

## EVALUATION OF PULP AND PAPER MAKING CHARACTERISTICS OF RICE STEM FIBERS PREPARED BY TWIN-SCREW EXTRUDER PULPING

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Twin-screw extrusion pulping is a new approach to the manufacture of pulp for paper production, designed for non-wood feedstocks. In this research, the production of pulp from rice stem with a newly fabricated twin-screw extruder was investigated. Extrusion pulping of rice stem was conducted following a central composite design using a two-level factorial plan involving three process variables (pretreatment NaOH concentration: 0.4, 0.8, 1.2%; extrusion temperature: 40, 60, 80 °C; and extruder rotational speed: 55, 70, 85 rpm). Responses of pulp and handsheets properties to the process variables were analyzed using statistical software (MINITAB 15). As the results show, pulping of rice stem fiber can be done at a relatively short pretreatment time about 4 hours and a low NaOH concentration about 0.8% by twin-screw extruder with limit extrusion temperature of about 80 °C and extruder rotational speed about 85 rpm. The effect of pretreatment solvent, NaOH, is greatly enhanced by increases in the extrusion temperature. Analysis of the results revealed that this process has suitable potential to be used to obtain a pulp with yields approximately equivalent to neutral sulfite semi-chemical pulping at fixed kappa number, which is applicable for fluting paper and linerboard production.

*Keywords: Pulping; Twin-Screw Extruder; Rice stem; Papermaking; Central composite design*

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### INTRODUCTION

Straw (of any kind, including wheat straw, rice straw, maize straw, etc.) and virtually any other agricultural residues may be used for production of good quality paper pulp. However, due to the bulky nature of these residues, a small scale pulp mill may be more economical as compared to traditional wood pulp mills. In this article a ground, twin-screw extruder, a system designed for non-wood feed-stocks, is considered as a new approach for producing paper pulp. This technology, having been attractive since the most recent three decades as a mini-mill system, will contribute to reducing the human ecological footprint through cleaner and more efficient use of local resources, whilst at the same time incorporating wider social benefits. In fact, instead of traditional pulping systems, e.g. batch digesters, the new pulping system works with a twin-screw extruder, a technology that is widely used in many industries, and allows good mixing, fast throughput, and high pressure and shear force operation (Riddlestone 2006).

Extruder pulping is a mechanical or chemi-mechanical pulping method in which fibers are processed by means of compression and shear forces. The basic principles of this pulping method have been developed with successful results on pilot and industrial applications involving annual agricultural plants such as cotton, hemp, wheat straw, rice straw, and bagasse. The original Bivis (French for twin-screw extruder) process was designed with two pulping extruders in series, the first for impregnation and partial cutting, the second for bleaching and additional cutting. This type of pulper has been generally developed and designed to make a quality printing and writing paper pulp (Westenbroek 2000) but it can also be used to make fluting or other paper grades (Harris et al. 2008).

The physical size of the twin-screw pulper is smaller for similar capacity than traditional pulping systems and consequently carries a lower capital cost as compared to competing technologies. An additional advantage of the new system is that the pulping is conducted with lower water consumption, thus black liquor with higher concentration is produced. This is a beneficial point in the black liquor treatment stage, as it reduces or removes the need for evaporation. Moreover, the material inside the co-rotating extruder barrel travels as an eight-shaped path and thus takes a longer route than if the screws were counter-rotating pulp (Westenbroek 2000). This method imparts less mechanical treatment to the fibers and thus minimizes fiber damage; this fact is important for ensuring high final paper quality (Harris et al. 2008).

It is expected that a fully digested pulp can be produced in a twin-screw extruder without requiring any additional treatment. This would enable rapid response times to be achieved. Therefore quick response will make it much easier to control pulp quality than current technologies (Harris et al. 2008).

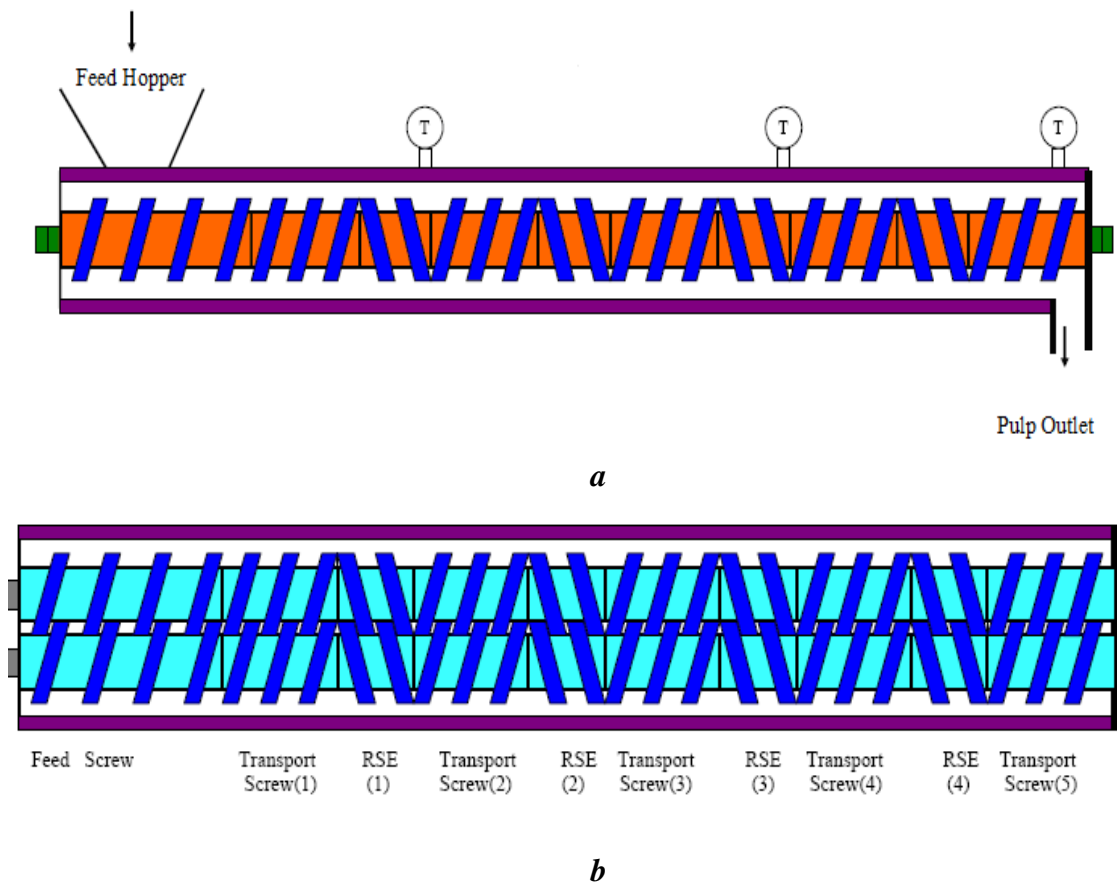
The fibrous material is subjected to alkaline solution impregnation as a common chemical for pretreatment of non-wood materials (Jiménez et al. 2005; Westenbroek 2000; McGovern 1988; Sharma et al. 1984) before entering the extrusion process.

In this research, taking into consideration the advantages of the twin-screw extruder technology and the potential of promoting its efficiency, a laboratory scale co-rotating twin-screw extruder was designed, fabricated, and installed for pulping of non-wood materials. Schematics of the twin-screw pulper are shown in Fig. 1. In this study, rice stem was treated by cold soda, and then pulped with twin-screw pulper. The effects of operational variables on the obtained properties of pulp and handsheets were evaluated.

## **EXPERIMENTAL**

### **Raw Material**

The rice stem used in this study was supplied from a local rice field in the northern region of Iran. Before pulping, the raw material was sun-dried, cleaned, and cut as pieces of approximately 3 cm length. 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, on an oven-dry weight basis (moisture content 9.56%). The deviations of these contents from their respective means were all less than 9.7%. In this research, cold soda was selected for pretreatment and delignification of rice straw at atmospheric pressure.



**Figure 1.** Schematic of the twin-screw extruder for pulping: a) Side view, b) Top view

### Pretreatment of Rice Stem

In a typical experiment, 300 g rice stem was impregnated with 0.4%, 0.8% and 1.2% sodium hydroxide solution in an 8:1 liquid: weight of rice stem ratio (L/W) at room temperature for 4 hours (Table 1). The rice stem was completely submerged and soaked in caustic solution for each run. After impregnation the liquid was allowed to drain through a perforated screen for about 5 minutes.

### Pulping and Papermaking

A series of experiments were done using a 110 mm outside diameter and 1400 mm long twin-screw extruder. The drained pretreated materials were used for pulping with a typical dry matter content of approximately 20%. This materials pulped by extruder under different temperature (40, 60, 80 °C) and speed of extruder (55, 70, 85 rpm). The time consumed during a pulping cycle in an extrusion step varies with the rate of extruder speed. At 70 rpm, it was about 20 minutes. Then the exiting pulp from the extruder was washed with warm water and disintegrated in a standard disintegrator (T 205 sp-95). Because of high initial CSF numbers of the extruded pulp, all pulp samples were beaten in a stainless steel PFI mill under standard conditions (T 248 sp-00) until the freeness reached an appropriate value (~300 CSF). Then handsheets were made, and the physical and mechanical properties of them were determined using standard procedures.

**Table 1.** Values of the Process Variables in the Pulping Process in the Extruder using the Proposed Experimental Pulp and Papermaking System\*

No.	Codes			Values		
	$X_T$	$X_C$	$X_{rpm}$	$T$ (°C)	$C$ (%)	rpm
1	0	0	0	60	0.80	70
2	0	0	0	60	0.80	70
3	0	0	0	60	0.80	70
4	-1	0	0	40	0.80	70
5	0	0	-1	60	0.80	55
6	-1	+1	+1	40	1.20	85
7	0	+1	0	60	1.20	70
8	+1	+1	+1	80	1.20	85
9	0	0	+1	60	0.80	85
10	+1	-1	-1	80	0.40	55
11	0	-1	0	60	0.40	70
12	-1	+1	-1	40	1.20	55
13	-1	-1	+1	40	0.40	85
14	-1	-1	-1	40	0.40	55
15	+1	+1	-1	80	1.20	55
16	+1	0	0	80	0.80	70
17	+1	-1	+1	80	0.40	85

\*  $X_T$ : Normalized temperature of Extruding;  $X_C$ : Normalized NaOH concentration;  $X_{rpm}$ : Normalized speed of Extruder;  $TE$ : Temperature of extruder;  $C$ : NaOH Concentration; rpm: speed of Extruder;  $PTT$ : Pre Treatment Time=240 min, L/W=8:1

### Analysis Methods of Raw Materials, Pulp and Paper Sheets

The starting materials and the products obtained from them were characterized according to the following standard methods: Klason lignin (TAPPI T 222 om-98), cellulose (Kurshchner-Hoffner Rowell 1984), and ethanol/ acetone extractables (TAPPI T 204 cm-97). Pulp yield was determined gravimetrically following drying at 105 °C ±2 for 24 h. Test methods of the technical association of the pulp and paper industry (2006-2007) were used for measurements of freeness (TAPPI T 227 om-99), and Kappa number (TAPPI T 236 om-99). In addition, handsheets of 60 g/m<sup>2</sup> were formed, and their properties were evaluated in accordance with the TAPPI standard methods. The handsheets were conditioned at 23 °C and 50% RH for at least 24 h before testing.

### Experimental Design

The tested model uses a series of points (experiments) around a central one (central experiment), and several additional points (additional experiments), to estimate the first- and second-order interaction terms of a polynomial. This design meets the general requirement that every parameter in the mathematical model can be estimated from a fairly small number of experiments. The total number of observations (experiments)  $N$  required for the three independent variables (viz. temperature - $T$ -, pretreatment NaOH solution concentration - $C$ - and rotational speed of extrusion motor -rpm-) was calculated from the following equation (Rezayati-Charani et al. 2005):

$$N = 2^k + (2 \times K) + 3 \quad (1)$$

The value of  $N$  was found to be 17. The parameter  $K$  in the equation is the number of independent variables. The experimental data were fitted to the following second-order polynomial,

$$Z = a + bX_T + cX_C + dX_{RPM} + eX_T^2 + fX_C^2 + gX_{RPM}^2 + hX_TX_C + iX_TX_{RPM} + jX_CX_{RPM} \quad (2)$$

where  $Z$  denotes the response variables (yield =  $YI$ , kappa number =  $KN$ , initial freeness =  $CSF$ ) for pulp, and (brightness =  $BR$ , breaking length =  $BL$ , burst index =  $BI$ , folding endurance =  $FE$ , tear index =  $TI$ , corrugated medium test =  $CMT$ , ring crush test =  $RCT$ ) for handsheet papers. The parameters  $X_T$ ,  $X_C$ , and  $X_{rpm}$  are the normalized values of  $T$ ,  $C$ , and  $rpm$ , and the letters  $a$  to  $j$  denote constants.

The values of the independent variables were normalized from  $-1$  to  $+1$  by using Eq. (3) in order to facilitate direct comparison of the coefficients and visualization of the effects of the individual independent variables on the response variable,

$$X_n = 2 \frac{X - X_{mean}}{X_{max} - X_{min}} \quad (3)$$

where  $X_n$  is the normalized value of  $T$ ,  $C$ , or  $rpm$ ,  $X$  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 values, respectively, of such a variable. This normalization also results in more accurate estimates of the regression coefficients as it reduces interrelationships between linear and quadratic terms (Rezayati-Charani et al. 2006).

The 17 experiments conducted, together with the corresponding normalized values for the independent variables, are given in Table 1. The values of responses obtained allow the calculation of 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 the statistical software MINITAB of Minitab, Inc., USA.

## RESULTS

### Response Surface Analysis of Pulping with Extruder and Handsheet Making

The characteristics of the pulp and handsheet paper obtained in the 17 pulping runs (each of runs with three repetitions) are summarized in Table 2. Data processing enabled estimation of the main effects and the interactions of the factors for the responses considered. The effect of every factor is the change in the response when it is changed from the low level ( $-1$ ) to the high level ( $+1$ ). The main effect of each factor estimates its average effect over all possible conditions of the other variables. Each of the responses analyzed can be affected only by the main effects of processing variables and interactions among them. The main effect of a variable should be individually interpreted only if there

is no evidence that the variable interacts with other variables. When there is evidence of one or more such interactions, the interacting variables should be considered jointly.

**Table 2.** Mechanical and Chemical Properties of the Pulp and Paper Obtained in the Pulping Process in the Extruder, Using the Proposed Experimental Pulp and Papermaking System \*

No.	Y(%)	G(g/m <sup>2</sup> )	KN	CSF-in (mL)	CSF_fi (mL)	BL (m)
1	76.55	60.33	103	483	302	2757
2	72.31	63.00	94	445	292	2690
3	76.90	62.67	88	463	283	1509
4	73.96	62.33	98	608	317	2787
5	75.99	61.67	99	585	312	2599
6	70.36	63.57	91	578	298	4068
7	72.63	60.67	75	462	285	3549
8	72.84	60.17	79	433	293	1886
9	76.88	60.17	83	477	302	1456
10	78.23	60.33	92	460	308	741
11	77.90	58.67	98	488	307	1331
12	70.57	64.67	84	513	282	3831
13	79.95	62.33	100	628	315	1036
14	79.28	61.00	112	605	210	719
15	75.23	60.33	75	438	287	2380
16	75.65	60.50	85	535	302	2361
17	85.17	60.00	93	448	308	1213

No.	BR (%ISO)	BI (kN/g)	TI (mNm <sup>2</sup> /g)	FE	CMT (kw/g)	RCT (kw/g)
1	25.60	2.68	4.88	4	35.00	0.23
2	25.70	0.75	4.88	4	38.33	0.25
3	27.70	1.62	7.65	4	40.00	0.22
4	24.50	0.74	7.49	2	47.00	0.30
5	25.70	2.59	7.33	1	40.00	0.20
6	22.30	2.46	4.88	12	48.00	0.25
7	25.00	0.55	2.44	4	42.00	0.25
8	20.27	1.49	2.28	8	38.67	0.25
9	26.77	1.48	7.65	12	29.67	0.20
10	23.53	1.61	4.88	1	29.67	0.20
11	26.90	2.56	2.12	3	28.00	0.20
12	21.97	0.76	2.12	3	52.00	0.35
13	25.30	1.53	4.88	12	35.00	0.20
14	27.37	1.64	4.88	1	30.00	0.20
15	24.20	0.68	7.33	1	56.67	0.27
16	23.90	2.17	4.88	3	36.67	0.28
17	26.40	1.38	2.44	17	29.67	0.39

<sup>a</sup> Y: Yield %; G: grammage; KN: Kappa Number; CSF\_in: Canadian freeness of Initial pulp (after extrusion); CSF\_fi: Canadian freeness of pulp after PFI Refining; BL: breaking length; BR: brightness of handsheet from unbleached pulp; BI: burst index; TI: tear index; FE: folding endurance; CMT: Corrugated medium test; RCT: Ring crush test.

A set of three preliminary experiments was conducted under the central operating conditions, namely: 60 °C, 0.8%, and 70 rpm. The experimental results obtained in the determinations of the dependent variables differed from the mean values, as shown in the last row of Table 1, by less than 5 to 10%. Subsequent tests, corresponding to the experimental design adopted, provided the results shown in the other rows. Based on the results of preliminary experiments, the operating variables were changed over the following ranges: 40–80 °C, 0.4–1.2 %, and 55–85 rpm in order to obtain pulps spanning the yield range 70 to 85% (i.e. at neutral sulfite semi-chemical pulps). The MINITAB 15 software suite was used to conduct a multiple polynomial regression analysis involving all the terms of Eq. (2) except those with alpha-to enter 0.15 and alpha to remove 0.15, which were left out using the stepwise method (Draper and Smith 1981). The following equations (4) to (13) and their coefficients are reduced models for each response:

$$\text{Yield} = 76.13 - 2.76C + 1.92TC - 1.04TR - 1.24T^2 \quad (4)$$

$$S = 0.95, R_{-sq} = 91.7, R_{-Sq(adj)} = 88.9, R_{-Sq(pred)} = 78.65$$

$$\text{Freeness}_{\text{initial}} = 4743 - 69.2T + 56.2T^2 - 20.5C - 13.1TR + 13.1TC + 10R - 16R^2 + 6CR \quad (5)$$

$$S = 9.19, R_{-sq} = 98.47, R_{-Sq(adj)} = 97.6, R_{-Sq(pred)} = 97.76$$

$$\text{Kappa Number} = 91.65 - 9.23C - 6.07T + 2.88CR - 6.28C^2 + 5.75R^2 + 1.29TR \quad (6)$$

$$S = 2.56, R_{-sq} = 94.42, R_{-Sq(adj)} = 67.94, R_{-Sq(pred)} = 92.12$$

$$\text{Breaking Length} = 2556 + 1067C - 479TC - 386T \quad (7)$$

$$S = 264, R_{-sq} = 94.97, R_{-Sq(adj)} = 93.29, R_{-Sq(pred)} = 89.59$$

$$\text{Fold Endurance} = 3.48 + 5.40R + 3.32R^2 - 1.38TC - 1.37CR - 0.67C \quad (8)$$

$$S = 1.07, R_{-sq} = 95.61, R_{-Sq(adj)} = 95.23, R_{-Sq(pred)} = 94.10$$

$$\text{Burst Index} = 1.55 + 0.881C - 009TC \quad (9)$$

$$S = 0.102, R_{-sq} = 98.2, R_{-Sq(adj)} = 97.91, R_{-Sq(pred)} = 97.19$$

$$\text{Tear Index} = 4.88 + 2.165C + 0.44T - 0.32C^2 - 0.16TC \quad (10)$$

$$S = 0.19, R_{-sq} = 99.14, R_{-Sq(adj)} = 98.85, R_{-Sq(pred)} = 98.37$$

$$\text{Brightness} = 26.20 - 2.36T^2 - 1.43C - 0.74CR + 0.43TC \quad (11)$$

$$S = 0.78, R_{-sq} = 83.88, R_{-Sq(adj)} = 82.48, R_{-Sq(pred)} = 79.86$$

$$\text{CMT} = 36.16 + 8.5C - 3.4CR - 2.7R + 4.3T^2 \quad (12)$$

$$S = 3.72, R_{-sq} = 85.28, R_{-Sq(adj)} = 80.38, R_{-Sq(pred)} = 69.51$$

$$\text{RCT} = 0.23 - 0.039CR - 0.035TC + 0.061T^2 + 0.033TR + 0.017C - 0.027R^2 \quad (13)$$

$$S = 0.02, R_{-sq} = 91.00, R_{-Sq(adj)} = 85.61, R_{-Sq(pred)} = 54.21$$

As can be seen, the statistical analysis (with the  $R$ -sq,  $R$ -sq(adjusted),  $S$ ,  $R$ -sq(predicted) values for the fitted lines) of equations (4) through (13) confirms the adequacy of the fitted models, where all models with the exception of CMT are R-sq at a level of 80 or more. According to method of this software, R-square value was reported between zero and hundred. Also shown are the coefficient estimates for each term in the selected models for all dependent variables.

Optimum (maximum) values of the dependent variables and variations with changes in the independent variables in the extruder pulping and handsheet making of rice stem are presented in Table 3.

**Table 3.** Optimum (maximum) Values of the Dependent Variables and Variations with Changes in the Independent Variables of the Extruder Pulping and Handsheet Making of Rice Stem

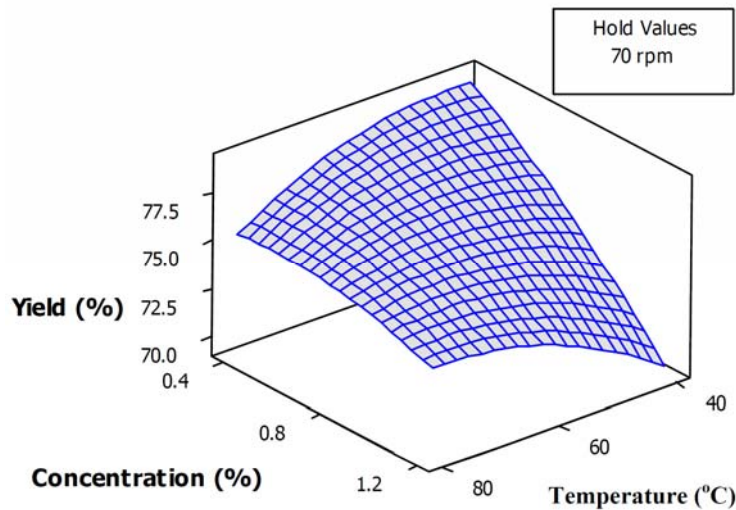
Variable dependent	Error percentage of the experimental values with respect to optimum values	Optimum (maximum) values	Normalized values of the independent variables leading to optimum values of the dependent variables			Changes in the dependent Variables (%) with changes in the independent variables (from -1 to +1)		
			$X_T$	$X_C$	$X_{rpm}$	$T$	$C$	$Rpm$
Yield (%)	0.44	79.58	-1	-1	+1	7.3	11.6	2.6
	2.02	(Min) 69.17	-1	+1	-1	8.5	13.5	3
Kappa number	1.58	111	-1	-1	-1	13	21.9	7.54
	4.21	(Min) 71.65	+1	+1	-1	20.5	33.8	11.6
Freeness (CSF)	0.69	(Min) 435	+1	+1	-1	19.8	6.2	1.3
Breaking length(m)	14.64	4488	-1	+1	-1	38.5	84	9.3
Burst index (KN/g)	1.97	2.53	-1	+1	+1	7.1	77	—
	2.79	7.33	-1	+1	-1	4.4	75.4	—
Tear index(mN <sup>2</sup> /g)	13.49	16	+1	-1	+1	18	44	87
	9.75	28	0	-1	0	3.1	9.5	5.4
Folding endurance	3.17	55.09	-1	+1	-1	—	43.2	22.1
	3.46	0.39	+1	+1	-1	40.7	40.7	45.7
Brightness(ISO)	5.6	—	-1	+1	-1	48	40.7	45.7
	3.46	—	+1	+1	-1	48	40.7	45.7
CMT(kW/g)	5.6	—	-1	+1	-1	48	40.7	45.7
RCT(kW/g)	9.8	0.39	-1	+1	-1	48	40.7	45.7

## Yield

The calculated yield values of pulp obtained from Eq. (4) reproduced their experimental counterparts with errors less than 1 ( $S = 0.95$ ). The steepest ascent method (Press et al. 1992) was applied to Eq. (4) in order to determine the highest yield over the ranges of process variables studied (normalized values from -1 to +1 for all); the calculated maximum yield obtained was 79.58% at low temperature and the NaOH concentration (a normalized value of -1 for both variables) and at in the high extruder speed (normalized value +1), which is the same as a typical yield of NSSC pulp. Also Eq. (4) allows the estimation of the variation of the yield with changes in each independent variable, over the range considered, while holding the other two variables constant under optimum condition. The greatest changes in yield resulted from variation of the NaOH concentration (11.61%) and the smallest ones from the speed of extruder (2.58%) under maximum yield. As a result, the yield was much more sensitive to changes in the NaOH concentration than in the extrusion temperature and the speed of extruder (Table 3). Although the extruder speed had no significant effect on the yield, interaction of this variable with the extrusion temperature appeared to be significant in a negative way. Also the interaction of the extrusion temperature with the NaOH concentration appeared significant in a positive way, relative to the yield. Figure 2 shows the variation of pulp



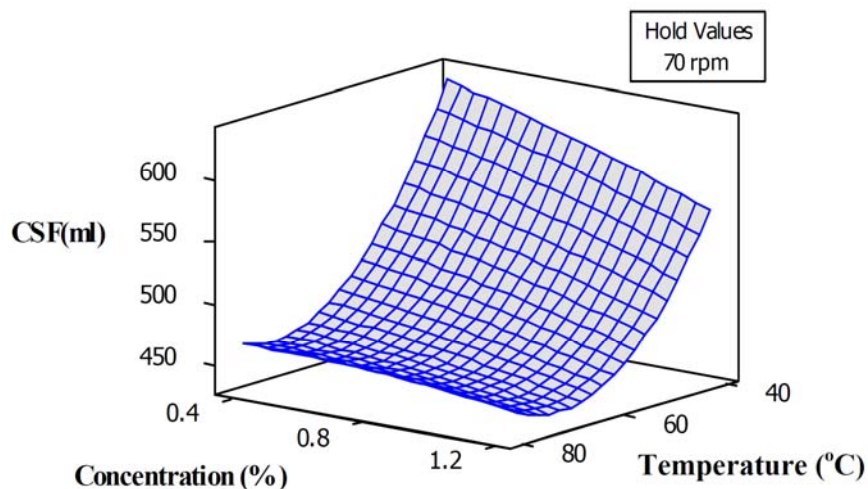
yield with the NaOH concentration and the extrusion temperature at a medium, constant speed of the extruder.



**Figure 2.** Variation of pulp yield with The NaOH concentration and the extrusion temperature at a medium, constant RPM of the extruder

### Initial Freeness

The initial freeness, a parameter related to the drainage of pulp, was obtained for pulp taken from the extruder without any refining. This pulp was refined to a freeness of about 300 mL CSF for papermaking. Of the three investigated factors, the most influential was the extrusion temperature (19.8%), with the NaOH concentration having a smaller role (6.2%) and the extruder speed the least effect (1.3%) on the initial freeness (Table 3). Statistical analyses of the effects of these factors showed that the extrusion temperature and the extruder speed appeared as second power terms, indicating that these parameters can be optimized to certain values.

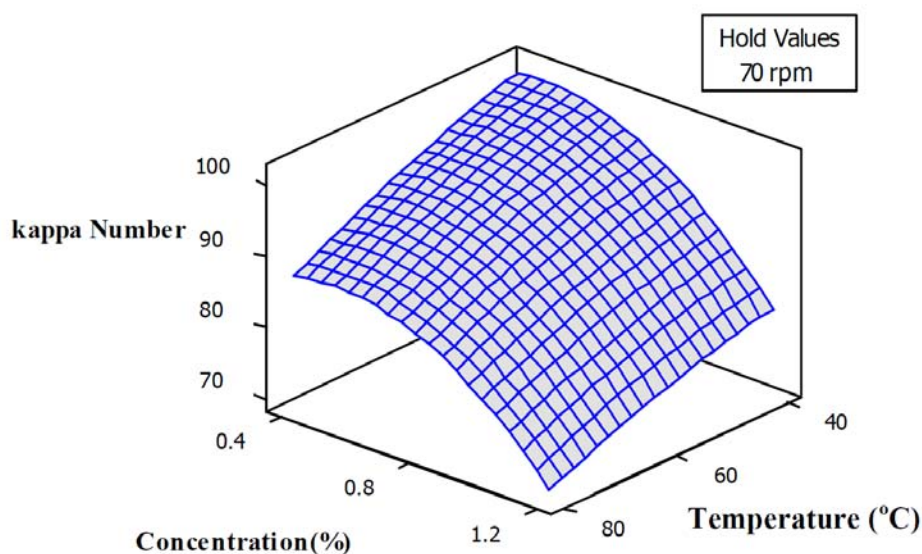


**Figure 3.** Variation of initial freeness with the NaOH concentration and the extrusion temperature at a medium, constant extruder speed

In fact, the positive coefficient of the extrusion temperature indicates a minimum level of freeness and for the extruder speed and vice versa. Also the interaction of extrusion temperature appeared significant in a negative way with the NaOH concentration on the initial freeness and in a positive way with the extruder speed (Eq. 6). The effect of extrusion temperature and the NaOH concentration at a constant extruder speed (70 rpm) is presented in Fig. 3.

### Kappa Number

The main effects of the extrusion temperature, the pretreatment NaOH concentration, and the extruder speed on Kappa number are shown in Table 3. It should be noted that the variation of the NaOH concentration had the most influence on reducing the lignin in the pulp, followed by the extrusion temperature, and the extruder speed the least. Statistical analyses of the interaction effects of the three factors showed significant interaction between the extruder speed with the pretreatment NaOH concentration and the extrusion temperature (Eq. 6). Also, the effect of pretreatment NaOH concentration appeared as a second power term. The responses of Kappa number to the extrusion temperature and the pretreatment NaOH concentration at a medium, constant extruder speed are shown in Fig. 4.

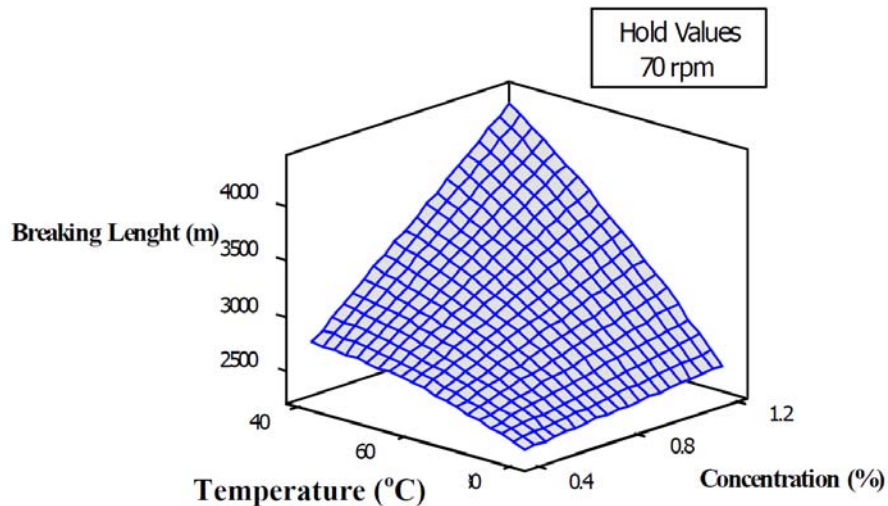


**Figure 4.** Variation of Kappa Number with the NaOH concentration and the extrusion temperature at a medium, constant extruder speed

### Breaking Length

The main effects of the extrusion temperature, the NaOH concentration, and the extruder speed on breaking length are shown in Table 3 and Eq. (7). It is apparent that the NaOH concentration is the most influential factor in relation to the breaking length of handsheets, followed by the extrusion temperature, in order of importance. The effect of the extruder speed was insignificant. Also, statistical analyses of the interaction of the three factors showed that the only significant interaction was related to the NaOH concentration with the extrusion temperature. This interaction had a negative effect on breaking length. For example, in Table 1 it is shown that the breaking length at 40 °C was significantly improved by the increase in the NaOH concentration from 0.4% to 1.2%.

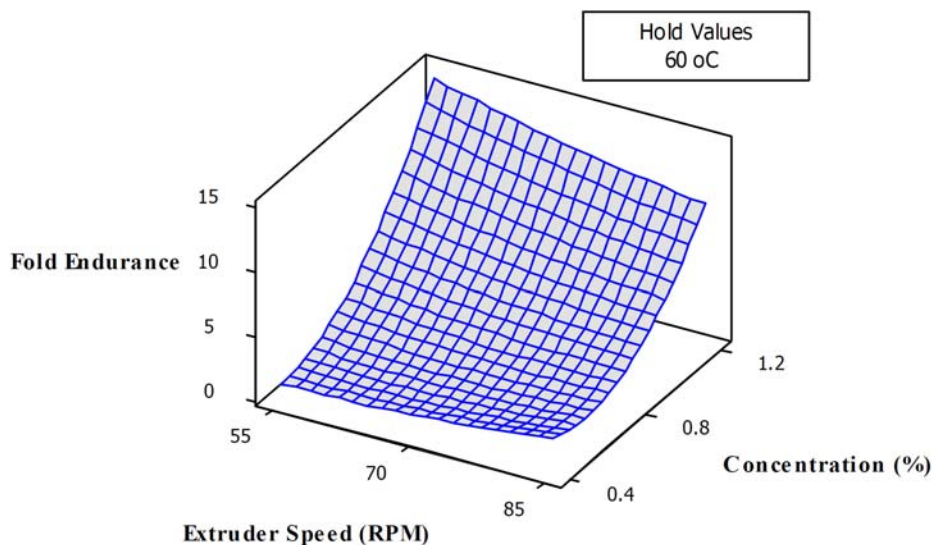
The influence of the extrusion temperature and the NaOH concentration variation at a medium constant extruder speed is presented in Fig. 5.



**Figure 5.** Variation of breaking length with the NaOH concentration and the extrusion temperature at a medium, constant extruder speed

### Folding Endurance

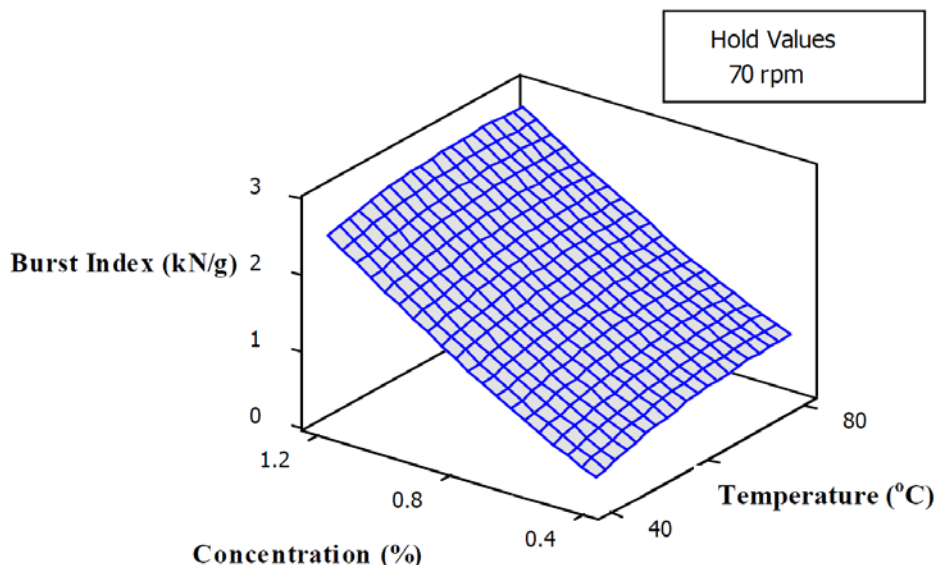
The main effects of the extrusion temperature, the NaOH concentration, and the extruder speed on folding endurance are shown in Table 3 and Eq. (8). It is obvious that the extruder speed was the most influential factor on the fold endurance of handsheets, followed by the NaOH concentration. The effect of the extrusion temperature was comparatively small. Statistical analyses of the interaction effects of the pulping variables showed that although the extrusion temperature had no main significant effect on handsheet fold endurance alone, it had significant negative interactions with the NaOH concentration and the extruder speed. A response surface graph showing the effect of variations on at 60 °C fold endurance is presented in Fig. 6.



**Figure 6.** Variation of fold endurance versus the extruder speed and the NaOH concentration at a medium, constant extrusion temperature.

## Burst Index

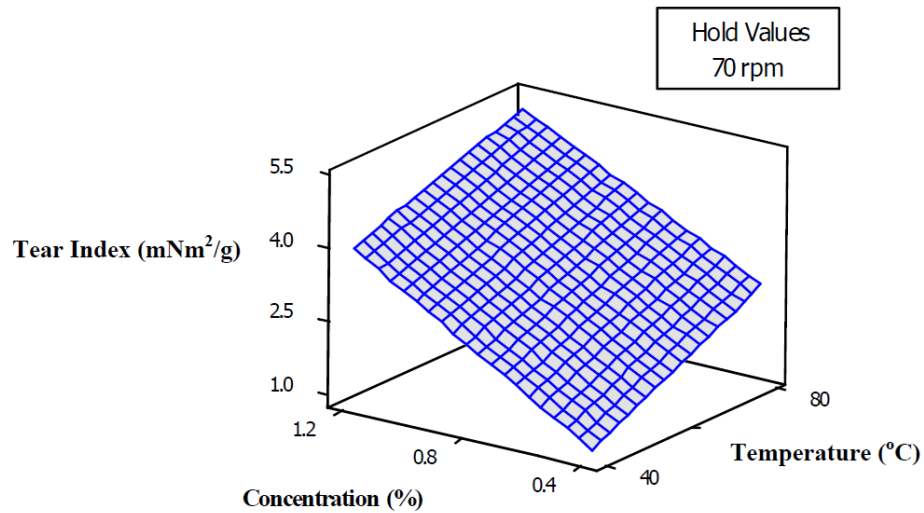
In terms of the main effects on burst index, it was found that the NaOH concentration had the greatest, followed by the extrusion temperature, while the extruder speed was the least influential (Table 3 and Eq. 9). Statistical analyses of the interaction effects of the three variables showed that neither the extruder speed nor the extrusion temperature had significant effects on handsheet burst index. Also, only interaction between the extrusion temperature and the NaOH concentration was negatively significant at all values of the extruder speed. The variation of the NaOH concentration and the extrusion temperature is shown in Fig. 7.



**Figure 7.** Variation of burst index with the extruder speed and the extrusion temperature at a medium, constant extruder speed.

## Tear Index

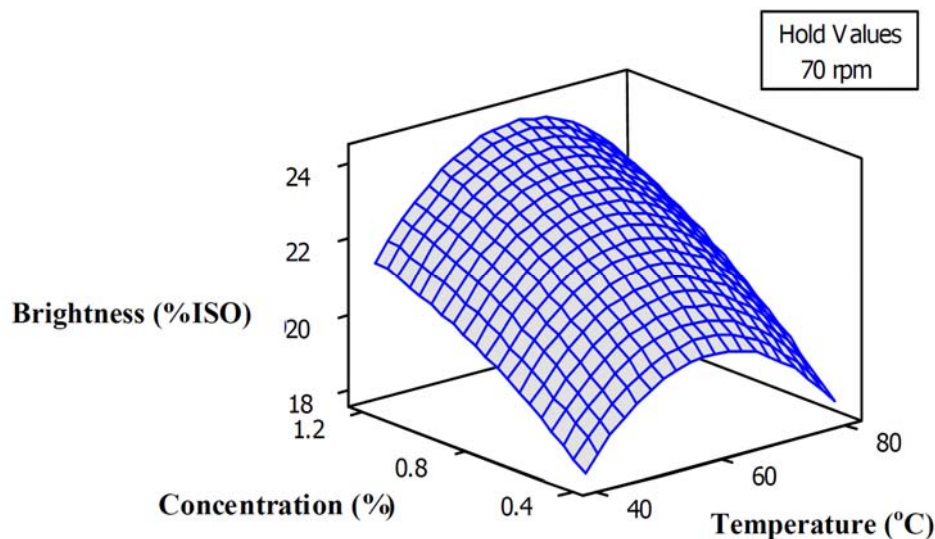
It is obvious from Eq. (10) and Table 3 that the NaOH concentration had the most significant effect on tear index, followed by the extrusion temperature, while the effect of the extruder speed was comparatively small and insignificant. Statistical analyses of effects of the three factors showed not only that the NaOH concentration and the extrusion temperature as main factors had direct positive influences on tear index, but they had negative significant interactions together at all values of extruder speed. Definitely, the effect of the extruder speed on handsheet tear index was insignificant. However, it has been established that tear strength is a function of both fiber strength and fiber bonding (Page and MacLeod 1992; Seth and Page 1988). So, the dependency of tear index on the pretreatment NaOH concentration is probably most related to these parameters. At low NaOH concentration, the degree of delignification is relatively small; hence an insignificant amount of lignin is still extracted from the pulp, resulting in lower bonding and consequently reduced tear strength. At the other hand, at the higher NaOH concentration some damage of fiber occurred, resulting in slightly reduced tear strength, but because the delignification was increased, improved bonding can contribute to increasing tear index. The effects of variation of process variables on tear index are shown in Fig. 8 in the form of a response surface graph.



**Figure 8.** Variation of tear index with the NaOH concentration and the extrusion temperature at a medium, constant extruder speed

### Brightness

In terms of the main effects on brightness of handsheets, Table 3 shows that the NaOH concentration had the greatest effect, followed by the extruder speed, while the extrusion temperature had the least effect. According to the results of right column of Table 3, if the parameters of extruder temperature, NaOH concentration, and extruder speed were held constant individually at their normalized values 0, -1, and 0, respectively, the greatest changes in brightness resulted from variation of the NaOH concentration (9.52%), followed by the extruder speed (5.36%), while the extruder temperature had the least effect (3.11%). The effect of the NaOH concentration and the extrusion temperature on brightness is shown in Fig. 9.

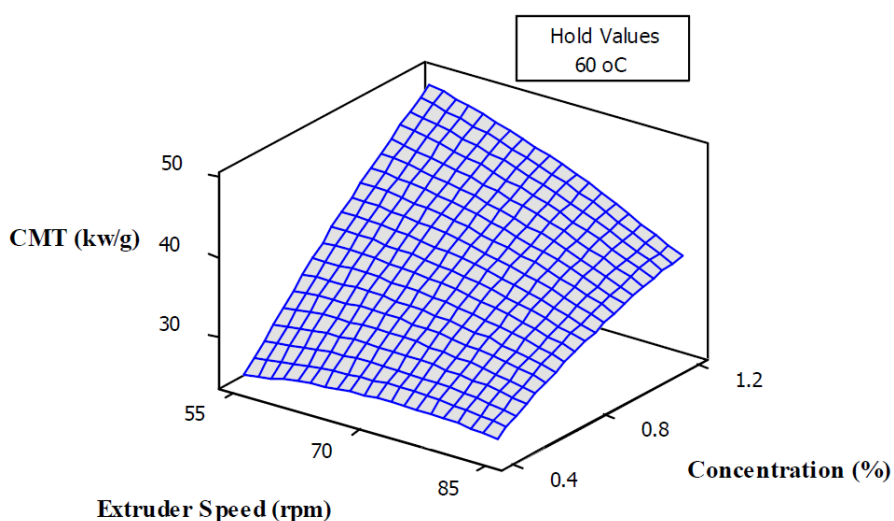


**Figure 9.** Variation of brightness with the NaOH concentration and the extrusion temperature at a medium, constant extruder speed

Statistical analysis of the effects of the three variables showed that the NaOH concentration was the only parameter that had a linear effect on brightness. Also, the extrusion temperature appeared as a second power term, indicating that this parameter can be optimized to a certain value. In fact, the negative coefficient of the extrusion temperature indicates a maximum level of brightness. In addition, the speed of extruder effect was only significant as an interaction with the NaOH concentration (Eq. 11).

### CMT

In terms of the main effects on corrugated medium test (CMT), it was observed that the NaOH concentration had the greatest effect, followed by extruder speed, while the extrusion temperature had the least effect (Table 3). As seen from Eq. (12), the extrusion temperature appeared as a second power term, indicating that this parameter can be optimized to a minimum value. Statistical analyses of the interaction effects of the three variables showed that only interaction between the NaOH concentration and the extruder speed was negatively significant. The interaction of the NaOH concentration and the extruder speed is shown in Fig. 10.

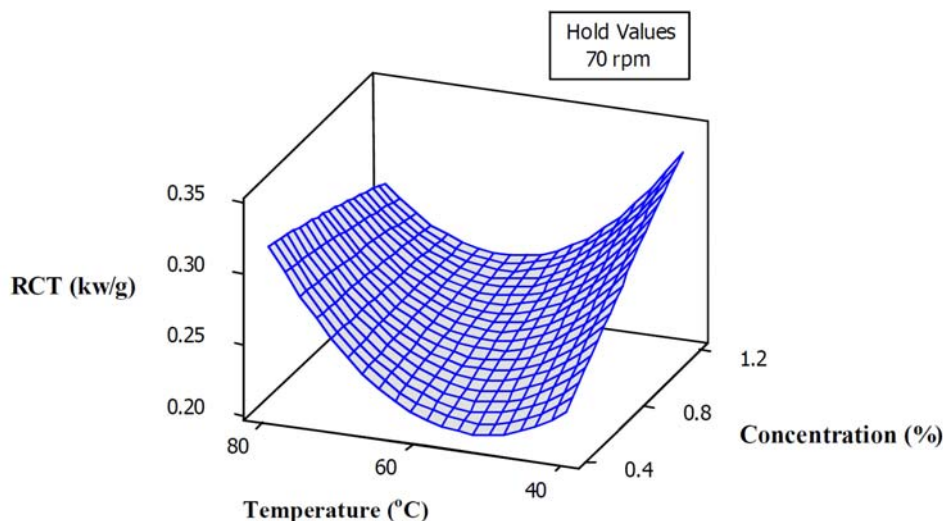


**Figure 10.** Variation of CMT with the extruder speed and the NaOH concentration at a medium, constant extrusion temperature

### RCT

The main effects of the extrusion temperature, the NaOH concentration, and the extruder speed on ring crush test (RCT) results are shown in Table 3. It is apparent that the NaOH concentration was the most influential factor in relation to the RCT of handsheets, followed nearly equally by the extrusion temperature and extruder speed. Statistical analyses of the effects of the three variables showed that just the NaOH concentration had main significant effect on RCT, all of the interactions of parameters were significant together too (Eq. 13). In addition, the extrusion temperature and the extruder speed appeared as second power terms, indicating that these parameters can be optimized to certain values. In fact, the positive coefficient of the extrusion temperature indicates a minimum level of CMT and for the extruder speed vice versa. A response

surface graph showing the effects of the NaOH concentration and the extrusion temperature on RCT at 70 rpm is presented in Fig. 11.



**Figure 11.** Variation of RCT with the NaOH concentration and the extrusion temperature at a medium, constant extrusion speed

## DISCUSSION

Studying the responses of pulp and paper properties to the process variables provides a useful means for optimizing the pulping conditions. It gives, however, little insight into the chemical and physical changes that the raw material has undergone during the process. In this investigation, the extrusion temperature and extrusion speed used are variables of extrusion that have a direct impact on the physical and chemical nature of the resulting pulp and paper.

As extrusion is often performed with pre-treated raw material, it is important to examine the influence of soaking liquids on the product of extruder pulping. Sodium hydroxide was used to decompose the lignin present within the fiber as well as at inter-fiber locations to facilitate defibration. Also, use of sodium hydroxide results in a more swollen fibre wall, as well as an easy slippage of elementary fibres along each other and a change in fibre cellulose structure (Westenbroek 2000). The statistical effect of different process conditions on the pulp and paper properties is summarized in Table 4.

On the base of these results, the trend of effects of extrusion parameters on raw material can be predicted. Consequently it is possible to control the extrusion process. As shown, pretreatment with sodium hydroxide appears directly to increase initial freeness, breaking length, burst index, folding endurance, and tear strength; these effects can be attributed to improved bonding and higher fiber length. Even so, by increasing the extrusion temperature, there were no typical effects of strengths, except for the initial freeness and tear index of the product. But, as Eqs. (4) to (14) show, this factor had significant interaction with both other factors.

**Table 4.** The Effect of Extrusion Pulping Conditions at Center Point (80 °C, 0.8% and 70 rpm) on Pulp and Paper Properties

	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Extrusion temperature	↓	↑	C	C	C	C	↓	↑	↓	↑	C	C	↓	↑
Screw speed (rpm)	C	C	↓	↑	C	C	↓	↑	C	C	↓	↑	↓	↑
NaOH concentration	C	C	C	C	↓	↑	C	C	↓	↑	↓	↑	↓	↑
Yield	-	-	+/-	+/-	+	-	-	-	+	-	+	-	+	-
Kappa Number	+	-	+	+	+	-	+	0+	++	--	++	-	++	--
Initial Freeness	++	0-	-	0-	+	+	++	-	++	-	+/-	0-	++	-
Breaking Length	0+	0-	+/-	+/-	-	+	0+	0-	0-	0+	-	+	0-	0+
Fold endurance	+/-	+/-	-	++	+/-	+/-	-	++	0-	0-	--	++	--	+
Burst Index	+/-	+/-	+/-	+/-	-	+	-	+	--	0+	-	+	--	0+
Tear Index	-	+	+/-	+/-	--	+	-	+	--	+	--	+	--	+
Brightness	+/-	+/-	+/-	+/-	+	-	+/-	+/-	+	-	+	--	-	--
CMT	+/-	+/-	+	-	--	++	+	-	--	++	--	+	--	+
RCT	+/-	+/-	+/-	+/-	0-	0+	+	+	0+	+	--	-	-	0-

++ more positively affected

↑ increase

↓ decrease

C constant

O+ slightly positively affected

-- more positively affected

+ positively affected

- negatively affected

+/- not affected

O- slightly negatively affected

Beside, the variation of the extruder speed did not significantly affect strengths, except for the predicted values of CMT and folding endurance. In other words, the throughput increases with increase of the extruder speed. Therefore, utilizing a high speed may be economically recommended.

In general, according to results of handsheet properties (Table 1), at these process conditions, extrusion pulping of rice stem can be used to make fluting paper and linerboard. In addition, as compared to the fluting paper properties, these pulps need to be passed through the same extruder or undergo beating action until achieving a sufficient freeness ( $\approx 300$  ml, CSF). High extrusion temperatures and speeds can be used for obtaining better handsheet strength.

## CONCLUSIONS

In this research, extrusion pulping of rice stem was performed with a newly fabricated twin-screw extruder, and the optimum conditions were obtained at a relatively short pretreatment time (about 4 hours), low NaOH concentration (about 0.8 %), moderate extrusion temperature (about 80 °C), and moderate extruder rotational speed (about 85 rpm). The effect of pretreatment NaOH solution is greatly enhanced by increasing the extrusion temperature. Analysis of the results revealed that this process can be used to obtain a pulp with yields equivalent to neutral sulfite semi-chemical pulping at fixed kappa number that is applicable for fluting paper and linerboard production.



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