

Comprehensive Evaluation of the Effect of Microwave-Assisted Alkali Pretreatment using Ammonia on Pineapple Core Waste

Evaluasi Komprehensif terhadap Pengaruh Pra-perlakuan Alkali Berbantuan Gelombang Mikro Menggunakan Amonia pada Limbah Bonggol Nanas

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ABSTRACT

This study aimed to comprehensively investigate the effect of microwave-assisted alkali pretreatment on delignification and changes in composition, functional groups, morphology, and crystallinity structure of pineapple core waste biomass. Pretreatment was performed using a 0.03 M ammonia solution under microwave irradiation at 600 W power for 5 minutes. Delignification efficiency and biomass composition were determined using biomass compositional analysis method from the National Renewable Energy Laboratory (NREL), functional groups were characterized by Fourier Transform Infrared Spectroscopy (FTIR), surface morphology was characterized using Scanning Electron Microscope (SEM), and crystallinity was characterized using X-Ray Diffraction (XRD) method. The results showed that microwave-assisted alkali pretreatment reduced the lignin content from 23.443% to 19.71%, increase the hemicellulose content from 39.903% to 45.967%, and maintained the cellulose content at 34.317% from 36.653% before pretreatment. FTIR analysis showed a weakening of the lignin aromatic band and changes in the hydroxyl group. SEM analysis revealed pronounced morphological changes in the biomass structure which was initially dense and regular to become rougher, cracked, and containing cavities. XRD analysis showed a decrease in the crystallinity index which indicates the occurrence of partial disruption of the cellulose crystal structure. Overall, microwaves-assisted alkali pretreatment using ammonia effectively modified the physicochemical characteristics of pineapple core biomass, especially in the delignification process and changes in biomass structure. The improvement in physicochemical characteristics obtained through these pretreatment indicate the potential to enhance the efficiency of biomass conversion processes for advanced stages, such as enzymatic hydrolysis and biofuel production.

Keywords: *pineapple core waste, lignocellulose, alkali pretreatment, microwave-assisted, characterization*

ABSTRAK

Penelitian ini bertujuan untuk menelaah secara komprehensif pengaruh perlakuan awal alkali dengan bantuan gelombang mikro terhadap delignifikasi dan perubahan komposisi, gugus fungsional, morfologi, dan struktur kristalinitas pada biomassa limbah bonggol nanas. Perlakuan awal dilakukan menggunakan larutan amonia 0,03 M melalui pemanasan gelombang mikro dengan daya 600 W selama 5 menit. Delignifikasi dan komposisi ditentukan menggunakan metode analisis komposisi biomassa dari *National Renewable Energy Laboratory* (NREL), gugus fungsional dikarakterisasi dengan *Fourier Transform Infrared Spectroscopy* (FTIR), morfologi permukaan dikarakterisasi menggunakan *Scanning Electron Microscope* (SEM), dan kristalinitas dikarakterisasi dengan metode *X-Ray Diffraction* (XRD). Hasil penelitian menunjukkan bahwa perlakuan awal alkali dengan gelombang mikro mampu mengurangi kadar lignin dari 23,443% menjadi 19,71%, meningkatkan kadar hemiselulosa dari 39,903% menjadi 45,967%, dan mempertahankan kadar selulosa pada 34,317% dari 36,653%. Analisis FTIR menunjukkan pelemahan pita aromatik lignin dan perubahan pada gugus hidroksil. Analisis SEM menunjukkan perubahan morfologi yang terlihat pada struktur biomassa yang awalnya padat dan secara teratur menjadi lebih kasar, retak, dan mengandung rongga. Analisis XRD menunjukkan penurunan indeks kristalinitas yang mengindikasikan terjadinya gangguan sebagian pada struktur kristal selulosa. Secara keseluruhan, perlakuan awal alkali dengan bantuan gelombang mikro menunjukkan efektivitas dalam memengaruhi karakteristik biomassa bonggol nanas, terutama dalam proses delignifikasi dan perubahan struktur biomassa. Peningkatan karakteristik fisikokimia yang diperoleh melalui perlakuan awal ini menunjukkan adanya potensi untuk meningkatkan efisiensi proses konversi biomassa untuk tahap lanjutan, seperti hidrolisis enzimatis dan produksi biofuel.

Kata kunci: bonggol nanas, lignoselulosa, perlakuan awal alkali, berbantuan gelombang mikro, karakterisasi

INTRODUCTION

Lignocellulosic agricultural waste is a renewable biomass resource with significant potential for conversion into bioenergy or bio-based chemicals (Widiastuty et al., 2021). Pineapple waste is an abundant agricultural by-product in Indonesia. According to data from the Central Statistics Agency (BPS), pineapple production in Indonesia reached 2.74 million tons in 2024 (Badan Pusat Statistik, 2024). This high production volume generates substantial quantities of organic waste from unutilized fruit biomass. Much

of this waste is discarded or left to degrade naturally, potentially causing environmental pollution (Méndez-durazo et al., 2025). One of the major waste fractions is the pineapple core, which are still underutilized. This biomass contains a high proportion of lignocellulosic components. However, the lignocellulose structure in pineapple core biomass is very complex due to the presence of lignin that protects the cellulose and hemicellulose, making the biomass difficult to hydrolyze directly without pretreatment (Baig, 2020; Saini et al., 2022).

Previous studies have shown that pretreatment can increase the accessibility of cellulose in lignocellulosic biomass by disrupting its structure and loosening the bonds between components thereby promoting lignin degradation (Ulises et al., 2019). Various pretreatment approaches have been carried out to increase the conversion of lignocellulosic biomass, one of which is physicochemical pretreatment which uses physical and chemical processes (Kumar and Sharma, 2017). Heating using microwaves is widely applied because it allows for fast and even heat distribution during the treatment process (Zhou et al., 2023) and alkaline reagents such as ammonia solution are capable of breaking the ester and ether bonds between lignin and hemicellulose (Jacqueline and Velvizhi, 2024).

A previous study reported that microwave-assisted alkali pretreatment on pineapple biomass was able to increase the openness of the biomass structure and producing higher reducing sugar yields after the hydrolysis process (Jacqueline and Velvizhi, 2024). Characterization of changes in biomass structure after pretreatment can be analyzed using Fourier Transform Infrared Spectroscopy (FTIR) to identify changes in functional groups, Scanning Electron Microscopy (SEM) to observe surface morphology and structural

disruption, and X-Ray Diffraction (XRD) to evaluate changes in cellulose crystallinity and crystal structure (Pratama et al., 2023; Saini et al., 2022).

Although numerous studies have been conducted on lignocellulosic biomass pretreatment, studies investigating pineapple core waste using microwave-assisted alkali pretreatment remain limited, particularly those that analyze changes in the overall structural characteristics of the biomass. Most previous studies have focused only on increasing sugar yield or on one biomass characterization after pretreatment. Therefore, there is still a research gap regarding the relationship between changes in the structure of pineapple core biomass due to pretreatment and increased cellulose accessibility to hydrolysis. The novelty of this study is a more comprehensive evaluation of the effect of microwave-assisted alkali pretreatment using ammonia on changes in lignocellulose content, functional groups, surface morphology, and crystallinity structure of pineapple core biomass. These characteristics are important because lower lignin content can increase biomass accessibility and conversion efficiency, while high lignin content can inhibit the hydrolysis process and subsequent utilization.

METHODS

Tools and Materials

The main equipment used in this study included the following: a drying oven (Drying Oven DX302), a 600-W microwave (Samsung), an analytical balance (Vibra SJ), a shaker incubator (Taitec), an autoclave (Tomy SX-700), a centrifuge (Sigma 2-16KL), a magnetic stirrer (Thermo Scientific Cimarec MG-320), a water bath (VWR 1217-2), a 40-mesh sieve, and characterization instruments, including Fourier Transform Infrared Spectroscopy (FTIR) (Thermo Scientific Nicolet iS-10 iD5 ATR), Scanning Electron Microscopy (SEM) (Jeol JSM IT 200 and Thermo Scientific Prisma E), and X-Ray Diffraction (XRD) (AERIS PANalytical).

The primary raw material was dried and ground pineapple core waste. The chemicals used included a 0.03 M ammonia (NH₃) solution (prepared from a 25% ammonia solution, analytical grade, Merck) as a pretreatment reagent, 0.5 M sodium hydroxide (NaOH), 7.3 M sulfuric acid (H₂SO₄), and barium chloride (BaCl₂). Biomass composition analysis was performed according to the National Renewable Energy Laboratory (NREL) standard method.

Pineapple Core Waste Preparation

Raw pineapple core waste was thoroughly washed and cut into small pieces. The biomass was then placed in an oven at 60°C for 12 hours. The dried biomass was milled and sieved through a 40-mesh sieve to obtain a uniform particle size (Pathanibul and Hongkulsup, 2021).

Microwave-Assisted Alkali

Pretreatment

Pretreatment was performed using a microwave-assisted alkali method. The biomass powder was mixed with 200 mL of 0.03 M ammonia solution as a pretreatment reagent, according to the optimized pretreatment conditions previously reported for pineapple biomass (Jacqueline and Velvizhi, 2024) using a biomass loading of 10% (w/v) (20 g). The mixture was heated in a microwave oven at 600 W for 5 minutes to disrupt the lignocellulosic structure of the biomass. After microwave treatment, the sample was cooled to room temperature and the biomass was repeatedly washed with distilled water until the pH reached approximately 6-7. During the washing process, phase separation was achieved by centrifuging the mixture at 4000 rpm for 10 minutes. The samples were then dried at 45°C for 24 hours using an oven, and then left in a desiccator until a constant weight was obtained (Jacqueline and Velvizhi, 2024).

Biomass Composition Analysis

Biomass composition analysis was performed on the samples before and after pretreatment using the National Renewable Energy Laboratory (NREL) method. The analyzed parameters included the contents of hemicellulose, lignin, and cellulose content (Palamae et al., 2014).

Hemicellulose Analysis

A total of 0.6 g of biomass was reacted with 6 mL of 0.5 M NaOH solution at 80°C for 3.5 hours in a water bath. The sample was then filtered and washed with distilled water until the pH reached 7, the resulting residue was then dried at 105°C for 5 hours and allowed to cool in a desiccator until a constant weight was achieved. The hemicellulose content was calculated using Equation (1).

$$H(\%) = \frac{W_1 - W_2}{W_1} \times 100\% \quad (1)$$

Where W_1 represents the biomass weight before treatment, while W_2 represents the biomass weight after treatment.

Lignin Analysis

A total of 0.2 g of biomass was reacted with 200 mL of 7.3 M H_2SO_4 solution. The mixture was incubated in a shaker incubator at 30°C for 1 hour. Afterward, 56 mL of distilled water was added, and the mixture was autoclaved at 121°C for 1 hour. The sample was then

washed with distilled water until no sulfate ions remained in the filtrate, as indicated by the solution becoming cloudy after being tested with $BaCl_2$ drops. The recovered biomass was subsequently dried at 105°C for 5 hours and cooled in a desiccator until a constant weight was achieved. Lignin content was calculated using Equation (2).

$$L(\%) = \frac{W_4}{W_3} \times 100\% \quad (2)$$

Where W_3 represents the biomass weight before treatment, while W_4 represents the biomass weight after treatment.

Cellulose Analysis

The Cellulose content was determined by calculating the difference between the total biomass components of the hemicellulose and lignin analysis results using Equation (3).

$$L(\%) = 100 - H(\%) - L(\%) \quad (3)$$

Where H (%) represents the hemicellulose content and L (%) represents the lignin content.

Biomass Characterization

Pineapple core biomass was characterized before and after pretreatment using Fourier Transform Infrared Spectroscopy (FTIR) (Thermo Scientific Nicolet iS-10 iD5 ATR) to identify functional groups, Scanning Electron Microscopy (SEM) (Jeol JSM IT 200 and

Thermo Scientific Prisma E) to observe changes in biomass surface morphology, and X-Ray Diffraction (XRD) (AERIS PANalytical) to evaluate changes in the crystallinity of the biomass.

RESULT AND DISCUSSION

Effect of Microwave-Assisted Alkali Pretreatment on the Lignocellulose Composition of Pineapple Core

As shown in **Table 1**, compositional analysis based on the National Renewable Energy Laboratory (NREL) method showed that the microwave-assisted alkali pretreatment process resulted in changes in the proportions of hemicellulose, lignin, and cellulose. The relatively small standard deviation values obtained for each parameter indicate that the data have a relatively small level of deviation, so the analysis results were consistent and representative.

The lignin content of pineapple core biomass decreased from 23.443% to 19.715% after microwave-assisted alkali pretreatment, indicating that the treatment was able to disrupt part of the lignin carbohydrates complex in the cell walls, so that the biomass structure became more accessible. These findings are consistent with previous reports that alkali pretreatment was effective in increasing the openness of biomass structures, although

the degree of delignification remained relatively limited (Madyira and Olatunji, 2025).

Table 1. Lignocellulose composition of pineapple core waste before and after pretreatment.

<i>Sample</i>	<i>Lignin (%)</i>	<i>Hemi cellulose (%)</i>	<i>Cellulose (%)</i>
<i>Before pretreatment</i>	23.443± 0.636	39.903 ± 3.240	36.653 ± 3.084
<i>After pretreatment</i>	19.715 ± 1.260	45.967 ± 2.284	34.317 ± 3.225

Hemicellulose content increased from 39.903% to 45.967% after pretreatment, indicating some lignin degradation, making the hemicellulose fraction more detectable. This finding is consistent with previous studies on alkaline pretreatment, where lignin reduction can alter the relative distribution of lignocellulosic components (Madyira and Olatunji, 2025).

Meanwhile, the analysis results showed a slight decrease in cellulose content, from 36.653% to 34.317%. This decrease indicates that a small portion of amorphous cellulose underwent changes during the pretreatment process. According to a previous study, microwave heating may induce swelling in cellulose fibers, thereby making the amorphous cellulose

fraction more susceptible to mild depolymerization or partial dissolution. Overall, these findings indicate that microwave-assisted alkali pretreatment can modify the lignocellulosic composition (Venegas-vásconez et al., 2025).

Effect of Microwave-Assisted Alkali Pretreatment on Functional Groups

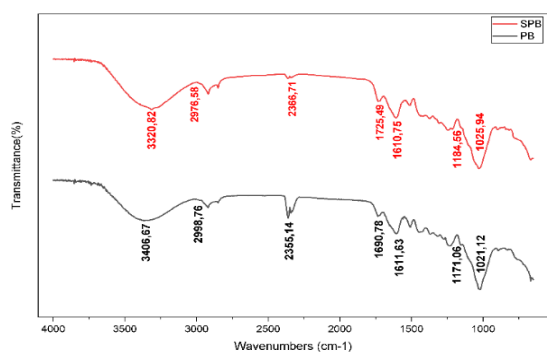


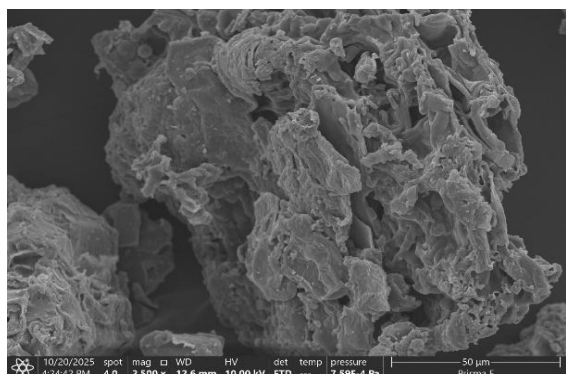
Figure 1. FTIR spectra analysis of pineapple core before and after microwave-assisted alkali pretreatment.

As shown in **Figure 1**, the Fourier Transform Infrared Spectroscopy (FTIR) analysis results indicate changes in the functional groups of the biomass after microwave-assisted alkali pretreatment. Before pretreatment (SPB), the absorption band at 3320.82 cm^{-1} corresponding to the stretching vibration of the hydroxyl group O-H of lignocellulose indicating the presence of strong hydrogen bonding in the lignocellulose matrix (Lestari et al., 2025). The absorption band at 2976.58 cm^{-1} corresponds to the aliphatic carbon-hydrogen C-H stretching vibration of lignin and polysaccharides, while the absorption bands at 1725.49 cm^{-1} and 1610.75 cm^{-1} are

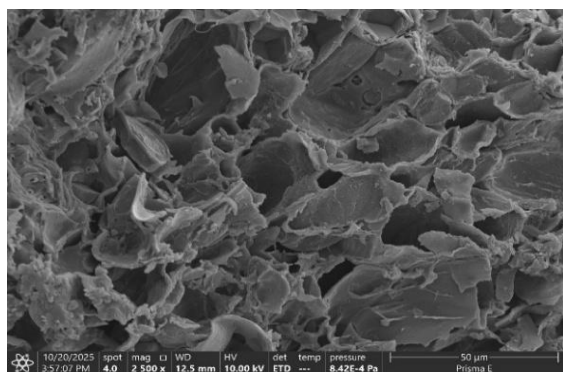
attributed to the vibrational modes of the carbonyl group C=O and the aromatic C=C stretching vibration of lignin, indicating that lignin was still dominant before pretreatment. In addition, the absorption bands at 1184.56 cm^{-1} and 1025.94 cm^{-1} are attributed C–O and C–O–C stretching vibrations associated with cellulose through β -1,4-glycosidic linkages bonds in the sample, indicating the presence of cellulose in the lignocellulosic matrix (Mandasari and Sitorus, 2023).

After pretreatment (PB), the absorption band of the hydroxyl group (O-H) shifted to a higher wavenumber and became broader 3406.67 cm^{-1} , suggesting weakened hydrogen bonding due to the opening of the lignocellulose matrix. The absorption band at 2998.76 cm^{-1} remained clearly visible, indicating that the main structure of cellulose was maintained despite the pretreatment. The absorption band at 1690.78 cm^{-1} showed reduced intensity, suggesting partial lignin degradation (Jacqueline and Velvizhi, 2024). The absorption bands at 1163.63 cm^{-1} , 1171.06 cm^{-1} , and 1021.12 cm^{-1} became more pronounced after pretreatment, indicating greater accessibility of the cellulose structure due to the partial removal of lignin from the biomass matrix.

Effect of Microwave-Assisted Alkali Pretreatment on Morphological Changes



(a)



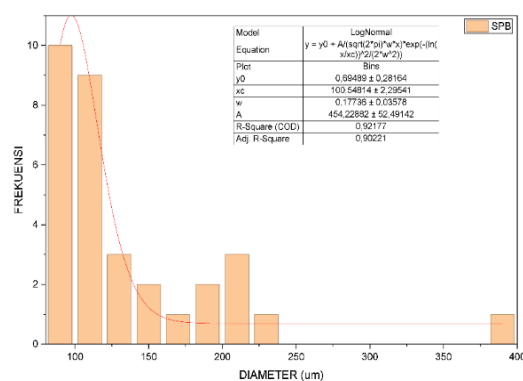
(b)

Figure 2. SEM micrographs of pineapple core biomass (a) before and (b) after microwave-assisted alkali pretreatment.

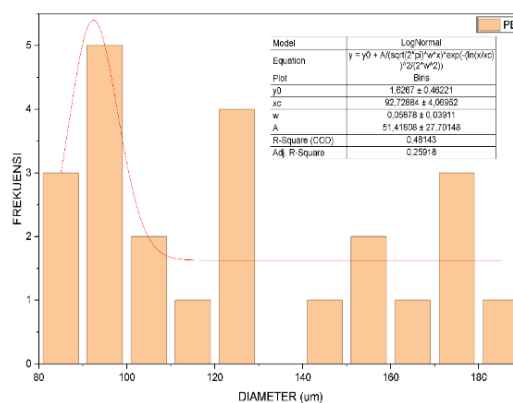
Based on SEM analysis presented in (Figure 2a), the sample before pretreatment exhibited a dense, relatively smooth, and non-porous surface morphology, indicating that lignin still functioned a matrix that binds the cell walls so that the surface area and accessibility of the biomass to reactants were limited. Biomass without pretreatment generally has a rigid surface and is less favorable for

subsequent hydrolysis (Sunkar and Bhukya, 2022).

After the pretreatment in (figure 2b) the surface morphology of the biomass underwent substantial morphological changes, characterized by a rougher, more fibrous, and more porous surface, as well as cracks and cavities. This observation indicates damage to the cell walls due to the release of some lignin, thereby increasing the surface area of the biomass and its accessibility to reactants (Sunkar and Bhukya, 2022).



(a)



(b)

Figure 3. Log-Normal SEM micrographs of pineapple core biomass (a) before and (b) after microwave-assisted alkali pretreatment.

This observation is further supported in the results of the log-normal distribution analysis with a coefficient of determination (R^2) value of 0.9277 in **(Figure 3a)** before pretreatment, which indicates that the particle size distribution remained relatively uniform and the biomass structure is not damaged. This finding is further supported by a decrease in the coefficient of determination (R^2) to 0.4814 in **(Figure 3b)** after pretreatment, indicating more heterogeneous biomass fragmentation.

Effect of Microwave-Assisted Alkali

Pretreatment on Changes in

Crystallization Structure

As shown in **Figure 4**, X-ray diffraction (XRD) analysis revealed that before pretreatment (SPB) and after pretreatment (PB) revealed the typical characteristics of lignocellulosic biomass, characterized by the presence of the main crystalline peak at a diffraction (2θ) of approximately 22.5° , indicating the presence of type I cellulose, and a broad diffraction region at approximately $18-20^\circ$, indicating the presence of an amorphous associated with the amorphous lignocellulosic phase. The crystallinity index (CRI) was calculated to be, 30.93% for the untreated sample (SPB) and decreased to 29.21% after pretreatment (PB). The decrease in the CRI value

indicates that the pretreatment process is able to modify the internal cellulose structure of the biomass without altering the cellulose crystal polymorph (Jang and Jeong, 2023).

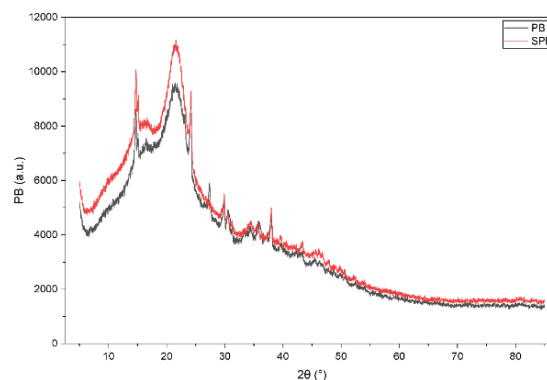


Figure 4. XRD patterns of pineapple core biomass before and after pretreatment.

The decrease in crystallinity index after pretreatment indicates disruption in the intra- and intermolecular hydrogen bonds that maintain the cellulose structure, resulting in partial distortion of the crystalline structure an increase in the amorphous biomass fraction has also been reported to decrease the crystallinity index due to the disruption of the cellulose crystalline structure after alkali pretreatment. Furthermore, the characteristic cellulose I diffraction peak observed in both samples indicates that the pretreatment did not induce polymorphic changes in cellulose. Lignocellulosic biomass generally contains amorphous lignin and hemicellulose phases. Overall, these findings demonstrate that the

pretreatment effectively altered the structural regularity of the biomass.

CONCLUSION

The findings of this study suggest that microwave-assisted alkali pretreatment can modify the physicochemical characteristics of pineapple core lignocellulosic biomass, as reflected in the structural and chemical changes of the biomass. These changes were reflected by a reduction in lignin content, indicating successful delignification, and changes in the relative proportions of hemicellulose and cellulose, indicating a redistribution of structural components due to pretreatment. FTIR analysis revealed changes in the position and intensity of functional group, absorption bands particularly in the hydroxyl and aromatic groups of lignin, suggesting weakened hydrogen bonding interactions and a reduction in lignin structure within the biomass matrix.

SEM analysis showed that the pretreated biomass exhibited a rougher surface morphology and more irregular, with the formation of cracks and pores, indicating improved accessibility to reactants for subsequent conversion processes. XRD analysis demonstrated a decrease in the crystallinity index, indicating disruption of the cellulose

crystalline structure due to pretreatment. The combination of alkali pretreatment and microwave heating is an effective approach in increasing the reactivity of lignocellulosic biomass. However, this study was limited to evaluating changes in the composition and structure of pineapple core biomass after pretreatment. Therefore, further studies are required to investigate the effect of this modification on subsequent conversion processes, such as enzymatic hydrolysis and biofuel production, as well as to optimize pretreatment parameters to improve the utilization of lignocellulosic biomass.

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