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A green approach to sustainable cellulose production: Optimizing microwave-assisted sodium hydroxide delignification of durian rind

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ABSTRACT

Cellulose from durian rind can be further utilized, including as advanced materials for the manufacture of bioplastics, as raw materials for bioethanol, and as raw materials for the pulp and paper industry. However, lignin hinders cellulose utilization. Lignin binds strongly to cellulose and hemicellulose, so it is necessary to prepare cellulose using a microwave-assisted delignification method. This study aimed to examine the optimal conditions for preparing durian rind cellulose by varying delignification time and sodium hydroxite (NaOH) concentration to produce durian rind fiber with high cellulose content. This study used Response Surface Methodology optimization techniques with a Central Composite Design, with 2 independent variables: NaOH concentration and time. The response or dependent variables observed were yield, as well as cellulose , hemicellulose , and lignin contents. The results of the study showed that cellulose content will increase along with NaOH concentration and the length of delignification time of durian skin, hemicellulose content will increase along with NaOH concentration and will decrease with the addition of delignification time, and lignin content will decrease along with the increase in NaOH concentration and will increase with the addition of delignification time. The optimum solution formula given is at a NaOH concentration of 0.79 M and a time of 33.25 minutes. The results of this study provide an alternative, environmentally friendly method for obtaining optimal cellulose.

Keywords: cellulose, delignification, lignin, microwaves, sodium hydroxite.

INTRODUCTION

Durian rind is an agricultural waste that needs to be optimally utilized. Durian rind is very rich in cellulose (60.45%). In addition to cellulose, durian rinds contain hemicellulose, lignin, and ash (Anggoro et al., 2015). The high cellulose content in durian rind offers opportunities for further utilization, including as an advanced material for the manufacture of bioplastics, a raw material for bioethanol production, as well as a raw material for the pulp and paper industry. However, lignin hinders the utilization of cellulose. Lignin binds strongly to cellulose and hemicellulose, so cellulose must be prepared by a process called delignification.

Delignification aims to degrade lignin from cellulose. Mechanical, physical, chemical, semichemical, and biological treatments are used for delignification (Fernandes et al., 2023; Ishfaq Bhat et al., 2022; Leny Fitriah & Dwi Agustini, 2024; Li et al., 2020; Permata et al., 2021, 2024).

Various lignin extraction methods are discussed, including sulfite pulping, Kraft, Organosolv, ozonolysis, steam explosion, ionic liquids, supercritical CO_2 extraction, microwave and ultrasonic-assisted extraction, and biological methods (Schieppati et al., 2023). Industrially, the two most widely used methods of delignification are sulfite and alkaline. Physical and chemical treatments use microwaves and chemical solvents, such as sodium hydroxite (NaOH). Several studies have shown that microwave pretreatment can reduce lignin levels in lignocellulosic materials (Kohli et al., 2020).

The effectiveness of microwave radiation in pretreating lignocellulosic biomass was confirmed for the waste from the food industry, cereal straw, distillery stillages, and exotic plant biomass (Amini et al., 2018; Bundhoo, 2018; Germec et al., 2017; Mikulski et al., 2019). Delignification of rice straw was performed using 950-watt microwaves with 0.5 M NaOH solvent for 40 minutes, with a straw powder size of 100 mesh. This process resulted in a 42.32% increase in cellulose content and a 4.27% decrease in lignin content (Dehani et al., 2013), indicating the potential for similar optimization in the preparation of durian fruit cellulose. Delignification of water hyacinth using microwaves at 700 W for 30 minutes with 2 M NaOH solvent can increase cellulose from 56% to 68.27% and reduce lignin from 12.01% to 11.50%. In addition, delignification of empty oil palm bunches using microwaves with 0.5 M NaOH as the solvent for 25 minutes can produce fibers with a cellulose content of 72.59% (Permata, 2023).

Previous studies have shown that delignification is influenced by solvent concentration and duration. However, no study has examined the influence of these factors on durian rind delignification using microwave-assisted delignification with NaOH as a solvent. Furthermore, conventional delignification methods typically require prolonged heating and excessive chemical consumption, which contradicts the principles of green chemistry. To address these limitations, this study introduced a highly efficient microwave-assisted extraction approach specifically designed to optimize the synergistic effects of NaOH concentration and exposure time on the decomposition of the durian rind matrix. The novelty of this study lies not only in the precise process optimization through experimental design, but also in its holistic framework that links micro-level biopolymer processing with macro-level sustainability goals. By establishing an energy-efficient biorefinery pathway to convert biowaste into high-purity cellulose, this study provided a strategy that supports SDG 12 (Responsible Consumption and Production) and promoted the transition to a circular economy.

MATERIALS AND METHODS

Research design

This research used response surface methodology (RSM) optimization techniques to optimize the durian rind delignification process. Stages in the optimization technique include:

- Design (selecting response factors/components, setting lower and upper limits of factors/components, drafting experimental design, and conducting experiments where expertise is crucial).
- Response model analysis (establishing the mathematical model of the response, creating contour/3D graphs, and creating normality graphs of the response data).
- Optimization (determination of response criteria and determination of optimum value).
- Verification (testing optimum conditions and comparing values and predictions).

Cellulose preparation

The dried durian rind was reduced in size to ± 1 cm^2. A total of 20 g of durian rind was mixed with 200 mL NaOH (Sigma Aldrich). Durian rind powder and NaOH were placed in an Erlenmeyer flask, then covered with cotton and aluminum foil to prevent evaporation; the sample was homogenized. After mixing, the sample was placed in the microwave oven (Sharp R-21D0(S)-IN, 450 W input) at the medium-high power level (~70% output, ±315 W effective output). After that, fiber bleaching was performed with 2% H_2O_2 at a 1:15 w/v ratio. The fiber and solution were placed on a hotplate at 60 °C for 2 hours, then the fiber was separated from the solution. The bleached fiber was washed using distilled water until the pH was neutral. The resulting material was measured for yield, cellulose, hemicellulose, and lignin content. The resulting material was analyzed for yield and the contents of cellulose, hemicellulose, and lignin. Optimization treatment in the cellulose preparation process is shown in Table 1.

Analysis of lignin, cellulose, and hemicellulose content using the Chesson method

A total of 1 gram of dry sample (a) was dissolved in 150 mL of aquadest and heated at a temperature of 90–100 °C for 1 hour. After heating, the solution was filtered, and the residue was rinsed with 300 mL of hot water. Then, the residue

Table 1. Cellulose isolation research design

Name	Unit	Low	High	-alpha	+alpha
NaOH concentration	M	0.5	1.5	0.292893	1.70711
Time	Minutes	25	35	22.9289	37.0711

was dried in an oven (Memmert) at 105 °C until it reached a constant weight, and was weighed (b). The dry residue was further processed by adding 150 mL of 1 N H_2SO_4 (Sigma Aldrich). The mixture was heated to 90–100 °C for 1 hour. After heating, the residue was filtered and washed with 300 mL of aquadest. The residue was returned to the oven at 105°C to dry until it reached a stable weight, then weighed (c). Furthermore, the dried residue was dissolved in 10 ml of 72% H_2SO_4 solution and soaked at room temperature for 4 hours. Then 150 ml of 1 N H_2SO_4 solution was added, and the mixture was heated for 1 hour at 90–100 °C. The residue was filtered again and washed with 400 mL of H_2O (Sigma Aldrich). Then, the residue was dried in an oven at 105 °C until it reached a stable weight and was weighed (d). The last step was to burn the residue at 600 °C using a furnace for 4 hours, and the final weight (e) was weighed. The formula for testing cellulose and lignin content is as follows:

$$Hemicellulose\ content = \frac{b-c}{a} \times 100\% \quad (1)$$

$$Cellulose\ content = \frac{c-d}{a} \times 100\% \quad (2)$$

$$Lignin\ content = \frac{d-e}{a} \times 100\% \quad (3)$$

RESULTS AND DISCUSSION

The cellulose preparation formulation used a central composite design (CCD) experimental design with 2 independent variables (k=2): NaOH concentration (X1) and time (X2). The response or dependent variables observed were yield (Y1), cellulose content (Y2), hemicellulose content (Y3), and lignin content (Y4). There were 13 experiments, and 5 center points.

Effect of NaOH concentration and time on yield response

The Design-Expert 11.0 model for the yield response indicates it is not significant. The statistical results are presented in Table 2.

The program's determination of the mean model is based on the lowest Sequential Model Sum of Squares value. The yield range value is 69.325% - 76.675%. The mean model shows that the yield response for each combination of delignification process conditions is the same, around 73.03% (Fig. 1). The mean yield response equation is as follows:

$$Y = 73.03462$$

$$Y = \textit{yield response}\ (\%)$$

The analysis of variance showed that none of the factors significantly affected yield. Thus, the formulated factors do not significantly affect yield.

Effect of NaOH concentration and time on the cellulose content response

Based on the Design-Expert 11.0 analysis, the best model for the cellulose content response is the mean model, with an R^2 value of 0.9116. The statistical results are presented in Table 3. Equation means the cellulose content response:

$$Y = 8.07330 + 38.91089\,A + 2.69639\,B - 0.925000\,AB - 4.76200\,A^2 - 0.025120\,B^2$$

where: Y – response of cellulose content (%), A – effect of NaOH concentration (M), B – time effect (minutes), AB – effect of NaOH and time interaction,

The model equation above shows that cellulose content increases along with NaOH

Table 2. ANOVA for a linear model on yield response

Model	0.0000	0				
Residual	56.76	12	4.73			
Lack of Fit	27.95	8	3.49	0.4852	0.8218	not significant
Pure Error	28.81	4	7.20			
Cor Total	56.76	12				

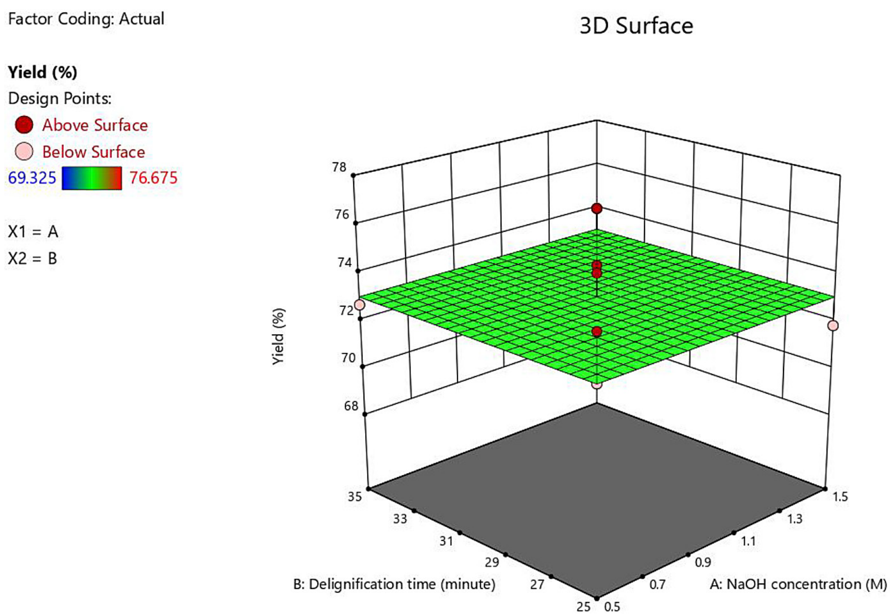

Figure 1. 3D response surface and contours of the effect of NaOH concentration and delignification time on the yield produced

Table 3. ANOVA for a linear model on the cellulose content response

Model	52.15	5	10.43	14.44	0.0014	significant
A-NaOH concentration	5.36	1	5.36	7.42	0.0296	
B-Delignification time	13.96	1	13.96	19.32	0.0032	
AB	21.39	1	21.39	29.61	0.0010	
A^2	9.86	1	9.86	13.65	0.0077	
B^2	2.74	1	2.74	3.80	0.0923	
Residual	5.06	7	0.7224			
Lack of Fit	2.55	3	0.8506	1.36	0.3751	not significant
Pure Error	2.50	4	0.6262			
Cor Total	57.21	12				

concentration and durian peel delignification duration. NaOH concentration is the most dominant factor. NaOH breaks down the β-O-4 and ester bonds in the Lignin-Carbohydrate Complex (LCC), thus dissolving lignin and exposing cellulose (Permata et al., 2021). The linear effect is very strong, but is limited by the quadratic effect of A^2. Likewise, delignification time has a positive, but weaker effect. Longer interaction times allow NaOH to react more completely in breaking down the lignin structure. However, if the time is too long (B^2), the cellulose content decreases (Fig. 2), indicating that both factors have an optimal point. After this point, adding NaOH or extending the time actually decreases the cellulose content. This phenomenon is caused by over-delignification, or cellulose degradation by excess NaOH over a long period.

The interaction between NaOH concentration and delignification time showed a negative coefficient. The combination of excessively high NaOH concentrations and long delignification times actually reduced cellulose content. This indicates that at high NaOH concentrations, increasing the time is ineffective and leads to saturation. The best process conditions obtained from the average model for the cellulose content response were 1 M NaOH concentration and 37 minutes (Fig. 3). This is consistent with the research on palm fiber delignification, which showed an increase in cellulose content at 30 minutes compared to 15 minutes (Sriana et al., 2021).

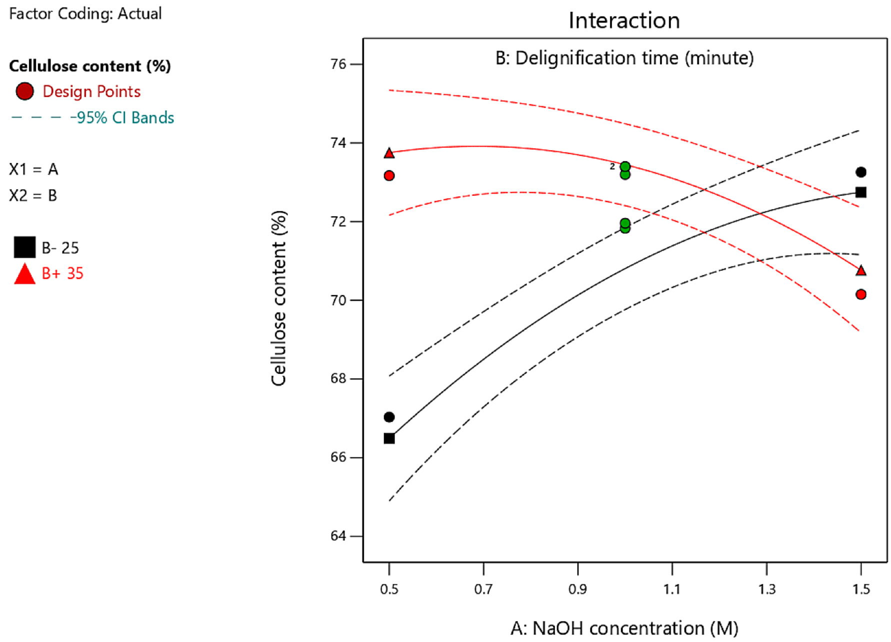

Figure 2. Interaction graph of the effect of NaOH concentration and delignification time on cellulose content

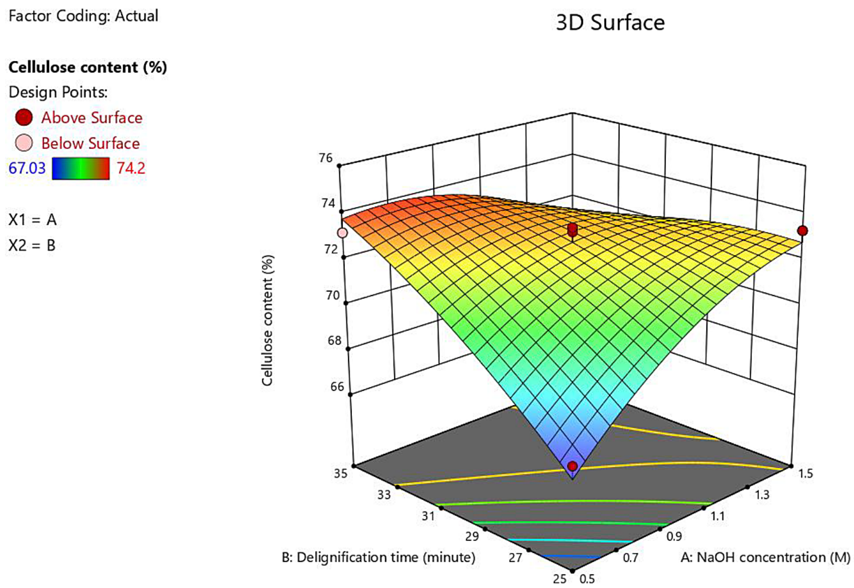

Figure 3. 3D surface response and contours of the effect of NaOH concentration and delignification time on cellulose content

Effect of NaOH concentration and time on hemicellulose content response

According to the Design-Expert 11.0 analysis, the quadratic model is the best fit for the hemicellulose content response (Table 4), with an R^2 of 0.9442. The linear equation for the hemicellulose content response is:

$$Y = 2.53963 - 0.438472\,A - 0.104939\,B + 0.039000\,AB - 0.248500\,A^2 + 0.000715\,B^2$$

where: Y – response of hemicellulose content (%), A – effect of NaOH concentration (M), B – time effect (minutes), AB – effect of NaOH and time interaction.

The mathematical equation shows that the response of hemicellulose content increases along with the interaction between NaOH concentration and delignification time, as shown in Figure 4. The increase in hemicellulose content due to the addition of NaOH may be due to the slower solubility

Table 4. ANOVA for a linear model on the hemicellulose content response

Model	0.2858	5	0.0572	23.70	0.0003	significant
A-NaOH concentration	0.1100	1	0.1100	45.62	0.0003	
B-Delignification time	0.1062	1	0.1062	44.02	0.0003	
AB	0.0380	1	0.0380	15.77	0.0054	
A^2	0.0268	1	0.0268	11.13	0.0125	
B^2	0.0022	1	0.0022	0.9217	0.3690	
Residual	0.0169	7	0.0024			
Lack of Fit	0.0138	3	0.0046	5.97	0.0585	not significant
Pure Error	0.0031	4	0.0008			
Cor Total	0.3027	12				

of carbohydrates compared to oxidation or reactions with other compounds, leading to a decrease in carbohydrate content in the medium. When carbohydrate solubility decreases, oxidation reactions form more complex compounds, which eventually accumulate, thus increasing the hemicellulose content (Mahdy et al., 2014). In Figure 5, the yellow color indicates a high hemicellulose content. According to the linear model, the optimal process conditions to reduce the hemicellulose content response are a NaOH concentration of 0.29 M and a time of 30 minutes (Figure 5).

Effect of NaOH concentration and time on the lignin content response

Based on the Design-Expert 11.0 analysis, the best model for lignin content is the quadratic model (Table 5), with an R^2 value of 0.9521. Linear equation of the lignin content response:

$$Y = 70.16062 - 20.52200\,A - 2.57475\,B + \\ + 0.332000\,AB + 4.62800\,A^2 + 0.038680\,B^2$$

where: Y – response of lignin content (%), A – effect of NaOH concentration (M), B – time effect (minutes), AB – effect of NaOH and time interaction.

The resulting equation shows that lignin content decreases with increasing NaOH concentration and increases along with delignification time, though the increase is not significant, as shown in Figure 6. This aligns with the delignification process of pineapple leaves, which states that the higher the NaOH concentration, the more lignin is degraded (Sumiati et al., 2023). The

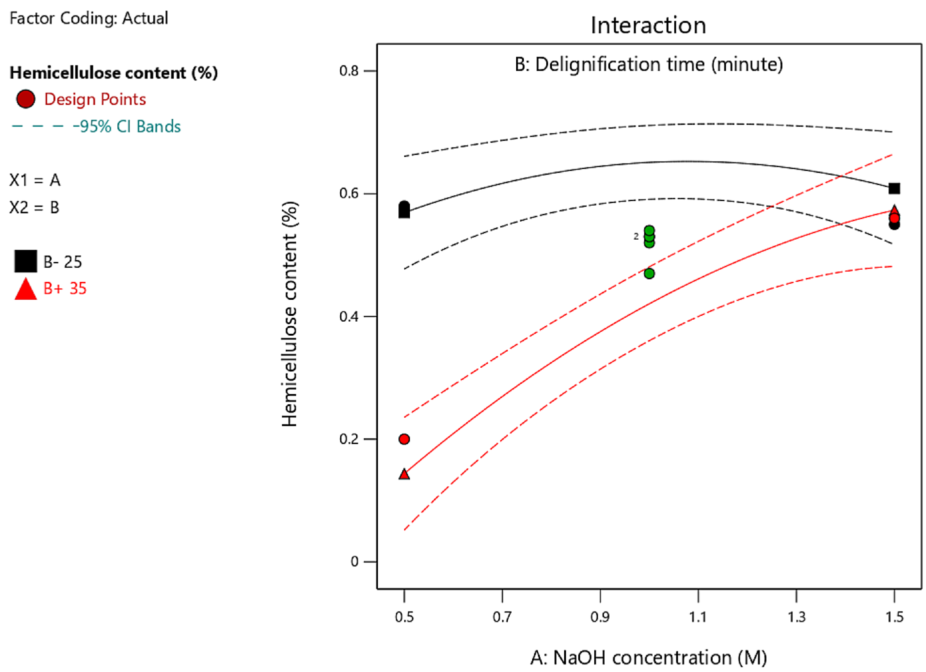

Figure 4. Effect of interaction between NaOH concentration and delignification time on hemicellulose content

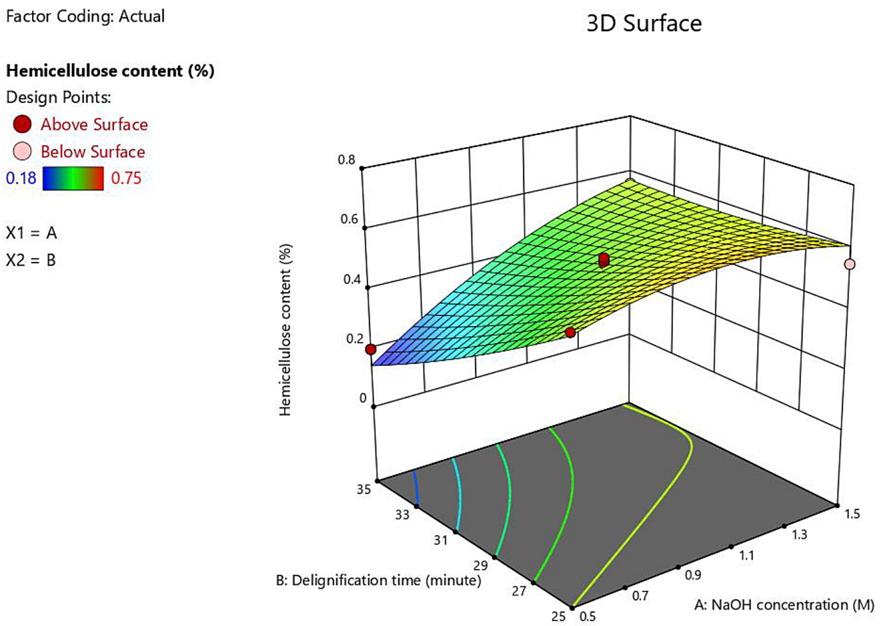

Figure 5. 3D surface response and contours of the effect of NaOH concentration and delignification time on hemicellulose content

Table 5. ANOVA for a linear model on the lignin content response

Model	21.41	5	4.28	27.81	0.0002	significant
A-NaOH concentration	3.41	1	3.41	22.15	0.0022	
B-Delignification time	1.22	1	1.22	7.91	0.0261	
AB	2.76	1	2.76	17.89	0.0039	
A^2	9.31	1	9.31	60.47	0.0001	
B^2	6.50	1	6.50	42.24	0.0003	
Residual	1.08	7	0.1540			
Lack of Fit	0.8365	3	0.2788	4.62	0.0868	not significant
Pure Error	0.2415	4	0.0604			
Cor Total	22.49	12				

more the chemicals are involved, the lower the yield and lignin content because chemical action degrades and solubilizes components of the lignocellulose, especially lignin and hemicelluloses (Bajpai, 2018). The mechanism of decreasing lignin content is consistent with that of increasing cellulose content. The decrease in lignin by alkali solution is mainly due to the lack of stable ester bonds between cellulose and lignin complexes. The released lignin then binds with alkali to form a water-soluble lignin-alkali complex (Fig. 7). This process involves several stages, beginning with the cleavage of phenolic bonds such as α-O-4, β-O-4, β-O-5, and β-β in lignin. Next, the non-phenolic β-O-4 bonds and carbon-carbon bonds in lignin are broken, and carbohydrates are also degraded (Mikulski & Kłosowski, 2022)by 57.68% in beech chips, and by 74.08% in wheat straw. After enzymatic hydrolysis was conducted, the highest concentration of glucose: 463.27 ± 11.25 mg glucose/g (hydrolysis yield 46.76 ± 1.14%. In addition, microwaves applied during delignification help break lignin into its water-soluble monomers. The use of microwaves involves the oscillatory motion of air dipoles, thereby stimulating the degradation of lignocellulosic biomass (Kohli et al., 2020). In addition, the bleaching process with H_2O_2 can also reduce lignin content in the fiber.

In this study, NaOH concentration and delignification time did not interact in the equation. The plot shown in Figure 4 provides information about the relationship between the variables. The color gradation on the graph determines the desired optimum point during the process. The best process condition obtained from the linear model for lignin

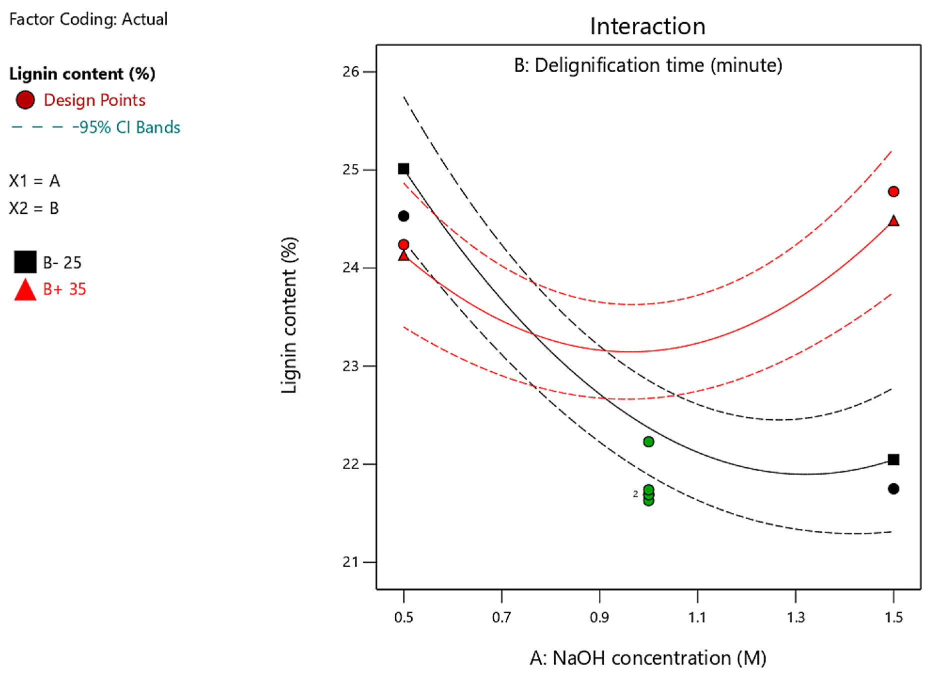

Figure 6. Effect of NaOH concentration (a) and delignification time (b) on lignin content

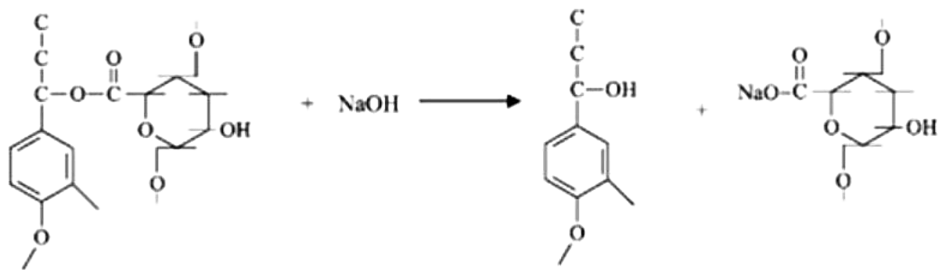

Figure 7. Mechanism of lignocellulose reacting with NaOH

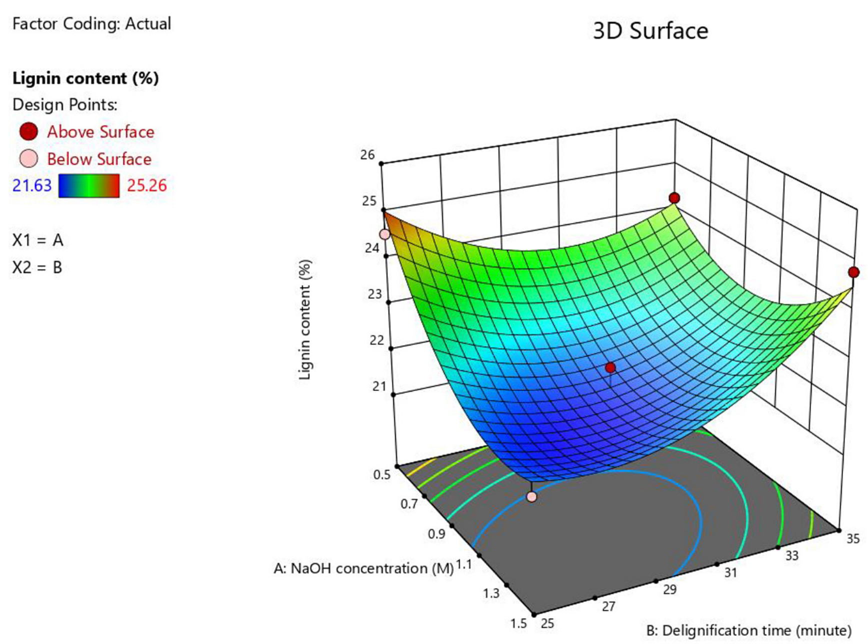

Figure 8. 3D surface response and contours of the effect of NaOH concentration and delignification time on lignin content

content was 1 M NaOH at 30 minutes (Fig. 8). The same was also observed in the siwalan fiber delignification process: the optimal time for lignin degradation was 30 minutes (Sriana et al., 2021).

Model validation

Optimization of the cellulose production process from durian peel was guided by two key parameters: NaOH concentration and time (minutes). Cellulose and lignin content were assigned the highest importance weight (5) because they are the primary indicators of delignification process quality. Hemicellulose content was given an importance weight of 4, while yield was given an importance weight of 3 to reflect its secondary role in process economics. The formula for durian peel cellulose production can reach optimal conditions when the desired response variable is close to one. The program provides a desirability value of 0.705, an important metric in this context. The optimal formula is 0.79 M NaOH for 33.25 minutes, producing a yield of 79.49%, cellulose content of 73.42%, hemicellulose content of 0.36%, and lignin content of 22.72%. This value differs from that of the durian rind that has not undergone delignification (cellulose content 49.9% and lignin content 29.21%). The overall response values met the predicted 95% confidence interval (CI) and 95% prediction interval (PI). These results indicate that the model provides good predictions for all responses studied in the RSM design.

Compared with other studies, this study isolates cellulose with a purity level that is not much different, and in some cases even higher (Table 6), while requiring fewer chemicals and less time, making this method more efficient and environmentally friendly. The microwave-assisted delignification process using NaOH solvent in this study inherently embodies green chemistry principles. Microwave-assisted pretreatment offers several advantages, including low operational costs, rapid processing, efficient volumetric heating, minimal use of solvent (NaOH), and a reduced likelihood of side reactions.

Four dimensions of excellence in this research, using a green technology approach, are as follows:

- Superior energy efficiency – while conventional heating transfers heat through the surface, microwaves generate heat directly within the material, reducing energy loss. Microwaves offer advantages in faster energy transfer, controlled heating, and shorter residence times.
- Renewable feedstock – this process offers an economical and environmentally friendly approach to reclaiming lignocellulosic biomass from agro-industrial activities.
- High selectivity and byproduct minimization – continuous innovation in process optimization and synergistic approaches collectively

Table 6. Comparison with other studies

Methods	Results	References	Deficiency
Delignification of pineapple rind using choline chloride and glycerol (1:9) with thermal treatment in an autoclave at 100 °C for 1 hour	65.54% cellulose, 16.09% hemicellulose, 8.05% lignin	(Yerizam, Ramayanti, et al., 2026)	Low cellulose content, long processing time
Delignification of durian skin using 6% NaOH with conventional heating at 100 °C for 105 min	81.31% cellulose and 10.01% lignin	(Yerizam, Yulianty, et al., 2026)	Long processing time, cellulose content is not much different
Ozonolysis of oil palm fruit mesocarp with a flow rate of 2 Lmin^{-1} for 15 minutes	cellulose content of 62.88% cellulose, 16.89% hemicellulose, and 9.53% lignin	(Asyeni et al., 2024)	Low cellulose content, high operating costs
Delignification of durian rind with acidic sodium chlorite and mercerization with 17.5% (w/v) sodium hydroxide (NaOH)	33.12 ± 0.108% cellulose	(Penjumras et al., 2014)	Low cellulose content
Delignification of durian rind with chemical treatment (HNO_3 3.5 wt.% and placed in the autoclave; refluxed with NaOH 2 wt.% at 70–80°C for 30 min; bleaching process using NaOCl 1.75 wt.% at 70–80 °C for 30 min)	Yield of up to 75%	(Lubis et al., 2020)	Excessive use of chemicals
Delignification of durian rind using $NaClO_4$ and alkalization process using NaOH	The cellulose obtained without the heating process has a yield of 47,95% and 42,7% that obtained with the heating process	(Lubis et al., 2019)	The yield obtained is lower
Delignification of durian rind with 1.75 wt.% $NaClO_2$ solution	Yield of cellulose from durian rinds was as high as 32.23%	(Xing et al., 2022)	The yield obtained is lower

emphasize the importance of microwaves as a key technology for sustainable extraction in the biorefinery and chemical industries. RSM-CCD modeling precisely defines operational safety zones.
- Experimental efficiency through statistical design – RSM-CCD with 13 runs replaces the wasteful full factorial approach, reflecting green chemistry, a process designed from the outset to minimize resource waste.

Overall, this study confirmed that microwave-assisted delignification using moderate NaOH concentrations optimized through RSM-CCD is a technically superior and environmentally responsible approach for producing cellulose from lignocellulosic biomass. This process complies with green chemistry principles, making it a strong candidate as a core technology in sustainable biorefinery schemes to be developed at pilot and industrial scales in the future.

CONCLUSIONS

The results showed that there are no factors that can significantly affect the yield; cellulose content will increase along with the amount of NaOH concentration and the length of time delignification of durian rind, hemicellulose content will increase along with NaOH concentration and will decrease with the addition of delignification time, and lignin content will decrease along with the increase in NaOH concentration and will increase with the addition of delignification time. The optimal formula solution is 0.79 M NaOH at 33.25 minutes. There was an increase in cellulose content from 49.9% to 73.17%, and lignin degradation was 17.15%.

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