Sintesis Asam Oksalat Dari Limbah Kulit Rambutan Dengan Metode Peleburan Alkali

Synthesis of Oxalic Acid from Rambutan Peel Waste Using the Alkaline Fusion Method

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ABSTRAK

Limbah kulit rambutan berpotensi sebagai bahan baku asam oksalat karena mengandung selulosa tinggi dan tersedia melimpah di Indonesia. Penelitian ini bertujuan mensintesis asam oksalat dari limbah kulit rambutan menggunakan metode peleburan alkali serta mengevaluasi pengaruh konsentrasi natrium hidroksida (NaOH) dan waktu reaksi terhadap rendemen. Kulit rambutan varietas binjai dicuci, dikeringkan, dihaluskan, dan diayak hingga ukuran 70 mesh. Sintesis dilakukan pada suhu 180°C menggunakan variasi NaOH 3M, 4M, 5M, dan 6M serta waktu reaksi 60, 75, 90, 105, dan 200 menit. Hasil menunjukkan kondisi optimum pada NaOH 6M selama 105 menit dengan rendemen tertinggi 21,5%. Perpanjangan waktu hingga 200 menit menurunkan rendemen menjadi 17,8%, yang diduga disebabkan oleh degradasi lanjutan dan over-oxidation. Kristal produk menunjukkan titik leleh 102°C. Karakterisasi Fourier Transform Infrared (FTIR) mengonfirmasi pembentukan asam oksalat melalui puncak O-H, C=O, dan C-O yang sesuai dengan standar. Hasil ini menunjukkan bahwa limbah kulit rambutan dapat dikonversi menjadi asam oksalat dengan rendemen yang kompetitif.

Kata kunci: asam oksalat, kulit rambutan, peleburan alkali, hidrolisis, biomassa

ABSTRACT

Rambutan peel waste is a promising raw material for oxalic acid because of its high cellulose content and abundant availability in Indonesia. This study synthesized oxalic acid from rambutan peel waste using the alkaline fusion method and evaluated the effects of sodium hydroxide (NaOH) concentration and reaction time on yield. Binjai rambutan peel was washed, dried, milled, and sieved to 70 mesh. The synthesis was carried out at 180°C with NaOH concentrations of 3M, 4M, 5M, and 6M and reaction times of 60, 75, 90, 105, and 200 min. The optimum condition was obtained at 6 M NaOH for 105 min, producing the highest yield of 21.5%. Extending the reaction time to 200 min decreased the yield to 17.8%, indicating further degradation and over-oxidation. The product crystals showed a melting point of 102°C, and FTIR analysis confirmed oxalic acid formation.

Keywords: *oxalic acid, rambutan peel, alkaline fusion, hydrolysis, biomass*

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INTRODUCTION

Oxalic acid ($C_2H_2O_4$) is a simple dicarboxylic acid with important industrial applications, including dyes, coatings, metal cleaning, textile mordants, laboratory analysis, electroplating, waxes, inks, photographic chemicals, and pharmaceutical intermediates (Febriaty et al., 2016; Puspita, 2015; Zulnazri et al., 2025). The domestic demand for oxalic acid in Indonesia is still largely met by imports, which creates an opportunity to develop local production using renewable and abundant biomass resources (Badan Pusat Statistik, 2020).

Indonesia produces large amounts of agricultural residues that can be converted into value-added chemicals. Rambutan (Nephelium lappaceum L.) is widely cultivated in tropical regions, and its peel is commonly discarded after fruit consumption or processing. This waste may cause environmental problems when left to decompose, yet it contains lignocellulosic components that can serve as substrates for chemical conversion.

Rambutan peel contains approximately 35-45% cellulose, 20-25% hemicellulose, 15-20% lignin, and 17.1% extractives (Oliveira et al., 2016). Its relatively high cellulose content and moderate lignin fraction make it a promising feedstock for alkaline hydrolysis. Under strong alkaline conditions and elevated temperature, cellulose can be depolymerized and oxidized into oxalic acid, while lignin-carbohydrate linkages are partly disrupted, increasing substrate accessibility (Fengel & Wegener, 1984; Kirk & Othmer, 1983).

Previous studies have reported oxalic acid synthesis from teak wood sawdust, palm fiber, cardboard waste, and mixed rice husk-coconut fiber using alkaline treatment or related routes (Desinta et al., 2020; Mufid et al., 2018; Utami et al., 2018; Wulandari et al., 2021). However, the utilization of rambutan peel waste as a raw material for oxalic acid synthesis by alkaline fusion remains insufficiently explored. Therefore, this study aims to synthesize oxalic acid from rambutan peel waste, evaluate the effects of NaOH concentration and reaction time on yield, and determine the optimum operating condition for the highest product yield.

MATERIALS AND METHODS

Materials

The materials used in this study were Binjai variety rambutan peel waste obtained from Sambas Regency, sodium hydroxide (NaOH) p.a., sulfuric acid (H_2SO_4) p.a., calcium chloride ($CaCl_2$), ethanol 96%, distilled water, and Whatman No. 1 filter paper.

Apparatus

The apparatus consisted of a 500 mL three-neck flask, reflux condenser, oil bath with magnetic hotplate stirrer, 200°C thermometer, distillation set, blender, 70 mesh sieve, Fourier Transform Infrared (FTIR) spectrophotometer, melting point apparatus (Stuart SMP10), and standard laboratory glassware.

Sample Preparation

Rambutan peels were washed with running water, sliced into 2-3 mm pieces, and air-dried at room temperature (28-30°C) for seven days until the moisture content reached 8-10%. Moisture content was determined gravimetrically at 105°C. The dried peels were milled using a blender and sieved to 70 mesh. The resulting powder was stored in an airtight glass container before use.

Oxalic Acid Synthesis

A 15 g portion of rambutan peel powder on an air-dried basis was placed in a 500 mL three-neck flask equipped with a reflux condenser and thermometer. A 250 mL NaOH solution at concentrations of 3M, 4M, 5M, and 6M was added. The mixture was heated at 180°C in an oil bath with constant stirring. Reaction times were varied at 60, 75, 90, 105, and 200 min. The reflux system minimized solvent loss during alkaline fusion.

After the reaction, the mixture was cooled gradually to 60°C and then cooled in an ice bath to room temperature. Hot water (80-90°C, approximately 150 mL) was added to dissolve the soluble salts formed during

alkaline treatment. The mixture was filtered through Whatman No. 1 filter paper.

The filtrate was treated dropwise with 10% $CaCl_2$ solution (approximately 50 mL or until no further precipitate formed) to precipitate oxalate ions as calcium oxalate. The white precipitate was filtered and washed with distilled water until the washing filtrate reached pH 6-7. The calcium oxalate precipitate was then reacted with 200 mL of 2M H_2SO_4 for 30 min at room temperature to liberate oxalic acid and precipitate $CaSO_4$. The filtrate was concentrated at 70°C to one-third of its initial volume and cooled slowly to form white needle-like oxalic acid crystals. The crystals were purified by recrystallization using 96% ethanol.

Melting Point Test

A small amount of recrystallized oxalic acid was placed in a capillary tube and analyzed using a Stuart SMP10 melting point apparatus. The heating rate was maintained at 2°C/min from 90°C until the crystals melted completely. Measurements were performed in triplicate, and the average value was compared with the reference melting point of oxalic acid dihydrate.

Product Characterization

The synthesized oxalic acid was characterized using FTIR spectroscopy in the range of 400-4000 cm^{-1} to identify functional groups and confirm product formation. The yield was calculated using the following equation:

$$Yield = \frac{\text{mass of oxalic acid crystals}}{\text{mass of dry rambutan peel powder}} x\ 100\% \quad (1)$$

RESULTS AND DISCUSSION

Preparation of Rambutan Peel Powder

The rambutan peel sample was obtained from Tamang Sagang Village, Sambas Regency, West Kalimantan. Washing removed adhering soil and impurities that could interfere with the reaction. Air drying for seven days produced brittle, yellowish-brown peels with a moisture content of approximately 8-10%. Milling and sieving to 70 mesh increased the surface area of the biomass, thereby improving contact between the lignocellulosic substrate and the alkaline solution.

Based on the lignocellulosic composition reported by Oliveira et al. (2016), rambutan peel contains a substantial cellulose fraction. This characteristic supports its use as a feedstock for oxalic acid synthesis, because cellulose is the main carbohydrate source that undergoes hydrolysis and subsequent oxidative cleavage. Direct compositional analysis of the sample used in this study was not performed; therefore, the literature composition was used as a theoretical basis and is acknowledged as a limitation.

Alkaline Fusion Mechanism

Oxalic acid formation from rambutan peel involves cellulose hydrolysis, depolymerization or chain scission, oxidation of carbohydrate fragments, precipitation of oxalate, and acid liberation. In the alkaline fusion step, NaOH acts as a hydrolyzing medium and promotes the breakdown of glycosidic bonds in cellulose. Strong alkali at high temperature can also disrupt ester and ether linkages in lignin, increasing cellulose accessibility (Fengel & Wegener, 1984; Kirk & Othmer, 1983).

$$(C_6H_{10}O_5)n + nH_2O \rightarrow nC_6H_{12}O_6 \quad (2)$$

$$C_6H_{12}O_6 + 2NaOH \rightarrow 3C_2H_2O_4 + H_2O \quad (3)$$

$$H_2C_2O_4 \rightarrow CO_2 + H_2O \quad (4)$$

$$H_2C_2O_4 + CaCl_2 \rightarrow CaC_2O_4\downarrow + 2HCl \quad (5)$$

$$CaC_2O_4 + H_2SO_4 \rightarrow H_2C_2O_4 + CaSO_4\downarrow \quad (6)$$

The glucose formed during hydrolysis may undergo retro-aldol fragmentation and oxidation under strongly alkaline conditions, producing two-carbon fragments that are further oxidized to oxalic acid (Febriaty et al., 2016; Harmaen et al., 2016). At excessive reaction time, oxalic acid may degrade further through decarboxylation or over-oxidation, causing a reduction in product yield. The addition of $CaCl_2$ selectively precipitated oxalate as calcium oxalate, while acidification with H_2SO_4 released oxalic acid and precipitated $CaSO_4$.

Effect of NaOH Concentration and Reaction Time

The yield of oxalic acid was affected by NaOH concentration and reaction time, as shown in Figure 1. Increasing NaOH

concentration from 3M to 6M improved conversion because a higher concentration of OH^- ions enhanced cellulose hydrolysis and promoted the oxidation of carbohydrate fragments. The highest yield was obtained at 6M NaOH and 105 min reaction time.

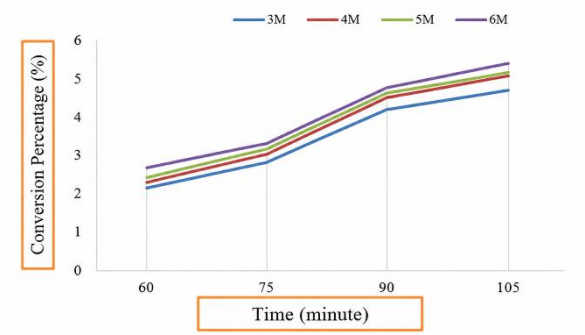

Figure 1. Effect of NaOH concentration and reaction time on oxalic acid yield

The optimum condition produced 21.5% oxalic acid yield. Increasing the reaction time from 60 to 105 min increased yield, indicating that longer contact allowed more complete degradation of cellulose into oxalate-forming fragments. However, extending the reaction to 200 min reduced yield to 17.8%. This decrease was attributed to further degradation and over-oxidation of oxalic acid into smaller products such as formic acid, CO_2, and H_2O, as also reported for alkaline processing of cardboard waste (Desinta et al., 2020).

The yield obtained in this study was higher than those reported for teak wood sawdust (18.5%), palm fiber (16.3%), and mixed rice husk-coconut fiber (19.7%) (Mufid et al., 2018; Utami et al., 2018; Wulandari et al., 2021). This result indicates that rambutan peel is a competitive lignocellulosic feedstock for oxalic acid production, presumably due to its relatively high cellulose content and moderate lignin fraction.

Melting Point of Synthesized Oxalic Acid

The synthesized crystals showed a melting point of 102°C, which is close to the reference value for oxalic acid dihydrate, approximately 101-102°C (Merck & Co., 2006). The correspondence between the observed and reference melting points indicates that the product had good preliminary purity. Significant impurities would normally cause a lower and broader melting range.

FTIR Characterization

FTIR analysis was performed to confirm the functional groups of the synthesized product. The comparison between the FTIR spectra of standard oxalic acid and synthesized oxalic acid is presented in Figure 2.

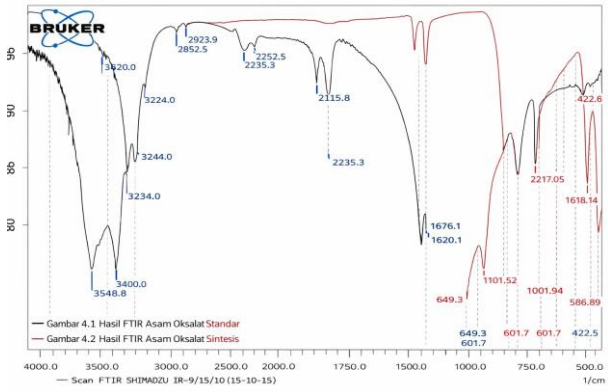

Figure 2. FTIR spectra comparison of standard oxalic acid and synthesized oxalic acid

The FTIR spectra showed a high similarity between the standard and synthesized samples. The broad O-H absorption of the synthesized product appeared at 3489.2 cm^{-1}, indicating hydrogen bonding typical of oxalic acid hydrate. The C=O stretching vibration appeared at 1680.8 cm^{-1}, while the C-O stretching vibration appeared at 1101.5 cm^{-1}. The fingerprint region showed peaks at 649.3 and 601.7 cm^{-1}, consistent with the standard spectrum.

Table 1. FTIR spectral data of standard and synthesized oxalic acid

Functional group	Standard (cm^{-1})	Synthesized (cm^{-1})
O-H stretching (broad)	3404.1	3489.2
C=O stretching	1687.6	1680.8
C-O stretching	1141.8	1101.5
O-H deformation	1298.4	1301.7
Fingerprint	649.3 / 601.7	649.3 / 601.7

The small shift in the C=O absorption from 1687.6 to 1680.8 cm^{-1} is within the

normal tolerance for FTIR analysis and may be associated with differences in hydrogen bonding and hydration state. The absence of significant additional peaks in the fingerprint region indicates that no major organic impurities remained in the product. Overall, the FTIR profile supports the successful synthesis of oxalic acid from rambutan peel waste.

CONCLUSION

Oxalic acid was successfully synthesized from rambutan peel waste using the alkaline fusion method. The optimum condition was obtained at 6M NaOH and 105 min reaction time at 180°C, yielding 21.5% oxalic acid. Extending the reaction time to 200 min reduced the yield to 17.8%, indicating further degradation and over-oxidation of the product. The synthesized crystals had a melting point of 102°C, which is consistent with oxalic acid dihydrate. FTIR analysis confirmed the formation of oxalic acid through characteristic O-H, C=O, C-O, and fingerprint absorptions. These results demonstrate that rambutan peel waste is a promising local biomass resource for producing oxalic acid and may contribute to organic waste valorization and import substitution.

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