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by Joelianingsih Joelianingsih

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Biodiesel production from palm frying oil using sulphated zirconia catalyst in a bubble column reactor

Is Sulistyati Purwaningsih[a*], Joelianingsih[a], Wahyudin[a]

[a]*Chemical Engineering Department, Institut Technology of Indonesia, Jl. Raya Puspiptek Serpong, Tangerang Selatan 15320, Indonesia*

Abstract

Homogeneous catalysts are promising for the transesterification reaction of vegetable oil to produce biodiesel since this catalyst offer certain advantages such as high activity, easily reached reaction condition and less expensive; however homogeneous catalyst has some drawbacks such as high energy consumption, costly separation of catalyst from the reaction mixture and the purification of the product. The use of bubble column reactor (BCR) in producing biodiesel fuel without catalyst has been developed. In the BCR, the role of catalyst was replaced by high operating temperature, while the role of agitation was taken over by the formed vapor bubbles. The experimental result concluded that the higher the operating temperature, the higher the product conversion as well as the reaction yield, although it lowers the biodiesel's purity. Nowadays, heterogeneous catalysts have been more widely favoured over the homogeneous one since they are easily separated from reaction mixture and reused for many times. In this study, transesterification reaction of refined palm oil (palm frying oil) was conducted in a bubble column reactor using sulphated zirconia (SO_4/ZrO_2)as the solid heterogeneous catalyst. The experiment was carried out at 250 ° C. At first, the influence of methanol flow rate towards vapor bubble formation was investigated, the experiments were then run catalytic and non-catalytically by varying the catalyst to reactant mass ratio. The experimental result showed at 5 mL/min of methanol flow rate, the amount of methanol vapor bubbles were continuously produced and uniformly distributed in the oil phase. This condition was then selected for the remaining study. It was noted that the highest yield of biodiesel product was achieved at 0.5 % (m/m) of catalyst concentration, meanwhile yield of product that run without catalyst was the lowest among all experimental results. However, at 1% (w/w) of catalyst to reactant mass ratio, the product phase was changed to solid.

Keywords: biodiesel; refined palm oil (palm frying oil); SO4/ZrO2 catalyst; bubble column reactor

1. Introduction

The catalytic alcoholysis of vegetable oil use to produce biodiesel, is an important industrial process. Biodiesel is a promising fuel for substitution or blending with petroleum based diesel fuel, while these two kinds of fuel share similar physical and chemical properties[1]. Some of the first industrial processes to produce biodiesel used either strong base or strong acid homogeneous catalyst for transesterification reaction, such as potassium and sodium hydroxides, sulfuric, hydrochloric or sulphonic acids. The homogeneous catalyst offer certain advantages such as high activity, easily reached reaction condition (25-130°C at atmospheric pressure) and less expensive, but face variety of technical difficulties[1]. Homogeneous catalysts are normally carried out in batch-mode processing. Furthermore, a drawback of homogeneous base-catalyzed transesterification is that the oil that contain significant amounts of free fatty acids could not be convert into biodiesel completely and remained as soap in high quantity[2]. Separation of the product from the soap and spent waste catalyst appears to be technically challenging and brings additional cost to the product. It was reported that generally acid catalyzed reactions are performed at high alcohol to oil molar ratios, low to moderate temperatures and pressures, and high acid catalyst concentrations. Unfortunately, ester yields do not equally increase with molar ratio of reactans. Despite its insensitiveness to free fatty acids, acid-catalyzed trans-esterificataion is relatively slow reaction rate[3]. However, Saka and Kusdiana[4] revealed that producing biodiesel fuel from rapeseed oil which was prepared in super-critical methanol (at 350 °C and 200 Bar) had highly reduced reaction time. Unfortunately, such kind of reactor is not only high risk but also high capital and operating cost.

Yamazaki et al.[5] and Joelianingsih et al.[6,7]have studied the production of non catalytic biodiesel oil employed by Bubble ColumnReactor (BCR) at atmospheric condition. In the semi-batch system, Joelianingsih[7] had varied the temperature reaction in the range between 250 and 290 °C with flow rate of methanol vapor set at 4 g/min. Transesterification reaction of triglycerides (TG) to form methyl

* Corresponding author. Tel.: +628125714335; fax: +62217560542.
E-mail address: ispurwaningsih@iti.ac.id

ester (ME) in the BCR showed that this reactor acts as reactive distillation (RD); the reactor is not only as a reaction place, but also as a product separator as well. The RD concept works appropriately on equilibrium reaction such as in vegetable oil transesterification reaction, because straight and continuous separation of reaction product will drive the equilibrium to the product side and raise the conversion. According to Yamazaki et al.[5] and Joelianingsih et al.[6], in the BCR, the role of catalyst was replaced by high operating temperature, while the role of agitation was taken over by the formed vapor bubbles. Their experimental result concluded that the higher the operating temperature, the higher the product conversion as well as the reaction yields although it lowers the biodiesel's purity. It was also revealed that BCR that runs with high temperature condition will cause more monoglyceride (MG)formed as impurity in the product. According tothe Indonesian National Standard[8], the ME content in biodiesel should be less than 96.5% (m/m). Many studies have been reported that utilization solid or heterogeneous catalyst can be done to improve quality and conversion of biodiesel product[1].

Producing biodiesel oil incorporated heterogeneous catalyst has the potential to offer some relief to the biodiesel producers by improving their ability to process other cheaper feedstocks and to use a shortened and less expensive manufacturing process[1]. In addition, compared to the homogeneous one, this catalyst can be easily separated from reaction mixture and reused for many times. Moreover, using heterogeneous catalyst, biodiesel processing can be run continuously. Several solid acid catalysts have been reported to have the potential to replace strong liquid acid. These catalysts are zeolites, heteropoly acids, functionalized zirconia & silica, and some metals oxide[3]. Zeolites normally can be synthesized with extensive variation of acidic properties. However due to their uniform pore structure, the hydrophobicity of the catalyst is still in the stage of trial and error[3,9]. Previous study[10], reported that sulphated zirconia (SO_4-ZrO_2) has been utilized as a catalyst for transesterification vegetable oil to produce biodiesel. Study by Kiss[1] revealed that this catalyst was the most active catalyst for esterification. Petchmala et al.[11] reported that transesterification of palm oil with SO_4-ZrO_2 catalyst in super-critical methanol at 250^{0}C and within 10 minutes reaction, its conversion reached to 90 %.

Based on the above information, in this study, experiment was carried atmospherically in a set of BCR apparatus incorporated with SO_4-ZrO_2as a catalyst. Previous study [5] revealed that transesterification reaction carried out in the BCR, run non catalytically at atmospheric pressure and at 250 °C temperature , produced biodiesel oil with high purity (almost reach the standrad value), but its conversion was very low (55%). However , the superiority of BCR over other type of reactors is based on the capability act as RD proces. Even though the rate of the reaction is very slow, the product is directly separated from the reactans and produces higher purity of ME. In addition, reaction can be proceed continuously although with low capacity. It is expected by combining the BCR technology with the advantage of sulphated zirconia as the catalyst in transesterification of vegetable oil[11], will increase quality and productivity of the biodiesel.

2. Experimental methods

2.1 Materials

Palm Frying Oil (PFO, Bimoli brand) was purchased at Alfa-Mart grocery store. The fatty acids composition of palm frying oil was determined by gas chromatography (GC) equipped with a flame ionization detector and a cyanopropylmethyl silicone column (60 m × 0.25 mm internal diameter and film thickness of 0.25 μm). Thecarrier gas was helium at 1 mL.min-1. The oven temperature was initially held at 160 ° C for 5 minutes then increased to 220 °C. This analysis was conducted at the Integrated Laboratory of Bogor Agricultural University. Methanol (MeOH) with 99.8 % purity (analytical grade) was produced by PT. Smart-Lab. Indonesia. Sulphated zirconia (SO_4-ZrO_2) catalyst.

2.2 Preparation of catalyst

Sulphated zirconia (SO_4-ZrO_2) catalyst was prepared by Center of Physics Research - The Indonesian Institute of Sciences, Puspiptek Serpong South Tangerang. As for catalyst preparation, 100 gram of $ZrOCl_2$ $8H_2O$ was disolved into 1000 mL of aquadest water. Ammonium hydroxide solution was added dropwise into well-stirred solution of $ZrOCl_2$ until pH reach to 9 at room temperature till ZrO_2 solid formed. The resulting precipitate was removed by filtration and then washed by aquadest until free of chlor. The solid was then dried in the oven at 120°C, for 16 hours. The sulphated zirconia was then prepared by impregnation of H_2SO_4 over$Zr(OH)_4$ by immersing in 1.0 N solution of H_2SO_4 (1 gram sample in 15 mL H_2SO_4) for 30 min.The solid was then filtered and dried at 120°C. Next, solid sample was calcined (650°C, 3.5 hr). Differential Thermal Analyzer (DTA) was applied to determine calcination temperature of catalyst. The sulphated zirconia catalyst was characterizied using the Fourier Transform Infra Red (FTIR) analyzer, the XRD and the Brunauer-Emmett-Teller (BET) value. The FTIR analysis was conducted prior impregnation the ZrO_2 with sulphate solution and after calcined at the ITI Chemical Engineering laboratory. The crystal structure was determined using X Ray Diffractometer at the Center of Integrated laboratory of University of Islam Negeri Syarif Hidayatullah Jakarta, while the BET analysis was carried out at the BATAN laboratory ,Puspiptek Serpong.

2.3 Apparatus

A photograph and a schematic diagram of a BCR apparatus are presented in Fig. 1 .The apparatus was equipped with a methanol tank, vaporizer, reactor, heater, and condenser columns.

2.3. Experimental procedures

Initially, the reactor was filled with PFO until reach the minimum mark level. Then another 250 ml of palm oil was filled into reactor, put the reactor lid and bring it into tight. Secondly, the vaporizer was filled with MeOH till the maximum mark level followed by filling up the MeOH tank. Next, switched the reactor heater on and held until reached the setting temperature. Afterwards, the super-heater was turned on until temperature reached to 250 °C, followed by running the water cooler. Then, switched the MeOH vaporizer on and adjusted its vapor flow rate, followed by turning MeOH pump on and adjusted its pumping rate according to the vapor rate. Reaction time was counted after the vapor bubble uniformly formed and evenly distributed in the reactor. Finally, samples were taken within 20 minutes interval time, and were analyzed using GC.

Fig 1(a). The photograph of biodiesel apparatus set up

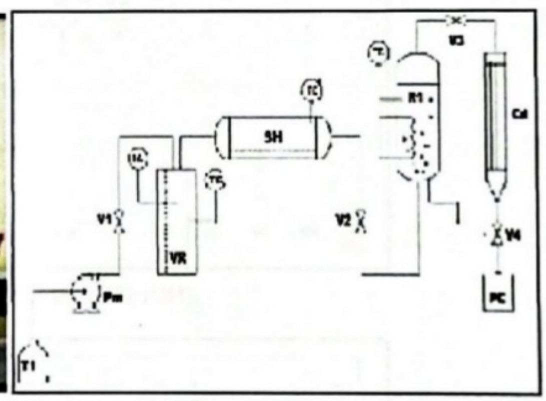


Fig 1(b). The schematic diagram of biodiesel experiment apparatus.
T1: MeOH tank, Pm: MeOH, pump, V1-V4: valves, VR: vaporizer, LC: level controller, TC: temperature controller, SH; superheater, R1: reactor, Cd: condenser, PC: product collector.

2.4. Product analysis

The biodiesel products analysis was carried out using a gas chromathograph, Shimadzu 2010 model with a flame ionization detector (FID), using helium as a carrier gas. The GC featured a capillary column (RTX-1 restex nonpolar phase; Cross bond® dimethyl polysiloxane, 30 M, 0.32 mm ID, 0.5μM). Method of analysis follows the modified EN 14105[12].

3. Results and discussion

3.1. Fatty Acid composition of palm frying oil material

The major fatty acid composition of PFO was tabulated in Table 1 and its result was compared to fatty acid composition of several palm oils analyzed by Petchmala et al. [11] and Kataren [13].

Table 1. Comparison of Fatty Acid Composition

Fatty Acids	Fatty Acids Composition (% w/m)		
	Sample (avg)	Reference [11]	Reference [13]
Miristic Acid	1.2	1.0	1.1 – 2.5
Palmitic Acid	43.9	45.6	40 – 46
Stearic Acid	3.9	3.8	3.6 – 4.7
Oleic Acid	41.7	33.3	39 – 45
Linoleic Acid	9.3	7.7	7 – 11

It can be observed that the fatty acid composition of PFO sample in this experiment is in accordance with the results of previous studies [11,13].

3.2. Catalyst characterization

Sulphated zirconia (SO_4-ZrO_2) catalyst that was prepared from the impregnation of H_2SO_4 over $Zr(OH)_4$ resulted in 48 % (w/w) yield. The FTIR analysis prior H_2SO_4 impregnation and after H_2SO_4 impregnation followed by catalyst cacination were presented in figures 2 and 3.

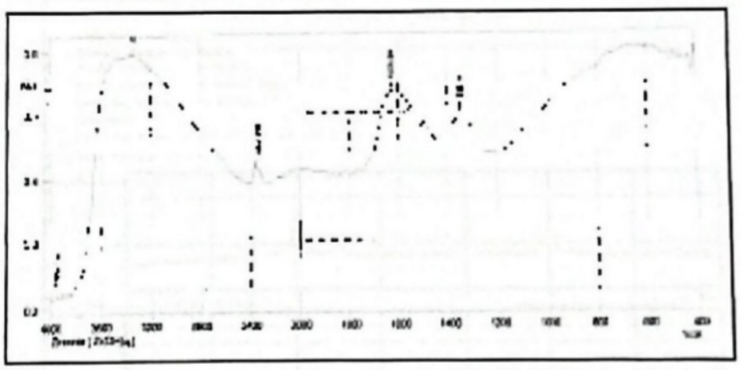


Fig 2. FTIR Spectrum of Zr $(OH)_4$

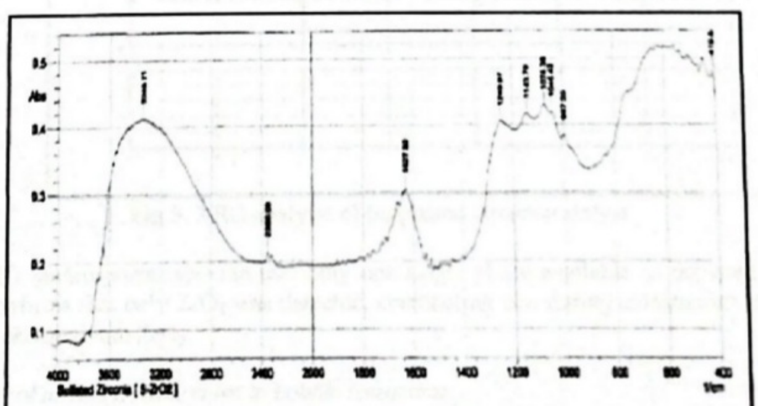


Fig 3. FTIR spectrum of SO_4-ZrO_2

Fig.2 indicates the spectrum of FTIR analysis of $Zr(OH)_4$ prior to H_2SO_4 impregnation and Fig. 3 shows the spectrum of FTIR analysis of SO_4-ZrO_2. It was noted that there is a difference absorbance profile between these two figures at the range of 1000 – 1280 cm^{-1}wave lengths. According to Stuart [14] the SO_2 and SO groups of sulphur compounds produce strong infra red band at wave length of 1000–1400cm^{-1}. It can be concluded that H_2SO_4 impregnation over the $Zr(OH)_4$occurred, since sulphates group were identified at 997,20; 1045,42; 1074,35; 1143,79 and 1249,87 cm^{-1} wave lengths.

The specific surface area of catalyst was determined by the BET method and found to be 53.26 m^2/g. This result is represented in fig 4.

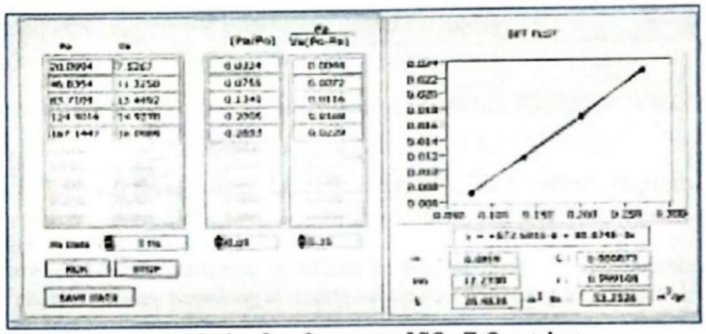


Fig 4. Analysis of surface area of SO_4-ZrO_2catalyst

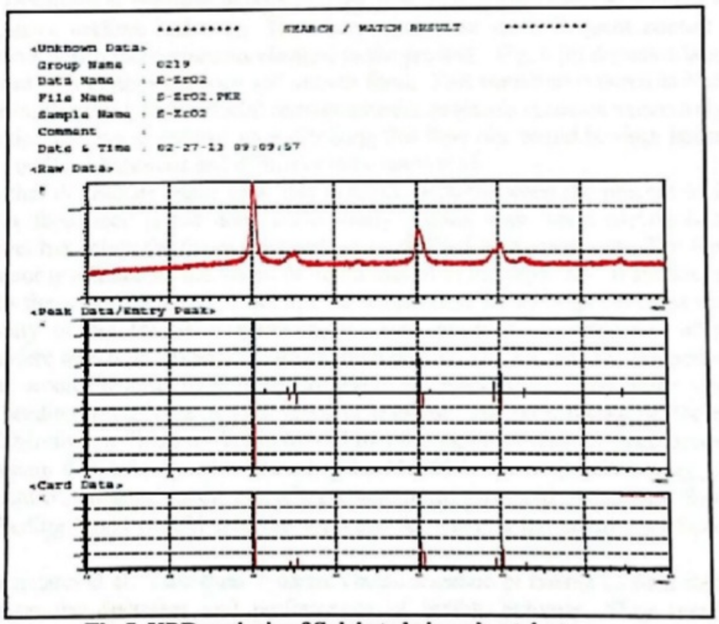
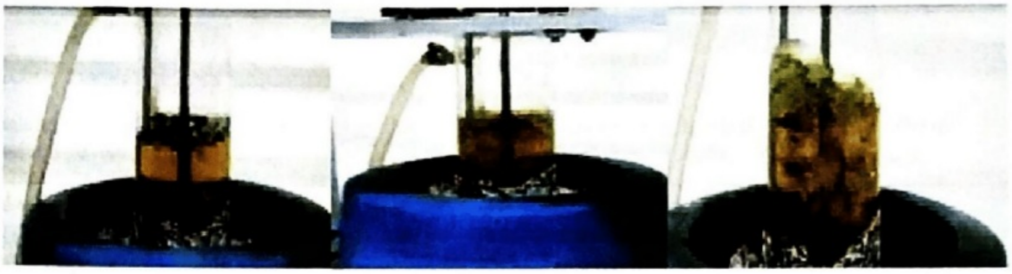


Fig 5. XRD analysis of Sulphated zirconiacatalyst

While XRD measurement showed that only one single phase available as depicted on Figure 5. This result confirms that only ZrO_2 was detected, considering that during calcinations process all the $Zr(OH)_4$ was changed into ZrO_2.

3.3. The effect of methanol flow rates in bubble formation

The effect of methanol flow rates in bubble formation are shown in Figure 6. These figures show the flow rate differences in bubble formation. The bubble sact as substitutes for stirring the biodiesel manufacturing process in which the reaction occurs at the contact interface of the bubbles. The methanol flow rates affect the amount and uniformity of the formed bubbles.



(a) (b) (c)

Fig 6. Bubble formation at methanol flow rate of 3,5 and 7 ml/min respectively

Fig. 6 (a) shows bubble formation at 3 ml/min of MeOH flow rate. The number of bubbles produced was slight and uneven. Resulting in interfacial contact between MeOH and oil is not optimal due to the lack of MeOH vapor flows into the reactor column. Consequently, the reactants which supposed to be changed into the product become even and less likely to be a product. Fig. 6 (b) indicates bubble formation at 5ml/min of MeOH flow rate. It is shown that the number ofbubbles formed are muchmore uniform and even. Therefore, there were more frequent contact interface between the reactants which more reactants changed to the product. Fig. 6 (c) depicts a large amount of bubbles produced with in homogeneous and uneven form. This condition occured as MeOH flow rate run at 7 ml/min. Resulting in interfacial contact between reactants becomes excessively fast and the product formation was not at optimal state. Utilizing this flow rate would be risky because of the numerous bubbles outburst happened and difficulty to be controlled.

It is observed that the MeOH vapor flow rate becomes unstable when the process of filling the liquid methanol as fresh feed is not done continuously. Filling with liquid MeOH is conducted whenthe liquid level has below the lineof fluid indicator specified in the vaporizer. The formation of bubbles in the reactor is affected by the height of liquid MeOH in the vaporizer. If sudden refilling of liquid MeOH into the vaporizer occured because of temperature of the vaporizer has reached the temperature stability of the MeOH evaporation, the cold physical characteristics of the liquid methanol will interfere with high temperature distribution process.The MeOH with temperature below evaporation point would require more time to reach its evaporation temperature causing the methanol's flow heading towards the reactor tends to wane or even none (based on the number of bubbles formed).Therefore, refilling the liquid MeOH into the vaporizer must be done continuouslyby generatingsuch system that refillingand evaporating the MeOH to occur simultaneously, in order to maintain the amount ofliquidmethanolat a stable predetermined threshold. Additional flow meter is required before feeding liquid MeOH into the vaporizer to maintain the continuous flow of liquid MeOH

According to Kantarci et al.[15], the fluid dynamic characterization of bubble column reactors has a significant effect on the operation and performance of bubble columns. They noted that the performance of bubble columns strictly depend on the regime prevailing in the column that can be classified and maintained according to the superficial gas velocity employed in the column. The methanol vapor superficial velocity obtained in this study was presented in Table 2.

Table 2. Superficial velocity of methanol vapor

Flow rate ($^{ml}/_{mint}$)	Vapor Velocity ($^{cm}/_{s}$)
3	1,370
5	2,300
7	3,219

Kantarci et al.[15] reported that the bubbly flow regime, also called the homogeneous flow regime is obtained at low superficial gas velocities, approximately less than 5 cm/s. This flow regime is characterized by bubbles of relatively uniform small sizes. In addition, a uniform bubble distribution and relatively gentle mixing is observed over the entire cross-sectional area of the column. Based on the above table, it was showed that the superficial velocities of methanol vapor employed in this study were in agreement with Kantarci et al.[15]. Based on the vizualisation of above figures obtained, and supported by literature data, the flow rate of 5ml/min of methanol was then selected for further studies.

3.4. The effect of catalyst to reactant mass ratio in biodiesel yield

Experimental study was conducted with and without catalyst. Amount of catalyst were varied in the range of 0.1 – 1.0 % (w/w) catalyst to reactant ratio when sulphated zirconia catalyst was employed. Experimental result is presented inTable3.

It was shown that average yield of product accumulation of biodesel run catalitycally is higher than that of non catalitycally (4.41% after 100 min run). Previous study conducted by Joelianingsih et al[7] at thesame temperature (250°C), non catalytically, revealed that product yield was 1.25% and 10.96 %(w/w) after 60 and 180 min run respectively. There is a significance difference if these data are compared to this non-catalytically experiment result. The yield obtained in previous study[7] was total yield of product in the vapor and liquid phases. Their experiment was conducted in a four-necked flask equipped with a condenser and a pipe for MeOH vapor feed, and the TLC/FID was used to analyze content of product. In this experiment, the yield obtained was yield in the vapor phase only. The BCR form is different to the reactor used in the previous study[7]. In addition, the GC was used to analyze the product in this experiment. Some differences mentioned above that might cause product yield run non-catalytically in this experiment lower than the previous study.

It can be observed also that the biodiesel product yield obtained run with catalyst were significantly low compared to the previous studies[6,7,11]. Petchmala et al.[11] indicated that the specific surface area and calcination temperature of sulphated zirconia catalyst play an important role in transesterification reaction of vegetable oil. The BET surface area of sulfated circonia employed in this study was only 52.26 m^2/g, with calcination temperature at 650 °C, while Petchmala et al.[11] concluded that the most active catalyst of their synthesized sulphated zirconia catalyst has the BET value of 234,9 m^2/g and calcination temperature at 500 °C. The specific surface area of sulphated zirconia considerably decreases as calcination temperature is increased from 500 to 700 °C, since the amount of active sites of catalyst was reduced. In addition, the low yield obtained in this study might be due to the short contact time between oil and methanol vapor, resulting the reactions to be incomplete. The mechanism reactions of transesterification of PFO can be presented as follows:

Table 3. Experiment results of biodiesel production (methanol flow rate at 5 ml/min, reactor temperature at 250°C, time 100 min)

Catalyst to reactant ratio, % (w/w)	Product yield, %	Product picture
0.00	4.41	
0.10	6.09	
0.50	7.91	
1.00	5.53	

$$TG + MeOH \leftrightarrow DG + FAME \quad (1)$$
$$DG + MeOH \leftrightarrow MG + FAME \quad (2)$$
$$MG + MeOH \leftrightarrow GL + FAME \quad (3)$$

The third reaction, in which MG with methanol turns to Fatty Acid Methyl Esters (FAME)/biodiesel and glycerol (GL), is considered the slowest reaction among all above reactions, because MG, as an intermediate compound, is the most stable compound compared to triglyceride and diglyceride[16], and resulted that the amount of MG formed was excessive.

It is shown from Table3 that the reaction run with 0.5 % (w/w) catalyst produces the highest yield than the run using 0.1 % and 1.0% (w/w) catalyst. Catalytic experiment run with 0.1% (w/w) sulphated zirconia resulted in 6.9 % of product yield, while with 0.5 % (w/w) catalyst, yield obtained was 7.91 %. Several literatures[3,9,11] revealed that the highest yield of biodiesel products was achieved at catalyst to reactants mass ratios between 0.1 and 0.5 %. These findings support our experimental results.

Since method of product analysis in this experiment follows the modified EN 14105[12], in which in the list of EN 14105 parameters standard noticed to calculate the impurities of biodiesel product, such as free GL, MG, DG, TG, and total GL, instead of purity of biodiesel product, so that in this study, purity of the product did not directly analyzed . However, it could be calculated using equation (1) to (3) with several assumptions.

Utilization of 1% catalyst to reactant mass ratio in this study causes biodieselproduct to change intosolid phase with the acquisition ofa lowyield. The low yield of product indicated that more MG present in the biodiesel. According to literature [17], melting and solidification points of MGs are higher than room temperature. This condition might be caused by the biodiesel product phase that changed into solid at room temperature. The result study confirms employee of 1% (w/w) catalysts to reactant ratio is unappropriate in transesterification of vegetable oil for biodiesel production as revealed by literatures[3,11].

Conclusion

Biodiesel production by transesterification of palm frying oil carried out in bubble column reactor in the presence of SO_4/ZrO_2 catalyst, unfortunately, beyond the expectation, because all experimental results showed lower biodiesel yield than previous studies. The highest yield was 7.91% run with 0.5 % (w/w) catalyst to reactant mass ratio.

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