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Abstract

The diminishing of fossil fuel reserve has raised a consideration on the renewable energy development. Biodiesel is among the promising renewable energy which is feasible for large-scale production. Biodiesel is generally synthesized through the alkaline-catalyst transesterification of vegetable oil. The common catalyst for biodiesel is homogeneous base catalysts which are active but show several drawbacks related to the environmental aspects. Therefore, development of heterogeneous alkaline catalyst for biodiesel production is critical. CaO catalyst is considered a favourable heterogeneous base catalyst for transesterification reaction and it can be derived from various natural resources. In this work, CaO catalyst from eggshell was synthesized from eggshell waste. To improve the catalyst activity, CaO was combined with ZnO active metal, resulting ZnO/CaO catalyst. In this research, the development, characterization, and application of ZnO/CaO catalyst for waste cooking oil (WCO) transesterification to produce biodiesel has been investigated. Various concentration of ZnO was combined with CaO to determine the best formulation of ZnO/CaO catalyst development. It was demonstrated that the addition of ZnO active metal on CaO catalyst could remarkably improve the biodiesel yield through WCO transesterification reaction. The addition of 6% ZnO active metal on CaO, forming ZnO/CaO 6% catalyst, has exhibited the optimal enhancement of biodiesel yield. Furthermore, it was found that the optimum amount of ZnO/CaO 6% catalyst added in the reaction system was 3% w/w catalyst/WCO.

INTRODUCTION

The issue on fossil fuel reserve depletion has significantly increased the attention on the renewable energy development. Among the prospective alternative energy that is feasible to be produced in mass-production scale is biodiesel. Biodiesel can be derived from various vegetable oils and animal fats (Kusumaningtyas et al., 2016). Waste cooking oil (WCO) is including the potential feedstock for biodiesel synthesis. WCO contains carcinogenic compounds, thus, from the health standpoint it is not suitable for human consumption. On the other hand, if WCO is thrown away as waste, it will cause the environmental problem. Therefore, utilization of WCO as biodiesel feedstock will provide an economical value-added (Garrigós, 1999).

One important factor to achieve a high yield on biodiesel production is catalyst. The most common catalyst in biodiesel production is homogeneous alkaline catalyst, such as potassium hydroxide or sodium hydroxide (Ehsan & Chowdury, 2014). However, homogeneous catalyst demonstrates several drawbacks such as it has low selectivity, corrosive property to the equipment and difficulty in product separation. Besides, it is not reusable so that it causes catalyst waste. Hence, development of heterogeneous catalyst becomes essential (Fan et al., 2018).

Among the heterogeneous alkaline catalyst for biodiesel synthesis, CaO has gained notable interest due to its advantages. CaO catalyst has a high basicity strength. It is also cheap, non-corrosive, and low environmental impact (Kaewdaeng et al., 2017). Kawashima et al. (2009)

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declared that base oxide in CaO has a high catalytic activity in transesterification reaction, increasing the biodiesel yield to 90%. CaO catalyst can be developed from various natural resources, among others is eggshell waste.

Eggshell waste contains 94% weight $CaCO_3$ (Arabhosseini & Faridi, 2018), which is potential as the feedstock of CaO. Based on the data released by the Statistics Indonesia (2016), the egg consumption in Indonesia is 1.289.718 tons per year. This consumption results in 1.000.000 tons eggshell waste. On the other hand, $CaCO_3$ contained in the eggshell can be converted to CaO through calcination process. Therefore, utilizing eggshell waste as the raw material for CaO catalyst will provide added-value on this waste.

However, CaO catalyst commonly exhibits lower catalytic activity compared to KOH and NaOH (Singh et al., 2011). It provides biodiesel yield of 81.83% in the transesterification of WCO, which is considered unsatisfying yet (Rahkadima & Abdi, 2016). To overcome this shortcoming, CaO can be combined with active metal oxide to improve its catalytic activity (Ali et al., 2016). Metal oxide from transition metal, such as Cu, Ni, Cr, Zn, Fe, are usually shows excellent performance as catalyst. Among this transition metal, metal oxide of Zn (ZnO) is cheaper and suitable for esterification reaction. Combination of ZnO and CaO can be carried out using precipitation or impregnation methods (Klinklom et al., 2013). Impregnation method is favorable since it needs a lower amount of reactant and cheaper equipment compared to precipitation method.

CaO from eggshell waste has been applied by several researchers for biodiesel production. However, impregnation of ZnO active metal on CaO from eggshell and its application for transesterification reaction has not been studied yet. Therefore, in this work, synthesis of ZnO/CaO catalyst from eggshell for the transesterification of WCO for biodiesel synthesis was investigated.

MATERIALS AND METHODS

Materials

Materials of the research were sulphuric acid (p.a.), oxalic acid (p.a.), $Zn(NO_3)_2.4H_2O$ (p.a.), potassium hydroxide (p.a.), ethanol (p.a.) and methanol (p.a.), which were purchased from Merck, eggshell waste and used frying oil which were obtained from local small-scale food enterprise, phenolphthalein indicator, and aquadest,

Methods

This work consisted of two steps, i.e. synthesis of ZnO/CaO catalyst from Eggshell waste and application of the catalyst for biodiesel production from used frying oil.

Synthesis of ZnO/CaO Catalyst from Eggshell

The first step is eggshell preparation. Initially, the eggshell was soaked in the water. It was then washed with aquadest for 15-20 minutes to remove the impurities. Subsequently, the eggshell was dried in the oven at the temperature of 100°C for 1 hour. The dried eggshell was then crushed and sieved to obtain the powder size of 35 mesh. The next process was calcination. The 100 gram of eggshell powder was calcinated in the furnace at the temperature of 900°C for 6 hours to obtain activated CaO catalyst powder.

To synthesize ZnO/CaO catalyst, primarily the $Zn(NO_3)_2.4H_2O$ solution was made by dissolving a certain amount of $Zn(NO_3)_2 4H_2O$ into 25 mL aquabidest in a 100 mL beaker glass. The mixture was stirred until it became homogeneous. The homogeneous $Zn(NO_3)_2.4H_2O$ solution was afterward mixed with a certain amount of CaO catalyst powder in a 100 mL beaker glass. Ratio of ZnO/CaO was varied at 2, 4, 6, and 8%.

The mixing was carried out together with heating process until the mixture turned into slurry. This ZnO/CaO catalyst slurry was then dried in the oven at 100°C until it reached constant weight. Afterwards, dried ZnO/CaO catalyst was activated through calcination process in the furnace at the temperature of 800°C for 6 hours. ZnO/CaO catalysts from eggshell waste were characterized using Scanning Electron Microscope (SEM) to reveal their structureand morphology, Energy Dispersive X-Ray (EDX) to determine the CaO and ZnO/CaO concentration, Fourier-transform infrared spectroscopy (FTIR) to disclose the chemical bond of the materials. The ZnO/CaO catalysts (ZnO/CaO 2%, ZnO/CaO 4%, ZnO/CaO 6%, ZnO/CaO 8%) were subsequently employed for enhancing transesterification of used frying oil for biodiesel production.

Application of ZnO/CaO Catalyst for Biodiesel Production

Production biodiesel from waste cooking oil (WCO) in the presence of ZnO/CaO catalyst from eggshell waste was performed through 2 steps, viz. esterification and transesterification reactions. Esterification reaction was a pre-treatment to reduce the free fatty acid (FFA) content of the oil until it reached the standard quality (below 2% of FFA). Esterification reaction was carried out in a batch reactor equipped with reflux condenser at the temperature of 60°C, molar ratio of oil and methanol of 1:6, and sulfuric acid catalyst of 2.5% w/w oil for 1 hour. The FFA content was then tested using standard acid-base titrimetric method. Esterification reaction was followed by transesterification to produce biodiesel.

Transesterification reaction of WCO with methanol in the presence of ZnO/CaO catalyst from Eggshell waste was accomplished in batch reactor equipped with reflux condenser and hot-plate heater. The reaction was carried out in 2 hours at the fixed reaction temperature of 65°C, molar ratio of oil and methanol of 1:6 and stirring speed of 300 rpm. On the other hand, the 4 different types of ZnO/CaO catalysts (ZnO/CaO 2%, ZnO/CaO 4%, ZnO/CaO 6%, ZnO/CaO 8%), were applied in this reaction.

Biodiesel product of this reaction was separated from the glycerol side-product by decantation process. To enhance the purity, biodiesel product was then washed with aquadest and dried by heating it at 110°C. The concentration of alkyl ester in biodiesel product was analyzed using Gas Chromatography – Mass Spectroscopy (GC-MS). The type of catalyst providing the best alky ester content in biodiesel was applied for transesterification reaction at different catalyst concentration (1, 3, 5, 7 % w/w oil) to determine the best ZnO/CaO catalyst concentration for biodiesel production.

RESULT AND DISCUSSION

ZnO/CaO Catalyst Synthesis

Synthesis of CaO catalyst was conducted through eggshell waste calcination. Calcination process aimed at removing the carbon dioxide content through the decomposition reaction of $CaCO_3$ in the eggshell. In this work, calcination process was performed at the temperature of 900°C for 6 hours. According to (Wei et al., 2009), the reaction occurs during the calcination process can be presented in Eq. (1):

$$CaCO_3 \longrightarrow CaO + CO_2 \qquad (1)$$

Once the calcination process accomplished, white CaO catalyst powder was formed. CaO catalyst was then characterized to reveal its properties.

Energy Dispersive Spectroscopy (EDX) Analysis

Energy Dispersive Spectroscopy (EDX) analysis was brought about to determine the compounds in CaO catalyst from eggshell calcination. The result of EDX analysis is exhibited in Figure 1, and its composition is presented in Table 1.

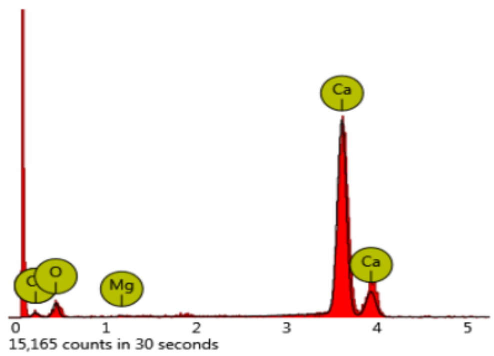

Figure 1. EDX Analysis of Eggshell CaO Catalyst.

Table 1. EDX Analysis of CaO Catalyst from Eggshell.

Element Number	Symbol	Name of the Element	Weight Percentage
20	Ca	Calcium	70.6
8	O	Oxygen	29.1
12	Mg	Magnesium	0.3

Based on the EDX analysis, it was disclosed that the CaO catalyst from eggshell composed of 70.6 % w/w calcium (Ca), 29.1 % w/w oxygen (O), and 0.3 % w/w magnesium (Mg). Magnesium element was detected in the sample since CaO catalyst was synthesized from eggshell which contained 0.84 % $MgCO_3$. This result agrees with the work of Santoso, Kristianto & Setyadi (2013), stating that the Ca content yielded by the eggshell calcination was 70.41%. Thus, it can be concluded that the eggshell can be utilized as raw material of CaO catalyst synthesis with Ca content of 70.6 %.

Scanning Electron Microscope (SEM) Analysis

Scanning Electron Microscope (SEM) analysis was aimed at determining the structure and morphology of the eggshell CaO catalyst surface. SEM analysis was conducted by magnification of 10.000 and 20.000 times as depicted in Figure 2.

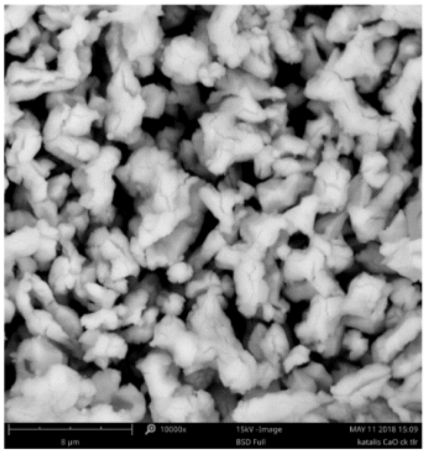

Figure 2. SEM Analysis of Eggshell CaO Catalyst by Magnification of 10.000 Times.

Figure 2 demonstrated that Eggshell CaO catalyst, which has been calcinated at 900°C, exhibited a mutually aggregated structure and different form of particle with the particle. The size of the CaO catalyst was found 3 micrometer. This result was in line with the work of Khalid et al. (2018) and Buasri et al. (2013), which observed that CaO catalyst from eggshell comprised irregular particles form and shape like rods (bars). Irregularity form of particles occurred since the calcination process was performed in the temperature below 1000°C.

To enhance the CaO catalyst activity, the addition of ZnO active metal was essential. The presence of ZnO in CaO catalyst was expected to provide superior catalyst activity and higher yield of biodiesel product. In this work, ZnO active metal addition was accomplished by using various mass percentage of ZnO in CaO catalyst (2%, 4%, 6%, and 8% w/w). the addition of ZnO active metal to CaO resulted in ZnO/ CaO catalyst. To disclose the optimum concentration of ZnO addition in improving the reaction yield, ZnO/CaO catalyst with various concentration of ZnO (2%, 4%, 6%, and 8% w/w) were applied in the biodiesel synthesis from waste cooking oil (WCO).

Application of ZnO/CaO for Biodiesel Synthesis

ZnO/CaO catalyst was applied to enhance the reaction of biodiesel synthesis from waste cooking oil (WCO). Initially, waste cooking oil as feedstock underwent esterification reaction in the presence of sulfuric acid catalyst to reduce the FFA content until it is less than 2%. Subsequently, transesterification reactions of WCO employing ZnO/CaO catalyst were performed at the fixed molar ratio of WCO to methanol of 1:6, temperature of 65°C, reaction time of 2 hours, and stirring speed of 300 rpm. The effects of ZnO concentration in ZnO/CaO catalyst and catalyst concentration were investigated in this work.

Effect of ZnO concentration in ZnO/CaO Catalyst on Biodiesel Yield

ZnO/CaO catalyst with various concentrations of ZnO in CaO (2%, 4%, 6%, and 8% w/w), which were so-called ZnO/CaO 2%, ZnO/CaO 4%, ZnO/CaO 6%, ZnO/CaO 8%, were applied in the biodiesel synthesis from WCO with the catalyst amount of 5% w/w catalyst/ WCO. As the control, the result was compared with the biodiesel yield obtained by using solely CaO Catalyst (Santoso et al., 2013). Biodiesel yield of each transesterification reaction was analyzed using GC-MS as presented in Table 2.

Table 2. Biodiesel Yielded by Various Types of ZnO/CaO Catalysts.

Catalyst	Biodiesel Yield, %
CaO	95.89
ZnO/CaO 2%	97.50
ZnO/CaO 4%	97.76
ZnO/CaO 6%	97.79
ZnO/CaO 8%	97.63

Table 2 shown that biodiesel yield from the transesterification reaction catalyzed by ZnO/CaO is higher than that achieved in the presence of CaO catalyst. It is due to the fact that the impregnation of ZnO on CaO increased the basicity property of the catalyst, leading to the higher catalyst activity. It consequently enhanced biodiesel yielded in the alkaline-catalyzed transesterification reaction. This phenomena agrees with the theory stating that catalyst activity is equal with the strength of alkaline catalyst (Lee, Park & Lee, 2009). The other factor affecting the yield of biodiesel is particle size of the catalyst. ZnO/CaO catalyst has small size of particle, providing higher surface area for reaction

(Zhang et al., 2019). It accordingly improves the reaction conversion and yield. Based on the GC-MS analysis, it was demonstrated that ZnO/CaO 6% exhibited the highest biodiesel yield of 97.79%. Therefore, this type of ZnO/CaO catalyst was then applied for the transesterification reaction at various catalyst amounts.

Fatty acid compositions of biodiesel obtained in the presence of ZnO/CaO 6% were detected using GC-MS as shown in Figure 3 and Table 3.

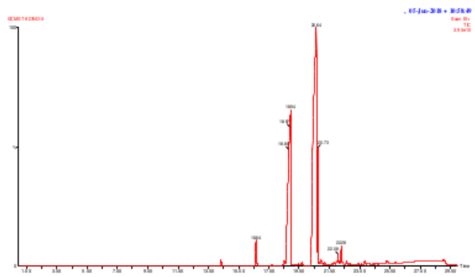

Figure 3. Biodiesel from WCO Chromatogram.

Table 3. Fatty Acid Composition of WCO Biodiesel Synthesized using ZnO/CaO 6% Catalysts.

Name of Fatty Acid	Retention Time	% Area
Dodecanoic acid, methyl ester	14.354	0.276
Methyl tetradecanoate	16.645	1.919
Hexadecanoic acid, methyl ester	18.936	32.184
9-Octadecenoic acid, methyl ester, (E)-	20.636	57.060
Octadecanoic acid, methyl ester	20.731	6.595
trans-13-Octadecenoic acid	20.951	0.288
Oxiraneoctanoic acid, 3-octyl-, methyl ester, cis-	21.967	0.318
cis-11-Eicosenoic acid, methyl ester	22.082	0.463
Eicosanoic acid, methyl ester	22.282	0.897

Table 3 shown that the most dominant compounds in WCO biodiesel was 9-Octadecenoic acid (oleic acid) and hexadecanoic acid (palmitic acid). This composition was in agreement with the findings of Anisah et al. (2018) in their work on biodiesel synthesis using used frying oil raw material.

Effect of ZnO/CaO 6% Catalyst Amount on the Biodiesel Yield

ZnO/CaO 6% catalyst was found as the best catalyst among the other types of ZnO/CaO catalyst. To ascertain the best amount of catalyst for biodiesel synthesis, various amounts of ZnO/CaO 6% (1%, 3%, 5% and 7% w/w catalyst/ WCO) were applied in the WCO transesterification reaction. The GC-MS analysis on biodiesel yields was shown in Table 4. It was found that the highest biodiesel yield was provided by employing ZnO/CaO 6% catalyst with the amount of 3% w/w. The higher amount catalyst shown the lower yield of biodiesel.

Table 4. Biodiesel Yielded by Various Amount of ZnO/CaO 6% Catalysts.

Amount of Catalyst, % w/w catalyst/ WCO	Biodiesel Yield (%)
1	97.625
3	97.804
5	97.793
7	97.014

Characterization of ZnO/CaO 6% Catalyst

SEM-EDX Analysis of ZnO/CaO 6% Catalyst

Based on the experiment, ZnO/CaO 6% catalyst brought about the highest biodiesel yield. To disclose the structure and morphology of catalyst surface, SEM-EDX analysis on ZnO/CaO 6% catalyst has been accomplished. SEM-EDX analysis could also reveal the composition and concentration of each compound comprised in the catalyst. The result of SEM analysis on ZnO/CaO 6% catalyst is presented in Figure 4.

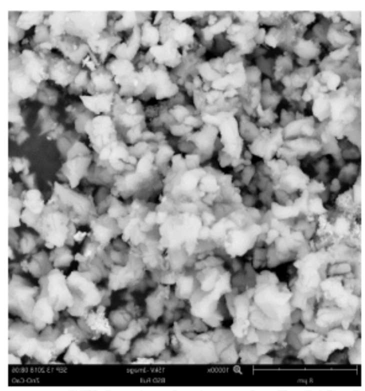

Figure 4. SEM Analysis of ZnO/CaO 6% Catalyst by Magnification of 10.000 Times.

SEM analysis in Figure 4 shown that ZnO/CaO 6% catalyst had a smaller structure compared to the CaO catalyst from eggshell which is presented in Figure 2. Moreover, ZnO/CaO 6% also demonstrated a denser and more solid structure than eggshell CaO. The size of ZnO/CaO 6% is found more various due to the impregnation of ZnO active metal on the CaO catalyst pores. It can be concluded that the impregnation of ZnO affected the structure of catalyst formed. This evidence is in line with the work of Yulianti *et al.* (2014), which found that the combination of ZnO and CaO exhibited uneven structure, and the catalyst shown solid form with different size.

To verify the presence of ZnO impregnated in the CaO catalyst, EDX analysis was conducted. EDX analysis aims at detecting the compounds comprised in the catalyst and determining the concentration of each compound. This can show whether CaO catalyst has been successfully impregnated by ZnO. EDX analysis is displayed in Figure 5 and Table 5.

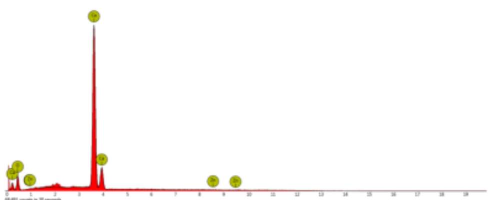

Figure 5. EDX Analysis of ZnO/CaO 6% Catalyst.

Table 5. EDX Analysis of ZnO/CaO 6% Catalyst.

Element Number	Symbol	Name of the Element	Weight Percentage
20	Ca	Calcium	66.8
8	O	Oxygen	32.5
30	Zn	Zinc	0.7

EDX analysis shown that ZnO/CaO 6% consists of calcium, oxygen, and zinc with composition of 66.8%, 32.5%, and 0.7%, respectively. It proved that ZnO active metal has been effectively impregnated in CaO catalyst. Concentration of Zn detected was also comparable with the concentration of ZnO impregnation in CaO.

FTIR Analysis of ZnO/CaO 6% Catalyst

FTIR analysis aims to reveal the infrared spectra pattern of the ZnO/CaO 6% catalyst, and identify the chemical bond of a compound at the certain wave length. The result of FTIR analysis was depicted in Figure 6 and Table 6.

FTIR analysis as shown in Figure 6 indicated the FTIR spectrum of ZnO/CaO 6% catalyst on the wavelength of 4000-400 cm-1. The infrared spectrum of the catalyst sample denoted the existence of ribbon on 3642.97 cm-1. It revealed that the O-H (hydroxyl) functional group has been formed, implying the formation of alkaline site in the ZnO/CaO 6% catalyst. Besides, carbonyl group was also formed on 1468.91 cm-1 wavelength. The addition of ZnO active metal resulted in the presence of peak of spectra on the wavelength of 550.35 cm-1. It indicated that ZnO active metal has been effectively impregnated in the CaO catalyst, it therefore affected the structure and characteristic of the catalyst. FTIR analysis on ZnO/CaO 6% was in accordance with the result of the work performed by Yulianti et al. (2014), uttering that there are several functional groups found in the ZnO /CaO catalysts such as hydroxyl, carbonyl and ZnO groups. The summary of the FTIR interpretation was shown in Table 6.

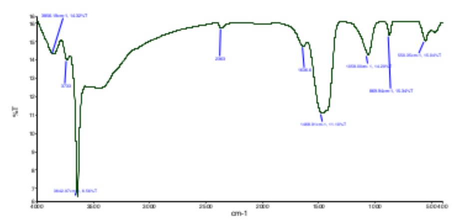

Figure 6. FTIR Analysis of ZnO/CaO 6% Catalyst.

Table 6. Interpretation of FTIR Analysis on ZnO/CaO 6% Catalysts.

X Axis	Y Axis	Functional Group
3642.97	6.56	Alcohol (OH)
1468.91	11.1	Nitro (-C-NO_2)
1058	14.29	Ester (>CO)
869.94	15.34	Alkene (CH)
550.35	15.04	Others (-$(CH_2)_n$)

CONCLUSION

The addition of ZnO active metal on CaO catalyst could significantly enhance the biodiesel yield on the alkaline-catalyzed transesterification reaction of WCO. The addition of 6% ZnO active metal on CaO, forming ZnO/CaO 6% catalyst, has demonstrated the best enhancement of biodiesel yield. The optimum amount of ZnO/CaO 6% catalyst employed in the reaction was found 3% w/w catalyst/WCO. The analysis using SEM-EDX has shown that ZnO/CaO 6% catalyst exhibiting a denser and irregular form of catalyst morphology.

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