Synthesis of Biodiesel in Low-Grade Palm Oil using Geopolymer-ZnO Catalyst

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ABSTRACT

This study aimed to synthesize biodiesel (fatty acid methyl ester) from low-grade palm oil using geopolymer-ZnO catalyst. The activity of catalyst was tested by mixing low-grade palm oil and methanol in a mole ratio of 1:10, with varying catalyst concentrations of 1% , 3%, and 5% at a temperature of 67°C and different time intervals. Subsequently, the progression of the reaction was monitored using thin-layer chromatography (TLC). The results showed that catalyst ratios of 1:0, 1:1, and 1:2 provided complete conversion at concentrations of 3% and 5%. Geopolymer-ZnO catalyst reuse test was carried out using a concentration of 5%, followed by washing with n-hexane. After drying at 100°C, the sample was reused in biodiesel synthesis. The reuse test findings showed optimum results at 1:2 variation and 2 hours of reaction time with total methyl ester conversion. In addition, the majority of the experiments performed were carried out using a 1:1 variation with a maximum of 3 repetitions, which consistently showed total conversion to methyl ester. Catalyst used was then characterized using FTIR, XRD, and XRF instruments, with the analysis results confirming that it was geopolymer-ZnO. Methyl ester obtained was analyzed using GC-MS, and the findings showed that the main compositions included methyl oleate (47.35%) and methyl palmitate (40.13%).

Keywords: Biodiesel; geopolymer-ZnO; low grade palm oil; transesterification

INTRODUCTION

The demand for fuel oil (BBM) in Indonesia is currently increasing, as evidenced by a 2.76% increment in national fuel consumption (71.58 to 73.56 million kiloliters) from 2015 to 2016. The largest increase occurred in the category of general fuel (JBU), which experienced a 9.45% increase (44.45 to 48.66 million kiloliters) in the same year (BPH Migas, 2017). Based on the Strategic Plan (Renstra) of the Ministry of Energy and Mineral Resources 2015 – 2019, Indonesian petroleum reserves are expected to experience a total

depletion in the next 13 years, with the remaining current reserves standing at 3.6 billion barrels (Sa'adah et al, 2017). Consequently, there is a pressing need to develop alternative renewable energy aimed at overcoming the problem of shortages. In this context, several studies have recommended the use of biodiesel as an alternative renewable energy. Biodiesel is an alkyl ester derived from vegetable oil (Bustaman, 2009), particularly palm oil in West Kalimantan.

According to previous reports, palm oil can be used as a biodiesel raw material through esterification or transesterification reactions. Esterification is a

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reaction between fatty oil and alcohol, which typically leads to the production of esters using acid catalysts, such as H_2SO_{4} , AlCl₃, and PTSA (Usman et al, 2019). This process is particularly suitable for oil with high free fatty acid (ALB/FFA) content (acid number \geq 5 mg-KOH/g). Meanwhile, transesterification is a reaction between alcohols and triglycerides using basic catalysts, including NaOH, KOH, and metal oxides to produce acyl esters as the main product and glycerol as a by-product (Harinda & Nur, 2019; (Usman et al, 2019). Vegetable oil used in the transesterification process often has low ALB levels of less than 1% (Usman et al, 2019 ; Astar, I., 2017). One of palm oil products with less than 1% free fatty acid content is bulk palm oil.

In line with previous studies, catalyst used for biodiesel production through esterification and transesterification is generally homogeneous. However, homogeneous catalyst has several disadvantages, such as being corrosive, complicated product separation, producing toxic waste, and not being used repeatedly, leading to economic inefficiency (Kondamudi et al , 2011). The material can also react with ALB to form soap, which causes high consumption. This indicates that there is a need to develop heterogeneous catalyst with non-corrosive and easy-to-separate properties for biodiesel production. This alternative is typically solid, easy to separate, reusable, cost-effective, environmentally friendly, non-corrosive, and has high catalyst activity (Aqhilla et al, 2017).

Several reports have explored the use of heterogeneous catalyst, such as Nugraha I & Umi K, (2017) which used geopolymer catalyst from rice husk ash and kaolin to synthesize biodiesel from *nyamplung* (*Calophyllum inophyllum*) seed oil. Geopolymer can serve as a basic catalyst, active metal support, and oxide catalyst. The material also has catalyst site accessibility, a large surface area (Alimuddin et al, 2017), and strong basic properties.

In the context of improving geopolymer performance, zinc (Zn) is an important concern because it has the potential to be developed as heterogeneous catalyst in biodiesel production. Over the years, geopolymer-ZnO has only been used in measuring microstructure and thermal properties (Zulkifli et al, 2019) as well as assessing the compressive strength of geopolymer (Rustan et al, 2015). However, the application as heterogeneous catalyst in biodiesel production has largely remained unexplored. According to Arifah et al, 2016, the use of ZnO catalyst in synthesizing biodiesel from palm oil produced a best conversion of 93.56%. Therefore, geopolymer-ZnO heterogeneous catalyst was used for the synthesis process in this study. Catalyst had high catalyst activity, alkaline properties, and could

be reused (regenerated), leading to superiority over conventional geopolymer or ZnO catalyst for synthesizing biodiesel using bulk palm oil.

METHODS

Materials

The materials used in this study were bulk palm oil, kaolin, distilled water, dichloromethane (CH_2Cl_2) p.a, ethanol (C₂H₅OH) p.a, potassium hydroxide (KOH) p.a, Whatman filter paper, methanol (CH₃OH) p. a, sodium hydroxide (NaOH) p.a, sodium chloride (NaCl) p.a, sodium silicate (Na₂SiO₂) p.a, n-hexane p.a, 60 GF $_{254}$ TLC plate, zinc oxide (ZnO) p.a, and cyclohexane p.a. Meanwhile, the tools used were a set of reflux apparatus, separatory funnel, pycnometer, thermometer, centrifugation, SHIMADZU Fourier transform infrared (FTIR) spectrophotometer, GC-MS QP2010S SHIMADZU, X-Ray Diffractometer (XRD), and X-Ray Fluorescence (XRF) PANalytical Epsilon 3.

Kaolin Preparation

Kaolin was prepared by washing the material using distilled water 3 times, followed by filtration. The wet kaolin was then dried at 80°C for 24 hours, crushed, and sieved with a 100 mesh sieve. Subsequently, the sample was calcined at 700°C for 3 hours and the product was obtained in the form of metakaolin (Wahyuni et al, 2019).

Figure 1. Biodiesel synthesis apparatus circuit

Geopolymer-ZnO Catalyst Synthesis

The preparation of geopolymer catalyst began with mixing metakaolin and ZnO by stirring with variations in the weight/weight ratio (b/b) of metakaolin:ZnO, namely 1:0, 2:1, 1:1, and 1:2. Alkaline solution was prepared by mixing sodium silicate (12.2 g), sodium hydroxide (1.1 g), and distilled water (0.7 g) until homogeneous. Subsequently, the alkaline solution was added in drops to the metakaolin:ZnO mixture until a homogeneous geopolymer paste mixture was obtained. The mixture was then heated in an oven at 60°C for 48 hours. Geopolymer formed was allowed to stand for 7 days and then crushed into powder. Catalyst formed was characterized using FTIR, XRD, and XRF (Nugraha et al. 2017; Rustan et al, 2015).

Determination of Free Fatty Acid Content

A total of 1 g bulk palm oil was weighed and placed into a 250 mL erlenmeyer. A total of 10 mL ethanol and 2 mL phenolphthalein (pp) indicator were added and titrated with 0.1 N NaOH until a pink solution was formed and did not disappear for 30 seconds. The process was continued with the recording of the volume of NaOH (Usman et al, 2019).

Catalyst Activity Test of Geopolymer-ZnO

Catalyst activity test consisted of two stages, namely (1) catalyst activity test of newly used geopolymer-ZnO and (2) catalyst activity test of reused geopolymer-ZnO that had been pretreated. Both testing stages were carried out using the same testing methodology, namely catalyst activity test through transesterification reaction by mixing methanol and catalyst in each variation with a percentage of 5%, 3%, and 1% (w/b). Subsequently, bulk palm oil was added in a mole ratio of 1:10 and heated at 67°C with a time variation of 1, 2, and 3 hours while stirring with a stirrer. The reaction was monitored by thin layer chromatography method using cyclohexane and dichloromethane eluent in the ratio of 2:1. The reaction

product was placed into a separatory funnel leading to three layers, namely catalyst layer (bottom), glycerol (middle), and methyl ester (top). Methyl ester product was washed with saturated NaCl solution, and washing was performed until the resulting product became neutral. The resulting product was characterized by product purity using GC-MS (Putri, P. C. A., dan Edy, 2020).

Geopolymer-ZnO Catalyst Reuse Test

Catalyst reuse test preparation was performed by filtering the 5% variation of geopolymer-ZnO that had been used with filter paper. Geopolymer-ZnO catalyst was washed with n-hexane with the ratio of catalyst weight to solvent volume that had been determined. In addition, geopolymer-ZnO catalyst was dried at 100 °C (Tambak, 2019).

RESULTS AND DISCUSSION

Geopolymer-ZnO Catalyst Synthesis

The synthesis of geopolymer-ZnO was carried out by mixing metakaolin as raw material with ZnO in a stirred manner at a variation of weight/weight ratio (b/b) of 1:0, 2:1, 1:1, and 1:2. The addition of ZnO served as a dopping in the transesterification reaction by increasing the activity of heterogeneous catalyst (Lestari, 2018). In addition, the alkaline solution was prepared by mixing sodium silicate (12.2 g), sodium hydroxide (1.1 g), and distilled water (0.7g) until homogeneous, which was used to activate geopolymer precursor, which polymerized to form a Si-O-Al-O polymer framework after being added to the metakaolin:ZnO mixture. The obtained geopolymer-Zn paste mixture was then placed into a mold and heated at 60°C for 48 hours to unite and strengthen the resulting geopolymer structure, as well as remove moisture content in geopolymer (Abdullah et al, 2018). The resulting geopolymer is presented in Figure 1.

Figure 2. Geopolymer-ZnO catalyst variations of (A) $1:0$, (B) $2:1$, (C) $1:1$, and (D) $1:2$

Geopolymer-ZnO Catalyst Test

Catalyst activity test was conducted by reacting methanol with geopolymer-ZnO catalyst to form methoxy methanol. In addition, the transesterification reaction used palm oil mixed with methanol (mole ratio 1: 10). The use of more methanol was performed, hence, the direction of the reaction moved towards the product. Methanol served as a source of alkyl groups in the transesterification reaction because it had high reactivity with biodiesel results in the form of fatty acids methyl ester (FAME) (Soerawidjaja, 2006 ; Priyambudi & Amirullah, 2017).

in ulian, 2017).
Geopolymer-ZnO catalyst was added with variations in the ratio of 5%, 3%, and 1% by weight (b/w), to bulk palm oil to determine the optimum concentration variation. The sample was then heated at 67°C with time variations of 1, 2, and 3 hours while stirring with a stirrer to examine an increase in the concentration of methyl ester formed as time increased. The reaction results were monitored by thin layer chromatography method on aluminum plates with silica gel 60 GF $_{254}$ adsorbent using cyclohexane and dichloromethane organic solvents as mobile phases in a ratio of 2:1. The chromatography results of TLC elution of methyl ester compounds with variations in time and concentration are presented in Figure 2.

Figure 2 showed that geopolymer could act as a catalyst in the synthesis of bulk palm oil into biodiesel as indicated by the separation of methyl ester and residual obtained oil stains. The application of 1% catalyst percentage, at of stams the appreasent of 1% catalyst percentage, at the composition of tax, and concentration in the samples. the presence of residual oil, but as the reaction time **the concentration catalyst Reuse**d. The reaction results were monitored by the reaction results were monitored by the reaction results were monitored by the reaction increased, methyl ester produced continued to increase. However, some of the oil had not been converted into methyl ester, which was indicated by the presence of residual oil stains at every hour. In the use of 3% and residual oil but as the reaction time **Geopolymer-ZnO Catalyst Reuse residual oil** but as the reaction time

5% catalyst, the monitoring results using TLC method showed a small amount of oil stains starting from the 1st hour, indicating that most of the oil had been converted into methyl ester.

Based on Table 1, the approximate methyl ester conversion results were obtained due to the unavailability of tools to analyze the percent conversion **Example 3 and 3 and 3 (3) of methyl ester specifically. The 5% geopolymer-ZnO** catalyst in all variations was able to convert methyl ester totally, while the use of 3% geopolymer-ZnO catalyst could convert totally in variations of 1:0, 1:1, r_{ref} (Socrawiaja), 2000, Friyambuar and 1:2. In addition, catalyst variation of 2:1 and 1:2 became the most optimum catalyst because it could convert more methyl esters at the use of 1% catalyst compared to other catalyst variations. Geopolymerumum concentration and ZnO catalyst using 1% in all variations was only able and the catalyst using 1% in all variations was only able to convert methyl ester partially. This was because ZnO could provide a large enough catalyst surface area to react with methanol and bulk palm oil. The addition of ZnO as doping increased methyl ester produced due to by thin layer chromatography an increase in the alkalinity of catalyst (Lee et al, 2009 ; Lestari, 2018).

The reaction product was placed into a separatory funnel leading to three layers, namely catalyst layer of TLC elution of methyl ester (bottom), glycerol (middle), and methyl ester (top). variations in time and concentration Methyl ester product was washed with saturated NaCl Figure 2. The use of more measured solution, and the resulting methyl ester was measured owed that geopolymer could act as a source fatty acid content and density, namely 0.0563% nthesis of bulk palm oil into biodiesel as for ALB and 0.8853 g/mL for density. Methyl ester obtained was characterized using GC-MS to determine of the composition of fatty acids contained with variation $\frac{1}{2}$ is the resolution of 5 $\frac{1}{2}$ is the ratio of 5 $\frac{1}{2}$ is the r

I ester produced continued to increase. The selection of heterogeneous catalyst in the of the oil had not been converted into vegetable oil transesterification process was based hich was indicated by the presence of con catalyst activity and ability to be reused in the reaction process, also known as regeneration. In

Table 1. Biodiesel conversion result using geopolymer-ZnO catalyst

Description: T reaction product was placed into a separatory function \mathcal{L} and \mathcal{L} and \mathcal{L}

 $\langle \cdot \rangle$ = not converted

 $(+)$ = slightly converted $(+)$

 $(++)$ = partially converted

 $(+++)$ = totally converted

Saturated NaCl Methyl ester

Figure 3. Transesterification results (A) methyl ester and glycerol (B) methyl ester and saturated NaCl

addition, regeneration was carried out in industry to reduce production costs as well as to determine the effectiveness of catalyst in the context of studies. Several studies also showed that it aimed to support data from previous conversion results.

Preparation of catalyst regeneration was performed by filtering geopolymer-ZnO that had been used with filter paper to separate geopolymer-ZnO catalyst from the reaction results in the form of methyl ester. Geopolymer-ZnO catalyst was washed with n-hexane

organic solvent to remove compounds adsorbed on catalyst. N-hexane was selected because it could dissolve methyl ester. Subsequently, geopolymer-ZnO catalyst was dried at 100 °C to remove the solvent content on geopolymer-ZnO catalyst (Tambak, 2019) After drying for 24 hours, catalyst could be reused for other procedures. Drying geopolymer-ZnO at 100°C for 24 hours was carried out to remove n-hexane as a washing solution, where it was known that n-hexane had a boiling point of 68.7 °C. Therefore, it could be evaporated at a drying temperature of 100 °C.

In this study, the used geopolymer-ZnO catalyst that had gone through the preparation stage was reused in the transesterification reaction to produce biodiesel with a concentration of 5% at geopolymer-ZnO mixture variation of 1:0, 1:1, 1:2, and 2:1, reaction time of 3 hours, and temperature of 67 °C. Regeneration of geopolymer-ZnO catalyst using 5% catalyst occurred because it could provide the best conversion results from the 1st hour. The reaction results were analyzed using a thin layer chromatography method on an aluminum plate with silica gel 60 GF $_{254}$ adsorbent using cyclohexane and dichloromethane organic solvents as mobile phases in a ratio of 2:1.

TLC test results showed that geopolymer-ZnO catalyst could repeatedly synthesize bulk palm oil into biodiesel, which was characterized by separating methyl ester and residual oil stains. The results of catalyst activity test through the regeneration test obtained the most optimum results in the use of 1:2 geopolymer-ZnO catalyst, which was indicated by the total conversion results at 2 hours of R1. Catalyst variation with the most regeneration outcome was carried out by the 1:1 variation with a repetition of 3 times, while the 1:0, 1:2, and 2:1 catalyst variations could only be used repeatedly 2 times. This result was due to the addition of ZnO in geopolymer manufacture, compared to the 1:0 catalyst variation without ZnO.

Characterization of Geopolymer-ZnO with FTIR

Characterization of Geopolymer-ZnO catalyst with FTIR was carried out to identify structural changes from the presence of functional groups and fingerprints of geopolymer-ZnO catalyst that had not been used in biodiesel synthesis. In addition, characterization with FTIR on geopolymer-ZnO catalyst showed a typical pattern with infrared wave number region in the range of 4000-350 because the typical spectrum of geopolymer and ZnO appeared in this region. Characterization of

| Geopolymer-ZnO catalyst variation | Catalyst regeneration | Reaction time (hour) | | | |
|--------------------------------------|--------------------------|--------------------------|--------------|----------------|-----------|
| | | $\mathbf 0$ | $\mathbf{1}$ | $\overline{2}$ | 3 |
| 1:0 | R ₀ | | $\ddot{}$ | $\ddot{}$ | $^{+++}$ |
| | R1 | | $\ddot{}$ | $^{+}$ | $\ddot{}$ |
| | R ₂ | | | | |
| 2:1 | R ₀ | | $\ddot{}$ | $+++$ | $^{+++}$ |
| | R1 | | $\ddot{}$ | $^{+}$ | $^{+++}$ |
| | R ₂ | | | | |
| 1:1 | R ₀ | | $\ddot{}$ | $^{+++}$ | $^{+++}$ |
| | R1 | | $\ddot{}$ | $\ddot{}$ | $^{+++}$ |
| | R ₂ | | $\ddot{}$ | $\ddot{}$ | $^{+++}$ |
| | R ₃ | | | | |
| 1:2 | R ₀ | $\overline{}$ | $+++$ | $^{+++}$ | $^{+++}$ |
| | R1 | | $\ddot{}$ | $^{+++}$ | $^{+++}$ |
| | R ₂ | | ۰ | | - |

Table 2. Regeneration results using geopolymer-ZnO catalyst

Description:

 $R0$ = The use of a new catalyst.

 $R1$ = The use of catalyst after one washing.

 $R2$ = The use of catalyst after two washes.

R3 = The use of catalyst after three washes.

Figure 4. FTIR spectra of geopolymer-ZnO catalyst of variations (A) 1:0, (B) 2:1, \overline{C} 3. Functional groups and \overline{C} (C) 1:1, and (D) 1:2.

geopolymer-ZnO with FTIR was performed on catalyst with variations of 1:0, 2:1, 1:1, and 1:2. The results \qquad without $\frac{1}{2}$ of geopolymer-ZnO characterization are presented in Figure 4.

Characterization of Geopolymer-ZnO with XRD

The initial (fresh) geopolymer-ZnO catalyst was characterized using XRD to determine the crystal structure of the resulting geopolymer-ZnO. Each mineral was detailed by a specific arrangement of atoms to create a field of marker atoms that could reflect X-rays. The measurement process was carried out from the angle of 26 diffraction angle 2 θ in the range $10 - 90^{\circ}$.

XRD characterization results of geopolymer catalyst without ZnO addition and geopolymer catalyst with ZnO addition are presented Figure 5. In **Characterization of Geopolymer-ZnO with XRD** addition, Figure 5 (A) showed the diffractogram of

pure geopolymer sample with metakaolin raw material without ZnO addition. Based on the analysis results, haracterization are presented in there were high peaks at angles $2\theta = 21.11^{\circ}$; 25.52° ;
 26.62° , 26.28° , 26.62° , 26.52° , 26.42° , 26.32° , 26.32° , 27.52° ; 26.62°; 26.8°; 42.68°; 50.33°; 60.12° and 68.29°. The highest diffractogram peak was at an angle of 2θ = θ 26.8° , indicating that quartz was the most dominating 1) geopolymer-ZnO catalyst was bhase in this geopolymer sample. XRD peak obtained was consistent with XRD results of geopolymer in the re accomme the crystal study conducted by Prasetya et al, 2018, showing that leopolymer-ZnO. Each mineral the 20 angle of geopolymer produced was relatively the same with the appearance of the highest peak at an angle of $2\theta = 26.72^\circ$ with the dominating component being $SiO₂$. $\sum_{i=1}^{\infty}$ and $\sum_{i=1}^{\infty}$ dominating

 5.5 \sim ₂.
Figure 5 (B) showed that the addition of ZnO with the ratio of metakaolin and ZnO 2:1 revealed high peaks at angles $2θ = 21.0°$; $26.5°$; $26.7°$; $29.5°$; $39.5°$, $47.6°$; 5 (A) showed the diffractogram of 48.6° and 50.2° . The highest diffractogram peak was

of atoms to create a field of marker atoms that could reflect \mathcal{F} Table 3. Functional groups and fingerprints of geopolymer-ZnO catalyst

Figure 5. Difractograms of geopolymer-ZnO catalyst variation (A) standard ZnO (ICDD, 2016) (B) 1:0, (C) 2:1, (D) 1:1 and (E) 1:2

found at angle 2θ = 26.7°, which indicated the quartz \qquad patterns almost the same as the 1:1 ratio wii phase of the metakaolin-based geopolymer component. The presence of angle $2\theta = 29.5^\circ$ showed the presence of zincite phase in geopolymer sample caused by the aliffractogram peak in the 1;2 variation was at ar addition of ZnO (Yunita et al, 2020). The addition of $\qquad \qquad$ of 2 θ = 29.6°, which showed the zincite phase fr ZnO to geopolymer mixture led to a shift of the zincite diffractogram peak from ZnO at the highest peak of 31.7° (ICCD, 2016). The higher SiO₂ peak indicated that in this variation, geopolymer produced had more SiO₂ $=$ 36.4° and 38.0°. components compared to ZnO component.

The diffractogram of geopolymer-ZnO with **Characterization of Geopolymer-ZnO with)** metakaolin ratio: ZnO 1:1 ratio showed that the Geopolymer-ZnO catalyst was characterized addition of ZnO with the same ratio as metakaolin led to a change in peak height at 2θ angle. High peaks for this ratio were obtained at angles $2\theta = 21.0^{\circ}$; 26.8°; 29.6°; 36.2°; 36.7°; 39.6°; 43.3°; 47.7°; 48.7°; and 50.3°. The highest diffractogram peak in the 1;1 variation was at an angle of $2\theta = 29.6^\circ$, which revealed the zincite phase from the addition of ZnO to geopolymer. In this diffractogram, there was a quartz phase shown at angles $2\theta = 21.0^{\circ}$; 26.8°, as well as calcite peaks presented at angles $2θ = 36.2°$ and $36.7°$.

The diffractogram of geopolymer-ZnO with metakaolin-ZnO ratio of 1:2 showed the effect of ZnO addition with a ratio of 1:2, which produced XRD

patterns almost the same as the 1:1 ratio with high of the metakaolin-based geopolymer component. $\qquad \qquad$ peaks at angles 2 θ = 26.8°; 29.6°; 32.4°; 36.2°; 36.4°; esence of angle 20 = 29.5° showed the presence \qquad 38.0°; 39.6°; 43.38°; 47.7° and 48.7°. The highest diffractogram peak in the 1;2 variation was at an angle of $2\theta = 29.6^{\circ}$, which showed the zincite phase from the addition of ZnO to geopolymer. In this diffractogram, beak from ZnO at the highest peak of there was a quartz phase shown at angle $2\theta = 26.8^\circ$, ICCD, 2016). The higher SiO, peak indicated that as well as calcite peaks presented at angles $2\theta = 36.2^{\circ}$; 36.4° and 38.0°.

Characterization of Geopolymer-ZnO with XRF

Geopolymer-ZnO catalyst was characterized using XRF to determine the amount of oxide components k height at 20 angle. High peaks for this a contained in catalyst. Characterization using XRF was ere obtained at angles $2\theta = 21.0^{\circ}$; 26.8°; 29.6°; carried out on catalyst that had not been used in $\frac{1}{2}$ 36.7°; 39.6°; 43.3°; 47.7°; 48.7°; and 50.3°. biodiesel synthesis with variations of 1:0, 2:1, 1:1, and The material control of the results of analysis using XRF, most phistogram peak in the 1;1 variation was at 1:2. Based on the results of analysis using XRF, most components in catalyst were SiO_2 at 34.367%, and ZnO at 35.153%.

Characterization of Methyl Ester by GC-MS

Testing of methyl ester produced from the initial (fresh) geopolymer-ZnO catalyst was carried out using GC-MS spectra. GC-MS analysis aimed to determine fatty acids comprising methyl ester and the quantity

Table 4. Oxide composition of geopolymer-ZnO catalyst

| Oxide | | Geopolymer-ZnO catalyst variation (%) | | | | | |
|--------------------------------|--------|---------------------------------------|--------|--------|--|--|--|
| | 1:0 | 2:1 | 1:1 | 1:2 | | | |
| AI_2O_3 | 20.001 | 12.989 | 10.355 | 6.723 | | | |
| SiO ₂ | 75.191 | 57.992 | 49.291 | 34.367 | | | |
| P ₂₀₅ | 1.552 | 1.584 | 1.323 | 1.253 | | | |
| K,O | 0.672 | 0.359 | 0.243 | 0.124 | | | |
| TiO ₂ | 1.037 | 0.738 | 0.628 | 0.474 | | | |
| Fe ₂ O ₃ | 0.883 | 0.65 | 0.527 | 0.428 | | | |
| 7nO | 0.126 | 14.898 | 22.574 | 35.153 | | | |
| CaO | 0.273 | 10.317 | 12.462 | 20.659 | | | |

Table 5. Components of methyl ester compounds from CPO

of each composition contained. Based on the results of GC-MS analysis, the main content of methyl ester from palm oil was methyl oleate with a percentage of 47.35% and methyl palmitate of 40.13%, as shown in Table 5.

CONCLUSION

In conclusion, the use of geopolymer-ZnO catalyst proved promising for the synthesis of biodiesel from bulk palm oil, demonstrating consistent efficiency across all variations tested, each yielding total conversion of biodiesel. Among these variations, the 1:2 catalyst ratio was particularly optimal, exhibiting complete biodiesel conversion with a 5% concentration, specifically when coupled with a 1-hour regeneration cycle. In addition, the 1:1 variation of geopolymer-ZnO catalyst showed

exceptional regenerative capability, allowing for up to three rounds of biodiesel synthesis. Characterization through FTIR, XRD, and XRF analyses confirmd the successful formation of geopolymer-ZnO. Methyl ester obtained was subjected to thorough analysis through GC-MS, revealing methyl oleate (47.35%) and methyl palmitate (40.13%) as the primary constituents.

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CONFLICT OF INTEREST

The authors declare no conflict of interest with other parties.

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