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Cellulose Derived from Alkaline Treatment of Garlic Waste: Effect of Sodium Hydroxide Concentration on Product Yield and Characteristics

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Abstract

Garlic waste, including straws, bulbs, and skins, is a common agricultural residue contributing to domestic waste. In fact, the cellulose-rich garlic waste can be converted into various value-added products through proper processing techniques. This study aimed to investigate the influence of sodium hydroxide (NaOH) concentration on the characteristics of the cellulose extracted from garlic waste using alkaline treatment. Various concentrations of NaOH solutions, namely 2%, 7%, 14%, and 20% wt., were used during the garlic waste alkaline delignification process. FTIR spectra of the delignified garlic waste sample revealed transmittance changes with the increase in NaOH concentration, indicating an observable reduction in lignin content. The cellulose resulting from garlic waste delignification using a NaOH concentration of 20% wt. exhibited a needle-like structure, while the garlic fibers displayed an amorphous structure with a clean surface. The highest cellulose extract yield from the alkaline delignification of garlic waste was 62.7%, achieved at an NaOH concentration of 2% wt. It was also observed from thermogravimetric analysis (TGA) that the decomposition temperature of the alkaline delignified garlic waste increased significantly due to efficient hemicellulose and lignin removal, which increased cellulose content. These findings demonstrate the potential of garlic waste as a cellulose source and its potential application for producing derivative products, such as thin films, optical fibers, and bioplastic raw materials.

Keywords: alkalization; cellulose; delignification; garlic waste

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INTRODUCTION

Indonesia is an agrarian country, with a significant portion of its population engaged in the agricultural sector. Consequently, agricultural waste has become a prominent issue within this domain. Agricultural waste refers to the residual or unused byproducts generated from agricultural activities. Waste management remains a significant challenge in Indonesia, as household waste accounts for 51% of total waste generation. Food waste and plastic contribute 28% and 23%, respectively. Waste handling at the household level remains inadequate, with only 1.2% of waste being recycled, while 68% is disposed of through open (Badan Pusat Statistik, 2018).

One type of agricultural waste that contributes to household waste is garlic waste, including garlic straws, bulb remnants, and peels. These materials are typically bulky, fibrous, poorly digestible, and low protein. Garlic waste is a globally available biological residue that can be readily sourced from the food processing industry (Choi et al., 2021; Lee et al., 2020). Approximately 76% of a garlic bulb's mass comprises cloves, with the remaining 24% consisting of its inner and outer peel layers (Kallel & Ellouz, 2017). These fibrous components are composed primarily of cellulose, hemicellulose, and lignin, and they hold great potential as renewable sources of biopolymers (Mahardika et al., 2018; Phanthong et al., 2018). Garlic waste is highly abundant and easily obtainable, and its utilization is promising, especially considering the projected garlic consumption in Indonesia, which is expected to reach 526,770 tons by 2024 (Agustina, 2020).

Cellulose is a renewable and abundant biopolymer widely utilized across various sectors, including energy, food, and environmental applications. Cellulose is a linear homopolysaccharide consisting of repeating $\beta\text{-D-glucopyranose}$ monomers, each bearing three hydroxyl functional groups (Xie et al., 2016). In nature, cellulose forms the primary structural component of plant cell walls and is associated with polysaccharides such as hemicellulose and lignin (Gian et al., 2017).

Cellulose has diverse applications, such as in the paper industry, pharmaceuticals (Syamsu, 2013), textile additives (Eriningsih et al., 2011), reinforcement in bioplastics (Maryam et al., 2019), and membrane production (Istirokhatun et al., 2015). One of the essential cellulose derivatives is cellulose acetate, which has excellent potential in material applications, particularly as a membrane material, due to its asymmetric structure and ultra-thin active layer that effectively filters solutes (Husni et al., 2018).

Alkaline treatment is a standard chemical method for extracting cellulose from lignocellulosic biomass (Kallel et al., 2016). This treatment removes non-cellulosic components such as hemicellulose and lignin, enhancing the resulting fibers' mechanical, physical, and thermal properties (Syafri et al., 2019). Sodium hydroxide treatment improves natural fiber

characteristics. For instance, Kallel et al. (2016) reported that a 2% NaOH treatment yielded a cellulose content of 63%, reducing lignin from 6.3% to 4.6%. Similarly, Hernández-Varela et al. (2021) observed the disappearance of a 1740 cm $^{-1}$ FTIR peak—associated with acetyl groups and α -keto carboxylic acids—after 5% NaOH treatment at 80°C, indicating hemicellulose and lignin removal. Reddy & Rhim (2018) extracted cellulose from garlic stalks and skins using 17.5% NaOH at 30°C, resulting in cellulose contents of 49.8% and 42.7%, respectively.

This study aims to investigate the effect of varying NaOH concentrations on the characteristics of cellulose extracted from garlic waste and to evaluate its potential for further applications in membrane or bioplastic synthesis.

RESEARCH METHOD

Materials

Garlic waste, including straws, bulbs, and skins, was collected from household sources in Way Huwi, South Lampung. Meanwhile, analytical-grade NaOH (99%, Merck) and distilled water were used throughout the experiment.

Preparation and Delignification Process

Garlic waste was washed to remove dirt and other contaminants before it was further sun-dried for three days until completely dry. The dried material was ground into a fine powder using a grinder and sieved through a 50-mesh screen to obtain uniform garlic waste powder.

The powdered garlic waste was soaked in NaOH solution for 4 hours with a solid-to-liquid ratio of 5:100 (w/v). The extraction was conducted using NaOH concentrations of 0%, 2%, 8%, 14%, and 20% wt. at a temperature of 60°C. After soaking, the samples were filtered, and the solid residues were rinsed repeatedly with distilled water until they were free from residual alkali. The filtered solids were then dried in an oven at 60°C for 12 hours.

Characterization

Functional Groups Analysis

Fourier-transform infrared (FTIR) spectroscopy was used to identify the functional groups attached to the fiber. In this study, the FTIR spectra of garlic waste fibers treated with different NaOH concentrations were obtained using a Varian 3100 Excalibur Series FTIR spectrometer (USA). Spectra were recorded in the range of 400–4000 cm⁻¹.

Morphological Analysis

Scanning Electron Microscopy (SEM) was used to examine the surface morphology of the garlic waste fibers. Images were captured using a JEOL JSM-6510 LA (Japan) scanning electron microscope. Microstructural observations of the fiber and cellulose microfibers were conducted under an accelerating voltage (Vacc) of 10 kV and an emission current (Ie) of 10 μA . Before imaging, samples were coated with osmium (Os) using a vacuum sputter coater.

Thermogravimetry Analysis

Thermal degradation behavior of the fiber samples was evaluated using a thermogravimetric analyzer (TGA/DSC 1, Mettler Toledo, Schwarzenbach, Switzerland). Approximately 11 mg of each specimen was heated from ambient temperature up to 500 °C at a heating rate of 10 °C per minute, under a nitrogen environment delivered continuously at a 50 cm³/min flow rate.

Yield

The yield measures the percentage of delignified garlic waste resulting from the alkalization process. It was calculated using equation (1).

$$\% \ yield = \frac{w_a}{w_b} \times 100\% \tag{1}$$

(1)

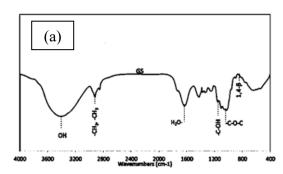
where,

 w_a = weight of sample before alkalization

 w_b = weight of sample after alkalization

RESULTS AND DISCUSSION Functional Groups Profile of Garlic Waste Fiber

FTIR analysis was conducted to observe the possible chemical structural changes in garlic waste fibers after alkaline treatment. The observed vibrational peaks are illustrated in Figure 1, and their corresponding wave numbers are summarized in Table 1.



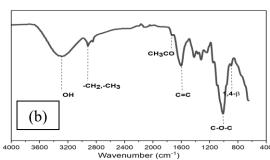


Figure 1. FTIR spectra of garlic waste fiber samples: (a) this study, (b) previous study by Kallel et al. (2016)

These structural changes are indicative of lignin removal, which leads to an increase in cellulose content. The FTIR spectra of untreated samples exhibited numerous unique peaks at various infrared

wave numbers, representing the major functional groups in lignocellulosic fibers: cellulose, hemicellulose, and lignin. For example, the β-1,4glycosidic linkage peak is associated with cellulose, the C=C bond indicates lignin, and the CH₃CO (acetyl) group is characteristic of hemicellulose. The hydroxyl (OH) group is commonly found in all major lignocellulosic components, with a higher concentration in cellulose.

Table 1. Functional groups identified in garlic waste fiber samples

| Eurotional Cusus | Wave number (cm ⁻¹) | | |
|----------------------|---------------------------------|------------|--|
| Functional Group | Kallel, dkk. 2016 | This study | |
| Cellulose (OH) | 3406- | 3327 | |
| | 3420 | | |
| Cellulose (1,4-β) | 904 | 892 | |
| Cellulose (C-O-C) | 1048 | 1008 | |
| Lignin (C=C) | 1630 | 1603 | |
| Hemicellulose | - | 1739 | |
| (CH ₃ CO) | | | |
| C-H Stretching | 2897- | 2907 | |
| - | 2923 | | |
| C-OH Bond | 1118 | 1156 | |

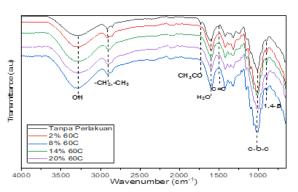


Figure 2. Chemical structure changes of garlic fiber treated with varying NaOH concentrations (60 °C, 300 rpm, 180 minutes)

Table 2. FTIR transmittance data for various NaOH concentrations at 60°C, 300 rpm for 180 minutes

| NaOH | Transmittance (%) | | | | |
|-------------------|--------------------|--------------------|--------------------|-------------------|--|
| Concentrati on | 1,4-β | C=C | CH ₃ CO | ОН | |
| | (892 | (1603 | (1739 | (3327c | |
| | cm ⁻¹) | cm ⁻¹) | cm ⁻¹) | m ⁻¹) | |
| Untreated | 87.7 | 94.41 | 95.87 | 90.39 | |
| | 9 | | | | |
| 2% wt. | 84.9 | 93.34 | 96.85 | 87.68 | |
| | 4 | | | | |
| 8% wt. | 83.1 | 93.09 | 96.74 | 86.11 | |
| | 5 | | | | |
| 14% wt. | 85.5 | 94.08 | 97.57 | 84.86 | |
| | 3 | | | | |
| 20% wt. | 81.7 | 92.30 | 95.62 | 84.38 | |
| | 5 | | | | |

The decrease in transmittance value at $892 \, \mathrm{cm^{-1}}$ indicates an increase in absorbance associated with β -glycosidic linkages, suggesting a higher cellulose content in fibers treated with higher NaOH concentrations. This is attributed to the dissolution of lignin by NaOH, which enhances cellulose purity. These results align with Dewanti (2018), who reported that NaOH facilitates delignification by removing non-cellulosic components, leading to higher-purity cellulose.

The peak at 1603 cm⁻¹ corresponds to the C=C bonds present in lignin. The transmittance of this peak increased with the rise of NaOH concentration by up to 8%wt. Nonetheless, the transmittance decreased as the NaOH concentration continued to increase beyond 8%, which indicated that lignin content initially increased in detectability before being significantly reduced as NaOH concentration increased, consistent with the finding reported by Yalç et al. (2019).

The 1739 cm⁻¹ peak, which represents the CH₃CO ester group in hemicellulose, showed minor fluctuations across the NaOH concentrations studied in this research, with a general trend of low intensity, suggesting a very low presence of acetyl. This observation supports Kallel et al. (2016), which noted that alkaline treatment effectively removes hemicellulose and lignin, thus purifying the cellulose.

The broad OH band around 3327 cm $^{-1}$ represents hydroxyl groups, primarily found in cellulose. The decreasing transmittance with the increase in NaOH concentration indicates stronger absorption, consistent with the increasing β -glycosidic peak intensity and, therefore, higher cellulose presence.

Effect of NaOH Concentration on Garlic Waste Fiber Morphology

Scanning Electron Microscopy (SEM) was used to examine the surface morphology of the resulting garlic waste fibers derived from garlic waste treated with different NaOH concentrations. The resulting SEM images are presented in Figure 3.

As seen in Figure 3, the surface of the untreated garlic fibers exhibits thick, irregular, and wavy surfaces, characterizing the general structure of amorphous lignin and hemicellulose. When garlic waste cooking was carried out using 2% wt. NaOH concentration, the fiber surface appears to be slightly straighter. However, the use of 8% wt. NaOH concentration as the cooking solution, the garlic fiber surface was rougher due to the thinning of the outermost layers. As expected, garlic waste cooked using 14% wt. of NaOH solution, resulting in a rougher garlic fiber surface. Surprisingly, the use of 20% wt. NaOH solution at the delignification stage, the garlic fiber surfaces appeared to be rougher, and they were visibly cleaner, indicating significant removal of amorphous materials. These results confirm that NaOH concentration significantly influences fiber surface structure. The use of higher NaOH concentrations leads to increased fiber roughness due to the extended removal of lignin, hemicellulose, and waxes. These findings are consistent with Reddy & Rhim (2018), which reported that alkaline treatment increases fiber surface roughness, improving fiber-polymer interfacial bonding for composite applications.

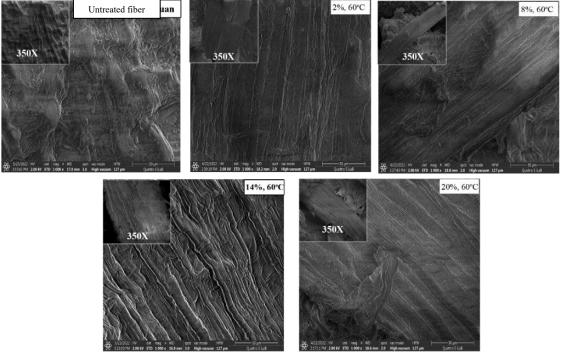
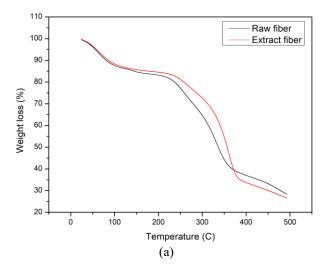


Figure 3. Surface morphology of garlic waste fibers treated with varying NaOH concentrations (60°C, 300 rpm, 180 minutes)

Thermal Properties of Garlic Waste Fiber Extract

Thermogravimetric analysis (TGA) provides essential insights into the alterations in thermal stability of a material resulting from particular thermal treatment conditions. The TGA and derivative thermogravimetric analysis (DTG) profiles of garlic samples, including both untreated and alkali-treated variants, are illustrated in Fig. 5.



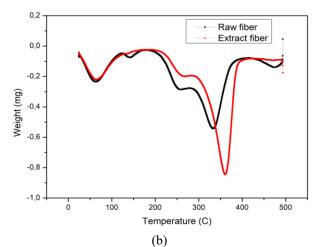


Figure 4. TGA (a) and DTG (b) curves of garlic fiber

The DGTA profiles display distinct peaks that are associated with a variety of thermal decomposition stages. A minor mass reduction observed in all samples at temperatures below 100°C is likely due to the evaporation of the physically adsorbed moisture (Collazo-Bigliardi et al., 2018). The additional peaks correspond to the thermal degradation of lignocellulosic compounds.

During the second degradation stage, which transpires between 225 and 350°C, the garlic-derived samples demonstrated analogous thermal behavior. This phase entails the condensation reactions among hydroxyl groups in the anhydroglucose chains, resulting in the cleavage of ether linkages and unsaturated moieties. The thermal decomposition of

starch, cellulose, and glycerol structures occurs simultaneously (Syafri et al., 2019).

Both samples demonstrated two separate decomposition events, which align with the progressive thermal degradation of various lignocellulosic constituents. Following alkali treatment, a significant increase in decomposition temperature was observed, likely due to the efficient extraction of hemicellulose and lignin, resulting in a slight increase in cellulose content.

Effect of NaOH Concentration on the Yield of Garlic Waste Fiber Extract

Delignification of garlic waste was conducted using the alkaline pulping method. This process was carried out using NaOH concentrations of 0%, 2%, 8%, 14%, and 20% wt. at a temperature of 60°C. The results of garlic waste delignification yield are presented in Figure 4.

Figure 4 displays the effect of NaOH concentration on the cellulose yield obtained from the alkaline pulping treatment. The results indicate that increasing the concentration of NaOH from 0% to 20% wt. leads to a remarkable decrease in cellulose yield. This decline is primarily attributed to effective removal of non-cellulosic components, such as lignin, waxes, and moisture from the fiber structure.

The cleavage mechanism of the bond between lignin and cellulose by nucleophilic OHbegins with the attack of the hydroxide ion (OHbegins with the susceptible hydrogen atoms bound to the phenolic hydroxyl group. The hydrogen atom exhibits acidic characteristics and is bonded to an oxygen atom with high electronegativity. Due to this high electronegativity, the oxygen atom withdraws electron density from the hydrogen atom, rendering the hydrogen partially positively charged and more easily dissociable as an Heben (Lestari et al., 2018). This mechanism is clearly illustrated in Figure 6.

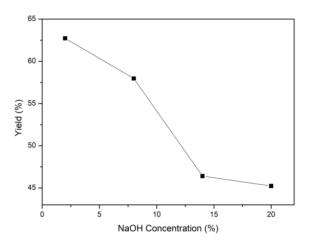


Figure 5. Yield recovery of the treated fibers with varying NaOH concentrations

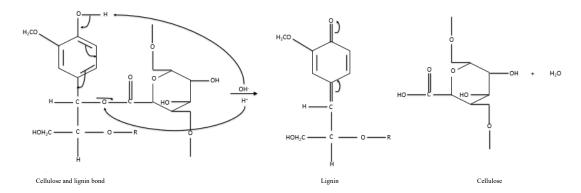


Figure 6. Mechanism breaking the bond between lignin and cellulose by an alkali compound

The reduction in cellulose yield is moderate at lower NaOH concentrations (2–8% wt.). However, there was a sharp decline between 8% and 14% wt. NaOH is observed, indicating a more substantial removal of matrix materials. This phenomenon can be explained by the effective disruption of ligninhemicellulose complex network during alkaline treatment. The lignin-hemicellulose complex network encapsulates the cellulose microfibrils. phenomenon leads to mass loss of the garlic waste. Sequentially, the effective fiber diameter is reduced, leading to a lower overall yield. These observations are consistent with Hasina Mamtaz et al. (2016) report. which states that alkaline treatment causes a reduction in fiber diameter through the elimination of lignin, pectin, hemicellulose, and water content. Finally, although higher NaOH concentrations enhance cellulose purity, they also cause significant reduction of the overall yield of the treated fibers.

CONCLUSION

Garlic processing by-products have been recognized as promising sources of biopolymers that contain a high concentration of cellulose (> 40%), making it a viable resource for cellulose. The extraction of cellulose from garlic waste through alkaline delignification has been proven to be effective. The highest fiber yield (62.7%) was obtained at a NaOH concentration of 2% wt., while higher concentrations led to a decrease in yield but enhanced cellulose purity and fiber surface morphology. FTIR analysis confirmed that garlic waste treatment using high NaOH concentration (20% wt.) increased β-glycosidic linkage absorbance, indicating higher cellulose content. In addition, SEM observation revealed that increasing concentration resulted in rougher fiber surfaces due to the excessive removal of lignin and hemicellulose. This research demonstrates that garlic waste offers a promising potential as a raw material for cellulose production, which can subsequently be converted into various value-added products.

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