IJFAC
Indonesian Journal of
Fundamental and Applied Chemistry

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Synthesis and Characterization of Zn-Terephthalate MOF as a Catalyst for Biodiesel Production from Waste Cooking Oil

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Abstract

Utilization of waste cooking oil as a raw material for biodiesel production is an effective solution to reduce environmental pollution and produce renewable energy. The high free fatty acid content in waste cooking oil is a major problem in the industry, so it requires an effective catalyst in the biodiesel production process. The Zn-terephthalate MOF has a large surface area, porous structure, and good thermal stability, making it very effective as a heterogeneous catalyst for the biodiesel esterification process. This study aimed to synthesize and characterize Zn-terephthalate MOF using a hydrothermal method with a Teflon autoclave, and evaluate its effectiveness as a catalyst. The analysis was carried out by measuring the acidity of the Zn-terephthalate MOF catalyst, and characterizing by XRD, TGA, PSA, and converting used cooking oil into biodiesel. The results showed that the synthesized Zn-terephthalate MOF had an acidity of 4.33 mmol/g, the XRD results showed pattern of Zn-terephthalate MOF shows the main peaks located at 9.972°, 14.885°, 15.85°, 18.189°, 19.402°, 26.36°, 32.673°, and 39.25°. TGA results showed that the Zn-terephthalate MOF catalyst had thermal stability up to 400°C, and PSA results showed particles distributed at 233.54 nm. The Zn-terephthalate MOF catalyst can reduce the FFA value of waste cooking oil and convert it into biodiesel with a conversion rate of 94.18%, and the catalyst can be reused up to four times with relatively stable performance.

Keywords: Zn-terephthalate MOF, waste cooking oil, esterification, biodiesel

Article Info

Received 12 September 2025
Received in revised 4 October 2025
Accepted 7 October 2025
Available Online 29 October 2025

Abstrak (Indonesian)

Pemanfaatan minyak goreng bekas sebagai bahan baku produksi biodiesel merupakan solusi efektif untuk mengurangi pencemaran lingkungan dan menghasilkan energi terbarukan. Kandungan asam lemak bebas yang tinggi pada limbah minyak goreng menjadi masalah utama dalam industri, sehingga diperlukan katalis yang efektif dalam proses produksi biodiesel. Zn-tereftalat MOF memiliki luas permukaan yang besar, struktur berpori, dan stabilitas termal yang baik, sehingga sangat efektif sebagai katalis heterogen untuk proses esterifikasi biodiesel. Penelitian ini bertujuan untuk mensintesis dan mengkarakterisasi Zn-tereftalat menggunakan metode hidrotermal dengan autoklaf Teflon, serta mengevaluasi efektivitasnya sebagai katalis. Analisis dilakukan dengan menguji keasaman katalis Zn-tereftalat MOF, dan dikarakterisasi dengan XRD, TGA, PSA, serta mengkonversi minyak goreng bekas menjadi biodiesel. Hasil penelitian menunjukkan bahwa Zn-tereftalat MOF memiliki keasaman 4,33 mmol/g, hasil XRD menunjukkan pola Zn-tereftalat MOF dengan puncak-puncak utama terletak pada 9,972°, 14,885°, 15,85°, 18,189°, 19,402°, 26,36°, 32,673°, dan 39,25°. Hasil TGA menunjukkan bahwa katalis memiliki stabilitas termal hingga 400°C, dan hasil PSA menunjukkan partikel terdistribusi pada 233,54 nm. Katalis Zn-tereftalat MOF dapat menurunkan nilai FFA minyak goreng bekas dan mengubahnya menjadi biodiesel dengan tingkat konversi 94,18%, serta katalis dapat digunakan kembali hingga empat kali dengan kinerja yang relatif stabil.

Kata Kunci: Zn-tereftalat MOF, limbah minyak goreng, esterifikasi, biodiesel

INTRODUCTION

Diesel is a fossil fuel that is essential for the public, both for vehicles and work equipment that use diesel engines [1]. The increasing demand makes the availability of diesel fuel dwindling, so there are other alternatives as renewable energy that are comparable to diesel [2]. This can be overcome by the existence of renewable and environmentally friendly energy that is very necessary, namely biodiesel [3]. Biodiesel can be used in diesel engines and diesel motor vehicles with or without the addition of diesel fuel [4]. Indonesia is implementing the B35 biodiesel blending program, namely a mixture of biodiesel with diesel fuel up to 35% in accordance with the Decree of the Minister of Energy and Mineral Resources (ESDM) No. 295.K/EK.01/MEM.e/2022. The main raw material for making biodiesel, namely palm oil, is a major problem in biodiesel production. This is because palm oil is also used for food needs [5]. However, the scarcity of raw materials and high production costs still make biodiesel more expensive than conventional fossil fuels[6]. One alternative raw material for biodiesel is by utilizing used cooking oil [7]. Waste cooking oil containing free fatty acids and triglycerides can be converted into biodiesel through esterification and transesterification processes [8]. Both require effective catalysts to accelerate the reaction and increase conversion yields [9]. Homogeneous acid catalysts such as sulfuric acid are often used for esterification, but have major drawbacks such as reactor corrosion, separation difficulties, and recycling difficulties [10]. In the transesterification stage, the use of homogeneous base catalysts is prone to soap formation, especially when the FFA content in the feedstock is quite high [11].

One alternative catalyst that has the advantages of being easily separated, reusable, and can increase conversion yields is a heterogeneous solid catalyst [12]. Metal–Organic Frameworks (MOFs) are crystalline materials consisting of metal ions or metal clusters connected by organic ligands that can be used as heterogeneous catalysts [13]. MOFs have a very large surface area, high porosity, and flexibility in active site design, making them ideal for various types of catalytic reactions [14].

Zn-terephthalate MOF is a heterogeneous catalyst of the Metal–Organic Frameworks (MOFs) type which is effective as a catalyst for the biodiesel production process [15]. Zn-terephthalate MOF is made of Zn^{2+} ions which act as Lewis acid sites and terephthalate ligands which form a stable porous structure [16]. Zn-terephthalate MOF allows the effective diffusion of large molecules in used cooking oil as well as the activation of carbonyl groups in FFA and triglycerides [17]. The Zn MOF structure has a large surface area, porous structure, and good thermal and chemical stability, so it is widely used as a catalyst acetylene acetoxylation [18], dye remover [19], xylene sensing [20], antitumor drug carrier [21], color degradation [22], fluorescence sensor [23].

The stability test of Zn-terephthalate MOF showed that it remained active when reused for several subsequent cycles [24]. With its advantages of high catalytic activity, structural stability, and environmentally friendly properties, as well as being reusable Zn-terephthalate MOF is a promising alternative to replace homogeneous acid catalysts such as sulfuric acid or conventional solid catalysts that are susceptible to degradation, reactor corrosion, difficult to separate, and difficult to recycle [7]. The many benefits of MOF are believed to actively facilitate esterification and transesterification reactions in waste cooking oil and maintain high activity and structural stability [25].

MATERIALS AND METHODS

Materials

Material used in this study include Zinc nitrate hexahydrate $Zn(NO_3)_2{\cdot}6H_2O$ (AR/p.a), Terephthalic Acid $C_6H_4(CO_2H)_2$ (Sigma Aldrich), N,N-Dimethylformamide (DMF) (Merck) KGaA, Triethylamine (Merck), Chloroform (Merck), Waste Cooking Oil (WCO was taken after 7 uses for 7 days), Sodium Hydroxide, Methanol pa. Phenolphthalein Indicator (Merck), Distilled Water, Oxalic Acid, Ammonia (Merck), and Hydrochloric Acid (Merck).

Synthesis of Zn-terephthalate MOF

The synthesis of Zn-terephthalate MOF was carried out with a ratio of 2:1 between 3.57 g (12 mmol) zinc nitrate hexahydrate and 0.9966 g (6 mmol) terephthalic acid dissolved separately in 15 mL DMF. The two solutions were mixed and magnetically stirred at 200 rpm for 5 min at room temperature, 0.223 mL of triethylamine was added dropwise. Next, the solution was transferred into a hydrothermal reactor which was autoclaved at 120 °C for 24 h [26]. After cooling to room temperature, the catalyst was filtered and washed with 5 mL of DMF three times. The catalyst was filtered and soaked in 15 mL of chloroform for 24 h, then dried and activated at 100 °C for 12 h [17]. The acidity of the Zn-terephthalate MOF catalyst was measured using ammonia adsorption combined with a gravimetric method to calculate the weight difference before and after adsorption using the following formula (1). 1 g of Zn-terephthalate MOF was acidified in a desiccator with flowing ammonia for 24 h. After adsorption, the catalyst was left at room temperature for 2 h and its weight was calculated [27].

$$\mathrm{Kal} = \frac{W_2 \times 1000}{M_b \times W_1} \tag{1}$$

Kal = solid acidity (mmole/g)
W_1 = solid weight (g)
W_2= weight of ammonia adsorbed (g),
M_b = molar mass of NH_3 (17.03 g/mole).

Characterization of Zn-terephthalate MOF catalyst
X-ray diffraction (XRD)

The measurements were carried out using Cu-Kα radiation (λ = 1.541 nm) at 30 kV and 10 mA in the 2θ scan range from 5 to 50. The obtained diffraction patterns were analyzed and compared with reference databases such as JCPDS no.96-432-6738 to confirm the crystal structure and phase of the material [22].

Thermogravimetric analysis (TGA)

TGA analysis was performed to compare the thermal stability of the materials. Heating from 30–1000°C under N_2 atmosphere was performed to determine the decomposition stage. The weight change of the samples with temperature was recorded to evaluate the thermal stability and identify the decomposition stage such as the release of terephthalate ligands or degradation of the MOF structure [23].

Particle size analyzer (PSA)

Analysis with PSA with particle size distribution measured using laser diffraction method. This data provides information about particle size and its distribution which influence catalyst activity [21].

Esterification reaction of waste cooking oil with Zn-terephthalate MOF catalyst

A total of 2.5 mL of waste cooking oil was added with 10 mL of methanol, stirred, and heated at 60 ºC. The solution was titrated using 0.1 N sodium hydroxide to calculate the free fatty acid number of waste cooking oil. A total of 25 g of waste cooking oil was added with 79 mL of methanol and 3 g of Zn-terephthalate MOF catalyst into a two-neck round-bottom flask by reflux method at 80 ºC for 150 minutes [28]. The esterification results were added with 50 mL of distilled water, then separated by centrifugation. The biodiesel product found at the top was washed again with distilled water 3 times, and finally dried by heating at 80 ºC for 15 minutes to remove residual methanol [17]. A total of 2.5 mL of biodiesel was added with 10 mL of methanol. The mixture was titrated with Phenolphthalein indicator using 0.1 N sodium hydroxide to determine the remaining free fatty acids from biodiesel. The conversion of the reaction results was determined based on the decrease in free fatty acids from the reaction results. Calculation of the conversion of reaction results using the formula:

$$\text{Conversion} = \left(\frac{\text{Initial \%FFA} - \text{Final \% FFA}}{\text{initial \%FFA}}\right) \times 100\ \% \tag{2}$$

Initial %FFA: Free Fatty Acids in Used Cooking Oil
Final %FFA : Free Fatty Acids after reaction

Reusability of Zn-terephthalate MOF catalyst

The Zn-terephthalate MOF catalyst was vacuum filtered and washed with alcohol four times after the esterification reaction. The catalyst was then dried for 12 h at 100 °C and stored for use in the next esterification cycle [17].

RESULTS AND DISCUSSION

Synthesis of Zn-terephthalate MOF

The synthesis of Zn-terephthalate MOF was carried out using a solvothermal method using DMF as the primary solvent. In the initial stage, zinc nitrate hexahydrate served as the source of Zn^{2+} ions, while terephthalic acid (H_2BDC) served as the bridging organic ligand. The addition of triethylamine increased the solubility of terephthalic acid through deprotonation of the-COOH group, thereby accelerating coordination with the Zn^{2+} ions. The solvothermal process at 120 °C for 24 hours enabled the formation of a three-dimensional crystalline structure [$Zn_4O(BDC)_3$] with a regular pore framework, typical of MOF-5. Washing with DMF and soaking in chloroform removed any remaining solvent molecules trapped in the pores, resulting in a material with a larger surface area. Activation at 100 °C for 12 hours subsequently ensured that the MOF pores were optimally open for catalytic applications.

The acidity test of the catalyst using the ammonia adsorption method showed a value of 4.3 mmol/g. This value indicates the presence of a fairly high acid site in Zn-terephthalate MOF, which generally comes from the Zn^{2+} metal center as Lewis acid sites. This acidity level is considered good to support catalytic reactions such as esterification reactions in biodiesel production. Compared with conventional heterogeneous catalysts, this MOF has a higher ammonia adsorption capacity, which confirms the potential of Zn-terephthalate MOF as a bifunctional catalyst [17].

X-ray diffraction (XRD)

The crystallinity characterization of Zn-terephthalate MOF through XRD analysis shows a typical diffraction pattern of MOF material with sharp intensity peaks in the low 2θ region shown in **Figure** 1, which corresponds to the $Zn_4O(BDC)_3$ framework structure. The existence of the main peaks in the diffraction pattern confirms the formation of a regular crystalline phase, indicating that the synthesis successfully produces Zn-terephthalate MOF with good crystal quality. This is in line with the JCPDS No. 96-432-6738 literature [22] which has dominant peaks in the low angle region due to the large unit cell size. The XRD pattern of Zn-terephthalate MOF in **Figure** 1 shows the main peaks located at 9.972°, 14.885°, 15.85°, 18.189°, 19.402°, 26.36°, 32.673°, and 39.25°. In accordance with JCPDS No. 96-432-6738 peaks at 9.7°, 14.67°, 15.76°, 17.59°, 19.18°, 26.19°, 32.38°, and 39.96° indicate the formation of MOF-5 used for XRD pattern analysis. The presence of peaks at 10° to 20° is likely due to changes in material morphology due to temperature and solvent variations. Another peak at 30° to 50° indicates the presence of a small amount of ZnO [29].

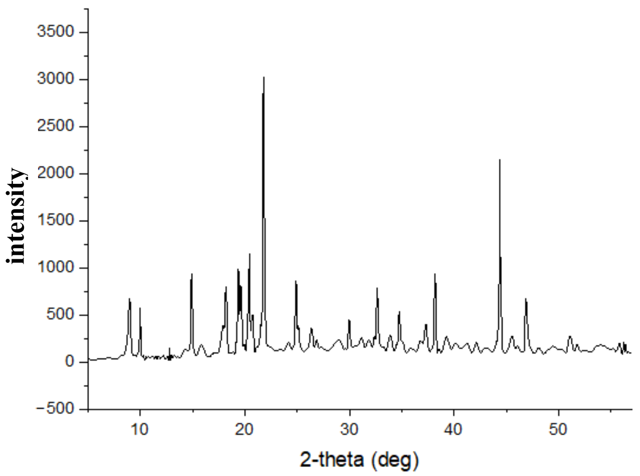

Figure 1. XRD pattern of Zn-terephthalate MOF

Thermogravimetric analysis (TGA)

Thermogravimetric analysis (TGA) showed several stages of mass decomposition in the sample described in **Figure** 2. The initial mass loss at 150 °C (-6.18%) occurred in the low temperature range, which can be attributed to the evaporation of water molecules trapped in the catalyst pores. The second stage at 180-300 °C with a mass loss of 25.57% indicates the loss of the remaining DMF solvent trapped in the catalyst [22]. The main decomposition stage occurred in the range of 300–500°C with a significant mass loss of about 25.43% and 3.04% which represents the degradation of terephthalate ligands (BDC) from the MOF framework. After 500 °C, there was an additional loss of 9.67% to nearly 1000°C, leaving a residue of about 29.92%, which is likely ZnO as the final product [10]. These TGA results confirm that Zn-terephthalate MOF has thermal stability up to around 400 °C, so it is still adequate for application in the biodiesel catalytic process which generally takes place at lower temperatures.

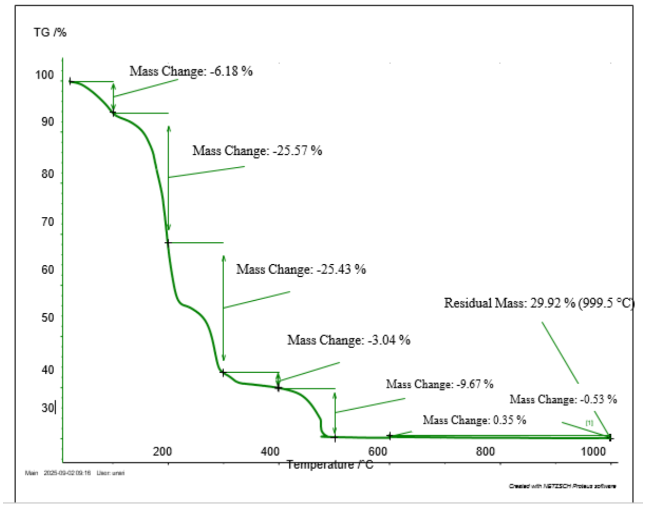

Figure 2. Thermogravimetric analysis curve of Zn-terephthalate MOF

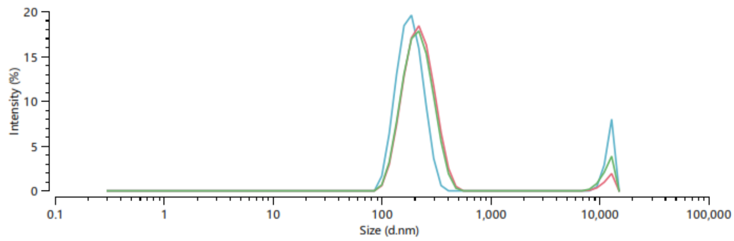

Figure 3. Particle Size Analyzer Zn-terephthalate MOF

Particle size analyzer (PSA)

The results of particle size analysis with PSA in **Figure** 3 show a relatively large Z-average particle value, namely in the range of 231.52–237.16 nm with a polydispersity index (PDI) between 0.025-0,295A. This value indicates that the material is on the nanometer scale, which indicates the successful formation of a MOF structure with a fine particle size. The polydispersity index (PDI) value of 0.025–0.295 (<0.5) indicates a relatively narrow and homogeneous particle size distribution, indicating good particle dispersion stability. This small particle size and uniform distribution have the potential to increase the active surface area of Zn-Terephthalate MOF, thereby strengthening its performance as a heterogeneous catalyst in the biodiesel transesterification reaction. [21].

Esterification reaction of waste cooking oil with Zn-terephthalate MOF catalyst

The esterification of waste cooking oil involves the reaction of free fatty acids (FFA) react with methanol with the help of a catalyst to form methyl ester (biodiesel) and water [30]. The results of the study showed that the high free fatty acids in waste cooking oil of 11.03% can be reduced to 0.64%, meaning that the sample can be converted into biodiesel as much as 94.18% with the Zn-terephthalate MOF catalyst. The Zn-terephthalate MOF catalyst plays an important role because it has an active center in the form of a Zn^{2+} ion which is a Lewis acid and an –OH group on the MOF framework which is a Brønsted acid. The initial stage of the reaction begins when the carbonyl group (C=O) of the fatty acid interacts with the Zn^{2+} Lewis acid site on the catalyst, causing a decrease in the electron density on the carbonyl carbon atom so that it becomes more electrophilic [31]. Next, the methanol molecule binds to the –OH group of the MOF which functions as a proton donor (Brønsted acid), so that the methanol is activated to become a more reactive nucleophile. The protonated methanol then attacks the electrophilic carbon atom on the fatty acid carbonyl group, forming a tetrahedral intermediate. This intermediate is unstable and reorganizes, releasing a water molecule as a byproduct, and producing an ester (methyl ester/biodiesel) as the main product [32]. With this mechanism, the Zn-terephthalate MOF catalyst acts dually: Zn^{2+} acts as a Lewis acid to activate the carbonyl group, while the –OH group in the organic framework acts as a Brønsted acid to facilitate the protonation of methanol. This bifunctionality makes the reaction faster and more selective, while suppressing side reactions such as fatty acid polymerization or the formation of unwanted products [17].

Reusability of Zn-terephthalate MOF catalyst

One of the main advantages of using Zn-terephthalate MOF as an esterification catalyst is its heterogeneous nature, which allows it to be easily separated from the reaction mixture using filtration or centrifugation [33]. The Zn-terephthalate MOF catalyst at the beginning of the reaction can reduce and convert free fatty acids from waste cooking oil into biodiesel by 94.18%. After the reaction is complete, the catalyst is washed using methanol to remove residual fatty acids, methyl esters, and glycerol that may be adsorbed on the surface or pores [17]. This washing process and drying the catalyst aims to reactivate the catalyst. With this simple treatment, the Zn-terephthalate MOF catalyst can be reused in the next reaction cycle without requiring complex reactivation. The results of the reusability conversion of the Zn-terephthalate MOF catalyst can be seen in **Figure** 4. In the next cycle, the catalyst can convert by 88.35%, 83.99%, and 75.74%. This proves that the Zn terephthalate catalyst is very effective as a biodiesel esterification catalyst and can be reused [17].

The stability of the crystalline structure of Zn-terephthalate MOF is able to maintain the metal-organic framework even though it is used repeatedly, so the reusability is very good [17]. Zn^{2+} ions that act as Lewis acid sites remain firmly bound to the central zinc oxide, while terephthalate ligands maintain the regularity of the pores so that the accessibility of methanol and fatty acids remains high. The decrease in catalyst activity after several uses is more often caused by pore blockage by large molecules or particle agglomeration that reduces the active surface area, rather than by permanent damage to the MOF structure [34]. Therefore, simple regeneration through washing and heating can restore most of the catalyst activity [35].

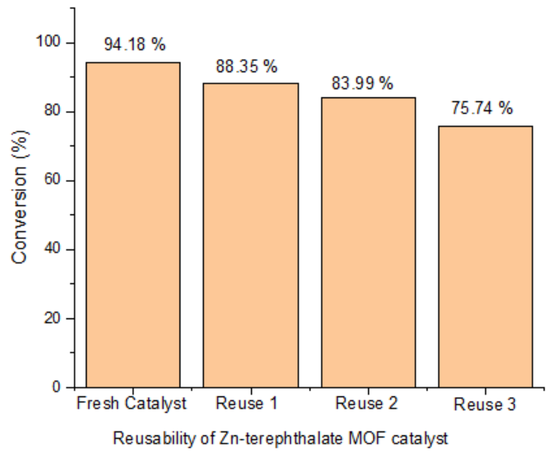

Figure 4. Reuse of Zn-terephthalate MOF catalyst

CONCLUSION

The synthesis of Zn–terephthalate MOF was successfully carried out using the hydrothermal method, as evidenced by several characterization results. The XRD pattern matched the reference data (JCPDS No. 96-432-6738), confirming the formation of a well-defined MOF crystalline structure. The TGA analysis demonstrated good thermal stability up to approximately 400°C, while PSA results revealed a uniform particle size distribution that supports catalytic performance. The catalyst exhibited a high biodiesel conversion efficiency of 94.18%, demonstrating its effectiveness in converting waste cooking oil. Furthermore, the catalyst maintained its performance after four consecutive reuse cycles with minimal loss of activity, indicating good durability. These findings suggest that Zn–terephthalate MOF possesses strong potential for industrial applications, both at the pilot scale and in integrated sustainable biodiesel production systems.

ACKNOWLEDGMENT

The author would like to thank Sriwijaya University for granting permission and fully supporting the author in conducting this research in the Physical Chemistry Laboratory.

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