharehkhani *et al.* 2015).

Table 4 and Figure 4 show the convenience of using the thermomechanical pulp from rapeseed stalks for fluting production. Rapeseed TMP was slightly refined, up to 1200 revolutions in the PFI mill, while virgin pulps from conventional materials usually undergo more than 2000 revolutions (Cheng *et al.* 2013), but it was enough to achieve high indicators of paper strength. The tear index decreased (Table 4), probably due to the diminishment in fibre length. As the gaps between fibres were filled by fines generated by refining, bulk and porosity of paper sheets decreased. Breaking length, internal bond, and burst index were notably increased with refining, given the increment in the bonding area. However, the Schopper-Riegler degree was considerably higher than that obtained for recycled and refined fluting, auguring a poor drainage behaviour in the paper machine. The breaking length obtained at a refining level of 800 revolutions (Fig. 4) was similar to that of the recycled fluting after 1000 revolutions, indicating that 800 PFI revolutions could be enough to produce fluting from high-yield pulps of rapeseed stalks.

Table 4. Properties of Isotropic Sheets from Rapeseed TMP *vs.* Refining Levels

Property	PFI Revolutions			
	0	400	800	1200
Internal bond (J/m^2)	321 ± 12	359 ± 12	404 ± 12	426 ± 16
Tear index (mNm^2/g)	3.2 ± 0.1	2.9 ± 0.1	2.60 ± 0.1	2.40 ± 0.2
Bulk (cm^3/g)	2.0 ± 0.1	1.9 ± 0.2	1.9 ± 0.4	1.8 ± 0.1
Porosity (%)	67.33	65.33	64.7	64.0
Schopper-Riegler degree (ºSR)	65	67	69	71

An asymptotic tendency was observed for refined TMP papers (Fig. 4), suggesting that pulps should not be further refined or, at least, that more PFI revolutions would not be translated into much higher mechanical properties. In addition, further refining, besides consuming more energy, could cause structural damages that limit the performance of the fibres (Hubbe 2014; Gharehkhani *et al*. 2015). This undesired behaviour as pulp is further refined has been also observed for other pulps, regardless of the origin of the raw material and the pulping process (González *et al*. 2012, 2013; Delgado-Aguilar *et al*. 2015).

In any case, the comparison between rapeseed TMP and commercial fluting indicate the feasibility of using alternative raw materials for the production of brown line paper, relieving both the recycling sector and wood resources exploitation. Refined TMP from rapeseed stalks could be also mixed with recycled fluting, extending the lifespan of the fibres.

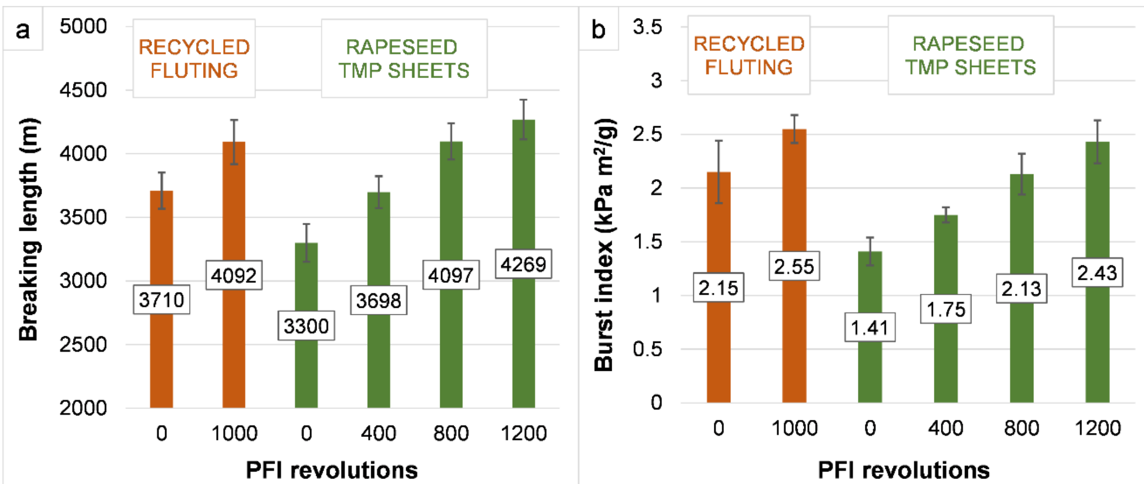

Fig. 4. Effect of refining in the breaking length and burst index of isotropic sheets from commercial fluting and from rapeseed stalk high-yield pulp.

CONCLUSIONS

1. High-yield pulps of rapeseed stalks, a material consisting mainly of holocellulose (68%) and lignin (18%), can be used for the production of packaging grade paper.
2. Thermomechanical pulping (TMP) is recommended over mechanical pulping, despite the lower yield (90%). The thick fibre bundles in the mechanical pulp from rapeseed stalks were translated into less strength.
3. The breaking length and the burst index of unrefined rapeseed TMP were higher than those of conventional high-yield pulps, pine MP and pine TMP, albeit lower than those of commercial fluting.
4. Refining generated fines and enhanced the internal bond strength, resulting in a better performance in tensile and burst tests. 800 PFI revolutions were enough to achieve a breaking length of 4.1 km when using sheets with a basis weight of 100 g/m^2.
5. The results showed the feasibility of exclusively using rapeseed pulp as raw material for the production of fluting paper or, at least, the convenience of incorporating moderate percentages of this pulp to the current recycled fibres.

ACKNOWLEDGMENTS

The authors acknowledge the Ministry of Science and Innovation of Spain for financial support from the Project CTQ2010-21660-C03-01.

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Article submitted: September 9, 2016; Peer review completed: November 20, 2016; Revised version received and accepted: January 11, 2017; Published: February 23, 2017.
DOI: 10.15376/biores.12.2.2792-2804