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Characterization of Cellulose Extracted from Garlic Waste via Alkaline Treatment and Its Effect on Yield

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Abstract

Garlic waste—including straws, bulbs, and skins—is an agricultural residue contributing to household waste. Rich in cellulose, garlic waste can be converted into various value-added products through proper processing. In this study, cellulose was extracted from garlic waste using alkaline treatment to investigate the influence of sodium hydroxide (NaOH) concentration on the characteristics of the resulting cellulose. NaOH solutions with varying concentrations of 2%, 7%, 14%, and 20% were used during the alkalization process. FTIR spectra revealed transmittance changes with increasing concentration, indicating a reduction in lignin content. The resulting cellulose exhibited needle-like structures, and garlic fibers with amorphous morphology showed cleaner surfaces at a NaOH concentration of 20%. The highest extract yield from the alkalization process was 62.7%, achieved at a NaOH concentration of 2%. These findings demonstrate the potential of garlic waste as a cellulose source and its applicability 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 by-products 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 (Lee *et al.*, 2020; Choi *et al.*, 2021). Approximately 76% of a garlic bulb's mass comprises cloves, with the remaining 24% consisting of its inner and outer peel layers (Kallel and 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 (Pertanian and Pertanian, 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 β -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, Farid and Ardhyanta, 2017).

Cellulose has diverse applications, such as in the paper industry, pharmaceuticals (Syamsu, 2013), textile additives (Eriningsih, Yulina and Mutia, 2011), reinforcement in bioplastics (Maryam, Rahmad and Yunizurwan, 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 membrane material, due to its asymmetric structure and ultrathin active layer that effectively filters solutes (Husni, Rahim and Ruslan, 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, thereby 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 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. Analytical-grade NaOH (99%, Merck) and distilled water were used throughout the experiment.

Preparation and Delignification Process

The garlic waste was washed to remove dirt and contaminants and then 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). Delignification was conducted using NaOH concentrations of 0%, 2%, 8%, 14%, and 20% 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

a. FTIR Analysis

Fourier-transform infrared (FTIR) spectroscopy was used to identify the functional groups on the fiber surface. 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 $400\text{--}4000\text{ cm}^{-1}$.

b. SEM 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 (V_{acc}) of 10 kV and an emission current (I_{e}) of 10 μA . Before imaging, samples were coated with osmium (Os) using a vacuum sputter coater.

c. Yield

The yield measures the proportion of delignification that results after the alkalization process. Yield calculations can be done using the following formula 1.

$$\% \text{ yield} = \frac{w_a}{w_b} \times 100\% \quad (1)$$

Where,

W_a = weight before alkalization

W_b = weight after alkalization

RESULTS AND DISCUSSION

3.1 Fourier Transform Infrared (FTIR) Spectroscopy Analysis

FTIR analysis was conducted to observe the chemical structural changes in garlic waste fibers after alkaline treatment. These structural changes are indicative of lignin removal, which leads to an increased cellulose content.

The FTIR spectra of untreated samples exhibited characteristic peaks at various infrared wavenumbers, representing the major functional groups in lignocellulosic fibers: cellulose, hemicellulose, and lignin. The β -1,4-glycosidic linkage peak is associated with cellulose, the C=C bond indicates lignin, and the CH_3CO (acetyl) group is characteristic of hemicellulose. The hydroxyl (OH) group is commonly found in all major lignocellulosic components, with a higher concentration in cellulose. The observed vibrational peaks are illustrated in Figure 1, and their corresponding wavenumbers are listed in Table 1.

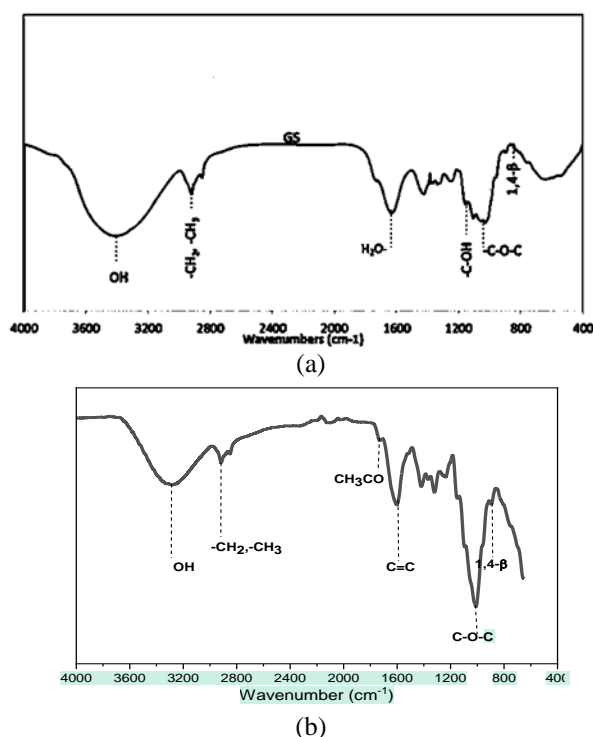


Figure 1. FTIR spectra of garlic waste fiber samples: (a) this study, (b) previous study by Kallel et al. (2016)

Table 1. Functional groups identified in garlic waste fiber samples

Functional Group	Wave number (cm ⁻¹)	
	Kallel, dkk. 2016	This study
Cellulose (OH)	3406–3420	3327
Cellulose (1,4- β)	904	892
Cellulose (C-O-C)	1048	1008
Lignin (C=C)	1630	1603
Hemicellulose	-	1739

(CH_3CO)		
C-H Stretching	2897–2923	2907
C-OH Bond	1118	1156

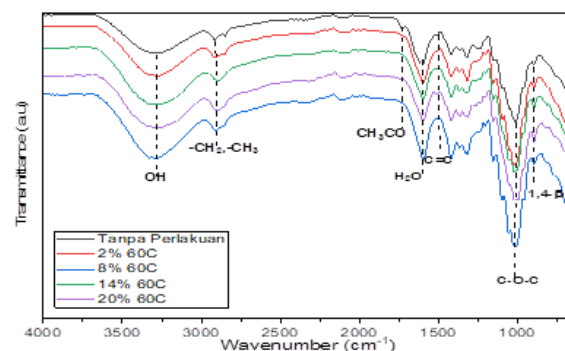


Figure 2. Chemical structure changes of garlic fiber treated with varying NaOH concentrations (60 °C, 300 rpm, 180 minutes)

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

NaOH Concentration	Transmittance (%)			
	1,4- β (892 cm ⁻¹)	C=C (1603 cm ⁻¹)	CH_3CO (1739 cm ⁻¹)	OH (3327 cm ⁻¹)
Untreated	87,79	94,41	95,87	90,39
2%	84,94	93,34	96,85	87,68
8%	83,15	93,09	96,74	86,11
14%	85,53	94,08	97,57	84,86
20%	81,75	92,30	95,62	84,38

The decrease in transmittance at 892 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 C=C bonds present in lignin. Transmittance increased to 8% NaOH, then decreased at higher concentrations, indicating that lignin content initially increased in detectability before being significantly reduced as NaOH concentration increased—consistent with findings by Yalç et al. (2019).

The 1739 cm⁻¹ peak, which represents the CH_3CO ester group in hemicellulose, showed minor fluctuations across concentrations, with a general trend of low intensity, suggesting minimal acetyl presence. This observation supports Kallel et al. (2016), who noted that alkaline treatment effectively removes hemicellulose and lignin, purifying the cellulose.

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

3.2 Effect of NaOH Concentration on Fiber Morphology

Scanning Electron Microscopy (SEM) examined the surface morphology of garlic waste fibers treated with different NaOH concentrations. SEM images are presented in Figure 3.

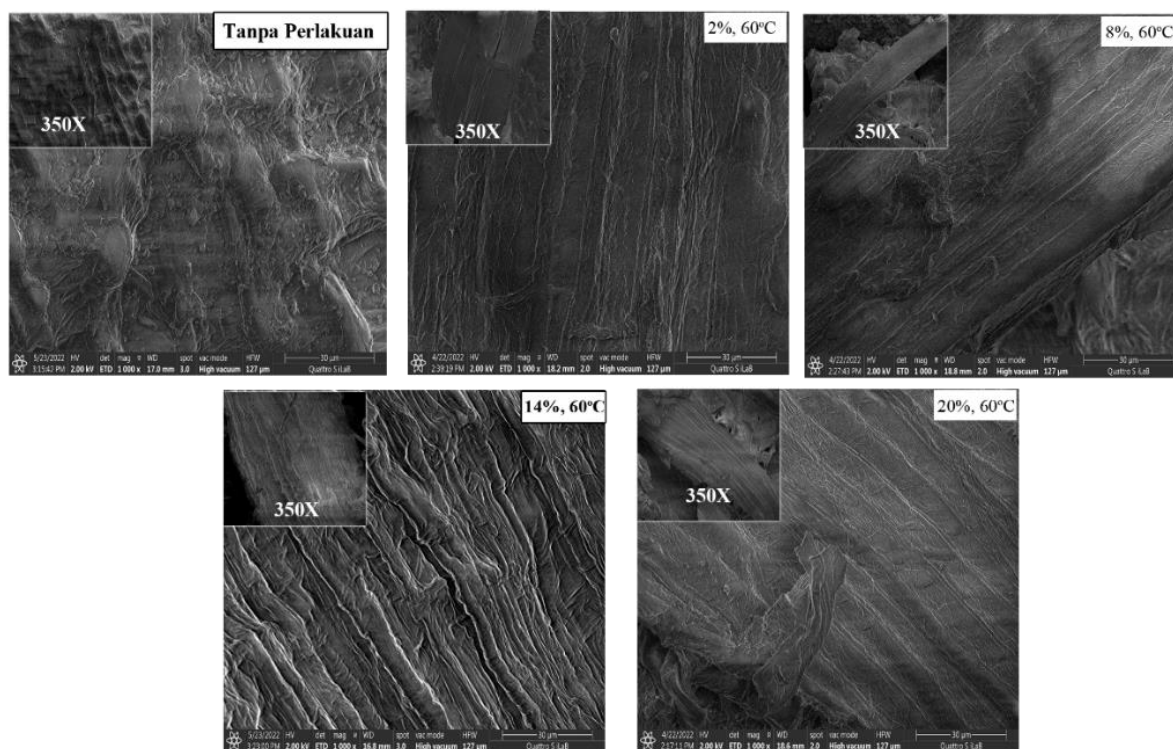


Figure 3. Surface morphology of garlic waste fibers treated with varying NaOH concentrations (60°C, 300 rpm, 180 minutes)

Untreated fibers exhibited thick, irregular, and wavy surfaces, characteristic of amorphous lignin and hemicellulose structures. At 2% NaOH, fibers appeared slightly straighter. At 8%, fiber surfaces became rougher due to the thinning of the outermost layers. At 14%, surfaces appeared rougher, and at 20%, the fibers were visibly cleaner, indicating significant removal of amorphous materials. These results confirm that NaOH concentration significantly influences fiber surface structure: higher concentrations lead to increased roughness due to removing lignin, hemicellulose, and waxes. These findings are consistent with Reddy & Rhim (2018), who reported that alkaline treatment increases fiber surface roughness, improving fiber-polymer interfacial bonding for composite applications.

3.3 Effect of NaOH Concentration on Yield Extract

Delignification of garlic waste is performed using the alkalization method. The alkalization process was conducted using NaOH concentrations of 0%, 2%, 8%, 14%, and 20% at a temperature of 60°C. The results of garlic waste delignification yield can be observed in Figure 4.

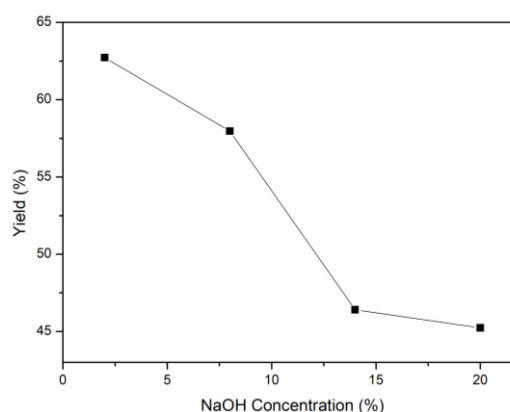


Figure 4. Yield recovery of the treated fibers with varying NaOH concentrations

Figure 4 presents the effect of NaOH concentration on the yield after alkaline treatment. The results indicate that increasing the concentration of NaOH from 0% to 20% leads to a notable decrease in yield. This decline is primarily attributed to removing non-cellulosic components such as lignin, waxes, hemicellulose, and moisture from the fiber structure. The cleavage mechanism of the bond between lignin and cellulose by nucleophilic OH^- begins with the attack of the hydroxide ion (OH^-) from NaOH on the hydrogen atom 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 H^+ ion (Lestari *et al.*, 2018). This mechanism is illustrated in Figure 5.

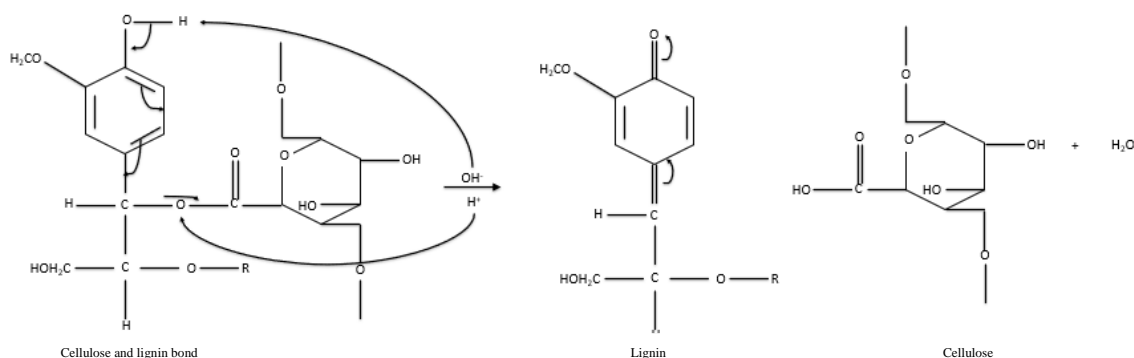


Figure 5. Mechanism breaking bond between lignin and cellulose by alkali compound

The reduction in yield is moderate at lower NaOH concentrations (2–8%). However, a sharp decline between 8% and 14% NaOH is observed, indicating a more substantial removal of matrix materials. This phenomenon can be explained by alkaline treatment disrupting the complex network of lignin and hemicellulose that encapsulates cellulose microfibrils, resulting in mass loss of the treated biomass. As these components are dissolved, the effective fiber diameter is reduced, leading to a lower overall yield.

These findings are consistent with Hasina Mamtaz *et al.* (2016) report, which states that alkaline treatment causes a reduction in fiber diameter by eliminating lignin, pectin, hemicellulose, and water content. Thus, while higher NaOH concentrations enhance cellulose purity, they also reduce the overall yield recovery of the treated fibers.

CONCLUSION

Garlic processing by-products have been recognized as a promising source of biopolymers. Agricultural waste, including garlic waste, contains a high concentration of cellulose (over 40%), making it a viable resource compared to conventional sources. The extraction of cellulose from garlic biomass through chemical treatment, particularly alkaline delignification, has proven effective. The highest fiber yield (62.7%) was obtained at a NaOH concentration of 2%, while higher concentrations led to a decrease in yield but improved cellulose purity and fiber surface morphology. FTIR analysis confirmed that treatment with a high NaOH concentration (20%) increased β -glycosidic linkage absorbance, indicating higher cellulose content. SEM observations revealed that increasing NaOH concentration resulted in rougher fiber surfaces due to the progressive removal of lignin and hemicellulose. These results demonstrate that garlic waste holds significant potential as a raw material for cellulose production, which can subsequently be developed into value-added products such as cellulose acetate membranes.

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