Optimization of soda cooking for cellulose production from sugarcane bagasse

Nhan T. T. Dang¹, Hong K. T. Tang^{1*}, & Dien Q. Le²

¹Faculty of Forestry, Nong Lam University, Ho Chi Minh City, Vietnam ²School of Chemistry and Life Sciences, Ha Noi University of Science and Technology, Ha Noi, Vietnam

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ABSTRACT

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*Corresponding author

Tang Thi Kim Hong Email: tangkimhong@hcmuaf.edu.vn

Sugarcane bagasse, an agricultural residue, is a fibrous material containing cellulose as its main component, produced in large quantities worldwide. The aim of this work was to investigate the production of unbleached cellulose pulp from sugarcane bagasse using the soda cooking process with sodium hydroxide as the alkaline reagent. The cooking conditions were investigated with dosages of sodium hydroxide from 20% to 25%, temperatures from 150°C to 170°C, and cooking time from 75 to 105 min. The response surface methodology was used to study the effect of pulping variables on observed parameters. The results indicated that the optimal cooking conditions achieved the highest yield of 46.4% w/w and the lowest kappa number of 20.6 at a sodium hydroxide dosage of 23%, a temperature of 155°C, and a cooking time of 93 min. Further analysis of paper produced from the investigated pulp, refined at varying revolutions (0 to 3000 rpm), revealed that optimal strength properties were achieved at a refining level of 31°SR, equivalent to 2500 rpm. At this refining level, handsheets with a basis weight of 85 gsm exhibited a tensile strength of 2 kN/m, a burst strength of 2.7 kgf/cm², and a ring crush strength of 6.9 kgf. These findings confirm that the mechanical properties of the refined pulp meet the strength requirements of commercially recycled kraft paper, demonstrating its suitability for similar applications.

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1. Introduction

Non-wood plant fibers, such as sugarcane bagasse, represent a promising and sustainable alternative for pulp and paper production. Sugarcane bagasse, a fibrous byproduct of the sugar extraction process, is characterized by a high cellulose content and can effectively substitute wood fibers in pulp production. Cultivated primarily in tropical and subtropical regions, sugarcane production globally amounts to approximately 2 billion tons annually (Okibe et al., 2023). Asia is the predominant producer, contributing nearly 60 million metric tons, with Vietnam producing about 11.5 million metric tons in 2023 (Nguyen, 2024). During the sugar extraction process, approximately 30% of each ton of sugarcane is converted into bagasse (Katakojwala & Mohan, 2022; Narisetty et al., 2022). Comprising fibrous residues dense in polymeric substances, sugarcane bagasse is recognized as a significant source of secondgeneration lignocellulosic biomass (Konde et al., 2021). As reported by Kumar et al. (2021), sugarcane bagasse contains substantial quantities of cellulose (32 - 45%), hemicellulose (20 - 32%), lignin (17 - 32%), ash (1.0 - 9.0%), and various extractives.

In the pulp and paper industry, wood is traditionally the primary raw material. The extensive consumption of wood, however, poses environmental challenges, including deforestation and associated greenhouse gas emissions. The utilization of alternative agricultural residues, such as rice straw, wheat straw, cotton linters, corn stalks, and notably sugarcane bagasse, can alleviate these environmental impacts. Among these alternatives, sugarcane bagasse is particularly favored for pulp production due to its superior fiber length (El-Sayed et al., 2022).

The growing demand for paper and paperbased packaging in Vietnam has intensified deforestation to meet increasing pulp requirements. Exploring and utilizing alternative raw materials, such as sugarcane bagasse, presents potential environmental benefits, including a reduction in deforestation, and adds economic value to this byproduct. Various chemical pulping methods can be applied to process sugarcane bagasse, including sulfate (kraft), sulfite, soda, and organosolv processes. While sulfate and sulfite processes are widely used, they involve sulfated chemical agents that contribute to environmental pollution through the release of sulfur compounds. Organosolv processes, which use organic solvents as delignifying agents and do not release sulfur compounds, have shown effectiveness only at the pilot scale. Consequently, this study employs the soda pulping process a traditional, sulfurfree method selected to minimize environmental impact. The objective of this study was to evaluate and optimize pulping conditions to produce nanocellulose from sugarcane bagasse for use in food packaging applications.

2. Materials and Methods

2.1. Materials and chemicals

Sugarcane bagasse (SCB) was collected from the An Khe Sugar Mill, Quang Ngai Sugar Joint Stock Company. The raw material underwent a pretreatment procedure that included cutting it into approximately 2 - 3 cm in length, cleaning, air drying, and sorting. For chemical composition analysis, the air-dried material was ground to a particle size of 0.25 to 0.50 mm. Main chemicals used for determining SCB chemical compositions and for cooking SCB were purchased from Sigma-Aldrich. Other chemicals were originally sourced from China.

2.2. Experimental design

The Central Composite Design (CCD) within the framework of Response Surface Methodology (RSM) was employed to assess the effects of three independent variables- X_1 : NaOH dosage (% w/w of oven-dry SCB), X_2 : cooking temperature (°C), and X_3 : cooking time (min)-on the properties of cellulose pulp. The response variables examined were cellulose pulp yield and kappa number. A 17-run CCD experimental design was utilized,

incorporating both axial and center points, and was conducted using Minitab (version 21.2). The variable ranges are provided in Table 1, with NaOH dosage varying from 20-25% w/w, cooking temperature from 150 -170°C, and cooking time from 75 - 105 min. Each independent variable was coded at five levels, as shown in Table 1. The soda cooking conditions generated through RSM with CCD are detailed in Table 2.

Table 1.	The range	and levels	of the	variables	in soda	a cooking stage
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Factor	Variable	Range and level of coded and actual values				
		-α	-1	0	+1	α
X ₁	Dosage of NaOH (%)	18	20	22.5	25	27
X_2	Cooking temperature (°C)	143	150	160	170	177
X ₃	Cooking time (mins)	65	75	90	105	115

Run	NaOH dosage (%)	Cooking temperature (°C)	Cooking time (min)
1	20	150	75
2	25	150	75
3	20	170	75
4	25	170	75
5	20	150	105
6	25	150	105
7	20	170	105
8	25	170	105
9	18	160	90
10	27	160	90
11	22.5	143	90
12	22.5	177	90
13	22.5	160	65
14	22.5	160	115
15	22.5	160	90
16	22.5	160	90
17	22.5	160	90

Table 2. The soda cooking schedules using central composite design

2.3. Cellulose production

Soda cooking was conducted in a laboratory rotating digester model Regmed AU/E-20. Soda cooking conditions for each experiment are detailed in Table 2, with a liquor-to-solid ratio of 7:1 (L/kg) for all experiments, using exclusively oven-dry SCB as the solid component. Following cooking, the unbleached cellulose pulp was filtered and washed thoroughly with distilled water.

2.4. Evaluation of chemical composition of SCB, cellulose pulp yield, and kappa number

The solubility and chemical composition of SCB were analyzed following TAPPI standards: TAPPI T 207 om-93 for hot and cold water solubility (TAPPI, 1993), TAPPI T 212 om-93 for 1% NaOH solubility (TAPPI, 1993), TAPPI T 17 wd-70 for cellulose content (TAPPI, 1970), TAPPI T 223 cm-23 for pentosan content (TAPPI, 2023), TAPPI T 222 om-88 for lignin content (TAPPI, 1988), and TAPPI T 211 om-93 for ash content (TAPPI, 1993).

The dry matter content of the washed pulp was determined according to ISO 638 to calculate cellulose pulp yield, kappa number, and for further testing (ISO, 2008). The kappa number was evaluated following the TAPPI T 236 om-13 standard (TAPPI, 2013).

2.5. Cellulose pulp refining and handsheet formation for analysis of cellulose pulp strength properties

The pulp at the optimum conditions of cooking was chosen for refining at different revolutions in PFI mill to study the effect of refining on pulp strength properties. The cooked pulp was refined at the revolutions of 0; 500; 1,000; 1,500;

2,000; 2,500; 3,000 rpm. After refining, the pulp drainability at different refining revolution was determined by measuring the Schopper Riegler degree (°SR). The refined cellulose pulps were made handsheets with a basis weight of 85 gsm were then prepared from the refined pulps using a sheet former. These handsheets were tested to evaluate tensile strength, burst strength, and ring crush strength, following TAPPI standard methods T 494, T 403 (TAPPI, 2022), and T 818 (TAPPI, 2018), respectively.

3. Results and Discussion

3.1. Chemical compositions of SCB

The main chemical components of SCB are presented in Table 3. Compared with the data reported by Kumar et al. (2021), the cellulose and lignin contents are relatively similar. As shown in Table 3, the cellulose content of SCB in this study is only 45.12%, which suggests that the cooked yield obtained is expected to be relatively low. The ash content is approximately 5%, which is significantly higher than that of wood and within the same range as that of nonwood plants (Alonso-Esteban et al., 2022). The presence of silica in SCB ash negatively impacts chemical recovery in the alkaline pulping process; therefore, this chemical component should be assessed when considering the valorization of this agricultural waste in papermaking.

Table 3 shows that SCB is soluble in the used solvents in the following decreasing order: 1% sodium hydroxide > hot water > cold water. This result indicates that SCB exhibits better solubility in alkaline conditions. Consequently, the soda process is selected as the pulping method for this study.

Colubility and common out	Results			
Solubility and component	This study	Kumar et al. (2021)		
Hot water solubility	5.01%			
Cold water solubility	4.37%			
1% NaOH solubility	35.65%			
Cellulose	45.12%	32 - 45%		
Pentosan	18.7%	20 - 32%		
Lignin	24.57%	17 - 32%		
Ash	5.49%	1.0 - 9.0%		

Table 3. Solubility and chemical composition of sugarcane bagasse (expressed as a percentage percent on a dry weight basis)

3.2. Effect of cooking conditions on yield and kappa number of cellulose pulp

The results of cooking are presented in Table 4, including cellulose pulp yield and the kappa number of the pulps. The models to predict each response variable for the samples produced by the soda cooking process were obtained using

Minitab 21.2. The final models for all variables are presented below as Equations (1) and (2). These equations are reduced models that include only significant terms, with a significance level or p-value of less than 0.05. The final equations, expressed in terms of the actual factors, are shown below:

 $Y_{Kappa number} = -104.388 - 0.7888x_1 + 2.1606x_2 - 0.515666667x_3 - 0.00694x_2^2 + 0.002648889x_3^2(1)$

 $Y_{v_{ield}}$ (%) = 89.68 - 1.0632 $x_1 - 0.0977 x_2 - 0.040133333 x_3$

Run	NaOH dosage	Cooking temperature	Cooking time	Cellulose yield	Kappa
	(%)	(°C)	(min)	(%)	number
1	20	150	75	49.8	24.5
2	25	150	75	44.2	19.6
3	20	170	75	47.5	22.4
4	25	170	75	43.5	19.3
5	20	150	105	49.5	22.8
6	25	150	105	44.0	19.3
7	20	170	105	47.2	21.8
8	25	170	105	43.0	19.0
9	18	160	90	50.1	25.0
10	27	160	90	40.1	17.5
11	22.5	143	90	49.1	20.2
12	22.5	177	90	44.8	17.5
13	22.5	160	65	48.6	24.0
14	22.5	160	115	44.5	21.0
15	22.5	160	90	46.7	20.9
16	22.5	160	90	46.8	20.9
17	22.5	160	90	46.0	20.9

Table 4. Soda cooking results

(2)

The models are verified for the adequacy of final equation by investigating values of multiple regressions (R^2), adjusted R^2 and ρ value of regression. In model (1), R^2 and adjusted R^2 are about 97.17% and 93.52%, respectively. In model (2) R^2 and adjusted R^2 are about 95.27% and 89.18%, respectively. The R^2 indicates how well the model explains the variation in the data, whereas the adjusted R^2 indicates how much R^2 overestimates the variance when another predictor is added in the model. The higher the adjusted R^2 , the better the goodness of fit.

Figures 1 to 6 and the corresponding data in Table 4 demonstrate that NaOH dosage, cooking temperature, and cooking time significantly affect both cellulose yield and kappa number. The effects of NaOH dosage and temperature on yield are shown in Figure 1 with surface plots (A) and contour plots (B). The results indicate that, at a constant temperature, an increase in NaOH dosage decreases the yield, while an increase in cooking temperature leads to a slight decrease in yield. This occurs because NaOH is used during cooking to remove lignin through reactions that degrade the lignin macromolecules before they can disrupt the cellulose chains.

The effects of NaOH dosage and cooking time on yield are illustrated in Figure 2 with surface plots (A) and contour plots (B). Increasing NaOH dosage up to 24% across a wide range of temperatures (140°C to 175°C) maintains a yield above 45%. However, higher cooking temperatures and longer cooking times decrease the yield (Figure 3). At low temperatures around 145°C and short reaction times around 70 min, the fibers are not completely separated after pulp washing.



Figure 1. The surface plots (A) and contour plots (B) of cooking yield as function of NaOH dosage and cooking temperature.



Figure 2. The surface plots (A) and contour plots (B) of cellulose yield as function of NaOH dosage and cooking time.



Figure 3. The surface plots (A) and contour plots (B) of cooking yield as function of cooking temperature and pulping time.

For the kappa number, a *P*-value of less than 0.05 indicates that all factors have significant effects on the kappa number, as shown in Figures 4, 5, and 6. Increasing NaOH dosage and cooking temperature reduces the kappa number. As discussed previously, higher NaOH dosage and cooking temperature reduce the lignin content in the cellulose pulp. When NaOH dosage was around 19%, the cooking temperature was below 165°C, and the cooking time was 90 min, the kappa number of the cellulose pulp remained above 24 (Figure 4), and shives were observed after washing under these conditions. Figure 6 shows that cooking at temperatures above 160°C with cooking times ranging from 70 to 120 min results in cellulose pulp with a lower kappa number.



Figure 4. The surface plots (A) and contour plots (B) of kappa number as function of NaOH dosage and cooking temperature.



Figure 5. The surface plots (A) and contour plots (B) of kappa number as function of NaOH dosage and cooking time.



Figure 6. The surface plots (A) and contour plots (B) of kappa number as function of temperature and time of cooking.

As shown in Figure 7, cooking with a NaOH dosage of 23% at 155°C for 93 min was identified as the optimal condition by Minitab 21.2,

resulting in the highest yield of 46.4% and the lowest kappa number of 20.6.



Figure 7. The cross-sectional surface meets the optimum point.

3.3. Effect of cellulose pulp refining on strength properties

The cellulose pulp obtained under optimal conditions was refined using a PFI mill at different revolutions: 0; 500; 1,000; 1,500; 2,000; 2,500 and 3,000 rpm. Cellulose refining is a promising approach to improve pulp quality by altering fiber characteristics (Gharehkhani et al., 2015). During PFI refining, mechanical treatment is primarily caused by the impulses



Figure 8. The relation between refining revolutions and °SR.

of the beating body bars, which predominantly induce internal fibrillation (Mandlez et al., 2022). The drainability of cellulose at different refining revolutions was assessed by measuring the Schopper-Riegler degree (°SR), and the results are shown in Figure 8. Increasing the refining revolutions resulted in a higher °SR and decreased cellulose drainability. The main reason for this result is the increase in fines produced during refining (Cole et al., 2008).



Figure 9. The relation between °SR and tensile strength of unbleached cellulose pulp from sugarcane bagasse.



Figure 10. The relation between °SR and burst strength of unbleached cellulose pulp from sugarcane bagasse.

The effects of refining on the strength properties of cellulose pulp, including tensile strength, burst strength, and ring crush strength, are illustrated in Figures 9, 10, and 11. The tensile strength of cellulose pulp may be influenced by various interacting factors, such as fiber length, fiber strength, coarseness, and specific bonding strength (Kerekes et al., 2021). Beyond a certain point, the limiting factor for strength is no longer fiber-to-fiber bonding but the strength of the individual fibers. Refining beyond this point begins to decrease strength properties. The results indicate that at a refining level of 31 °SR (corresponding to 2500 rpm), the strength properties of handsheets (basis weight 85 gsm) reached their maximum values, with a tensile strength of 2 kN/m, burst strength of 2.7 kgf/ cm², and ring crush strength of 6.9 kgf.

4. Conclusions

The soda cooking process has been successfully applied to produce unbleached cellulose pulp from sugarcane bagasse. Optimal conditions, determined using Minitab 21.2, were found to maximize yield and minimize kappa number: a sodium hydroxide dosage of



Figure 11. The relation between °SR and ring crush strength of unbleached cellulose pulp from sugarcane bagasse.

23% w/w, a cooking temperature of 155°C, and a cooking time of 93 min. Under these conditions, the cellulose pulp obtained a tensile strength of 2 kN/m, burst strength of 2.7 kgf/cm², and ring crush strength of 6.9 kgf. These properties are comparable to those of cellulose obtained from recycled commercial kraft paper.

Conflict of interest

The authors have no conflict of interest to declare.

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