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PREPARATION OF POLYGLYCEROL FROM PALM-BIODIESEL CRUDE GLYCERIN

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ABSTRACT

The demand for green energy derived from plant feedstock has been the determining factor for the abundant supply of glycerol. Concerted efforts have been made to expand the current scope of glycerol application. This study describes the rapid polymerisation of crude glycerol directly obtained from the biodiesel process to produce polyglycerol via microwave heating technology. Crude glycerol used in this study was obtained from four different biodiesel pilot plants. Commercially pure glycerol was used as the control in all the parameters studied. The highest percent yield of polyglycerol obtained was 94.94% when heated at 250°C for 60 min under microwave irradiation. Crude glycerol that gave the highest polyglycerol percentage was found to contain the highest percent of soap (12.5%). It was anticipated that a high conversion of glycerol was due to the soap contained in the crude glycerol. This study showed that biodiesel-based crude glycerol with appropriate soap content could be used directly as a raw material in polyglycerol production.

Keywords: crude glycerol, polyglycerol, soap, dehydroplymerisation, microwave.

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INTRODUCTION

The utilisation of glycerol for the synthesis of value-added chemical is a topic of great industrial interest because glycerol can be formed in large amounts during the production of biodiesel. For every 3 moles of methyl ester produced, 1 mole of glycerol is obtained as a by-product, which is equivalent to approximately 10% of the total product by mass (Smirnovs *et al.*, 2008). Increasing demand of biodiesel will lead to larger amount of glycerol accumulated as by-products of which there is currently insufficient conventional use. Its effective utilisation will be a key factor to the success of biodiesel commercialisation and further development (Beltramini and Zhou, 2010).

Crude glycerol generated during the biodiesel production process contains impurities such as methanol, water, inorganic salts (catalyst residue), free fatty acids, unreacted mono-, di- and triglycerides, methyl esters and a variety of other organic materials (Chiu *et al.*, 2005; Yori *et al.*, 2007). In order for biodiesel-based crude glycerol to be use in the industrial application such as for food, cosmetics, or pharmaceuticals, it requires expensive purification method. As a consequence, crude glycerol is claimed to be a waste with an associated disposal cost (Pinar Calik *et al.*, 2008). Evidently, the development of new routes to convert crude glycerol into higher value products is very crucial.

Crude glycerol has been studied with respect to wide range of fields such as combustion (Johnson *et al.*, 2007), anaerobic digestion (Holm-Nielson *et al.*, 2008), or feeding for various animals such as pigs (Kijora *et al.*, 1996), broiler chickens (Simon *et al.*, 1996), and laying hens (Yalcin *et al.*, 2010). Altering crude glycerol into value-added products through thermochemical methods (Alhanash *et al.*, 2008;

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Dasari *et al.*, 2005; Chiu *et al.*, 2006) or biological methods (Lee *et al.*, 2001; Papanikolaou *et al.*, 2002; 2004; Rymowicz *et al.*, 2006) is another alternative for utilising this waste stream.

In the present study, polyglycerol from crude glycerol was synthesised. This study was undertaken to investigate the polymerisation of biodiesel-based crude glycerol via microwave irradiation. Preparation of polyglycerol was known to be carried out at a reaction time of minimum 5 hr to 72 hr and high reaction temperature required (Endo and Omori, 2008; Jacobson *et al.*, 1990; Stuhler, 1985; Eshuis *et al.*, 1998; Lemke, 2002). The long reaction time and high reaction temperature can be considered a drawback. To circumvent the problem microwave heating method was used to expedite the self-polymerisation of the crude glycerol. There have been many recent reports of remarkable decreases in reaction time for reactions carried out under microwave irradiation (Pelle *et al.*, 2001; A Sosnik *et al.*, 2011; Vanier, 2007; Rostami *et al.*, 2012). The effect of reaction time and temperature on the polymerisation reaction and the effect of crude glycerol with different characteristic in the formation of polyglycerol oligomers were also studied.

Polyglycerol has been used as feed material for the production of polyglycerol esters, a nonionic surfactant commonly incorporated as emulsifier in food products. Polyglycerol is commonly prepared by mixing and heating glycerol in the presence of an alkali catalyst, such as sodium or potassium hydroxide; alkaline carbonates such as potassium carbonate; and alkaline earth metal hydroxide such as calcium hydroxide at an elevated temperature from 100°C to 300°C (Endo and Omori, 2008; Johnson and Tocani, 2007; Stuhler, 1985; Eshuis *et al.*, 1998; Babayan, 1967; Lemke, 2002). A variation in temperature and the duration of the process determines the degree of polymerisation. Generally, polymerisation of glycerol results in the formation of ether bonds between the hydroxyl groups of two glycerol molecules, while water is released as a by-product.

MATERIALS AND METHODS

Materials

Five samples of crude glycerol of various qualities were collected from local biodiesel plants. The crude glycerol obtained was analysed for different physical properties such as ash value [ISO 2098 - 1972 (E)], soap content [Emery Oleochemical (Malaysia) Sdn Bhd in-house method number QC007:1997], moisture content [Standard method, ISO 662:1998(E) (Karl Fischer titration)] and glycerol content (International Standard, ISO 2879-1975). The pH of crude glycerol in 10% solution was determined using Hanna instrument, HI 9021 microprocessor pH meter.

Methods

Crude glycerol polymerisations were carried out in a two-neck round bottom flask equipped with a mechanical stirrer and condenser system. The second neck was connected to a temperature sensor of the microwave reactor (Model CEM MARSXpress, CEM Co., Matthew, North Carolina, USA). The reaction was at atmospheric pressure and without any addition of catalyst. Crude glycerol (125 g) was heated under microwaves irradiation to the desired temperature and reaction time. Commercial pure glycerol was used as the control raw material to produce polyglycerol. The distribution of polyglycerol monomer obtained was analysed using high performance liquid chromatography (HPLC). Polyglycerol sample of 0.02 g was diluted in 1.5 ml of distilled water and was stirred using vortex mixer. A 100 μl sample was eluted through Hypersil 10 μm NH_2 (250 mm x 4.6 mm) column using 85% acetonitrile solution at a flow rate of 1 ml min^{-1}. The resolved components were detected by evaporated light scattering detector (ELSD) 800 (Alltech). The HPLC was calibrated using tripalmitin as internal standard.

RESULTS AND DISCUSSION

Properties of Crude Glycerol

The composition and quality of crude glycerol collected from local biodiesel plants varied in terms of glycerol content, pH, soap and moisture content. This was because the feed material used were different and subsequently led to a variation in the quality of their by-product, glycerol. All glycerol samples collected were randomly termed as Cg-A, Cg-B, Cg-C, Cg-D and Cg-E. Their characteristics are as tabulated in *Table 1.*

Sample Cg-A had the highest percentage of ash content since the biodiesel process used crude palm oil (CPO) as feed material. CPO contains higher amounts of impurities or other components each as fruit fibres, nut shells, free fatty acids, phospholipids, trace metals, carotenoids, tocopherols or tocotrienols, oxidation products and sterols. These impurities may contribute to the high ash content of 7.12%. The rest of the crude glycerol samples were produced from the biodiesel process which used refined, bleached and deodorised (RBD) palm oil as feed material. Their ash content was in the range of 3.04% to 5.26%.

TABLE 1. PROPERTIES OF CRUDE GLYCEROL FROM LOCAL BIODIESEL PLANTS AND PURE GLYCEROL

Analysis	Glycerol content (%)	Moisture content (%)	Ash content (%)	Soap content (%)	pH value	Appearance	Feedstock
Commercial pure glycerol	99.5	0.5	0	0	6.7	Colourless liquid	Commercial sample – depend on the particular company
Cg-A	88.56	3.41	7.12	0.91	6.68	Dark brown liquid	CPO
Cg-B	85.2	8.11	5.26	1.43	7.73	Dark brown semi solid	RBDPO
Cg-C	93.4	1.43	3.43	1.74	12.13	Dark brown semi solid	RBDPO
Cg-D	88.61	2.24	3.04	6.11	9.00	Dark brown paste	RBDPO
Cg-E	75.1	6.88	5.52	12.5	8.84	Dark brown paste	RBDPO

Note: RBDPO - refined, bleached and deodorised palm oil.
CPO - Crude palm oil.

Among all the five crude glycerol samples, Cg-C had the highest glycerol content of 93.4% and surprisingly, the pH was also very high, indicating that the crude glycerol might have undergone some pre-treatment process. The soap content of this sample, however, was amongst the lower range of soap content which is 1.74%. Nevertheless, in terms of soap content, Cg-E had the highest soap content of 12.5%, while giving the least amount of glycerol content of only 75.1%.

Polymerisation of Crude Glycerol to Produce Polyglycerol

The polymerisation of crude glycerol to produce polyglycerol was done under microwave irradiation using five different types of crude glycerol distinguished by their characteristics. The highest polyglycerol % yield was 94.94% at reaction temperature of 250°C in 60 min. Microwave assisted organic synthesis is known for the spectacular accelerations produced in many reactions as a consequence of heating rates that cannot be reproduced by classical heating. As a result, higher yield, milder reaction conditions and shorter reaction times can be achieved (Loupy, 2003; De La Hoz *et al.*, 2004). This effect is particularly important in the preparation of polyglycerol that is known to have a long reaction time (Endo and Omori, 2008; Jacobson *et al.*, 1990; Stuhler, 1985; Eshuis *et al.*, 1998; Lemke, 2002).

The results of the time course study are presented in *Figure 1*. It is shown here that as reaction time increased, the yield of crude glycerol to polyglycerol increased with each sample, thus giving a different degree of conversion of the crude glycerol to polyglycerol.

Effect of Reaction Time and Temperature

The highest yield of polyglycerol for Cg-A, Cg-B, Cg-C, Cg-D and Cg-E is at 60 min under microwave irradiation. It was reported that basically the reaction time for polymerisation of glycerol was dependent in particular on the reaction temperature and on the content of the catalyst. The reaction time was relatively short at a higher reaction temperature and appropriate amount of catalyst (Stuhler, 1985).

Reaction temperatures which ranged from 150°C to 250°C were used to observe the effect of temperatures on the polymerisation of crude glycerol without a catalyst for 60 min. *Figure 2* demonstrates the yield of the reaction product mixtures. All samples exhibited similar trends where the yield increased when reaction temperatures increased. For Cg-A, Cg-B and pure glycerol there were no conversions when the reaction was conducted at temperatures below 200°C. For Cg-C, the reaction started between 150°C to 180°C, but with only a minimal amount of polyglycerol yield. For Cg-D and Cg-E, both crude glycerols started to polymerise at a temperature as low as 150°C. Polymerisation proceeded slowly below 190°C (Stuhler, 1985). This study shows that the conversion of all the crude glycerol used in this study is below 15% when the reaction temperature is at 180°C under microwave irradiation.

Effect of Soap Content and pH of Crude Glycerol

The polymerisation of five crude glycerol samples by microwave irradiation at 250°C for 60 min indicated that the yield of polyglycerol produced, varied for each crude glycerol tested.

As depicted in *Table 2*, the catalytic effect of soap on yield of crude glycerol to polyglycerol for all five

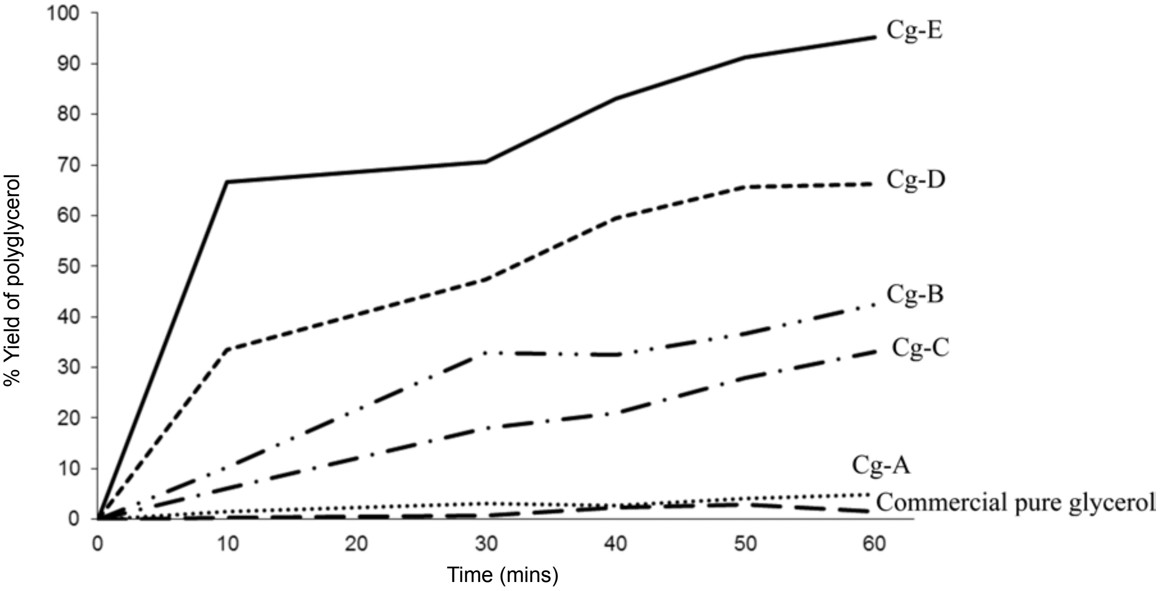


Figure 1. The effect of reaction time on the conversion of crude glycerol to polyglycerol.
The reaction was done at 250°C at different reaction time (as in Figure 1*) without any addition of catalyst.*

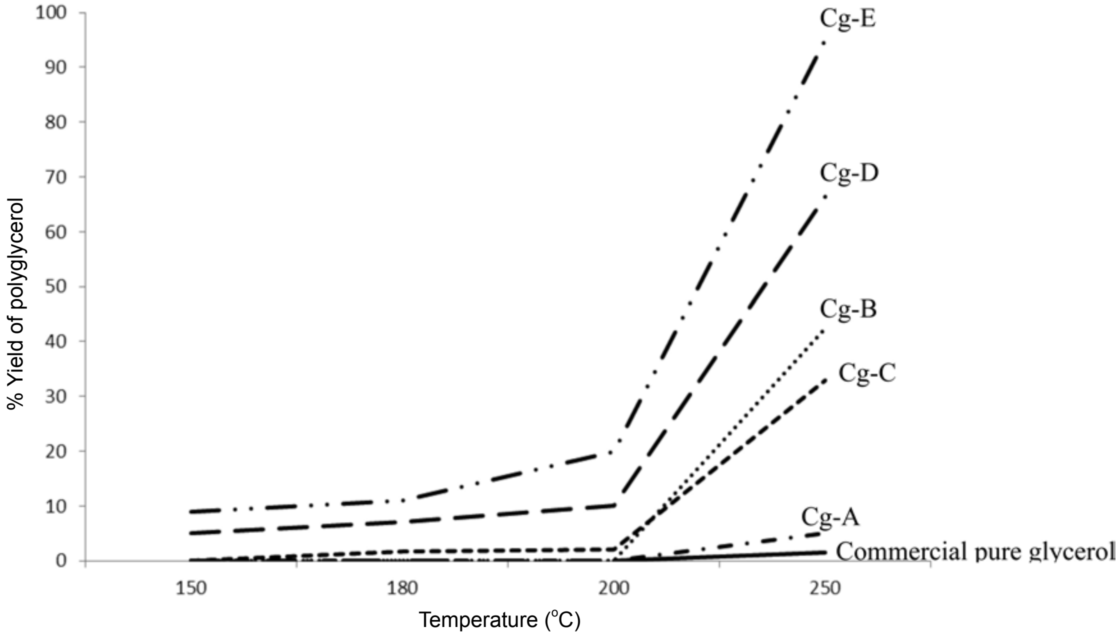


Figure 2. The effect of reaction temperature on the conversion of crude glycerol to polyglycerol. The reaction was done at multiple reaction temperature (as in Figure 2*) for 60 min without any addition of catalyst.*

crude glycerol samples followed the succeeding order of increasing soap content:

$$\text{Cg-A} < \text{Cg-B} \approx \text{Cg-C} < \text{Cg-D} < \text{Cg-E}$$
$$\xrightarrow{\hspace{6cm}}$$
Crude glycerol with increasing soap content

This was in agreement with the results reported by Soi *et al.*, in 2010, where they had discovered that fatty acid salts of alkaline metal (soap) gave good yield and selectivity in producing polyglycerol from crude glycerol. Garti *et al.*, in 1981 in their study pointed out, alkaline conditions were needed for catalysis in polyglycerol preparation from glycerol. Martin and Richter, in 2011 reported that, crude glycerol of biodiesel by-product contain high salt with the presence of $RCOO^{-}Na^{+}$ formed from sodium hydroxide and triglycerides. Therefore, the salt will contribute to the base characteristic of the crude glycerol.

TABLE 2. COMPOSITION OF REACTION PRODUCT MIXTURES USING VARIOUS SOURCES OF CRUDE GLYCEROL

Crude glycerol	Soap content (%)	Polyglycerol (%)					Total oligomers (%)
		Unreacted glycerol	Di-	Tri-	Tetra-	Penta-	
Commercial pure glycerol	0	98.70	1.3	0	0	0	1.30
Cg-A	0.91	95.02	4.98	0	0	0	4.98
Cg-B	1.43	57.64	39.96	2.41	0	0	42.36
Cg-C	1.74	66.64	31.60	1.76	0	0	33.36
Cg-D	6.11	33.74	55.08	9.58	1.60	0	66.26
Cg-E	12.50	5.06	47.99	26.92	14.01	6.02	94.94

Note: The reaction was done at 250°C for 60 min without any addition of catalyst.

Figure 4 shows the yield of polyglycerol versus soap content. As the soap content of the crude glycerol samples increase the polyglycerol yield increases. The highest polyglycerol yield obtained from Cg-E which is 94.94% with soap content of 12.5%. While Cg-A with 0.91% of soap content gives only 4.98% polgycerol yield.

Polyglycerol oligomers produced range from diglycerol, triglycerol, tetraglycerol and pentaglycerol. From *Table 2*, it was observed those crude glycerols that contained soap of less than 1%, gave only a small amount of diglycerol (4.98), 2.00% of soap would give diglycerol and triglycerol, but more selective to diglycerol oligomer (31.60% to 39.96%) and diminutive amounts of triglycerol (1.76% to 2.41%). Crude glycerol that contains soap content of 6.11% gave polyglycerol oligomers of diglycerol (55.08%), triglycerol (9.58%) and small amount of tetraglycerol (1.6%). Cg-E that had a soap content of 12.50% gave glycerol oligomers of diglycerol, triglycerol, tetraglycerol and pentaglycerol with the distribution of 47.99%, 29.92%, 14.01% and 6.02% respectively. It was seen that a longer chain of polyglycerol was formed as the percentage of soap increased. *Figure 5* showed the HPLC chromatogram for the polyglycerol prepared from Cg-E, while in *Figure 6* showed the HPLC chromatogram for commercial sample as the standard sample. Both chromatograms showed the polyglycerol oligomers appear at almost similar retention time.

Figure 3 illustrates that at pH 8.84, yield of polyglycerol was 94.94%. At higher pH, there is no increase in polyglycerol yield. Garti *et al.*, in 1981 reported in their study of glycerol polymerisation using glycerol anhydrous with several carbonates, hydroxides and oxides as the polymerisation

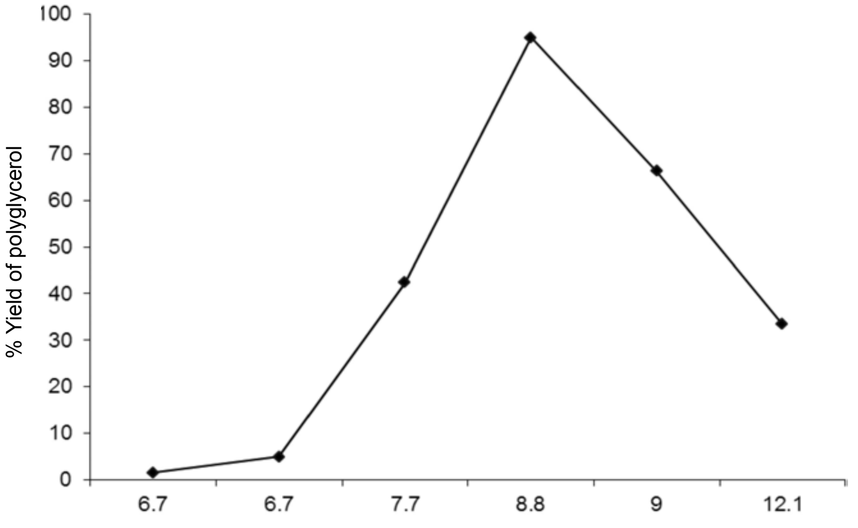
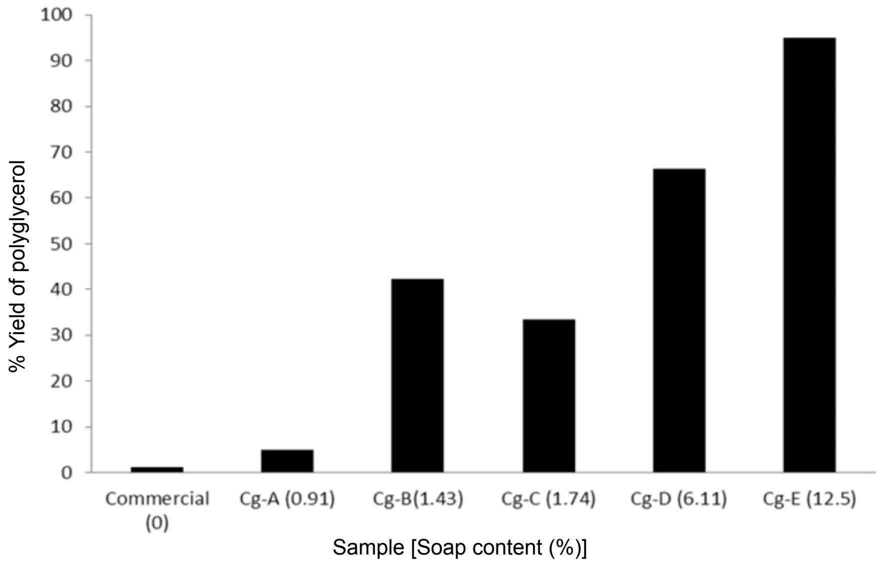


Figure 3. The pH of crude glycerol and pure glycerol versus % yield of polyglycerol.
The reaction was done at 250°C for 60 min without any addition of catalyst to the crude glycerol sample.



Note: *The reaction was done at 250°C for 60 min without any addition of catalyst.
**Commercial sample is a commercial pure glycerol sample.

Figure 4. The % yield of total oligomers in polyglycerol produced versus soap content of pure glycerol and each crude glycerols.

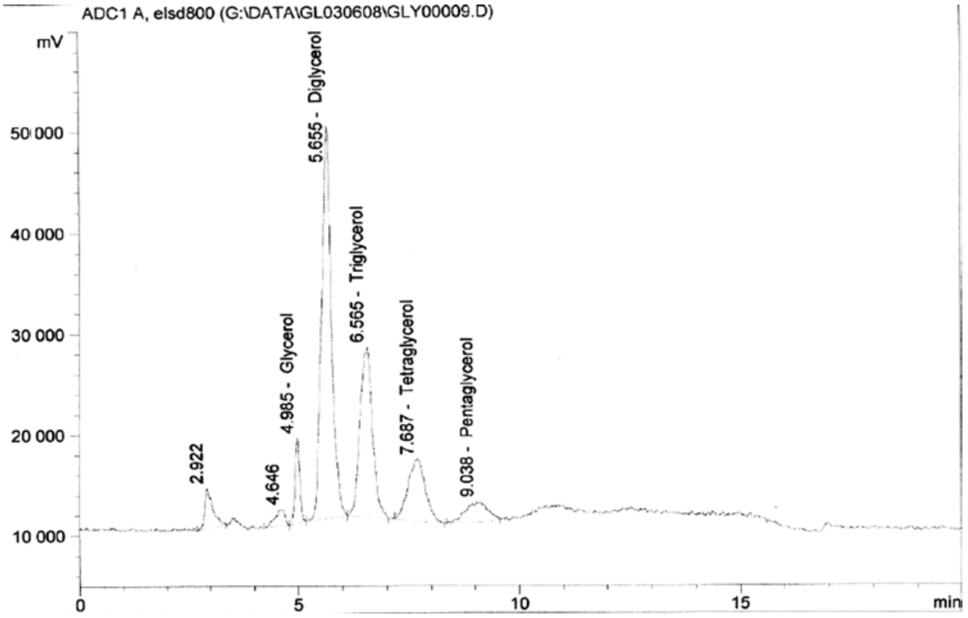


Figure 5. The high performance liquid chromatography (HPLC) chromatogram of polyglycerol obtained from Cg-E. The reaction was done at 250°C for 60 min without any addition of catalyst.

catalyst, indicated that although hydroxides were better bases, which had higher pH than carbonates; solubility of the catalyst in the glycerol and in the polymeric product at elevated temperatures influenced the rate of reaction. Thus, it was believed that the reaction was dependent on the solubility of the soap that was anticipated to catalyse the reaction. Therefore, we expect that pH of the crude glycerol maybe not be correlated with its soap content in giving a high yield of polyglycerol. Hence, the solubility of the soap might be the crucial factor. The pH of pure glycerol used in controlled experiments is 6.7 with the yield of polyglycerol is 1.5%.

Reproducibility of Crude Glycerol Polymerisation using Cg-E

The results showed that Cg-E contained catalytic amount of soap (12.50%) polymerised well at 250°C for an hour to produce 94.94% of polyglycerol comprising of four types of oligomers. *Table 3* shows that the process was reproducible with average

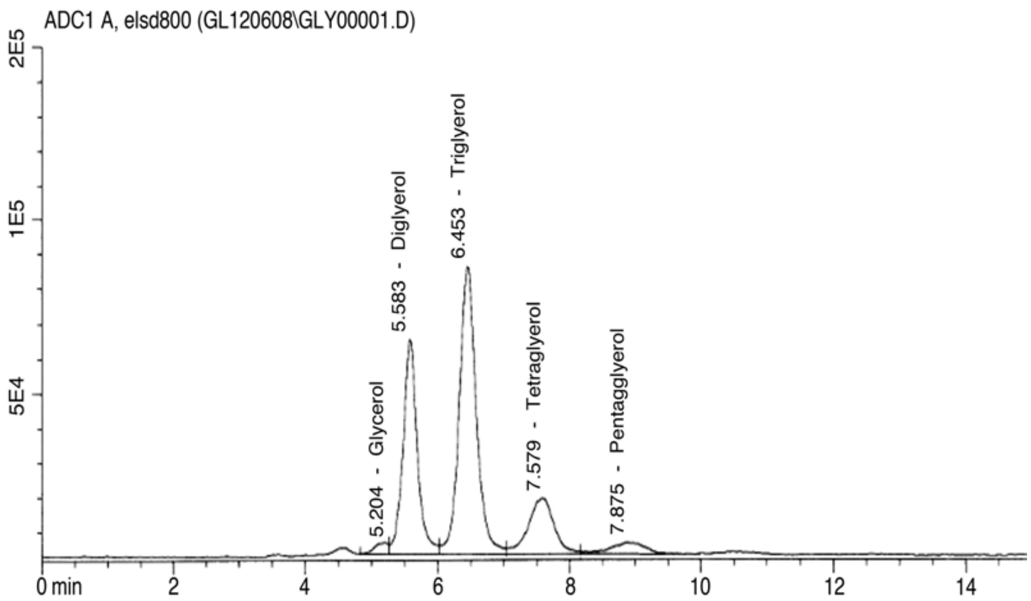


Figure 6. The high performance liquid chromatography (HPLC) chromatogram of commercial polyglycerol. The reaction was done at 250°C for 60 min without any addition of catalyst.

TABLE 3. REPRODUCIBILITY OF POLYGLYCEROL FROM CG-E

Trials	Total oligomers (%)
1	94.42 ± 0.322
2	94.35 ± 0.322
3	94.94 ± 0.322
Average	94.57
Commercial sample	100

Note: The reaction was done at 250°C for 60 min without any addition of catalyst.

percent total oligomers of polyglycerol were 94.57% after three times reproduction. However, variation in distribution of polyglycerol oligomers was identified. The study on the variation of the percent distribution of the oligomers is still in the laboratory stage. Preliminary hypothesis from the study maybe due to the stability of the crude glycerol properties, especially the moisture content of the crude glycerol. Moisture content in the crude glycerol may give impact on the way microwave react to the crude glycerol.

CONCLUSION

Polyglycerol was successfully synthesised using crude glycerol from biodiesel waste via microwave irradiation. This study has shown that 94.94% of polyglycerol could be obtained from crude glycerol with a soap content of 12.50% at a temperature of 250°C for a period of 60 min under atmospheric pressure through microwave irradiation. This study showed that biodiesel-based crude glycerol with appropriate soap content could be used directly as a raw material in polyglycerol production, where no additional catalyst is required to aid the reaction. Hence, biodiesel-based crude glycerol can be a cheapest candidate in polyglycerol production.

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4 TIMES A YEAR

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