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Investigation on Pulping Potential of Iranian Rapeseed Residue in the Paper Industrial

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Abstract: This study examined the impact of pulping conditions on properties of NSSC pulp from rapeseed residue. Rapeseed residue was collected from the research field in the Babul city that located in north of Iran. The celluloses, lignin, Extractives soluble in alcohol- acetone and ash were measured as 44±0.87%, 19.21±0.58%, 6±0.53% and 13±0.19%, respectively. Fiber length, diameter, lumen and cell wall thickness of rapeseed residue fibers were examined as 0.96±0.18 mm, 24.21±6.07 µ, 15.47±5.23 µ and 4.37±1.97 µ. Three pulping durations of 20, 30 and 40 min and 3 chemical charges of 10%, 15% and 20% chemical charge was used for NSSC paper industry. Cooking temperature at 170°C, Na_2SO_3 to Na_2CO_3 ratio of 2.91:1 and liquor to residue ratio of 10:1 were kept constant. The highest yield (82%) was obtained applying the treatment combination of 40 min pulping time and 10% chemical charge and the lowest yield (63.67%) was observed with 30 min pulping time and 15% chemical. After the Pulping, The pulp prepared to be refined up to 400±50 ml CSF and 127 gm^{-2} hand sheets were prepared. The results of pulping and hand sheet evaluation showed that 20% chemical charge, 30 min pulping at 170 °C can be considered as the optimum pulping condition for rapeseed residues. Corrugating medium test, ring crush test, tensile, tear and burst indices of this pulp were measured as 232 KN m^{-1}, 1.96 KN m^{-1}, 35.56 N.mg^{-1}, 6.19 mN m^2g^{-1} and 2.29 kPa m^2g^{-1}, respectively. According to the results of this study, it was found the rapeseed residue is suitable for pulping.

Key words: Corrugating Medium test · NSSC · Rapeseed · Ring crush test · Strength · Yield

INTRODUCTION

Paper consumption is continuously increasing across the world. It amounted to 52.45 Kg per person in 2004 and is expected to rise above 400 million tons by the year 2010 [1]. However, during the late 20^{th} century, global shortage of wood emerged and so, paper industry was forced to look for new alternatives for fibrous raw material. Furthermore, the fiber supply in regions like Asia and Middle East is so tense that these locations concentrated on utilization of nonwoods as raw material. Iran is concentrating on rapeseed plantation to produce vegetable oil for its growing population. Paper industry worldwide is facing shortage of suitable fibers and this limitation is not only the issue for fiber deficient countries but also North America as well as Europe has started considering nonwood fiber utilization [2, 3]. Therefore, nonwood pulping picked up the momentum during the last 40 years and the share of nonwood pulp increased from 7% to 12% [4]. To the best of our knowledge, no activity is available on rapeseed pulping worldwide and only research groups in Iran showed interest in this subject. Soda pulping of rapeseed residue was tested by Sefidgaran (2003) in an attempt to use the produced pulp in corrugating paper production and Molaii (2007) Soda pulping on this raw material [5, 6]. Rapeseed Straw (*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

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for Rapeseed Straw increases continuously. In 2003, the worldwide planted area of Rapeseed Straw was more than 23 million hectares. China, Canada, India, France, Germany and Australia are the major producers of Rapeseed Straw. The total amounts of biomass produced per unit area by Rapeseed Straw depends on irrigation and varies from 5 to 10 t ha^{-1}. About 20 percent of Rapeseed Straw biomass includes the stem portion that remains in the field after Rapeseed Straw seed collection [7]. Thus, it can be concluded that at least 34 million tons of Rapeseed Straw stalks are available annually, which could be used in various products, including pulp and paper productions. The properties of rapeseed residue fibers for pulp and paper production have never been explored in the literature.

In this study we aimed at Investigating the biometry, chemical and Neutral sulfite semi-chemical pulping properties of Rapeseed residue in the pulp and paper production.

MATERIALS AND METHODS

Raw Material: Rapeseed residue was collected from the research field in Babul city. Samples were cleaned and leaves and debris were separated and then the stems were depithed carefully by hand. Depithed material was cut into 2-4 cm length chips. Chips were dried at ambient temperature and after reaching equilibrium moisture content, chips were stored in plastic bags until used.

Fiber Biometry Characteristics: The pieces of Rapeseed residue were defibrated using the technique developed by Franklin (1954) [8] and then the fiber length, fiber diameter and lumen width were measured with a microscope equipped with a Leica Image Analysis (Quantimeta 100+). The fiber wall thickness was calculated as a difference of fiber diameter and lumen width divided in half. For dimensions of 120 fibers were randomly measured. From these data, the average fiber dimensions were calculated and then the following derived indexes were determined:

Runkel ratio = 2 × (Wall thickness/Lumen width)
Flexibility ratio = (Lumen width of fiber/Diameter of fiber) × 100
Slenderness ratio = (Length fiber/Diameter of fiber)

Chemical Composition: The lignin, ash and ethanol/acetone extractable of Rapeseed residue were determined according to TAPPI Test Methods. The cellulose Content of Rapeseed residue was determined according to the nitric acid [9] method, Ash; T211-om 93: Extractives soluble in alcoholacetone; T264-om 97: Lignin; T222-om 98: respectively. All measurements were repeated three times.

Pulping: After transfer Rapeseed residue to laboratory Research Center of Mazandaran Wood and Paper factory, Pulping neutral sulfite semi-chemical (NSSC) using experimental rotating digester (HATTO) was performed. Using 500 grams of depithed residue in each trial and pulping time was measured after reaching pulping temperature. For cooking of Rapeseed residue from white liqueurs of Mazandaran Wood and Paper Factory include sodium sulfite (Na_2SO_3) and sodium carbonate (Na_2CO_3), with a weight ratio of 2.91 to 1 (sulfite to carbonate) were used. Rapeseed residue cooking conditions are shown in Table 1. For each combination of variables, three replica pulps were prepared. At the end of each cook, the content of each cylinder was discharged into 20 liter container and mixed with a sufficient volume of boiling water and then defibered using laboratory single disk refiner. Pulp suspension was screened using 12 mesh screen on top and 200 mesh screen at the bottom. Material remained on the 12 mesh screen was considered as reject (shives) and the fibers passed the 12 mesh screen and remained on the 200 mesh screen was considered acceptable. The dry weight of each part is measured and reported. For making handsheets paper, the freeness was required that it was estimated almost 400 ± 50 CSF in the present study. For this reason, to achieve the desired freeness PFI Mill was used. From NSSC pulp products, according to the T 205

Table 1: Optimum conditions for producing neutral sulfite semi-chemical pulping from rapeseed residues

Cooking condition	Neutral sulfite semi-chemical pulping	Cooking condition	Neutral sulfite semi-chemical pulping
Chemical charge (%)	10, 15 and 20	Liquor-to-chips ratio	10:1
Pressure (bar)	9.3	Time Impregnated (min)	30
pH Before Pulping	9.95	Na_2O (gr/l)	126
pH After pulping	9.01	SO_2 (gr/l)	85
Yields (%)	61 to 82	(°C) cooking temperature	170
Chemical charge	sodium sulfite (Na_2SO_3)	Cooking time at maximum temperature (min)	30, 40 and 50

om-88 standards, the number of eight hand sheet paper (with basic weight 127 gr m^{-2}) was maked. The sheet's paper was conditioned at 23°C and 50% RH for 24 hours. Then, the corrugating medium test (CMT), ring crush test (RCT), tensile index, tear index and burst index of paper sheets were determined according to TAPPI T809 om-99, T 818 om-87, T240 Om-92, SCAN P11:73 and T 403 om-91 Test Method, respectively. The reported results represent the average values of five handsheets. Finally, in order to compare the results of paper strength test, two-way analysis of variance test and Duncan's multiple range using SPSS software was performed.

RESULTS

Chemical Composition and Fiber Dimensions: The percentage of cellulose, lignin, extractive soluble in alcohol-acetone and ash is summarized in Table 2 and the fiber dimensions as well as calculated runkel, felting and flexibility coefficients are summarized in Table 3. The cellulose content of rapeseed residues is 44%, which is satisfactory for pulp production (close to or above 40%). Each value in Table 2 is the average of three measurements and each value in Table 3 is the average of 120 measurements. Standard deviation of the measurements is also given in both tables to show the variations of the given property.

Pulp and Paper Properties: Table 4 shows the results of the descriptive statistics of the pulp and paper strength properties and the Duncan’s mean separation test for Pulp and paper strength is shown in Table 5.

Yield: The analysis of variance shows the effects of cooking time on the yield of pulp and paper were significant at the 95 percent confidence level (F = 271.75, Sig = 0.001). The Duncan's tables show that significant differences exist among the levels of cooking time index (Table 5). In addition, the value of yield in time 20 min is more than the other cooking times. The analysis of variance shows the effects of chemical charge on the yield of pulp and paper were significant at the 95 percent confidence level (F = 173.179, Sig = 0.001). The Duncan’s mean separation test reveals a significant difference in

Table 2: Chemical composition of the rapeseed residues

Component	Value %	Coefficient of Variation %
Cellulose	44±0.87	1.97
Lignin	19.21±0.58	1.84
Extractives soluble in alcohol acetone	6±0.53	8.67
Ash	13±0.19	1.49

Table 3: Fiber dimensions and morphological coefficients of rapeseed residues

Fiber dimension	Value %	Coefficient of Variation %
Length	0.96±0.18	19.31
Width	24.21±6.07	11.5
Diameter of cellular cavity	15.47±5.23	12
Cell wall thickness	4.37±1.97	14.5
Slenderness ratio	39.47	
Flexibility ratio	63.93	
Runkel ratio	0.56	

Table 4: Different yields of rapeseed NSSC pulps (Constant pulping temperature: 170°C)

Pulping variable							
Chemical charge (%)	Pulping time (min)	Total yield (%)	Corrugating Medium Test (Knm^{-1})	Ring crush Test (Knm^{-1})	Tensile index ($N\ mg^{-1}$)	Tear index ($mN.m^2g^{-1}$)	Burst index ($kPa\ m^2g^{-1}$)
10	20	73.33 ± 1.53	167 ± 2.65	1.63 ± 0.02	25.64±0.17	6.87 ± 0.1	1.88 ± 0.28
15		78.67 ± 0.58	190± 1.00	1.74 ± 0.02	25.2±1.15	7.1 ± 0.13	1.74 ± 0.23
20		75 ± 1	191 ± 2.65	1.67 ± 0.02	28.44±0.51	6.56 ± 0.3	2.07 ± 0.15
10	30	69 ± 0	181± 1.73	1.49 ± 0.04	27.98±0.38	6.02 ± 0.16	2.26 ± 0.14
15		63.67 ± 0.58	170 ± 2.0	1.98 ± 0.03	32.16±1.19	5.59 ± 0.26	1.94 ± 0.07
20		66 ± 0	232 ± 1.73	1.96 ± 0.04	35.56±2.32	6.19 ± 0.16	2.29 ± 0.27
10	40	79.67 ± 1.15	165 ± 2.65	1.39 ± 0.02	26.21±0.82	7.31 ± 0.05	1.54 ± 0.09
15		82 ± 1	189 ± 3.0	1.73 ± 0.03	26.91±1.16	6.4 ± 0.14	1.73 ± 0.18
20		61 ± 1.73	233 ± 2.0	1.91±0.02	29.65±1.13	6.66±0.11	1.97 ± 0.16

Table 5: Duncan multiple range test for independent effect of Pulping time and Chemical charge on resistance and strength characteristics of hand sheet from NSSC process of rapeseed

Variable		Total yield (%)	Corrugating Medium Test (Knm^{-1})	Ring crush Test (Knm^{-1})	Tensile index (N mg^{-1})	Tear index (mN.m^{2}g^{-1})	Burst index (kPa m^{2}g^{-1})
Pulping time (min)	20	77a	182.67a	1.68a	26.43a	6.84b	1.9a
	30	66.22b	194.33b	1.81b	31.9c	5.93a	2.16b
	40	74.22c	195.67b	1.68a	27.59b	6.79b	1.75a
Chemical charge (%)	10	75.33a	171a	1.5a	26.61a	6.73b	1.89a
	15	74.78a	183b	1.82b	28.09b	6.36a	1.8a
	20	66.33b	218.67c	1.84c	31.22c	6.47a	2.11b

the yield between chemical charge indexes 10 and 20% and between 15 and 20% (Table 5). With increasing chemical charge, the amount of yield is decreased. In addition, the effects of interaction cooking time × chemical charge on yield was significant (F = 120.357, Sig = 0.001).

Corrugating Medium Test (CMT): The analysis of variance shows the effects of cooking time on the CMT were significant at the 95 percent confidence level (F = 92.067, Sig = 0.001). The Duncan's mean separation test reveals a significant difference in the CMT between cooking time indexes 20 and 30 min and 20 and 40 min (Table 5). The value of CMT in time 40 min is more than the other cooking times. The analysis of variance shows the effects of chemical charge on the CMT of pulp and paper were significant at the 95 percent confidence level (F = 1106.467, Sig = 0.001). The Duncan's tables show that significant differences exist among the levels of chemical charge index (Table 5). With increasing chemical charge, the mean of CMT is increased. In addition, the effects of interaction cooking time × chemical charge on CMT was significant (F = 187.167, Sig = 0.001).

Ring Crush Test (RCT): The analysis of variance shows the effects of cooking time on the RCT of pulp and paper were significant at the 95 percent confidence level (F = 83.625, sig = 0.001). The Duncan's mean separation test reveals a significant difference in the CMT between cooking time indexes 20 and 30 min and 30 and 40 min (Table 5). The value of RCT in time 30 min is more than the other cooking times. The analysis of variance shows the effects of chemical charge on the RCT of pulp and paper were significant at the 95 percent confidence level (F = 523.018, Sig = 0.001). The Duncan's tables show that significant differences exist among the levels of chemical charge index (Table 5). With increasing chemical charge, the mean of RCT is increased. In addition, the effects of interaction cooking time × chemical charge on CMT was significant (F = 99.455, Sig = 0.001).

Tensile Index: The analysis of variance shows the effects of cooking time on the tensile strength of pulp and paper were significant at the 95 percent confidence level (F = 56.706, Sig = 0.001). The Duncan's tables show that significant differences exist among the levels of cooking time index (Table 5). In addition, the value of tensile strength in time 30 min is more than other cooking times.

The analysis of variance shows the effects of chemical charge on the tensile strength pulp and paper were significant at the 95 percent confidence level (F = 37.724, Sig = 0.001). The Duncan's tables show that significant differences exist among the levels of chemical charge index (Table 5). With increasing chemical charge, the amount of RCT is increased. In addition, the effects of interaction cooking time × chemical charge on paper tensile strength was significant (F = 4.761, Sig = 0.001).

Tear Index: The analysis of variance shows the effects of cooking time on the tear strength were significant at the 95 percent confidence level (F = 79.12, Sig = 0.001). The Duncan's mean separation test reveals a significant difference in the tear strength between cooking time indexes 20 and 30 min and 30 and 40 min (Table 5). The value of tear strength in time 20 min is more than the other cooking times. The analysis of variance shows the effects of chemical charge on the tear strength of pulp and paper were significant at the 95 percent confidence level (F = 11, Sig = 0.001). The Duncan's mean separation test reveals a significant difference in the tear strength between chemical charge indexes 10 and 15% and between 10 and 20% (Table 5). With increasing chemical charge, the mean of yield is decreased. In addition, the effects of interaction cooking time × chemical charge on the paper strength properties was significant (F = 14.155, Sig = 0.001).

Burst Index: The analysis of variance shows the effects of cooking time on the burst strength were significant at the 95 percent confidence level (F = 11.337, Sig = 0.001). The Duncan's mean separation test reveals a significant difference in the burst strength between cooking time indexes 20 and 30 min and 30 and 40 min (Table 5). The value of tear strength in time 30 min is more than the other cooking times. The analysis of variance shows the effects of chemical charge on the burst strength of pulp and paper were significant at the 95 percent confidence level (F = 6.325, Sig = 0.005). The Duncan's mean separation test reveals a significant difference in the burst strength between chemical charge indexes 10 and 20% and between 15 and 20%. With increasing chemical charge, the mean of yield is increased. In addition, the effects of interaction cooking time × chemical charge on the paper strength properties wasn't significant (F = 1.568, Sig = 0.226).

DISCUSSION

The results show that cellulose content of rapeseed residues shorter than that bagasse (52.42%) [10] and is higher than that of rice straw (41.20%) [11] and wheat straw (38.20%) [12]. the lignin content of rapeseed residues is lower than that of rice straw (21.90%) and Egyptian cotton stalks (22.50%) [13]. The average of fiber length of rapeseed residue is o.96 mm which is longer than cotton stalks (0.83 mm) [5] and is shorter than wheat straw (1.73 mm) [2]. In addition, cell wall thickness of rapeseed residues fibers is thicker than aspen (1.93 μm) [14] and cotton stalks (3.40 μm) [5]. So, the calculated Runkel ratio for rapeseed residues fibers (0.56) is shorter than that of cotton stalks (0.84), date palm rachis (0.8) fibers. The slenderness ratio of rapeseed residues fibers is 39.47 and is shorter than that of cotton stalks (42.35) and aspen (46.15) fibers. Generally, the acceptable value for Slenderness ratio of papermaking is more than 33, respectively [15]. The flexibility coefficient of rapeseed residues fibers are less than both cotton stalks (65.31) and aspen (81.44). This indicates good sheet formation potential of these fibers. The performance of rapeseed residues in different pulping, such as soda and NSSC pulping varies considerably and usually a low yield is observed. Even though it was possible to reach digester yield between the lowest value of 61% and the highest value of 79.67%, but reaching a high yield is difficult with this material. The yield of soda-AQ pulping of rapeseed residue was measured as 36.8% [3] and for soda as 36.1% [6]. The results exhibited that higher chemical charge as well as pulping time reduce digester yield. It should be noted that defibration usually reduces the pulping yield. This is the consequence of fine generation and the presence of pith particles, which are very short and small cubical. However, without defibration, such as fine generation is limited. The results show that at higher chemical charge or pulping duration, Paper strength improved and the influence of chemical charge is more effective than pulping duration. Changing the chemical charge from 10% to 20% increases the Corrugating Medium Test from 171 to 218.67 KN m^{-1}, Ring crush Test from 1.5 to 1.84 KN m^{-1}, tensile index from 26.61 to 31.22 N.mg^{-1} and burst index from 1.89 to 2.11 kPa m^2g^{-1} and decrease tear index from 6.73 to 6.47 mN m^2g^{-1}. The tensile and burst indices of rapeseed residue NSSC pulp is lower than wheat straw Kraft pulps (89.15 ± 3 Nmg-1 and 3.32 ± kPam^2g^{-1}), its tear index is almost identical [4]. Since the strength of either soda or soda/AQ pulps from rapeseed residue are lower than NSSC, it can be concluded that rapeseed residues is suitable for unbleached NSSC pulp production.

CONCLUSION

- Chemical composition analysis showed the lignin content of rapeseed residues was comparable to other nonwood papermaking fiber resources. These positive factors in the selection of rapeseed residues as raw material in paper industry are considered.
- The results of biometrical study showed that rapeseed residues contained average fibers with similar biometrical properties to the hardwood fibers, except the cell wall of rapeseed residues was thicker.
- Then NSSC pulping trials were performed applying different chemical charges and pulping times. The results showed that rapeseed residues are suitable for NSSC pulping and application of 20% Chemical charge, 30 min pulping time at 170°C pulping temperature will produce pulp suitable as supplement pulp for unbleached paper production. Corrugating Medium Test, Ring crush Test, Tensile index, tear index and burst index of this pulp (66% total yield) were measured as 232 KNm^{-1}, 1.96 KNm^{-1}, 35.56 N.mg^{-1}, 6.19 mN m^2g^{-1} and 2.29 kPa m^2g^{-1}.
- The overall results showed that rapeseed residues have a promising potential to be used in combination with softwood or hardwood pulps in papermaking.

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