EEET ECOLOGICAL ENGINEERING & ENVIRONMENTAL TECHNOLOGY

Ecological Engineering & Environmental Technology 2024, 25(10), 359–367
https://doi.org/10.12912/27197050/192038
ISSN 2299–8993, License CC-BY 4.0

Received: 2024.07.29
Accepted: 2024.08.15
Published: 2024.09.01

Optimization of Sugar Palm Starch Waste Delignification Process for Membran Production

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ABSTRACT

Sugar palm starch waste (SPSW) was biomass that produce from processing of sugar palm starch. SPSW contain high cellulose, hemicellulose, and lignin. Using lignocellulose from SPSW needs a pretreatment method to obtain high-purity cellulose and create an environmentally friendly method. The investigation focuses on determining the optimal NaOH concentration, temperature, and duration for hydrothermal-alkali treatment to remove hemicellulose and lignin, achieving high-purity cellulose. There is no further research regarding optimal conditions related to NaOH concentration, temperature, and duration for hydrothermal-alkali treatment to remove hemicellulose and lignin, achieving high-purity cellulose. This study aims to define optimal conditions for lignin and hemicellulose removal to obtain cellulose with high purity. The delignification process is carried out by hydrothermal and hydrothermal alkali activation. The hydrothermal activation removes hemicellulose, whereas the hydrothermal alkali activation removes lignin. In this study, Response surface methodology (RSM) was used to determine optimum conditions. The results were then structural and morphological identification using XRD, FTIR, and SEM. The optimization results obtained parameter values of 2% NaOH, temperature 120 °C for 120 minutes. The desirability value was 0.774, with a response value of 2.9 ± 1.7% for hemicellulose, 92.35 ± 1.06% for cellulose, and 2.45 ± 0.1% for lignin. FTIR results showed that lignin was absent during the delignification process. Meanwhile, in XRD, during the delignification process, the crystallization index (CI) decreases, and the crystallization size (CS) increases. SEM analysis results show that the morphology of the delignified SPSW becomes smoother after pretreatment, which indicates a decrease in hemicellulose and lignin content and a high level of cellulose purity. Further research is needed on other parameters, such as cellulose yield, energy consumption, and environmental impact. This research should also consider other potential pretreatment factors, such as pressure and catalysts in the hydrothermal process.

Keywords: sugar palm starch waste, hydrothermal alkali activation, delignification, lignin, cellulose.

INTRODUCTION

Sugar palm (*Arenga pinnata*) is one of Indonesia's most important commodities and production has increased annually. Sugar palm is primarily used for its sap and sugar industry (Ansar et al., 2021; Imraan et al., 2023), while palm steam is utilized for starch production (Ilyas et al., 2020). Processing palm steam for starch yields approximately 10%, with the remaining portion being SPSW. SPSW is commonly utilized for animal feed and as a planting

medium, but its full potential has yet to be fully realized. Moreover, the SPSW is high in lignocellulose. SPSW contains 43.71% cellulose, 32.20% hemicellulose, and 26.33% lignin (Aisyadea et al., 2023). The abundance of cellulose in SPSW makes it promising for membrane production. However, in its natural state, cellulose tends to bind to lignin, impeding cellulose isolation. Lignin is an adhesive that binds various lignocellulosic components together, rendering them insoluble in water (Keshav et al., 2023). Due to its association with cellulose microfibrils, lignin is a significant barrier to the enzymatic hydrolysis of lignocellulosic materials. Therefore, delignification is required to eliminate lignin and simplify cellulose hydrolysis.

Dayatmo and HS (2015) show that the delignification of palm starch dregs using 30% H_2SO_4 (v/v) at 121 °C for 30 minutes removed 48.20% lignin. In contrast, Purnawan (2011) found that delignification with 7.5% HNO_3 and 7.5% NaOH at approximately 103 °C for 1.5 hours resulted in 95.74% α-cellulose. Khalili and Amiri (2020) suggest increasing the temperature during sorghum dregs delignification at 150 °C and 180 °C for 30 and 60 minutes can reduce lignin. Additionally, Li et al. (2020) demonstrated that a hydrothermal process involving 55% ethanol, 3% NaOH, and 3% H_2O_2 at 180 °C for 90 minutes led to a significant reduction in hemicellulose by 94.4% and lignin by 83.5%.

The investigation of the optimal NaOH concentration, temperature, and duration for hydrothermal-alkali treatment of lignocellulose content is ongoing. Combining hydrothermal and hydrothermal alkali activation conditions holds great promise for achieving these objectives. The hydrothermal method aids in hemicellulose removal, while the hydrothermal alkali activation method assists in lignin removal. This process has significant potential for enhancing material-alkali source interaction at elevated temperatures (Liu et al., 2022). This study aims to identify the optimal delignification process point using the low-concentration NaOH hydrothermal alkali activation method on lignocellulose compounds.

MATERIALS AND METHODS

Sugar palm starch preparation

The sugar palm starch waste was obtained from a sugar palm starch industrial in Central Java, Indonesia. Approximately 200 gr of SPSW were washed with distilled water multiple times to eliminate pollutants and dried at 60 °C for 24 hours. After drying, the SPSW was ground to a particle size of 150–250 μm and stored for the delignification process.

Delignification process

The delignification process consists of two stages: hydrothermal and alkali activation. Approximately 3 gr of samples are placed in a hydrothermal reactor, and distilled water with a ratio of 1:35 (w/v) is added. The samples are then heated to 195 °C for 36 minutes. After cooling, the samples are filtered using filter papers and subsequently dried. Approximately 3 grams of the dried samples are mixed with a NaOH solution with a ratio of 1:25 (w/v) and heated using an oil bath. The percentages of NaOH concentration, temperature, and heating time are shown in Table 1.

Lignocelulose analysis

The lignocellulose analysis was performed using the Chesson method. Approximately 1 gram (W_1) of dry sample was refluxed at 100 °C for

Table 1. The experimental design (NaOH concentrations, temperature, and time)

Experiment	NaOH concentration (%)	Temperature (°C)	Time (Min)
1	2	80	120
2	3	100	39.5462
3	4	80	120
4	3	133.636	90
5	3	100	140.454
6	3	66.3641	90
7	4.68179	100	90
8	1.31821	100	90
9	4	120	120
10	3	100	90
11	2	120	60
12	3	100	90
13	3	100	90
14	4	80	60
15	3	100	90
16	3	100	90
17	3	100	90
18	2	120	120
19	4	120	60
20	2	80	60

1 hour and then treated with 150 ml of distilled water. The resulting mixture was filtered and washed with hot water. Subsequently, the sample was oven-dried until a constant weight (W_2) was achieved, followed by refluxing with 150 ml of 1 N H_2SO_4 for 1 hour at 100 °C. As in the previous stage, the sample was filtered, washed with hot water, and oven-dried until a constant weight was reached (W_3). The hemicellulose content was calculated using Equation 1.

$$Hemicellulose = [(W_2 - W_3)/W_1] \times 100\% \quad (1)$$

To obtain cellulose, the sample is immersed in 10 mL of 72% H_2SO_4 for 4 hours at room temperature. Subsequently, it is refluxed by adding 150 mL of 1 N H_2SO_4 at 100 °C for 1 hour. The resulting sample is filtered, washed with hot water, and dried in an oven until a constant weight (W_4) is achieved. The cellulose content is determined using Equation 2.

$$Cellulose = [(W_3 - W_4)/W_1] \times 100\% \quad (2)$$

In the last step, the sample is placed in the ashing furnace chamber, and the resulting ash is weighed (W_5). The lignin content is calculated using Equation 3.

$$Lignin = [(W_4 - W_5)/W_1] \times 100\% \quad (3)$$

Structural and morphological identification

The morphology of the SPSW was determined using a Scanning Electron Microscope (SEM) (Brand: FEI Inspect-S50 type). The samples were coated with a thin layer of gold during scanning at an acceleration voltage of 20.00 kV and a magnification of 1000×. The SPSW's crystallinity index was determined using X-ray diffraction (X-RD) (Brand: PANalytical, Type: X'Pert PRO). The instrument was operated with a Cu radiation source over a 2θ range from 10° to 90°. Additionally, the molecular bonds of the SPSW were analyzed using Attenuated Total Reflection-Fourier Transform Infrared Spectroscopy (FTIR) (Bruker 200546 Model Alpha).

Statistical analysis

Response surface methods (RSM) central composite design (CCD) was used with three replicates for each cellulose, hemicellulose, and lignin content sample. This method determined the optimal % NaOH concentration, temperature, and time of the delignification process. Analysis of variance (ANOVA) was applied to assess the significance of the treatments. All analyses were performed using design expert software (version 11).

RESULTS AND DISCUSSION

Lignocelulose content

Lignocellulose is the main component of plant biomass, making lignocellulose an abundant and sustainable source of raw materials. Lignocellulose is formulated of lignin, cellulose, and hemicellulose. The lignocellulose content differs for each material, depending on the type of biomass. The lignocellulose content in rice straw will differ from wheat straw, as will the SPSW. The results of measuring the lignocellulosic content of SPSW in this study were hemicellulose at 32.30%, cellulose at 48.90%, and lignin at 16.26%. (Fitriana et al., 2020) reported that sugar palm fibre has hemicellulose at 35.00%, cellulose at 50.68%, and lignin at 13.45%.

The high lignin content in SPSW significantly impacts the cellulose extraction process. Therefore, the delignification process to obtain high-purity cellulose was carried out. The delignification process consists of two stages: hydrothermal and alkali activation. In the hydrothermal stage, the pretreatment process focuses on dissolving hemicellulose, while the hydrothermal alkali activation focuses on dissolving lignin. The hydrothermal process is accomplished without the addition of the chemicals. In subcritical conditions, water is the solvent used in hydrothermal pretreatment at high temperatures (120–230 °C), and this method effectively removes hemicellulose (Bharadwaj et al., 2023).

This research conducted the SPSW hydrothermal process at 195 °C for 36 minutes. This process produces SPSW with a hemicellulose of 5.9%, cellulose of 72.6%, and lignin of 17.58%. Furthermore, hemicellulose decreased from 32.3% to 5.9%, and solubility reached 81.73%. This method is more significant than (Morales et al., 2022) and lower than Fiorentini et al. (Fiorentini et al., 2022). The wheat straw hydrothermal at 195 °C for 15 minutes can dissolve hemicellulose up to 88.96% (Fiorentini et al., 2022), whereas the walnut shells hydrothermal at 195 °C for 65 minutes were able to dissolve hemicellulose by 53.43% (Morales et al., 2022).

SPSW is then carried out with hydrothermal alkali activation using NaOH. At this stage, the

lignin removal process occurs. Table 2 shows the lignocellulose content of SPSW after the delignification process with several treatments. The results showed that the cellulose content was 86.7–92.9%, hemicellulose 1.7–3.3%, and lignin 0.53–7.71%. At this stage, the lignin removal reached 56.14–96.98%. Lignin reduction is more optimal at temperatures above 100 °C. The high temperature and pressure during hydrothermal processes force the lignin structure to be unconsolidated and break down into smaller fragments.

Furthermore, with increasing temperature, water ionic products increase while increasing the concentration of hydroxyl and hydronium ions, which can act as catalysts in lignin delignification (Scherzinger and Kaltschmitt, 2019). Using low-concentration NaOH significantly reduces the lignin content compared to other SPSW alkaline hydrolysis research (27–48.20%) (Erliana et al., 2020; Fitriana et al., 2020). It shows that using low concentrations of NaOH in hydrothermal alkali activation effectively reduces the lignin content and produces high-purity cellulose.

The optimization of the delignification process

This study's delignification process reduces hemicellulose and lignin content and increases cellulose. The analysis results show that all model responses are significant (p-value < 0.05). In contrast, the lack of fit values was insignificant (p-value > 0.05), indicating that the model was accepted. NaOH concentration had a significant impact in altering the amount of hemicellulose (p-value < 0.05), while temperature and time did not significantly influence hemicellulose content (p-value > 0.05). Otherwise, for cellulose content, temperature and time had a significant impact (p-value < 0.05). The changes in these factors affected the amount of cellulose present. NaOH concentration did not significantly influence cellulose content (p-value > 0.05). Furthermore, all three parameters (temperature, time, and NaOH concentration) had a significant influence on the amount of lignin content (p-value < 0.05).

The results of the model show an equation that describes the relationship between NaOH concentration (X_1), temperature (X_2), and time (X_3). In Hemicellulose (Y_h), the relationship between the parameters and hemicellulose content is described by a straightforward, linear model. Whereas Cellulose (Y_c) and Lignin (Y_l), these components exhibit a more complex relationship with the factors, requiring a quadratic model. The following Equation represents this relationship in a coded form:

$$Y_h = 2.54 + 0.2969\ X_1 + 0.1984\ X_2 – 0.0018X_3$$

Table 2. The lignocellulose content of SPSW after the delignification process with several treatments

Std	Run	% NaOH Concentration	Temperature (°C)	Time (Min)	Hemicellulose(%)	Cellulose (%)	Lignin (%)
5	1	2	80	120	2.1	89.65	6.12
13	2	3	100	39.5462	2.25	87.70	6.32
6	3	4	80	120	2.90	89.20	6.23
12	4	3	133.636	90	2.45	93.60	0.53
14	5	3	100	140.454	1.70	90.50	4.71
11	6	3	66.3641	90	1.85	88.55	7.71
10	7	4.68179	100	90	3.20	89.60	4.75
9	8	1.31821	100	90	1.80	89.30	6.03
8	9	4	120	120	3.10	92.30	0.82
18	10	3	100	90	2.35	89.25	5.46
3	11	2	120	60	2.45	89.65	3.88
17	12	3	100	90	3.30	88.70	5.38
19	13	3	100	90	2.65	88.50	5.39
2	14	4	80	60	2.45	87.40	5.98
20	15	3	100	90	2.50	89.15	4.98
16	16	3	100	90	2.40	89.50	5.35
15	17	3	100	90	3.30	89.20	4.72
7	18	2	120	120	2.90	92.35	2.45
4	19	4	120	60	2.95	90.30	2.15

$$Y_c = 89.05 - 0.0143X_1 + 1.36X_2 + 0.9233X_3 + \\ + 0{,}2375X_1X_2 - 0.0375X_1X_3 + 0.1875X_2X_3 + \\ + 0.1335X_1^2 + 0.7080\,X_2^2 + 0.0098X_3^2$$

$$Y_1 = 5.24 - 0.4556X_1 - 2.04X_2 + 0.4318X_3 - \\ - 0.3312X_1X_2 + 0.1287X_1X_3 - 0.2913X_2X_3 - \\ - 0.0836X_1^2 - 0.5326X_2^2 - 0.0394X_3^2$$

Figure 1 shows higher temperatures and longer processing times are needed to maximize cellulose production. However, an optimal NaOH concentration is required to balance cellulose yield with lignin removal. Otherwise, a higher NaOH concentration and processing temperature

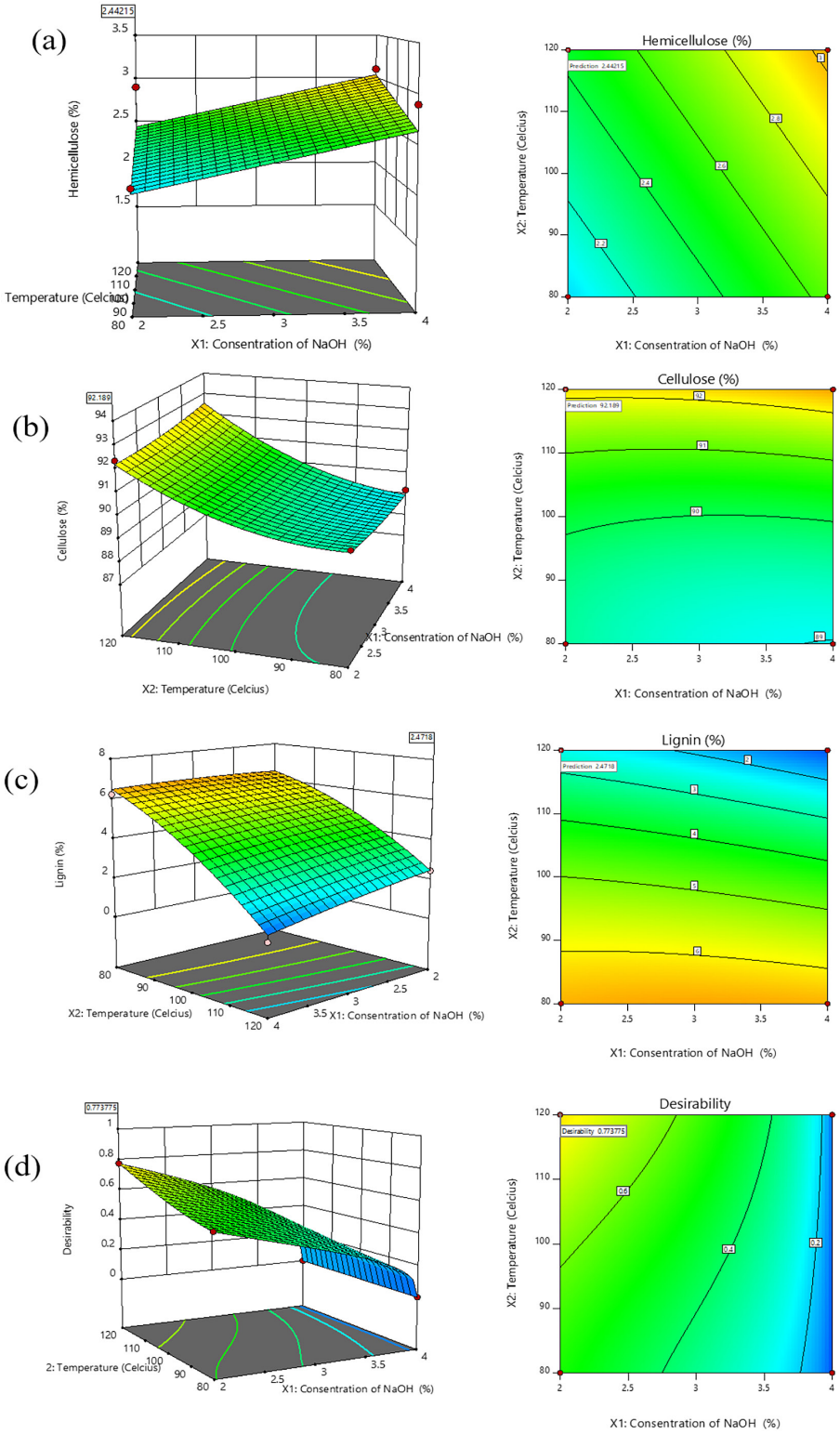


Figure 1. Contour and response surface model of (a) hemicellulose, (b) cellulose, (c) lignin, and (d) desirability

are needed to minimize lignin content. However, longer processing times have the potential to reduce lignin removal.

Table 3 shows the ideal conditions identified through optimizing the delignification process. These conditions performed a suitable proportion of NaOH concentration of 2%, temperature 120 °C, and time 120 minutes. The optimization process achieved a desirability score of 0.774 with a response value of hemicellulose 2.9 ± 1.7%, cellulose 92.35 ± 1.06%, and lignin 2.45 ± 0.1%.

The actual values obtained (Table 3) are compared with the predicted values (Table 4). The actual results are within the 95% prediction interval of the model, or the conditions for the delignification process to obtain optimal results are consistent. In other words, duplicating the process using 2% NaOH, 120 °C, and 120 minutes can predict and achieve similar results for hemicellulose, cellulose, and lignin content within the expected range.

Structural and morphological identification

Using FTIR, XRD, and SEM, the structure and morphology of SPSW were identified to validate the significance of the delignification process. Several peaks in FTIR were obtained in the SPSW spectra, which indicated the presence of lignin, cellulose, and hemicellulose (Fig. 2). All peaks indicated in untreated SPSW and delignified SPSW are all wood components. Figure 2 shows that untreated SPSW detected the presence of cellulose, hemicellulose, and lignin, whereas, in delignified SPSW, lignin was absent. The cellulose's presence is indicated by peak wavelength intensity ranging from 900 cm^{-1} to 1050 cm^{-1} (Maryana et al., 2014; Xu et al., 2013). Further, the presence of cellulose and hemicellulose is suggested by peaks at wavelengths around 3200–3600 cm^{-1} (O-H stretching) and 2800–2950 cm^{-1} (C-H stretching) (Cherdkeattikul et al., 2023). Furthermore, the presence of lignin is evident at peak wavelengths around 1465 cm^{-1} and 1700 cm^{-1} (Xu et al., 2013). It indicates that hydrothermal alkali's delignification process significantly removes lignin in SPSW.

X-ray diffraction analysis aims to determine approximate changes in the microstructure of cellulose. The sample is analyzed using crystal structure diffraction under X-rays. In cellulose, there are crystalline (2θ intensity 22–23°) and amorphous (2θ intensity 15–17°) regions (Fig. 3); the results of the ratio of the two are used to determine crystallinity index (Ci) (Wang et al., 2019).

During the delignification process, there is a decreasing crystallization index (CI) and an increasing crystallization size (CS) (Table 5). The decreasing CI is the same as to several other lignocellulosic material pretreatments, including sugar palm bagasse (Maryana et al., 2014), corn straw (Wang et al., 2019), rice husks (Jiang and Hu, 2019), and pussy willow flowers (Han et al., 2020). CI and CS are two crucial parameters that determine the cellulose quality. Cellulose CI decreased due to oxidative delignification treatment. A decrease in cellulose CI makes the substrate more appropriate for further processing. Meanwhile, the delignification process affects lignin removal, which wraps the cellulose crystals, thereby increasing CS.

Table 3. Constraint data optimation

Name	Goal	Lower limit	Upper limit	Lower weight	Upper weight	Importance
X_1: Consentration of NaOH	Minimize	2	4	1	1	5
X_2: Temperature	Is in range	80	120	1	1	3
X_3: Time	Is in range	60	120	1	1	3
Hemicellulose	Minimize	1.7	3.3	1	1	3
Cellulose	Maximize	87.4	93.6	1	1	3
Lignin	Minimize	0.53	7.71	1	1	3

Table 4. Confirmation of the optimization point

Respon	Predicted mean (%)	Observed (%)	95% CI low for the mean	95% CI high for the mean
Hemicellulose	2.44215	2.9 ± 1.7	1.98367	2.90063
Cellulose	92.189	92.35 ± 1.06	91.55	92.828
Lignin	2.4718	2.45 ± 0.1	1.61483	3.32876

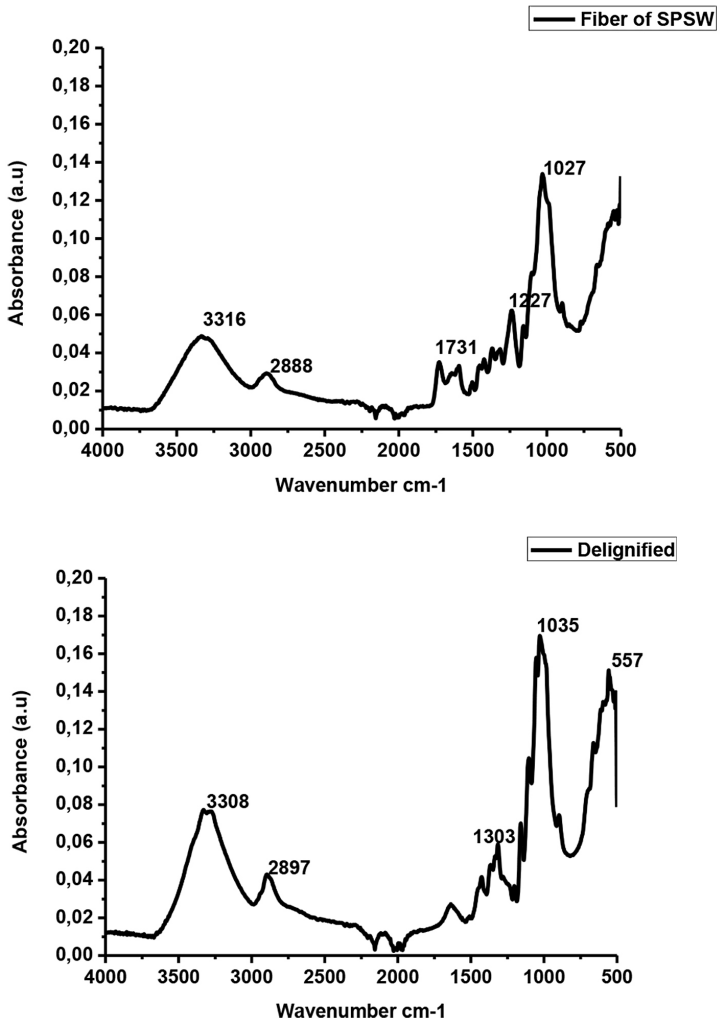


Figure 2. FTIR analysis of SPSW untreated and delignified

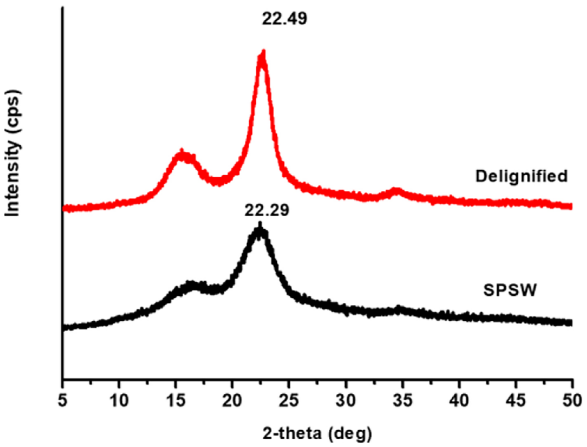
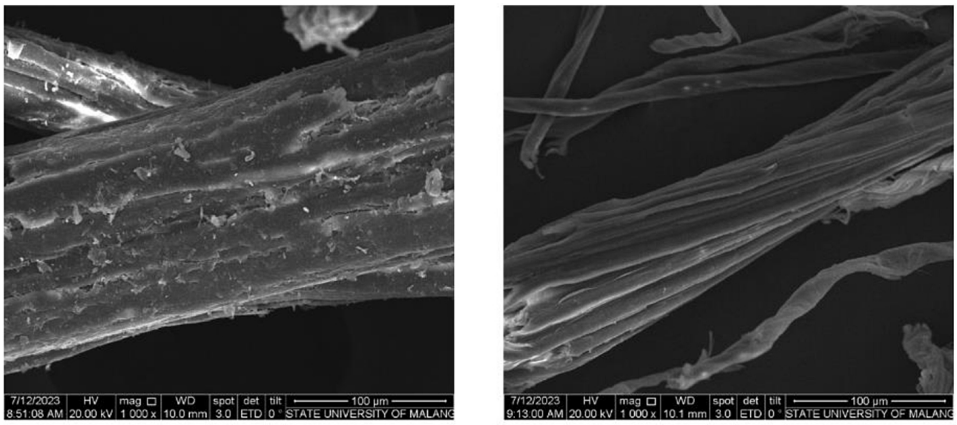


Figure 3. XRD analysis

SEM analysis indicates surface modifications induced by the treatment (Fig. 4). The pretreatment process induces alterations in the lignocellulose structure and significantly modifies the amount of hemicellulose, cellulose, and lignin. However, it will also cause morphological changes. SPSW samples that have not been treated show rigidly linked fibrils with a rough morphology. This condition is because the samples that have not received treatment still contain a lot of hemicellulose and lignin, which are amorphous. The morphology of the delignified SPSW becomes smoother after pretreatment, which reduces the hemicellulose and lignin content, meaning

Table 5. X-ray difraction

Source	2θ (I_{002})	2θ (I_{am})	Crystallinity index (%)	Crystallite size (nm)
Control	22.42	16.11	82.23	1.98
Delignified	22.59	15.46	66.81	3.88



(a) (b)

Figure 4. Morphology of SPSW untreated (a) and delignified (b) using SEM

that the material has a high level of cellulose purity. The pretreatment process causes fibril separation in the sample, which did not occur before the pretreatment.

CONCLUSIONS

In this study to investigate the optimal conditions for lignin and hemicellulose removal to obtain cellulose with high purity. Using Combining hydrothermal and hydrothermal alkali activation pretreatment is effective in the SPSW delignification process. The study obtained optimum conditions for hydrothermal alkali activation at NaOH concentration of 2%, temperature 120 °C, and time 120 minutes, with purity cellulose 92.18%, hemicellulose 2.44%, and lignin 2.47%. This method is more effective in producing cellulose and reducing lignin than hydrothermal. This study demonstrates cellulose production with high purity > 80%. The research indicates that the concentration of NaOH substantially impacts the levels of hemicellulose and lignin. In contrast, temperature and time significantly affect cellulose and lignin. Using low-concentration NaOH combined with hydrothermal treatment proves to be more environmentally friendly and effective in decreasing lignin levels. FTIR analysis shows hemicellulose, cellulose, and lignin content in the untreated SPSW and the absence of lignin in the delignification SPSW. XRD gave results of a decrease of the crystallinity index of the cellulose obtained as 66.81%, and the crystallite size was 3.88 nm. The morphological SPSW using SEM shows that the pretreatment process can remove hemicellulose and lignin to obtain high-purity cellulose. These results show that SPSW has the potential for cellulose production. Further research is needed to improve cellulose yield, reduce energy consumption, and analyze environmental impact. This research should also consider other potential pretreatment factors, such as pressure and catalysts in the hydrothermal process, to improve the cellulose purity and yield.

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