

OXALIC ACID PRODUCTION OF BANANA PEEL (*Musa textilia*) THROUGH OPTIMUM TEMPERATURE AND TIME with ALKALINE METHOD

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ABSTRACT

The chemical compound has the formula $H_2C_2O_4$ is oxalic acid. Every year especially, the industrial sector in Indonesia requires oxalic acid made, which increases annually. Oxalic acid is valuable because it may be used to clean (rust remover), make colors, protect metals from corrosives, and other things. Alkaline melting is one way for creating oxalic acid from cellulose-containing materials. Other methods include hydrolysis, $CaCl_2$ precipitation, and acidification using H_2SO_4 . In Indonesia, cellulose-containing materials are widely available. Banana peels are one of the substances that contain cellulose. In addition to identifying the properties of oxalic acid produced using the alkaline smelting process, this study attempts to find the best temperature and duration for producing oxalic acid from discarded banana peels. The ideal conditions include a temperature of $60^\circ C$, a period of 60 minutes, and a yield of 5.42%. Oxalic acid from banana peel waste has the properties of having a melting point, hydroxyl, C=C, C-O, and C-H group absorption at a wave number of $104.6^\circ C$, 3422.06 cm^{-1} , 1682.48 cm^{-1} , 1103.42 cm^{-1} , and 666.36 cm^{-1} respectively. Based on the strain vibration results from the FTIR test and melting point test.

1. INTRODUCTION

Indonesia still imports raw materials and chemical products in large quantities rather than producing its own to meet domestic demand and exports abroad. Of the significant import of these chemicals, the state expenditure or expenditure is getting bigger. Therefore, it is necessary to make efforts to meet the production of domestic substances and to reduce the consumption of chemicals from abroad. Among other reasons, the growth of a chemical industry that produces goods is crucial because it can lessen Indonesia's reliance on foreign industries. The costs of importing these materials in terms of foreign cash include oxalic acid.

Today's market for oxalic acid is 350 000 t yr⁻¹, but in the future, oxalic acid may serve as the starting point for a variety of high-value and high-volume compounds. Formic acid/formate (market size: 900 000 t/yr) and MEG (market size: 30 000 000 t/yr) are two examples. Of course, we can make these molecules from various sources, but by 2050, the only carbon sources that will substitute for fossil fuels are CO₂ and biomass (Centi et al., 2019; Perathoner & Centi, 2019). It can be a powerful platform for carbon materials, such as novel polymer classes (Valderrama et al., 2019). The need for oxalic acid in Indonesia is expected to continue to increase by the number of industries that use it; therefore, establishing a domestic oxalic acid factory is also expected to open up new jobs.

The banana plant is a climacteric fruit and is one of the most extensive fruit plantations in the world (Amini Khoozani et al., 2019). The plantations, which cover an area of more than 2.3 million hectares, are the largest in the world (FAO, 2021; Cordenunsi & Lajolo, 1995). Bananas account for 16.8% of global fruit availability, followed by apples and oranges at 11.4% (FAO, 2021). Bananas are tropical fruit often consumed with high nutritional value (Vu et al., 2017; Gómez et al., 2019). Bananas can be processed into various products, such as pesticides, snacks, or food coloring (Farahmandfar et al., 2017; Majaliwa et al., 2019; Yan et al., 2016; Sartori et al., 2017).

The existence of banana peel waste is often found in the surrounding environment, so it can pollute the environment. Thus the utilization of banana peel waste still needs to be improved. Many banana peels will have high economic value if used properly. Banana peel is a waste from the rest of the production of snacks (such as banana chips and banana sales) which, in most cases, are only used as animal fodder. According to a comparative study, peels are high in total dietary fiber (64.33 g/100 g), folate (33.12 mg/100 g), and potassium (35.61 mg/100 g) (Arun et al., 2015). Another research also mentioned that banana peels contained some compositions such as protein (3.74%), carbohydrates (56.35%), followed by ash (12.62%), fiber (15.30%), crude fat (4.34%), and moisture content (7.65%) (Singanusong et al., 2013).

In the process of using alkaline smelting, the materials used contain cellulose which will then be reacted with alkali hydroxides such as sodium hydroxide (NaOH) or potassium hydroxide (KOH). In the alkaline smelting process from carbohydrates this material is melted with sodium hydroxide or potassium hydroxide at a temperature of 240–285° C, which usually uses NaOH because NaOH has a more efficient price than KOH, besides that the concentration of alkali used can also affect the process of making oxalic acid because the lower the concentration alkali is used, the reaction is slow (Cinantya, 2015).

Banana peel is a raw material used to make oxalic acid. Cellulose in banana peels reacted with strong alkalis to produce oxalic acid, acetic acid, and formic acid. This reaction with strong alkali is often called hydrolysis or smelting. This process is carried out by variable cooking time and temperature, consistency or comparison between the banana peel and the

solution added in the cooking process, and acid concentration in the hydrolysis process (Fatmawati et al., 2017).

This treatment is intended to take advantage of optimal operating conditions to obtain maximum results. Because the formation of oxalic acid is influenced by the melting time and the concentration of NaOH, based on the description above, in this research, The alkaline fusion process will be used to produce oxalic acid from banana peels. Variables in this procedure include the number of banana peels used, the consistency of the solution added during the cooking phase, and the amount of acid used during the hydrolysis step. This procedure aims to maximize outcomes by utilizing the best possible working circumstances.

2. METHODS

This research methodology chapter, it is explained the time and place of the research. The equipment and supplies utilized in the research on the manufacture of oxalic acid from banana peels using the alkaline smelting method, an explanation of the process flow, variables used, analysis, and various characteristics to be carried out in the research.

2.1. Research Time and Place

The preparation time and this research were from October 2021 to February 2022. This research was carried out at the Physical Chemistry Laboratory of the Semen Indonesia International University. Implement the melting point test in the material physics laboratory at Brawijaya University, Malang. While FTIR testing is carried out in the energy and environment laboratory at the Sepuluh Nopember Institute of Technology (ITS) Surabaya.

2.2. Tools and materials

2.2.1. Research Tools

The equipment used in this study was a series of reflux apparatuses, three neck flasks, stative, clamps, a bulb, an analytical balance, a beaker, a watch glass, an erlenmeyer 250 mL, erlenmeyer 500 mL, a spatula, a hotplate, volume pipette, Buchner funnel, aqua dest bottle, reagent bottle and 1000 mL volumetric flask.

2.2.2. Research Material

The materials used in the research were banana peel waste, sodium hydroxide (NaOH) 40%, saturated calcium chloride (CaCl_2), ethanol ($\text{C}_2\text{H}_5\text{OH}$) 96%, sulfuric acid (H_2SO_4) 4 N, aqua dest (H_2O), filter paper *Whatman*, aluminum foil.

2.3. Research methods

2.3.1. Research Variables

The variables studied in the research on the manufacture of oxalic acid from banana peels using the alkaline smelting method are as follows:

Fixed variable: the number of materials

Changed variable: time and temperature

Table 1 Table of process variables in the manufacture of oxalic acid from banana peels using the alkaline smelting method

Variables	Units	Treatments
Temperatures	°C	60, 80, 100
Times	Minutes	50, 60, 70, 80

2.3.2. Oxalic Acid Manufacturing Process(Iriany et al., 2015)

Banana Peel Hydrolysis

The following are the steps of work carried out in the hydrolysis of banana peels(Iriany et al., 2015):

1. Cut the banana peel into small pieces
2. Dry the banana peel until the water content is gone
3. Smooth the banana peel until it becomes powder
4. Weigh as much as 15 grams
5. Assemble tools, set the time and desired temperature
6. Put the weighed raw materials into a three-neck flask, then add NaOH with a concentration of 40% as much as 200 mL
7. Set the time used, namely 50, 60, 70, and 80 minutes at temperatures of 60° C, 80° C, and 100° C, respectively, then heat the three-neck flask
8. The solution is cooled after the heating is finished
9. Filter the filtrate into a 500 mL beaker then the remaining precipitate is washed using hot distilled water into a beaker containing the filtrate up to 400 mL
10. Repeat the procedure with variations of each time and temperature

2.3.3. Oxalic Acid Crystallization

The following steps work for oxalic acid crystallization(Iriany et al., 2015) :

1. Pipette 25 mL of the filtrate that has been produced by hydrolysis, then add 10% CaCl₂
2. Filter the precipitate and then add 100 mL of H₂SO₄4N
3. Heating the filtrate to a temperature of 70° C.
4. Cool the filtrate in ice water for approximately 24 hours to form oxalic acid crystals.
5. Wash oxalic acid using 96% ethanol and heat at 70° C.
6. Cool the filtrate in ice water for about 24 hours to form oxalic acid crystals.
7. Filter oxalic acid crystals in the oven at 80° C for 30 minutes.
8. Weighing the yield of oxalic acid

Calculation of the weight of oxalic acid (gr)

$$\text{Crystal weight} = \text{filter paper} + \text{crystal} - \text{blank paper}$$

2.3.4. Oxalic Acid Testing Process

FTIR Test

The following are the working steps of the FTIR test in the oxalic acid testing process.

The process of the FTIR spectroscopy instrument is as follows (Syafiqoh, 2015):

1. Energy source: Infrared energy is emitted from a glowing black-body source, and the resulting beam is passed through a slit which can control the amount of energy hitting the sample.
2. Interferometer: Light enters the interferometer where it will be converted into an interferogram signal that will exit the interferometer.
3. Sample: Light enters the sample chamber and is then transmitted/reflected from the sample surface depending on the type of analysis used.
4. Detector: The beam is passed to the detector as the final measurement.
5. Computer: The measured signal will be read/recorded on the computer as a chromatogram.

2.3.5. Melting Point Test

Melting point determination following the standard method (Young, 2013) with a slight change, namely + 1 gram of oxalic acid, is included into the capillary tube placed on the melting point determination plate, then the tool is turned on. Then observed and recorded the melting point temperature.

3. RESULTS AND DISCUSSION

3.1. The Effect of Time and Temperature on the Yield of Oxalic Acid Produced

This study used temperature variables (60°C, 80°C, and 100°C) and time variables (50, 60, 70, and 80 minutes). The purpose of the temperature and time variables in this study is to ascertain how the temperature and melting time affect the yield of oxalic acid. The relationship between temperature and time and the yield of oxalic acid produced is direct.

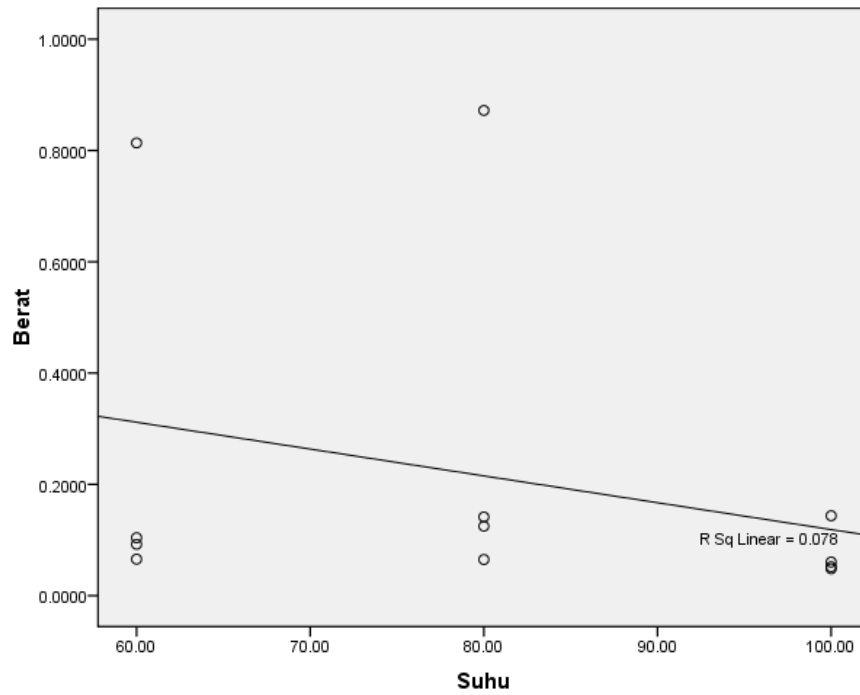


Figure 1 Diagram of the Relationship Temperature to the Yield of Oxalic Acid Formed (%)

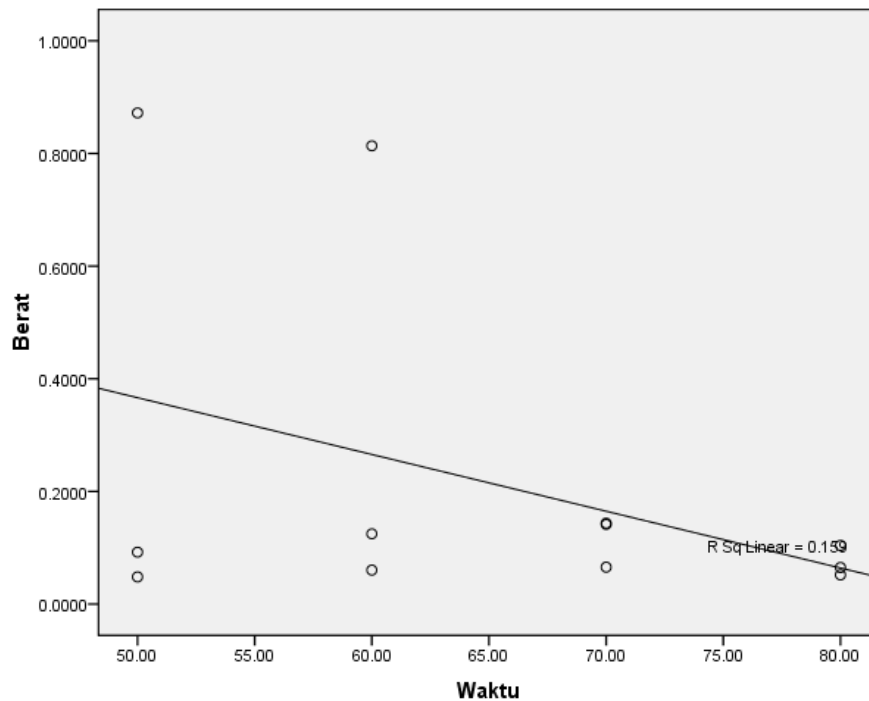


Figure 2 Diagram of the Relationship Temperature to the Yield of Oxalic Acid Formed (%)

The research shows that the first and second graphs define optimum smelting temperature and smelting time, affecting the weight or yield of the resulting oxalic acid. However, at 2 certain points, the yield of oxalic acid increased, namely at 60°C for 60 minutes by 0.8136, and at 80°C for 50 minutes by 0.872. If the temperature is high, the reaction rate

constant gets bigger, so it is getting bigger too. However, too high a temperature decomposes oxalic acid and affects the yield. An extended reaction time will produce a large amount of oxalate, but if the time is too long, it will cause a further reaction, reducing the result (Nurul et al., 2017).

With the results that have been known, the higher the temperature and time used, the greater the yield of oxalic acid produced, but at a temperature of 60 °C and a time of 60 minutes there was a very significant jump in results compared to other variables. Different treatments occurred from other variables. This treatment occurred at the crystallization stage, which was more than the specified time, namely 24 hours, but at 60°C, and the 60-minute time was up to 48 hours. Therefore, the oxalic acid used was obtained more than the other variables; this can happen because if, within 24 hours, the oxalic acid crystals formed are less than optimal, the results obtained are very different.

In this experiment, the next stage is smelting, namely reacting 15 grams of plantain peel with 200 ml of 40% NaOH. Using NaOH accelerates the hydrolysis process or the breakdown of lignocellulose; the solution obtained from this hydrolysis stage is a simple mother liquor, where the product is formed—namely sodium oxalate with by-products namely sodium acetate and sodium formate. Cellulose undergoes a hydrolysis process in an alkaline solution, and degradation of cellulose by an alkaline process occurs as a base-catalyzed cleavage of glycosidic bonds (hydrolysis) (Siswanto et al., 2022). The sedimentation and filtering stages are carried out to remove the by-products.

3.2. Sodium Oxalate (COONa)₂

In the hydrolysis stage by adding 200 ml of 40% NaOH with time variables of 50, 60, 70, and 80 minutes and temperatures of 60°C, 80°C, and 100°C, aiming to hydrolyze the cellulose contained in the banana peel or break it from lignocellulose. Which will form sodium oxalate (COONa)₂ and by-products sodium acetate (CH₃COONa) and sodium formate (HCOONa) with the following reaction :



After hydrolysis, the plantain peel using NaOH is separated from the by-products by filtering the hydrolysis results and mixing 200 ml of distilled water to facilitate the filtering process of the filtrate and sediment contained in the filtrate will be separated from the sediment or by-product of the hydrolysis process.

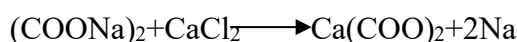
In addition to filtering the separation of by-products, precipitation is added with 10% calcium chloride (CaCl₂), which has a function to bind sodium oxalate (COONa)₂ from by-products during hydrolysis, namely sodium acetate (CH₃COONa) and sodium formate (HCOONa) which will form a white precipitate.

3.3. Oxalic Acid (COOH)₂

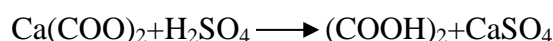
This study's precipitation stage was adding 10% calcium chloride (CaCl₂) solution. The function of adding 10% (CaCl₂) is so that the solution forms calcium oxalate (COO)₂Ca and sodium chloride (NaCl) solution. Then filtering is carried out, which aims to separate calcium oxalate (COO)₂Ca and sodium chloride (NaCl) solution, at the addition of 10% (CaCl₂) with calcium oxalate filtrate with a ratio of 1:1, namely 25 ml of CaCl₂ and 25 ml of potassium oxalate filtrate. No precipitate will appear when adding 10% (CaCl₂) to more than 25 ml of the solution. The CaCl₂ exceeds the total filtrate taken by only 25 ml from 400 ml of filtering.

A white precipitate indicates that potassium oxalate has been bound by calcium chloride (CaCl_2) and separated from the by-products of hydrolysis.

The addition of 10% CaCl_2 will form calcium oxalate $(\text{COO})_2\text{Ca}$ and sodium chloride solution (NaCl) with the following reaction :



After calcium oxalate $(\text{COONa})_2$ is formed, the next step is to acidify it by adding 100 ml of sulfuric acid (H_2SO_4) 4N; the addition of sulfuric acid is carried out after the calcium oxalate is filtered to separate the calcium oxalate $(\text{COO})_2\text{Ca}$ and sodium chloride solution (NaCl). After the separation, the next step is the acidification of the calcium oxalate obtained by adding 100 ml of 4N sulfuric acid (H_2SO_4), which will form oxalic acid $(\text{COOH})_2$ and calcium sulfate CaSO_4 with the following reaction:



The supersaturation of the oxalic acid solution allows for the formation of oxalic acid due to the solvent's diminished capacity to dissolve the solute. Evaporation and chilling are two methods for reducing the amount of solvent.

3.4. Fourier Transform Infrared (FTIR)

Samples in the form of oxalic acid crystals from banana peel waste were obtained from the alkaline smelting method (NaOH) and identified the presence of oxalic acid compounds using Fourier Transform Infra-Red (FTIR). Fourier Transform Infra-Red (FTIR) was used to test the presence of oxalic acid groups in the resulting sample. The way this FTIR works is that the sample is scanned so that infrared rays will pass through the sample; then, the wave transmitted by the sample will be detected by a detector connected to a computer so that it will provide an overview of the absorption spectrum of the sample (Sjahfirdi et al., 2015).

This analysis is founded on a study of the wavelength of a sample's distinguishing peaks. The wavelength of these peaks indicates the presence of specific functional groups in the sample because each has characteristic peaks specific to certain functional groups. The infrared spectrum of standard oxalic acid and synthesized oxalic acid can be seen in the following figure:

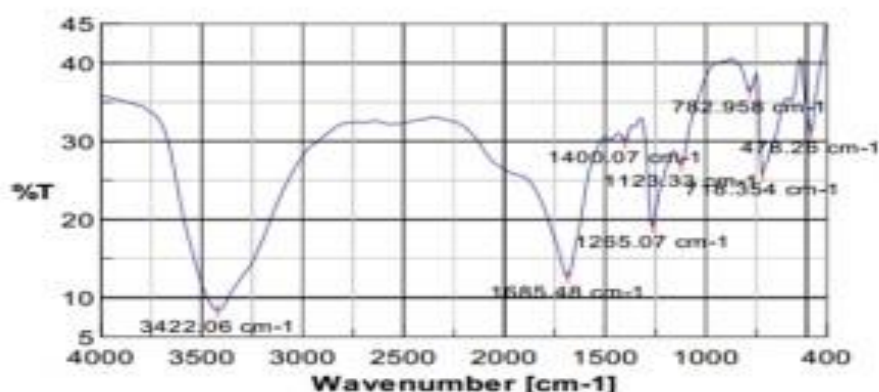


Figure 2. Infrared Spectrum of Standard Oxalic Acid

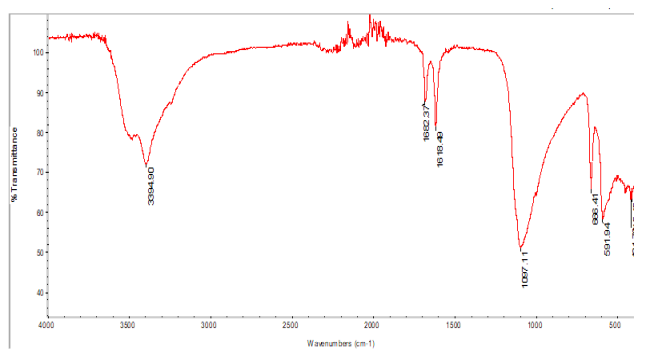


Figure 3 Results of Infrared (IR) Absorption of Oxalic Acid from Banana Peel Waste

In the FTIR test, the oxalic acid, which was analyzed for purity, was produced in the experiment with variable 60 with a temperature of 60°C. Based on the figure, it is known that the strain vibration of the O-H group of standard oxalic acid is 3422.06 cm⁻¹. For the results of the O-H group on oxalic acid from plantain peel 3396.24 cm⁻¹. Then for the strain vibration of C=C standard oxalic acid at wave number 1685.48 cm⁻¹ for synthetic oxalic acid at wave number 1682.48 cm⁻¹. The strain vibration of the C-O group of standard oxalic acid is found at a wave number of 1123.33 cm⁻¹, then for synthetic oxalic acid at a wave number of 1103.42 cm⁻¹. The strain vibration of the C-H group of standard oxalic acid is found at a wave number of 718.35 cm⁻¹; for standard oxalic acid, it is 666.36 cm⁻¹. The results of oxalic acid absorption from banana peel waste are pretty close, different from that of acid absorption standard oxalate. The result obtained is oxalic acid. There is a difference in absorption between standard oxalic acid and oxalic acid from waste banana peels caused because there are still impurities so that they can be it was concluded that the oxalic acid obtained was still not pure.

3.5. Melting Point Analysis

The melting point is the temperature at which solid changes into a liquid at the pressure of one atmosphere. Oxalic acid crystals tested for melting point analysis in this study were 80 minutes with a temperature of 60°C, and the resulting melting point was 104.6°C. Pure oxalic acid had a melting point of 101.5°C (MSDS, 2021). From the results of the analysis of the melting point test that has been carried out, the results of the synthesis have a value that is not much different from pure oxalic acid, and it can be concluded that the resulting product is oxalic acid.

4. CONCLUSION

The conclusions obtained based on the research that has been done are that the research shows that first and second graphs define optimum smelting temperature and smelting time, affecting the weight or yield of the resulting oxalic acid. However, at 2 certain points, the yield of oxalic acid increased, namely at 60°C for 60 minutes by 0.8136, and at 80°C for 50 minutes by 0.872. Based on this result, we know the optimal temperature and time for oxalic acid production. Furthermore, the characteristics of oxalic acid experienced decreased with high temperature and long time.

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