The Potential Use of Non-Edible Kesambi oil for Producing Biodiesel Using Zinc Oxide Based Composite Catalyst: A Comparison Study

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Abstract. **The non edible kesambi (*Scheleichera oleosa L*) oil which is coded by SO has been converted into biodiesel using composites catalyst. There are three types of zinc oxide based composite catalysts that have been used to convert that oil, namely $ZnO/\gamma-Al_2O_3$, $ZnO-CuO/\gamma-Al_2O_3$ and $ZnO/MWCNTs$ which are coded by ZA, CZA and ZCNTs, respectively. The aim of this study is comparing the activities of those three catalysts in converting kesambi oil (SO) into biodiesel. The effect of various reaction variables including the amount of catalyst (%w/w of oil) and reaction time (h) on yield (%) of Kesambi oil's biodiesel (BSO) were investigated. The results confirmed that SO potentially can be converted into biodiesel using those three of catalyst (ZA, CZA and ZCNTs). The catalytic activity test carried out through the transesterification reaction of SO with methanol showed that the ZA catalyst has the highest activity followed by CZA and ZCNTs.**

Keywords - comparison study, composite catalyst, non edible kesambi oil, zinc oxide

I. INTRODUCTION

Currently the world's energy needs are largely dependent on non-renewable fossil fuels which are the main source of CO2 greenhouse gas emissions which have a major impact on global warming [1]-[4]. Renewable energy systems appear to be a very promising solution to overcome these problems in order to ensure a secure world energy supply and a friendly environment [4]. There are many reasons for interest in bio-fuels including: 1) mitigating climate change and reducing dependence on energy imports, 2) a global commitment to reduce greenhouse gas (GGE) emissions, 3) to substitute conventional fossil fuels, 4) local resource development (support for small-scale producers, welfare and value added), 5) significant cost savings in the transportation sector [5].

Renewable energy sources such as solar energy, wind energy, hydro energy, wave energy, energy from biomass and waste have been successfully developed and used by various countries

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to replace fossil fuels [5]. However, according to a statement from the International Energy Agency (IEA), only energy that comes from renewable sources and waste has a high potential compared to other renewable sources. In Indonesia, the use of fuel oil in the transportation sector from 2003 to 2025 is estimated to increase by an average of about 6% per year. Diesel consumption ranks second after gasoline, which is 44%. Considering these reason related to renewable energy sources, biodiesel is a promising alternative for replacing petroleum-based diesel fuel[2] [4]. Biodiesel consists of vegetable oil methyl ester (RCOOCH3), in which the hydrocarbon triglyceride chains of crude vegetable oil are chemically converted into fatty acid esters through a transesterification reaction. This reaction is also often referred to as methanolysis. Biodiesel has several advantages over petroleum diesel fuel include: derived from renewable resources, it has a cetane number higher than diesel oil, it doesn't require modification on the diesel engine, it is a non-toxic and biodegradable fuel, low gas emission (CO, CO2 and NOx) [3][6].

The biggest challenge in producing biodiesel is related to the use of raw materials and their conversion or processing techniques. Today, the most common and simplest way for the production of biodiesels is through the transesterification process of vegetable oils on a homogeneous sodium hydroxide base catalyst. In Indonesia, vegetable oils that are commonly used are refined vegetable oils, especially palm oil. It is known, the cost of raw materials contributes between 70-90% of the total cost of biodiesel production [1][2][6][7]. The using of refined edible oil as the raw material for producing biodiesel is not an attractive idea. The fact, it is being expensive it can also compete with food industry's sector, so it can interfere with national food stability [2][5][7]

There are two ways to overcome these challenges, namely by using alternative raw materials as a substitute for refined vegetable oil and replacing homogeneous catalysts with heterogeneous catalysts. Non-edible oil (NEOs) such as Jatropha [8][9], castor oil [9], Kapok seed oil [3], Kesambi oil [2] and low-grade oil (LGO) such as used cooking oil [7] [10], palm fatty acid distillate (PFAD) [1], and municipal waste mud [1] [11] seemly to be a promising alternative feedstock for producing of biodiesel. Among those NEOs, kesambi oil has the most potential because it is cheaper than edible oil and its abundance, considering that currently kesambi oil is being cultivated by the Indonesian forestry ministry [2].

Heterogeneous catalysts are believed to be able to overcome the weaknesses of homogeneous catalysts that have been used to

produce biodiesel on an industrial scale. The advantages of heterogeneous catalysts include being easily separated from products, regenerable (reused), not producing soap, and not requiring a washing process, produce minimum liquid waste and lower energy consumption [13]. As a result, total production costs can be reduced, and the price of biodiesel can compete with diesel oil prices. Likewise homogeneous catalysts, there are 2 types of heterogeneous catalysts, namely basic and acid heterogeneous catalysts. The alkaline catalysts are very suitable for transesterifying high purity vegetable oils with low FFA content because of their higher activity than that of solid acid catalysts. However, heterogeneous base catalysts are more intolerant to FFA and water content, which is the favorable FFA content <= 2% (meanwhile for homogeneous base catalyst FFA content of feedstock should be =<0.5%). On contrary, solid acid catalysts are much more tolerant to both of them. Therefore, it can convert LGO with a high content of FFA and water into biodiesel with good and stable performance.

Considering kesambi oil is included in a non-edible oil which in fact has lower quality than refined edible oil, especially its high free fatty acid (FFA) and water content, therefore solid acid catalysts is more appropriate to be used in converting it into biodiesel. It's possible because the insensitivity of solid acid catalysts to FFA content is higher than that of solid base catalysts. They have the potential to catalyze both esterification and transesterification process simultaneously [2] [4]. Therefore, the application of heterogeneous acid catalysts in biodiesel production needs to be studied to overcome the problems caused by homogeneous acid catalysts.

Research related to acid solid catalysts has been carried out by many previous researchers. There are different types of them used in producing biodiesel through esterification and transesterification processes include tungsten oxides, sulphonated zirconia (SZ) [14], ZnO/γ-Alumina [2], ZnO-CuO/ γ-Alumina [3][13], sulphonated multi-walled Carbon Nanotubes [1]. Asri et al. also have been prepared multi-walled carbon anotubes supported zinc oxide catalyst for transesterification of kesambi oil (Schleichera oleosa) [15]. Meanwhile, heterogeneous acid catalyst titanium-doped amorphous zirconia has been observed for transesterifying rapeseed oil found a 65% biodiesel yield from rapeseed with 40:1 methanol to oil ratio [16]. Due to the presence of acid sites with different strengths of Bronsted or Lewis acidity in heterogeneous acid catalysts, industrially it has been considered more effective in a contrast to homogeneous acid catalysts. Zinc oxide, titanium oxide, tungstated zirconia and sulphated zirconia are known as solid acid catalysts having enough acid site strength and are particularly preferable as catalysts during the transesterification reaction for biodiesel production [17]. From these references, it can be seen that the catalyst activity varies greatly depending on the type of catalyst, the synthetic method used, the kinds of promoters and supports, the type of oil used as raw material, and the transesterification parameters. Therefore, it is necessary to further develop an appropriate solid acid catalyst to transesterify non-edible oils kesambi oil (SO). Moreover, studies on the application of solid acid catalysts to convert SO into biodiesel are still very rare.

In this study we focused on studying zinc oxide based solid-acid catalysts with several modifications. There are three type of solid acid catalysts include γ-alumina supported zinc oxide (ZnO/γ-Al$_2$O$_3$), γ-alumina supported zinc oxide-Copper oxide (ZnO-CuO/γ-Al$_2$O$_3$) and multi wall carbon nanotube zinc oxide catalyst (Z/MWCNTs), which coded by ZA, CZA and ZCNTs, respectively. The activities of those synthesized catalysts were compared in the transesterification of kesambi oil (SO) with various parameters, namely the dose of the catalyst, the molar ratio of the catalyst to the SO, the reaction time, and the reaction temperature.

II. PROCEDURE

A. Materials

Analytical grade zinc chloride and cupric sulfate as reactants for synthesizing the precursors of zinc methoxide and cooper methoxide were obtained from Merck, Germany. γ-Al$_2$O$_3$ and MWCNTs as a catalyst support were purchased from Merck, Germany, and Advanced Material Esoterica, China, respectively. Aside from, other analytical grade materials without facing any further treatment such as sodium hydroxide, ammonia, ethanol, and all standard reagents for Gas Chromatography (GC) analysis were also provided by Merck, Germany. Kesambi oil (SO) as non edible oil (NEOs) feedstock was purchased from the local vendor. Meanwhile, commercial grade methanol as a reactant for transesterification process was supplied by local supplier.

B. Methods
Preparation of zinc oxide based solid acid catalyst of ZA, CZA and ZCNTs

ZA catalyst was prepared by precipitation and gel methods. CZA catalyst was synthesized by precipitation, impregnation and gel methods. Meanwhile, ZCNTs was prepared through modified Strober process [18] then followed by precipitation process. The catalysts synthesized in this study were ZA catalyst with 70% ZnO content, catalyst (wt.% To alumina), CZA with 20% ZnO content (wt.% To Alumina) and ZCNTs catalyst with 20% ZNO content (wt. % against MWCNT) [2] [3] [15]. The synthesizing procedure of those three type catalysts of ZA, CZA, and ZCNTs have been described in detail on previous work at [2] [13] [15]. The synthesized catalysts were characterizing by mean Brunauer-Emmett-Teller (BET), XRD, SEM-EDX, and the XRF methods. The composition of catalyst was measured by XRF methods. Meanwhile, to determine the average surface area, morphology, and the presence of crystals on the surface of the catalysts were analyzed using the BET and XRD methods, respectively

Transesterification of SO into BSO

It has been described in the introduction section, that SO is NEOs with high FFA and also other impurities content. Therefore, it should be treated prior transesterification process. The treating process was done to reduce FFA content and its impurities such as gum, water, and unwanted material.. The purification process includes several stages, namely preheating for 30 minutes at a temperature of 100-105 C, further heating for

4 hours at the same temperature to reduce the FFA levels. The final step is the degumming process to remove the gum content in the oil. This treatment procedure has been described in detail in previous studies [2] [13]. After the treatment process is complete, then SO is ready to be processed into biodiesel through a transserification process using 3 types of synthesized zinc oxide-based composite catalysts, namely, ZA, CZA and ZCNTs.

III. RESULT AND DISCUSSION

A. Catalyst characterization

The synthesized catalyst before being used for transesterification must be characterized in advance to determine its properties, including crystallinity, surface area, morphology and composition of the catalyst. As described in the method sub section, the catalyst characteristics were analyzed using the X-RD, BET, SEM-EDX and XRF methods. The results of catalyst characterization have been elaboarted in detail in previous research, namely [2] [13] [15] (Asri et al., 2018), Asri et al., 2020 (ICBB), Asri et al., 2020 UNPAR) for ZA, CZA and ZCNTs catalysts, respectively).

B. The effect of catalysts dosage on yield of BSO (%)

Catalyst has a very important role in a reaction including the transesterication reaction, because the catalyst can reduce the activation energy which in turn can increase the constant rate of the reaction. Arrhenius's law states that the rate of reaction increases with increasing reaction rate constant [19]. Although advances in biodiesel technology are currently experiencing significant developments, the success of the process on an industrial scale still depends on the suitability; effectiveness, and efficiency of catalysts used. It is influencing by many factors including the type of promoter, the type of support, the method of synthesis, and other factors [13]. This is closely related to the amount of catalyst needed in the transesterification process. To investigate the effect of catalysts dosage of on BSO yield the experiment were performed with varied catalyst dosage of 1–7% (wt% to SO) in 1% of range. The other parameters namely the reaction temperature, molar ratio of SO to methanol and reaction time was kept constant at 55 °C, 1:12 and 4 h, respectively. The one-variable-at-a-time (OVAT) approach was using in this study in which one parameter was varying in the experiment, and the others parameters was fixed [13].

The influence of catalyst dosage on yield of BSO is shown on Fig. 1. It is clearly shows that BSO yield significantly increases by increasing the catalyst dosage. It can be ascertained that biodiesel is not formed without the presence of catalyst in the transesterification process (the figure is not shown). On the other hand, in the addition of 1% catalyst (with fixed other parameters), the BSO yield of 30.12% was obtained for the ZA catalyst, while the BSO yield of 22.87 and 26.36% for the other two catalysts CZA and ZCNTs, respectively. This evidence shows that the catalyst has a very important role in accelerating the reaction rate (Levenspiel, 1999). All catalysts show the same pattern, namely the greater the catalyst dose, the greater the yield of SBO up to a certain dose. For ZA catalyst the highest SBO yield of 79.27% was achieved at a dose of 5%. Whereas

for CZA and ZCNTs catalyst the highest BSO yield was 56.66 and 39.55%, which were obtained at doses of 5% and 4%. Conversely, the use of a catalyst that exceeds that dose, the yield of SBO obtained decreases. It was caused the three-phase mixture of SO, methanol, and the heterogeneous catalyst become too viscous so that it interferes with the mixing process which ultimately hinders the mass transfer process between phases.

The maximum SBO yield for ZA and CZA catalysts is obtained for the same catalyst dose of 5%, because it uses the same support, namely γ- alumina, while for ZCNT catalysts the maximum yield is obtained at lower doses, this makes sense because it uses a MWCNTS support, which in fact has a bulk density much lower than γ- alumina. The maximum BSO yield of 79.27% using ZA catalyst with 5% of dosage is lower than that of the previous study using AZO catalyst [2]. However, it should be noted that the AZO catalyst used to transestrify kesambi oil (SOO) with a lower FFA content (<3%) than the FFA SO content used in this case (> 5% 2.9%) (after treatment process). However, it should be noted that AZO catalyst was used to transestrify kesambi oil (SOO) with a lower FFA content (<3%) than the FFA SO content used in this case (> 5% 2.9%) (after treatment process). In addition, the reaction time for ZA catalysts is 2 hours shorter than the reaction time for AZO catalysts. Another researcher [20], studied the transesterification of soybean oil with SO_4^{2-}-ZnO catalyst and their highest yield of 80.1% was achieved at 4 h, and molar ratio of oil; methanol 1:6 , respectively. The result proved that kesambi (SOO) oil has good potential for producing biodiesel using alumina supported zinc oxide (AZO) solid as catalyst.

C. The effect of molar ratio SO to methanol on BSO yieldl

Another factor that is no less important in the process of methanolysislysis triglycerides (TG) into biodiesel is the molar ratio of oil to methanol. Theoretically, the molar ratio of oil to methanol is 1: 3 [2] [6]. Considering that the transesterification reaction is an equilibrium reaction, to shift the reaction towards the product, excess methanol is needed. Practically, the transesterification of LGO with homogeneous catalyst needs the molar ratio of 1:6 [6]. However, on heterogeneous system, much higher molar ratio (within range 1:9–1:42) is needed compared to conventional catalyst [2].
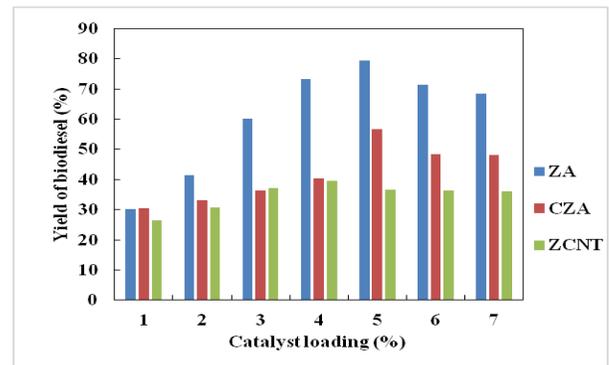

Fig. 1 The influence of catalyst doses (wt % to SO) on BSO Yield (%)

This occurs because heterogeneous systems involve 3 insoluble phases so that mass transfer is being limited. However, the use of too much excess methanol is not worth it, and enhancing the total production cost of biodiesel [2] [6].

In this work the influence of the molar ratio of SO to methanol on the BSO yield was studied by varying the ratio from 1:9 to 1:24 at a reaction temperature of 65 ∘C, a reaction time of 4 h and a catalyst amount of 4%. Figure 2 depicts the influence of the molar ratio of oil to methanol on the yield of BSO. It shows that the molar ratio of SO to methanol significantly affected the yield of biodiesel. It was observed that yield of biodiesel increased along with the increase of molar ratio from 1:9 to 1:12 by using ZA catalyst. The highest yield of 73.25% was achieved at molar ratio 1:12. However, at a molar ratio of 1:15, it was slightly decreased to 73.24% and further gradually decreased to 70.29% at a molar ratio of 1:24. For CZA and ZCNTs catalysts have different performance from ZA catalysts, but the pattern is the same, the BSO yield increases with the increase in the molar ratio of methanol oil to a certain extent.

The highest BSO yield was obtained at the molar ratio of SO: methanol 1:15, namely 56.12 and 39.52 for CZA and ACNTs, respectively. This is possible because the three catalysts have different characteristics. ZA is a catalyst with a single promoter ZnO supported by alumina, while CZA uses a double promoter CuO and ZnO with the same support as ZA. On the other hand, the ZCNTs catalyst uses the same promoter but the supports are different, namely MWCNTs. Therefore, the optimum ratio should be determined to eliminate excess of methanol usage for economic point of views.
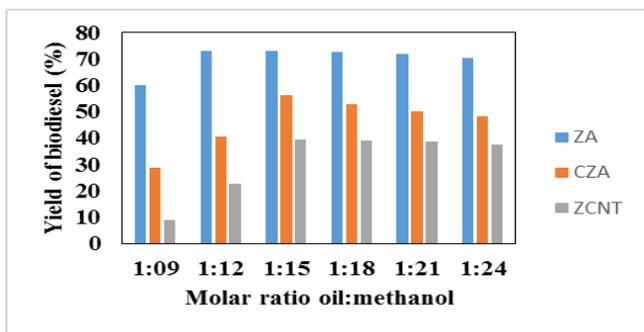

Fig. 2 The influence of molar ratio SO to methanol (wt.% to SO) on BSO yield (%)

D.The effect of reaction time and reaction temperature on BSO (%)

Reaction time and reaction temperature are two parameters that significantly affecting transesterification performance. Theoretically, the reaction temperature is significantly influence the rate of transesterification reaction, because the reaction temperature significantly influenced the rate constant of the transesterification reaction.

According to Arrhenius law the rate constant of reaction increased with the increase of reaction temperature. The higher the rate constant of the reaction, the faster the rate of reaction, because of the reaction rate directly proportional to the rate constant of reaction [5]. The impact of reaction temperature on

yield of biodiesel on transesterification of SO using ZA, CZA and ZCNTs catalysts were studied also using OVAT approach. The reaction temperatures were varied from 55-75 °C with interval of 10 °C under constant stirring speed. Meanwhile, the others conditions such as reaction time, molar ratio of KO to methanol, amount of catalyst were kept constant, at 4 h, 1:12, and 5% (wt. % to KO), respectively.

Similarly, the effect of reaction time was also conducted with the OVAT approach by varying the reaction time from 1-7 h with a range of 1 h. The fixed parameters are reaction temperature, catalyst dosage, and a molar ratio of SO to methanol. Figure 3 shows the effect of time reaction on yield of BSO (%). As expected, BSO yield increase along with the increase of reaction time. At the initial stage (1-3 h) the low yield of BSO was achieved for all types of catalysts. Possibly, due to the time was not enough to well mixing three phases of reactant (SO, methanol and catalyst). However, at 3 h up to 5 h there was a significant increase on yield of BSO. Especially for ZA catalyst it increases is exponentially from 3 to 5 h, followed by stationary at 5 to 7 h). On the other side, a linear increase was found in the CZA catalyst. Meanwhile, for ZCNTs there was not a significant increase in BSO yield by the increasing of reaction tim. The highest yield of 78.26% was achieved at 6 h and 55 ∘C for ZA catalyst.

The effect of reaction temperature on BSO yield was shown in Fig. 4. Its effect is very significant on the yield of BSO. Similarly with others parameter, the best performance was shown by the ZA catalyst, followed by ZCA and ZCNTs was the last. The highest yield of 92.29 was achieved by ZA catalyst at 65 ∘C, 5% of catalyst dosage, and 4 h of reaction time. From the overall results it can be concluded that the performance of heterogeneous acid catalysts with different types varies greatly in performance, this is very dependent on many factors including the type of promoter, the type of support, the method of preparation, and many other factors.
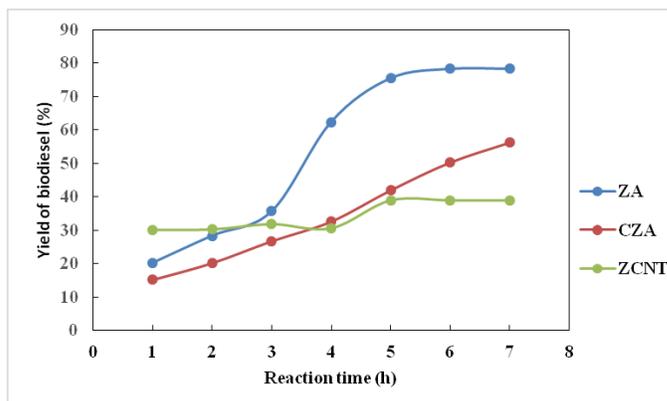

Fig. 3 The influence of molar ratio SO to methanol (wt.% to SO) on BSO yield (%)

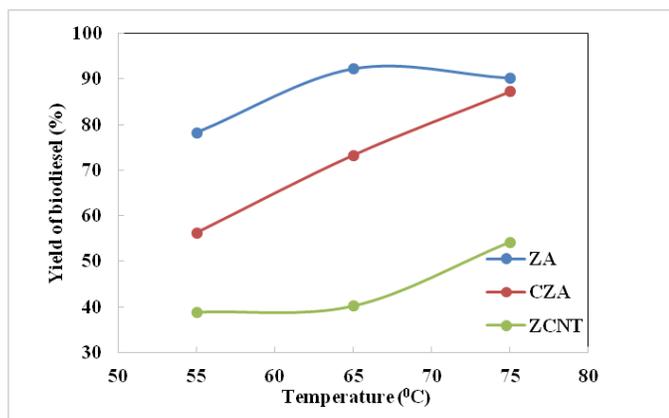Fig. 3 The effect of reaction time (h) on BSO yield (%)

Fig. 4 The Influence of reaction temperature (°C) on BSO yield (%)

IV. CONCLUSION

Three type of synthesized catalyst include ZA, CZA and ZCNTs have been used to convert kesambi Oil (SO) in to biodiesel. SO was potentially converted into biodiesel using those catalysts. However, the performances of each catalyst are different. ZA catalyst has the best performance with highest yield of 92.29 %was achieved at reaction temperature 65 ∘C, catalyst dosage was 5%, reaction time of 4 h. Meanwhile, the highest BSO yield of 87.24 and 54,2 was achieved by CZA and ZCNTs at different condition namely 75∘C, 6% .

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