Process Parameters Optimization of *Casuarina equisetifolia* for Enhanced Production of Bleachable Grade Kraft Pulp through RSM

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Casuarina equisetifolia (beach sheoak), a fast-growing deciduous tropical hardwood tree, was utilized to assess its suitability for pulp and paper production. Response surface methodology (RSM) based on central composite design (CCD) was used to optimize the kraft pulping process by varying the alkali doses, reaction temperature, and time. Using the best optimum pulping conditions with alkali charge 17.9%, temperature 170.2 °C, and pulping time 82.1 min, a high screened pulp yield of 52.4% with low kappa number of 18.1 and pulp viscosity of 974.5 cm^3/g could be achieved. The laboratory handsheets prepared with kraft pulp under optimum conditions showed good mechanical strength properties at an optimum beating level of 45 °SR.

Keywords: Casuarina equisetifolia; Response surface methodology; Kraft pulping; Central composite design

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INTRODUCTION

With the rapidly increasing world's population and literacy rate, paper industries are facing a serious lack of better quality wood fibres. An inadequate supply of wood fibre due to rapid depletion of forest wealth is forcing the pulp and paper industries located in southern part of Asia to use other alternate fibrous resources such as non-woody plants, agricultural residues, fast-growing trees, and waste papers. Many fast-growing plants have been cultivated and studied for their potential as an alternative source of raw materials for the pulp and paper industry (Malik *et al.* 2004; Lal *et al*. 2010).

Casuarina equisetifolia, commonly known as beach sheoak or iron wood, is a tall and fast-growing hardwood tree belonging to the family Casuarinaceae that can grow easily on infertile, highly alkaline soils, as well as in highly degraded habitats. Moreover, this plant has an additional importance because of its nitrogen-fixing ability, which is due to its symbiotic association with the bacteria *Frankia* species. In the past, only a few studies have been done on the pulping process of *C. equisetifolia* to test its suitability for pulp and paper making (Guha and Sharma 1970; Guha and Karira 1981). These studies were based on one-variable-at-a-time (OVAT) approach. However, pulping is a complex heterogeneous process, involving the interaction effects of many variables. The OVAT approach is inefficient for optimization of multi-variable system because it does not illuminate the interactions between different variables and eventually, the complete effect of the variables on the process cannot be observed (Myers *et al.* 1995). Response surface methodology

(RSM) can eliminate these limitations of a single variable optimisation process (Bas and Boyaci 2007). RSM has been used in many pulping processes to optimize the pulping conditions using various raw materials (Wanrosli *et al*. 2004; Shirkolaee *et al*. 2008). With this background, pulping and bleaching of *C. equisetifolia* were conducted (Poonam and Dutt 2014; Maan and Dutt 2017).

To the best of our knowledge, this is the first study to determine the optimum kraft pulping conditions leading to maximum screened yield, low kappa number (approximately lower than 20), and acceptable viscosity by using RSM for *C. equisetifolia*. In the present study, RSM based on central composite design (CCD) has been adopted for evaluating the effective factors, building models, studying interaction between the variables, and for determining the optimum conditions of variables for the desirable responses. Statistical software Minitab-16 was used to generate CCD table, facilitate the analysis and optimize the above mentioned three variables.

EXPERIMENTAL

Raw Materials

Wood chips of *C. equisetifolia* were provided by ITC Limited, Bhadrachalam, Andhra Pradesh, India.

Response Surface Methodology

The kraft pulping process for *C. equisetifolia* was optimized by using RSM with a fully randomized factorial design (2^3 central composite designs). Three parameters including, alkali charge (as Na_2O), process temperature, and time were selected as the independent variables and tested at five different levels (Table 1) to fit the second order response surface. The effect of alkali charge was measured in the range from 11.95 to 22%, temperature from 153 to 186.8 °C, and time from 39.6 to 140.4 min. The boundary limits of each variable were determined from preliminary experiments (Poonam and Dutt 2014). The influence of the above mentioned variables was measured by four response variables *i.e.*, screened yield, screening rejects, kappa number, and viscosity. Table 2 represents the experimental design of 20 runs with the experimental results of this study. The variables alkali charge, process temperature, and time are called natural variables, as they are expressed in their natural units of measurement. The transformation of these natural variables to coded variables was calculated by equations 1, 2, and 3,

$$X_1 = (A - A_o)/(A_o - A_{-1}) \quad (1)$$

$$X_2 = (T - T_o)/(T_o - T_{-1}) \quad (2)$$

$$X_3 = (t - t_o)/(t_o - t_{-1}) \quad (3)$$

where, X_1, X_2, X_3 are the coded form of the natural variables *A*, *T*, and *t*, respectively; *A*, *T*, and *t* are alkali charge (%), process temperature (°C), and time (minutes), respectively; 0 and -1 are the medium and low levels of variables.

The low and high levels of variables X_1, X_2, and X_3 are denoted by –1 and +1, respectively. The factorial and central points are placed at (±1, ±1, ±1) and (0, 0, 0), respectively, and axial points are positioned at (±1.68, 0, 0), (0, ±1.68, 0) and (0, 0, ±1.68). The 20 pulping experiments were conducted in triplicate and the average values were used.

The experimental data was fitted with a quadratic/polynomial equation (second degree). The model form is shown next,

$$Y = \beta_o + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{11} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3 + \beta_{11} {X_1}^2 + \beta_{22} {X_2}^2 + \beta_{33} {X_3}^2 \qquad (4)$$

where Y is the predicted response; β_0 is a constant; β_1, β_2, and β_3 are the linear coefficients; β_{11}, β_{22}, and β_{33} are the quadratic coefficients; and β_{12}, β_{13}, and β_{23} are the two-factor interaction coefficients. The significance of the regression coefficients was determined by the t-test and p-value. Analysis of variance (ANOVA) was used to determine the overall significance of the models. The contour plots were also plotted to locate the optimum conditions of the process.

Pulping Process

The pulping process was carried out in a WEVERK electrically heated rotary digester of 0.02 m^3 capacity with four bombs of 1-L each. Pulping conditions: liquor to wood ratio, sulphidity, anthraquinone (AQ), and Tween 20, were maintained at 3:1, 20%, 0.05%, and 0.1%, respectively, as established by the preceding test (Poonam and Dutt 2014). After completion of pulping process, the bombs were cooled by placing in water tank. After pulping, the liquor was separated and pulp was washed on a flat stationary screen of 300 mesh size wire bottom followed by isolation of screening rejects by disintegrating (2000 rpm for 3 min) and screening of pulp using vibratory flat screen (0.15 mm). The screened pulp was analyzed for screened pulp yield, kappa number (TAPPI T 236 cm-85), and viscosity (TAPPI T 230 om-04).

Preparation, Evaluation, and Microscopic Observations of Laboratory Handsheets

The unbleached pulp sample was beaten at different beating levels in a PFI mill (TAPPI T 200 sp-96). Laboratory handsheets of 60 g/m^2 were prepared on a British hand sheet former (TAPPI T 221 cm-99), and the sheets were air dried and tested for tensile index (TAPPI 494 om-01), tear index (TAPPI T 414 om-98), burst index (TAPPI T 403 om-97), and double fold (TAPPI T 423 cm-98).

RESULTS AND DISCUSSION

Response surface analysis

The pulping process was analyzed by multiple regressions through the least squares method, and the following models were obtained by substituting the experimental data into equation 4:

$$\text{Screened yield} = 50.81 + 1.48 * X_1 - 0.26 * X_2 0 - 1.10 * X_3 - 2.98 * {X_1}^2 - 2.39 * {X_2}^2 - 4.04 * {X_3}^2 + 1.03 * X_1 X_2 - 1.70 * X_1 X_3 + 1.10 * X_2 X_3 \qquad (5)$$

$$\text{Kappa number} = 19.03 - 7.18 * X_1 - 6.76 * X_2 - 4.22 * X_3 + 2.05 * {X_1}^2 + 3.45 * {X_2}^2 + 2.40 * {X_3}^2 - 1.41 * X_1X_2 + 2.29 * X_1X_3 - 0.09 * X_2X_3 \quad (6)$$

$$\text{Viscosity} = 993.62 + 14.61 * X_1 - 15.61 * X_2 - 21.38 * X_3 - 52.85 * {X_1}^2 - 101.14 * {X_2}^2 - 29.53 * {X_3}^2 + 32.95 * X_1X_2 - 10.15 * X_1X_3 + 27.32 * X_2X_3 \quad (7)$$

Table 3 represents the regression analysis of Eqs. 5 to 7 and the significance of each coefficient (main effects, quadratic, and interaction between factors) has been determined by student's t-test and p-value. The coefficient showing the larger the magnitude of t-test and smaller the p-value, was considered as more significant. The goodness of fit of all three models was examined by determination coefficient (R^2). All three values of R^2 for equations 5 to 7 were found to be near to 1 indicating a close agreement between the experimental results and the values predicted by the model equations. The values of adjusted determination coefficient (R^2_{adj}) were also high enough to indicate the significance of the models. The values of predicted R^2 were in reasonable agreement with the values of adjusted determination coefficient (R^2_{adj}) indicating a good adjustment between the experimental and predicted values of the responses. The p-values of lack of fit for all three models were more than 0.1 (non-significant), which indicated that all the models were significant. The regression equations were statistically significant at the 0.95 ($p<0.05$) confidence level. ANOVA evaluates the significance of model equations, and a value of $p>F$ below 0.05 indicates that the model terms are significant. Overall, the models were significant and able to predict the response variables. The interaction effects of operating variables *i.e.*, active alkali, maximum temperature and process time during kraft pulping of *Casuarina* was studied by plotting Contour plot against any two independent variables, while keeping another variable at its central (0) level.

Table 1. Variables and their Levels for Central Composite Experimental Design

Independent variables	Coded levels of variables				
	-α	-1	0	+1	+α
Active alkali charge, *A* (%)	11.95	14	17	20	22.05
Temperature, *T* (°C)	153.18	160	170	180	186.82
Time, *t* (min)	39.55	60	90	120	140.45

Rejects

The undigested material, remaining after pulping process, is known as rejects. Since, the screened pulp used for paper making process is free from rejects, the rejects content have not been considered for statistical analysis.

Contour Plots of Screened Pulp Yield, Kappa Number, and Pulp Viscosity

Figure 1 shows the contour plots of response variables as a function of two independent variables while keeping the third variable at a constant level. Figure 1a shows that there were many combinations of active alkali and temperature that produced the average screened pulp yield of >50%. The screened pulp yield reached a maximum (50.9%) at about alkali level of 0.04 (17.1%) and temperature level of –0.13 (168.7°C). Starting from the above mentioned maximum points, the screened pulp yield decreases by stepping in any direction.

Table 2. Central Composite Design and the Corresponding Responses

Run	Pulping variables						Response variables			
	Coded			Real						
	X_1	X_2	X_3	A (%)	T (°C)	t (min)	Screened yield (%)	Rejects (%)	Kappa number	Viscosity (cm^3/gm)
1	1	1	1	20.0	180.0	120.0	41.7	1.10	10.8	835.5
2	-1	-1	1	14.0	160.0	120.0	41.2	3.31	35.2	795.8
3	0	0	-1.68	17.0	170.0	39.6	40.5	1.80	34.1	945.5
4	1	-1	-1	20.0	160.0	60.0	46.8	2.80	29.5	870.4
5	0	0	0	17.0	170.0	90.0	52.2	0.10	17.9	1002.4
6	0	0	0	17.0	170.0	90.0	51.1	0.20	20.4	985.6
7	1	-1	1	20.0	160.0	120.0	38.7	1.60	25.4	745.5
8	0	-1.68	0	17.0	153.2	90.0	43.2	3.50	40.1	738.4
9	0	1.68	0	17.0	186.8	90.0	44.1	1.10	16.8	688.5
10	1	1	-1	20.0	180.0	60.0	45.4	0.52	11.9	847.6
11	0	0	0	17.0	170.0	90.0	49.8	0.09	18.2	978.6
12	0	0	0	17.0	170.0	90.0	52.5	0.08	18.0	995.5
13	-1	-1	-1	14.0	160.0	60.0	42.5	4.10	45.1	876.6
14	0	0	1.68	17.0	170.0	140.5	37.5	1.60	16.9	886.5
15	1.68	0	0	22.1	170.0	90.0	44.5	0.91	13.8	864.6
16	-1	1	1	14.0	180.0	120.0	40.1	2.50	22.9	750.5
17	0	0	0	17.0	170.0	90.0	48.8	0.15	20.1	987.6
18	-1	1	-1	14.0	180.0	60.0	37.0	1.50	36.5	725.5
19	0	0	0	17.0	170.0	90.0	50.6	0.18	19.7	1010.0
20	-1.68	0	0	11.9	170.0	90.0	39.5	3.40	35.2	835.5

Cooking conditions: liquor to wood ratio = 3:1, sulphidity = 20%, AQ = 0.05%, Tween-20 = 0.1%, time from room temperature to 105±2 °C = 45 min, time from 105 °C to maximum temperature = 45 min

Table 3. Regression Analysis for Screened Pulp Yield, Kappa Number, and Pulp Viscosity

Para-meters	Screened yield			Kappa number			Viscosity		
	CE	t-ratio	p-value	CE	t-ratio	p-value	CE	t-ratio	p-value
constant	50.81			19.03			993.62		
X_1	1.48	4.53	0.001	-7.18	-15.95	0.000	14.61	4.38	0.001
X_2	-0.26	-0.78	0.452	-6.76	-15.01	0.000	-15.61	-4.68	0.001
X_3	-1.10	-3.38	0.007	-4.22	-9.37	0.000	-21.38	-6.41	0.000
X_1^2	-2.98	-9.37	0.000	2.05	4.68	0.001	-52.85	-16.27	0.000
X_2^2	-2.39	-7.54	0.000	3.45	7.86	0.000	-101.14	-31.13	0.000
X_3^2	-4.04	-12.71	0.000	2.40	5.48	0.000	-29.53	-9.09	0.000
X_1*X_2	1.03	2.40	0.037	-1.41	-2.40	0.037	32.95	7.56	0.000
X_1*X_3	-1.70	-3.99	0.003	2.29	3.89	0.003	-10.15	-2.33	0.042
X_2*X_3	1.10	2.58	0.027	-0.09	-0.15	0.885	27.32	6.27	0.000
R^2	0.9697			0.9856			0.9927		
R^2 (pred)	0.8979			0.9069			0.9643		
R^2 (adj)	0.9424			0.9727			0.9860		

As shown by the plot, the screened pulp yield was found maximum in the vicinity of centre levels of both the variables (alkali charge and temperature). This is most likely due to the poor delignification and high content of screening rejects, which lead to a reduction in screened pulp yield at low temperature and alkali charge.

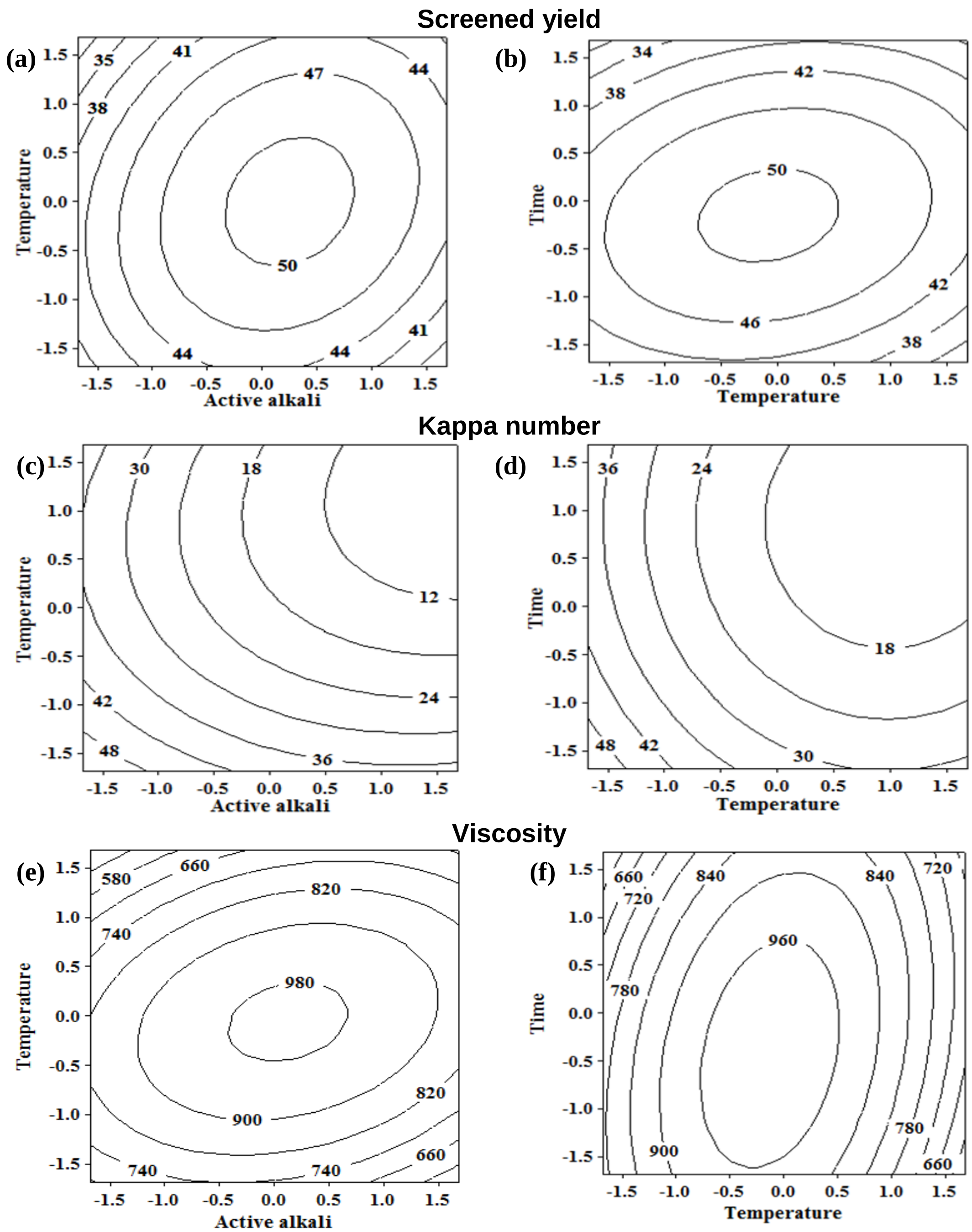

Fig. 1. Contour plots of screened yield as a relationship of (a) alkali charge (active alkali as Na_2O) and maximum temperature, at a constant cooking time of 90 min; (b) maximum temperature and cooking time, at a constant alkali charge of 17%. Contour plots of the kappa number as a relationship of (c) alkali charge (active alkali as Na_2O) and maximum temperature, at a constant cooking time of 90 min (d) maximum temperature and cooking time, at a constant alkali charge of 17%. Contour plots of the pulp viscosity as a relationship of (e) alkali charge (active alkali as Na_2O) and maximum temperature, at a constant cooking time of 90 min; (f) maximum temperature and cooking time, at a constant alkali charge of 17%.

On the contrary, high temperature and alkali charge may cause a severe dissolution of cellulose and hemicelluloses, and hence, the screened pulp yield decreases. Similarly, Wanrosli *et al*. (2004) reported a decrease in screened pulp yield in combination of very high and very low alkali levels or temperatures.

Similar to Fig. 1a, Fig. 1b shows the low screened pulp yield, predicted at very high and very low levels of process time and temperature. An increase in time of pulping up to a level of about 0 (90 min), initially increases the screened pulp yield, and beyond that level the screened pulp yield starts decreasing sharply, which may be due to degradation of the carbohydrates because of prolonged cooking (Jiang 1994). Moreover, at the time levels of >0.5 (>105 min), the contour lines are almost parallel to the temperature axis, indicates the variation in temperature at a fixed time and essentially does not affect the screened pulp yield in that region. The results reveal that the optimum values for the screened pulp yield are found in the close vicinity of the intermediate levels for all the three variables; active alkali, temperature and process time. If delignification is carried out at these conditions; the maximum screened pulp yield is obtained due to less dissolution of polysaccharides. It is, therefore, advisable to operate the process under intermediate operating conditions in order to obtain the maximum yield.

Figure 1c indicates a falling ridge of pulp kappa number with respect to alkali charge and temperature. This plot also indicates that the kappa number decreases sharply at high temperature with increasing the alkali charge, while decrease in kappa number is not to a great extent at lower temperature. At a fix central level of alkali charge, 0 (17% as Na_2O), the kappa number decreases rapidly from 40 to 19, with increasing temperature from level –1.68 (153 °C) to 0 (170 °C). This means pulping temperature plays an important role between these two levels. The solubilisation of lignin in this region may be related to bulk delignification phase of pulping. Bulk delignification took place in the temperature range of 150 to 170 °C (Chakar and Ragauskas 2004). On the other hand, in the region >170 °C, while keeping the alkali charge at central level, 0 (17% as Na_2O), the decrease in kappa number is insignificant, indicating the slow solubilisation of residual lignin (residual delignification). The residual lignin is resistant to delignification because it is chemically linked to carbohydrates (Iversen and Wannstrom 1986). Moreover, at the high levels of alkali (>17%) and temperature (>170 °C), the contour lines become almost parallel with the respective axis indicating that the solubilisation of lignin from the wood chips gets slow down. This also indicates that the bulk delignification is up to the above-mentioned transition points and it is not economical to continue the delignification beyond those optimum levels of active alkali (17%) and maximum temperature (170 °C).

The effect of variations in temperature and process time on kappa number is shown in Fig. 1-d. In the lower left region of the contour plot, the kappa number is reduced sharply from 53 to 19, with increasing the level of both temperature and process time from –1.68 to 0. However, at the higher levels of process time >0.5 (105 min), the contour lines are approximately parallel to the time axis indicating that variation in time at a fixed temperature does not affect kappa number and temperature plays a more important role in promoting delignification. Also, at higher temperatures, the contour lines are more widely spaced, indicating a decrease in kappa number with a slower rate (residual delignification). It is evidenced from the results that there are many combinations of alkali charge, temperature, and process time in the vicinity of their intermediate levels that can produce the desirable value of response (kappa number < 20) without decreasing the screened yield. These results are in close agreement with study conducted by Lal *et al.* (2010), who reported 16% alkali charge, 165 °C temperature, 90 min time and 20% sulphidity as the optimum conditions for the kraft pulping of *Anthocephalus cadamba*.

Figure 1e shows that there are many combinations of active alkali and temperature in the experimental range that produce the average pulp viscosity of >900. The pulp viscosity reaches maximum at about central level of all of three variables (active alkali, temperature and process time), as also shown in Table 2. With increasing temperature from its central level, the pulp viscosity starts decreasing which corresponds to the depolymerisation of cellulose. Similarly, the pulping at higher levels of alkali dose resulted in lower pulp viscosity, which may be due to cellulose chain scission reactions caused by alkaline hydrolysis.

The contour in Figure 1f indicates that the pulp viscosity initially increases with increasing temperature and then follows a decline trend. The lowest viscosity was observed for run 9, which was performed at medium concentration of alkali, high temperature, and medium time. In this case, the high temperature may cause degradation of cellulose resulting into the low viscosity of pulp obtained. This is clear from the results that the optimum viscosity occurs when alkali charge, temperature, and process time are at medium levels.

Validation

The response optimizer module in Minitab helps to identify the combination of independent variable settings that jointly optimize a single response or a set of responses. The best optimum conditions determined by the response optimizer were X_1 = 0.3, X_2 = 0.02, and X_3 = –0.26, which correspond to an actual alkali charge of 17.9%, temperature of 170.2 °C, and process time of 82.1 min, respectively. The experiments have been performed under the aforementioned optimized conditions and the predicted values for screened yield (51.1%), kappa number (17.9), and viscosity (997.1 cm^3/g) were quite close to the experimental values, *i.e*. 52.4%, 18.1, and 974.5 cm^3/g for the corresponding response variables. The closeness of predicted values to the experimental values further proves the validity of the models.

Mechanical Strength Properties of Paper

The mechanical strength properties *i.e.*, tensile index, burst index and double fold number of kraft pulp of *C. equisetifolia* prepared under the optimized conditions were calculated and plotted at different beating levels varying from 15 to 50 °SR, as shown in Fig. 2. The fibre length of kraft pulp was also calculated. The fibre length was found as 0.74 mm, which is quite comparable with other hardwoods. Tensile index, burst index, and double fold number increased up to a beating level of 45 °SR, except tear index. Tear index first increased up to a beating level of 30 °SR and then decreased. In a weakly bonded sheet (lower Schopper Reigler), more fibres are pulled out than break in the tear zone. Hence, the tearing resistance is controlled mainly by the number of bonds that break along the length of fibres (Seth and Page 1988). Therefore, tearing resistance depends strongly on the fibre length. Conversely, in a well bonded sheet (higher Schopper Reigler), more fibres break that pull out in tear zone (Seth and Page 1988). The °SR of pulp fibres shows the bonding ability, which increases gradually with pulp beating/refining and creates hydrogen bonds between fibres due to increased specific surface area and volume as a result of gradual beating (Norell *et al.* 1999; Hubbe and Heitmann 2007). Therefore, the strength properties like tensile index, burst index, and double fold numbers, *etc*. governed by hydrogen bonding, get improved. On the other hand, fibre length decreases slightly with increasing °SR, which may contribute to decreasing tear index beyond 30 °SR. Therefore, the beating level of 45 °SR was found optimum as tensile index, burst index and double fold numbers are increased up to this level and then start to decrease while tear index also exhibit the acceptable value at 45 °SR .

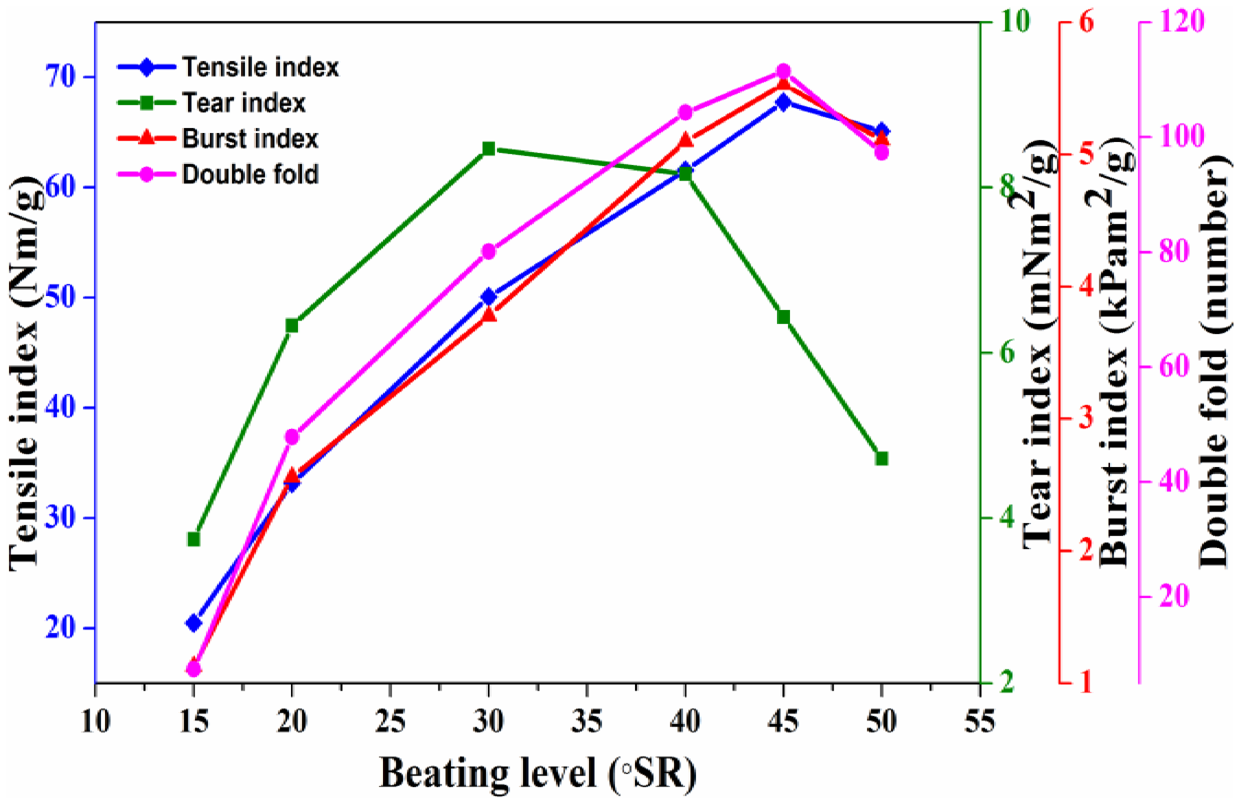

Fig. 2. Mechanical strength properties of optimized kraft pulp of *C. equisetifolia* at different beating levels (°SR).

CONCLUSIONS

1. The alkali charge, temperature, and cooking time had a strong influence on kraft pulping process.
2. The setting up of variables in the vicinity of their intermediate levels was the more suitable conditions to obtain acceptable responses.
3. Under the optimal conditions *i.e.*, alkali charge of 17.9%, temperature of 170.2 °C, and process time of 82.1 min, the predicted responses agreed very well with the experimental data, and confirmed the fitness and applicability of the models.
4. CCD and regression analysis methods were effective in determining the optimized alkali charge, temperature and process time for pulping of *Casuarina*.
5. Therefore, it is concluded that the dearth of short wood fibers can be addressed to some extent by fast growing wood species like *C. equisetifolia* through social forestry.

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