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Enhanced Oxalic Acid Production from Peanut Shell Waste via Nitric Acid Oxidation Using Coal Ash Silica Catalyst

Erin Dimyati, Elfira Rosa Pane*, M. Mahfudz Fauzi Syamsuri
Department of Chemistry, Universitas Islam Negeri Raden Fatah Palembang Sumatera Selatan, 30257., Indonesia.

Corresponding author*
elfirarosapane_uin@radenfatah.ac.id

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Abstract

The increasing industrial demand for oxalic acid has encouraged the search for more sustainable and environmentally friendly production methods. In this study, peanut shell waste (*Arachis hypogaea* L.) was explored as a renewable raw material for oxalic acid production through nitric acid oxidation, with particular attention given to the catalytic role of coal ash-derived materials. The oxidation reaction was carried out using a sulfuric acid–nitric acid mixture under controlled heating conditions. To evaluate catalytic performance, three reaction systems were compared: a non-catalytic system, a system using coal ash as the catalyst, and another employing silica extracted from coal ash. The resulting products were characterized by Fourier Transform Infrared Spectroscopy (FTIR) and X-ray Diffraction (XRD). The results showed that the addition of catalysts enhanced oxalic acid production, with the silica catalyst providing the highest yield of 49.90%. FTIR analysis confirmed the characteristic functional groups of oxalic acid, while XRD results indicated a crystallinity of 39.59%. Overall, the findings demonstrate that silica recovered from coal ash is a promising catalyst for improving the conversion of lignocellulosic biomass into oxalic acid, while also offering a value-added application for an industrial waste material.

Keywords: Peanut peel; catalysts; coal ash; oxidation; nitric acid.

INTRODUCTION

Oxalic acid is an important dicarboxylic acid widely applied in textile processing, metal cleaning, pharmaceuticals, and analytical chemistry. The increasing industrial demand has encouraged the development of alternative and sustainable production pathways (Dube et al., 1982; Yoanda et al., 2022). Conventional production methods commonly utilize carbohydrate-based raw materials such as sucrose, which may compete with food resources and raise sustainability concerns (Utami et al., 2018). Lignocellulosic biomass has emerged as a promising alternative due to its abundance, renewability, and high cellulose content. Agricultural residues such as peanut shells are particularly attractive because they contain significant cellulose fractions and are produced in large quantities but remain underutilized (Sulistyani et al., 2018; Fitriani, 2010). Improper disposal practices, including open burning, further highlight the need for valorization strategies.

Several studies have demonstrated that cellulose-rich biomass can be converted into oxalic acid through alkaline fusion or oxidative processes, although the conversion efficiency varies considerably depending on the reaction conditions employed (Iriany et al., 2015; Wiyani et al., 2022). More recently, the use of catalysts has attracted increasing attention as an effective strategy for improving oxalic acid production. In particular, catalysts derived from industrial waste, such as coal fly ash, have shown considerable promise because they not only enhance the conversion process but also provide an environmentally and economically beneficial use for waste materials (Kuipa et al., 2021). Building on these findings, the present study investigates the production of oxalic acid from peanut shell waste via nitric acid oxidation, with emphasis on the catalytic performance of coal ash and its derived silica.

MATERIALS AND METHODS

Procedures

Materials

Sulfuric acid (H2SO4) 97% Merck brand, Nitric Acid (HNO3) 65%, aquades (H2O), HCl 37%, NaOH, peanut shell waste (Arachis hypogaea), coal fly ash and filter paper.

Tools

Mettler Toledo ME204E brand analytical balance, LG brand freezer, Memmert UN 55 brand oven, thermometer, hot plate, beaker cup, measuring cup,

volume pipette, dropper pipette, blender, stirring rod, spatula, glass mouthpiece, FT-IR and XRD instruments

Procedure

The cellulose oxidation carried out in this study follows the way Kuipa (2021) works, namely peanut shell powder is weighed as much as 2 grams, then put into a 100 ml beaker glass then added a solution of 4 ml of 98% H2SO4 and 6 ml of HNO3 65%. Then it is heated on a hotplate at a temperature of 70°C for 4 minutes, this process is stopped when the gas changes color to light yellow or pale then desired to a temperature of 0°C then filtered and put in the freezer for 30 minutes at a temperature of -15 °C, the filtrate is reheated at a temperature of 50 °C for 10 minutes then cooled again at a temperature of 0 °C and put back into the freezer for 12 hours at a temperature of -15 °C and filtered Then the crystals obtained are dried at a temperature of 65 °C for 30 minutes, the procedure is repeated using an ALB catalyst and silica as much as 0.5 grams. The final products were analyzed using FTIR spectroscopy to identify functional groups and XRD to determine crystallinity.

Data analysis

The final products were analyzed using FTIR spectroscopy to identify functional groups and XRD to determine crystallinity

RESULTS AND DISCUSSION

The conversion of cellulose into oxalic acid involves a sequence of hydrolysis and oxidation reactions. Initially, cellulose is hydrolyzed into glucose under acidic conditions. The glucose is subsequently oxidized by nitric acid to form intermediate compounds such as glucaric acid and glyoxylic acid, which are further converted into oxalic acid through oxidative cleavage (Dube et al., 1982; Utami et al., 2018).

Oxalix acid yield

The experimental results demonstrate that the presence of catalysts significantly enhances oxalic acid yield. The silica catalyst derived from coal ash exhibited the highest yield (49.90%), compared to non-catalytic conditions (31.68%). This improvement can be attributed to the role of silica as a heterogeneous catalyst that facilitates bond cleavage and enhances the interaction between oxidizing agents and substrate molecules (Kuipa et al., 2021) Table 1

Table 1. Results of oxalic acid.

Sample	*Mass (gr)*	*%*
Oxalic Acid Without Catalyst	0,6336	31,68
Oxalic acid digests ALB	0,8621	43,10
Oxalic acid with Silica Catalyst	0,9781	49,90

Similar findings have been reported in previous studies, where the use of solid catalysts improved biomass conversion efficiency by increasing reaction rates and promoting selective oxidation pathways (Wiyani et al., 2022). In addition, silica-based catalysts are known to possess high surface area and active sites, which contribute to improved catalytic performance.

FTIR Analysis

FTIR analysis confirmed the presence of characteristic functional groups of oxalic acid, including hydroxyl (O–H), carbonyl (C=O), and carboxyl groups. These findings are consistent with previous reports on oxalic acid characterization (Yoanda et al., 2022). Furthermore, XRD analysis indicated a semi-crystalline structure with a crystallinity degree of 39.59%, which falls within the typical range reported for oxalic acid synthesized from biomass (Iriany et al., 2015).

$$C_6H_{12}O_{6(s)} + 6HNO_3(l) \longrightarrow 3(COOH)_{2(s)} + 6\,H_2O_{(l)} + 6NO_{2(g)}$$

The vibrational range of functional groups in oxalic acid and oxalic acid derived from peanut shells,both without a catalyst, with coal fly ash leaching, and with silica bladder leaching, exhibited similar peak patterns. This indicates that the synthesized compounds are indeed oxalic acid. These findings are further supported by the results of X-ray Diffraction (XRD) analysis.

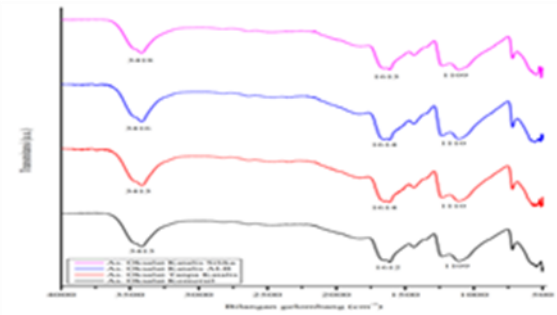


Figure 1. Results of oxalic acid analysis using FT-IR

XRD Analysis

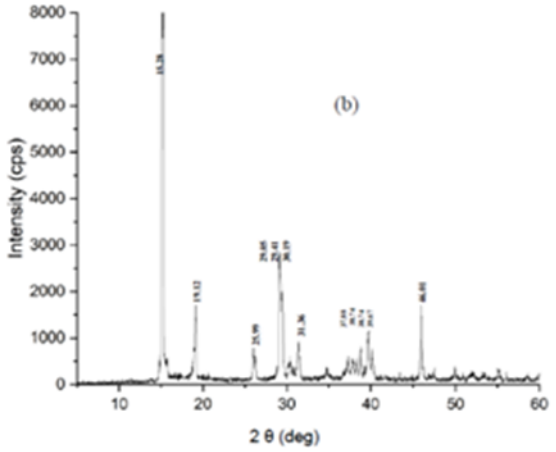


Figure 2. Results of oxalic acid analysis using XRD.

The instrument produces a degree structure of oxalic acid crystals that have the position 2θº at 15.28º, 19.12º; 26.02º; 29.16º; 30.26º; 31.36º; 34,72º; 37.32º; 37.82º; 38.36º; 39.75º; 40.24º dan 46.01º. In oxalic acid as a comparison, namely by using the results of research from C. Muthuselvi ddk, which has the position of 2θº at 14.58º; 15.10º; 18.69º; 25.82º; 28.87º; 30.98 º; 34.42º; 37.41º; 39.67º; 41.34º dan 46.82º. Based on the data mentioned above, which is then processed with the Origin application with an output value of % crystallinity of 39.59 % From the results of oxalic acid, silica is lysed by the nitric acid oxidation method. As well as the moderate quantity possessed in oxalic acid bilified silica from ALB, this is due to the range of crystallinity degrees between 10%-80% not to 100%

Discussion

The oxidation of peanut shell biomass using nitric acid successfully produced oxalic acid, as confirmed by FT-IR and XRD analyses. The FT-IR spectrum of the synthesized product showed characteristic absorption bands corresponding to oxalic acid functional groups, which were consistent with those observed in the reference oxalic acid. The presence of broad O–H stretching vibrations in the region of approximately 3200–3500 cm^{-1}, strong C=O stretching vibrations around 1680–1720 cm^{-1}, and C–O stretching vibrations in the range of 1200–1300 cm^{-1} indicates the successful formation of oxalic acid (O.Kuipa, G. K., 2021, iriany et al., 2015). The similarity between the synthesized sample and the reference material proves that the oxidation process effectively converted lignocellulosic components of peanut shells into oxalic acid.

The crystallinity of the synthesized oxalic acid was evaluated further using X-ray diffraction (XRD). The diffraction pattern showed the characteristic crystalline peaks 39.59%, indicates a moderate degree of crystallinity, suggesting that the synthesized product was not completely crystalline, and may be attributed to the presence of residual organic compounds or incomplete crystallization during the recovery process (Štejfa, et al, 2021). Factors such as crystallization temperature, cooling rate, solvent evaporation, and impurities originating from biomass degradation can significantly influence crystal formation (Štejfa, et al, 2021). Although the crystallinity is lower than that of commercially available oxalic acid, the XRD results confirm the successful synthesis of crystalline oxalic acid from peanut shell biomass.

The addition of catalysts affected the production yield of oxalic acid. Without a catalyst, the oxidation process produced only 0.6336 g of oxalic acid. The use of ALB catalyst increased the yield to 0.8621 g, representing an increase of approximately 36% compared to the non-catalytic reaction. Furthermore, the application of silica catalyst derived from coal ash enhanced the oxalic acid yield to 0.9781 g, corresponding to an increase of approximately 54% relative to the reaction without catalyst and about 13% higher than that obtained using the ALB catalyst.

The catalytic performance of silica derived from coal ash can be attributed to its physicochemical characteristics. Amorphous silica contains abundant surface silanol (Si–OH) groups together with a highly porous framework, providing numerous accessible adsorption sites for reactant molecules and facilitating catalytic interactions. These surface hydroxyl groups are recognized as the primary active sites responsible for adsorption and surface reactions on silica-based catalysts (Adam et al., 2012; Al Othman, 2012). The utilization of silica recovered from coal ash also demonstrates an important environmental advantage. Coal ash is an industrial waste generated in large quantities and commonly poses disposal and environmental challenges. Converting this waste into a value-added catalyst not only improves the efficiency of biomass conversion but also supports the principles of waste valorization and circular economy. The integration of agricultural waste (peanut shells) and industrial waste (coal ash) in this study therefore represents a sustainable approach for producing value-added chemicals.

CONCLUSIONS

The results of oxalic acid using silica catalyst from coal ash have been characterized using FT-IR with a vibration absorption band of the functional group which proves that the oxalic acid obtained is in accordance with the comparative oxalic acid, and characterized using XRD with a % value of 39.59% of oxalic acid crystals made from peanut shells by the nitric acid oxidation method. Oxalic acid was obtained from peanut shells by the nitric acid oxidation method without a catalyst with a yield of 0.6336 g, with an ALB catalyst of 0.8621 g and with a silica catalyst from ALB of 0.9781 g.

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Authors' Contributions: Author Contributions: Erin Dimyati: Investigation, Methodology, Data Collection, Validation, Writing – Original Draft; Elfira Rosa Pane: Conceptualization, Methodology, Supervision, Writing – Review & Editing; Mahfudz: Conceptualization, Data Curation, Formal Analysis, Methodology, Supervision. All authors have read and approved the final version of the manuscript.

Competing Interests: The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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