ECO-FRIENDLY PULPING OF RICE STEM BY TWIN-SCREW EXTRUDER: OPTIMIZATION OF COOKING TIME AND NaOH CONCENTRATION

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In this study, rice stem pulping was performed using a co-rotating intermeshing twin-screw extruder as an eco-friendly method. The effects of NaOH concentration and cooking time during alkali pre-treatment on the chemical compositions and contents of the resulting pulp, and handsheet physical characteristics were investigated. Using a two-level factorial design and the Minitab software suite, equations relating each dependent variable to the independent variables were obtained. These equations predicted the experimental results for the dependent variables with errors of less than 30% for cooking times and NaOH concentrations in the ranges of 15-75 min and 1-2%, respectively. The results showed that the NaOH concentration had a greater effect on pulp and handsheet properties compared to cooking time. The rice stem could be pulped at a low NaOH concentration (1%) and low cooking time (15 minutes) using the twin-screw extruder at atmospheric boiling point, generating a pulp with a Kappa number of approximately57, which is suitable for fluting paper, linerboard and nanocellulose production. A pulp with a lower Kappa number was also produced under intensified process conditions (75 min cooking time and 2% NaOH concentration).

Keywords: twin-screw extruder, rice stem, papermaking, process optimization, nanocellulose

INTRODUCTION

Current pulp output is inadequate to meet the increasing demand in most countries. This has contributed to shortage of wood, the gradual deforestation of some areas in the world, andenvironmental pollution. Non-wood materials such as wheat straw, rice straw, and various other agricultural residues, which are abundant in Iran, are attractive alternatives for pulp production. Nowadays, non-wood materials are widely used in pulp and paper industries in several countries due to their advantages over wood.[1-4] In fact, from 2008 to 2012, the use of non-wood fiber for pulp and paper production increased by 79%, whereas that of wood fiber decreasedby 3%.[5] The greaterincrease in the use of non-wood pulp is a result of the need to cut costs and avoid environmental problems using alternative raw materials.[6]

Generally, non-wood materials used for production of unbleached and bleached pulps require moderate cooking conditions compared to wood materials.[7,8] However, one of the main problems with non-woodsis the relatively low quality of the resulting paper. However, straw is a good source of raw material in areas where wood supplies are scarce.

Currently, straw is the largest non-wood source for the paper industry,[4] and rice straw seems to be one of the most suitable cereal straws for papermaking.[9,10] However, there are economic and environmental disadvantages to pulping rice straw usinga traditional pulping system, such aslow pulp quality, low effluent recovery, and low industrial capacity. Therefore, recent researches have attempted to find alternative methods for pulping rice straw, such as organosolv pulping[11-16] and extrusion pulping.[8,17-19]

Extrusion pulping occurs in twin-screw extruders, atechnology that is extensively used in the polymer industry andallows for good mixing,

fast throughput, and operation at high pressures.[17]Itis a mechanical or chemi-mechanical pulping method in which fibers are processed by means of compression and shear force using different elements,[20] which can reduce the fiber size and also separate fibers.[18] The reverse screw elements move the material in opposite directions and effectively resist material flow.[21] The basic principles of this pulping method have been recently upgraded from the original BiVis process, with successful results on pilot and industrial applications involving annual agricultural plants, such as bamboo, hemp, wheat straw, rice straw, and bagasse.[17] Furthermore, it is predicted that extrusion pulping of non-wood raw materials can be used not only as an eco-friendly method for papermaking, but also as an effectivepre-treatment method for production of nanocellulose. In fact, pulping of non-wood by a co-rotating intermeshing twin-screw extruder results in the pulp with small particle sizes, which can facilitate subsequent mechanical and chemical treatments, contributing to economic production of nanocellulose from non-wood raw materials.

In this project, rice straw was subjected to alkaline solution impregnation, which has previously been used for pre-treatment of non-wood materials,[17,20] before entering the extrusion process. Various conditions for the extrusion pulping of rice stem were investigated with reference to our previous research[8] for process optimization.

EXPERIMENT

Material

Air-seasoned rice stem was obtained from the northern region of Iran. Itwas cleaned and cut into 3cm long pieces before pulping. The chemical composition of the rice stem was determined as follows: 46.51% cellulose, 16.03% lignin, 14.61% ash, and 3.10% ethanol/acetone extractable, based on oven-dry weight.

Methods

Alkali pre-treatment of rice stem

Samples of rice stem weighing 400 g were treated with 1%, 1.5% and 2% sodium hydroxide at atmospheric boiling point with a liquid to rice stem ratio of 8:1 for varying amounts of time (15, 30, 45, 60 and 75 min) in a mini-digester. Followingthe treatment, the liquid was allowed to drain for 10 minutes through a perforated screen with a mesh size of 18.

Pulping and handsheetmaking

The drained pre-treatedrice stem used for pulping had a typical dry matter content of approximately 20%. Pulping was done using a co-rotating intermeshing twin-screw extruder with a length of 1400 mm and an external diameter of 110 mm. Pulp was extruded at constant temperature and speed (60 ^{0}C, 70 rpm) with an extrusion time of about 15 minutes for each run. Exiting pulp was washed with warm water and disintegrated in a standard disintegrator (TAPPI T 205 sp-95). As the freeness of the extruded pulp was initially high, all pulp samples were beaten in a stainless steel PFI mill under standard conditions (TAPPI T 248 sp-00) until the freeness reached 300 ml CSF. Handsheets of 60 g/m^{2}were then prepared from the pulp, and their physical and mechanical properties were determined using standard procedures.

Analysis of raw materials, pulp and paper sheets

Handsheets were conditioned at 23 ℃ and 50% RH for at least 24 hoursbefore testing. The starting material, pulp, and handsheets were all characterized according to the following standard methods: Klason lignin (TAPPI T 222 om-98), cellulose, 22 ethanol/acetone extractable (TAPPI T 204 cm-97), and ash (TAPPI T413 om-93). Pulps were also used for measurements of freeness (TAPPI T 227 om-99), Kappa number (TAPPI T 236 om-99) and classification of fiber length (TAPPI T 233 co-06).

Modeling

A central composite factor design using a series of points (experiments) was used around a central point (central experiment), and several additional points (additional experiments) were usedto estimate the first and second-order interaction terms of a polynomial. This design meets the general requirement forevery parameter in the mathematical model tobe estimated from a fairly small number of experiments. The total number of observations, N, required for the two process variables (concentration of NaOH solution, C, and cooking time, t) was calculated by the multi-level factorial design including 2 variables in 15 runs. The experimental data were fitted to the following second-order polynomial:

$$Z = a + bX_C + cX_t + dX_C^2 + eX_t^2 + fX_C X_t \qquad (1)$$

where Z denotes the response variables of the pulp (classification of fiber length as shown by R_{14}, $P_{14}R_{100}$, P_{100}; Kappa number = KN, initial freeness = CSF, cellulose, ash) and of the handsheets (burst factor=BF, breaking length=BL, tear factor=TF, folding endurance = FE, brightness = BR). The parameters X_t and X_C are the normalized values of t and c, and the coefficients *a*, *b*, *c*, *d*, *e*, and *f* are constants.The values of the independent variables as process variables were normalized to -2, -1, 0,+1 ,+2 using Equation 2 in order to facilitate direct comparison of coefficients and showthe individual effects of the independent variables on each dependent variable:

$$X_n = 2\frac{X - X_{mean}}{X_{max} - X_{min}} \quad (2)$$

whereX_n is the normalized value of*t* or *c*, *X* is the absolute experimental value of the variable concerned, X_{mean} is the mean of all the experimental values for the variable in question, and X_{max} and X_{min} are the maximum and minimum valuesof such a variable, respectively. This normalization results in more accurate estimates of the regression coefficients as it reduces the interrelationships between linear and quadratic terms.[8,14] The 15 experiments conducted, together with the corresponding normalized values for the independent variables, are given in Table 1. The values obtained were utilized for determining the mathematical estimation models for each response, which were subsequently used to characterize the nature of the response surface. All statistical analyses were carried out using MINITAB statistical software. Data processing enabled estimation of the liner and quadratic (main) effects, and the interactions of the process variables for the responses were considered. Based on experimental conditions, the effect of a factor is the change in response when the factor is changed from a low (-1) to a high (+1) value. The effect of each variable estimates its average effect over all possible conditions of the other variables. Each of the analyzed responses can be affected by the main and quadratic effects of processing variables and interactions among them. The main effects of a variable should be individually interpreted only if there is no evidence that it interacts with other variables. When there is evidence of one or more such interactions, the interacting variables should be considered together.[14] Also, quadratic significant effects of independent variables imply the existence of effective maximum or minimum levels for dependent variables that aremarked according to the quadratic coefficients of the independent variables (i.e. a positive sign implies the minimum level and a negative sign implies the maximum level).

Table 1
Values of the process variables during the pulping process in the extruder using the proposedexperimental pulp system*

Run No.	Codes		Values	
	X_t	X_C	t(min)	C (%)
1	-2	-1	15	1
2	-1	-1	30	1
3	0	-1	45	1
4	1	-1	60	1
5	2	-1	75	1
6	-2	0	15	1.5
7	-1	0	30	1.5
8	0	0	45	1.5
9	1	0	60	1.5
10	2	0	75	1.5
11	-2	1	15	2
12	-1	1	30	2
13	0	1	45	2
14	1	1	60	2
15	2	1	75	2

*X_t: normalized cooking time; X_C: normalized NaOH concentration; *t*: cooking time; *C*: NaOH concentration

RESULTS AND DISCUSSION

Results

The characteristics of the pulp and handsheets obtained from the 15 pulping runs (15 different runs each with three replicates) are summarized in Tables 2 and 3. The dependent variables, as shown in Tables 4 and 5, differed from their mean values by less than 30%. The MINITAB software suite was used to conduct a multiple polynomial regression analysis involving all the terms of Equation1 except those with alpha (0.15, 0.15), which were left out using the stepwise method.[23] Equations 3 to 14 below are reduced models for each response.

$$R_{14} = 1.391 - 0.81X_c - 0.482X_t + 0.154X_t^2 \quad (3)$$

$$P_{14}R_{100} = 59.60 - 3.37X_c - 1.92X_t - 8.60X_c^2 - 0.96X_t^2 \quad (4)$$

$$P_{100} = 47.44 + 3.40X_c + 6.85X_t - 3.40X_cX_t \quad (5)$$
$$Kappa\ Number = 25.41 - 12.6X_c - 4.47X_t + 5.60X_c^2 + 0.92X_t^2 \quad (6)$$
$$Cellulose = 75.20 + 1.65X_t^2 + 2.84X_cX_t \quad (7)$$
$$Ash = 5.17 - 1.79X_c + 1.80X_c^2 + 0.81X_cX_t \quad (8)$$
$$CSF = 414 - 29X_c - 24.2X_t - 10X_c^2 + 2X_t^2 \quad (9)$$
$$Burst\ Factor = 41.52 + 11.40X_c \quad (10)$$
$$Breaking\ Length = 4.88 + 1.42X_c \quad (11)$$
$$Fold\ Endurance = 67.40 + 71.20X_c + 31X_c^2 \quad (12)$$
$$Tear\ Factor = 84.65 - 1.73X_c - 7.10X_c^2 + 0.76X_t^2 \quad (13)$$
$$Brightness = 40.39 + 3.92X_c + 2.53X_t - 2.40X_c^2 - 0.73X_t^2 \quad (14)$$

Some of the parameters (R-sq, R-sq (adj), and S values) of Equations 3-14 are summarized in Table 4. These results confirm the adequacy of the fitted models, where all models have R-sq greater than 80% except for breaking length and tear factor. Also, the coefficient estimates for each term in the selected models are shown for every dependent variable.

The effects of the independent variables on the dependent variables were also investigated under optimum conditions by comparing the experimental results with the equations (Table 5). However, the predictions given by the equations were not always verified by the measurements. For example, the Kappa number of pulp obtained by extrusion had 7.1% error with respect to optimum values. However, process optimizations of independent variables have predicted dependent variables with errors ofless than 10%, such as burst factor with 5.05%.

Table 2
Chemical properties of the pulp obtained from the extruder pulping process, using the proposed experimental pulp system*

Run No.	R_{14} (%)	P_{14}-P_{10} (%)	P_{100} (%)	KN	CSF (ml)	Cellulose (%)	Ash (%)
1	6.1	56.1	37.8	57.0	495	76.43	7.15
2	2.7	53.8	43.5	49.3	450	73.08	7.65
3	2.6	53.4	44.0	42.5	430	75.46	9.3
4	2.3	52.7	45.0	44.7	410	88.84	9.8
5	1.4	46.4	52.2	34.2	400	88.73	9.85
6	3.2	37.5	32.1	41.5	460	78.10	4.95
7	2.1	58.5	39.4	27.0	440	78.05	4.55
8	1.0	62.2	46.0	24.0	425	74.15	5.3
9	0.9	41.0	58.1	24.0	395	77.85	5.4
10	0.8	51.4	57.0	19.8	370	82.46	5.63
11	2.3	72.7	25.0	30.4	440	76.90	3.4
12	0.5	52.7	46.8	20.5	400	75.53	4.1
13	0.7	46.8	52.5	20.4	375	73.92	5.7
14	0.6	46.2	53.2	15.7	350	73.90	5.9
15	0.6	39.4	60.0	14.0	330	69.11	5.0

*R_{14}: fibers on a 14 mesh screen; P_{14}-P_{100}: retained fibers between 14 and 100 mesh screen; P_{100}: fibers pass through a 100 mesh screen; KN: Kappa number; CSF: Canadian freeness of initial pulp (after extrusion); Cellulose: cellulose percentage of each sample; Ash: ash percentage of each sample

Table 3
Properties of the handsheets obtained from the extruder pulping process, using the proposed experimental pulp system *

Run No.	BF	BL (km)	TF	FE	BR (ISO)
1	25.8	3.390	82.0	18	26.5
2	31.2	3.432	83.4	33	31.4
3	24.5	2.980	78.0	21	35.2
4	31.9	3.550	80.6	32	34.3
5	26.0	3.535	80.0	32	35.8
6	26.5	3.600	86.6	41	32.8
7	39.5	5.111	86.4	64	38.4
8	45.8	5.343	83.0	93	39.3
9	47.9	5.620	85.3	76	36.2
10	54.5	5.608	89.6	63	42.0
11	56.9	6.820	74.2	168	31.4
12	41.0	5.295	76.0	66	41.0
13	52.3	6.464	73.0	147	40.4
14	56.2	7.183	80.0	194	43.2
15	42.8	5.282	83.5	61	46.4

BF: burst factor; BL: breaking length; TF: rear factor; FE: folding endurance; BR: brightness of handsheet from unbleached pulp

Table 4
Coefficients of the equations that relate dependent and independent variables, *R-sq*, R-sq (adj), R-sq (pred) and s[a]

CV		Equations											
		R_{14}[3]	P_{14}-P_{100}[4]	P100 [5]	KN [6]	Cell[7]	Ash[8]	CSF[9]	BF[10]	BL[11]	FE[12]	TF[13]	BR[14]
A	1	+ 1.39	+59.60	+47.44	+25.41	+75.20	+5.17	414	+41.10	+4.88	+67.40	+ 84.65	+40.39
B	X_c	-0.81	-3.37	+3.40	-12.67	0	-1.79	-29	+12.40	+1.42	+71.20	-1.73	+3.92
C	X_t	-0.43	-1.92	6.85	-4.47	0	0	-24.2	+6.40	0	0	0	+2.53
D	X^2_c	0	-8.60	0	+5.60	0	+1.80	-10	0	0	+31.10	-7.10	-2.40
E	X^2_t	+0.15	-0.96	0	+0.92	+1.65	0	2	0	0	0	+0.76	-0.73
F	X_cX_t	0	0	-3.40	0	+2.84	+0.81	0	-7.70	0	0	0	0
S		0.41	1.92	2.03	3.01	1.66	0.42	7.33	4.50	0.70	16.60	2.87	1.75
R-Sq%		85.55	94.69	92.67	96.29	87.87	96.85	98.01	89.77	75.92	93.31	71.3	92.90
R-Sq(adj) %		81.21	91.15	90.47	94.80	85.18	95.90	97.21	86.71	74.07	91.97	63.25	89.74
R-sq(pred) %		70.95	67.43	82.93	91.59	74.70	93.85	94.89	79.33	68.54	87.70	44.65	82.07

CV: Coefficients of equations and values of regression analysis; KN: Kappa number; Cell:Cellulose; BF: burst factor; BL: breaking length; FE: fold endurance; TF: tear factor; B: brightness; S (sum of error =$MES^{1/2}$), R-Sq, R-Sq (adj) and R-sq (pred) used to compare the full model to a model with a subset of predictors that were estimated for each model by regression analysis

Table 5
Optimum (maximum) values of dependent variables and effects of dependent variables on independent variables for the extruder pulping process and handsheet making using resulting pulp

Dependent variable	Error (%) of the experimentalvalues with respect to optimum values	Optimum values	Normalized values of theindependent variables leadingto optimum values of thedependent variables		Maximum changes in dependent variables (in units andpercentages with respect to optimum values, shown in brackets) with changes in independentvariables (from -2 to +2 for cooking time and -1 to +1 for NaOH concentration)	
			Xt	Xc	t	C
R14 (%)	158	0.23 (min)	+2	+1	1.9 (827)	1.6 (695)
P_{14}-P_{100} (%)	28	77 (max)	-2	-1	11.5 (15)	23.9 (31)
P_{100} (%)	5.8	37.8 (min)	-2	-1	41 (114)	20 (57)
KN	1.24	56.3 (max)	-2	-1	17.9 (137)	25.3 (194)
	7.1	13.1 (min)	+2	+1		
Cellulose (%)	12.63	87.48 (max)	-2	-1	13.5 (15)	11.7 (13)
Ash (%)	0.16	4.49 (min)	-2	+1	0.8 (23)	6.8 (192)
CSF (ml)	1.51	335 (min)	+2	+1	58 (17)	96 (29)
BF	5.05	53.50 (max)	+1	+1		24.8 (46)
BL (km)	14	6.30 (max)	+1	+1		2.8 (45)
FE	14.4	170 (max)	+1	+1		142 (84)
TF	2.16	87.69 (max)	+2	0	2.3 (3)	8.8 (10)
			-2			
BR (% ISO)	1.1	45.90 (max)	+2	+1	8 (17)	7.8 (17)

KN: Kappa number; BF: burst factor; BL: breaking length; FE: fold endurance; TF: tear factor; BR: brightness

Properties of pulp

R_{14}

The effects of cooking time and NaOH concentration on R_{14} are shown in Table 4 and Equation 3. Usually, R_{14} is related to the amount of oversized pulp fibers that remain on size 14 mesh screens as shives.[18] Variations in R_{14} content with NaOH concentration and cooking time have been modeled by Equation 3 with an error of less than 1 (S = 0.41). The steepest ascent method[24] was applied to Equation 3 in order to determine the minimum value of R_{14} over the range of the process variables studied (normalized values from −2 to +2 for cooking time and -1 to +1 for NaOH concentration); the minimum R_{14} of 0.23% was thus achieved using a high NaOH concentration and a long cooking time (a normalized value of +2 for cooking time and +1 for NaOH concentration) with an error of 158% compared to the experimental results (Table 5). The high error of the predicted R_{14} is likely influenced by other factors, in addition to the two process variables under consideration.

$P_{14}R_{100}$

The effects of cooking time and NaOH concentration on pulp quality, which is related to the amount of fibers that pass through size 14 mesh, but are retained by size 100 mesh ($P_{14}R_{100}$), are shown in Equation 4 and Table 4. According to Equation 4, the two variables affecting $P_{14}R_{100}$ were main and quadratic significant effects of NaOH concentration and cooking time. Based on the intercept of Equation 4, it appears that changing process variables do not considerably impact the amount of quality pulp. However, there is a level for maximum positive effect of both process variables, above which the amount of quality pulp decreases. In order to reach the maximum amount of acceptable pulp, it is necessary to use a low value for both cooking time and NaOH content. Using Equation 4, this condition predicted the maximum amount of acceptable pulp as 77% with an error of 28% compared to the experimental results (Table 5). Table 5 and statistical analyses of the effects of the pulping variables under optimum condition show that the changes in the acceptable pulp amount resulting from variations in NaOH concentration are twice greater than those resulting from cooking time variations.

P_{100}

The effects of cooking time and NaOH concentration on the P_{100} are shown in Equation 5 and Table 4. P_{100}isthe amount of undersized fibers of pulps that pass through size 100 mesh screen. According to Equation 5, both of the process variableshad significant main effects on P_{100} with positive coefficients. The process variablesalso hadsignificant interactions with P_{100}. However, according to the intercept of 47.44 of Equation 5, it seems the impactsof varying process variables on P_{100} are not considerable. Moreover, in order to obtain the minimum amounts of fines, it is necessary to use low cooking time and chemical charge. The minimum value of P_{100} was predicted to be 35.7% by Equation 5, achieved using low cooking time and low chemical concentration, with an error of 5.8% compared to the experimental results (Table 5). Table 5 and statistical analyses of the interaction effects of the pulping variables show that the largest change in $P_{100}$is due to variations in cooking time (40%).

Kappa number

Cooking time and NaOH concentration had significant main and quadratic effects on Kappa number (Table 4, Equation 6).As expected, Kappa number, as a function of pulp lignin content, decreased with an increase in each of the process variables. Therefore, the minimum Kappa number was obtained using maximum values for cooking time and chemical concentration. Using these values, the minimum Kappa number of pulps was predicted to be13.1 based onEquation 6, with an error of 7.1% compared to the experimental results (Table 5). Moreover, the maximum predicted Kappa number of the pulpswas57, which is suitable for fluting paper and linerboard for containerboard products.Statistical analyses of the effects of the pulping variables show that compared to cooking time, NaOH concentration has a greater effect on Kappa number (Table 5).

Cellulose

The effects of cooking time and NaOH concentration on cellulose are shown in Table 4 and Equation 7. Based on Equation 7, small coefficients of process variables compared to the intercept value (75.2) imply only small significant effects of each of these variables. However, there is a significant interaction effect between the variables. Also, cooking time is significant as a quadratic function with a positive coefficient, which indicates a more complicated effect than NaOH concentration. Based on Table 5, in order to reach the maximum amount of cellulose, it is necessary to use a short cooking time and low chemical concentration. Under these conditions,

the optimum amount of cellulose of pulp predicted by Equation 7 was 87.48% with an error of 12.63%.

Ash

Higher amounts of ash in pulp do not have any positive effects on paper properties,but do reduce fiber-bonding. Similar to the fillers used in papermaking, ash reduces the number of bonding sites accessible on the fiber interface by filling in the spaces between fibers.[7] Excessive amounts of ash in the pulp may cause undesirable abrasion of metal parts that repeatedly contact the paper, such as punches, dies, knives, or type. The amount of ash in rice stem ishigh in comparison to other non-wood materials, and this is one of the main obstacles to pulping rice stem using conventional methods.[25] The effects of cooking time and NaOH concentration on ash content are shown in Table 4 and modeled by Equation 8. Based on Equation 8, the ash content of pulp is directly affected by NaOH, with a negative coefficient and as a quadratic function with a positive coefficient. In order to obtain the minimum amount of ash, it is necessary to use a low value for cooking time and a high NaOH concentration (Table 5). The minimum amount of ash in the pulp was predicted to be around 0.16% by Equation 8, with an error of 4.49% compared to the experimental results. Based on this investigation, the largest change in ash content is due to variations of the NaOH concentration, which contributes toeight times as much change as cooking time.

CSF

The effects of cooking time and NaOH concentration on CSF are shown in Table 4 and Equation 9. Based on Equation 9, small coefficients of process variables compared to the intercept value of 414 imply only small significant effects of each of these variables. However, there is no significant interaction effect between the variables. Also, both process variablesare significant as a quadratic function. According to Table 5, in order to reach the minimum amount of freeness, CSF, it is necessary to use a long cooking time and high chemical concentration. Under these conditions, the optimum amount of freeness of pulp predicted by Equation 9 was 335 ml with an error of 1.5%.

Paper properties

Burst factor

Equation 10 predicts that NaOH concentration significantly affects burst factor, while cooking time does not significantly affect this property, when considered separately. This equation predicted the burst factor of paper with correlation coefficients of about 90% and error of less than 5% (Table 4).However, to achieve the maximum burst factor, it is necessary to use high values of both process variables. Using these high values, Equation 10 predicted a maximum burst factor of 53.5, with an error of about 5%, compared to the experimental results (Table 5).

Breaking length

As for burst factor, NaOH concentration significantly affects breaking length, while cooking time does not significantly affect this property, when considered separately (Table 4 and Equation 11). Generally, this property is positively related tothe fiber length. It also seems that fibers are softened with increasing NaOH concentration, resulting in better extruding during passage through screws. In order to reach the maximum breaking length, it is necessary to use a longer cooking time and higher NaOH concentration. Using these high values, Equation 11 predicted an optimum breaking length of 6.3 km, with an error of about 14%, compared to the experimental results (Table 5).

Folding endurance

As for burst factor and breaking length, NaOH concentration significantly affects folding endurance, while cooking time does not significantly affect this property, when considered separately (Equation 12).Here, there was no significant interaction between these process variables. In order to achieve the optimum folding endurance, it is necessary to use higher values of cooking time and NaOH concentration in investigating areas (Table 5). Using these conditions, Equation 12 predicts the optimum folding endurance as170, with an error of about 14%, compared to the experimental results. Based on this statistical analysis, folding endurance varies by 84% with changing NaOH concentration under optimum conditions.

Tear factor

The effects of NaOH concentration and cooking time on tear factor are shown in Table 4 and Equation 13. Based on the high intercept value of Equation 13, tear factor is not seriously affected either by NaOH concentration or by cooking time. It has however been established that tear strength is a function of both fiber strength and fiber bonding.[18,26] Statistical investigations of the process variables under optimum conditions show that the maximum tear factor can be obtained using a moderate NaOH concentration

with either a long or short cooking time (Table 5). The largest change in tear factor resulted from variations of the NaOH concentration.

Brightness

Both cooking time and NaOH concentration had noticeable effects on the brightness of handsheets. Here, the interactions of process variables were significantly influential (Table 4 and Equation 14). The maximum brightness of handsheets were predicted to be 45.90 by Equation 14 using a long cooking time and high NaOH concentration, with an error of about 1%, compared to the experimental results (Table 5). As expected, the brightness of paper increases with higher values of these process variables.

Discussion

The study of the responses of pulp and handsheet properties to process variables provides useful means for optimizing the manufacturing of pulp and paper. It gives, however, little insight into the chemical and physical changes that the raw material has undergone during the pulping process. In this investigation, NaOH concentration had a direct impact on the physical and chemical nature of the resulting pulp, while cooking time did not. Generally, cooking time maintains the chemical reactions, and has no direct influence on the characteristics of the resulting pulp. However, the raw material is softened by increasing cooking time, resulting in better extrusion and thus achieving superior pulp, as confirmed by the $P_{14}R_{100}$.

The development of each pulp property by extruder pulping has its own limitations, which are dictated by both the chemical and morphological characteristics of the constituent elements of the raw material, *i.e.* individual fibers. For instance, the range of strength properties, such as breaking length, burst and tear factors, are limited for each particular type of fiber. The increase in one property is not necessarily coordinatedwith the changes in other properties of the same pulp. For example, the breaking length is mainly associated with the effective number of bonding sites (hydrogen bonds) available on the fiber surface, while the tear index depends mainly on fiber length and strength. However, the mean length of fibers of a particular raw material would not remain unchanged during extrusion of pulp. In addition, the degree of hydrogen bonding, which is finite in nature, may depend, to a certain extent, on the severity of chemical treatment or the extent of dissolution of fiber constituents.[27] Indeed, the behavior of the extruder has a direct relationship with the pre-treatment of pulp under constant extruding conditions. Therefore, it seems that reducing lignin content results in softer pulp, which isbetter disintegrated and refined during extrusion. However, according to intercepts of equations and the results of Table 5, process variable variation does not drastically change cellulosecontent and tear factor. The analysis of the results of Table 5 revealed that the effects of cooking time and NaOH concentration were the same in terms of obtaining high brightness under optimum conditions. Additionally, using longer cooking times and higher amount of alkali results in better overall strength, which involves fundamentally higher costs. Therefore, it is necessary to extend the process conditions to comply with the quality requirements of the handsheets.

Generally, refining of pulp is used to decrease fiber size before the production of microfibrillated cellulose, MFC.[28] Pre-treatment methods such as PFI-mil[29-31] and double disc laboratory refining[32] are used to reduce the energy consumption during MFC production to acceptable levels, which has been identified as one of the major requirements for large scale production of MFC. Beating of initial pulp is a necessary pre-treatment that causes reduced Shopper-Riegler freeness of the obtained pulp. Therefore, using a process for production of initial pulp with low freeness can help to reduce energy consumption during pre-treatment of initial pulp for MFC production. Based on the results of P_{100} and CSF of initial pulp in this investigation, it is suggested that co-rotating intermeshing twin-screw extruder pulping be usedinstead of conventional pulping for production of MFC from non-wood raw materials.Table 6 shows the freeness of initial pulps produced by different processes.

Table 6
Properties of pulps obtained by different pulping processes from rice straw

Process	Yield, %	Freeness, ml	Kappa number	Reference
Kraft	42	669	21	[10]
	32	651	18	
Potassium hydroxide	36	717	19	
Soda	35	627	16	
Soda-antraquinone	37	669	18	
Soda-*para*- benzoquinone	32	651	18	
Twin screw extruder (co-rotating intermeshing)	35	330	14	[This report]
	75	438	75	[8]

CONCLUSION

Pulping of rice stem by a co-rotating intermeshing twin-screw extruder required a relatively short cooking time (about 15-70 min) and a limited NaOH concentration (1-2%). This study demonstrates that hot NaOH pre-treatment can enhance co-rotating intermeshing twin-screw extruder pulping of rice stem fibers. Furthermore, the extrusion of treated rice stem resulted in a medium Kappa number (57) using a short cooking time (15 min) with low chemical content (1% NaOH) at atmospheric boiling point, indicating that the desired properties of the final product dictate the optimized pulping conditions for general packaging applications. Moreover, it is possible to reduce Kappa number to about 15 by increasing the process variables to 75 min and 2% at the atmospheric boiling point, which can be used to bleach pulp without seriously decreasing the pulp and paper properties. Additionally, co-rotating intermeshing twin-screw extruder pulping can be used as an alternative to conventional pulping to reduce energy consumptionfor the production of MFC from non-wood raw materials.

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