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Reaction Mechanism in Standardized α-Cellulose Content Test: Study from *Boehmeria nivea* Fiber

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***Abstract*—**In defense industry, α-cellulose is the main component of nitrocellulose propellant. However, a detailed description of the reaction mechanism of each treatment step in SNI 0444-2009 is still very scarce. This study addresses this gap by presenting the reaction mechanisms of each treatment and the symbols used in the SNI 0444-2009 procedure. The separation of lignin from α-cellulose occurred by breaking the C–O bond linking them. This bond was broken by the –OH group of NaOH via a hydrolysis reaction. The reaction was initiated with the elimination of a hydrogen atom from the lignin structure by the hydroxyl ion (–OH), and the C–O bond was broken by a hydrolysis reaction. The breaking of this bond was indicated by the disappearance of the IR peaks at wavenumbers 1049 and 1190 cm^{-1} in the filtrate after extraction. The SNI 0444-2009 method for the α-cellulose content test was carried out by a redox back titration of Cr(VI) with Fe(II) from ferrous ammonium sulfate. This titration was conducted to calculate the amount of Cr(VI) ions in potassium dichromate or Cr(VI) that did not react with lignin or beta cellulose in the filtrate. Understanding the contribution and reaction mechanisms of each compound involved in the SNI 0444-2009 procedure contributed to obtaining accurate data on α-cellulose content. In this study, the calculated α-cellulose content of the flax fiber was 96.75%. Furthermore, the detailed mechanism of the redox reaction was discussed in detail in this paper.

***Keywords*—** *Cellulose content test; Extraction; Flax fiber; Reaction mechanism; SNI 0444-2009*

1. INTRODUCTION

In the defense industry, the quality of single-base and double-base propellants is determined by the importance of the nitrogen content of the nitrocellulose raw material. These compounds must contain at least 12% nitrogen to produce propellants with high thrust power and specific impulse [1]. Another reason for this emphasis is the requirement for qualification standards for the raw material of the propellant. The nitrogen content in nitrocellulose is associated with the quality of cellulose compounds. Cellulose with a high α-cellulose content facilitates the nitration reaction more optimally through the substitution of hydroxyl groups at C2, C3, and C6 in cellulose with nitro groups. However, the presence of other compounds accompanying cellulose, such as lignin and hemicellulose, can impede the optimization of the nitration reaction. Cellulose, an extractive compound, is found in linked with lignin and hemicellulose as lignocellulose [2]. Therefore, the separation of cellulose compounds from these accompanying compounds has attracted immense interest.

Cellulose isolation process depends on the cellulose source. Cellulose as extractive compound can be obtained from plant fibers, animal cell walls, and several minerals [3]. However, the main source of cellulose in nature is plant fibers. Plant fibers can be processed from various parts of plants, such as fibers obtained from seeds (kapok and cotton) [4], fibers obtained from leaves (pineapple) [5], fibers obtained from bark and stems (jute, flax, hemp) [6], and fibers obtained from fruit (coconut) [7]. Cellulose sourced from plant stems generally has a middle-level cellulose content in the range of 30%. In fact, the natural cellulose source is produced from fibers obtained from both woody and non-woody stems [8]. Flax fiber from the *Boehmeria nivea* plant is known for its high α-cellulose content. This is confirmed by research conducted by Arafah et al. (2021), which states

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that the α-cellulose content of flax fiber is 75%, and by research conducted by Fauziati et al. (2020), which claims that the α-cellulose content of flax fiber is 72-97% [8,9]. Generally, the process of isolating α-cellulose from biomass involves several procedures, such as sample preparation, hydrolysis, delignification, and bleaching. The high content of α-cellulose correlates with low levels of lignin and other matrix substances. This allows for a simpler α-cellulose isolation procedure (without repetition) compared to other biomass sources, such as oil palm empty bunches and coconut fiber, which require more complext methods.

The determination of α-cellulose content is an equally crucial procedure in the process of extracting cellulose from plant fibers. This provides valuable information regarding the effectiveness of the cellulose isolation process. The α-cellulose content test can be conducted using several internationally standardized procedures, such as those outlined by the American Society for Testing Materials (ASTM) and the International Union of Pure and Applied Chemistry (IUPAC) methods. However, in general, most countries, including Indonesia, have their own national standards that serve as a reference for all forms of technical activities in the industry and manufacturing, food and beverage production, environmental management, energy production, agriculture, health, security and all sectors related to public welfare requirement. In Indonesia, the technical method for testing α-cellulose content is outlined in SNI 0444-2009, which includes α-cellulose, β-cellulose, and γ-cellulose content test methods [9]. The procedures and principles of the α-cellulose content test in SNI 0444-2009 are similar to those established by the international standards like ASTM and IUPAC. The α-cellulose content test is based on the isolation of compounds with accompanying compounds contained in the cellulose source sample, such as lignin, hemicellulose, β-cellulose, γ-cellulose, and other extractive materials. The isolation result of α-cellulose is calculated using the equation that is formulated. Notably, the α-cellulose separation method employed in SNI 0444-2009 utilizes various types of compounds, such as oxidizing agents, reducing agents, and several additive compounds. However, the role of each chemical compound and its reaction mechanism have not been described in SNI 0444-2009 and other studies. This limited understanding of the individual contributions and reaction mechanisms of each compound can negatively impact the accuracy of the test, leading to higher error rates [10].

The α-cellulose content test using the standardized SNI 0444-2009 method has been widely used by researchers to determine α-cellulose content present in a sample. However, research literature remains scarce on the reaction mechanisms and treatment function of any chemical compounds used in the α-cellulose content test procedure. In this study, we present the reaction mechanism of each treatment given in the α-cellulose content test method in accordance with SNI 0444-2009, which consists of α-cellulose extraction and the redox process.

2. EXPERIMENTAL SECTION

2.1. Materials

In this study, flax fibers from *Boehmeria nivea* (obtained from Sleman, Indonesia) were used as a sample to investigate their α-cellulose content. The sample size was reduced to two centimeters. A 17.5% NaOH solution was used to extract other compounds that accompany the α-cellulose in the sample. To initiate the redox reaction of these compounds, potassium dichromate was used, assisted by concentrated sulfuric acid as the catalyst. For the titration process, a ferroin indicator was used as a marker of the redox reaction and 0.5 N ferro ammonium sulphate was employed as a titrant.

2.2. Instrumentations

Attenuated total reflectance infrared (ATR-IR Bruker Invenio-R) was used to investigate the compound structure of the extraction result flax fiber with NaOH solution in liquid form and the sample before extraction in powder form. A UV-VIS spectrophotometer (Jenway 6105) was used to investigate the redox reactions that occurred during the process of determining the α-cellulose content.

2.3. α-Cellulose Content Test

The method was based on the SNI 0444-2009 in Ikhtiarini et al. (2022) [9]. The procedure is as follows: 2 g sample was dried in an oven at 100 °C for 30 min to remove the water contained in the sample. The sample was then soaked in 100 mL of 17.5% NaOH at 25 °C. The mixture was stirred until fully dispersed; the formation of air bubbles was avoided during the stirring process. Next, distilled water (100 mL) was added to the mixture and stirred for 30 min. The mixture was then separated using a filter paper to obtain the filtrate.

The titration process was initiated by carefully mixing 25 mL of filtrate, 10 mL of potassium dichromate solution (0.5 N), and 50 mL of concentrated sulfuric acid. The mixture was heated to a temperature range of 125-135 °C and after heating, 50 mL of distilled water was added to the mixture. Once the mixture cooled to room temperature, 2-4 drops of the ferroin indicator were added. The mixture was then titrated with 0,1 N ferro ammonium sulfate until the color changed from green to violet. The titration was also conducted on the blank solution, which consist of 12.5 mL distilled water and 12.5 mL of 17.5% NaOH. The α-cellulose content was calculated using Equation (1). where X is the α-cellulose content (%), V_1 is the blank titration volume (mL), V_2 is the filtrate titration volume (mL), N is the

normality of the ferro ammonium sulfate solution (N), A is the volume of filtrate (mL), and *W is* the mass dried of the analyzed sample (g).

$$X = 100 - \frac{6.85\ (V_1 - V_2) \times N \times 20}{A \times W} \quad (1)$$

2.4. Determination of The Compound Structure Before and After NaOH Extraction

The compound structure before and after extraction was investigated using ATR-IR spectroscopy. Before the extraction process, 2 g of flax fiber was crushed using a grinder for sample preparation. The samples were then investigated using ATR-IR. After the extraction process, 2 mL of the filtrate was investigated using ATR-IR. The spectra of the two samples were analyzed by comparing the bands produced by each functional group.

3. RESULT AND DISCUSSION

3.1. Determination of Compound Structure Before and After NaOH Extraction

This determination is essential to validate the reaction mechanism of the α-cellulose content test. The lignocellulose in the flax fiber reacts with the OH^- group from the NaOH solution. The hydroxyl group breaks the bond between cellulose and lignin by reacting with hydrogen, which bonds with phenolics in lignin compounds. The difference in electronegativity in the hydrogen and phenolic bonding causes the hydrogen to have a high-positive dipole moment. Therefore, the hydroxyl group tends to attack that bond by eliminating the reaction. The removal of hydrogen atoms from lignin compounds leads to electron rearrangement, which breaks the bonds between lignin and cellulose through hydrolysis. The resulting lignin compound does not have electron stability. Therefore, there is a rearrangement of electrons that produces new nucleophilic lignin compounds. Thus, the reaction of NaOH with lignocellulosic compounds produces cellulose compounds as the residue and lignin compounds dissolved in NaOH as the filtrate. The proposed reaction mechanism is illustrated in **Fig. 1.**

To validate the proposed reaction mechanism, ATR-IR analysis was conducted on the resulting filtrate (**Fig. 2a**). A fair sharp and broad peak was obtained at wave numbers 3334, 1633, and 653 cm^{-1}. The peak at 3334 cm^{-1} indicated the existence of –OH stretching vibrations of alcohol and phenol groups [11,12]. The peak at 1633 cm^{-1} was correspond to the aromatic ring C=C group [13,14]. The peak at 653 cm^{-1} showed the presence of C–H bending of alkenes and the characteristics of wagging, deformation, and twisting of anhydrous pyranose rings [15].

ATR-IR analysis of the flax fiber powder (**Fig. 2b**) showed peaks at 3334, 1633, 1190, and 1049 and a fingerprint area at 653 cm^{-1}. Compared to **Fig. 2a**, there were two peaks at 1190 and 1049 cm^{-1}, which were not observed in **Fig. 2b**. The peaks at 1049 and 1190 cm^{-1} indicated the C–O stretching of aliphatic ether groups [16]. The absence of these two peaks in **Fig. 2a** represents the bond-breaking of C–O aliphatic ether that links the lignin with the cellulose during NaOH treatment [17].

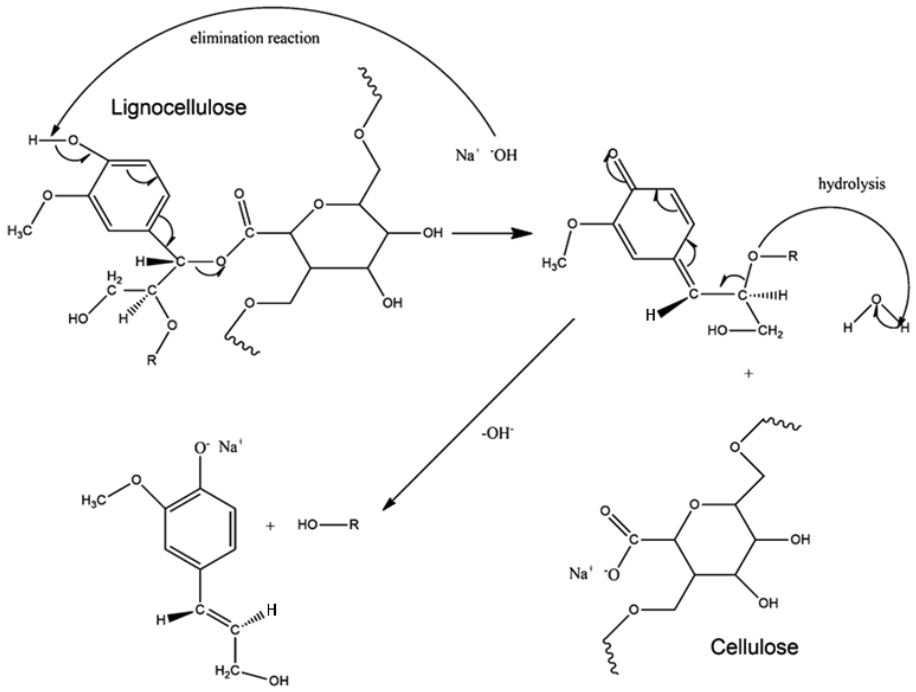

Fig. 1. Reaction mechanism of lignin bond breaking by NaOH

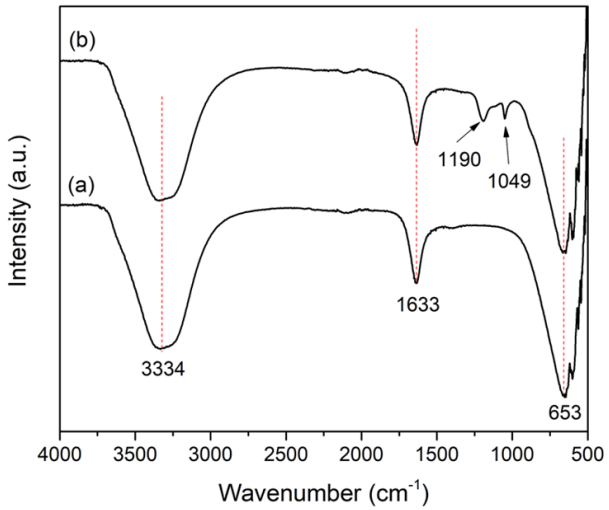

Fig. 2. The ATR-IR analysis of (a) filtrate extraction using 17.5% NaOH and (b) flax fiber powder

3.2. Investigation of Redox Titration

A redox reaction is involved in the determination of α-cellulose content in the sample after the sample extraction stage using NaOH. The filtrate obtained from the extraction was reacted with $K_2C_2O_7$ and concentrated sulfuric acid. The reaction between $K_2C_2O_7$ solution with lignin and β-cellulose compounds contained in the filtrate causes a reduction reaction of Cr (VI) ions in $K_2Cr_2O_7$. In the first reaction (with lignin), adding the $K_2C_2O_7$ solution to the filtrate changes the color of the solution. The originally bright orange $K_2C_2O_7$ solution turns into a yellowish color after being added to the filtrate. The color change is caused by a change in pH from 4 to 14 due to the NaOH content in the filtrate. The reaction mechanism of reduction-oxidation between lignin and $K_2C_2O_7$ can be found in **Fig. 3**.

Then, after the addition of H_2SO_4 as a catalyst, a significant color change occurs, transforming the solution from yellowish to a leafy green. The color shift occurs due to the change in oxidation state from Cr (VI) to Cr (IV). Changes in the oxidation state of chromium ions occur through redox reactions between dichromate ions with lignin and beta-cellulose compounds present in the filtrate. The redox reaction mechanism between β-cellulose and potassium dichromate is shown in **Fig. 4.**

The filtrate, after reacting with potassium dichromate and concentrated sulfuric acid, is titrated with a ferro ammonium sulfate solution. The titration determines the amount of Cr(VI) ions in the potassium dichromate or the Cr(VI) that do not react with lignin or beta cellulose in the filtrate. The reaction between the residual Cr(VI) and ferro ammonium sulfate is shown in Equation (2).

$$2Fe^{2+}\,(aq) + CrO_4^{2-}\,(aq) + 8H^{+}\,(aq) \rightarrow Cr^{3+}\,(aq) + 2Fe^{3+}\,(aq) + 4H_2O\,(l) \qquad (2)$$

The reaction between the blank and the ferro ammonium sulfate solution is also crucial for the determination of alpha-cellulose content. To determine the amount of Cr(VI) ions that react redoxally with lignin and β-cellulose compounds in the filtrate, it is necessary to know the amount of Cr(VI) ions that react with ferro ammonium sulfate as a whole without lignin and β-cellulose compounds. By calculating the difference between the amount of Cr(VI) ions used in the blank titration and the remaining Cr(VI) ions, the amount of Cr(VI) ions used can be determined.

The change in the oxidation state of chromium ion in the filtrate before and after titration using ferro ammonium sulfate is shown in **Fig. 5**. There is a change

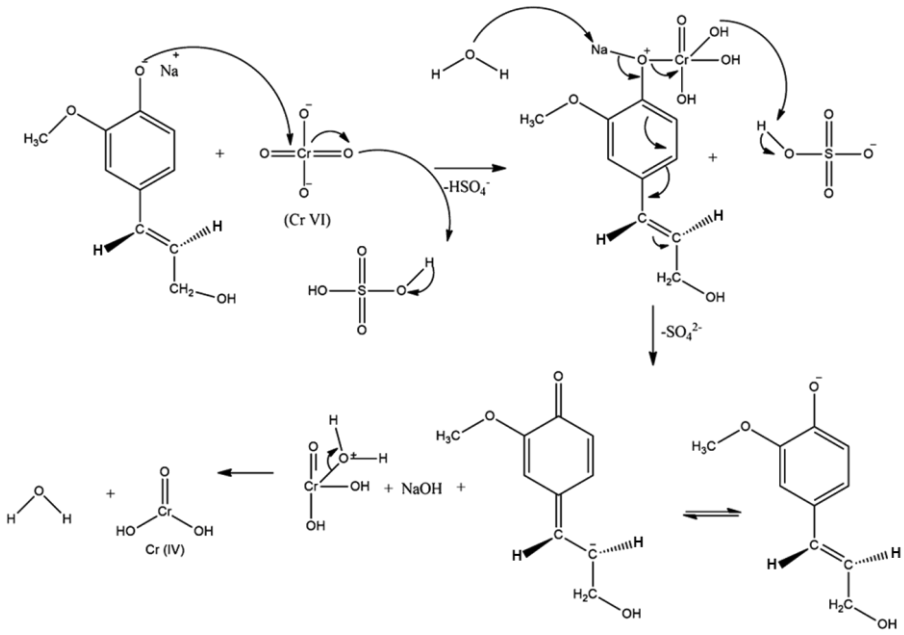

Fig. 3 Mechanism of redox reaction between lignin and $K_2C_2O_7$

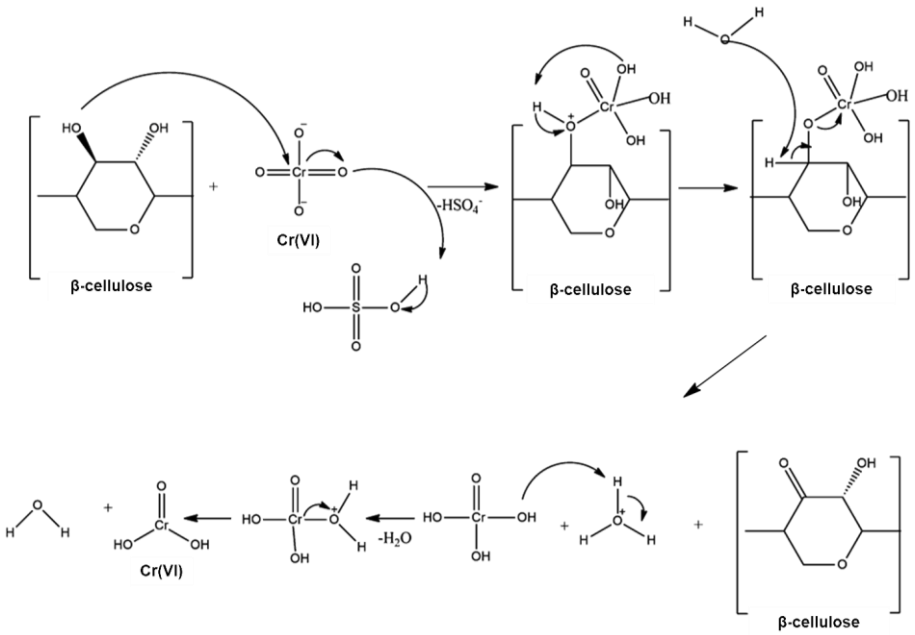

Fig. 4 Mechanism of redox reaction between β-cellulose and $K_2C_2O_7$

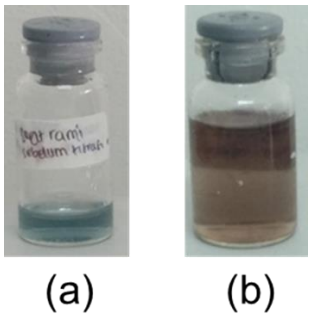

Fig. 5 The different color of filtrate before titration (a) and after titration (b)

in color from green to a brownish-purple solution. The brownish-purple indicates the formation of Fe(III) ions, as detected by the ferroin indicator [18]. In contrast, the green color is characteristic of the Fe(II) ions.

The equation from SNI 0444-2009 (Equation (1)) to determine the α-cellulose content can be described: *X* is the amount of α-cellulose in the sample, a reduction number of 100 is used because the determination of α-cellulose content with this method is done indirectly (back titration) through the calculation of compounds other than α-cellulose. V_1 is the volume of titrant used for the blank, representing the amount of Cr(VI) ions reduced by the species in the blank. V_2 is the volume of titrant used for the sample, representing the remaining Cr (VI) that does not react with lignin and β-cellulose). *N* is the normality of the ferro ammonium sulfate used as titrant. *A* is the volume of titrate (analyzed filtrate). *W* is the weight of the analyzed sample (flax fiber). Theoretically, 1 millieqivalent $K_2Cr_2O_7$ is equal to 6.75 mg of cellulose and other hexosanes, and equal to 6.60 mg pentosanes. However, during this test, less oxidant is consumed, so 1 milliequivalent of $K_2Cr_2O_7$ is considered equivalent to 6.85 mg of cellulose and other dissolved carbohydrates [19].

The α-cellulose content of the flax (*Boehmeria nivea*) fiber calculated in this study was 96.75%. This value is comparable to studies reported by Mongiovi et al. (2021) [20], Kopania et al. (2012) [21], and Lazic et al. (2018) [22] who reported the α-cellulose content in flax fiber of 73%, 60%, 93%, respectively. To produce nitrocellulose with a minimum 12% nitrogen content (military grade), a minimum α-cellulose content of 92% is required. Based on these results, the flax fiber has a high potential to be a source of α-cellulose because it is cheap, easy to obtain, and abundant.

CONCLUSION

The reaction mechanism of each step in the National Standard Indonesia (SNI) 0444-2009 procedure for the α-cellulose content test had been described. There were two steps in this procedure, firstly extraction of lignin with NaOH and secondly, redox reaction followed by the back titration. Extraction of lignin separated lignin from α-cellulose by breaking the C–O bond that links the lignin and α-cellulose by OH^- of NaOH. The breaking of this bond was indicated by disappearing peaks at wavenumber 1049 and 1190 cm^{-1} in filtrate after extraction, as confirmed by ATR-IR analysis. The α-cellulose content test of SNI 0444-2009 was conducted to calculate the amount of Cr(VI) ions in the potassium dichromate or the Cr(VI) that did not react with lignin or beta cellulose in the filtrate. The calculated α-cellulose content of flax (*Boehmeria nivea*) fiber in this study was 96.75%. Therefore, flax fiber is a highly promising source of α-cellulose due to being cheap, easy to get, and abundant. Furthermore, beyond obtaining accurate

data, a clear understanding of the reaction mechanisms and roles of each compound in the SNI 0444-2009 procedure could contribute to the development of new methods for α-cellulose content determination.

SUPPORTING INFORMATION

There is no supporting information in this paper. The data that support the findings of this study are available on request from the corresponding author (R. Basuki).

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CONFLICT OF INTEREST

The authors have no conflict of interest in this publication.

AUTHOR CONTRIBUTIONS

FR, VAN, and AS performed the experiment and wrote the early manuscript. RB supervise the experiment, data calculations, and revise the manuscript. MF, YBA, TK, and TI collaborated on writing and revising the manuscript. All authors approved the final version of the manuscript.

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