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Production of biodiesel over ZnO-TiO₂ bifunctional oxide catalyst supported on natural zeolite

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Abstract. In this paper, biodiesel is produced from palm oil and methanol via the transesterification process over the heterogeneous catalyst. The metal oxide ZnO-TiO₂ was impregnated into natural zeolite over the dry impregnation method. The ZnO-TiO₂/NZ catalyst was prepared over the dry impregnation method. The catalysts' characteristic was characterized by X-ray diffraction (XRD) and Scanning Electron Microscope (SEM). The biodiesel was analyzed by Fourier-Transform Infrared Spectroscopy (FTIR) and Gas Chromatography-Mass Spectrometry (GC-MS). The results showed a functional group of methyl ester C=O, carbonyl group at 1744.51 cm⁻¹. The components of methyl ester such as hexadecanoic acid methyl ester (C₁₇), cis-9-heptadecanoic acid methyl ester (C₁₈), oleic acid methyl ester (C₁₉), are present in the biodiesel with the percent area of 0.05%, 0.08%, 0.63%, 7.06%, 16.3%, respectively over ZnO-TiO₂/NZ catalyst with a metal ratio of 1:1. This catalyst was successful in the transesterification of palm oil to produce biodiesel.

1. Introduction

Nowadays, fossil fuels are the main source of energy used as fuel. However, the combustion of fossil fuels produces gases such as nitrogen oxides (NO_x), sulfur oxides (SO_x), carbon monoxide (CO), carbon dioxide (CO₂) and other pollutants that cause environmental effects. Renewable energy sources are required to resolve this issue [1-3]. One of the potential renewable energy sources is biodiesel. Biodiesel is a promising alternative energy. Renewable raw materials such as edible and inedible vegetable oils, animal fats, and oils from algae can produce biodiesel [4].

Biodiesel production generally uses a transesterification reaction, which requires alcohol to break the triglyceride chains contained in vegetable oils to form esters and glycerol in the product. Methanol and ethanol are commonly used in this transesterification reaction. During this time, the production of biodiesel using homogeneous catalysts, such as acids (HCl, H₂SO₄, BF₃ and sulfonic acids) [5], and



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basic alkaline catalysts (KOH, NaOH, CH₃OK, CH₃ONa, and CH₃CH₂ONa) [6]. The use of this homogeneous catalyst causes problems in the product. For example, it still contains a catalyst, which must be separated again, and the formation of emulsions and soaps, difficult to separate and purification of catalyst and products [7].

This homogeneous catalyst can be replaced with heterogeneous catalysts that are more environmentally friendly, stable at high temperatures, have large pores inexpensive. These problems can be minimized by the use of heterogeneous catalysts in the transesterification process. The use of heterogeneous catalysts is economical and has several advantages, such as being non-corrosive, environmentally friendly, and easy to separate from liquid products and activities. The selectivity is relatively high [8]. Heterogeneous catalysts show high potency, strength and durability, up to 40% free fatty acids [9]. Heterogeneous catalysts used in transesterification of vegetable oils are zirconia oxide, titanium, zeolite, potassium zirconia, oxides of alkaline earth metals and derivatives (CaO, MgO, SrO), boron and group elements carbon (generally loaded on alumina), etc [10-11].

So far, both natural zeolite and synthetic are promising catalysts to produce biodiesel via the transesterification process. Natural zeolite is an abundant resource, low-cost, environmentally friendly, and easily available alternative as a heterogeneous catalyst for biodiesel production. Several studies on biodiesel production by natural zeolite have been conducted. Mierczynski et al., [12-13] reported the production of biodiesel over Natural zeolite from Gunungkidul (Yogyakarta, Indonesia) with the addition of CaO, MgO and SrO oxides and noble metals (Pt, Pd, Ru, and Ag) onto the natural zeolite. The result showed that the catalyst improves the triglycerides conversion and fatty acid methyl ester (FAME).

This study aimed to determine the effect of ZnO-TiO₂/NZ catalyst on its catalytic in the transesterification reaction. The properties of the catalyst were investigated XRD, SEM and the product was analyzed over FT-IR and GC-MS.

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2. Materials and methods

2.1 Materials

In this study obtained natural zeolite from South Lampung, Lampung and supplied from a local supplier. The commercial palm oil is purchased from the local market and directly used in transesterification. Metal oxide such as titanium oxide (TiO₂) and zinc oxide (ZnO) was purchased from Merck. The methanol solvent for transesterification was purchased from Merck.

2.2 Preparation of catalysts

The preparation of catalyst following the method previously by Al Muttaqii et al., [14-16] with the incipient wetness impregnation method. Natural zeolite was modified over physical and chemical activation. First, the natural zeolite was calcined at 500 °C for 3 h to remove the water and open the pore. Then, it was dealumination over HCl solution (1M) and desilication over NaOH solution (0.5M). The powders and the solution were mixed and heated at a temperature of 70 °C for 1 h. The natural zeolite was washed using distilled water to a neutral pH (pH = 7) and dried at a temperature of 130 °C for 5 h. The modified natural zeolite was obtained. Next, the natural zeolite was added with metal oxide precursors such as titanium oxide (TiO₂) and zinc oxide (ZnO). The metal loading in the bifunctional oxide catalyst was 5% with metal ratios of 1:1 and 1:3. The catalyst was allowed to stand overnight and dried at a temperature of 130 °C for 3 h. The bifunctional metal oxide catalyst was calcined at a temperature of 500 °C for 5 h. The catalysts were characterized over XRD (X-ray diffraction) and SEM- (Scanning Electron Microscopy).

2.3 The Transesterification Process

The transesterification process was performed in the batch reactor. 3 g of catalyst and the mole ratio of methanol and vegetable oil of 6:1 were used for each running. Then, it was heated to 60 °C for 3 h. After the reaction, the sample was cooled to room temperature. Then, the product was analyzed by FT-IR Fourier Transform Infra-Red (FT-IR) and Gas Chromatography-Mass Spectrometry (GC-MS). Fourier

Transform Infra-Red (FT-IR) was used to determine the functional groups of biodiesel. Gas Chromatography-Mass Spectrometry (GC-MS) was used to determine the chemical composition of the liquid product.

3. Results and discussion

3.1 The Properties of Catalyst

The diffractograms of the natural zeolite and ZnO-TiO₂/NZ catalyst are shown in Figure 1. Figure 1 shows the diffraction peak of natural zeolite at 2θ of 22.395°, 28.097°, and 22.793°. According to JCPDS No. 25-1349, the type of natural zeolite from Lampung is clinoptilolite [12-13]. After the impregnation process of metal oxide on natural zeolite, ZnO metal showed the presence with JCPDS No. 036-1451 at 2θ of 34.42° and 36.24°, and TiO₂ with JCPDS No. 29-1360 at 2θ of 54.29° and 56.58°. The presence of ZnO and TiO₂ did not change the crystallinity structure of natural zeolite but reduced the diffraction peak intensity [17]. This result indicates ZnO and TiO₂ were obtained on the natural zeolite.

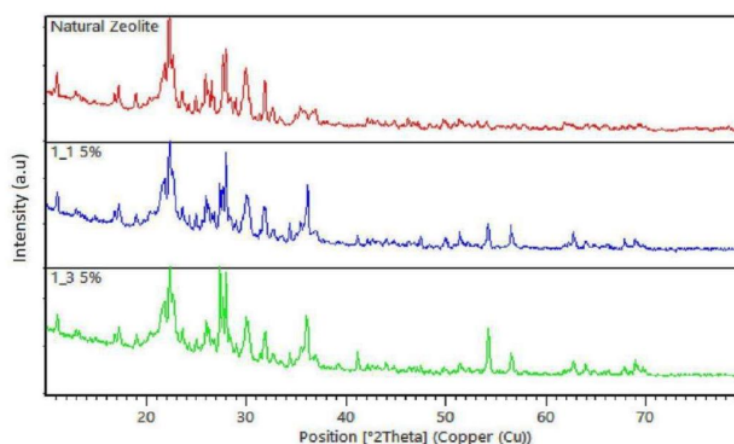


Figure 1. The diffractogram of natural zeolite and ZnO-TiO₂/NZ catalyst with metal loading 5% and metal ratios of 1:1 and 1:3

Figure 2 shows the results of the scanning electron micrograph of natural zeolite and ZnO-TiO₂ catalyst. The results showed natural zeolite has spherical and agglomerates and consists of non-uniform size. After impregnation, obtained amount of metal oxide with irregular form shapes and the dispersion of metals on the surface is not uniform [13].

FT-IR is used to determine the functional groups contained in biodiesel products. FTIR characterization was carried out at wavenumbers 4000–600 cm⁻¹. The spectrum of biodiesel compounds can be seen in Figure 3. In Figure 3 the transesterification reaction produces a product with a typical functional group of methyl ester, such as the vibration of the C=O (carbonyl) group that appears in the absorption region of 1744.51 cm⁻¹. The vibration of the C-O at 1162.9 cm⁻¹, the vibration of the C-C at 1461.1 cm⁻¹. The C-H group appears at the absorption of 2855.1 and 2922.2 cm⁻¹. So, it can be seen that there is an ester compound formed from the transesterification reaction process [18].

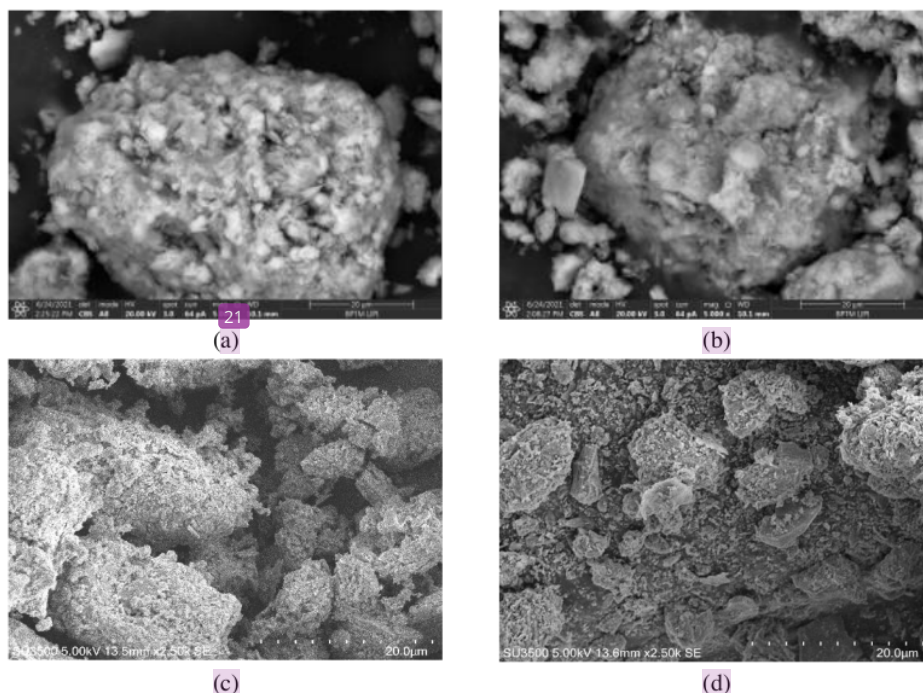


Figure 2. Scanning electron micrograph of natural zeolite and ZnO-TiO₂/NZ catalyst with metal loading 5% and metal ratios of 1:1 and 1:3

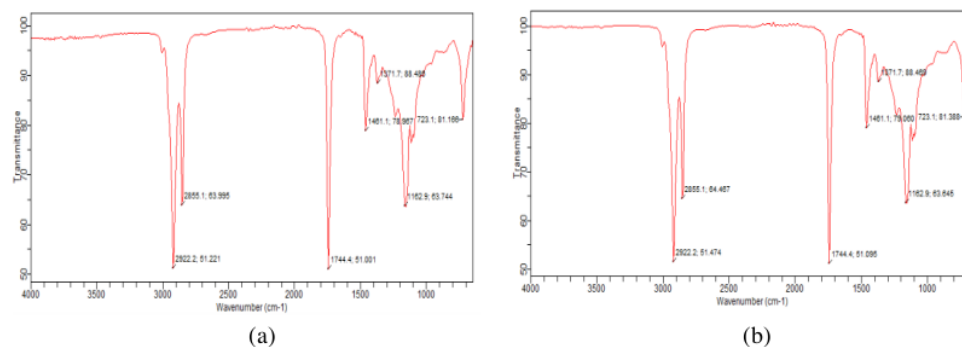


Figure 3. The FT-IR spectrum of biodiesel over ZnO-TiO₂/NZ catalyst with metal loading 5% and metal ratios of 1:1 and 1:3

Figure 4 shows a chromatogram of the biodiesel product produced using ZnO-TiO₂/NZ catalyst. Over ZnO-TiO₂/NZ catalyst with a metal ratio of 1:1, the components of heptanoic acid methyl ester (C₈), Octanoic acid, methyl ester (C₉), nonanoic acid, 9-oxo- methyl ester (C₁₀), hexadecanoic acid methyl ester (C₁₇), cis-9-heptadecanoic acid methyl ester (C₁₈), oleic acid methyl ester (C₁₉), are present in the biodiesel with the percent area of 0.05%, 0.08%, 0.63%, 7.06%, 16.3%, respectively. Comparison to ZnO-TiO₂/NZ catalyst with a metal ratio of 1:1, the components of hexadecanoic acid methyl ester (C₁₇) and oleic acid methyl ester (C₁₉) are present in the biodiesel with the percent area of 6.85% and

6.46%, respectively. According to Putra et al. [19] the biodiesel obtained from the transesterification process contains fatty acids in the range of C₁₆-C₂₀.

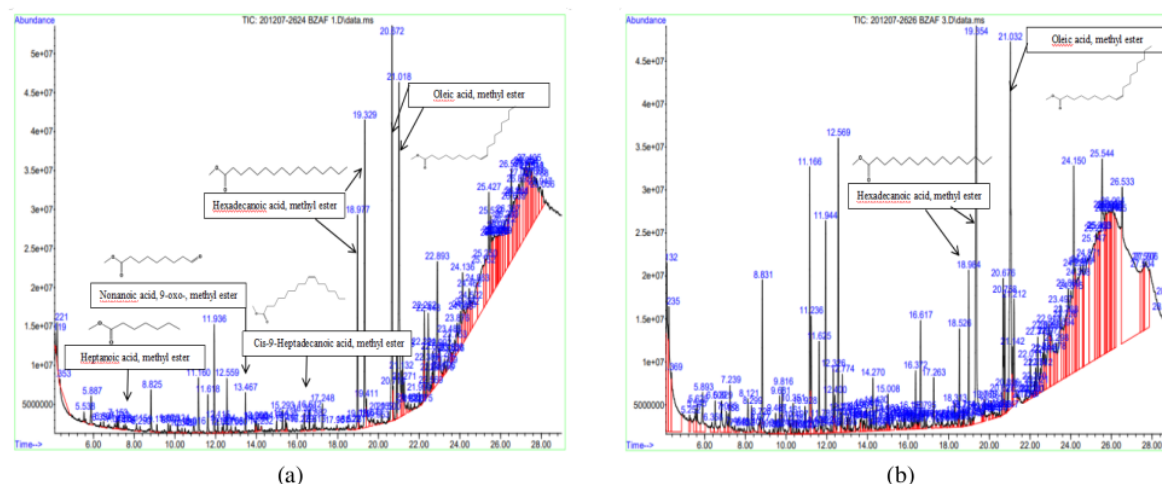


Figure 4. The chromatogram of biodiesel over ZnO-TiO₂/NZ catalyst with metal loading 5% and metal ratios of 1:1 and 1:3

4. Conclusions

This study has shown that natural zeolite from Lampung is Clinoptilolite type. Metal oxide ZnO and TiO₂ supported on the natural zeolite were prepared by the incipient wetness impregnation method. After impregnation, the crystallinity of natural zeolite did not change. The functional groups contained in biodiesel products showed a typical functional group of methyl ester (C=O) at 1744.51 cm⁻¹. In the range of C₁₆-C₂₀, the biodiesel obtained from the transesterification process mostly of hexadecanoic acid methyl ester (C₁₇) and oleic acid methyl ester (C₁₉). The developed ZnO-TiO₂/NZ catalyst showed a potential catalyst to produce biodiesel in the transesterification process.

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Authors Contributions

MAM is the main contributor with analysis of catalyst and product, writing original draft, review, and editing. MA, EP, RA, LM are member contributors with analysis of the data and editing.

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