

# Durian Peel Waste as An Alternative Material for Oxalic Acid Using The Nitric Acid Oxidation Method

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### ABSTRACT

Durian (Durio zibethinus murr) was a tropical fruit from Southeast Asia. Based on the content, the durian peel waste has the potential to be used as a raw material in the process of making oxalic acid. The cellulose content found in durian peel is 50-60%, and there was lignin and starch content of 5% for each. Oxalic acid is an organic compound with the formula C<sub>2</sub>H<sub>2</sub>O4. This study aimed to determine the effect of nitric acid concentration and oxidation time of durian peel on yield, oxalic acid content, and the quality of the oxalic acid produced. The method used in this study was the oxidation process. The influential variables in the oxidation process with nitric acid were the concentration and heating time. The higher the nitric acid concentration, the greater the % yield of oxalic acid produced. The independent variables used were nitric acid concentration and time. This research used a factorial Completely Randomized Design (CRD) method with two factors, namely nitric acid concentration (HNO3) and reaction time, and nine treatment combinations were obtained. The concentrations used were 3 M, 4 M, and 5 M then the times used were 60, 70, and 80 minutes. The ratio between nitric acid and durian peel was 1:10 with a temperature of 75°C. The optimum yield occurred at a concentration of 4M HNO3 with a heating time of 80 minutes, where the yield obtained was 22.50%. Oxalic acid from a citric acid concentration of 5M and a reaction time of 80 minutes resulted in oxalic acid crystals melting at 98.6°C. From the results of this analysis, the synthesized oxalic acid has the same characteristics as pure oxalic acid, so it can be concluded that the product produced from this research is oxalic acid and is classified as dihydrate oxalic acid.

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### **1. Introduction**

Durian has various morphologies, depending on where the durian grows. In Indonesia, there are seven variations of the shape of the durian. Among them are round, ovoid, oval, round flat tip, elliptical, ovoid, and obovoid. In general, durian fruit has a fruit peel color that is green to brown. The market has various types, shapes, and colors of durian for sale. Durian has a fruit peel with sharp, thick, and hexagonal-shaped spines. Several types of durian are popular in Indonesia. Durian has an attractive flesh color and a distinctive aroma (Ho & Bhat, 2015). The use of durian is only limited to the fruit. Many people need to understand that durian peel has a high cellulose content. The cellulose content found in durian peel is 50-60%, and there is a lignin and starch content of 5% each (Hatta, 2007). Cellulose is the most abundant organic material currently produced in the biosphere, with  $5 \times 10^{11}$  tons annually produced (Qiu & Hu, 2013). The community has made several innovations from durian peel waste. Some examples of innovations that the community has made. Based on the content contained in durian peel, durian peel waste can be used as a raw material to make oxalic acid. Oxalic acid is an organic compound with the chemical formula C<sub>2</sub>H<sub>2</sub>O<sub>4</sub>. Oxalic acid has a physical form, a colorless crystalline solid. Oxalic acid has the property of being easily wrinkled in water, and the resulting solution is also colorless. Oxalic acid has various uses in the industrial world, including as a metal cleaner, and can be used as a reagent in chemical analysis processes (Coniwanti et al., 2008).

Previous research was carried out to produce oxalic acid from rice husks by nitric acid oxidation method. Based on the research conducted, the yield was 81% for rice husk and 79.9% for paddy with a raw material ratio of 5:1, the concentration of nitric acid used was 68%, the concentration of sulfuric acid used was 98%, the temperature used is  $75^{\circ}$ C, and the time used is 1.3 hours (Oghome et al., 2012). Other studies were also carried out but derived from palm fronds as raw material using the nitric acid method using variations in temperature and reaction time. In the research, the palm fronds reacted with 40% nitric acid at the ratio of 1:6 (w/v) to obtain the desired oxalic acid. The research process was carried out in several stages: the oxidation reaction stage, filtration, precipitation using calcium chloride (CaCl<sub>2</sub>), acidification using sulfuric acid (H<sub>2</sub>SO<sub>4</sub>), and crystallization stage. Based on the research that has been done, a yield of 23.2% is obtained using a raw material ratio of 6:1, the nitric acid concentration used is 40%, the temperature used is 80°C, and the time is 50 minutes (Ambarita et al., 2015).

The chemical content of durian peel that can be utilized is oxalic acid, where the raw material for manufacturing oxalic acid is glucose contained in cellulose found in the yellowish-white inner fruit peel commonly called the durian albedo. The results showed that durian peels indicated that the material could be used as a mixture of processed food raw materials and processed as a raw material to produce other products. In addition, durian peel waste contains fiber cells with long dimensions and thick enough fiber walls so that they will be able to bond correctly when an adhesive containing synthetic or mineral is added (Arlofa, 2015). Cellulose is a polysaccharide composed of glucose polymers connected by glycosidic bonds that form straight chains. Disaccharides will be produced by partial hydrolysis of cellulose, and D-glucose will be more easily oxidized to produce oxalic acid with strong acids (Melwita & Kurniadi, 2014).

Based on these research studies, it is necessary to research manufacturing oxalic acid from durian peel waste (*Durio zibethinus Murr*). This study aimed to determine the effect of nitric acid concentration and oxidation time of durian peel on yield, oxalic acid content, and the quality of the oxalic acid produced. Oxalic acid obtained from durian peel waste (*Durio zibethinus murr*) will be determined for its properties or characteristics, such as Fourier Transform Infrared Spectroscopy (FTIR) analysis to test the presence of functional groups in

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oxalic acid compounds, analysis of cellulose content as a qualitative analysis of oxalic acid, and determination of the melting point as a test for the physical properties of oxalic acid.

### 2. Methods

Durian peel was collected by purchasing from durian traders in the market. Durian peel preparation begins by cutting the durian peel into small pieces to speed up the drying process, then drying the durian peel in an oven for 30 minutes at 105°C to remove the moisture content. After that, the durian peel was put into the desiccator for 10 minutes, then weighed. The next stage is drying until a constant mass of durian peel is obtained, and the last stage is smoothing the durian peel using a blender and sifting using a 50-mesh sieve. This research used a factorial Completely Randomized Design (CRD) method with two factors, namely nitric acid concentration (HNO3) and reaction time. It was obtained nine treatment combinations as follows:

No.	Nitric Acid Concentration (%)	Reaction Time (Minutes)	<b>Reaction</b> Temperature (°C)	Rasio durian peel: HNO3 (w/v)
1	214	60		
2	JIVI	70		
3		80		
4		60	-	
5	4M	70	75	1:10
6		80		
7		60	-	
8	5M	70		
9		80		

Table 1	Experimental	design
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In synthesizing oxalic acid from durian peel, a delignification process is required using 15 grams of durian peel powder, which is put into a 250 mL three-neck flask and added with 40% NaOH. The reflux process was carried out for 1 hour using a temperature of 100°C. After that, the samples were washed using distilled water until the pH was neutral. After that, the samples were dried in an oven for 1 hour at 105 °C. The hydrolysis process was begun by taking 15 grams of delignified durian peel powder in a 250 mL three-neck flask, then refluxing after adding 150 mL of  $H_2SO_4 2$  N solution for 1 hour at 100°C then filtering the solution using and taking the filtrate.

The oxidation reaction stage was carried out by incorporating 50 mL of the hydrolyzed filtrate into a 250 mL three-neck flask and then adding 150 mL of 3 M nitric acid. After that, reflux at 75°C for 60 minutes and continue with filtering and washing until the filtrate becomes apparent; the resulting residue is used for the cellulose content analysis test stage. The process of adding a 10% CaCl<sub>2</sub> solution to the filtrate was then allowed to stand for 12 hours until a precipitate was formed. The precipitate formed was dissolved in 200 mL of 2 N H<sub>2</sub>SO<sub>4</sub>, then filtered and washed using 96% ethanol. After that, a water bath evaporated the filtrate at 80°C for about 1 hour. A precipitate of oxalic acid in the form of white needle crystals will be produced after cooling the filtrate for 24 hours. The important thing that needs to be done is to purify the results that have been obtained by using a recrystallization process using 96% ethanol solvent and repeating all the process steps with a predetermined variation of nitric acid

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concentration (3M, 4M, 5M) and different reaction times (60, 70, 80 minutes) (Coniwanti et al., 2008; Yenti & Herman, 2012).

The recrystallization stage was carried out by dissolving the crystals obtained using 200 mL of 96% ethanol. The ratio of ethanol is based on the high solubility of oxalic acid crystals in ethanol, which is 400 mg/ml. The following process is heating the solution until the crystals dissolve entirely and filtering the hot solution little by little. After that, cool the filtrate until new oxalic acid crystals form, then filter the solution and dry the crystals in a desiccator.

## 2.1. Analysis of Water Content

The water content analysis process is carried out using oven drying, namely as follows:

- 1. Cut the durian skin into small pieces.
- 2. Weigh the durian skin and chop the mass obtained (a).
- 3. Dry the durian skin using an oven at a temperature of 105°C.
- 4. Put the dried durian skin into the desiccator.
- 5. Weigh the dried durian skin using an analytical balance and record the mass obtained (b).
- 6. Calculation of water content using the following equation:

Water content = 
$$\frac{a-b}{a} \times 100\%$$
 .....(3.1)

## 2.2. Cellulose Content Analysis

The process of analyzing cellulose content is carried out using the Chesson-Datta method (Dzikro, 2013), which is as follows:

- 1. Add 150 mL  $H_2O$  to 1 gram (a) of dried durian skin and reflux at a temperature of  $100^{\circ}C$  using a water bath for 1.
- 2. Filter the mixture and wash the residue with 300 mL of hot water.
- 3. Dry the residue using an oven until a constant weight is obtained (b).
- 4. Add 150 mL of 1 N H<sub>2</sub>SO<sub>4</sub> to the residue and conduct a reflux process in a water bath for 1 hour at 100 100°C.
- 5. Filter the results obtained until they are neutral or 300 mL, and carry out the drying process (c).
- 6. Add 10 mL of 72% H<sub>2</sub>SO<sub>4</sub> to the dry residue and carry out the soaking process for 4 hours at room temperature.
- 7. Add 150 mL of 1N H<sub>2</sub>SO<sub>4</sub> and reflux using a water bath for 1 hour in reverse cooling.
- 8. Filter the residue and wash using H<sub>2</sub>O until neutral or 400 mL.
- 9. Heat using an oven at 105°C.
- 10. Weighing the results obtained using an analytical balance (d)
- 11. Calculation of cellulose content using the following equation:

Cellulose content = 
$$\frac{c-d}{a} \times 100\%$$
 .....(3.2).

## 2.3. Yield Analysis

The yield analysis procedure was carried out by adopting the procedure carried out by previous researchers (Zultiniar, 2014), namely as follows:

- 1. Weigh the oxalic acid crystals by first weighing the mass of the filter paper used using an analytical balance.
- 2. Calculate the oxalic acid yield using the following equation:

$$Yield (\%) = \frac{mass of oxalic acid crystal}{mass of durian peel} \times 100\% \dots (3.3) (Atikah, 2017).$$

## 2.4. Melting Point Analysis

The melting point analysis procedure adopts the procedure carried out by previous researchers (Zultiniar, 2011), which is as follows:

- 1. Insert oxalic acid crystals into the capillary tube.
- 2. Place the capillary tube on the heating part of the melting point determination tool and cover the other holes using metal.
- 3. Turn on the heater.
- 4. Adjust the heater by adjusting the coarse and fine temperature control buttons so that the heater speed shows a temperature increase of 1-2°C per minute.
- 5. Observe and record the temperature of the oxalic acid crystals being analyzed, then press the display button when the crystals start to melt until the oxalic acid crystals melt.

## 2.5. Purity Analysis

The process stage of analyzing the purity of oxalic acid uses a tool called infrared spectrophotometry (infrared spectroscopy). The purity analysis stage using infrared spectrophotometry follows the work method carried out by previous researchers (Elmila, 2011), namely as follows:

- 1. Add several grams of KBr to the oxalic acid sample and grind until smooth.
- 2. Insert into the pellet and press evenly.
- 3. Carefully move the formed pellets into the cell holder using a spatula.
- 4. Set the Infrared Spectrophotometer (FTIR) with the paper speed in the "normal" position. If the scale of the paper is correct, in the same way, a red spectrum is made from the prepared sample and determines its functional groups.

## 3. Results and Discussion

### 3.1. Results

Oxalic acid has a white solid form and has no odor (PT. Smart Lab Indonesia, 2019). The oxalic acid produced in this research has the same form as pure oxalic acid. The tests used in this research are quantitative testing and qualitative testing. This is what was done in many previous studies. Quantitative testing, namely by calculating the % yield, while qualitative testing uses experimental methods, namely FTIR analysis and melting point analysis. Another qualitative test for oxalic acid analysis is the permanganometric titration and acid-base titration methods.

## 3.1.1. Water Content

Water content analysis was carried out using the oven-drying method. The oven or drying method is one of the methods that is used to measure the water content in food with the principle that the water contained in a material will evaporate if the material is heated at a temperature of 105°C for a specific time and the difference between the weight before and after heating is the content water of the material. The precision and accuracy of determining water content values using the oven method have become a reference for the Indonesian National Standard (01-2891-1992) regarding water content using the oven method (Prasetyo et al., 2019). From the results of the analysis of the water content in durian peel, it was found that the weight of the durian peel before using the oven method was 16.1991gr, then after the drying process using an oven for 90 minutes, the weight of the durian peel was 10.3751 gr so that the water content in the durian peel was 36%.

#### 3.1.2. Cellulose Content

Durian peel contains lignocellulose, which consists of cellulose, hemicellulose, and lignin (Arlofa, 2015). The cellulose content in durian skin must be analyzed to determine its potential as a raw material for making oxalic acid. The cellulose contained in durian peel waste will be synthesized into oxalic acid through an oxidation reaction using nitric acid. Cellulose content analysis was carried out using the Chesson-Datta method. For international standards for analysis of cellulose content, follow the SNI 14-04444-1998 method. The cellulose content of durian skin waste obtained from the analysis results was 63%. The results of this analysis are close to the results in the literature, which state that the cellulose content of oil palm fronds is 60%. Differences in growing location, soil structure, and age of the durian tree cause this difference in results.

### 3.1.3. Effect of Reaction Time

The resulting yield will be higher for longer reaction times, but if the reaction time is too long, the yield will continue to decrease. The decrease in yield was caused by a further oxidation reaction, where the oxalic acid produced from the oxidation of  $HNO_3$  against carbohydrates contained in durian skin then underwent an oxidation reaction to produce  $CO_2$  gas,  $NO_2$  gas, NO gas, and  $H_2O$ . The effect of reaction time on oxalic acid yield can be seen in the following picture:



Figure 2 The Relationship Between Yield and Time for The Comparison Of HNO3 with Reaction Time



Figure 3 Yield Versus Time Relationship For The Ratio Of HNO<sub>3</sub> – Reaction Time at A Concentration Of 3M

Based on Figure 3, it can be seen that the relationship between yield and time increases to a certain point, where when heating at a temperature of 60 to 70 minutes, the yield percentage increases and 70 minutes is the optimum heating time. This is because HNO<sub>3</sub> completely oxidizes the glucose content in the skin. If heating is continued for longer, the percent yield obtained will decrease. This is because a further oxidation reaction occurs, which produces  $CO_2$  gas,  $NO_2$  gas, NO gas, and  $H_2O$  at a concentration of 3M HNO<sub>3</sub>, an optimum yield was obtained, namely 3.12% for 70 minutes, with a weight ratio between durian skin powder and HNO<sub>3</sub>, namely 1 10. The longer the reaction time, the higher the resulting yield. However, if the reaction time is shorter, the yield will continue to decrease (Atikah, 2017).



Figure 4 Yield Versus Time Relationship for The Comparison Of  $HNO_3$  – Reaction Time at A Concentration Of 4M

Based on Figure 4, it can be seen that the relationship between yield and time increases to a certain point, where when heating at a temperature of 60 to 80 minutes, the yield percentage increases and 80 minutes is the optimum heating time. This is because HNO<sub>3</sub> completely oxidizes the glucose content in the skin. If heating is continued for longer, the percent yield obtained will decrease. This is because a further oxidation reaction occurs, which produces CO<sub>2</sub> gas, NO<sub>2</sub> gas, NO gas, and H<sub>2</sub>O. At a concentration of 4M HNO<sub>3</sub>, the optimum yield was obtained, namely 4.33% for 80 minutes with a weight ratio between durian skin powder and with an increase in concentration HNO3, namely 1: 10. The longer the reaction time, the higher the yield. However, if the reaction time is shorter, the yield will continue to decrease (Atikah, 2017).



Figure 5 Yield Versus Time Relationship for The Ratio Of HNO<sub>3</sub> – Reaction Time at A Concentration Of 5M

Based on Figure 5, it can be seen that the relationship between yield and time increases to a certain point, where when heating at a temperature of 60 to 80 minutes, the yield

percentage increases and 80 minutes is the optimum heating time. This is because  $HNO_3$  completely oxidizes the glucose content in the skin. If heating is continued for longer, the percent yield obtained will decrease. This is because a further oxidation reaction occurs, which produces  $CO_2$  gas,  $NO_2$  gas, NO gas, and  $H_2O$ . At a concentration of 5M HNO<sub>3</sub>, the optimum yield was obtained, namely 3.73% for 80 minutes, with a weight ratio between durian skin powder and  $HNO_3$ , namely 1 10. The longer the reaction time, the higher the resulting yield. However, if the reaction time is shorter, the yield will continue to decrease (Atikah, 2017).

### 3.2. Discussion

#### 3.2.1. Effect of Nitric Acid Concentration

The higher the concentration of HNO<sub>3</sub>, the higher the yield of oxalic acid produced will be at a certain point. An increased concentration or increasingly concentrated HNO<sub>3</sub> means that the amount of HNO<sub>3</sub> available to oxidize the glucose contained in durian skin will increase. The graph of the relationship between time and yield in Figure 4 shows this. The optimum yield occurs at a concentration of 4M HNO<sub>3</sub> with a heating time of 80 minutes, where the yield obtained is 4.33%, and if the heating is carried out for a longer time, then the yield obtained will decrease. This is because the oxalic acid formed from durian powder will further oxidize HNO<sub>3</sub>, so the results obtained will decrease (Atikah, 2017).

#### 3.2.2. Purity Analysis

The purity of oxalic acid was analyzed by comparing the infrared spectrum of standard oxalic acid with the results of synthetic oxalic acid using FTIR (Fourier-transform infrared spectroscopy). FTIR analysis aims to identify functional groups from the chemical structure in a compound at specific wavelengths. The infrared spectrum of standard oxalic acid and oxalic acid synthesized in this research is as follows:





Figure 7 Infrared Spectrum of Oxalic Acid Synthesized from Durian Peel

In the FTIR test, there is a typical absorption based on an analysis of the wavelength of the characteristic peaks of a sample. The wavelength of these peaks indicates the presence of specific functional groups in the sample because each functional group has characteristic peaks specific to certain functional groups. The following are some typical absorptions of several groups, namely as follows:

No.	Functional Group	Type of Compound	Absorption Area
1.	O-H	Alcohol (phenol)	200-3600 cm <sup>-1</sup>
2.	C=C	Alkenes	1640-1680 cm <sup>-1</sup>
3.	C-0	Alcohols, Ethers, Carboxylic acids	1080-1300 cm <sup>-1</sup>
4.	C-H	Alkenes	675-1000 cm <sup>-1</sup>
5.	C-H	Aromatic	675-870 cm <sup>-1</sup>

Table 2 Typical absorption of some compounds

The oxalic acid whose purity analysis was tested was the oxalic acid produced in the experiment with the variable 5M 80 minutes. Figure 7 shows that the stretching vibration of the hydroxyl group (O-H) of standard oxalic acid is at a wave number of 3200-3700 cm<sup>-1</sup>. The hydroxyl group is characterized by strong and sharp absorption at 3422.06 cm<sup>-1</sup>. Meanwhile, based on Figure 4.5, it can be seen that the oxalic acid synthesized from durian skin has a stretching vibration of the hydroxyl group at a wave number of 3400.91 cm<sup>-1</sup>. The stretching vibration of the C=C group of standard oxalic acid is at a wave number of 1685.48 cm<sup>-1</sup>, while synthetic oxalic acid is at a wave number of 1681.18 cm<sup>-1</sup>. The stretching vibration of the C-O group of standard oxalic acid is at a wave number of 1123.33 cm<sup>-1</sup>, while for synthetic oxalic acid is at a wave number of 718.35 cm<sup>-1</sup>. The stretching vibration of the C-H group of standard oxalic acid is at a wave number of 5400.90 cm<sup>-1</sup>.

No	Functional Group	Standard Oxalic Acid	Synthesized Oxalic Acid from Durian Peel
1.	O-H	3422,06 cm <sup>-1</sup>	3400,91 cm <sup>-1</sup>
2.	C=C	1685,48 cm <sup>-1</sup>	1681,18 cm <sup>-1</sup>
3.	C-0	1123,33 cm <sup>-1</sup>	1099,97 cm <sup>-1</sup>
4.	C-H	718,35 cm <sup>-1</sup>	665,90 cm <sup>-1</sup>

Table 3 Comparison of the infrared spectrum of standard oxalic acid with oxalic acid synthesized from palm oil fronds

Based on Table 3, a comparison of the stretching vibration results between standard oxalic acid and the vibration results of oxalic acid synthesized from durian peel has no different peaks. This proves that in this research, the compound produced was oxalic acid. Other peaks found in the FTIR analysis of synthetic oxalic acid indicate that the oxalic acid obtained is still not pure because there are still impurities in the crystals. The way to find out the impurities contained in oxalic acid crystals is by the recrystallization stage. Recrystallization is a technique for purifying a solid substance from its mixture or impurities, which is carried out by re-crystallizing the substance after dissolving it in a suitable solvent. Impurities that may be present in oxalic acid crystals are cellulose that has yet to be wholly reacted, calcium oxalate precipitates, and calcium sulfate precipitates produced in previous reactions. The basic principle of recrystallization is the difference in solubility between the purified substance and the mixing agent or impurity solubility.

The advantage of using this instrument is that it can test samples in liquid, solution, paste, powder, or gas. The applications of infrared spectroscopy are extensive for quantitative and qualitative analysis. The most important use is for identifying organic compounds because their spectrum is very complex, consisting of many peaks. The advantages of using FTIR are that it is accurate, safe, fast, and sensitive. FTIR can specifically recognize functional groups in a component based on its working principle. Each functional group can be recorded at a specific wavelength. With the FTIR method, each group of components will be detected at different wavelengths and absorbance values.

## 3.2.3. Melting point analysis

Melting point analysis is carried out to determine purity and identify a solid material. The oxalic acid crystals tested for melting point analysis in this study were produced in experiments with a variable concentration of 5M Nitric Acid and a reaction time of 80 minutes. The results were that oxalic acid crystals from durian skin waste had a melting point of 98.6°C. Pure oxalic acid has a melting point of 101.5 °C. From the results of this analysis, the synthesized oxalic acid has the same characteristics as pure oxalic acid, so it can be concluded that the product produced from this research is oxalic acid and is classified as dihydrate oxalic acid.

### 4. Conclusion

In durian skin waste, the water content using the drying method was found to be 36%, and then for the cellulose content using the Chesson method, it was found to be 63%. The concentration of the oxidizing agent  $HNO_3$  in the carbohydrate oxidation process using durian peel as raw material influences the oxalic acid yield obtained. This research shows that the optimum conditions are 80 minutes with a ratio of 1:10 with 4M HNO<sub>3</sub> content, resulting in a

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yield of 4.33%. The properties of the oxalic acid produced by the properties of standard oxalic acid crystals are based on the results of stretching vibrations in FTIR testing.

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