

Effect of Temperature and Cellulose Mol Ratio on the Optimization of Oxalic Acid Production from Kapok Fiber

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Article Information	ABSTRACT
Article History Received : February 12, 2025 Revised : March 01, 2025 Published : April 04, 2025	Until now, kapok fiber has not been fully utilized. However, kapok fiber has a high cellulose content that can be used as a basic material in the manufacture of oxalic acid. The purpose of this study was to determine how the hydrolysis temperature and the number of moles of cellulose affect the oxalic acid content produced from kapok fiber. This research was conducted in four stages, namely hydrolysis, precipitation, acidification, and crystallization. Oxalic acid crystals were then analyzed using permanganometric titration to determine the levels contained, melting point analysis, and pH to compare the characteristics of the synthesized oxalic acid with standard oxalic acid. In addition, FTIR spectroscopy analysis to determine the functional groups contained in oxalic acid. The results showed that the highest oxalic acid content was obtained in the condition of 1 mole cellulose and 70°C hydrolysis temperature, which amounted to 17.45%. The FTIR analysis results of synthetic oxalic acid and standard oxalic acid show similarities, as seen from the O-H group length of 3406.29 cm ⁻¹ , the C=O group length of 1687.71 cm ⁻¹ , the C-O group length of 1132.21 cm ⁻¹ , and the C-H group length of 669.30 cm ⁻¹ .
Keywords: <i>Alkaline Hydrolysis; Cellulose; Oxalic Acid.</i>	

INTRODUCTION

Oxalic acid (H₂C₂O₄) is a dicarboxylic acid widely used in various industries, including rust removal, natural rubber purification, textile cleaning and dyeing, as well as a reagent in chemical analysis (Conuwanti, 2019). The demand for oxalic acid in Indonesia continues to rise, as indicated by import data from the Central Bureau of Statistics (2024), which recorded an increase from 1,702,738 tons in 2019 to 2,343,946 tons in 2022. This reliance on imports highlights the need for domestic oxalic acid production to reduce dependence on foreign supplies. One of the primary raw materials for oxalic acid production is cellulose, which is more readily available than oxalate salts such as calcium oxalate. Cellulose is abundant in Indonesia, with one potential source being the kapok tree (*Ceiba pentandra*). Kapok trees are widely distributed across Indonesia, particularly in East Java. They can thrive in dry areas. Kapok fibers are enclosed, smooth, slippery, porous, and relatively large in size (Chandra, 2022). Traditionally, kapok has been used as a filling material for mattresses, pillows, and flotation devices (Arifin, 2020). However, due to declining demand for kapok-based products, alternative applications are needed to optimize its potential. Kapok fiber contains 32% cellulose, 38% hemicellulose, and 20% lignin by weight (Rahmatullah, 2020), making it a promising alternative raw material for oxalic acid synthesis.

The process of making acid can be done by several methods, namely alkaline hydrolysis, oxidation of carbohydrates using nitric acid (HNO_3) and glucose fermentation (Atikah, 2017). Among several methods, the method that is often used is alkaline hydrolysis. This is because the method is simple and the results obtained are also more compared to other methods (Nurfadila, 2017). In the hydrolysis method, cellulose compounds will be melted with strong alkaline solutions, such as NaOH, KOH and $\text{Ca}(\text{OH})_2$. In the industrial world, NaOH is often used because the price is cheaper (Utami, 2018). In addition, the use of NaOH solution can also reduce lignin levels so that more cellulose conversion is obtained (Rahmatullah, 2020).

The process of making oxalic acid uses an alkaline hydrolysis method with raw materials containing cellulose later melted with sodium hydroxide solution. The melted product is added with sulfuric acid solution to form oxalic acid (Subyakto, 2021). According to (Febriaty, 2016), explained that there are several stages in making oxalic acid using the alkaline hydrolysis method, namely the first stage is the hydrolysis and heating stage. In the hydrolysis process, there is a breakdown of polysaccharides from the cellulose content in the raw material using NaOH solution. This stage will obtain glucose using the heating process. In the heating process there will be an oxidation process and the decomposition of glucose into oxalate, acetate and formate salts. The second stage is the precipitation stage, where the filtrate from hydrolysis will be reacted with calcium chloride solution. The results of this reaction will form calcium oxalate precipitates. The third stage is the acidification stage, calcium oxalate precipitate will be added with excess sulfuric acid solution to acidify calcium oxalate into oxalic acid.

There are several factors that affect the hydrolysis process in the manufacture of oxalic acid including hydrolysis temperature, hydrolysis time, solution composition, and stirring speed. If the hydrolysis temperature is high, it will increase the reaction speed constant so that the reaction that occurs will be faster and the levels obtained will also be more. This was proven by (Nurul, 2017) that the weight of oxalic acid from wastepaper was 1.8043 grams under optimum temperature conditions of 105°C . Similar to temperature, the longer the hydrolysis time, the more cellulose content will occur due to the collision of more reacting molecules. In research (Tambunan, 2021) obtained the highest cellulose content of 50% at 180 minutes while at 120 minutes the cellulose content obtained was 30%.

In research conducted by (Chamidy, 2021) the highest cellulose content was 82.8% in the composition of alkali with raw materials of 20:1. Meanwhile, in the composition of the alkaline solution with raw materials of 30:1, the cellulose content decreased to 63.4%. So that the greater the composition of the solution with the raw material, the less product obtained. In addition, stirring can accelerate the occurrence of reactions because stirring will produce a collision between particles so that the speed of the reaction that occurs will also be faster. This was proven by (Afriandi, 2015) that the highest oxalic acid weight was 4.067% at a stirring speed of 600 rpm.

The produced oxalic acid can be analyzed using Fourier Transform Infrared (FTIR) spectroscopy, which operates based on spectroscopic principles. Infrared spectroscopy is beneficial for identifying organic compounds due to its highly complex spectrum, consisting of multiple peaks. These peaks indicate the presence of functional groups, which are characterized by specific wave numbers (Sanjiwani, 2020). In infrared spectroscopy, infrared radiation is passed through the sample. Some of the radiation is absorbed, while the rest is transmitted. The sample is placed in a holder, and the corresponding spectrum is recorded. The result is a diffractogram that illustrates the relationship between wave number and intensity (Satriawan, 2017).

The objective of this study is to produce oxalic acid from kapok fiber using the alkaline hydrolysis method and to determine the optimal hydrolysis temperature and cellulose molar quantity for the highest yield. This study is expected to contribute to innovations in oxalic acid production for the chemical industry while expanding the utilization of kapok fiber beyond its conventional applications.

RESEARCH METHODS

In this study, the raw material used was kapok fiber obtained from Jalan Sawo, Sawo Village, Dukun District, Gresik Regency. The equipment used for the alkaline hydrolysis process consists of a thermometer, stirring motor, beaker glass, stative and clamp. While the equipment for titration is composed of erlenmeyer, burette, stative and clamps. As well as other additional tools namely pH meters, filter paper, measuring cups and spatulas. First, a flowchart depicting the kapok fiber preparation process is presented in Figure 1.

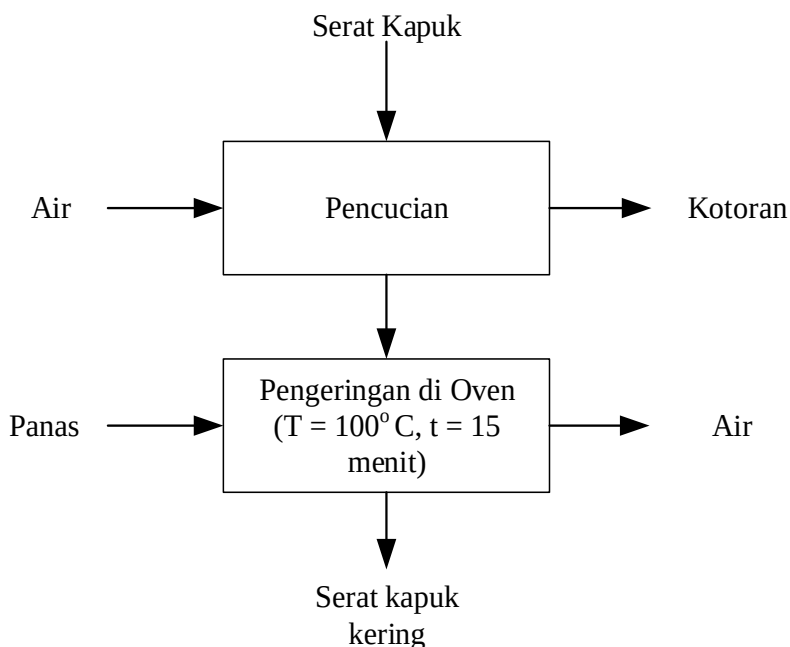


Figure 1. Flowchart of Material Preparation Process

The research procedure begins with the preparation of raw materials, namely kapok fiber. The kapok fiber used will be analyzed to determine the cellulose content. However, the kapok fibers is first processed by washing to remove impurities. It is then dried using an oven at temperature of 100°C for 15 minutes. Secondly, the flowchart illustrating the oxalic acid production process is presented in Figure 2.

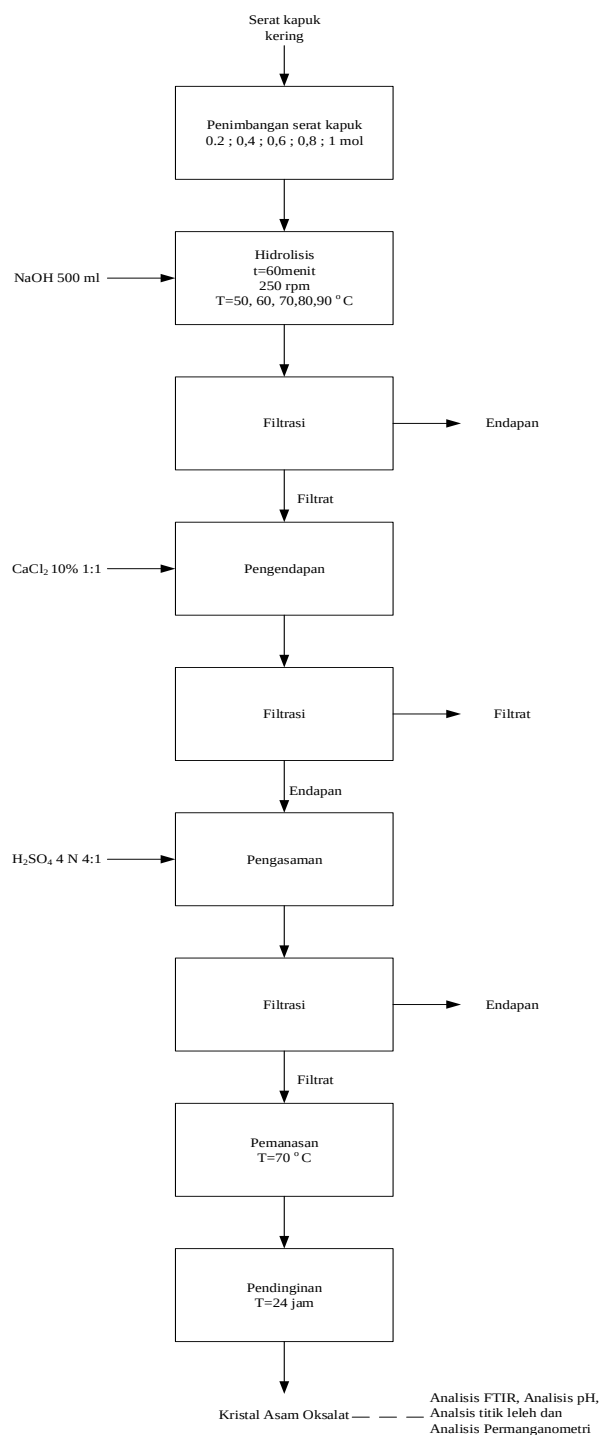


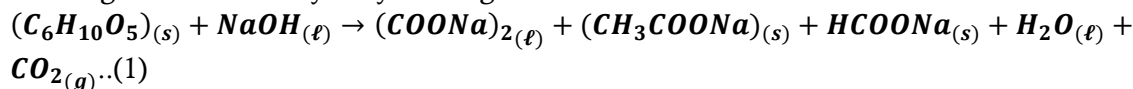
Figure 2. Flowchart of Oxalic Acid Manufacturing Process

The next stage, the dried kapok fiber is weighed with a number of moles of cellulose of 0,2; 0,4; 0,6; 0,8 and 1 mol. Then put into a beaker glass that already has 750 ml of 3N NaOH solution. Then heating was carried out for 45 minutes with temperatures of 50, 60, 70, 80, 90°C and stirring using a stirring motor at a speed of 250 rpm. After the hydrolysis process, the mixture was cooled for 10 minutes and then filtered to separate the precipitate from the filtrate. The filtered filtrate was added 10% CaCl₂ solution with a volume ratio of 1:1 which would later form calcium oxalate precipitate, then filtered. The precipitate was added 4N H₂SO₄ with a volume ratio of 4:1 to the initial filtrate volume and then filtered again to produce oxalic acid filtrate. The precipitate obtained was washed using ethanol. Furthermore, the filtrate was heated at 70°C and then cooled using ice water for 24 hours until the formation of oxalic acid crystals.

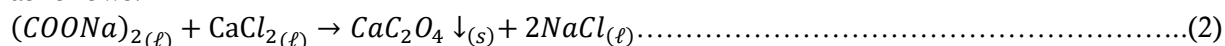
RESULTS AND DISCUSSION

The pre-prepared kapok fiber was analyzed for its cellulose content at the Nutrition Laboratory of the Faculty of Public Health, Airlangga University, with a cellulose content of 37.24%. This cellulose content was then used to calculate the required weight of kapok fiber for each molar variable. According to Nurul (2017), the reaction mechanism in the production of oxalic acid from cellulose feedstock through alkaline hydrolysis can be written as follows.

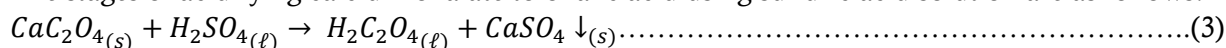
The stages of cellulose hydrolysis using NaOH to form oxalate salt are as follows:



The stages of precipitating sodium oxalate to calcium oxalate using calcium chloride solution are as follows:



The stages of acidifying calcium oxalate to oxalic acid using sulfuric acid solution are as follows:



Effect of Hydrolysis Temperature on Oxalic Acid Concentration

In this study, to determine the oxalic acid content produced, a permanganometric titration analysis was conducted. In the permanganometric analysis, 0.3 grams of oxalic acid crystals were dissolved in 10 ml of water and added with 10 ml of 4N sulfuric acid (H_2SO_4) solution. The solution was then titrated using 0.1N potassium permanganate ($KMnO_4$) solution until a pink color appeared. The oxalic acid content at various temperatures and cellulose moles is presented in Table 1.

Tabel 1. Results of Oxalic Acid Concentration Analysis at Various Temperatures ($^{\circ}C$) and Cellulose Mol (mol)

Mole of Cellulose (mol)	Temperature ($^{\circ}C$)	Oxalic Acid Concentration (%)
0,2 mol	50	6,30
	60	8,82
	70	13,02
	80	10,08
	90	8,82
0,4 mol	50	9,24
	60	10,50
	70	13,23
	80	10,71
	90	9,45
0,6 mol	50	10,08
	60	11,62
	70	14,00
	80	13,04
	90	13,02
0,8 mol	50	12,60
	60	13,82
	70	16,13
	80	14,05
	90	13,23
1 mol	50	13,55
	60	14,72
	70	17,45
	80	15,52
	90	14,99

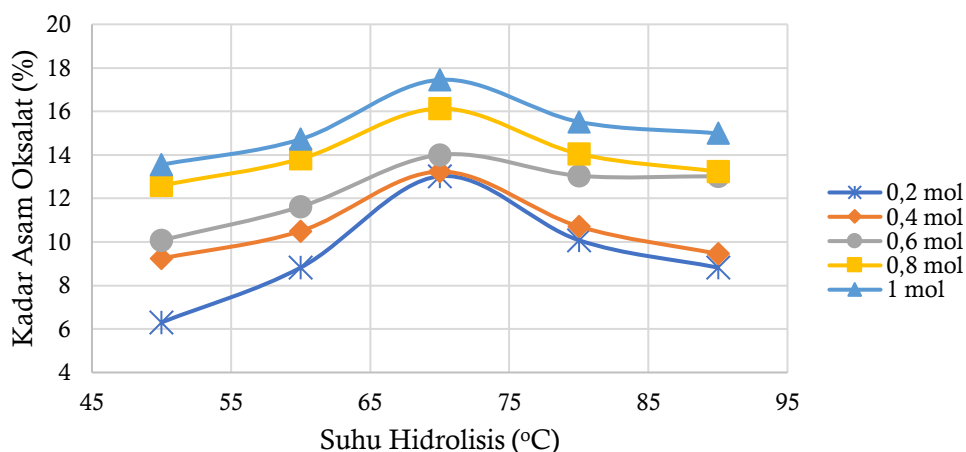


Figure 3. Relationship Between Hydrolysis Temperature (°C) and Oxalic Acid Concentration at Different Cellulose Moles (Mol)

Figure 1 shows that the concentration of oxalic acid increases with the increase in the number of moles of cellulose. This increase occurs because, during the hydrolysis process, the amount of cellulose moles influences the conversion rate of oxalic acid produced. However, in this study, not all cellulose moles were fully converted into oxalic acid, resulting in a decrease in yield after the cellulose conversion reached its optimum point at 70°C. The decline in oxalic acid production is attributed to further decomposition or decarboxylation reactions. Similarly, with the influence of cellulose moles, the temperature variable also shows an increase in oxalic acid concentration as the temperature rises. When the hydrolysis process exceeds the optimum temperature of 70°C, the concentration of oxalic acid formed decreases. This is due to the high sensitivity of the basic catalyst at elevated temperatures, which triggers further reactions with oxalic acid, known as decarboxylation. In the decarboxylation reaction, oxalic acid is oxidized into carbon dioxide and formic acid. Based on the research by (Nurul, 2017), the optimum results were obtained at a temperature of 70°C with a hydrolysis time of 60 minutes. When the temperature exceeded 70°C, the oxalic acid yield decreased. This finding is supported by (Wulandari, 2021) research, which states that the relationship between cellulose mole conversion and temperature on oxalic acid yield is directly proportional. However, once cellulose reaches its optimum point, the product yield decreases due to decarboxylation reactions.

Effect of Hydrolysis Temperature on Oxalic Acid pH

The obtained oxalic acid was then subjected to pH analysis using a pH meter, with each sample tested in triplicate to ensure greater accuracy in the pH readings. The results of the pH testing are presented in Figure 2

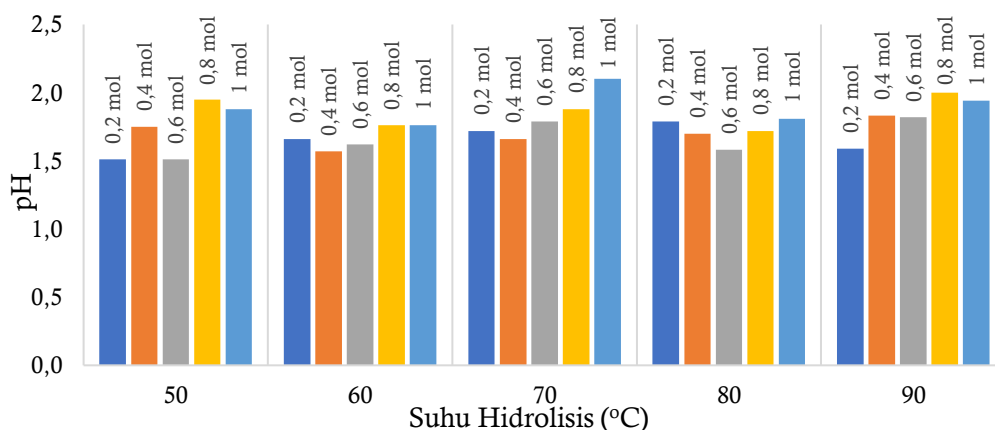


Figure 4. Relationship Between Hydrolysis Temperature (°C) and Oxalic Acid pH at Different Cellulose Moles (Mol)

Figure 2 shows the relationship between hydrolysis temperature and pH of oxalic acid formed fluctuates. This is due to the decreasing ability of sulfuric acid to convert calcium oxalate into oxalic acid, resulting in a higher pH of the formed oxalic acid at the optimum temperature compared to other temperatures. According to (Ecolab, 2020), standard oxalic acid crystals have a pH range of 1,1–2,1. Meanwhile, the pH of oxalic acid derived from kapok fiber falls within a range of 1,51–2,1. This confirms that the product obtained is oxalic acid.

Melting Point Analysis Results

Melting point testing is a quantitative analytical technique used to identify the chemical substances formed and to provide information about the purity and physical properties of a chemical compound. In this study, the melting point of oxalic acid from kapok fiber as 101,5°C, which aligns with the melting point of oxalic acid reported by (Perry et al. 1997) as 101,5°C. This confirms that the crystals obtained are indeed oxalic acid.

FTIR Analysis Results

In Fourier Transform Infrared (FTIR) testing, samples in the form of crystalline powder are analyzed using the KBr method. The wavelength of the spectral peak indicates the presence of specific functional groups in the sample, which is the basis of this analysis. This FTIR analysis was carried out at the Pharmacy Laboratory, Faculty of Pharmacy, Universitas Airlangga.

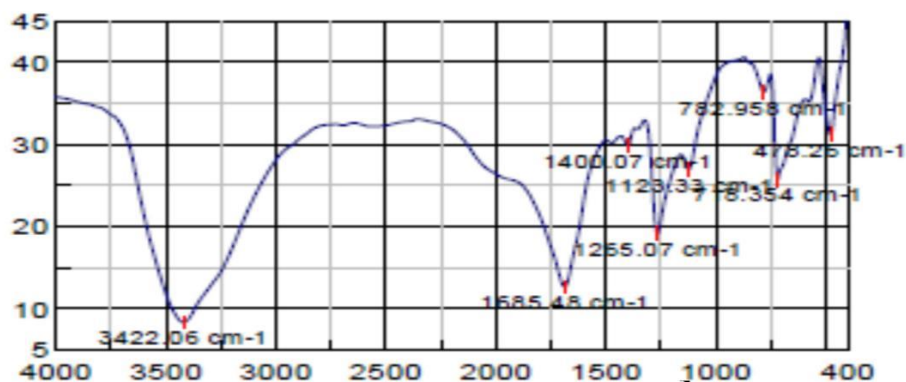


Figure 5. Infrared Spectrum (FTIR) of Standard Oxalic Acid (Sumber: Iriany, 2015)

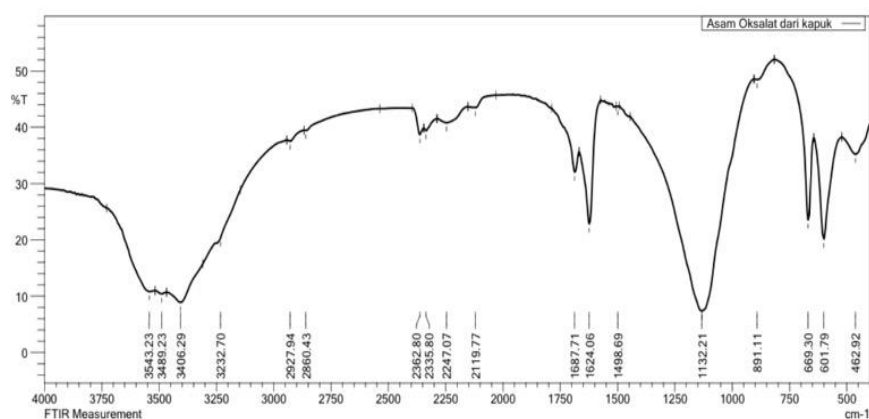


Figure 6. Infrared Spectrum (FTIR) of Synthesized Oxalic Acid

Figure 3 shows the infrared (IR) spectrum of standard oxalic acid, while Figure 4 displays the infrared (IR) spectrum of synthesized oxalic acid for several functional groups. For the hydroxyl group (O-H), the standard oxalic acid exhibited a peak at a wavelength of 3422.06 cm⁻¹, whereas the synthesized oxalic acid showed a peak at 3406.29 cm⁻¹. For the carbonyl group (C=O), the standard oxalic acid displayed a peak at 1685.48 cm⁻¹, and the synthesized oxalic acid showed a peak at 1687.71 cm⁻¹. For the ether group (C-O), the standard oxalic acid had a peak at 1123.33 cm⁻¹, while the synthesized oxalic acid showed a peak at 1132.21 cm⁻¹. Finally, for the

aromatic ring group (C-H), the standard oxalic acid exhibited a peak at 718.35 cm^{-1} , and the synthesized oxalic acid showed a peak at 669.30 cm^{-1} .

The comparative analysis of functional groups in standard oxalic acid and synthetic oxalic acid revealed distinct characteristics, as detailed in Table 2.

Tabel 2. Wavelength Comparison of Synthesized Oxalic Acid with Standard Oxalic Acid

Cluster	Wavelength (cm^{-1})	
	Synthesized Oxalic Acid	Standard Oxalic Acid
O-H	3406,29	3422,06
C=O	1687,71	1685,48
C-O	1132,21	1123,33
C-H	669,30	718,35

Table 2 shows a comparison of the wavelengths of synthesized oxalic acid and standard oxalic acid. The (C=O) and (C-O) functional groups did not exhibit significant differences in wavelength. However, significant differences were observed in the hydroxyl (O-H) and aromatic ring (C-H) functional groups. The (O-H) group showed a broadening of the vibration spectrum due to the alcohol content in the analyzed sample. The results of the Fourier Transform Infrared (FTIR) test confirmed that this study successfully produced oxalic acid. However, the oxalic acid derived from kapok fiber is not yet fully pure, as impurities are still present in the compound.

CONCLUSION

Based on the research results, the highest oxalic acid content of 17.45% was obtained at 1 mol of cellulose and a hydrolysis temperature of 70°C , while the lowest content of 6.3% was found at 0.2 mol of cellulose and a temperature of 50°C . The higher the temperature and the amount of cellulose, the more oxalic acid is produced. To improve yield, future research should implement a delignification process to obtain purer cellulose. These findings have potential applications in the textile, pharmaceutical, metallurgy, and metal-cleaning industries. Utilizing kapok fiber as an alternative raw material offers an environmentally friendly solution compared to chemical synthesis methods. Practically, this research can support industrial-scale production by increasing efficiency, reducing agricultural waste, and promoting the use of renewable resources for high-value products.

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