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Conversion Crude Palm Oil to FAME Over ZnO Catalyst Precipitant Zinc Carbonate

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ABSTRACT

Crude palm oil has a high free fatty acid over 2% is called CPO off-grade. This oil as an alternative energy sources are less costly in the held in one stage by a heterogeneous catalyst in order to produce a biodiesel or fatty acid methyl ester (FAME) over a ZnO catalyst. In this research, ZnO is made from zinc carbonate precipitant. Yield of conversion oil to FAME found approximately 96% under condition temperatures reaction 65 ±2°C, ratio moles of reactants 1:18 v/v, and the amount of catalyst 0.5%. ZnO catalyst is found as a potential in the biodiesel production. The catalytic activity of catalyst is determined by regenerating the spent catalyst, then it re-used four times in the transesterification reaction cycles. Results, the catalytic activity of ZnO catalyst till retain in the 4 th reaction cycle giving the yield of crude biodiesel in the range of 96 –75 %.

KEY WORDS: *CPO; Yield; ZnO.*

NOMENCLATURE

CPO	Crude Palm Oil
FFA	Free Fatty Acid
ZnO	Zinc Oxide

1.0 INTRODUCTION

CPO off-grade, crude palm oil has a high free fatty acid over 5% (FFA > 5%). This oil produced from the palm fruit off-grade. The fruit is categorized includes abnormal size, immature, pass to mature, and decay. CPO off-grade can be processed to become fatty acid methyl ester (FAME) or biodiesel [1, 2 & 3].

The use of biodiesel in diesel engines can reduce emissions imperfect burning of hydrocarbons, such as carbon monoxide, sulfates, polycyclic aromatic hydrocarbons, nitrate polycyclic aromatic hydrocarbons and solid particles. Biodiesel, a fuel oil has calorie value 128.000 Btu [4] almost many as a petroleum of diesel 130.000 Btu [5], It can produce a torque engine and horse power as same as a petroleum of diesel [6]. In room temperatures, biodiesel more safe to kept and used to be than petroleum of diesel, because it is not condense and releases dangerous gas emission such as carbon monoxide, sulfates, polycyclic aromatic hydrocarbons, nitrate polycyclic aromatic hydrocarbons and solid particles. Biodiesel contains oxygen molecules, so it is flash point higher than petroleum, also it is not flammable easily [7].

The application of CPO off grade into alternative energy sources are less costly, transesterifcation and esterification reaction must be held in one stage by a heterogeneous catalyst. This catalyst should be easily synthesized, the price of raw material is cheap and it is performs well. ZnO-based catalyst has been synthesized and used in the esterification and transesterification of vegetable oils in one step [1]. ZnO catalyst also should have a high surface area and uniform pore size. ZnO catalyst nano-sized particle has been synthesized and characterized, and it used in the transesterification reaction of corn oil into FAME [8]. In addition, ZnO-based catalyst, more properly called an absorbent, for the removal of H_2S that has been synthesized by Helianty et. al. (1998) through the precipitation of zinc sulfate with sodium carbonate, it is perform for the removal of H_2S as well as perform of a commercial catalysts.

In this study, a formula to obtain ZnO catalyst has been developed in order to get it is performs well. This catalyst was prepared from zinc carbonate precipitant, then it is used to produce biodiesel from CPO off-grade. Production costs will be competitive with that of biodiesel from CPO off-grade into alternative energy sources are reliable. Further emphasized,

especially for the production of small and medium-scale biodiesel economical, low energy consumption with the concept of zero waste management. The catalytic activity of ZnO is evaluated by regenerating the catalyst after used and it is reused four times in the transesterification reaction cycles.

2.0 METHODS

2.1 Materials

Methanol, H_2SO_4 (±98.99% purity), ethanol p.a, toluene p.a., kalium hydroxide pellet (±99.95% purity, Merck), phenolphtalein indicator were used. Crude palm oil is obtained from a CPO industry of PTPN V Sei. Pagar Riau-Indonesia. Several stages have been done in this research such as CPO sampling, CPO characterization, and the conversion CPO to FAME or a crude biodiesel, determination of the quality crude biodiesel product, finally the catalytic activity of catalyst by regeneration of the spent catalyst and re-used many times in the transesterification reaction.

2.2 CPO Characterization

Several characterization over CPO sample has been determined, such as Ph (universal indicator, Merck), water content (British 1016 Part 104.1:1999**)**, bulk density at 40°C (SNI 01-2891-1992**),** kinematic viscousity at 40°C (ASTM D445**)**, and Free Fatty Acid content (RSB, 2008).

2.3 Conversion Oli to FAME

Conversion oil to FAME has been carried out by two sreps, esterification and transesterification reactions**.** The experiment was conducted using a necked boiling flask with the type blade-turbin-machine impeller (Heidolph). A thermometer was immersed in the boiling flask by using a metal stand, one neck of boiling flask use for input a certain amount reactans and catalysts. The capacity of boiling flask 1 litre, 18.5cm height, outside diameter (OD) 8.725cm and inside diameter (ID) 12.95cm dimensions. The reactor is combined with a water bath/hot plate.

The esterification reaction of oil with methanol was carried out over H_2SO_4 as a catalyst in batch reactor. Catalyst H_2SO_4 and methanolic 0.5% was mixed in a bulb glass of batch reactor. This mixture was agitated and heated until the reaction temperature reached 65°C ± 2 °C, then it added oil and proceed for 30 minutes prior. Afterward, ZnO catalyst 0.5% was poured into the reaction mixture and heated at 65°C ± 2 °C for 1 hour prior to complete. The transesterifcation reaction parameters are keeping identical condition under molar ratio variation between oil and methanol (1:6, 1:12 and 1:18), also variation the amount of catalyst (0.3, 0.4 & 0.5 wt.%).

2.4 Determination Catalytic Activity of The Catalyst

Conversion oil to FAME has been carried out by two steps, esterification and transesterification reactions**.** The experiment was conducted using a necked boiling flask with the type blade-turbine-machine impeller (Heidolph). A thermometer was immersed in the boiling flask by using a metal stand, one neck of boiling flask use for input a certain amount reactants and catalysts. The capacity of boiling flask 1 liter, 18.5cm height, outside diameter (OD) 8.725cm and inside diameter (ID) 12.95cm dimensions. The reactor is combined with a water bath/hot plate.

2.5 Determination Quality of Crude Biodiesel

The product was analyzed such as density at 40°C (ASTM D 1298**)**, kinematic viscosity at 40°C (ASTM 1298), glycerol total (ASTM D6584), and alkyl ester content (FBI-A03-03).

3.0 RESULTS AND DISCUSSION

The amount of FFA in is significantly high approximately 14.5% (Table 1). Based on this result, the conversion oil to biodiesel has been done by two steps the esterification and then transesterification reactions. Edible oil contains high FFA > 5% was converted to biodiesel by two steps processes reactions is applied by many researches [1, 2, 3, 9 & 10].

Table 1: Characterization results of CPO

Properties	Results
Water content	3.19 % dry weight
pH	6.98
FFA content	14.5 %
Oil content	22.86 % dry weight

Table 2: Crude Biodiesel product by using ZnO catalyst 0.3%

Parameters	Crude biodiesel product			Biodiesel Standard
	Ratio of reactant 1:6	Ratio of reactant 1: 12	Ratio of reactant 1: 18	
Yield (%)	56.35	63.40	64.05	
Kinematic Viscosity (40°C , cSt)	6.6	6.3	6.3	2.3 – 6.0 (ASTM D 445)
Densityy (40°C, kg/m^3)	875	858	864	850 – 890 (ASTM D 1298)
Alkyl Ester content (% w/v)	95.70	96.64	97.86	96.0 min
Glycerole total (%, Wt.)	4.52	2.48	2.60	0.24 max. (ASTM D 6584)

Table 3: Crude Biodiesel product by using ZnO catalyst 0.4%

Parameters	Crude biodiesel product			Biodiesel Standard
	Ratio of reactant 1:6	Ratio of reactant 1: 12	Ratio of reactant 1: 18	
Yield (%)	71.72	83.19	93.67	
Kinematic Viscosity (40°C , cSt)	6.6	6.3	6.3	2.3 – 6.0 (ASTM D 445)
Densityy (40°C, kg/m^3)	875	858	864	850 – 890 (ASTM D 1298)
Alkyl Ester content (% w/v)	96.85	97.75	97.03	99.9
Glycerole total (%, Wt.)	4.52	2.48	2.60	0.24 max. (ASTM D 6584)

Table 4: Crude Biodiesel product by using ZnO catalyst 0.5%

Parameters	Crude biodiesel product			Biodiesel Standard
	Ratio of reactant 1:6	Ratio of reactant 1: 12	Ratio of reactant 1: 18	
Yield (%)	88.79	90.09	96.03	
Kinematic Viscosity (40^oC , cSt)	6.6	6.3	6.3	2.3 – 6.0 (ASTM D 445)
Densityy (40^oC, kg/m^3)	875	858	864	850 – 890 (ASTM D 1298)
Alkyl Ester content (% w/v)	97.4	98.24	98.51	99.9
Glycerole total (%, Wt.)	4.52	2.48	2.60	0.24 max. (ASTM D 6584)

Results of conversion oil to FAME as can be seen in the Table 2 – 4. Overall, the stoichiometry reactions between oil and methanol is affected to yield of crude biodiesel production. Yield of crude biodiesel has been obtained maximally ± 96.03% (Table 4). In the reaction using molar ratio of reactants 1:6 is the lowest yield of FAME found than another conditions. This yield is not different significantly than yield of the reaction using molar ratio of reactants 1:12. Others found that the stoichiometry reactions 1:6 is the best produced yield of biodiesel 90 – 98% [1, 2, 12, 13 & 14]. The consideration for this cause CPO off-grade contains high Free Fatty Acid (FFA 14.5%), so in the reaction FFA can contribute to produce high glycerol content as a byproduct (Table 2 – 4).

In this research, H_2SO_4 1% is applied in order to minimaze formed of by product, then continue for 1 hours in the transesterification reaction over ZnO. Metal oxide catalyst ZnO is applied in the reaction because of it is reactivity higher than other acid catalyst, and reaction can be operated under medium temperature [15 & 16], also it is cheaper than other catalysts. Some studies believe that ZnO as a catalyst has high the activity and stability for application to an intensified method of biodiesel production [17]. Also it is surface contains high the acidity and basicity as a metal oxide [18]**,** so it can generate high conversion CPO off-grade to FAME.

Overall, kinematic viscosity of biodiesel is quite higher than biodiesel standard (6.0 cSt) because the amount of existing glycerol in crude biodiesels are high (2.48 – 4.56%). Glycerol as a byproduct is influenced to viscosity of biodiesel be viscous, In addition, in the other research found similar condition, because a certain amount of triglyceride turn to be monoglyceride and triglyceride compounds biodiesel [1, 3 & 19].

An arrangement reaction temperature is very important with an assumption that reactant particles for collusion has more potential of increasing each other, so the activation of energy for reaction will be reduced and the reaction acceleration will be rising. Reaction temperature in the conversion CPO off-grade to crude biodiesel is 65^oC. Generally, the limitation of temperature reaction usually which is used by several researches in biodiesel production is around 50^oC - 65^oC [2, 3, 13 & 14]. If the reaction temperature higher than boiling point of methanol (68^oC), methanol tends more vapored easily, if conversion's temperature is under 50^oC, viscosity of biodiesel of biodiesel will increase [20 & 21].

Molar ratio of alcohol to triglyceride present in oil is an important parameter to be considered while screening the catalyst performance and in maximizing the fatty acid alkyl ester/glycerol yield biodiesel [1, 2, 3 & 9]. Theoretically, the transesterfication of oil requires three moles of methanol per mole of triglyceride. Since transesterification of triglyceride is a reversible reaction, the excess of methanol shifts in the equilibrium towards the direction of methyl ester formation. In the presence of excess alcohol, the forward reaction is product formation. In addition, usage of excess alcohol is one of the better options to improve the rate of the transesterification reaction catalyzed by heterogeneous catalysts [1, 2, 3, 9 & 11]. In the present work, with pre-optimized reaction parameters, molar methanol to oil ratio was varied in the range of 6:1, 12:1 and 18:1 and its influence on fatty acid alkyl ester yield.

The highest conversion of 96% was registered at molar ratio of 18:1. For molar ratio of reactants from 1:12 to 1:18, further increase in the methanol amount has not showed any significant improvement in the conversion. Probably, higher concentration of methanol interferes with the separation of glycerin because of its increased solubility. It is also possible that presence of glycerin in the solution might drive equilibrium to backward direction yielding lower contribution of glycerin in the product or may cause esterification of glycerol in presence of methanol. Thus, under the mentioned reactions conditions, methanol to oil molar ratio of 18:1 seems to be the most appropriate.

In order to understand the catalytic performance of ZnO, this catalyst was evaluated for optimizing the catalyst preparation method and reaction parameters, it is desired to check the reusability/recyclability of the catalyst. Results transesterification reaction using a catalyst re-used compare to transesterification reaction with a parent catalyst shown in Table 5.

Table 5: Comparison Crude Biodiesel product by using several reactions

Parameters	Crude biodiesel product			Biodiesel Standard
	Reaction I*	Reaction II**	Reaction III***	
Yield (%)	90.78	85.46	75.68	
Kinematic Viscosity (40^oC , cSt)	6.6	6.3	6.3	2.3 – 6.0 (ASTM D 445)
Densityy (40^oC, kg/m^3)	875	858	864	850 – 890 (ASTM D 1298)
Alkyl Ester content (% w/v)	93.48	96.56	96.39	99.9
Glycerole total (%, Wt.)	4.52	2.48	2.60	0.24 max. (ASTM D 6584)

*** transesterification reaction over a fresh catalyst in the first cycle.**
**** transesterification reaction over the spent regenerated catalyst in the second cycle.**
***** transesterification reaction over the spent regenerated catalyst in the third cycle.**

Results shows, the catalytic activity of ZnO catalyst till 4th reaction cycle giving the yield of crude biodiesel in the range of 90-70% (Table 5), which indicates that the sites of the catalyst are not deactivated in the regenerated catalyst. Overall, the conversion oil to crude biodiesel was observed to be reduced from 90.78% to 70.98%. This is indicate that the fresh catalyst gets little deactivated after used. The reduction in conversion could probably be due to the deposition of reactants and/or products on the active sites, and/or loss of the active sites during the reaction. The yield decreased after four reaction cycles, which is attributed due to the deposition of carbonaceous material on the external surface of the used catalyst.

But, yield biodiesel of transesterification reaction over the spent catalyst regenerated were still high for two and three times cycles. Endelaw et.al (2014), found that activity catalytic of ZnO catalyst in the transesterification reaction is high significantly. The ZnO nanoparticles were reused 17 times without any activity loss in a batch stirred reactor an average yield of FAME was around 93.7% [1]. It can be concluded that ZnO catalyst is potential as a catalyst in the biodiesel production. For this evidence, results of reaction over a catalyst regenerated for second and third cycles reactions such as kinematic viscosity at 40°C, density at 40°C, alkyl ester contents are still suitable with standard biodiesel for each parameters.

ZnO catalyst nanoparticles can be prepared from several methods including: sol-gel processes, precipitation, spray pyrolysis, thermal evaporation and mechanochemical synthesis. Prespitasi homogeneous often considered economically appropriate for the preparation of monodisperse particles of metal oxides with different sizes and shapes. This method also gives control of the morphological and chemical characteristics better [Bhattacharjee et al, 2011]. In the precipitation method, ZnO nano-particles are usually obtained from the wet chemical reaction using a solution of the compound $ZnSO_4.H_2O$, $ZnSO_4.6H_2O$, $ZnSO_4.7H_2O$, Zinc acetate, and $ZnCl_2$, NaOH or Na_2CO_3 as starting materials (precursors). Reaction with this precursor produced a form of intermediate carbonate compounds [Twigg, 1996]

These intermediates are used as carbonate salt solubility is so low that very high level of saturation. These properties cause the particle size on the precipitation produced is very small. Na2CO3 precursor solubility in water is high, and can be used as a concentrated solution. Carbonate compounds directly decomposed by heat into oxides with high surface area without leaving any residue on calcination sulphate sulfur. The sulfur compounds can cause deactivation of the catalyst. Moreover calcination carbonate compounds do not cause problems in the environment. [Twigg, 1996].

Table 6: Biodiesel yield for several solid Zinc oxide based catalysts

Catalyst type	Oil source	FFA content wt.%	Reaction conditions	Yield, %	Ref.
ZnO (99.0% purity purchased from Fluka)	PKO	0.5	200°C; 6:1; 50 min; 3 wt.%;350 rpm	86.1	Jitputti
ZnO pellet	SO	8.14	67°C; 18:1; 2h; 1 wt.%;360 rpm	96	Kumar
ZnO (nano particle)	Corn oil	10	90°C; 3:1; 2h; 3wt.%; 300 rpm	60	Peres
ZnO/La	PKO	8	170-220°C; 18:1; 3h;3 wt.%;600 rpm	96	Shulli
Zn/KF	CtO	15	80-90°C; 3:1; 2.5h; 3 wt.%;600 rpm, 55 psi.	97.3	Anastupoulos,
La_2O3-ZnO	JCO	9	60°C; 6:1; 3h; 5 wt.%;300 rpm	30	Endalew
SO_4^{-2}/ZnO	CPO	14.5	65°C; 18:1; 1h; 0.5 wt.%;300 rpm	96	

PO=palm kernel oil; CPO= crude palm oil; CO=corn oil; SYO=soya bean oil; WCO= waste cooking oil; JCO=jatropha curcas oil; SO=sorghum oil; CtO=cotton's seed oil.

Table 6 shows some edible oil contains high FFA >5 wt.% can be converted to biodiesel over Zinc oxide based as catalysts [1, 2 & 3]. Based on those research, in order to get biodiesel yield around 70% - 98%, the temperature reaction so high approximately 160 °C - 200°C, it need high energy to running the transesterification reaction, and very expensive cost. Yet, results in this research under the mild temperature reaction condition 65°C ± 2°C , molar ratio of reactants 18:1, reaction time 1 hour and amount of catalyst 0.5% is obtained biodiesel yield 90 wt.%, also Endelaw et al (2014) found biodiesel yield 100%, under a mild condition (Table 5).

Several methods have been proposed to resolve the issue. For example, the use of biocatalyst (enzyme), an acid catalyst and pre-esterification. Transesterification using enzymes could be held at moderate temperatures and produces high yields, but it cannot be used in the industry because of the high cost of procurement of enzymes, and deactivation of catalyst occurred by impurities contained in the raw material [23, 24, 25 & 26]. Meanwhile, the use of acid catalysts in the reaction produce problems associated with the corrosive nature of the acid catalyst, and the high amount of byproduct produced. The disadvantages of pre-esterification method is to separate the product from the acid catalyst after reaction need expensive cost [9, 10]. So, the solution to improve the process once again is to use a heterogeneous esterification catalyst.

4.0 CONCLUSION

Yield of FAME in the conversion CPO to crude biodiesel found around 96% maximally. Under condition temperatures reaction 65°C ± 2°C, ratio moles of reactants oil: methanol 1:18 v/v and the amount of catalyst 0.5%. Yield conversion extracted oil to biodiesel by using the spent catalyst has been obtained maximally ±96%. The catalyst ZnO was found highly active for transesterification of CPO off-grade methanol giving high yield of crude biodiesel (96%) after 2 hours. ZnO catalyst is potential as a catalyst in the biodiesel production. It is clear that the use of a heterogeneous catalyst in the production of biodiesel can reduce the cost of production, so that biodiesel can be competitive to petroleum diesel in terms of economy.

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