

Multivariate Optimization of Soda Pulping of *Musa paradisiaca* Pseudostem using Response Surface Analysis

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Abstract

This study evaluated the effects of sodium hydroxide concentration, cooking temperature, and cooking time on the soda pulping of *Musa paradisiaca* pseudostem using a three-factor Box–Behnken design and quadratic response-surface models. The reported experimental matrix was first checked for consistency with the stated design; the sodium hydroxide center level was corrected from 10% to 14%, and two apparent transcription errors in the design matrix were corrected to 140 °C for Run 6 and 180 min for Run 7 so that the matrix conforms to the stated 3-level Box–Behnken structure. Reanalysis of the supplied response data gave quadratic-model R^2 values of 0.9876 for pulp yield and 0.9923 for kappa number, with adjusted R^2 values of 0.9717 and 0.9824, respectively. Sodium hydroxide concentration, temperature, and time were significant linear terms for both responses ($p < 0.001$), while several quadratic terms were significant. However, lack-of-fit tests were significant for both models ($p = 0.0015$ for pulp yield and $p = 0.0003$ for kappa number), indicating that the fitted quadratic equations should not be treated as validated predictive models. A model-based compromise point within the studied region was approximately 13.76% NaOH, 139.04 °C, and 124.60 min, with predicted yield of 54.11% and kappa number of 17.35; this point requires experimental confirmation before being described as an optimum. The results nevertheless demonstrate the strong influence of pulping severity on delignification and pulp retention and support further controlled optimization of banana pseudostem as a non-wood fiber resource.

Keywords: Musa Paradisiaca, Soda Pulping, Response Surface Methodology, Box–Behnken Design, Banana Pseudostem, Pulp Yield, Kappa Number

1. INTRODUCTION

The global pulp and paper industry has traditionally relied on wood as the principal raw material for pulp production. Increasing pressure on forest resources, together with environmental concerns and growing demand for fiber, has stimulated research into non-wood lignocellulosic materials. Agricultural residues are attractive because they are renewable, widely available, relatively inexpensive, biodegradable, and potentially suitable for fiber production.

Banana and plantain pseudostems are generated in large quantities after fruit harvesting. Although commonly treated as agricultural waste, these materials contain cellulose and hemicellulose together with comparatively modest lignin levels, making them potential feedstocks for pulp production. Cordeiro *et al.* (2004) reported favorable chemical composition and pulping characteristics for banana pseudostems, while Ververis *et al.* (2004) demonstrated the suitability of several agricultural residues for paper production.

Soda pulping uses sodium hydroxide to promote lignin removal and fiber separation. Process severity is governed by variables such as alkali concentration, temperature, and residence time. Increasing severity can improve delignification but may also increase carbohydrate dissolution and cellulose degradation, thereby reducing pulp yield. An appropriate balance between lignin removal and carbohydrate retention is therefore necessary.

Response Surface Methodology (RSM) provides a systematic way to evaluate several process variables simultaneously and to estimate their main, quadratic, and interaction effects. Previous studies have applied RSM and related approaches to banana-stem extraction and pulping. Mohamad and Jai (2022) used RSM for NaOH–EDTA extraction, while Bedru *et al.* (2023) optimized microwave-assisted soda pulping of banana pseudostem. However, the conventional soda-pulping response surface of *Musa paradisiaca* pseudostem requires further verification using a statistically coherent experimental design.

The objective of this study was therefore to evaluate the effects of NaOH concentration, cooking temperature, and cooking time on pulp yield and kappa number using a Box–Behnken design, to refit and assess quadratic response-surface models, and to identify a model-based compromise operating region. Because model adequacy is essential for RSM optimization, lack-of-fit testing and coefficient-level significance were included in the revised analysis.

2. EXPERIMENTAL

2.1 Raw material preparation

Fresh *Musa paradisiaca* pseudostems were collected from local banana farms immediately after fruit harvesting. The pseudostems were washed with distilled water, chopped into approximately 2–3 cm pieces, air-dried at room temperature for seven days, and then oven-dried at 60 °C to constant mass. The dried material was milled and screened to obtain relatively uniform particles. Analytical-grade sodium hydroxide pellets and distilled water were used for preparation of the cooking liquor.

2.2 Experimental design and pulping procedure

A three-factor, three-level Box–Behnken design was used. The factors were NaOH concentration (10–18%), cooking temperature (120–160 °C), and cooking time (60–180 min). For consistency with a centered three-level design, the center level of NaOH was 14%. The intended Box–Behnken matrix contains 12 edge points and five replicated center points. Run 6 was reported as 40 °C and Run 7 as 80 min; these values are inconsistent with the stated design and with the corresponding factor pattern. They were treated as transcription errors and corrected to 140 °C and 180 min, respectively, for the statistical reanalysis.

Table 1: Independent variables and experimental levels

Variable	Symbol	Low (−1)	Center (0)	High (+1)
NaOH concentration (%)	X_1	10	14	18
Temperature (°C)	X_2	120	140	160
Cooking time (min)	X_3	60	120	180

For each run, 50 g oven-dried pseudostem was charged into a stainless-steel laboratory digester. Cooking liquor was added at a liquor-to-solid ratio of 10:1. The digester was heated to the specified temperature and held for the designated time. After cooking, the material was cooled, filtered through muslin cloth, and washed with distilled water to approximately neutral pH. The pulp was mechanically disintegrated and screened to remove uncooked particles and shives, then oven-dried at 60 °C to constant mass.

2.3 Response measurements

Pulp yield was calculated as the mass of oven-dried pulp divided by the initial oven-dried raw-material mass, expressed as a percentage. Kappa number was determined using a standard TAPPI-based procedure as an index of residual lignin. Lower kappa numbers indicate greater delignification. The statistical analysis was performed by fitting second-order polynomial models to the supplied response data using coded factor values. The general model was $Y = \beta + \sum \beta_i X_i + \sum \beta_{ii} X_i^2 + \sum \beta_{ij} X_i X_j$. Model significance, individual coefficient significance, coefficient of determination, adjusted R^2 , residual error, and lack of fit were evaluated at $\alpha = 0.05$.

3. RESULTS AND DISCUSSION

Table 2: Box–Behnken data

Run	NaOH (%)	Temperature (°C)	Time (min)	Pulp yield (%)	Kappa number
1	10	120	120	58.4	29.8
2	18	120	120	49.2	18.7
3	10	160	120	52.5	21.3
4	18	160	120	41.6	12.5
5	10	140	60	60.1	31.5
6	18	140	60	50.8	20.6
7	10	140	180	54.7	23.9
8	18	140	180	43.5	13.2
9	14	120	60	57.8	27.1
10	14	160	60	50.2	18.9
11	14	120	180	55.1	22.5
12	14	160	180	44.3	11.8
13	14	140	120	53.6	17.2

14	14	140	120	53.8	17.1
15	14	140	120	54.1	16.9
16	14	140	120	53.7	17.0
17	14	140	120	53.9	17.1

Pulp yield ranged from 41.6 to 60.1%, while kappa number ranged from 11.8 to 31.5. The highest observed yield was 60.1% at 10% NaOH, 140 °C, and 60 min. The lowest observed kappa number was 11.8 at 14% NaOH, 160 °C, and 180 min. These observations show the expected trade-off between fiber retention and delignification: mild conditions retained more pulp but left more residual lignin, whereas severe conditions reduced kappa number at the expense of yield.

The response pattern is consistent with the established chemistry of alkaline pulping. Greater NaOH concentration, temperature, and residence time increase the accessibility and dissolution of lignin and hemicellulose, but prolonged exposure can also increase carbohydrate loss. Similar trends have been reported for banana pseudostem and other agricultural residues (Cordeiro *et al.*, 2004; Finell and Nilsson, 2004; Bedru *et al.*, 2023).

3.1 Quadratic response-surface models

The refitted quadratic model for pulp yield was:

$$\text{Yield (\%)} = 53.820 - 5.075X_1 - 3.988X_2 - 2.663X_3 - 1.485X_1^2 - 1.910X_2^2 - 0.060X_3^2 - 0.425X_1X_2 - 0.475X_1X_3 - 0.800X_2X_3$$

The corresponding model for kappa number was:

$$\text{Kappa} = 17.060 - 5.188X_1 - 4.200X_2 - 3.338X_3 + 2.870X_1^2 + 0.645X_2^2 + 2.370X_3^2 + 0.575X_1X_2 + 0.050X_1X_3 - 0.625X_2X_3$$

Here, X_1 , X_2 , and X_3 are coded NaOH concentration, temperature, and cooking time, respectively. The overall quadratic models were statistically significant ($p < 0.001$ for both responses).

Table 3: Results for quadratic model

Response	R ²	Adjusted R ²	RMSE	Model F	Model p
Pulp yield	0.9876	0.9717	0.8677	65.05	<0.001
Kappa number	0.9923	0.9824	0.7540	90.37	<0.001

The high R^2 values indicate that the fitted equations reproduce most of the variation in the supplied observations. However, R^2 alone is insufficient to establish model adequacy, particularly for an RSM model with replicated center points. The lack-of-fit results below are therefore critical to interpretation.

Table 4: Coefficient-level statistical output

Term	Yield coefficient	SE	t	p-value	Kappa coefficient	SE	t	p-value
Intercept	53.820	0.388	138.69	<0.001	17.060	0.337	50.59	<0.001

X_1	-5.075	0.307	-16.5	<0.001	-5.188	0.267	-19.4	<0.001
			4				6	
X_2	-3.988	0.307	-13.0	<0.001	-4.200	0.267	-15.7	<0.001
			0				6	
X_3	-2.663	0.307	-8.68	<0.001	-3.338	0.267	-12.5	<0.001
							2	
X_1^2	-1.485	0.423	-3.51	0.0098	2.870	0.367	7.81	0.0001
X_2^2	-1.910	0.423	-4.52	0.0027	0.645	0.367	1.76	0.1226
X_3^2	-0.060	0.423	-0.14	0.8912	2.370	0.367	6.45	0.0004
X_1X_2	-0.425	0.434	-0.98	0.3599	0.575	0.377	1.53	0.1710
X_1X_3	-0.475	0.434	-1.09	0.3098	0.050	0.377	0.13	0.8982
X_2X_3	-0.800	0.434	-1.84	0.1077	-0.625	0.377	-1.66	0.1413

For pulp yield, all three linear terms and the X_1^2 and X_2^2 terms were significant. For kappa number, all three linear terms and X_1^2 and X_3^2 were significant. None of the three two-factor interaction terms reached the 0.05 significance level for either response. Thus, the previous statement that the NaOH $\times$ temperature interaction was statistically significant is not supported by the reanalysis of the supplied data and has been removed.

Table 5: ANOVA and lack-of-fit assessment

Source	Yield SS	df	F	p	Kappa SS	df	F	p
Model	420.3789	9	62.07	<0.001	513.6358	9	100.55	<0.001
Residual	5.2705	7	—	—	3.9795	7	—	—
Pure error	0.1480	4	—	—	0.0520	4	—	—
Lack of fit	5.1225	3	46.15	0.0015	3.9275	3	100.71	0.0003
Total	425.6494	16	—	—	517.6153	16	—	—

Although the overall models are highly significant, both lack-of-fit tests are significant. This means that the unexplained variation is greater than would be expected from pure experimental error alone and that the quadratic equations do not adequately represent the response surfaces across the entire design region. Consequently, the earlier claims that the models were fully adequate and that the predicted optimum was experimentally validated are not justified by the supplied data. Additional experiments or a re-examination of the raw measurements are required before the model can be used for definitive process optimization.

3.2 Response trends and comparison with previous work

The negative linear coefficients for all three factors in the yield model show that increasing NaOH concentration, temperature, or time generally reduced pulp yield over the investigated region. The most influential linear factor was NaOH concentration, followed by temperature and time. This behavior is

consistent with increased dissolution of hemicellulose and partial carbohydrate degradation under more severe alkaline conditions. Cordeiro *et al.* (2004) and Mohamad and Jai (2022) similarly reported reduced carbohydrate recovery at higher alkaline severity.

For kappa number, all three linear coefficients were negative, confirming that increasing process severity reduced residual lignin. The positive X_1^2 and X_3^2 terms indicate curvature in the NaOH and time responses, whereas the temperature quadratic term was not significant. The lowest observed kappa number, 11.8, occurred at 14% NaOH, 160 °C, and 180 min. This agrees qualitatively with reports that increased alkaline severity improves delignification of non-wood fibers (Goswami *et al.*, 2008; Bedru *et al.*, 2023).

The observed yield of 60.1% is within the broad range reported for banana-derived feedstocks, although direct comparison among studies should be made cautiously because raw-material species, chemical systems, liquor ratios, pretreatments, and yield definitions differ. Mohamad and Jai (2022) reported higher cellulose recovery using a NaOH–EDTA system, while Bedru *et al.* (2023) reported a lower optimized yield for microwave-assisted soda pulping. These differences emphasize that the present results should be interpreted as specific to the stated conventional soda-pulping conditions.

3.3 Model-based compromise operating point

Because the fitted models show significant lack of fit, a numerical optimum should not be presented as a validated optimum. Nevertheless, an exploratory equal-weight desirability calculation, maximizing pulp yield while minimizing kappa number within the studied factor ranges, gives a compromise point of approximately 13.76% NaOH, 139.04 °C, and 124.60 min. The corresponding model predictions are 54.11% pulp yield and kappa number 17.35. This point lies inside the experimental region and represents a model-based compromise rather than a confirmed optimum.

Table 6: Exploratory compromise value for some parameters

Parameter	Exploratory compromise value
NaOH concentration (%)	13.76
Temperature (°C)	139.04
Cooking time (min)	124.60
Predicted pulp yield (%)	54.11
Predicted kappa number	17.35

The compromise point should be tested experimentally, preferably with independent replicate runs at or near the proposed conditions. A confirmation experiment is necessary to determine whether the observed responses agree with the model predictions and to establish whether the model can support optimization after refinement.

4. CONCLUSION

The supplied experimental data demonstrate clear effects of NaOH concentration, cooking temperature, and cooking time on the soda pulping of *Musa paradisiaca* pseudostem. Increasing process severity generally reduced pulp yield while improving delignification, as reflected by lower kappa number. Reanalysis of the corrected Box–Behnken matrix produced statistically significant quadratic models with high R^2 values (0.9876 for yield and 0.9923 for kappa number).

However, significant lack of fit was detected for both responses. Therefore, the quadratic equations should be regarded as exploratory descriptions of the supplied data rather than validated predictive optimization models. The previously reported optimum of 13.5% NaOH, 145 °C, and 105 min and its associated predictions have consequently been replaced by an exploratory compromise point of approximately 13.76% NaOH, 139.04 °C, and 124.60 min, predicted to give 54.11% yield and kappa number 17.35. These predictions require independent experimental confirmation.

Additional confirmation experiments around the proposed compromise region, together with complete diagnostic assessment, would strengthen the statistical basis for optimization. Future work should also examine bleaching response, paper strength, fiber morphology, and the effect of pulping additives such as anthraquinone or EDTA.

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