

Synthesis and characterization of heterogeneous catalysts (MgO, CaO/Zeolite-Y) via hydrothermal method for biodiesel production

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Abstract. The global energy crisis and increasing greenhouse gas emissions have driven the development of sustainable energy alternatives, among which biodiesel has gained considerable attention. However, conventional biodiesel production is often limited by homogeneous catalysts that suffer from separation difficulties, low reusability, and secondary waste generation. In this work, heterogeneous MgO and CaO supported Zeolite-Y catalysts were synthesized via a hydrothermal method to enhance metal dispersion and catalyst stability. Structural characterization by XRD confirmed the preservation of the Faujasite-Y framework after metal incorporation, whereas SEM and SEM-EDX analyses revealed a typical octahedral morphology and uniform distribution of MgO and CaO on the Zeolite surface. Acidity measurements verified the availability of active sites suitable for transesterification. The catalysts were evaluated in sono-transesterification of waste cooking oil with methanol at 65 °C for 2 h. MgO/Zeolite-Y achieved a biodiesel yield of 63.824%, while CaO/Zeolite-Y exhibited superior catalytic performance with a yield of 79.344%. The produced biodiesel complied with SNI 7182:2015 and ASTM D6751 standards. These results indicate that CaO/Zeolite-Y is a promising heterogeneous catalyst for biodiesel production, demonstrating superior performance compared to MgO/Zeolite-Y under optimised reaction conditions.

1 Introduction

Biodiesel has attracted increasing attention as a renewable alternative to fossil diesel in response to the depletion of petroleum reserves and rising environmental concerns. Biodiesel is a long-chain fatty acid methyl ester produced through the transesterification of vegetable oils or animal fats with methanol [1]. Compared with conventional fossil fuels, biodiesel offers several advantages, including reduced carbon and sulphur emissions, biodegradability, lower toxicity, and a reduced environmental impact on water and soil systems [2].

Among various feedstocks, waste cooking oil (WCO) has emerged as an attractive raw material due to its low cost, wide availability, and contribution to waste management [3] WCO is classified as hazardous waste generated mainly from households and the food service industry, with global production exceeding 190 million tonnes per year and approximately 1 million tonnes

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annually in the European Union [4]. The presence of free fatty acids such as oleic, palmitic, linoleic, stearic, myristic, and linolenic acids makes WCO a viable feedstock for biodiesel production when an appropriate catalytic system is employed [5].

Catalysts play a critical role in transesterification reactions. Although homogeneous catalysts exhibit high activity, their industrial application is limited by separation difficulties, poor reusability, corrosion, and wastewater generation. Consequently, heterogeneous catalysts have received increasing attention due to their ease of recovery, reusability, low corrosivity, and improved environmental compatibility [6]. Alkaline earth metal oxides such as Calcium Oxide (CaO) and Magnesium Oxide (MgO) are widely recognised as effective heterogeneous base catalysts; however, their application in bulk form is often constrained by poor dispersion of active sites, catalyst leaching, limited stability, and mass-transfer limitations [7].

Supporting metal oxides on porous materials has been proposed as an effective strategy to overcome these drawbacks. Zeolite-Y, a microporous crystalline aluminosilicate with a Faujasite structure, offers a large surface area, high thermal stability, tunable acid base properties, and an average pore diameter of approximately 7.4 Å, making it suitable for transesterification of bulky triglyceride molecules [8]. In addition to catalyst design, sono-transesterification has emerged as a promising reaction intensification technique. Ultrasonic irradiation enhances mass transfer between immiscible reactants through acoustic cavitation, micro mixing, and interfacial disruption, leading to reduced reaction time and improved biodiesel yield [9]. However, studies combining hydrothermally synthesised zeolite supported alkaline earth metal oxide catalysts with sono-transesterification remain limited.

Therefore, this study aims to develop heterogeneous MgO and CaO impregnated Zeolite-Y catalysts via a hydrothermal synthesis route to improve metal dispersion and catalyst stability, while directly addressing mass transfer limitations through Sono-transesterification. The catalysts were comprehensively characterised to elucidate their structural and physicochemical properties, and their catalytic performance was evaluated in the sono-transesterification of waste cooking oil using methanol, with particular emphasis on reaction time and biodiesel yield.

2 Methodology

2.1 Material and Instruments

The equipment used in this study consisted of 100 mL beakers (Iwaki), 50 mL burettes (Iwaki), measuring cylinders (Iwaki), racks and clamps, Erlenmeyer flasks (Pyrex), glass funnels (Pyrex), porcelain dishes, a digital balance (Shimadzu ATX224), volumetric pipettes (Iwaki), separating funnels (Pyrex), watch glasses (Pyrex), a 100°C thermometer, condenser (Iwaki), heating plate, magnetic stirrer, furnace (Thermo), oven (Memmert, Type: UN 55), Oswald viscometer, pycnometer (Iwaki), ultrasonic water bath (Branson 1510), Cleveland cup, filter paper, and universal indicator paper. The instruments used were XRD (PANalytical Type: X'Pert PRO), SEM-EDX (FEI, Type: Inspect-S50). The scales used were Ohaus scales with an accuracy of 0.01 and a set of Buchner filter apparatus. The materials used in this study were commercial NaY Zeolite, waste cooking oil obtained from fried chicken vendors in Malang (Indonesia), 95% p.a. methanol (Merck), ethanol, 37% p.a. HCl (Sigma Aldrich), dihydrate oxalic acid p.a. (Sigma Aldrich), phenolphthalein indicator, KOH p.a. (Sigma Aldrich), distilled water, MgO (Merck), CaO 90%, NH₄Cl, ammonia, and anhydrous MgSO₄.

2.2 Activation and Characterisation of Zeolite-Y

Commercial NaY Zeolite (50 g) was dispersed in 100 mL of 1 M NH₄Cl solution and agitated using a shaker at 300 rpm for 2 h to obtain the protonated form of zeolite. The suspension was

filtered and repeatedly washed with distilled water until a neutral pH was achieved, followed by drying at 120 °C for 2 h 30 min and calcination at 500 °C for 4 h to produce Zeolite-Y [10] [27]. The activated zeolite was characterised by X-ray diffraction (XRD) to confirm structural integrity and scanning electron microscopy (SEM) to examine surface morphology.

2.3 Catalyst Preparation (CaO, MgO/Zeolite-Y)

Preparation of CaO/Zeolite-Y and MgO/Zeolite-Y catalysts was carried out by adding Zeolite-Y to an aqueous suspension of Calcium Oxide (CaO) or Magnesium Oxide (MgO) with a metal oxide concentration of 30% (w/w), using a solution to zeolite weight ratio of 1:4. The mixture was stirred continuously for 24 h at room temperature under atmospheric pressure. The resulting mixture was then filtered and dried at 80 °C for 24 h, followed by calcination at 450 °C for 4 h [10]. The synthesised catalysts were characterised by SEM–EDX analysis and zeolite acidity analysis..

2.4 Processing of Waste cooking oil

The purification of waste cooking oil was initiated by heating for 1 h, followed by cooling and filtration to remove solid impurities. The filtered oil was then visually inspected for colour changes, and its free fatty acid (FFA) content was determined [9].

2.5 Sono-Transesterifikasi Waste cooking oil

Sono-transesterification was carried out using 25 g of waste cooking oil placed in an Erlenmeyer flask and preheated to 40 °C. The oil was then added to a preheated mixture of methanol and catalyst at 65 °C, with a catalyst loading of 4 wt.% relative to oil and a methanol to oil molar ratio of 12:1. The resulting mixture was initially shaken and subsequently subjected to sono-transesterification in an ultrasonic bath maintained at 65 °C for reaction times of 1, 1.5, and 2 h. After completion of the reaction, the mixture was centrifuged at 3000 rpm for 30 min and then allowed to settle in a separating funnel for 24 h. Two distinct layers were formed, with the upper layer identified as methyl esters (biodiesel) and the lower layer consisting primarily of glycerol. The biodiesel phase was washed with warm distilled water and subsequently heated at 90–100 °C to remove residual moisture. Anhydrous MgSO₄ was then added as a drying agent. Finally, the synthesised methyl ester was analysed for density, kinematic viscosity, acid number, and free fatty acid content in accordance with SNI 7182:2015 [10].

3 Results and discussion

3.1 Zeolite-Y activation

The activation of Zeolite-Y is carried out in two stages, namely physical activation to remove impurities and increase surface area, and chemical activation to reduce inorganic impurities and enlarge pores. In the chemical stage, zeolite is dispersed in an NH₄Cl solution so that ion exchange occurs between metal cations (Na⁺, K⁺, Ca²⁺) and H⁺ ions. The NH₄⁺ ions then decompose into NH₃ when heated to 500 °C, producing Zeolite-Y [10].

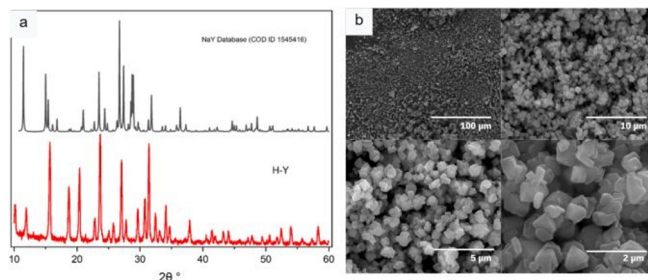


Fig. 1. (a) XRD patterns of activated Zeolite-Y and reference NaY (ICDD PDF No. 15-4476). (b) SEM images of activated Zeolite-Y recorded at different magnifications (scale bars: 100 μm , 10 μm , 5 μm , and 2 μm) after calcination at 500 $^{\circ}\text{C}$

The crystal structure of the Zeolite-Y catalyst was characterised using X-ray Diffraction (XRD) techniques and compared with reference patterns from the FAU Database (**Figure 1(a)**). The analysis results show that the main peaks on the Zeolite-Y catalyst ($2\theta = 10.16^{\circ}$, 11.94° , 15.68° , 18.76° , 20.39° , 22.81° , 23.65° , 25.79° , etc.) showed significant similarity to the peaks in the FAU Database, indicating that the crystal structure of the synthesised Zeolite-Y catalyst was consistent with the FAU standard structure. Furthermore, the crystal structure of Zeolite-Y is maintained after the synthesis process, and that small crystal size can increase its catalytic activity [11].

Morphological characterisation of the Zeolite-Y catalyst was also performed using SEM, as shown in **Figure 1(b)**. The SEM results indicate that at magnifications of 100 μm to 2 μm , the Zeolite-Y crystal particles have a uniform size and homogeneous distribution, with well-organised crystals. This structure supports an increase in active surface area, which is important for catalytic applications, particularly in transesterification reactions. These SEM results are consistent with the XRD results, which show agreement with the FAU Database pattern, confirming that the resulting Zeolite-Y microstructure is stable and suitable [10].

3.2 Characterization of Katalis (CaO, MgO/Zeolit-Y)

SEM-EDX analysis was used to observe the morphology and distribution of elements in metal oxide-impregnated Zeolite-Y catalysts. SEM results show changes in surface structure due to CaO and MgO impregnation, while EDX confirms the success of metal deposition and the stability of the aluminosilicate framework. This characterisation proves that the interaction between metal oxides and zeolite increases the base strength and thermal stability of the catalyst [12].

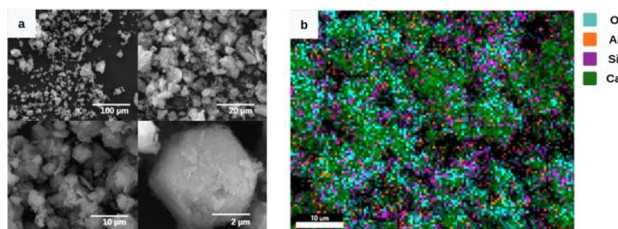


Fig. 2. (a) SEM images of CaO/Zeolite-Y at different magnifications (scale bars: 100 μm , 10 μm , 5 μm , and 2 μm). (b) EDX elemental mapping showing the distribution of O, Al, Si, and Ca on the CaO/Zeolite Y surface after calcination at 450 $^{\circ}\text{C}$.

Table 1. EDX elemental composition of CaO/Zeolite-Y catalyst after calcination at 450 °C.

| Element | Weight (%) | Atomic (%) |
|---------|------------|------------|
| O | 61.07 | 78.18 |
| Al | 2.81 | 2.14 |
| Si | 5.63 | 4.11 |
| Ca | 30.49 | 15.58 |

Figure 2(a), shows the SEM results of CaO/Zeolite-Y material with irregular particle morphology that forms micron to submicron sized aggregates. CaO particles are distributed relatively evenly on the zeolite surface and partially cover the pores. High magnification observations reveal a dense surface with fine crystallite agglomerations, confirming the success of CaO impregnation. These results are in line with the report by Yang and Yu, which describe that metal oxide incorporation can reduce external porosity through the development of an oxide layer along the pore surfaces, potentially influencing the accessibility of surface active sites [13]. Meanwhile, the EDX mapping results in Table 1, show a homogeneous distribution of O, Al, Si, and Ca elements across the surface, indicating successful impregnation without damaging the zeolite framework. The high oxygen content indicates the presence of Ca in the form of CaO, which acts as a strong base site in the catalyst [6].

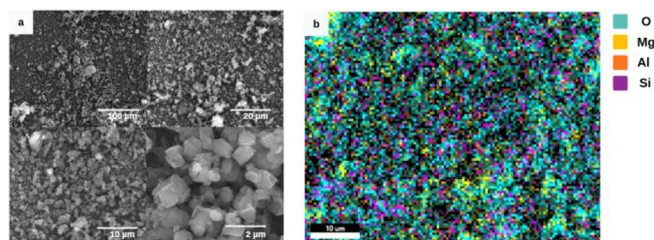


Fig. 3. (a) SEM images of MgO/Zeolite-Y at different magnifications (scale bars: 100 μm, 10 μm, 5 μm, and 2 μm). (b) EDX elemental mapping showing the distribution of O, Al, Si, and Ca on the CaO/Zeolite Y surface after calcination at 450 °C.

Table 2. EDX elemental composition of CaO/Zeolite Y catalyst after calcination at 450 °C.

| Element | Weight (wt.%) | Atomic (%) |
|---------|---------------|------------|
| O | 66.45 | 76.31 |
| Mg | 15.70 | 11.87 |
| Al | 5.39 | 3.67 |
| Si | 12.45 | 8.14 |

Figure 3(a) shows the SEM results of MgO/Zeolite-Y material with uneven particle morphology, dense aggregation, and a rougher and more porous surface than pure Zeolite-Y. This characteristic indicates that the MgO impregnation process successfully produced an oxide layer that covered part of the zeolite surface. At high magnification (2 μm), fine nanometer-sized particles were evenly distributed, indicating that MgO was homogeneously dispersed in the zeolite matrix. The EDX element mapping results shown in Table 2 indicate an even distribution of O, Mg, Al, and Si, indicating that the impregnation was successful without damaging the aluminosilicate framework and producing a catalyst that is stable at high temperatures [12]. The high oxygen content confirms that magnesium is dominant in the form of MgO, which acts as a strong electron donor and increases the number and strength of active base sites. This MgO layer enhances the catalyst's performance in transesterification and hydrocarbon cracking reactions. As

reported by Li et al., the interaction between MgO and zeolite base sites increases the activity and thermal stability of the catalyst compared to pure Zeolite-Y [14].

These characteristics confirm the successful impregnation of metal oxides onto Zeolite-Y and demonstrate the transformation of the catalyst surface from predominantly acidic to base dominated, which is essential for transesterification reactions. Metal oxide modification is widely reported to enhance catalytic performance by increasing base site density, strengthening basic active centres, and improving catalyst stability under reaction conditions [15]. Consequently, the observed reduction in acidity after impregnation reflects the effective neutralisation of Bronsted acid sites and the development of surface basicity, which promotes methanol activation and accelerates the transesterification process.

Catalyst acidity was evaluated using ammonia adsorption due to its small molecular size, allowing effective penetration into the zeolite pore system. The total acidity values, calculated from the mass difference before and after ammonia adsorption, are summarised in **Table 3**. The acidity of Zeolite-Y decreased substantially from 96.8 mmol g⁻¹ to 5.48 mmol g⁻¹ for MgO/Zeolite-Y and 6.45 mmol g⁻¹ for CaO/Zeolite-Y after metal oxide impregnation. This reduction indicates the replacement of acidic H⁺ sites by metal oxygen (M–O) basic species, resulting in a surface environment dominated by basic active sites that favour triglyceride conversion during transesterification [13]. The slightly higher residual acidity observed for CaO/Zeolite-Y compared to MgO/Zeolite-Y suggests a more balanced acid base interaction, which may contribute to improved catalytic performance by preventing excessive site neutralisation and maintaining reaction efficiency [10].

Table 3. Total acidity of Zeolite-Y before and after metal oxide loading determined by NH₃ adsorption

| Zeolite | Initial Acidity (mmol g ⁻¹) | Acidity After Impregnation | |
|-----------|---|----------------------------|------|
| | | MgO | CaO |
| Zeolite-Y | 96.8 | 5.48 | 6.45 |

3.3 Preparation of waste cooking oil

The purification process of waste cooking oil is carried out by heating it for one hour, then allowing it to cool and filtering it to remove solid impurities. The filtered waste cooking oil is then observed for colour changes and measured for free fatty acid (FFA) content as an indicator of purity [10]. **Table 4** shows the FFA content results in waste cooking oil to be below 2%, in accordance with the maximum limit recommended for the transesterification process. A low FFA value indicates that the waste cooking oil is suitable for use as a catalytic raw material without the risk of soap formation. Conversely, FFA levels above 2% can trigger saponification and hydrolysis reactions, which reduce the yield and purity of biodiesel. Therefore, the purification stage is a crucial step in obtaining stable oil that is ready for use in the conversion process [9].

Table 4. Physicochemical properties of purified waste cooking oil.

| Sample | Acid number (mg KOH g ⁻¹ , max) | Free Fatty Acids (%) | Kinematic viscosity at 40 °C (cSt) | Density (kg m ⁻³) |
|-------------------|--|----------------------|------------------------------------|-------------------------------|
| Waste Cooking Oil | 0.5 | 0.07 | 7.1 | 983 |

3.4 Characterization of The Synthesized Methyl Esters

Biodiesel synthesis was conducted using waste cooking oil that had been pretreated by heating and filtration to remove moisture and impurities, resulting in a reduced free fatty acid content of 0.077%, which is suitable for the sono-transesterification process. The reaction was performed using MgO/Zeolite-Y and CaO/Zeolite-Y catalysts at a catalyst loading of 4 wt.% relative to oil, with an oil to methanol molar ratio of 1:12, at 65 °C. Sono-transesterification was carried out for 1, 1.5, and 2 h to determine the optimum reaction conditions. The process resulted in the formation of two distinct phases, consisting of an upper methyl ester (biodiesel) layer and a lower glycerol layer Figure 4(a). due to ultrasonic waves producing cavitation that forms an emulsion between oil and methanol, thereby increasing the interfacial area, mixing efficiency, and accelerating the reaction [9].

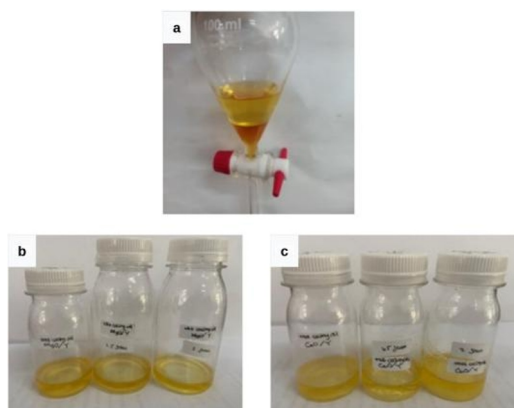


Fig. 4. Results of the Sono-transesterification reaction of waste cooking oil: (a) shows the separation of the upper layer (methyl ester) and lower layer (glycerol and residue). (b) and (c) show the results with MgO and CaO/Zeolite-Y catalysts at reaction times of 1, 1.5, and 2 hours, from left to right.

The results of the sono-transesterification of waste cooking oil summarised in Table 5 indicate that sono-transesterification time affects catalytic performance differently depending on the catalyst system. For MgO/Zeolite-Y, the biodiesel yield decreased from 72.87% at 1 h to 71.12% at 1.5 h and further declined to 63.82% at 2 h, suggesting that prolonged ultrasonic exposure may induce partial methyl ester degradation or soap formation. Although certain fuel properties improved at 2 h, as reflected by a kinematic viscosity of 5.48 cSt and a density of 880 kg m⁻³ that fall within the acceptable ranges of SNI 7182:2015 and ASTM D6751, some quality parameters at shorter sono-transesterification times remained borderline or non-compliant, indicating incomplete transesterification under these conditions. In contrast, the CaO/Zeolite-Y catalyst exhibited superior performance, achieving the highest biodiesel yield of 79.34% at 2 h of sono-transesterification together with favourable physicochemical properties, including a density of 853 kg m⁻³, kinematic viscosity of 4.70 cSt, acid number of 0.52 mg KOH g⁻¹, and free fatty acid content of 0.07%, all of which satisfy the requirements of both fuel standards. This superior performance is attributed to the stronger basicity of CaO, faster formation of methoxide ions, different dispersion and pore clogging characteristics, and more effective interaction of Ca²⁺ with the zeolite framework [6], [13]. These findings confirm that ultrasonic irradiation enhances cavitation-driven mass transfer and conversion efficiency when an appropriate basic catalyst is employed, while excessive sono-transesterification can negatively affect yield [9]. Overall, only the optimised reaction condition using CaO/Zeolite-Y at a sono-transesterification time of 2 h

fully meets both SNI 7182:2015 and ASTM D6751 specifications, demonstrating the superior catalytic performance of CaO/Zeolite-Y compared to MgO/Zeolite-Y.

Table 5. The characterization results of biodiesel produced through sono-transesterification of used cooking oil using MgO/Zeolite-Y and CaO/Zeolite-Y catalysts, compared with SNI 7182:2015 and ASTM D6751.

| Catalyst | Sono-transesterification time (h) | Yield (%) | Density (kg m ⁻³) | Kinematic viscosity at 40 °C (cSt) | Acid number (mg KOH g ⁻¹ , max) | Free Fatty Acids (%) |
|---------------|-----------------------------------|-----------|-------------------------------|------------------------------------|--|----------------------|
| | SNI 7182:2015 | | 850-890 | 2.3 – 6.0 | 0.8 | < 0.5 |
| | ASTM D6751 | | 820-900 | 1.6 – 5.8 | 0.5 | < 0.45 |
| MgO/Zeolite-Y | 1 | 72.87 | 998 | 7.07 | 0.7 | 0.11 |
| CaO/Zeolite-Y | 1 | 74.53 | 895 | 6.10 | 1.5 | 0.23 |
| MgO/Zeolite-Y | 1.5 | 71.12 | 983 | 6.12 | 4.3 | 0.66 |
| CaO/Zeolite-Y | 1.5 | 72.32 | 866 | 5.31 | 0.5 | 0.08 |
| MgO/Zeolite-Y | 2 | 63.82 | 880 | 5.48 | 0.7 | 0.11 |
| CaO/Zeolite-Y | 2 | 79.34 | 853 | 4.70 | 0.5 | 0.07 |

4 Conclusion

This study successfully synthesized heterogeneous catalysts MgO/Zeolite-Y and CaO/Zeolite-Y through a hydrothermal impregnation method. XRD analysis confirmed that the Faujasite Y crystal framework of Zeolite remained stable after activation and metal oxide impregnation, while SEM-EDX results showed a homogeneous dispersion of MgO and CaO on the zeolite surface. In the sono-transesterification of waste cooking oil, MgO/Zeolite-Y achieved its highest biodiesel yield of 71.12% at 1.5 h, before declining to 63.82% at 2 h, likely due to partial methyl ester degradation or soap formation caused by prolonged ultrasonic exposure. Despite the lower yield, the biodiesel quality remained high, with a viscosity of 5.48 cSt and a density of 880 kg m⁻³, both meeting the SNI 7182:2015 and ASTM D6751 standards. In comparison, CaO/Zeolite Y exhibited superior catalytic performance, producing a 79.34% yield, density of 853 kg m⁻³, viscosity of 4.70 cSt, acid value of 0.52 mg KOH g⁻¹, and FFA of 0.07%, indicating the most efficient reaction. These findings confirm that ultrasonic cavitation enhances the transesterification rate by improving the interfacial contact between methanol and oil; however, excessive sono-transesterification duration may reduce yield. Overall, CaO/Zeolite-Y demonstrated higher basic strength, better catalytic efficiency, and greater product purity than MgO/Zeolite-Y, making it a promising and environmentally benign catalyst for industrial biodiesel production and sustainable energy applications.

Acknowledgment

This work was supported by LPPM Universitas Negeri Malang and the authors acknowledge the assistance provided by the staff of the Chemistry Department during this study.

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