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Influence of Operating Variables on Soda Pulping of *Musa paradisiaca* Mid-Rib

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Abstract: The effect of time, temperature, liquor to solid ratio and concentration of cooking liquor on the pulp yield and residual lignin of *Musa paradisiaca* mid-rib was studied. The mid-rib chips were delignified in a laboratory pulp wood digester under the soda pulping process. Experimental values of the cooking variables were fitted into a second-order polynomial regression equation which reproduced the pulp yield and residual lignin content with error less than 2 and 4%, respectively. Pulping at high temperature for a short cooking with low to medium soda concentration makes the best compromise for high pulp yields and low residual lignin contents. Ratios of the dissolved lignin to the weight losses were relatively constant irrespective of the duration of cooking.

Key words: *Musa paradisiaca* mid-rib, soda pulping, pulp yield, residual lignin

INTRODUCTION

Pulp and paper industries are economically important in developing and developed countries alike because their products are consumed nearly in all facets of human endeavours. The consumption of paper per capital does not only correlate with the gross domestic product but also with the standard of welfare and education (Diesen, 1998; Wardle, 1995).

In Nigeria, one of the problems in pulp and paper industries is lack of adequate supply of long fiber pulpwood for paper production (RMRDC, 1996). The great value of the forest resources and the increasing demand for paper and paper board in Nigeria call for an overall plan for the utilization of these resources and for the development of plantation of *species* most suitable for the various sectors of the forest industry.

Apart from raphia palms proposed by Odeyemi (1995), there exist potentials to produce long fiber pulp (even at small-scale basis) from certain annuals. Osadare (1995) suggested the use of bast fibers like kenaf (*Hibiscus cannabinus*), roselle (*Hibiscus sabdariffa*) and plantain (*Musa paradisiaca*) as a way of alleviating the present long fiber shortage. Towards this end, the pulp and paper characteristics of some Nigerian grown *Musa* cultivars were examined (Oluwadare, 1998; Omotoso and Ogunsile, 2000). The results revealed that the cultivars are potential sources of long fiber pulp. The average fiber length compared favourable well with those obtained from soft wood species. The lignin contents were desirably low, while the cellulose content was a reflection of an

average but tolerable pulp yields. Similar results including pulping studies were also reported in the literature but not much has been done on modeling of pulping parameters (Sankia *et al.*, 1997; Cordeiro *et al.*, 2004; Kalpana *et al.*, 2005).

Kinetic modeling based on mathematical design have been used as a way of optimizing operating condition in pulping processes (Tjeerdsma *et al.*, 1994). However, this model becomes too complex when more than two independent variables are involved. A more recent approach is the use of central composite factorial design (Aknazarova and Kafarov, 1982). The factor design is preferred to kinetic modeling because of its simplicity especially when severally variables are involved. It allows the development of empirical models involving several independent variables in order to identify patterns of variation in the dependent variables of various pulping processes as applied to diverse vegetable materials (Vacquez *et al.*, 1995; Vega *et al.*, 1997; Gilarianz *et al.*, 1999; Jimenez *et al.*, 2000).

In the present study, a 2nd order factorial design was used to evaluate the effects of operating variables, namely, temperature, time, liquor to solid ratio and concentration of cooking liquors on the pulp yields and residual lignin content of a Nigerian grown *Musa paradisiaca* under the soda pulping process.

MATERIALS AND METHODS

Musa paradisiaca plants were collected at the Department of Agronomy of the University of Ibadan.

The study was conducted between 2002 and 2004. The part of the plant considered was the mid-rib which has high pulp yield. The composition of the sun-dried sample was determined to be 53.1% cellulose, 12.4% lignin and 6.9% ash. The alcohol-benzene extractive, cold water solubles, hot water solubles and 1% NaOH solubles were 8.2, 6.4, 9.8 and 24.9%, respectively. The fiber was 4.0mm long, 31.7 μm wide and 13.2 μm thick (Ogunsile, 2005)

Pulping: The mid-rib chips were digested in a 10-litre electrically heated stainless steel digester design after the method of Grant (1961). The plant chips were charged into the digester with the required amount of chemical solution at Liquor to Solid (LS) ratios of 7:1 and 10:1. The digester was heated to the operating temperatures (150 and 170°C), which was then maintained throughout the experiment for the different cooking times (30-150 min). At the end of each cooking scheme, the pulp was washed several times with water and dried at 102°C in the oven in order to determine the yield. The pulps were analyzed for Kappa number as described in TAPPI T236 cm-85 1993. The Residual Klason Lignin (RKL) was estimated from the relationship:

$$\text{RKL} = \text{Kappa number} * 0.13 \text{ (TAPPI, 1993)}$$

The second-order factorial design: The central composite factorial design was employed to evaluate and quantify the effect of the operational variables on the pulp yields and Residual Klason Lignin (RKL). The effect of the variables were quantified more precisely by choosing part of the experimental results and grouping them to form a first order full factorial design, with variables at two levels (2⁴). The factorial design gives expressions that relate the effect of each independent variable and their interactions on the dependent variables. Using this design, some of the experimental data were fitted to a first order polynomial regression equation as implemented in the MATHCAD 2001 Professional statistical package. Individual and second order interaction influences over the response surface of the independent variables were evaluated (Aknazarova and Kafarov, 1982).

The mathematical model was:

$$Y = a_0 + a_1x_1 + a_2x_2 + a_3x_3 + a_4x_4 + a_{12} x_1x_2 + a_{13}x_1x_3 + a_{14} x_1x_4 + a_{23} x_2x_3 + a_{24}x_2x_4 \quad (1)$$

The response variable Y represents the pulp yield and Residual Klason Lignin (RKL). The independent variables X₁, X₂, X₃ and X₄ correspond to liquor to solid ratio, time, temperature and concentration of cooking liquor, respectively. The ranges of values for each

independent variable were: LS, 7:1 to 10:1, time, 30-150 min, temperature, 150-170°C and concentration, 6-8% NaOH solution.

The values of the independent variables were normalized from -1 to +1 by using the equation:

$$X_n = 2(X - \bar{X}) / (X_{\max} - X_{\min}) \quad (2)$$

Where:

X_n is the normalized value of LS, time, temperature and concentration,

X is the absolute experimental value of the variable.

\bar{X} is the mean of all the experimental values for the variable in question

X_{max} and X_{min} are the maximum and minimum value, respectively of such a variable.

RESULTS AND DISCUSSION

The experimental design (2⁴ factorial designs) together with the pulp yield and the residual klason lignin are presented in Table 1. The coefficients of the model equations and the statistical parameters establishing their validity are summarized in Table 2. The correlation graphs between experimental and calculated yield and Residual Klason Lignin (RKL) content are shown in Fig. 1a and b respectively, while the ratio of the dissolved lignin to weight loses are estimated in Table 3.

Experiments 1-16 of Table 1 allowed the calculation of different parameters in the regression equations a_i and a_{ij}. These were subsequently subjected to a t-test to check their significance at 90 to 95% confidence level using the experimental error estimated from the replication at the central point of the design, that is, experiments 17, 18 and 19 of Table 1. The central point of the design corresponds to the following reaction conditions:

Liquor to solid ratio, LS = 8.5,
Time = 90 min,
Temperature = 160°C
Concentration = 7% soda

All normalized independent variables for the central points of the design are zero.

The dependent variables (pulp yield and Residual Klason Lignin (RKL)) were related to the independent variables through the following equations:

$$\text{Yield} = 37.62 - 2.5x_1 - 2.94x_2 - 4.28x_3 - 1.96x_4 + 0.65x_1x_2 - 0.69x_1x_3 + 0.8x_1x_4 - 0.93x_3x_4 \quad (3)$$

$$\text{RKL} = 3.10 - 0.48x_1 - 0.45x_2 - 0.76x_3 - 0.35x_4 + 0.48x_1x_3 + 0.25x_1x_4 \quad (4)$$

Table 1: Experimental design and result for yield and residual lignin

Experiment	LS (x ₁)	Time (x ₂)	Temperature (x ₃)	Concentration (x ₄)	Yield (%)	Lignin (%)
1	-1	-1	-1	-1	49.1	6.23
2	1	-1	-1	-1	42.4	3.21
3	-1	1	-1	-1	41.8	5.27
4	1	1	-1	-1	38.2	2.75
5	-1	-1	1	-1	43.0	3.20
6	1	-1	1	-1	34.2	2.91
7	-1	1	1	-1	37.4	2.17
8	1	1	1	-1	30.1	2.19
9	-1	-1	-1	1	44.5	4.44
10	1	-1	-1	1	42.4	3.49
11	-1	1	-1	1	39.2	3.47
12	1	1	-1	1	37.1	2.30
13	-1	-1	1	1	38.0	2.59
14	1	-1	1	1	30.4	2.62
15	-1	1	1	1	27.5	1.57
16	1	1	1	1	25.7	1.83
17	0	0	0	0	38.7	2.93
18	0	0	0	0	38.2	2.96
19	0	0	0	0	36.8	2.76

Table 2: Significant regression parameters

	Yield	Residual Klason Lignin (RKL)
a ₀	37.620	3.100
a ₁	-2.500	-0.480
a ₂	-2.940	-0.450
a ₃	-4.280	-0.760
a ₄	-1.960	-0.350
a ₁₂	0.650	(-0.051)
a ₁₃	-0.690	0.480
a ₁₄	0.800	0.250
a ₂₃	(-0.18)	(-0.001)
a ₂₄	(-0.29)	(-0.05)
a ₃₄	-0.930	(-0.120)

The non-significant parameters at 0.1 level are in parenthesis

Table 3: Ratio of the dissolved lignin to weight losses for the mid-ribpulp

Cooking liquor	Time (min)/ Temp. (°C)	Time (min)				
		30	60	90	120	150
6% soda	150	0.15	0.15	0.14	0.14	0.14
	160	0.16	0.15	0.16	0.15	0.15
	170	0.16	0.16	0.15	0.16	0.15
8% soda	150	0.15	0.16	0.14	0.15	0.15
	160	0.15	0.15	0.15	0.15	0.15
	170	0.15	0.14	0.14	0.14	0.13

Values calculated from the respective polynomial equations above were plotted with the experimental results for the different response variables as shown from Fig. 1a and b. The results showed very high correlation between the experimental values and those predicted by the models with errors less than 2% for the pulp yield and 4% for the Residual Klason Lignin (RKL).

Pulp yield: Equation 3 allows the estimation of the variation of the yield with changes in each independent variable over the range considered while the other three remain constant. Non-significant parameters were dropped to obtain simpler equations. The equation showed that there were significant interactions between LS and time, temperature and concentration and also

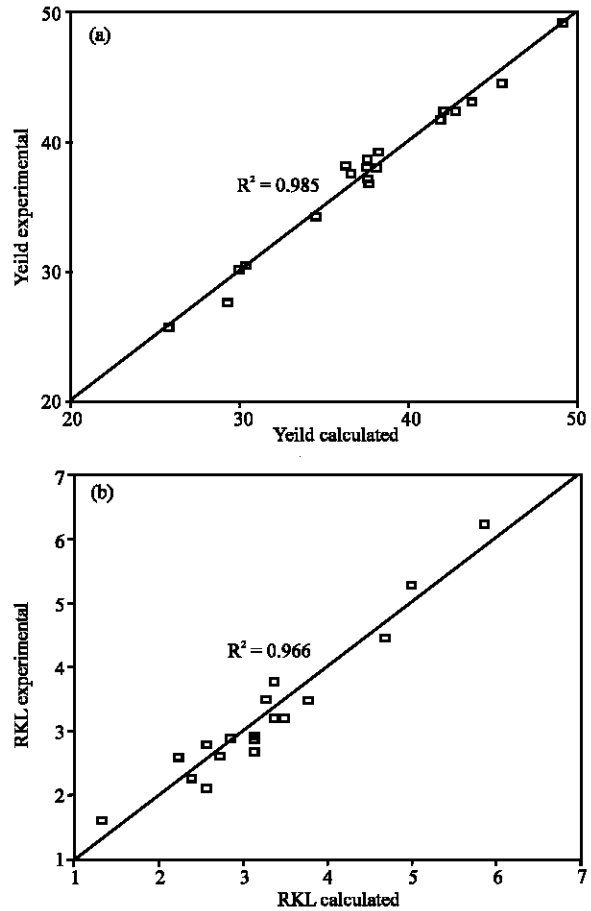


Fig. 1: Correlation graph between experimental and calculated values of (a) pulp yield and (b) Residual Klason Lignin (RKL)

between concentration and temperature. The lowest yield (25.8%) was obtained at large values of the four process

variables (i.e., +1 for all). If yield similar to chemical pulp is desired, then all the process variables must be at normalized values of +1. However, this lowest yield can be raised from 25.8 to 37.6% by using a lower concentration of cooking liquor (-1 normalized value). The use of a lower concentration of cooking liquor would also result in some chemical savings in addition to increasing the pulp yield. The greatest and smallest variations in the lowest pulp yield were caused by changes in temperature (11.8 units) and LS (3.48 units) respectively while the effect of time (4.58 units) and concentration (4.18 units) lie in between. The highest obtainable yield was 49.13%, which occurs at low values of the process variables (-1 for all). The maximum and minimum variations in the highest pulp yield were caused by changes in time (a change of 7.18 units) and concentration (a change of 3.7 units), respectively. The effect of LS (6.52 units) and temperature (5.32 units) lies in between the previous two.

Delignification: The minimum lignin content of the soda pulp according to Eq. (4) was obtained at high values of cooking time, temperature and concentration and a low value of the LS. Under this condition, the maximum variation in the minimum RKL value is caused by a change in temperature (2.48 units) while the minimum is caused by a change in LS (0.5 units). Changes in time (0.9 units) and concentration (0.7 units) lie in between. Pulping at high temperatures for a short cooking time with low to medium concentration of pulping liquor would give the best compromise for both pulp yield and Residual Klason Lignin (RKL) content.

Ratio of dissolved lignin to weight losses: The ratio of the dissolved lignin to the weight losses as presented in Table 3 can be used to estimate the quantity of lignin present in the pulp provided the pulp yield is known. The results showed that the values are relatively constant irrespective of the duration or time of cooking. The average ratio of the dissolved lignin to the weight loss was 0.14. The constant values seem to suggest that solubilization and delignification take place at the same rate.

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