Journal of Applied Chemistry and Biomedical Laboratory Research
https://doi.org/00.0000/0000-000-000
eISSN 3125-1404

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Synthesis of High-Value Oxalic Acid from Sugarcane Bagasse Waste by Alkaline Melting and Nitric Acid Oxidation

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Received: May 6, 2026
Accepted: June 29, 2026
Published online: June 30, 2026

ABSTRACT

This study aimed to synthesize oxalic acid from sugarcane bagasse using two methods: alkali fusion and direct oxidation with concentrated nitric acid, and to compare the results from both methods. Sugarcane bagasse was selected as the raw material due to its high cellulose content (26-43%), making it a potential alternative source for oxalic acid production. The alkali fusion method utilized 4N sodium hydroxide as the solvent and 4N sulfuric acid for acidification, while the direct oxidation method employed concentrated nitric acid. The results indicated that the alkali fusion method with sulfuric acid produced oxalic acid yields of 1.93% and 1.94%, whereas the alkali fusion method with nitric acid did not yield solid oxalic acid as it decomposed into other compounds, resulting in a water-soluble form of oxalic acid. In contrast, the direct oxidation method with concentrated nitric acid yielded the highest results, at 2.16% and 2.15%. Product characterization using melting point tests, thin-layer chromatography (TLC), and FT-IR confirmed that the synthesized oxalic acid had purity levels approaching the standard, although some variations were observed due to impurities. These findings demonstrate that the direct oxidation method with concentrated nitric acid is more effective and efficient for producing oxalic acid from sugarcane bagasse compared to the alkali fusion method.

Keywords: Sugarcane bagasse, Oxalic acid, Alkali fusion, HNO_3 oxidation, Yield, Characterization

Citation:
Yuliani E, Rahaju D, Salam D, Kurnia D. Synthesis of High-Value Oxalic Acid from Sugarcane Bagasse Waste by Alkaline Melting and Nitric Acid Oxidation. J Appl Chem Biomed Lab Res (JACBioLab). 2026;2(1):1-6. https://doi

INTRODUCTION

Sugarcane (*Saccharum officinarum*) is a plantation commodity that plays a role in improving the Indonesian economy. Sugarcane can grow in lowlands such as tropical regions and also in some subtropical areas. According to the Ministry of Agriculture, the current sugarcane plantation area reaches 420 thousand hectares spread across various regions in Indonesia [1]. Sugarcane has a primary benefit as a raw material for making granulated sugar. Based on data from the Central Statistics Agency (BPS), national sugar consumption currently reaches 5.1 million tons and continues to increase annually [2]. Sugar production in factories produces waste in the form of bagasse. Each year, the bagasse produced from sugarcane milling is estimated to reach 10.5 million tons throughout Indonesia. Approximately 0.3 million tons is dumped around the factory grounds. Bagasse contains cellulose, lignin, pentosan, and hemicellulose compounds. The cellulose content in bagasse ranges from 26-43%. Cellulose is a long-chain carbon compound that can be broken down into simpler carbon compounds. Cellulose has two reactive groups: a hydroxyl group and a reducing group [3, 4].

Oxalic acid is a chemical compound with the molecular formula $H_2C_2O_4$ and has the systematic name ethadionic acid. Oxalic acid is one of the simplest dicarboxylic acids and can be described by the formula HOOC-COOH, where each C atom is bound to one hydroxyl group. Oxalic acid is a relatively strong organic acid, 10,000 times stronger than acetic acid. This acid has a rhombic pyramidal crystal shape, is colorless or transparent, odorless, and hygroscopic [5]. Oxalic acid has many functions in the industrial world, including use as metal treatment, oxalate coating, anodizing, metal cleaning, and textiles. Oxalic acid is also found in foods such as chocolate, coffee, strawberries, nuts, spinach, and so on [1, 4].

Research on the manufacture of oxalic acid has been conducted by various researchers with various materials and methods [6]. Based on research conducted by on the effect of time and temperature on the manufacture of oxalic acid from HVS waste using the alkali melting method, the optimum temperature was obtained at 105°C and the optimum time at 70 minutes with a weight of oxalic acid obtained of 1.8043 grams and a yield of oxalic acid after the permanganometric titration test of 6.8537% [7]. Magfiroh (2025) [5] conducted research on the manufacture of oxalic acid from oil palm fronds using the alkali melting method, the optimum temperature was obtained at 90°C and the optimum time was obtained at 60 minutes with a yield of 59.6%. Yoanda *et.al* (2022) [3] conducted research on the effect of melting time on the extraction of oxalic acid manufacture, the highest yield of oxalic acid was obtained at 0.4086 at a melting time of 70 minutes. Nurjanah *et.al* (2026) [8] conducted a study on the manufacture of oxalic acid from cogongrass using an alkaline melting method at a cooking temperature of 98°C, obtaining an oxalic acid content of 44.39% with an optimum sodium hydroxide concentration of 4N and a cooking time of 60 minutes [9, 10 ,11]. Based on the explanation above, an innovation will be made in the manufacture of oxalic acid using an alkaline melting process with bagasse as the basic material. This is done based on the consideration that the alkaline melting process is a process that can produce oxalic acid with a high content and the cellulose content of bagasse is quite high, so it can be used as another alternative in the manufacture of oxalic acid.

MATERIALS AND METHODS

Raw Material Preparation (Pre-treatment)

The pre-treatment process involves preparing the bagasse according to the desired requirements. Cut the bagasse into pieces using a knife or similar tool to facilitate the boiling and grinding process. Boil the bagasse in water for 5 minutes to remove impurities. The bagasse is dried in an oven at 105°C for 30 minutes to reduce the water content. Grind the bagasse as finely as possible using an evaporator to facilitate the melting process.

Melting Process

Solvent: 4N sodium hydroxide and acidification: 4N sulfuric acid. Weigh 50 grams of bagasse into a three-necked flask containing 500 ml of 4N sodium hydroxide solution. The mixture is heated for 60 minutes at 90–98°C. After the melting process, the mixture is cooled to room temperature to facilitate the filtration process. The mixture was filtered to obtain a precipitate and filtrate.

Precipitation and Acidification Process

The filtrate obtained was added with a 10% calcium chloride solution until a calcium oxalate precipitate formed. Then, 100 ml of 4N sulfuric acid was added and allowed to stand for 24 hours until a calcium sulfate precipitate formed. The precipitate was then filtered and washed with 15 ml of 96% ethanol until a filtrate was formed. The filtrate was heated at 70°C for approximately 1 hour. The filtrate was allowed to stand for 24 hours in a container filled with ice cubes until an oxalate precipitate formed.

Melting Process

Solvent: 4N sodium hydroxide and acidification: 4N nitric acid. Weigh 50 grams of bagasse into a three-necked flask containing 500 ml of 4N sodium hydroxide solution. The mixture was heated for 60 minutes at 90–98°C. After the melting process, the mixture was cooled to room temperature to facilitate the filtration process. The mixture was filtered to obtain a precipitate and filtrate.

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Direct Oxidation of Concentrated Nitric Acid: Oxidation Process

Weigh 50 grams of bagasse and place it in a three-necked flask containing 500 ml of concentrated nitric acid solution. The mixture is digested until brown vapors appear. The reaction is continued by heating for 15 minutes. Pour the resulting reaction into a 100 ml beaker and rinse with 10 ml of distilled water.

Acidification Process

The resulting precipitate is then added with 10 ml of concentrated nitric acid. Evaporate with water at 70°C until 20 ml remains. Add 20 ml of distilled water and evaporate again until 20 ml remains. Cool with ice water while stirring until crystals form. The crystals are filtered and recrystallized with hot water, then cooled. Filter and dry for further analysis.

Analysis of Oxalic Acid Dihydrate Content

Oxalic acid dihydrate powder was weighed to determine the yield. Several grams were taken for melting point and TLC testing, and 0.5 grams were taken for FT-IR readings.
Yield Calculation:

$$yield = \frac{mass\ of\ oxalic\ acid}{initial\ mass} \times 100\%$$

RESULTS AND DISCUSSION

The following are the results of the synthesis of oxalic acid from sugarcane bagasse, in the form of a yield table, characterization of oxalic acid based on melting point, thin layer chromatography, and with an FT-IR instrument to determine the oxalic acid produced.

Table 1. Yield of Oxalic Acid

Reagents and Replications		Initial Mass (gram)	Oxalic acid mass (gram)	Yields (%)
A	1	50,031	0,9656	1,93
	2	50,025	0,972	1,94
B	1	50,0101	-	-
	2	50,02	-	-
C	1	50,201	1,0863	2,16
	2	50,019	1,0801	2,15

Note: A=NaOH + H2SO4 B=NaOH + HNO3 C=Oxidation of HNO3

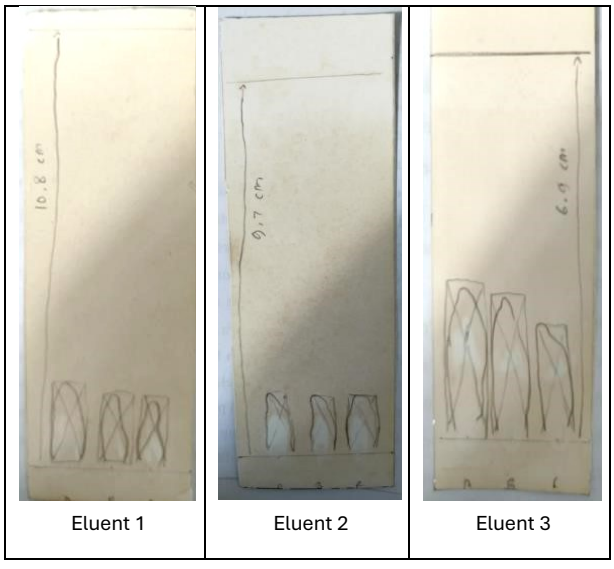

Figure 1. TLC Shows the RF Values of Each Oxalic Acid Dihydrate

Table 2. TLC Determination Data of Oxalic Acid Dihydrate

Sample	Eluent	Distance of Eluent (cm)	Distance of Sample (cm)	RF Value
Oxalic acid (Blank)	Eluent 1	10,8	1,3	0,12
	Eluent 2	9,7	1,1	0,11
	Eluent 3	6,9	1,95	0,28
Alkaline Smelting	Eluent 1	10,8	1,2	0,11
	Eluent 2	9,7	1,1	0,11
	Eluent 3	6,9	1,6	0,16

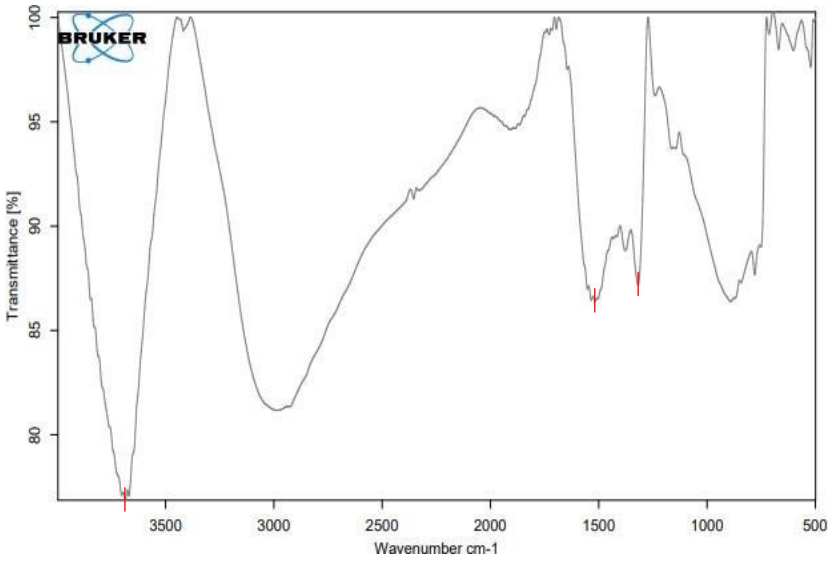

Figure 2. FT-IR Results from Alkaline Smelting of Bagasse

Table 4. FT-IR Results from Nitric Acid Oxidation of Bagasse

Group	Compound	Absorbance Area
O-H	Hydroxyl	3200-3600cm^{-1} ↑
C=O	Carbonyl from Carboxylic Acid	1600-1750 cm^{-1}
C-O	Carboxyl	1000-1300cm^{-1}

The results of oxalic acid from this study can be seen in Table 1. This shows that in the results of alkali melting with heating at a temperature range between 90 – 98°C and a melting time of 60 minutes with an acidification process using 4 N sulfuric acid, oxalic acid was produced at 1.93% and 1.94%. In the direct oxidation process using concentrated nitric acid, oxalic acid yields were obtained at 2.16% and 2.15%. The oxalic acid values obtained from the two procedures indicate that the best method or the oxalic acid yields from this study can be seen in Table 1. This shows that the alkali smelting process, heated at a temperature between 90 and 98°C and a melting time of 60 minutes with an acidification process using 4 N sulfuric acid, produced oxalic acid of 1.93% and 1.94%. In the direct oxidation process using concentrated nitric acid, the oxalic acid yields were 2.16% and 2.15%. The oxalic acid values obtained from these two procedures indicate that the best method or efficient work and the best results are using the oxidation process using concentrated nitric acid in the synthesis of oxalic acid, one of which is from the bagasse sample as in this study.

The melting point test results on the samples were used to determine the purity of the results obtained by comparing them with the control (oxalic acid p.a.) and the melting point literature for pure oxalic acid. The melting point of pure oxalic acid is 101°C, while the melting points of the samples slightly different. For alkaline smelting process the mean of melting point of oxalic acid is 104.8°C and for nitric acid oxidation it is 102.3°C. There is a slight difference in the melting point temperatures of the standard and the samples due to the presence of impurities in the samples obtained and the amount of sample in the capillary tube.

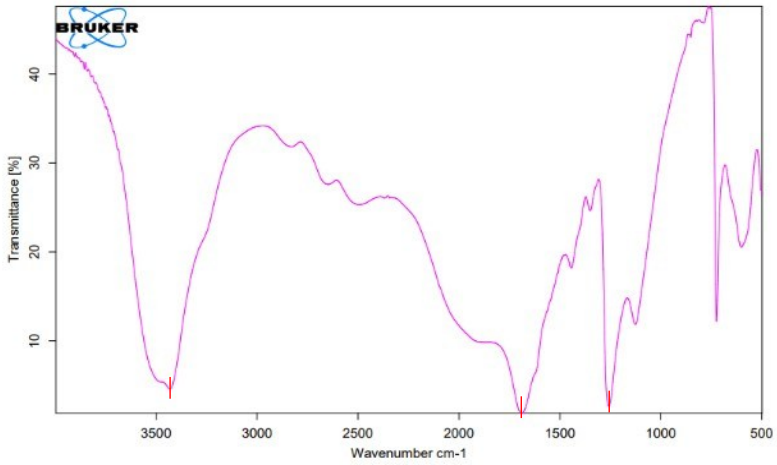

Figure 3. FT-IR Results from Nitric Acid Oxidation of Bagasse

In thin-layer chromatography testing to determine the RF value of oxalic acid, the presence of oxalic acid on the TLC plate cannot be seen directly with the naked eye because this compound is colorless. Therefore, a special detection agent is required to make it visible, a process called UV light visualization. Oxalic acid does not fluoresce under UV light. However, if the TLC plate used contains a fluorescence indicator (such as silica gel GF254), the non-fluorescent compound will appear as a dark spot that absorbs UV light against a bright green background. The results are indicated by a dark spot under UV light at a wavelength of 254 nm. Potassium permanganate. A purple $KMnO_4$ solution can be used to spray plates. Oxalic acid is reduced by $KMnO_4$ to produce yellow spots on a purple background.

In this study, three different solvents (eluents) were used, as follows: Eluent 1 (ethyl acetate: methanol: acetic acid) in a ratio of (7:2.5:1). This solvent system obtained an RF value of 0.12 for the oxalic acid sample (blank), and an RF value of 0.11 for the oxalic acid sample from the melting and oxidation process. Eluent 2 (n-butanol: acetic acid) in a ratio of (8:2). This eluent obtained an RF value of 0.11 for the oxalic acid sample (blank) and the melting process, and an RF value of 0.1 for the oxidized process. Eluent 3 (n-butanol: acetic acid: distilled water) in a ratio of (4:1:5). In this eluent, the RF value for the oxalic acid sample (blank) was 0.28, the melting product was 0.16, and the oxidation product was 0.2.

For Fourier Transform Infrared (FT-IR) test: The sample consisted of powdered oxalic acid obtained from the alkali (NaOH) melting method, and the oxidation product was concentrated nitric acid. The presence of oxalic acid compounds was identified using Fourier Transform Infrared (FTIR). The KBr disc method was chosen because the sample was in a solid powder form and easy to perform. The oxalic acid sample was added to KBr at a ratio of 1:10. FTIR readings using a wavelength of 400-4000 cm-1 on the oxalic acid dihydrate sample as a standard revealed compounds or groups detected by the instrument. The FT-IR results are shown in Table 4. The O-H group (hydroxyl from carboxyl) has an absorption range of approximately 2500-3500 cm-1. The C=O group (carbonyl of carboxylic acid) has an absorption range of around 1650–1750 cm^{-1} and C–O (1200–1300 cm^{-1}). These results are consistent with the chemical structure of oxalic acid, which contains two carboxyl groups. In the dihydric oxalic acid sample, the results of alkali melting and 4N sulfuric acid acidification yielded compounds or groups that were read by the FT-IR instrument. The results can be seen in Table 5. O–H Group (Hydroxyl Group) Absorption Range 3200–3600 cm^{-1}. C=O Group (Carbonyl Group of Carboxylic Acid) Absorption Range 1600–1750 cm^{-1}. C–O Group (from Carboxyl Group) Absorption Range 1200–1400 cm^{-1}.

In the dihydroxyl oxalic acid sample, the direct oxidation of concentrated nitric acid yielded compounds or groups that were detected by the FT-IR instrument.

CONCLUSIONS

Sugarcane bagasse contains relatively high levels of cellulose. The cellulose in bagasse can be processed into oxalic acid using carbohydrate oxidation methods, including alkali smelting and direct oxidation with concentrated nitric acid. The direct oxidation method with concentrated nitric acid can produce a yield of 2.15–2.16%.

Author Contributions: E.Y was in experimental sections, D.S was in determining the yields and TLC processing, processing the results of melting points, D.K was in FT-IR handling, D.R. was in writing reports and exploring literatures.

Funding: "This research received no external funding"
Conflicts of Interest: There are no conflict of interests to be declared.

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