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Article

Effects of Reactor Loading and Solvent Addition on Catalyst-Free Glycerolysis of Palm Oil

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Abstract. Glycerol is a by-product of biodiesel production. Every three moles of biodiesel produced, glycerol is released in one mole, which is around 10 wt.% of the total products. The crude glycerol from supercritical transesterification has a higher purity than that from alkaline transesterification. Monoglyceride is an anionic surfactant widely used in many applications. In this work, the glycerolysis reaction of palm oil and glycerol was studied using isopropanol as the solvent. The investigated important parameters in this study were reaction time in range of 30-150 minutes, reactor loading in range of 40-80 %, and molar ratio of isopropanol to glycerol to palm oil in range of 0-30. The glycerol to palm oil molar ratio was constant at 5 to 1. The results showed that parameters affected conversions and yields were reactor loading and solvent addition. The highest monoglyceride yield, 37.4%, was obtained at 260 ˚C in 150 minutes and 40 % of reactor loading. Molar ratio of glycerol to palm oil to isopropanol is 5:1:15. A central composite design (CCD) of 48 experiments investigated the effects on monoglyceride content (%MG) of temperature (220 to 260 ˚C), reaction duration (30 to 150 min), and molar ratio of IPA to palm oil (0:1 to 30:1). The %MG was substantially and statistically significantly enhanced ($p < 0.0001$) at higher temperatures and longer reaction duration. An analysis of variance confirmed that the molar ratio of IPA to palm oil had a much less significant effect ($p = 0.0255$) on %MG. The crude glycerol obtained from a biodiesel production plant was compared with pure glycerol at the optimal condition. A %MG of 46.58% was observed using crude glycerol as reactant because of the yield-limiting effects of water in crude glycerol.

Keywords: Glycerolysis, palm oil, glycerol, isopropanol.

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1. Introduction

The fast growth in biodiesel production lead to excess of glycerol the main by product in this process. The transesterification process release biodiesel and glycerol at a molar ratio of 3:1. Between 2008 and 2012, glycerol production around the world increased by 9.3% per year, and the growth will estimate to be 3.5% per year in 2025. Glycerol is generated ~10% by weight of biodiesel. Because rapidly grows of biodiesel production worldwide, the amount of glycerol in the world market is over-supplying nowadays.

The humectant properties of glycerol have long been and utilized in sweets, cakes, meats, and cheeses. In addition, glycerol has been used as solvent, sweetener and preservative in beverages and foods. Also, using glycerol to give the flavor and color in foods and soft drinks [1, 2]. However, the degree of purity is the restricted usability of glycerol.

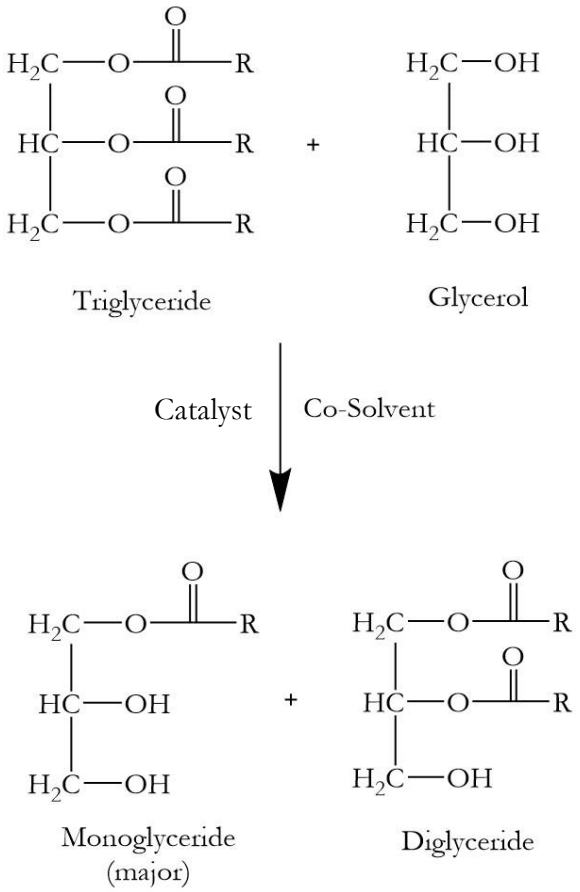

Fig 1. Glycerolysis reaction between triglyceride and glycerol.

Conversion of glycerol to monoglyceride is an option to utilize glycerol as bio-based feedstock to solve the over-supply problem. Monoglyceride has many applications in cosmetics, pharmaceuticals, and food industries as surfactants. The glycerolysis of triglyceride (TG) with glycerol is the process that produced monoglycerides (MG) and their derivatives as illustrated in Fig. 1. Monoglyceride is a raw material in various applications because of their emulsifying properties [3]. The optimal condition for monoglyceride production as found in the literatures is illustrated in Table 1.

The feedstocks for monoglyceride production are triglyceride, fatty acids, and fatty acid methyl esters. Using methyl ester as feedstock generates methanol as by-product that is not suitable for food and cosmetic application. The basic catalysts, KOH, and $Ca(OH)_2$, are conventionally used in this process. Enzymatic and ionic liquid were reported as alternative catalysts that provide the high %MG. The production of monoglyceride conducts with solvent or without solvent systems. Recently, the non-catalytic processes, using acetone and isopropanol (IPA) as solvents, were reported with the competitive %MG.

The typically glycerolysis process is carries out at high temperature (220-260 ºC), with an alkali catalyst, pressurized under nitrogen atmosphere. The major drawback of using alkali catalyst in glycerolysis reduction is the presence of free fatty acids (FFA), which would result in the formation of metallic soap, leading to flavor and odor troubles when blend in food products [4].

To avoid such problems, isopropanol under subcritical condition was employed as solvent for the glycerolysis of palm oil without the use of conventional catalyst. According to Liu et. al, the authors demonstrated that isopropanol was the most effective solvent in glycerolysis reaction when compared to methanol and ethanol [4]. Even though three lower alcohols could reduce viscosities of reactant components, isopropanol provided the highest yield of monoglyceride. Apart from the highest yield obtained, isopropanol does not occur the side reaction while the others (methanol and ethanol) generate fatty acid ester, which is an unwanted by-product. Therefore, the objective of this study is to investigate the monoglyceride production from palm oil and glycerol in non-catalytic and solvent-free system, comparing to the IPA adding system. The glycerol conversion was measured for screening the suitable reactor loading and volume of solvent at constant temperature (260 ºC) in 30-90 minutes.

2. Materials and methods

2.1. Materials

The n-heptane and IPA were purchased from Fischer Chemical. High-purity (99%) reagents, including tripalmitin, dipalmitin, monopalmitin, triolein, diolein, monoolein, palmitic acid, and oleic acid were supplied by Sigma-Aldirch. The refined palm oil was purchased from Morakot Industries Public Co., Ltd. The average molecular weight of palm oil was 840 as describe in our previous work [12]. The crude glycerol sample and its certificate of analysis were donated from Bangchak Biofuel Co., Ltd.

Table 1 Optimal condition for monoglyceride production as reported in the literatures.

Feedstock	Catalyst	Solvent	Optimal condition			%MG
			Temperature (°C)	Time (hr.)	Molar ratio (Feed:glycerol)	
Olive oil [5]	Immobilized Lipase (*Candida rugose*)	Isopropanol/ *tert*-butanol	40	4	1:2	55.8
Palmitic acid [6]	Novozyme 435	Act-ECO_2 [b]	50	5	1:3	58.68
Soybean oil [7]	[Bmim]Im [a]	–	200	3	1:6	69
Palm oil [8]	CuO-nano	–	240	1	2:1	25
Palm oil [8]	CuO-nano/ NaOH 0.3 wt%	–	240	0.5	2:1	~71
Methyl oleate [9]	MgO	–	250	2	1:4.5	77
Triglyceride [3]	KOH, $Ca(OH)_2$	N/R	255	N/R	N/R	50
Canola oil [10]	-	$CO_2/N_2/H_2O$ 4–8% (w/w)	250	9–10	1:34	66–71
Corn oil [11]	0.1 wt% CaO-CeO_2	–	200	1	1:3	90
Sunflower oil [4]	–	Acetone	250	2	1:5	52
Triolein [3]	–	Isopropanol	260	2.5	1:5	67.80
Triolein [3]	–	–	260	1.5	1:5	44.45
Palm oil [4]	–	–	240	1	2:1	5

N/R is not reported.
[a] [Bmim]Im is 1-Butyl-3-Methylimidazolium imidazolide.
[b] Act-ECO2 is acetone expanded carbon dioxide.

2.2. Glycerolysis of Triglyceride (Palm Oil)

Predetermined amounts of palm oil, glycerol and isopropanol were added into a batch reactor (4.2 mL) and placed in a high-temperature fluidized sand bath as show in Fig. 1. The temperature in the reactor was controlled to constant temperature at 260 °C with heating time of 30-150 min. The reaction was carried out with constant shaking (60 times per min) by a mechanical arm during the experiment progress. After the reaction complete, the reactor was rapidly cooled in water bath and the product in the reactor was collected at room temperature. In case of IPA added, the IPA was evaporated in a water bath at 85 °C for 8 hours before analysis.

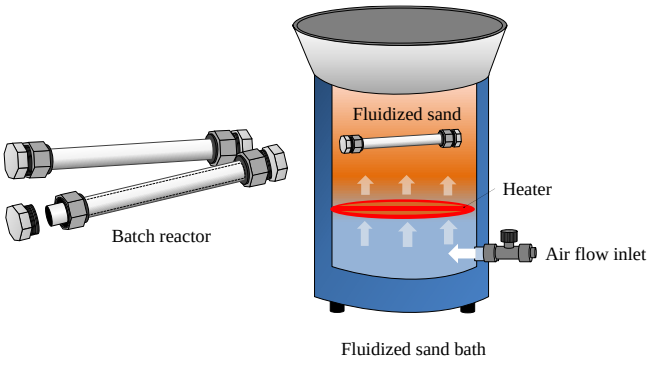

Fig. 2. The fluidized sand bath and the tube batch reactor.

2.3. GC Analysis

Amounts of monoglyceride, diglyceride, and triglyceride were standardized by the external standard method. The samples diluted in ethanol were analyzed by gas chromatography (7890A, Agilent). The GC was equipped with capillary column MXT-WAX (30 m, 250 µm ID, 0.1 µm, Agilent Technologies, Inc.). The column initial temperature is 50 °C, and the final constant temperature is 370 °C. Both injector and flame ionization detector (FID) were set at 380 °C. Glycerol conversion was calculated using Eq. (1).

$$\text{Conversion } (\%) = \left(1 - \frac{\text{Glycerol}_{\text{final}}}{\text{Glycerol}_{\text{initial}}}\right) \times 100\ \% \quad (1)$$

2.4. GC-MS Analysis

The sample was qualitatively analyzed by GC-MS (Shimudzu, model QP2010) to identify the fatty acid esters. The analytical conditions were reported in detail elsewhere [13]. In brief, the capillary column, J&W Scientific, model DB-5ms (30 m × 0.25 µm × 0.25 mm) was employed. The column oven was held at 50 °C for 5 min, and then increased to 200 °C at 15 °C/min. The molecular weight scan range was 50-850 m/z. Injection

port and interface temperature were set at 200 °C and 230 °C, respectively.

2.5. Experimental Design

To estimate the effects of temperature, reaction time, and molar ratio of reactants, triplicate central composite design (CCD), with an overall experiment of 48 runs, was employed to investigate the effects of the parameters shown in Table 2. The parameters and levels were selected to lie within ranges similar to those reported in studies of non-catalytic systems [4, 5]. To minimize uncontrolled factors, all experiments were conducted in random order, as shown in Table 3.

Table 2. The selected parameters and levels for the CCD.

Factors	Parameter	Unit	Levels		
			-1	0	1
A	Temperature	°C	220	240	260
B	Reaction time	min	30	90	150
C	Isopropanol molar ratio	-	0	15	30

The CCD experimental data were used to develop a polynomial equation that correlates %monoglyceride content (%MG), %diglyceride content (%DG), %fatty acid content (%FA), and %triglyceride content (%TG) as the responses. Equation (2) shows the general form of the second-order polynomial equation [18]:

$$y = \beta_0 + \sum_{(j=1)}^{k} \beta_j X_j + \sum_{(i<j)} \sum \beta_{ij} X_i X_j + \sum_{(j=1)}^{k} \beta_{jj} X_j^2 + \varepsilon \qquad (2)$$

where y is the value of the response, and *Xj* and *XiXj* are the main effects and interaction effects, respectively. β_0 is the overall mean. β_j, β_{jj}, and β_{ij} are the coefficients of linear, quadratic, and interaction effects, respectively. ε is random error.

3. Results and Discussion

3.1. Effects of Reaction Time

A monoglyceride yield (MG Yield) of 0.1% was observed at reaction time of 30 minutes as can be seen in Fig. 3.

The glycerolysis reaction at reaction time of 90 and 150 min produced the higher MG yield. Notes that a reaction time of 30 minutes is too short for the glycerolysis of palm oil. Similar to those reported in literatures [4, 5, 14], for catalyst-free glycerolysis of oils were similarly process, which required reaction time up to 45 minutes. It was reported that the glycerolysis of triolein in non-catalytic solvent-free system has monoglyceride content of 44.45 ± 0.35 % [3].

Table 3. Experimental data for the central composite design (CCD) and results.

Run order	Temp. (°C)	Reaction time (min)	Molar ratio (X:5:1)	%MG	%TG
9	220	30	0	0.02	94.11
1	220	30	0	0.05	95.83
34	220	30	0	0.01	95.80
37	260	30	0	0.04	95.02
48	260	30	0	0.00	98.20
29	260	30	0	0.01	94.11
21	220	150	0	0.42	94.72
2	220	150	0	0.13	98.08
47	220	150	0	0.19	95.94
16[a]	260	150	0	54.72	23.97
30[a]	260	150	0	51.12	24.09
22[a]	260	150	0	54.31	22.60
14	220	30	30	0.00	93.01
26	220	30	30	0.00	94.21
17	220	30	30	0.00	96.15
6	260	30	30	0.16	76.40
7	260	30	30	0.11	83.51
39	260	30	30	0.31	76.15
35	220	150	30	0.02	93.96
42	220	150	30	0.92	82.85
38	220	150	30	0.04	77.89
33	260	150	30	37.30	13.82
25	260	150	30	37.37	24.05
12	260	150	30	37.77	14.06
24	220	90	15	0.02	93.32
46	220	90	15	0.07	86.96
11	220	90	15	0.69	84.83
23	260	90	15	11.77	62.34
15	260	90	15	13.65	59.85
45	260	90	15	14.53	56.58
4	240	30	15	0.00	91.94
18	240	30	15	0.00	92.55
10	240	30	15	0.00	97.41
5	240	150	15	30.85	37.41
20	240	150	15	31.82	53.73
19	240	150	15	28.65	56.85
40	240	90	0	1.11	94.42
8	240	90	0	0.72	91.54
36	240	90	0	1.30	88.67
13	240	90	30	4.19	81.40
44	240	90	30	3.17	88.61
27	240	90	30	3.55	76.38
43	240	90	15	4.95	68.81
32	240	90	15	5.54	69.80
28	240	90	15	7.08	74.41
31	240	90	15	6.55	57.42
41	240	90	15	3.24	69.99
3	240	90	15	6.60	78.66

[a] Maximum monoglyceride content (%MG)

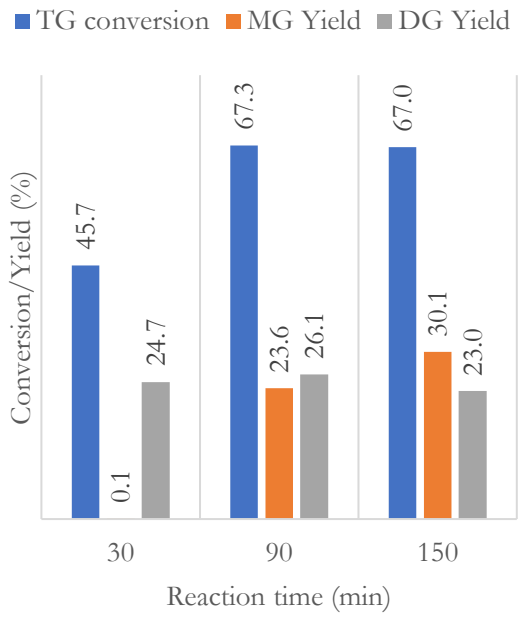

Fig. 3. Effect of reaction time on glycerolysis of palm oil at 260 °C at 60 % reactor loading, molar ratio of oil to glycerol to isopropanol (1:5:15).

To enhance the triglyceride conversion, increasing of temperature was generally employed in the non-catalytic process. However, the excess temperature could promote thermal degradation of triglyceride and glycerol. It was reported that thermal degradation of triglyceride takes place at 280 °C. The products of the degradation reaction are small molecules of fatty acids [15]. Those products could contaminate in the glycerolysis reaction because the fatty acids are the intermediate of this reaction [3]. For glycerol, the degradation temperature is approximately 250 – 300 °C depending on the reaction time. The products of glycerol degradation are water and acrolein that formed by ionic and radical reactions. When the reaction is prolonged or the temperature is enhanced, the acrolein could decompose to acrylic acid, acrylamide, and polyarolein [16]. Therefore, the maximum reaction temperature in this study is limited at 260 °C.

3.2. Effects of Reactor Loading

To comparison of the MG yield obtained from 40% to 80% reactor loading, the molar ratio of palm oil to glycerol to isopropanol was fixed at 1:5:15. The results are depicted in Fig 4. The glycerolysis reaction with a low reactor loading (40%) gave a higher glycerol conversion when compared to 60 % and 80 % reactor loading. A 37.4% of MG yield was observed when the reactor loading is 40%; whereas the MG yield decreased to 28.6% when use the 80% of reactor loading in 150 minutes of reaction time.

The drawing of the reactants in a tube batch reactor is shown in Fig. 5. The minimal head free space suggests that a small volume of isopropanol could be vaporized to reside in their space. The added isopropanol is mostly relied on the liquid phase of the mixture reactants during the reaction. When increasing of reactor loading at a same molar ratio (oil: glycerol: isopropanol = 1:5:15), the head free space was decreased; thus, less isopropanol can vaporize into head free space.

Likewise, the effects of dilution of the reactants is due to excess amount of isopropanol in reactor. The isopropanol molecule could obstruct the reaction between triglyceride and glycerol. Furthermore, the isopropanol is reactive at supercritical condition. Consequently, the transesterification of triglyceride and isopropanol could take place [17]. The GC-MS analysis was preformed to proof this hypothesis as shown in Section 3.5.

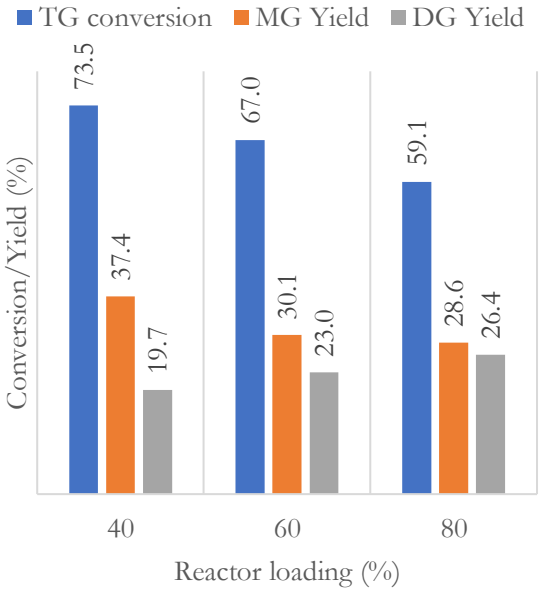

Fig. 4. Effect of reaction time on glycerolysis of palm oil at 260 °C, 150 min. Molar ratio of oil to glycerol to isopropanol (1:5:15).

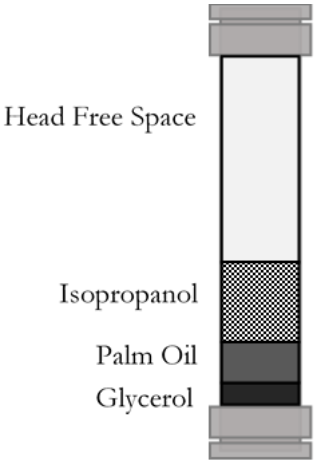

Fig. 5. The Proportion of reactants in a tube batch reactor at ambient conditions.

3.3. Effect of Isopropanol Addition

In this section, the experiments were studied by reactor loading to 60% using a palm oil to glycerol molar ratio of 1:5 and vary molar ratios of isopropanol from 0-30 with 150 minutes of reaction time. The results are illustrated in Fig. 6. After reaction complete, the 30.1% of a MG yield was observed at molar ratio of palm oil: glycerol: isopropanol of 1:5:15. The reaction without adding of isopropanol shown the lowest MG yield (0.3%). It can be noted that the addition of isopropanol resulted in significant increase in glycerol conversion. The presence of isopropanol, which exists as vapor within head free space of the reactor enhances the pressure in the reactor and improving the MG yield.

To understand the effect of isopropanol addition on glycerol conversion, the molar ratio of palm oil: glycerol: isopropanol was increased to 1:5:30 with 150 minutes of reaction time. The results are presented in Fig. 6. When amount of isopropanol is increased, the amounts of glycerol and palm oil that could be load to the reactor decreases resulting in the dilution of glycerol and palm oil in the reaction thus decreasing the MG yield (21.1%). Moreover, the transesterification of isopropanol and palm oil could occur as mention in Section 3.2.

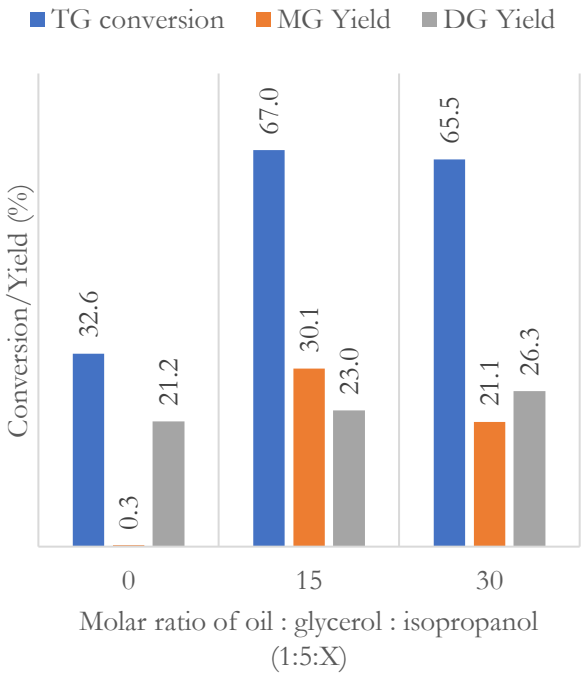

Fig. 6. Effect of isopropanol addition on glycerolysis of palm oil at 260 °C, 150 min and 60% reactor loading. Molar ratio of oil to glycerol to isopropanol (1:5:X).

3.4. Analysis of Variance and Regression Model

The experimental results (Table 3) were subjected to an analysis of variance (ANOVA). From this analysis, an ANOVA table was obtained for each output as a response variable: monoglyceride (%MG) and triglyceride (%TG) contents. The ANOVAs employing %MG and %TG as the response variable yielded statistically meaningful regression models, which are shown in Eq. (3) and (4), respectively:

$$\%MG = 6.21 + 10.32A + 12.13B - 1.27C + 11.22AB - 1.93AC - 1.95BC + 9.04B^2 - 3.84C^2 \quad (3)$$

$$\%TG = 74.18 - 17.78A - 18.03B - 5.14C - 14.81AB - 7.07B^2 + 8.11C^2 \quad (4)$$

where A represents temperature, B represents reaction duration, and C represents the molar ratio of IPA to palm oil. All parameters are in coded unit.

The coefficients of determination (R^2) of Eq. (3) and (4) are 0.97 and 0.94, respectively, both quite high. It is clear from the results in Tables 4 and 5 that temperature's effect (A) is slightly weaker than that of reaction duration (B). For both %MG and %TG analyses, the molar ratio of IPA to palm oil shows the weakest effect (C) of the three effects. In Eq. (3), the negative signs on the coefficients of all three of these effects indicate that %TG is reduced when any of these factors is increased.

The ANOVA tables for %MG and %TG are provided in Tables 4 and 5, respectively. It should be noted that for a statistical significance level of $p < 0.01$, the molar ratio of IPA to palm oil shows a significant effect on %TG but an insignificant effect on %MG.

Table 4. Analysis of variance (ANOVA) employing monoglyceride as response.

Source	Sum of Squares	df	Mean Square	F Value	p–Value
Model	11576.72	8	1447.09	160.86	< 0.0001
A	3193.43	1	3193.43	354.99	< 0.0001
B	4412.85	1	4412.85	490.54	< 0.0001
C	48.57	1	48.57	5.40	0.0255
AB	3020.72	1	3020.72	335.79	< 0.0001
AC	89.17	1	89.17	9.91	0.0031
BC	91.52	1	91.52	10.17	0.0028
B^2	718.87	1	718.87	79.91	< 0.0001
C^2	129.81	1	129.81	14.43	0.0005
Residual	350.84	39	9.00		
Lack of Fit	326.81	6	54.47	74.79	< 0.0001
Pure Error	24.03	33	0.73		
Core Total	11927.57	47			

However, for products obtained from the reaction at optimal conditions of 260 °C and 150 min, the %MG values achieved in the solvent-free process (runs 16, 30, and 22) are approximately 15% higher than those achieved in the IPA-added process (runs 33, 25, and 12). For comparison, the glycerolysis of triolein in a non-catalytic, solvent-free system has been reported to yield a monoglyceride content of 44.45 ± 0.35 % [3].

The maximum monoglyceride content (%MG) was observed at the highest measured levels of temperature and reaction duration. At these levels, the triglyceride content (%TG) was approximately 20%. Further increase in temperature is not suitable because triglyceride is prone to decompose at 280 °C. Furthermore, extending the reaction duration to 180 min did not significantly enhance the %MG. Furthermore, at this duration the %TG fell below 20%, indicating triglyceride degradation. Therefore, the maximum reaction temperature and duration were limited at 260 °C and 150 min, respectively.

Table 5. Analysis of variance (ANOVA) employing triglyceride as response.

Source	Sum of Squares	df	Mean Square	F Value	p–Value
Model	25989.81	6	4331.64	100.60	< 0.0001
A	9480.31	1	9480.31	220.17	< 0.0001
B	9749.18	1	9749.18	226.41	< 0.0001
C	792.86	1	792.86	18.41	0.0001
AB	5266.69	1	5266.69	122.31	< 0.0001
B^2	440.32	1	440.32	10.23	0.0027
C^2	579.17	1	579.17	13.45	0.0007
Residual	1765.43	41	43.06		
Lack of Fit	832.42	8	104.05	3.68	0.0036
Pure Error	933.01	33	28.27		
Core Total	27755.25	47			

3.5. GC-MS analysis

The addition of isopropanol can reduce the viscosity of reaction mixture for the continuous process, but the exceeding amount of isopropanol reduces the %MG and generates the fatty acid propyl esters as notice by GC-MS analysis as shown in Fig. 7. It is clear that the reaction product obtained at a reaction temperature of 260 °C, a molar ratio of glycerol to palm oil of 5:1, a molar ratio of isopropanol to palm oil of 30:1 contaminates by isopropyl fatty acid esters.

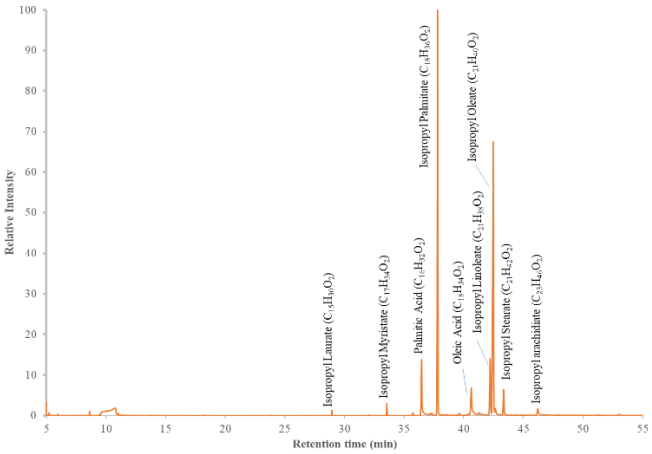

Fig. 7. Chromatogram of product obtained at a reaction temperature of 260 °C, a molar ratio of glycerol to palm oil of 5:1, a molar ratio of isopropanol to palm oil of 30:1, and a reaction duration of 150 min.

However, even product obtained using the isopropanol added process is probably suitable for biolubricant applications because it contains the isopropyl fatty acid esters [19]. In the isopropanol added process, palm oil as a feedstock yields a lower %MG than triolein is cited in the literature as yielding. This is probably because of the effects of saturated FAs (palmitic and stearic acids) in palm oil.

3.6. Preliminary Study of Monoglyceride Production with Crude Glycerol

After the optimal condition for monoglyceride production by pure glycerol was obtained, the crude glycerol was employed as raw material. The properties crude glycerol is shown in Table 6.

Table 6. Properties of crude glycerol obtained biodiesel production plant.

Property	Testing method	Limit	Value
Glycerol content (%wt.)	BS 5711 part 3:1979	80 Min.	80.82
Water content (%wt.)	ISO 2097–1972	-	13.44
Methanol content (%wt.)	BS 5711 part 11: 1979	0.5 Max.	0.007
Density at 30 °C	Digital Density Meter	-	1.25
Salt content	BS 5711 part 12: 1979	7 Max.	3.58
Ash content	ISO 2098–1972	-	1.05
MONG [a]	ISO 2464–1973	-	1.10
pH		-	5.78

[a] Organic matters (non-glycerol)

The crude glycerol has the water content of 13.44 %wt. and the glycerol content of 80.82 %wt. Thus, the lower %MG in the product could be expected. Furthermore, the crude glycerol contaminates with 0.007%wt. of methanol. The formation of methyl ester could occur because the reaction temperature is higher than critical temperature of methanol. The density of crude glycerol is slightly higher than that of 99% pure glycerol (1.26 g/cm^3). In Bangchak Biofuel Co., Ltd., the salts in crude glycerol becomes from the alkaline catalyst and neutralization acid which are KOH and H_3PO_4, respectively. After complete combustion, the ash could be used as the fertilizer because it consists of K_2PO_3. The monoglyceride non-glycerol (MONG) is calculated from 100 subtract by the summation of glycerol, water, methanol, salt, and ash contents. The reaction product obtained from crude glycerol is revealed in Fig. 7.

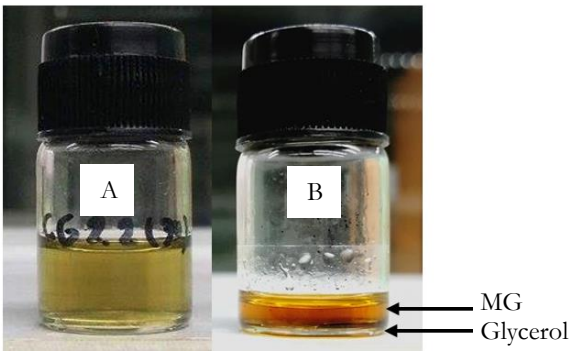

Fig. 7. The reaction products obtained at a reaction temperature of 260 ºC, a molar ratio of glycerol to palm oil of 5:1, a molar ratio of isopropanol to palm oil of 30:1, and a reaction duration of 150 min (A) product obtained from the complete reaction, and (B) product after isopropanol was evaporated.

At reaction temperature of 260 ºC, molar ratio between palm oil to glycerol of 1:5, and reaction time of 150 min, the %MG obtained from pure glycerol was 53.03% while the %MG of 46.58% was observed when using crude glycerol as reactant. The product was contaminated by 0.5%wt. of fatty acid methyl esters. The effects of water in crude glycerol would be investigated in the further study.

4. Conclusion

Isopropanol has been shown as an effective solvent for the glycerolysis of palm oil. The isopropanol addition and reactor loading are the parameters that affecting to glycerol conversion. The optimal conditions were set as 260 ºC in 150 minutes and 60 % reactor loading, giving the highest monoglyceride yield (37.4%). The molar ratio of glycerol to palm oil to isopropanol is 5:1:15, respectively. Furthermore, the IPA-added process generated <10% FA propyl esters when conducted at optimal temperature and duration (260 °C and 150 min). Temperature and reaction duration show significant effects on monoglyceride content (%MG) and triglyceride content (%TG), while the ratio of isopropanol (IPA) to palm oil shows the weakest influence on %MG and %TG. Monoglyceride yield is approximately 15% higher in the solvent-free process than in the IPA-added process. Furthermore, the IPA-added process generated < 10% FA propyl esters when conducted at optimal temperature and duration (260 °C and 150 min).

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