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Synthesis and Characterization of Corn Husk (*Zea Mays* L.) Cellulose Using Microwave-Assisted Extraction (MAE)

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Abstract

This research focuses on synthesizing cellulose from corn husks using microwave-assisted extraction (MAE), which aims to produce cellulose from corn husks through liquefaction, delignification, and bleaching processes. The total time required is only 14 minutes, thus shortening the process time. This study used three power variations: 450 W, 600 W, and 800 W. The results showed that the optimum conditions occurred at 800 W power. Hence, the higher the power caused the high cellulose content obtained, which amounted to 75.23% and decreased the lignin value to 15.78%, along with the increase in microwave power. FTIR analysis also confirmed the cellulose results obtained and indicated that the lignin groups were weakened or lost. Furthermore, morphological analysis of cellulose fibers by SEM showed that the surface of cellulose fibers was rough, oval, and irregular due to cracks caused by erosion. Then, the result of cellulose crystallinity using XRD was 45.8%. Compared to conventional methods, microwave-assisted cellulose synthesis exhibits better and more promising potential. This is because microwave radiation can be used to improve process time efficiency and achieve higher yields. Moreover, the proposed method is easier, faster, and straightforward.

1. Introduction

Biomass has abundant reserves and is environmentally friendly. Biomass accounts for about 1.5×10^{12} tons of the total biomass production per year. Biomass is considered a renewable resource [1], and the proportion of cellulose as the main component comes from cell-based lignocellulose, which forms the plant wall along with hemicellulose, lignin, pectin, and wax [2]. One of the sources of cellulose is corn husk waste, known for its high cellulose fiber content. The chemical composition of corn husks contains 5.09% ash, 4.57% cyclohexane alcohol, 15% lignin, and 44.08% cellulose [3]. Corn husks contain relatively high cellulose compared to corn cobs, with 28.77% cellulose [4]; corn stalks contain 42.6% cellulose [5], and corn leaves contain 38.66% cellulose [6], so corn husks have the potential to be used as a source of cellulose raw materials [7].

Cellulose can be synthesized by various methods, such as using alkalis and organic alkalis [8, 9], acid hydrolysis [10, 11], enzymatic [12, 13], steam explosion [14], and reactive extrusion [15]. All those methods are conventional, time-consuming, inefficient in solvent usage, and necessitate elevated temperatures. In particular, the supercritical method is conducted under high pressure, incurring significant costs. Therefore, there is a need for an alternative approach, a straightforward and cost-effective method employing microwave-assisted extraction (MAE) [16]. MAE works by directing microwave radiation to a material, causing the molecules within the material to absorb electromagnetic energy. Microwave heating is more targeted and selective, generating heat from within the material rather than transferring it from outside. This method has several advantages over conventional methods, including producing high yields, low costs, high selectivity, and faster extraction times [17].

Zou *et al.* [18] reported that extraction using the conventional method achieved 67% recovery within one hour of extraction time, while the MAE method achieved 82% recovery within 132 seconds. The same thing was

also expressed by Xie *et al.* [19], who conducted research utilizing microwaves as an alternative to conventional heating. Their findings indicated that microwave heating could accelerate the reaction rate. This occurs because microwave energy directly interacts with the reaction mixture, leading to a faster, energy-efficient, and more effective heating process. Conversely, the traditional heating systems require more energy [20].

The same opinion was expressed by Orhan *et al.* [21] and Khandanlou *et al.* [22] reported that using microwaves was superior to conventional methods. Iliyin *et al.* [16], Xie *et al.* [19], and Li *et al.* [23] revealed that microwave heating has the ability to increase cellulose content while reducing lignin content. Based on previous studies, microwave-assisted extraction (MAE) has not been employed for cellulose synthesis from corn husks. Therefore, this study aims to synthesize cellulose from corn husks using MAE and evaluate the impact of microwave power on cellulose content.

2. Experimental

2.1. Materials and Tools

The materials were corn husk from Tanah Laut, South Kalimantan, methanol (Merck, 99.8%), H_2SO_4 (Merck, 96-97%), glycerol (Merck, 85%), H_2O_2 (Merck, 30%), NaOH (Merck), and distilled water.

The tools used in this research were Pyrex glassware (measuring cup, conical beaker, beaker, stirring rod, porcelain cup, and volumetric flask), oven, microwave (Samsung, ME731K/XEU Solo, 20 L), Fourier Transform Infrared (FTIR) spectrometer (Merck Shimadzu type IRPrestige 21) in the Laboratory of Chemistry Department FMIPA IPB University, Scanning Electron Microscope (SEM) (Zeiss Evo) in the Laboratory of Forestry Research Center, X-ray Diffraction (XRD) (Rigaku Benchtop Miniflex 600) in the Laboratory of FMIPA Yogyakarta State University.

2.2. Synthesis of Cellulose from Corn Husk

The method was modified from previous studies [16]. Synthesis was initially conducted by cleansing and sun-drying corn husk. Subsequently, the dried corn husks were finely pulverized using a grinding machine and sieved to achieve a finer powder. Then, 10 g of corn husk powder was put into a beaker and added with 150 mL of solution (methanol: glycerol = 1:2) and 35 mL of 1.75% H_2SO_4. The mixture was heated using MAE for 3 minutes and then filtered. The residue was dried in an oven at 105°C. After drying, the residue was added with 10% NaOH and heated using MAE for 3 minutes. The resulting slurry was filtered and washed with distilled water until the pH was neutral, then dried in an oven at 50°C until constant weight. The residue was added with 5% H_2O_2 and heated for 4 minutes using MAE. The resulting pulp was filtered and washed with distilled water until the pH was neutral. The cellulose was dried in an oven at 60°C and then stored in a desiccator. The power transformers of MAE used were 450 W, 600 W, and 800 W.

2.3. Fourier Transform Infrared (FTIR) Spectroscopy Analysis

Before analysis, 1 mg of the sample was mixed with 200 mg of KBr powder and compressed into a transparent film with a pressure of 10 tons (2000 psi). The sample was placed in a holder to measure infrared absorption in the wavenumber range of 4000-400 cm^{-1}. From the analysis, a diffraction pattern was obtained, showing the relationship between wavenumber and intensity.

2.4. Scanning Electron Microscopy (SEM) Analysis

Morphological analysis was performed using SEM. The pattern was fixed to the base with double-sided tape and then vacuum-coated with gold. Afterward, the sample was placed in the SEM, and then the topography image was observed and magnified 5000 times.

2.5. X-ray Diffraction (XRD) Analysis

Samples were analyzed by wide-angle XRD (Bruker D 5000). Data were generated with a diffractometer at 40 kV and 30 mA Cu-K electricity (= 1.54 A), with a time step of 2.0 s and an angular range of 2θ = 10–40°. The crystallinity index (Cr l) was determined using Equation (1).

$$\text{Cr l} = \frac{I_c}{I_c + I_a} \times 100\% \quad (1)$$

where, I_c is crystal intensity, and I_a is amorphous intensity.

3. Results and Discussion

Cellulose synthesis from corn husks using microwave technology can be accomplished in 14 minutes, encompassing three distinct stages: liquefaction, delignification, and bleaching. This represents a significant time savings compared to the conventional method, where the delignification step extends over 9 hours, and the subsequent bleaching stage consumes 15 minutes, resulting in a total processing time of 9 hours and 15 minutes [24]. According to Handaratri and Yuniati [17] and Xie *et al.* [19], microwave heating offers advantages such as volumetric, cost-effective, and efficient heating, resulting in a faster extraction process and making it a viable alternative to traditional heating methods.

Liquefaction removes waxes, colorants, fats, tannins, and other organic matter [25]. The liquefaction process prevents the formation of condensation products with lignin during synthesis. Waxes, fats, tannins, and colorants are extracted with organic solvents. During liquefaction, methanol, and glycerol cause the breakdown of ester bonds between lignin and carbohydrates and the depolymerization of lignin, while the addition of sulfuric acid serves to activate oxygen atoms in glycosidic bonds in cellulose chains, thus facilitating the breaking of bonds between cellulose and lignin in the delignification process heating.

The delignification process employed NaOH, which served the purpose of breaking the lignin structure, thereby liberating the cellulose present in the material, including both its crystalline and amorphous components. Additionally, this process lead to the

separation of some lignin and hemicellulose components and the causation of swelling of the cellulose structure [26]. During the heating process, hemicellulose undergoes a softening phase, resulting in the fibers previously separated becoming sticky. In addition, NaOH also dissolves extractive substances, such as pectin, wax, and other impurities. Figure 2 depicts the delignification process employing NaOH, where the chemical reaction involves the cleavage of ester bonds between carbohydrates and lignin.

In this study, 10% NaOH was utilized to effectively permeate the amorphous cellulose region for the optimal dissolution of cellulose. Permatasari *et al.* [27] used 4% NaOH for delignification to remove 5.81% of lignin, and maximum solubility occurred at a concentration of 8-10% NaOH. In the delignification process, the dissolution of lignin is marked by a change in color to blackish brown. However, this indicates that lignin compounds remain, so the lignin must be separated by the bleaching stage.

The remaining lignin is dissolved during the bleaching process by applying an oxidizing agent, H_2O_2. Hydrogen peroxide is chosen for its strong oxidizing properties, making it an effective choice for bleaching. Peroxide anion (HOO^-) is an active species that removes lignin chromophores. Peroxide anions are highly nucleophilic and attack vinyl and carbonyl groups on lignin molecules, converting them into chromophore-free species [28]. During bleaching, long lignin chains are broken down into shorter ones, resulting in lignin dissolution during leaching. The process of lignin decomposition by H_2O_2 compounds is depicted in Figure 3. Equations (2) to (4) are the reaction mechanism of the bleaching process using hydrogen peroxide.

$$H_2O_2 \rightarrow 2OH\cdot \quad (2)$$

$$H_2O_2 + OH\cdot \rightarrow OOH\cdot + H_2O \quad (3)$$

$$OOH\cdot + OH\cdot \rightarrow H_2O + 2O_n \quad (4)$$

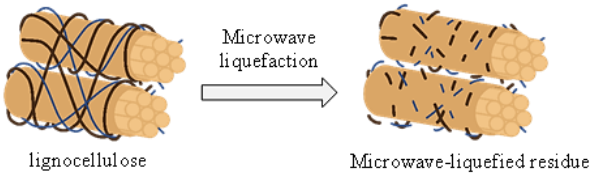

Figure 1. Liquefaction process

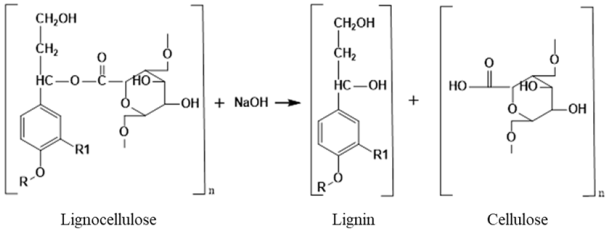

Figure 2. Delignification process [29]

Hydrogen peroxide oxidizes non-phenolic lignin units by releasing electrons and forming cationic radicals, which undergo chemical decomposition. Non-phenolic units account for approximately 90% of the lignin structure. Hydrogen peroxide can break the Cα-Cβ bonds of lignin molecules and can open lignin rings and carry out other reactions. Hydrogen peroxide catalyzes the oxidation of non-phenolic aromatic compounds of lignin to form aryl cationic radicals. Hydrogen catalyzes the oxidation of non-phenolic lignin compounds by converting veratrol to veratraldehyde. Peroxide is a strong oxidant and can also oxidize phenolic compounds, amines, aromatic ethers, and polycyclic aromatic compounds. H_2O_2-catalyzed oxidation of the lignin substructure begins with the splitting of electrons from the aromatic ring of the donor substrate. It generates arylcationic radicals, which undergo various post-enzymatic reactions [30].

Besides being a bleaching agent, H_2O_2 is an environmentally friendly chemical that does not damage cellulose compared to NaOCl. Chlorine compounds and their derivatives give rise to several other chlorinated compounds that cause environmental problems [31]. This bleaching process makes cellulose fibers white, reducing non-cellulose components and other impurities such as hemicellulose and lignin. The color change that occurs can be seen in Figure 4.

Based on Figure 4, it can be seen that the color of corn husk powder changes to reddish brown in the liquefaction process. This indicates that the O atom in the glycosidic bond has been activated. In the delignification process, the color becomes blackish brown, indicating the separation of some lignin and hemicellulose. During bleaching, the fiber color turns white, indicating that lignin and hemicellulose are reduced. This color change indicates that most non-cellulose material and impurities have been removed.

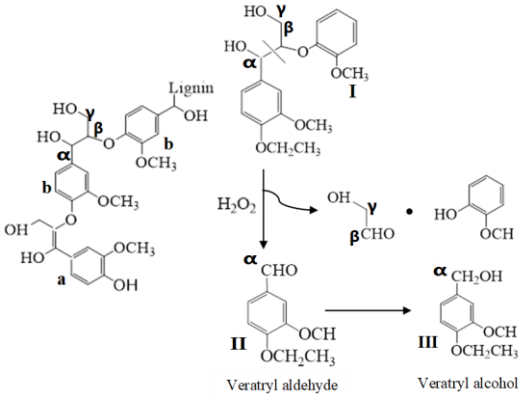

Figure 3. Reaction of lignin decomposition by H_2O_2 [32]

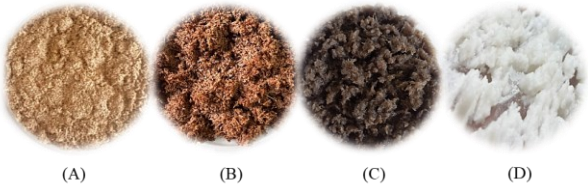

Figure 4. A) corn husk powder, (B) liquefaction result, (C) delignification result, (D) bleaching result

This study synthesized cellulose at varying microwave powers of 450 W, 600 W, and 800 W. According to the analysis results, a significant reduction in lignin content was observed, decreasing from an initial 36.92% to 0.94% at a power level of 800 W (Figure 5). The success of cellulose synthesis using microwave irradiation was evaluated based on the decrease in lignin content and the increase in cellulose content obtained from the initial material. The cellulose content increased from 36.92% of the starting material to 72.1% at 450 W power and increased to 75.23% at 800W power (Figure 5). Thus, this study showed that the highest cellulose content of 75.23% in corn husk was achieved at 800 W power.

The outcomes are notably superior to the conventional method, as revealed by Nawangsari [33], which yielded a cellulose content of 26.03%. In comparison, Setyaningsih *et al.* [24] reported a cellulose content of 18.12%. The increase in cellulose content is directly proportional to the increase in microwave power. According to Zhou *et al.* [34], microwave heating has high efficiency and selectivity for biomass processing, causing the high purity of cellulose obtained in this study.

According to the results, using MAE in the cellulose synthesis process demonstrated its effectiveness and efficiency in achieving better outcomes. This advantage is attributed to the rapid and controlled microwave heating mechanism. Additionally, microwave exposure accelerates chemical reactions by directly and uniformly heating the material, leading to the even breakdown of non-cellulose molecules. The microwave heating process relies on the speed of rotation and collision of polar molecules, leading to the conversion of kinetic energy into heat.

The heating effect is produced by the electric field of the microwaves, forcing the dipoles to rotate and collide with each other. Increased performance results in increased energy and temperature, which damages fibers and causes the breakdown of lignin [20]. During the microwave interaction process with molecules in NaOH and H_2SO_4 solutions, it causes a decrease in hemicellulose content that binds cellulose and decreases lignin content in cell walls that block cellulose.

On the other hand, conventional methods result in lower and uneven heating rates as heating occurs through the reactor walls, resulting in non-uniform heat distribution and, consequently, non-uniform molecular damage. The success of cellulose synthesis is further monitored through FTIR analysis. FTIR is a valuable method for investigating the primary structure and chemical alterations in lignocellulose derived from biomass.

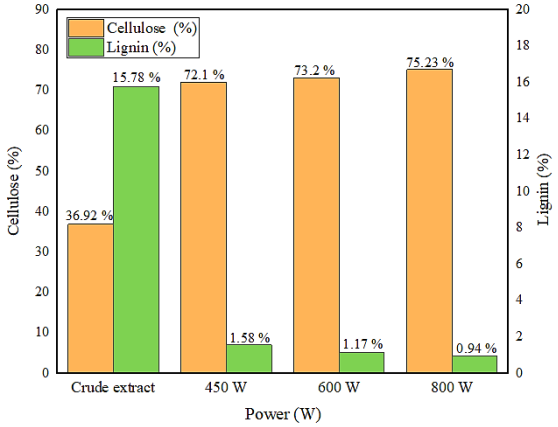

Figure 5. Effect of power on chemical components

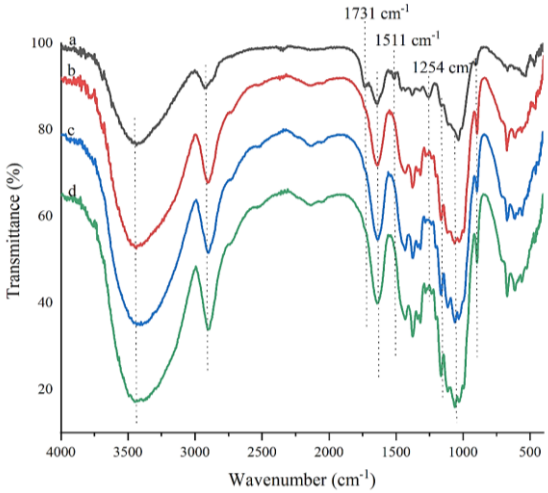

Figure 6. FTIR spectra of (A) corn husk powder, (B) cellulose at 450 W, (C) cellulose at 600 W, and (D) cellulose at 800 W

Table 1. Wavenumbers of FTIR spectra of corn husk and cellulose samples at 450 W, 600 W, and 800W power

Wavenumber (cm^{-1})	Functional group	Wavenumber (cm^{-1})			
		Corn husk powder	Cellulose (450 W)	Cellulose (600 W)	Cellulose (800 W)
900 [35]	C-H at β-glycosidic	902	903	903	903
1030 [36]	C-O	1030	1023	1027	1025
1161 [36]	C-O-C	1163	1167	1168	1168
1253 [36]	C=O of hemicellulose and lignin, C-O-C vibrations	1254	-	-	-
1512 [37]	C=C of aromatic ring lignin	1511	-	-	-
		1740	-	-	-
1600-1640 [35]	O-H bending	1637	1637	1632	1635
1730 [38]	C=O of hemicellulose and lignin	1731	-	-	-
2800-3000 [35]	C-H of cellulose	2913	2898	2899	2899
3300-3700 [35]	O-H of cellulose	3439	3435	3430	3437

The FTIR instrument was employed to analyze corn husk powder and cellulose samples at power levels of 450 W, 600 W, and 800 W. In Figure 6, discernible peaks are observed, signifying functional groups within their wavenumber regions. The FTIR analysis results are presented in Table 1.

Figure 5 and Table 1 show peak data as symbol markers for the presence of functional groups in the range of 4000 cm^{-1} to 500 cm^{-1}. The presence of cellulose is suggested by the peak at the wavenumber of 902 cm^{-1}, which indicates the presence of CH groups (proximity to 900 cm-1 is characteristic of CH groups). Meanwhile, the peak at 903 cm^{-1} signifies the presence of cellulose glucose units, particularly β-1,4 glycosidic bonds [37]. The increase in absorption at these wavenumbers is an excellent example of cellulose samples. A wavenumber of 1030 cm^{-1} indicates the presence of the CO group [39] and the stretching of the COC pyranose ring vibration (approximately 1161 cm^{-1}). The peaks at 1731 cm^{-1} and 1254 cm^{-1} are the COC vibrations representing carbonyl (C=O) lignin segments and the hemicellulose and aromatic ether bonds in lignin [38].

Moreover, the 1731 cm^{-1} peak in corn flour represents the carboxyl linkage between the acetyl and uronic acid ester groups in hemicellulose or the ferulic acid and p- coumaric acid ester groups in lignin. The decrease of this peak in the samples subjected to delignification and bleaching processes indicates that the ester bonds of non-cellulosic material are broken [40]. A peak of 1254 cm^{-1} in corn bran powder indicates the presence of lignin. The same results were observed at wavenumber 1511 cm^{-1}, indicating C=C for aromatic ring lignins [41]. In the spectrum of corn husk cellulose, a notable absence of lignin absorption peaks or weak absorption is observed. This observation concludes that a significant portion of the lignin and hemicellulose components have been reduced [38].

The surface morphology structures of cellulose after delignification and bleaching are shown in Figure 7. This observation aligns with the findings reported by Jufrinaldi [31] and Tajalla *et al.* [42]; SEM analysis revealed that the morphology of the cellulose derived from corn husks is fibrous. Cellulose fibers frequently aggregate, causing the apparent enlargement of fiber size. This aggregation occurs due to the melting process, leading to a rougher, irregular, and hollow surface. As per the study of Li *et al.* [43], this phenomenon suggests the release of a significant portion of hemicellulose. Nevertheless, there are still numerous small fragments remaining. The alkaline microwave delignification process also leads to the disruption and removal of the outer surface, which exposes the interior fibers and generates cracks due to erosion. According to Singh *et al.* [44], this erosion is attributed to the microturbulence and sound waves generated by cavitation bubbles.

The XRD pattern shows wavy dotted lines due to the refraction of X-rays by the crystals in the test material particles. The peak of the refraction line indicates the crystallinity of the material. In Figure 8, the XRD diffraction pattern shows that cellulose has the highest peaks at intensities of 2θ = 16.6°, 22.26°, and 35°. The typical cellulose patterns obtained correspond to the JCPDS (Joint Committee on Powder Diffraction Standards) data for cellulose.

The results obtained are similar to those of Fawcett *et al.* [45]. Here, typical modes for cellulose I with broad amorphous and crystalline peaks are around 2θ = 16°, 22°, and 35°. Cellulose adopts a crystalline structure due to the interaction of hydrogen bonding and van der Waals forces between adjacent molecules. The percent crystallinity of cellulose is calculated by looking at the integrated area of the crystals and dividing by the integrated area of the crystalline and amorphous components [45]. Thus, the calculated crystallinity index gave a quantitative value of 45.8% for the crystallinity of cellulose in the diffractogram.

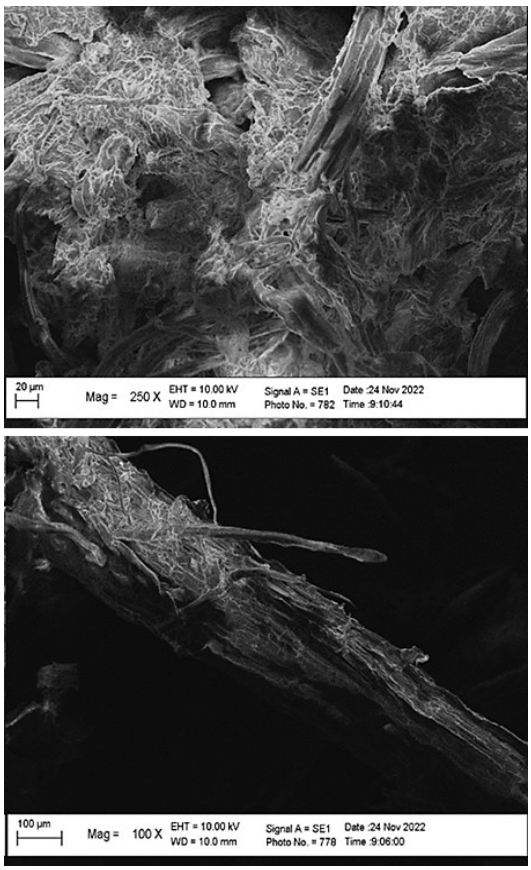

Figure 7. SEM images of cellulose fiber

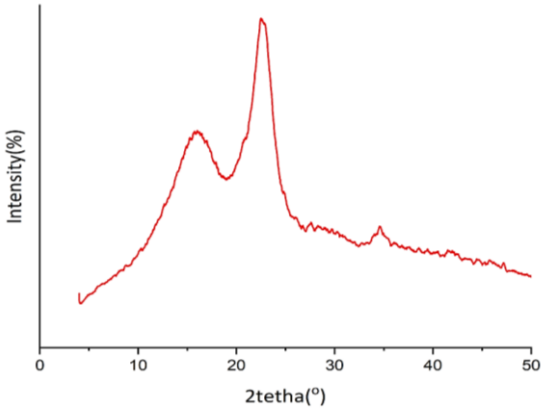

Figure 8. XRD diffractogram of cellulose

4. Conclusion

Cellulose was successfully synthesized from corn husk using MAE and proved notably effective and efficient compared to conventional methods. The results showed that the lignin content decreased to 0.98%, and cellulose increased to 75.23% at the optimum power condition of 800 W. These results were also confirmed by FTIR spectra, which showed the presence of cellulose compounds and the disappearance of lignin peaks. Analysis of cellulose surface morphology using SEM showed that cellulose fibers have rough, irregular, and hollow surfaces. The crystallinity of cellulose analyzed using XRD was 45.8%. The resulting cellulose possesses a broad range of potential applications, including its use as an alternative raw material in industries such as textiles, pulp production, bioplastics, innovative packaging, and various other sectors.

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