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RESEARCH ARTICLE

Modified ZnO to Indonesia limestone as heterogeneous catalysts for biodiesel production from *Reutealis trisperma* oil

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Graphical abstract

Abstract

The Bukit Jaddih Madura limestone has been modified as a heterogeneous catalyst in the transesterification reaction for biodiesel production. Limestone has been calcined at 900°C for 3 hours (CL) and it has been modified with ZnO using a wet impregnation method (ZnO/CL) to improve its catalytic activity. Catalysts were characterized by infrared spectroscopy (FTIR), X-ray diffraction (XRD), SEM-EDX, and basicity test. FTIR analysis showed that BKK and ZnO/CL were easily hydrated and carbonated. It was determined that CL and ZnO/CL catalysts contained CaO, MgO, and a little Ca(OH)2 from the XRD analysis. Next, from SEM analysis, we are able to observe that CL and ZnO/CL had different morphologies and sizes. Basicity test showed base strength of CL was 15,0 <H_ <18,4 and ZnO/CL was 8,2 <H_ <15,0. The catalyst activity on the transesterification reaction of *Reutealis trisperma* oil with methanol showed that biodiesel yield increased with increasing ZnO concentration.

Keywords: Limestone, ZnO/CL, *Reutealis trisperma* oil, biodiesel

INTRODUCTION

Currently, energy needs have increased which is in line with the rapid development in technological advancement. However, seeing as the supply of energy sources from fossil fuels is limited, more researches are being conducted in developing alternative energy sources that are renewable and environmentally friendly (Demirbas, 2007). One of the promising renewable energy sources is biodiesel, alternative diesel fuel with a good profile of combustion emissions and produces less carbon monoxide, sulfur dioxide and non-combustible hydrocarbons than diesel (Borges and Diaz, 2012). Biodiesel also has more oxygen in the complete combustion process, biodegradable, higher flash points, and contains lubricants (Gebremariam and Marchetti, 2018).

Chemically, biodiesel consists of mono-alkyl esters of long chain fatty acids derived from vegetable oils or animal fats. Some vegetable oils such as canola oil, palm oil, palm kernel, sunflower, and coconut have been studied as raw materials for biodiesel production (Math et al., 2010). The disadvantages of biodiesel production from raw materials for vegetable oils are the high price of oil and causing an imbalance between the use of oil as food and fuel. These problems can be reduced by the use of non-edible oil. Some non-edible oils used for biodiesel production such as jatropha oil (Jatropha curcas L.), Karanja (Pongamia pinnata L.), Castor (Ricinus communis), Palanga (Calophyllum inophyllum L.), Croton of megalocarpus, and Moringa oleifera. There is also new oil as the base material for biodiesel production which is interesting to study, namely *Reutealis trisperma* oil (Atabani et al., 2013).

Biodiesel production generally uses homogeneous catalysis, because of fast reaction, high conversion, and a small amount of catalyst (Manuale et al., 2015). However, it causes saponification, difficult to separate and cannot be reused (Talukder et al., 2010).

Therefore, heterogeneous acid and base catalysts are used for transesterification reactions. Heterogeneous acid catalysts can be used for low-quality oil raw materials because they are less sensitive to FFA (Free Fatty acid). However, it requires a higher reaction temperature which increases energy consumption (Fonseca et al., 2019). On the other hand, heterogeneous base catalysts have the advantages of being easily separated, reusability, form little soap, no corrosion, can be used under high temperatures and pressures, high activity, high selectivity, and longer lifetime (Marinkovic et al., 2016).

Calcium oxide (CaO) is used as a heterogeneous base catalyst because it has high basicity, high activity, non-toxic, low solubility in methanol, long lifetime, and requires moderate reaction (Roschat et al., 2016). CaO can be obtained from commercial sources such as calcium acetate, carbonate, hydroxide, nitrate, and oxalate (Cho et al., 2009). In addition, CaO can be obtained from natural sources such as egg shells (Borah et al., 2019;), snail shells (Kaewdang et al., 2017), fish bones (Widiarti et al., 2016), and limestone (Suprapto et al., 2016). Although various precursors are available to obtain CaO catalysts, their catalytic activities differ depends on precursors which have different decomposition. The catalytic activity of CaO can decrease when the oil with high FFA content, caused by non-activation of the active site on the surface of the catalyst and forming soap (Dias et al., 2013). One way to increase catalytic activity is by modification with transition metal oxides that have different acidity or basicity. Kumar and Ali (2013) impregnated transition metal ions (Mn, Fe, Co, Cu, Ni, Zn, and Cd) on the surface of the CaO catalyst to increase catalyst activity. Zn/CaO catalyst showed activity (99% yield) because it had high alkalinity and better resistance to moisture than CaO. ZnO is an amphoteric oxide that has an acidic and basic side. This is suitable for transesterification of oil with high FFA content because occur transesterification and esterification simultaneously thereby increasing yield (Navas et al., 2018).

In this paper, we investigated the modification of ZnO to Indonesian limestone as a heterogeneous catalyst in the transesterification reaction of *Reutealis trisperma* oil. The modification is expected to increase its catalyticactivity.

EXPERIMENTAL

Materials and catalysts preparation

In this work, *Reutealis trisperma* oil was supplied by PT. Kemiri Sunan Drajat as a source of oil, methanol (Merck, 98%) as a reactant in the esterification and transesterification reactions, H2SO4 (Merck, 98%) as a catalyst in the esterification reaction, zinc acetate (Merck, 99%), n-hexane (Merck, 99%), distilled water, phenolphthalein (H_=8,2), 2,4-dinitrianiline (H_=15,0), 4-nitroaniline (H_=18,4), benzoic acid, and limestone from Madura island, Indonesia. The limestone was dried overnight in an oven at 110°C and then crushed and sieved. Next, it was calcined at 900°C in air for 3 h. The powder was labeled calcined limestone (CL).

The ZnO/CL was prepared by wet impregnation method. CL was suspended in deionized water. To this, an aqueous solution of zinc acetate of the desired concentration was added. The concentration of the zinc acetate was varied to obtain a 1−7 wt % ZnO concentration in CL. The slurry thus obtained was stirred for 3 h, air-dried, calcined in a muffle furnace at 550 °C for 12 h, and calcined at 800 °C for 3 h for activation beforeused.

Transesterification procedure

Transesterification reaction of *Reutealis trisperma* oil was carried out in three bottom batch reactors round neck equipped volume 100 mL with condenser reflux, magnetic stirrer with a speed of 300 rpm, and thermometer. Previously the oil was treated with esterification to reduce its FFA content using a sulfuric acid catalyst. The esterification was carryout out with methanol oil mass ratio 3:1 and 3% w catalyst/oil was preheated at 65 ° C and added to 10 g of oil, that also heated. The transesterification reaction was carried out with the methanol to oil molar ratio of 15: 1 and catalyst loading amount 5% wt to the oil. Biodiesel was analyzed by gas chromatography with a GC-2010 Plus Tracera, equipped with an FID detector and a capillary column (Select Biodiesel for glycerides ultimetal 10 m x 0.32 mm x 0.10µm).

RESULTS AND DISCUSSION

Catalysts characterization

Limestone analyzed by thermogravimetric analysis (TGA) showed a total weight loss of about 7% by weight at 900 °C which indicated the decomposition of CaCO3 to CaO (Fig. 1). Weight loss is caused by the process of releasing chemical bonds from water molecules contained in limestone and CO2 as a result of the reaction (Granados et al., 2007). Modification of limestone shows three stages of decomposition, they are around 300 °C decomposition of Zn(OH)2 to ZnO (weight loss of 1%), 450 °C decomposition of Mg(OH)2 to MgO (weight loss of 5%), and 600-750 °C decomposition of Ca(OH)2 to CaO (weight loss 9.4%). This result suitable with the research of Navas et al., (2018) who studied the decomposition of carbonates from Ca, Mg and Zn from their oxides. Modification of limestone causes an amount of H2O and CO2 released in the ZnCO3 to ZnO decomposition process, thus facilitating the decomposition of CaCO3 to CaO. A number of pores formed through ZnCO3 decomposition will facilitate the transfer of heat to the interior of particles and produce gas diffusion. Therefore, the temperature needed to form CaO is lower (Ngamcharussrivichai et al., 2008).

The XRD pattern of limestone, CL and ZnO/CL are presented in Fig. 2. As seen in the XRD spectra all of samples, the characteristic peaks of both CaO (2θ=32.2°,37.3°,53.8°,64.1°and 67.3°:JCPDS file no.77-2376) and MgO(2θ=36.4°:42.9 °and 62.2°;JCPDS file no. 78-0430) were present, indicating that after calcination at 900 °C for 3 hours showed that the limestone was completely decomposed into CaO (Roschat et al., 2016) and peak of MgO causes in the limestone of a large component of MgCO3 which is not lost during washing or calcination.

The peak character of MgO is similar to the results of the analysis with XRF which shows limestone precursors containing Ca and Mg by 86% and 13%, respectively. ZnO modification does not affect the character because no ZnO characteristic peaks at 2θ = 31.8°, 34.46°, 36.3°, 47.6°, 56.6°, 62.9°, 66.37°and 69.1°(JCPDS891397) for all modifications. However, crystallinity of CL decreases with the addition of ZnO concentration. It was caused by ZnO which has been distributed evenly on the surface of CL and can be attributed to the formation of ZnO/CaO where Zn2+ has replaced the Ca2+ site. Zn2+ concentration further leads to the formation of hexagonal phase ZnO in Zn/CaO and CaO-ZnO (Kumar and Ali, 2013).

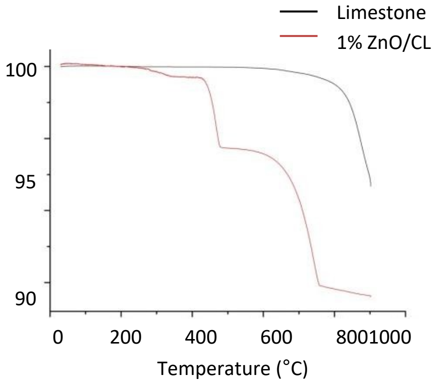

Fig. 1 Thermogram of Limestone and 1% ZnO/CL

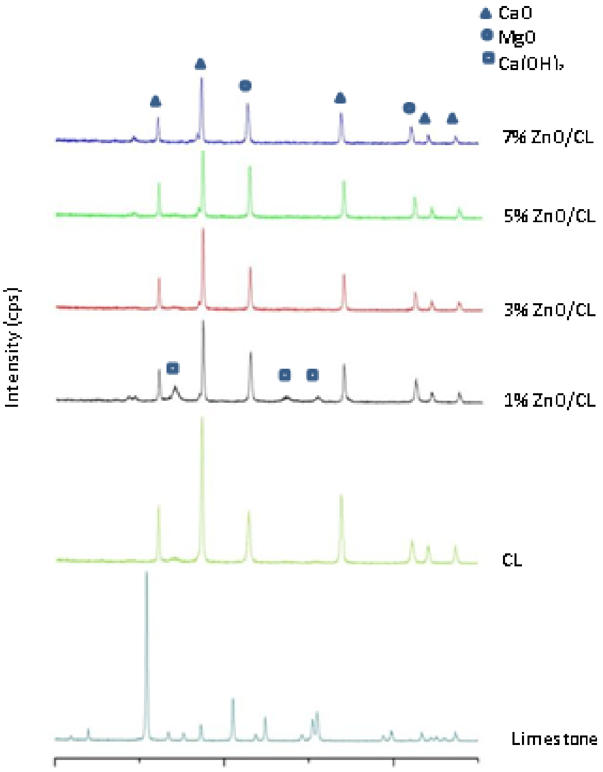

Fig. 2 Comparative powder XRD patterns of limestone, CL and ZnO/CL with ZnO concentration (1-7%)

From Fig. 3, the spectrogram pattern of limestone, CL, and ZnO/CL indicate stretching of O-H from water and Ca(OH)2 in the band widened around 3400-3500 cm-1 and band tapered at 3600 cm-1 (Granados et al., 2007). Major adsorption at 1400 cm-1 and minor adsorption at 850 and 700 cm-1 shows the presence of asymmetric sthrecing vibrations, vibrations in and out of the plane of the CO 2- group (Maneerung et al., 2016). The presence of a vibration peak at wave number 405 cm-1 shows the vibration of Ca-O (Alba-rubio et al., 2016). These results indicate that CaO synthesized from limestone has not obtained pure CaO compounds because the synthesized CaO has been contaminated with CO2 and H2O from the air. CaO is an unstable compound, easily reacts with air and water vapor to form Ca(OH)2 and CaCO3 which is likely to be obtained during preparation before CaO is analyzed by FTIR (Granados et al., 2007). This is different from the XRD results which shows that CaO obtained was pure even though it contained MgO. However, limestone contains a mixture of Ca(OH)2 and CaCO3 which shows lower catalytic activity (Granados et al., 2007). Therefore, calcination is very important to turn limestone into CaO (Kouzu et al., 2008). Fig. 3 also shows that ZnO has not been seen in FTIR spectra for all modified samples because the amount of ZnO is reduced slightly so that ZnO has not been completely distributed on the surface of CaO. These results are the same as the XRD analysis with no ZnO characteristic peaks.

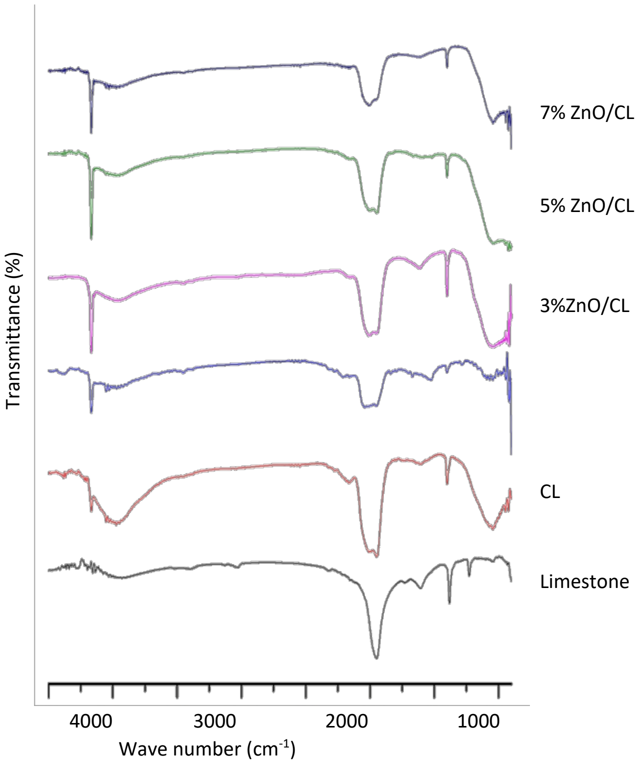

Fig. 3 IR spectra of natural calcite, CaO.MgO and ZnO/CaO.MgO with a varying ZnO concentration (1-7%).

Fig. 4 is the result of SEM analysis which shows that the CL catalyst generally looks homogeneous as large particles of various sizes which are shaped like cubic with a non-smooth surface. This is in accordance with a research reported by Yoosuk et al. (2010) that calcite has large particles shaped like a cubic with a smooth plane. CL particles have a larger and uniform size compared to ZnO/CL particles. In ZnO/CL, a spherical particle covers all surfaces. These ball particles are active sites of ZnO that have been attached to the CL surface (Borah et al., 2019). Ball particle size increases with increasing ZnO concentration. At 5% ZnO/CL, large spherical particles have covered the entire surface of the catalyst. In addition, an increase in ZnO concentration changes the morphological shape of the sample from cubic to hexagonal (Joshi et al., 2016)

The EDX analysis results show that the sample consisted of elements of Ca, Mg, Zn, O, and C (Table 1). This identifies that no other element formed during the synthesis process. The content of Ca and Mg elements decreases with increasing ZnO concentration, otherwise the amount of Zn element decreases. It is because Zn^{2+} has replaced the Ca^{2+} and Mg^{2+} sites. The contents of Ca and Mg are almost the same, this is in accordance with the XRD results which show a high peak of MgO. The presence of element C identifies that this sample has undergone a decarbonation process. The content of C is getting bigger as a result of increasing contact time with air. CaO catalysts at room temperature are very easily hydrated and carbonated (Granados et al., 2007).

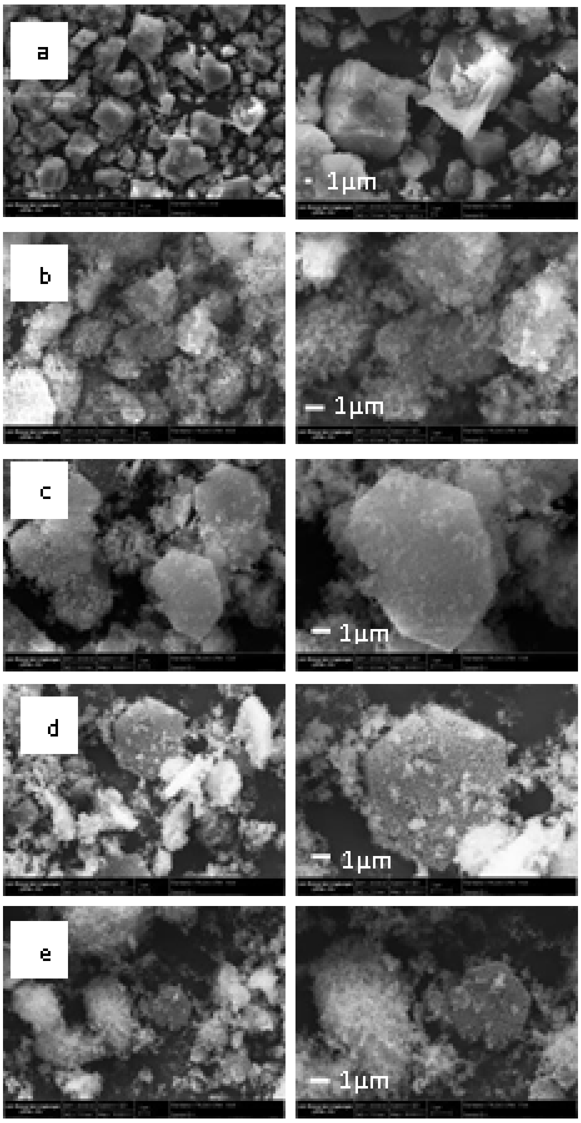

Fig. 4 SEM of CL (a), 1% ZnO/CL (b), 3% ZnO/CL (c), 5% ZnO/CL (d) and 7% ZnO/CL (e) catalyst.

Table 1 The composition of elements in CL and ZnO/CL.

Sample	Elemental content (%weight)				
	Ca	Mg	Zn	O	C
CL	15,70	11,17	0	53,29	19,84
1% ZnO/CL	13,56	12,89	0,75	57,54	15,26
3% ZnO/CL	14,32	12,58	1,50	54,56	17,04
5% ZnO/CL	9,85	8,64	1,75	52,23	27,54
7% ZnO/CL	8,86	9,12	2,67	49,84	29,51

The base strength of the catalyst can be analyzed qualitatively using the Hammett indicator method while quantitatively using the titration method with benzoic acid (Table 2). The results show that CL has basicity of 15,0<H_<18,4 and ZnO/CL has basicity of 8,2<H_<15,0. The addition of ZnO concentration decreases the level of basicity. It is because ZnO is an amphoteric oxide which has both acid and base sides (Navas et al., 2018). In addition, calcination temperature also affects the base strength of a catalyst. Kumar and Ali (2013) reported that the calcination process from 350 to 550 °C increased basicity from 11,1<H_<15,0 to 15,0<H_<18,4. This increase in basicity is due to the formation of lewis base (−O−) on the surface of Zn/CaO. The calcination process at temperatures higher than 750 °C will reduce basicity from 15,0<H_<18,4 to 11,1<H_<15,0.

Table 2 Base strengths of CL and ZnO / CL by titration method.

Sample	Bacisity (mmol/gr)
CL	15,9140
1% ZnO/CL	13,6036
3% ZnO/CL	12,4856
5% ZnO/CL	10,5048
7% ZnO/CL	9,9796

Effects of catalysts in transesterification

Fig. 5 shows the effect of the ZnO concenration on the FAME yield during transesterification reaction of *Reutealis trisperma* oil and methanol at a constant methanol:oil molar ratio of 15:1, 65 °C and catalyst loading 5%. The results showed that the yield of methyl esters without ZnO modification was very low at 9.27%. It is because CL contains almost the same Ca and Mg (EDX analysis). High MgO content in CaO catalyst can reduce catalytic activity because MgO has lower base strength than CaO (Wang et al., 2009). Modification of CL with ZnO can increase yields by 52.34%. It shows that ZnO plays an important role in the transesterification reaction process.

ZnO is an amphoteric oxide which has both acid and base sides. It is suitable for the transesterification reaction of vegetable oils with high FFA content because it allows transesterification and esterification reactions to occur simultaneously (Navas et al., 2018). Kumar and Ali (2013) reported that Zn/CaO catalysts have well moisture resistance and FFA.

Optimization of the amount of catalyst weight is determined because a low amount of catalyst will not be enough to complete the reaction whereas an increase in catalyst weight is higher due to bad mixing between the solid phase (i.e. catalyst) with the liquid phase (i.e. methanol and oil) and the system viscosity becomes higher so as to reduce yield (Sudsakorn et al., 2017). In addition, the amount of weight of the catalyst affects the active side of CaO which produces anion methoxide from methanol. As a reactive nucleophilic molecule, anion methoxide attacks electrophilic carbon carbonyl in triglycerides to produce biodiesel (Liu et al., 2008).

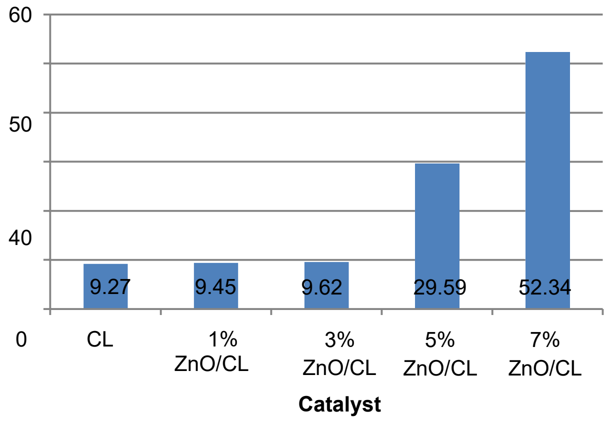

Fig. 5 Influence of catalyst modification (catalyst weight 5%, molar ratio of oil to methanol 1:15, reaction temperature 65°C and reaction time 3 h).

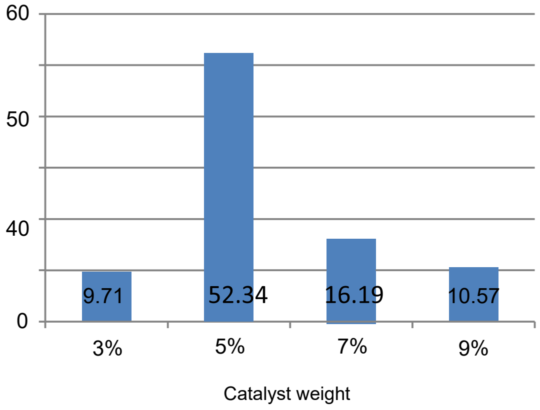

Fig. 6 Influence of catalyst weight (7% ZnO/CL, oil molar ratio to methanol 1:15, reaction temperature 65°C and reaction time 3 h)

CONCLUSION

Bukit Jaddih Madura limestone through the process of thermal decomposition with high temperature calcination can be used as a heterogeneous catalyst for the production of biodiesel from Reutealis trisperm oil. Modification of limestone with ZnO (ZnO/CL) using the wet impregnation method aimed to increase the catalytic activity. XRD analysis shows that CL and ZnO/CL contain CaO, MgO and a little Ca(OH)2. CaO is an unstable compound, easily reacts with air and water vapor to form Ca(OH)2 and CaCO3 which appear in FTIR spectra. SEM analysis shows BKK and ZnO/CL catalysts have different morphology and size. Basicity test shows the base strength of the BKK catalyst is 15,0 <H_ <18,4 while the ZnO/CL catalyst is 8,2 <H_ <15,0. The addition of ZnO concentration decreases crystallinity, decreases basicity and increases biodiesel yield. Biodiesel production of 52.34% was obtained at a reaction temperature of 65°C for 3 hours, a molar ratio of methanol to oil of 1:15 and a weight of 5% catalyst using a 7% ZnO/CLcatalyst.

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