PAPERMAKING POTENTIAL OF CANOLA STALKS

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A fundamental study was carried out to explore the properties of canola stalks with regards to pulp and paper production. In this study the morphological properties, chemical composition, and soda pulping properties of canola stalks were investigated. The mean values of length, diameter, and cell wall thickness of canola stalks fibers were measured as 1.17 mm, 23.02 μm, and 5.26 μm, respectively. The morphological properties analysis indicated that despite the thicker cell wall, the morphological properties of canola stalks fibers were comparable to those of non-woods and hardwoods fibers. The holocelluloses, alpha-cellulose, lignin, and ash contents of canola stalks were determined to be 73.6, 42.0, 17.3, and 8.2 wt%, respectively. The hot water and dilute alkali extractives of canola stalks were determined as 18 and 46.1 wt%. In comparison to most other non-wood papermaking raw materials, soda pulping of canola stalks required higher chemical charge and cooking time. Soda pulping of canola stalks gave a low yield bleachable grade pulp. The strength properties of bleached canola stalks soda pulp appeared to be similar to those of common non-wood papermaking resources. The overall results showed that canola has a promising potential to be used in pulp and paper production.

Keywords: Fiber resources; Canola stalks; Fiber morphology; Chemical composites; Soda pulping; ECF Bleaching

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INTRODUCTION

The worldwide consumption of paper and board products increases continuously due to several reasons, e.g., population growth, better literacy, development of communication, and industrialization in developing countries. Although wood is the major source of fiber supply for papermaking, the lack of forest resource in many areas of the world imposes the use non-woods fibers as important alternative fibrous sources for papermaking industries. In 2005, global production of virgin pulp for paper and paperboard was 187.6 million metric tons, from which 17.4 million metric tons made from non-wood fibers, accounted for 9.27% (Bowyer et al. 2007).

At present some agricultural residues such as wheat and rice straws, sorghum stalks, and some annuals plants such as hemp and jute are used as raw materials for pulp and paper production (Rousu et al. 2002; Ashori 2006). Moreover, numerous studies have been performed to introduce new lignocellulosic fiber resources for pulp and paper industries (Antunes et al. 2000; Law et al. 2001; Sarwar Jahan et al. 2006; Shatalov and Pereria 2006; Tran 2006; Wanrosli et al. 2007).

Canola (*Brassica napus L.*) is widely cultivated throughout the world and used as a major oilseed for vegetable oils and biodiesel production. As a result of high demand for vegetable oils and biodiesel, the worldwide planted area for canola increases continuously. In addition to oil production from canola seed, the leaves of canola can be used as an animal feed (Banuelos et al. 2002). In 2003, the worldwide planted area of canola was more than 23 million hectares. China, Canada, India, France, Germany, and Australia are the major producers of Canola (FAO 2003). The total amounts of biomass produced per unit area by canola depends on irrigation and varies from 5 to 10 t/ha. Approximately 20 percent of canola biomass includes the stem portion that remains in the field after canola seed collection (Banuelos et al. 2002). Thus, it can be concluded that at least 34 million tons of canola stalks is available annually which could be used in various products, including pulp and paper productions.

The properties of canola stalks fibers for pulp and paper production have never been explored in the literature. Therefore, in this study the morphological properties, chemical composition, and soda pulping properties of canola stalks were investigated to evaluate the potential utilization of canola stalks fibers in pulp and paper production.

EXPERIMENTAL

Raw Material

Air-dried canola stalks used in this study were collected in the spring of 2007 from a recently harvested canola field at Mazandaran province in the north of Iran. The air-dried canola stalks mainly consisted of stems and side branches (Fig. 1). Air-dried canola stems were 1-2 cm in diameter and about 80-100 cm in length with a moisture content of 7.5%.

Moreover, canola stalks contain a pith portion, which is located in the centre of the canola stem. The pith content was completely removed manually, and its percentage was calculated gravimetrically as dry pith over the initial dry canola stalks. The pith content was determined to be 7.9 wt% of dry weight of canola stalks.

Generally, the pith of non-wood plants is composed mainly of parenchyma cells and causes serious problems such as increasing chemical consumption in pulping and bleaching, washing problems in pulp washing, and drainage problems in papermaking. Before pulping canola stalks, the pith content of canola stalks was removed. Briefly, air-dried canola stalks were chipped in a hammer mill to obtain depithed chips with the length of 2-3 cm and pith particles with a 0.1-0.5 cm in diameter. Thereafter, the light density pith particles were removed using an air-separator from depithed chips. This technique was found to be simple and effective for depithing of canola stalks, since 95 wt% canola stalks pith content was separated from the canola stalks chips.

FIG. 1. Photograph of air-dried canola stalks

Fiber Morphology Analysis

Initially, several depithed chips were chosen randomly and were cut into about 10 mm pieces. The transverse section of canola stalks (about 30 μm in thickness) was prepared from stalks that immersed in distilled water at room temperature for 24 hours, using a microtome (Sledge Microtome G.S.L.1, Schenkung Dapples, Switzerland) and stained with 1% o-safranin. The microtome samples of transversal section were viewed by light microscopy equipped a CCA camera. For fibers separation without fiber breaking, the pieces of canola stalks chips were treated in a mixed solution (50/50, v/v) of acetic acid and hydrogen peroxide (solution 33%) for a period of 24 hours at 60°C. Then, the samples were washed by distilled water three times, and the fibers were separated into individual fibers by gentle shaking. The percentage (weight basis) of fiber was calculated gravimetrically. The obtained individualized fibers were stained with 1% aqueous safranin-o solution and placed on 20 glass microscope slides (cell walls retain a characteristic reddish color). The length of fibers was measured using a projection microscope. The fiber diameter and lumen width were measured with a microscope equipped with a filar micrometer eyepiece with a magnification of 400X. The fiber wall thickness was calculated as a difference of fiber diameter and lumen width divided in half. For each measurement 10 unbroken fibers were measured on each slide, resulting in 200 fibers measured. From these data, the average fiber dimensions were calculated and then the following derived indexes were determined.

$$\text{Runkel ratio} = 2 \times \frac{\text{Wall thickness}}{\text{Lumen width}} \quad (1)$$

$$\text{Flexibility ratio} = \frac{\text{Lumen width of fiber}}{\text{Diameter of fiber}} \times 100 \quad (2)$$

$$\text{Slenderness ratio} = \frac{\text{Length of fiber}}{\text{Diameter of fiber}} \quad (3)$$

Chemical Composition

The depithed canola stalks (40-60 mesh) were initially extracted according to modified T 264 cm-97 TAPPI Test Method using acetone instead of ethanol-benzene (1:2, v/v). The chemical compositions of extractive free canola stalks were determined according to the following TAPPI Standard Methods.

Hot water solubility, T207 cm-99
1% NaOH solubility, T212 om-98
Acetone extractives, T204 cm-97
Ash content, T211 om-93
Klason lignin, T222 om-98

Holocelluloses (sum of the cellulose and hemicelluloses) content of the extractive-free sample was determined according to Wise et al. (1946). Alpha-cellulose content was measured according to T203 cm-99 TAPPI Test Method. All measurements were repeated three times.

Pulping, Bleaching and Handsheet Forming

In general, the soda pulping process is known as a more suitable chemical pulping process for annual plants (Antunes et al. 2000; Nezamoleslami et al. 1998). The soda pulping of canola stalks was performed in a 2.8 liter pressurized rotating digester heated in thermostatic oil bath. Soda pulping of canola stalks was performed under the following conditions to obtain bleachable grade pulps:

- Alkali charge (% NaOH): 15 to 22% based on dry chips of canola stalks,
- Liquor-to-chips ratio: 8:1,
- Maximum cooking temperature: 170 ^{o}C,
- Time to maximum cooking temperature: 90 minutes,
- Cooking time at maximum temperature: 60, 80, and 100 minutes.

At the end of cooking the digester was cooled rapidly using tap water. A sample of 100 ml of the black liquor was taken for pH measurement. The reactor contents were filtered, and the resulting solid was washed with tap water on a sieve of 200 mesh. Then, the obtained pulp was mechanically disintegrated (with 3000 rpm and 2.5 min) in a standard disintegrator (TAPPI T 205 sp-95). Total yield of pulps was calculated as the percentage of the dry pulp over the initial dry raw material. Screen yield of pulps was determined based on dry pulp which passed through a screen of 0.15 mm mesh size. The Kappa number of pulps was determined according to T 236 om-06 TAPPI Test Method. The pulp viscosity, which is indicative of the damage of the cellulosic chain, was determined according to the T230 om-89 TAPPI Test Method using cupriethylendiamine (CED) as a solvent and a capillary viscometer. The degree of polymerization (DP) of the fibers was calculated using the following equation, where μ is the measured TAPPI viscosity (cP).

$$DP^{0.905} = [0.75\,(954 \log \mu - 325)] \quad (4)$$

Based on the principle of higher accepted yield, higher viscosity of pulp, and lower kappa number, the optimum pulp was chosen for bleaching and handsheet evaluation. At present, the ECF bleaching processes are known as well-established technology for bleaching of chemical pulps (Hamzeh et al. 2007). The ECF bleaching sequences have been applied successfully for bleaching of chemical pulps from non-woods plants (Jimenez et al. 1996; Riddlestone, 2001; Braunstein, 2004; Tschirner et al. 2007). The obtained soda pulp of canola stalks was bleached in a DEpD sequence. The applied pure ClO_2 solution was prepared by acidification of a $NaClO_2$ solution. The D_0 and Ep stages were performed at 10% consistency in polyethylene bags at 70°C in a temperature controlled water bath for 60 minutes. The D_1 stage was performed at 10% consistency, 70°C for 150 minutes. Unrefined bleached pulp was used to make 60 g/m^2 handsheets. The handsheets were made according to T 220 sp-06 TAPPI Test Method. Then they were conditioned at 23 ^{o}C and 50% R.H. for 24 hours according to TAPPI T205 sp-95. The burst, tear, and tensile strengths of handsheets were determined according to T 403 om-97, T 414 om-98, and T494 om-96 TAPPI Test Methods, respectively. The brightness of the handsheets was measured using a Technibrite Micro TB-1C instrument according to T452 om-98 TAPPI Test Method. The reported results represent the average values of 5 handsheets.

RESULTS AND DISCUSSION

Fiber Dimensions and Derived Indexes

Figure 2 shows individual fibers as well as other cell types of canola stalks, including parenchyma cells and vessels.

Fig. 2. Morphological features of canola stalks fibers (LM×40), (F; fibers, P; parenchyma cells and V; vessels).

The general structure of cross section of canola stalks from bark to core is displayed in Fig. 3. Phloem (bark), cambium, xylem, and pith sections are shown together. Phloem occupies a narrow space and its outer part is covered by epidermis cells. Cortex is present under the epidermis. The cambium layer is distinguishable between phloem and xylem. Xylem cells are distinguishable by their corrugated shape. The vessels (tracheal tubes) are diffused through the xylem. The pith part is large and consists of thin-walled parenchyma cells. The average fiber content of canola stalks is 61% (weight basis).

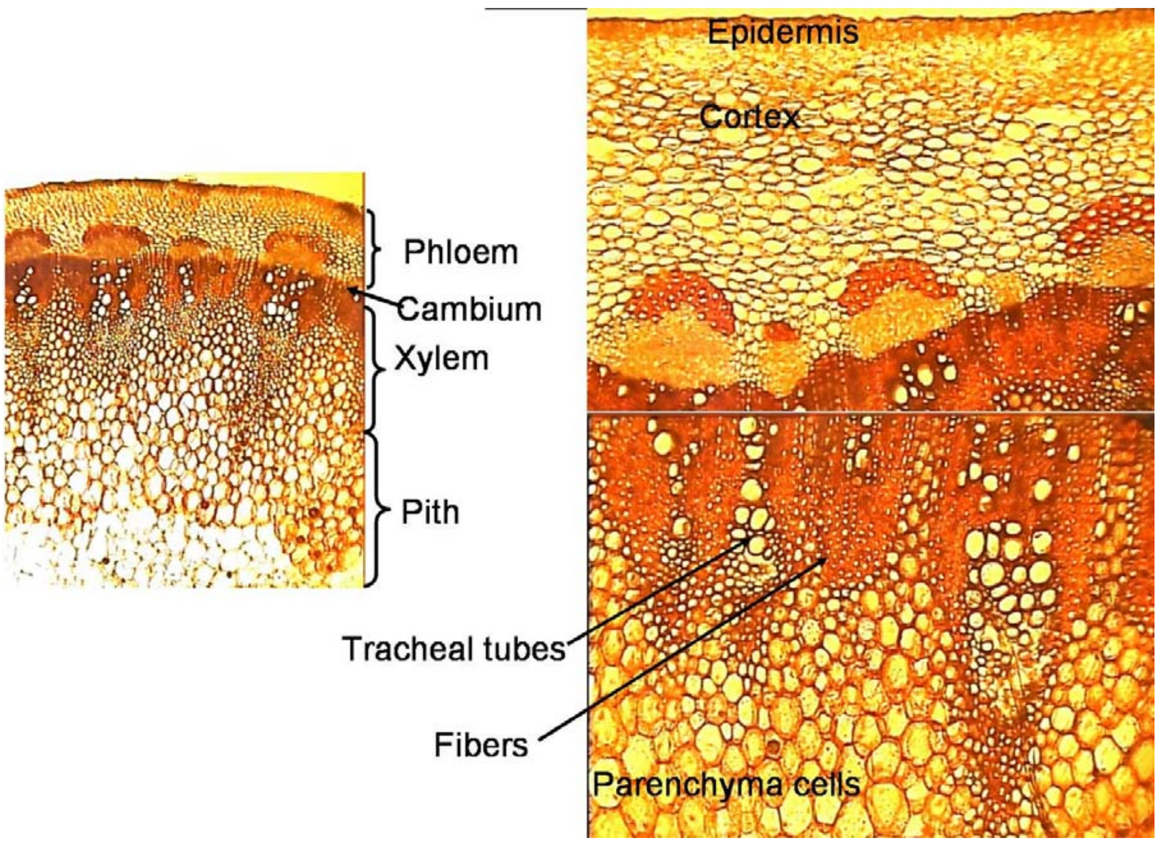

Fig. 3. Structure of stem tissue of canola (magnification of 10X at left and magnification of 40X at right)

The morphological properties of canola stalks and their comparison with common papermaking fiber resources are summarized in Table 1. The results show that the canola stalks contained short fibers with a mean length of 1.17 mm. The canola stalks fibers are as long as the short fibers of non-wood plants such as bagasse. However, they are longer than wheat straw, cotton stalks, and aspen fibers. The fiber diameter and lumen width of canola stalks fibers are similar to those of aspen, bagasse, and cotton stalks fibers. On the other hand, cell wall thickness of canola stalks fibers is thicker than those of cited wood and non-wood fibers. Consequently, the calculated Runkel ratio for canola stalks fibers (0.84) is higher than that of bagasse, cotton stalks, and aspen fibers. The slenderness ratio of canola stalks fibers is 50.6 and is comparable with that of bagasse and aspen fibers and higher than that of cotton stalks fibers. Generally, the acceptable values for slenderness ratio and Rankle ratio of papermaking fibers are more than 33 and less than 1, respectively (Xu et al. 2006). Referring to this and morphological properties of canola stalks fibers, it can be deduced that the canola stalks fibers can be collapsed to form ribbon like structures in the paper and that the overall morphological properties of canola stalks fibers are satisfactory for papermaking, although they would be classified as short fibers.

Table 1. The Mean Values of Canola Stalk Fiber Dimensions and Derived Indexes - Comparison with Common Papermaking Fibers

Fiber properties	Canola stalks	Wheat straw [(a)]	Bagasse[(b)]	Cotton stalks[(c)]	Aspen [(d)]
Length (mm)	1.17	0.74	1.13	0.83	0.96
Diameter (µm)	23.02	13.20	20.00	19.60	20.80
Lumen width (µm)	12.50	4.02	12.00	12.80	16.94
Cell wall thickness (µm)	5.26	4.59	4.00	3.40	1.93
Runkel ratio	0.84	2.28	0.67	0.53	0.23
Slenderness ratio	50.83	56.06	56.50	42.35	46.15
Flexibility ratio	54.30	30.45	60.00	65.31	81.44
a: (Deniz et al. 2004), b: (Sanjuan et al. 2001), c: (Ververis et al. 2004), d: (Law and Jiang, 2001).					

Chemical Characteristics

Table 2 shows the mean values of chemical composition of canola stalks and its comparison with wheat straw (Deniz et al. 2004), fiber fraction of bagasse (Wang and Patt, 1989), Egyptian cotton stalks (Ali et al. 2001), rice straw (Rodriguez et al. 2008), and poplar (Law et al. 2001).

Table 2. The Chemical Composition of Canola Stalks and some Typical Papermaking Raw Materials (% on OD basis)

Component	Canola stalks	Wheat straw	Depithed bagasse	Egyptian cotton stalks	Rice straw	Poplar
Holocelluloses	73.60	74.50	n/a	n/a	60.70	76.60
Alpha-cellulose	42.00	38.20 [(a)]	51.90	48.83	41.20	n/a
Lignin	17.30	15.30[(a)]	18.70	22.50	21.90	18.10
Ash content	8.20	4.70	2.60	1.84	9.20	0.54
Extractives ;						
Acetone	2.50	7.80 [(b)]	3.40[(c)]	2.93[(b)]	0.56[(b)]	2.50 [(b)]
Hot water	18.00	13.99	0.80	10.77	7.30	2.40
1 % NaOH	46.10	40.59	31.80	39.60	57.7	20.00
n/a: not available, a: Ash free, b: Alcohol benzene extractives, c: Cyclohexane-Ethanol extractives						

With a nearly similar holocellulose content of canola stalks and wheat straw, the holocellulose content of canola stalks is lower than that of poplar and higher than that of rice straw. The alpha-cellulose content of canola stalks is 42%, which is satisfactory for pulp production (close to or above 40%). However it is higher than that of wheat and rice straws and lower than that of bagasse and cotton stalks. The lignin content of canola stalks is fairly low and is comparable to that of poplar and depithed bagasse, but lower than that of rice straw and cotton stalks. The organic solvent extractives of canola stalks are similar to those of cotton stalks and poplar and lower than those of wheat straw and depithed bagasse. As indicated in Table 2, canola stalks contain considerably higher content of soluble compounds in hot water and dilute alkali. These decrease the pulping yield of canola stalks and contribute higher chemical consumption in pulping and higher load in pulping effluent. The ash content of canola stalks is also high.

Pulping Results

For pulping study the air-dried canola stalks were initially depithed using the presented procedure. The pulping conditions and some properties of obtained pulps are shown in Table 3.

Table 3. Soda Pulping of Canola Stalks (Cooking experiments were carried out using the 250 g dry chips, liquor to chips ratio = 8:1, time to max. temp. 90 min and max. cooking temp. 170 °C)

Cooking no.	NaOH charge (%)	Cook in time at max. temperature (Min)	Black liquor pH	Screen yield (%)	Reject yield (%)	Kappa number	Degree of polymerization
1	15	60	11.0	35.2	14.5	101.2	-
2	15	80	10.7	36.9	12.7	97.3	-
3	15	100	10.6	37.0	11.0	94.6	-
4	18	60	11.1	43.3	4.6	89.8	-
5	18	80	10.8	42.6	3.5	83.5	-
6	18	100	10.7	42.2	3.1	82	-
7	20	60	11.5	40.5	1.5	70.7	1435
8	20	80	12.1	39.1	1.4	54	1511
9	20	100	12.0	38.5	0.9	36.0	1553
10	22	60	12.2	37.3	0.6	40.0	1579
11	22	80	12.3	37.2	0.4	25.9	1448
12	22	100	12.2	36.6	0.3	24.2	1408

The pulping results indicated a predominant effect of alkali charge on pulp properties. The results showed that the soda pulping of canola stalks using 15% alkali charge yielded a high amount of uncooked material, even with 100 minutes cooking at maximum cooking temperature. Increasing the alkali charge to 18% led to more uniform cooking, and the uncooked portion decreased. Nevertheless, the residual lignin in the obtained pulps using 18% alkali charge was high, and increasing the cooking time at maximum temperature did not notably affect delignification degree. At 20% and 22% alkali charges, more uniform pulps were achieved, and increasing the cooking time from 60 minutes to 100 minutes led to a significant decreasing in kappa number. Despite the use of different pulps with kappa number ranging from 70.1 to 36, cooking of canola stalks chips using alkali charge of 20% did not yield bleachable grades pulps. Bleachable grade pulps only were produced using 22% alkali charge for 100 minutes cooking times.

The effects of pulping conditions on pulp viscosity indicated that the higher-intensity cooking conditions (higher alkali charge and longer cooking time) resulted in an increase in the pulp viscosity. This phenomenon could be attributed to the hemicelluloses dissolving during the high-intensity cooking conditions that led to higher average molecular weight of the obtained cellulose pulp. However, the results indicated that some cellulose depolymerization took place at 22% alkali charge with a cooking time of 100 minutes. Referring to the delignification degree and viscosity of obtained pulps, it can be deduced that the bleachable soda pulp of canola stalks could be obtained using 22% alkali charge with cooking time of 100 minutes.

Referring to the pulping results, it can be deduced that soda pulping of canola stalks yielded generally moderate-yield pulps. This is largely because of high soluble content of canola stalks in hot water and dilute alkali. Compared to soda pulp from bagasse (Khristova et al. 2006a), soda pulping of canola stalks produced lower yield pulp at a similar kappa number. However, the optimum yield for soda pulp from canola stalks was similar to that of soda pulp from Cynara cardunculus L. (Antunes et al. 2000) and better than soda pulp from rice straw (Rodriguez et al. 2008) and vine shoots (Jiménez et al. 2006).

Bleaching and Handsheet Paper Properties

With respect to pulp yield, polymerization degree of cellulose, and kappa number, the obtained pulp using 22% active alkali and 100 min cooking at maximum temperature was selected for bleaching and handsheet strength investigation. Table 4 summarizes the bleaching response of canola stalks soda pulp in DEpD sequence. The bleaching results indicated that the obtained pulp was bleached to an acceptable brightness in DEpD three stage bleaching sequence using a total chlorine dioxide charge of 28 kg/ton and a charge of hydrogen peroxide of 3 kg/ton of dry pulp under the conditions shown. Note that the chemical charges and bleaching conditions need to be optimized.

Table 4. D_0EpD_1 Bleaching of Canola Stalks Soda Pulp (initial kappa number and brightness was 23.8 and 36.5% ISO, respectively)

Bleaching stage	D_0	Ep	D_1
ClO_2 (% on dry pulp)	1.7		1.1
H_2O_2 (% on dry pulp)	-	0.3	-
NaOH (% on dry pulp)	-	1.2	-
Final pH	2.2	9.8	3.2
Kappa number	-	<2.0	-
Brightness (% ISO)		66.1	78.4

Table 5 shows the tensile index, burst index, tear index of handsheet paper obtained from bleached and unbleached canola stalks soda pulps. The strength properties of canola stalks soda pulps was almost similar to that of other non-woods and birch chemical pulps (Thykesson et al. 1998). The burst and tensile strengths of soda pulp from canola stalks was similar to that of unrefined rice straw soda pulp. The tear strength of unrefined soda pulp from canola stalks was higher than that of unrefined rice straw soda pulp, vine shoots kraft pulp, and eucalyptus kraft pulp.

Table 5. Mean Values of Strength Properties of Unrefined Unbleached and Bleached Canola Stalks Soda Pulp

	Freeness (ml CSF)	Tensile index (Nm/g)	Burst index (k Pa.m^2/g)	Tear index (m N.m^2/g)
Unbleached canola	480	24	1.22	5.07
Bleached canola	443	23.1	1.39	4.76
Rice straw soda pulp [(a)]	660	26.11	1.20	0.31
Vine shoots kraft pulp [(b)]	600	6.45	1.01	0.9
Eucalyptus kraft pulp [(c)]	650		1.4	1.9
a: (Rodriguez et al. 2008), b: (Jimenez et al. 2006), c: (Khristova et al. 2006b).				

CONCLUSIONS

1. The results of morphological study showed that canola stalks contained short fibers with similar morphological properties to the common non-woods fibers, except that the cell wall of canola fibers was much thicker.
2. Chemical composition analysis showed that the lignin content of canola stalks was comparable to other non-wood papermaking fiber resources. It was found that the canola stalks contained high amounts of hot water and dilute alkali extractives.
3. The soda pulping results showed that unlike to the other non-woods, canola stalks required high chemical charge with high H factor and produced low-yield bleachable grade pulp. The soda pulp of canola bleached easily in a three stage ECF sequence.
4. The strength properties of unrefined bleached canola stalks soda pulp were determined to be comparable with those of hardwood and typical non-woods papermaking raw materials.
5. The overall results showed that canola has a promising potential to be used in combination with softwood or hardwood pulps in papermaking.

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