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Enhancement of Pineapple Industrial Waste Delignification Through the Effect of Microwave Irradiation Duration with Alkali Assistance

Livia Rhea Alvita [a,*], Vida Elsyana[a], Rahmat Hidayat[a], Anggi Saputra[a], Yeni Variyana[b], Rafli Rustiawan Jody[a]

[a] Study Program of Applied Chemistry, Department of Agricultural Technology, Politeknik Negeri Lampung
[b] Industrial Chemical Engineering Technology, Department of Engineering, Politeknik Negeri Lampung
* corresponding author: liviarhea@polinela.ac.id
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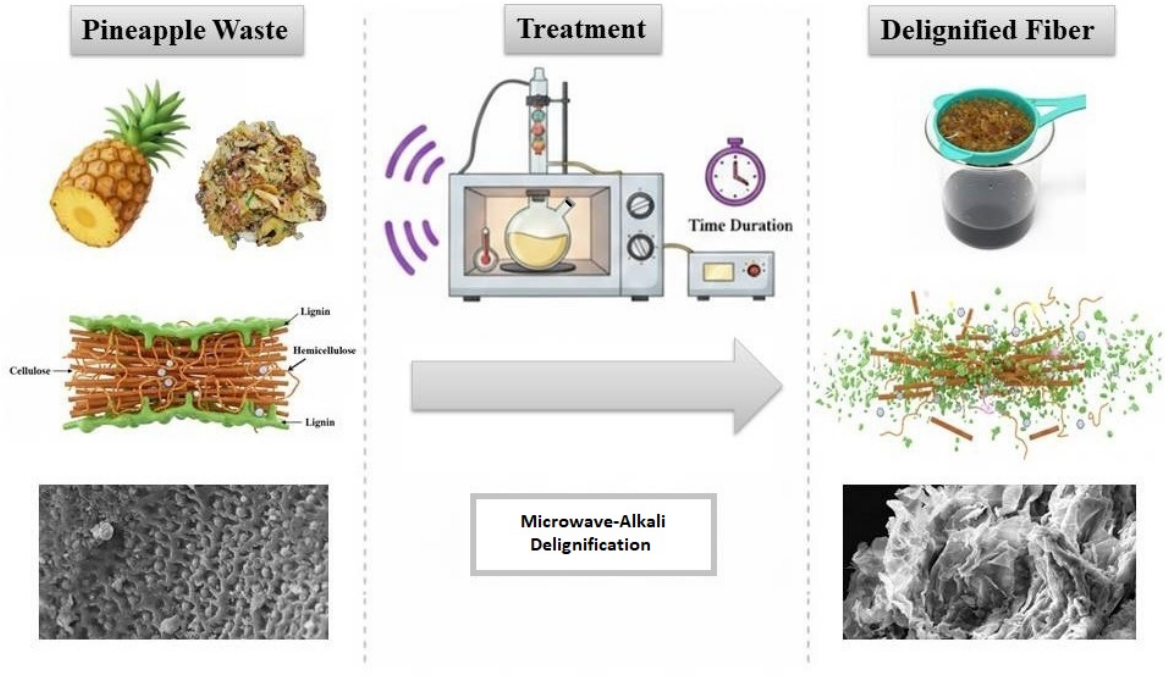
GRAPHICAL ABSTRACT



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ABSTRACT

Delignification is an important step for removing lignin, increasing the accessibility of cellulose and hemicellulose for various industrial applications. This study investigated the effect of microwave irradiation duration on the alkaline-assisted delignification of pineapple peel waste using a Completely Randomized Design (CRD) with irradiation times of 0, 15, 30, and 45 minutes at a constant power of 45 W. Compositional analysis was performed using the Chesson–Datta method, while structural and chemical modifications were evaluated through Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM). The results indicated that increasing irradiation time significantly improved delignification efficiency

($P < 0.05$), with the highest lignin reduction of 57.6% achieved at 45 minutes, accompanied by increases in cellulose and hemicellulose contents of 62.79% and 55.8%, respectively. FTIR spectra confirmed the removal of lignin through the reduction of characteristic functional group intensities. Moreover, SEM analysis revealed pronounced structural disruption, increased surface irregularity, and enhanced porosity after treatment, indicating effective breakdown of the lignocellulosic matrix and improved exposure of cellulose fibers. These findings demonstrate that microwave-assisted alkaline treatment is a promising approach for enhancing the valorization of pineapple industry waste.

1. INTRODUCTION

The rapid growth of agroindustrial activities has led to a significant increase in lignocellulosic waste, particularly from the pineapple processing industry. Pineapple peel waste contains relatively high lignin content, which limits its direct conversion into value-added products such as bioethanol and bio-composites [1]. Therefore, an effective delignification process is an essential step to improve cellulose accessibility and support efficient biomass valorization. Conventional alkaline pretreatment methods, although widely applied, generally require long heating times and high energy consumption, highlighting the need for more efficient alternatives [2].

Microwave-assisted pretreatment has emerged as a promising technology due to its rapid volumetric heating mechanism, improved energy efficiency, and enhanced reaction kinetics compared with conventional heating systems [3]. The synergistic combination of microwave irradiation and alkaline agents has been widely investigated for various lignocellulosic biomasses, demonstrating improved lignin removal under milder processing conditions and shorter treatment times [4, 5]. However, studies specifically addressing pineapple peel biomass remain limited, and most available research focuses on microwave treatment without chemical additives or on alternative chemical systems rather than controlled microwave-assisted alkaline processes.

Furthermore, systematic evaluation of the influence of microwave irradiation duration at specific power levels and alkaline concentrations has not been extensively explored for pineapple peel biomass. Irradiation time is a critical factor affecting the efficiency of lignocellulosic biomass delignification, as it determines the effectiveness of microwave penetration and the cleavage of lignin linkages. Insufficient irradiation time may result in incomplete delignification, whereas excessive exposure may lead to undesirable degradation and reduced quality of the resulting biomass fractions [6, 7].

This study aims to evaluate the effect of microwave irradiation duration on the alkaline-assisted delignification of pineapple processing waste from Lampung Province, one of Indonesia's major pineapple production centers. Identifying the optimal irradiation duration within the tested range is expected to enhance lignin removal efficiency while preserving cellulose quality, thereby offering a potentially energy-efficient and environmentally sustainable approach to agroindustrial waste valorization.

2. EXPERIMENTAL METHODS

2.1. Materials

The materials used in this study included pineapple peel waste obtained from a pineapple processing company in Lampung Province, Indonesia, which served as the primary lignocellulosic raw material. Sodium hydroxide (NaOH, Merck) was employed as the alkaline reagent in the delignification process. Sulfuric acid (H_2SO_4, Merck) was used to prepare 0.5 M and 72% H_2SO_4 solutions required for compositional analysis. In addition, reagents for carbohydrate determination by the Luff–Schoorl method were used in this study.

2.2. Physically Pretreatment of Pineapple Peel Waste

The initial stage is fiscal pretreatment of pineapple peel waste through chopping it into small pieces, blending it into a fine pulp, and then separating the juice from the filtered pulp by squeezing. The resulting pulp enters the delignification stage, but is first dried and analyzed for moisture content and carbohydrate analysis using the Luff-Schoorl method.

2.3. Delignification of Pineapple Peel Waste

A total of 50 g of dried pineapple peel was mixed with 250 mL of 0.8 N NaOH solution at a solid-to-liquid ratio of 1:5 (w/v). The mixture was subjected to microwave irradiation at 45 W for 15, 30, and 45 minutes. The selection of 45 W was based on preliminary experiments conducted at power levels ranging from 45 to 135 W, which showed no significant differences in delignification performance; therefore, the lowest tested power was chosen to improve energy efficiency and process control. Power levels above 135 W were not applied, as they tended to induce excessive and unstable boiling (bumping), particularly in the presence of 0.8 N NaOH solution.

Pretreatment was performed in a modified laboratory-scale microwave reactor for alkaline-assisted delignification. A Sharp microwave oven was used as the irradiation source, and the reaction was conducted in a round-bottom flask placed within the microwave cavity. The flask was connected to a vertical spiral condenser equipped with a continuous water circulation system to maintain reflux conditions, prevent solvent loss, and minimize bumping caused by rapid microwave heating. The system operated under a semi-closed configuration, and the reaction temperature was monitored and maintained below 100°C to avoid excessive thermal degradation and ensure controlled processing conditions. After irradiation, the slurry was filtered and thoroughly washed with distilled water until the pH was neutral. The obtained solid residue was dried to constant weight and subsequently analyzed for lignin, cellulose, hemicellulose, ash content, °Brix, and reducing sugar.

Morphological and structural characterizations were performed both before and after the delignification treatment. The surface morphology of untreated and delignified samples was examined using Scanning Electron Microscopy (SEM) to observe structural changes in the lignocellulosic matrix. Functional group analysis was performed using Fourier Transform Infrared Spectroscopy (FTIR) over 4000–500 cm^{-1} to identify chemical structure modifications associated with lignin removal and cellulose exposure.

2.4. Cellulose, Hemicellulose, Lignin and Ash Content Analysis (Chesson Data Method)

The sample to be analyzed is weighed (a), then put in a boiling flask that has been installed on a heating mantle, and then distilled water is added to the flask as much as the sample used times 150 ml. The condenser is assembled, and then the flask is refluxed at 100 °C for 2 hours. The refluxed product is filtered and then placed in the oven at 105 °C until constant weight. The filter paper was weighed until it reached a constant weight (b). The residue was refluxed with 0.5 M H_2SO_4 until the volume was 150 ml per g sample, then filtered with distilled water until the pH was neutral, and dried in the oven. The residue was weighed to the nearest 0.1 g (c). Soaking the residue with 72% H_2SO_4 as much as 10 ml times the sample at room temperature for 4 hours, then adding 0.5 M H_2SO_4 as much as 150 ml times the sample to be diluted, refluxed again, then the residue is filtered and washed with distilled water until neutral, then dried until constant and weighed. The dried residue is then ignited in a furnace at 575 °C for approximately 3 hours until it reaches a constant weight (e) [8].

1) Hemicellulose (%) $= \frac{b-c}{a} \text{ X } 100\%$ (1)

2) Cellulose (%) $= \frac{c-d}{a} \text{ X } 100\%$ (2)

3) Lignin (%) $= \frac{d-e}{a} \text{ X } 100\%$ (3)

4) Ash Content (%) $= \frac{e}{f} \text{ X } 100\%$ (4)

Description:
a = The mass of the beginning sample

b = Dry sample residue after refluxing aquadest
c = Dry sample residue after refluxed H_2SO_4 0.5 M
d = Dry sample residue after soaking with 72% H_2SO_4 + 0.5 M H_2SO_4
e = Ash from sample residue after heating in furnace
f = The mass of the beginning sample [(evaporating dish + sample) - empty evaporating dish]

3. RESULTS AND DISCUSSIONS

3.1. Characteristics of Pineapple Peel Waste Raw Material

The chemical composition of the analyzed pineapple peel waste shows that the lignin content of 35.85% is one of the main challenges in the delignification process. Lignin, as a complex polymer that forms a protective structure on plant cell walls, serves as a barrier to the accessibility of cellulose and hemicellulose [9]. The relatively high lignin content requires an effective pretreatment method to break the lignin-cellulose bonds.

TABLE I. Characteristics of pineapple peel waste before delignification

Parameter	Result of Analysis
Lignin	35.85%
Hemicellulose	19.58%
Cellulose	10.45%
Ash Content	2.50%
Water Content	10.79%
Carbohydrate	30.03%

The hemicellulose content of 19.58% and cellulose content of 10.45% in this sample provide an idea of the potential biomass that can be converted after lignin removal. However, delignification efficiency must be considered to avoid damage to cellulose and hemicellulose components. The high water content (10.79%) and ash content (2.50%) also need to be considered in the delignification process, as they can affect heating efficiency and chemical reactivity during pretreatment. In addition, the total carbohydrate content of 30.03% indicates the potential of this biomass as a renewable energy source, which can be enhanced through effective lignin separation, thereby increasing the accessibility of carbohydrate components for bioethanol applications or other biochemical products.

3.2. Results of Delignification Process

The results of the delignification process showed a significant decrease in lignin content ($P < 0.05$), at P = 0,.028 as the duration of microwave irradiation increased (Figure 1). The longer the irradiation duration, the greater the energy absorbed by the molecules in the lignocellulosic chain, which leads to an increase in temperature in the cell matrix. This increase in temperature accelerates the reaction of breaking the ester and ether bonds between lignin and cellulose or hemicellulose, so that lignin is more easily detached from the plant cell wall structure [10]. In this process, microwave irradiation also increases the mobility of the added alkali ions (NaOH), which helps break the chemical bonds between lignin and carbohydrates [11, 12].

Extended irradiation increases heat penetration into biomass tissues and accelerates the deconstruction of lignin structures. However, although longer irradiation improves delignification efficiency, excessive exposure may affect the integrity of cellulose and hemicellulose [13, 14]. Within the experimental range evaluated in this study (15–45 minutes), the 45-minute irradiation produced the lowest lignin content, indicating the most effective delignification among the tested durations. However, this condition should not be interpreted as a definitive optimal duration, since only three irradiation times were investigated. Additional experiments with irradiation times beyond 45 minutes are therefore recommended to determine whether further lignin removal can be achieved and to establish the true optimal processing condition.

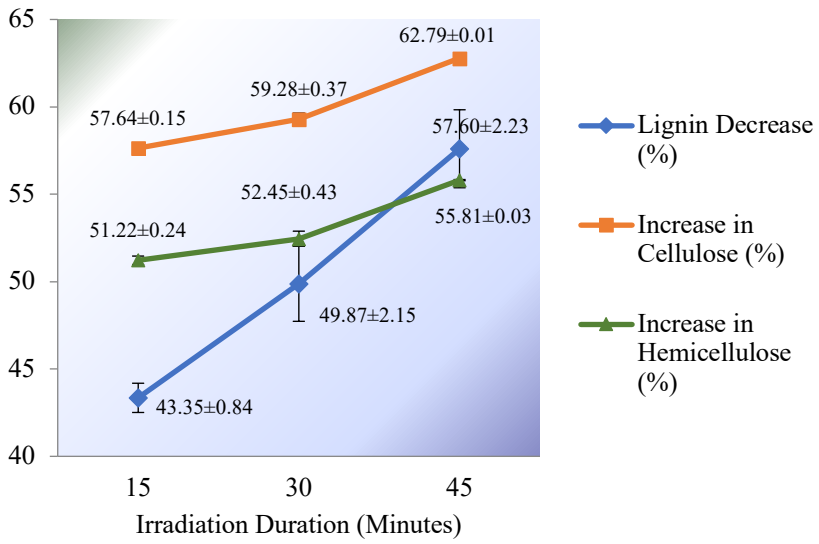

Figure 1. Test results after delignification using variations in microwave irradiation duration

Based on one-way ANOVA, cellulose and hemicellulose contents showed a significant increase ($P < 0.05$; $P = 0.001$ and $P = 0.003$, respectively) with increasing irradiation time. It is important to clarify that these increases do not indicate the formation of new cellulose or hemicellulose, but rather reflect a relative enrichment effect resulting from lignin removal. As lignin is progressively removed, the proportion of structural carbohydrates increases in the remaining biomass fraction. Therefore, the observed increase represents improved accessibility and a higher relative composition of cellulose and hemicellulose due to reduced lignin content, rather than the actual synthesis of these polymers [15].

In addition to compositional changes, the energy efficiency of the microwave-assisted alkaline treatment was considered. Using 45 W for 45 minutes to treat 50 g of dried pineapple peel, the estimated energy consumption was 0.03375 kWh, or approximately 0.000675 kWh per gram of biomass. These results indicate that the method is not only effective for delignification but also energy-efficient and potentially more sustainable than conventional heating approaches.

3.3. FTIR Result of Delignification Process

The functional groups of lignin, cellulose, and hemicellulose were identified through infrared spectra of pineapple peel waste samples before and after delignification. Before delignification, three absorbances with significantly different vibrational curves at 3280.1 cm^{-1}, 2914.8 cm^{-1}, and 1028.7 cm^{-1} were observed, while after delignification, there were only two prominent absorbances at 3310 cm^{-1} and 1028.7 cm^{-1}. The peak shift from 3280 cm^{-1} to 3310 cm^{-1} indicates a change in hydrogen interactions due to the degradation of lignin, which is rich in hydroxyl (O-H) groups in phenolic and aliphatic structures [16]. This spectrum can also overlap with O-H on cellulose and hemicellulose as polysaccharides [17]. In addition to peak shifts, noticeable changes in peak intensity were observed after delignification, indicating structural modification of the lignocellulosic matrix.

TABLE II. Results of wave numbers in the FTIR test

No	Wave Numbers (cm^{-1})			
	Before Delignification (Pineapple Peel)		After Delignification	
	Function Groups	Numbers	Function Groups	Numbers
1	O-H	3280,1	O-H	3310,0
2	C-H	2914,8	C - H	2892,4
3	C = O	1736,9		
4	C = C	1640,0	C = C	1625,1
5	C – O (ester)	1237,5	C – O (ester)	1312,0
6	C – O - C	1028,7	C – O - C	1028,7

The aliphatic C-H stretching vibration in pineapple peel appeared at 2914.8 cm^{-1}, while after delignification, this vibration shifted to 2892.4 cm^{-1}, indicating the degradation of some organic components, where the C-H stretching in the aliphatic methylene group, which can be derived from fatty acids contained in the lignin preparation [18]. Moreover, the intensity of the C–H band

decreased after treatment, suggesting the partial removal of lignin-associated aliphatic structures during alkaline microwave delignification.

The C=O (ester) peak of lignin at 1736.9 cm^{-1} also disappeared after delignification, indicating the breakdown of such groups during the process. Wavelengths of 1640.0 cm^{-1} in pineapple peel and 1625.1 cm^{-1} after delignification indicate aromatic C=C vibrations, typical of lignin. A clear reduction in the intensity of this aromatic band was observed after treatment, providing quantitative support for the decrease in lignin content detected in the compositional analysis. The peak at 1028.7 cm^{-1} in both samples indicates the absorption (C–O–C stretching) of glycosidic bonds in cellulose and hemicellulose related to glucose ring stretching [8, 19]. The relatively strong and stable intensity of this peak after delignification suggests that the polysaccharide backbone of cellulose and hemicellulose remained largely preserved during the treatment.

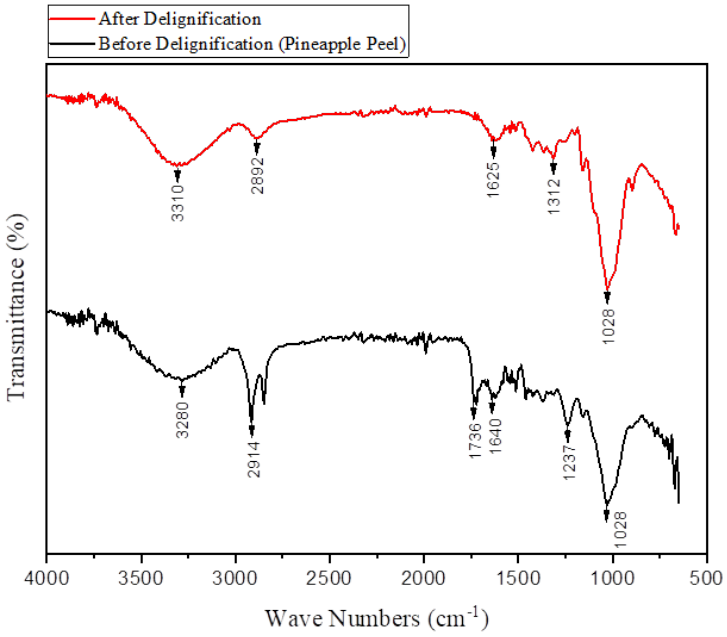

Figure 2. FTIR results of pineapple peel waste before and after delignification process

The FTIR results in Figure 2 show that the microwave irradiation treatment for delignification successfully removed most of the lignin. This conclusion is supported not only by the disappearance of characteristic lignin peaks (such as C=O at 1736.9 cm^{-1}) but also by the significant reduction in the intensity of aromatic lignin-related bands, while carbohydrate-associated peaks remained relatively stable. As a result, the spectra confirm that lignin was selectively removed while the structural framework of cellulose and hemicellulose was largely maintained, as indicated by the persistent C–O–C peak at 1028 cm^{-1}.

3.4. SEM Result of Delignification Process

The SEM images of pineapple peel samples prior to delignification revealed a relatively compact and layered surface morphology, in which the fibrillar structure remained embedded within the lignin–hemicellulose matrix. This morphology is consistent with the findings of Dong et al. (2019) in their study on lignocellulosic biomass pretreatment, in which untreated biomass exhibited a dense, continuous surface with limited pore openings. Such structural integrity is attributed to the dominance of lignin and hemicellulose, which envelop the cellulose microfibrils. The smooth, compact surface structure indicates low accessibility to chemical reagents or enzymatic attack, as strong intermolecular interactions among lignocellulosic components remain intact [20]. As shown in Figure 3A1 (500× magnification, scale bar = 10 µm), the untreated biomass exhibits a relatively smooth and compact surface with minimal visible pores. At higher magnification (Figure 3A2, 1500×), the fibrillar components remain tightly packed within the lignin–hemicellulose matrix, indicating limited structural disruption prior to treatment.

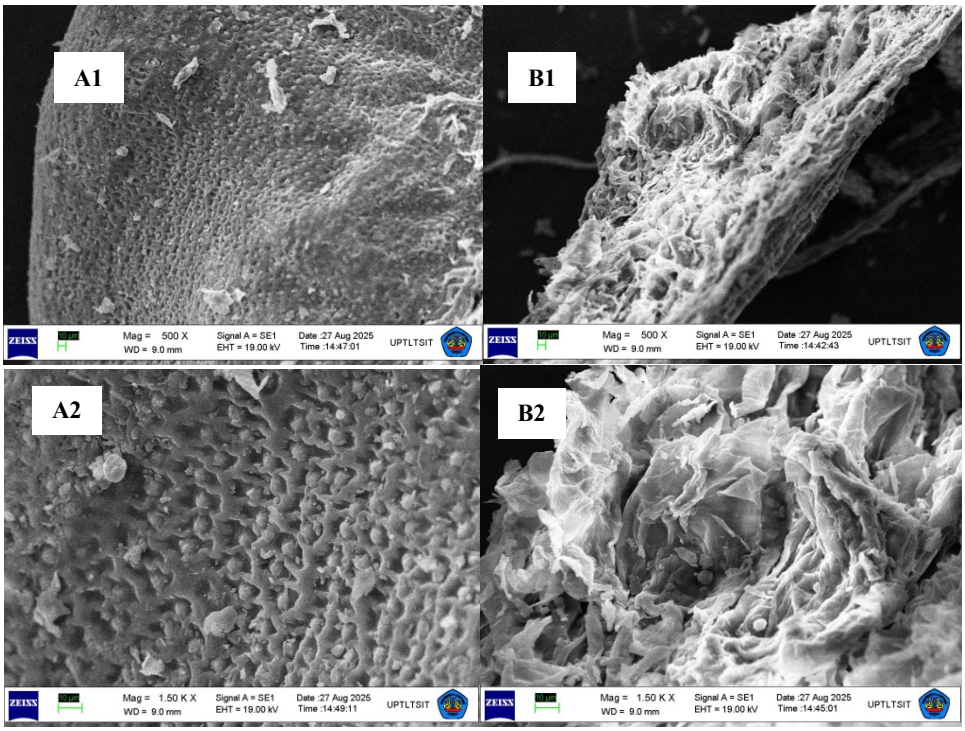

Figure 3. SEM micrographs of pineapple peel waste (A) before and (B) after microwave-assisted alkaline delignification at different magnifications: (A1, B1) 500× and (A2, B2) 1500× magnifications (scale bar = 10 µm).

Following microwave-assisted alkaline delignification, SEM micrographs showed pronounced morphological alterations, characterized by a rougher, more irregular surface, along with the formation of cracks and inter-fiber voids. These observations are consistent with those reported by Shakeel et al. (2024), who showed that alkaline pretreatment induces substantial structural disruption of lignocellulosic biomass, leading to increased porosity and enhanced exposure of cellulose fibrils. At 500× magnification (Figure 3B1), larger surface cracks and voids are clearly distributed across the biomass surface. Meanwhile, at 1500× magnification (Figure 3B2), finer microcavities and partial separation of fibrillar structures become more evident, indicating significant disruption of the lignocellulosic matrix after delignification. The presence of these micrometer-scale cavities suggests the removal of amorphous components, particularly lignin, from the cell wall matrix, thereby increasing the specific surface area and microporous structure. Furthermore, the peeling effect and layer delamination observed after delignification have also been documented as clear indicators of lignocellulosic matrix degradation resulting from alkaline pretreatment [4, 21].

4. CONCLUSIONS

Microwave-assisted alkaline treatment effectively enhanced the delignification of pineapple peel waste. At a treatment duration of 45 minutes and a microwave power of 45 W, lignin content decreased by 57.6%, while cellulose and hemicellulose contents increased by 62.79% and 55.8%, respectively. FTIR and SEM analyses confirmed lignin removal, structural disruption, increased surface irregularity, and enhanced porosity. These findings indicate the potential applicability of microwave-assisted alkaline treatment for valorizing pineapple peel waste at larger scales, while further studies are needed to assess process scalability, energy requirements, and economic feasibility.

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