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Synthesis of Glycerol from Peanut Oil (*Arachis hypogaea*) by Esterification Transesterification Process with Methanol

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Abstract: The synthesis of glycerol from peanut oil has been successfully carried out (*Arachis hypogaea*) This study aims to determine the glycerol content that can be obtained from the synthesis results through the esterification stage using a catalyst H2SO4 (1%) followed by trans-esterification with a catalyst KOH (1% w/v). The results of the study show that glycerol as a by-product of esterification - trans-esterification can be synthesized from peanut oil from Kendari city and methanol. The glycerol obtained showed unique physical characteristics with a clear color (not blackish brown) which gave a positive response with the Dunstan test for qualitative testing in the form of a faded purple color when heated and a color that returned to light when cooled. The results of the FTIR analysis showed OH absorption at a wavelength of 3340.71 cm-1and the CO group at 1028.13 cm-1, two specific functional groups that did not appear in the FTIR analysis results for peanut oil. The GCMS analysis results confirmed the presence of glycerol as indicated by a peak at a retention time of 15.781 minutes with a content of 79.71%.

Keywords: *Peanut, Oil, MES, Esterification, Trans-Esterification, Sulfonation.*

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I. INTRODUCTION

Efforts to increase the price of this commodity can be done by trying to convert peanut products into other, more valuable products (1). Conversion will at least cause commodity prices to remain stable even though production increases due to the availability of alternative consumption spaces. One conversion effort that can be done currently is synthesizing it into glycerol, a byproduct of the esterification of peanut oil to produce methyl ester, a biodiesel (2). Although a byproduct, this does not mean that glycerol has no importance (3). Glycerol is a raw material that can be used in many fields such as the pharmaceutical, cosmetics, food, and chemical industries (4). In addition, glycerol is also used for the synthesis of advanced products such as esters, carbonates, or other compounds with high economic value (5).

Glycerol is used as a sweetener, preservative, thickening agent, and humectant. It is often added to low-fat foods to improve texture and to baked goods to retain moisture (6). In addition, glycerol functions as a solvent for food colorings and flavorings (7). Glycerol is used in various medicines and medical products (8). It can also be used as a solution for injectable drugs and as a solvent in some pharmaceutical products. Glycerol is a key ingredient in many skin care and cosmetic products, such as soaps, lotions, and moisturizers (9). Glycerol helps moisturize the skin and maintain moisture, as a preservative, and as a thickener (10).

Esterification itself is a procedure commonly used to produce glycerol and is common in chemistry. However, due to the type of reaction, which is an equilibrium or reversible reaction, in many cases the esterification reaction is not necessarily successful (11). Therefore, to be successful, data such as the temperature used, the catalyst used, the ratio of oil to alcohol (ethanol) used, including kinetic treatments during synthesis, such as stirring speed, are absolutely necessary if we want to repeat an esterification process (12). In other words, an esterification process, especially when using a new type of oil, is not guaranteed to be successful. In line with all of that, the things that become obstacles are the oil structure contained in a type of carboxylic acid (oil) from each source is always different. Based on these things, a study will be conducted with the title: "Synthesis of Glycerol from Peanut

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Oil (*Arachis hypogaea*) by Esterification -Transesterification Process with Methanol".

II. METHOD

A. Tools and Materials

➢ *Tools*

The tools used in this research are a set of reflux tools, FTIR (Fourier Transform Infrared), hot plate, ruler, measuring cup (Pyrex) various sizes, beakers (Pyrex) various sizes, Erlenmeyer (Pyrex) various sizes, funnels (Pyrex), separating funnel (Pyrex), spatula, volumetric pipette, dropper pipette, vial bottle, dark bottle, stirring rod, thermometer, spray bottle, oven, stand, clamp, analytical balance and magnetic stirrer.

➢ *Materials*

The ingredients that will be used are peanut seeds, methanol (CH_3OH) pa, potassium hydroxide (KOH) pa, sulfuric acid (H_2SO_4) pa, sodium hydroxide (NaOH), filter paper, universal indicator, aluminum foil, tissue, and distilled water.

B. Procedures

- Sample Preparation

The peanuts used are obtained from the Konda area of South Konawe, Southeast Sulawesi.

➢ *Peanut Oil Isolation*

Isolation is done by soxhletation, namely making a sheath with a heavy weight. sample 125 g and measure the n-hexane solution using a 250 mL measuring cylinder with 4:1 ratio of sample weight. The n-hexane solution was placed in a 500 mL round-bottom flask. The solution was heated to 60-80°C for 8 hours, cooled, and then filtered with filter paper. The soxhletation results were then evaporated to obtain pure oil and solvent. The density of the pure mahogany oil obtained was determined using a pycnometer. The empty pycnometer was weighed, after which the sample was inserted into the pycnometer and reweighed. The same treatment was carried out three times (13-16).

➢ *Identification of Compounds in Peanut Oil with FTIR and GCMS*

Peanut oil will be determined its compound structure using FTIR. The composition of the compounds that make up mahogany oil and methyl ester sulfonate (MES) synthesized was identified using an IR Buck M500 scientific FTIR spectrophotometer with a wavelength of 4000-400 cm^{-1} with the equipment's condition specifications, namely scan: 32 sec/scan, resolution: 4 and pressure: 80 Torr. Interpretation of the obtained IR spectrum data was carried out by comparing the absorption of functional groups in the IR spectrum of the sample with the wavenumbers of literature or libraries (17,18).

Identification using GCMS will be obtained in the form of a chromatogram and mass spectrum. The chromatogram is useful for determining the amount of chemical components of peanut oil, while the mass spectrum is used to determine the structure of the components that make up the peanut oil. A sample of clove essential oil was taken as much as 0.05 µL. The operational conditions of the instrument were a Restek Rtx-5MS column (5% diphenyl-95% dimethyl polysiloxane), 30 m, and an injector temperature of 310°C, initial temperature 70°C (5 min), final temperature 230°C, increase 10°C/min, ion source temperature 250°C, interface temperature 300°C, pressure 28.0 kPa, total gas flow rate 91.6 mL/min, gas flow in column 0.63 mL/min, split ratio 139.1, carrier gas helium. The data were interpreted by comparing them with the literature (19,20).

➢ *Esterification and Trans-esterification*

The esterification stage is carried out by reacting 50 mL of mahogany seed oil and 33.5 methanol using sulfuric acid catalyst (H_2SO_4) 1%. The oil resulting from soxhletation was placed in a two-necked flask equipped with a condenser, thermometer, and hot plate. The sample was heated at a temperature of 65°C, then slowly added methanol and acid catalyst. The solution was stirred continuously using a magnetic stirrer for 1 hour, after which the solution was transferred to a separating funnel and left for 24 hours until two phases formed. The upper phase was free fatty acids and the lower phase was fatty acids (21-24).

The trans-esterification process is carried out by reacting the esterified oil and methanol with a molar ratio of 1:6 of the oil weight using a catalyst. KOH 1% (w/v). The esterified oil was placed in a two-necked flask equipped with a condenser, thermometer, and heater. The sample was then heated to a temperature of 60°C and methanol with KOH catalyst was added. The solution was stirred continuously using a magnetic stirrer for 1 hour, after which the solution was transferred to a separating funnel and left for 24 hours to form two phases. The upper phase was methyl ester and the lower phase was glycerol (25).

➢ *Characterization of Glycerol Products with FTIR/GCMS*

The test is similar to that for the characterization of peanut oil, with the only difference being the sample being worked on.

III. RESULTS AND DISCUSSION

An esterification reaction is a reaction between a carboxylic acid and an alcohol to form an ester. Esterification is a very slow, reversible reaction, but equilibrium is reached quickly in the presence of an acid catalyst (26). Esterification with an acid catalyst is necessary if the vegetable oil contains high levels of FFA. If the oil contains >5% FFA, it is directly transesterified with a base catalyst, where the FFA will react with the catalyst to form soap. The relatively high levels of free fatty acids (FFA) in peanut oil require an esterification process before transesterification (27).

Esterification was carried out by reacting peanut seed oil with methanol at a molar ratio of 1:15, using a strong acid catalyst, $NaHSO_3$, at 1% of the oil mass. The reaction was carried out at 65°C for one hour with continuous stirring to accelerate the reaction rate and ensure optimal interaction of the catalyst with the reactants. After the reaction stage was completed, the mixture was then left for 24 hours to allow for more effective separation of the solvent and triglycerides (28,29).

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Transesterification is a reaction that converts a triglyceride, which is an ester, into an alkyl ester and glycerol (30). Glycerol itself is actually a byproduct of transesterification, converting triglycerides (vegetable oil) into alkyl esters (31). The transesterification process in this study used a 1:6 mole ratio of oil and methanol at a temperature of 60°C for one hour using a KOH base catalyst of approximately 1% (w/v) to accelerate the reaction (11). The addition of excess methanol will shift the reaction equilibrium to the right so that the resulting biodiesel product will be greater (32). A mole ratio of oil/methanol of 1:6 is considered more effective in increasing methyl ester yield (33).

The esterification-transesterification reaction of peanut oil with methanol in this study produced a separate glycerol fraction as a secondary product. Although it is a secondary product, the use of glycerol as a value-added product is supported by recent studies on transesterification and biodiesel by-product management (34). The glycerol produced in this study can be seen in Figure 2. Figure 2 shows the presence of two phases of methyl ester and glycerol as is always found in transesterification products, however, the glycerol synthesized from peanut oil has a unique physical appearance, namely clear (not blackish brown) as is commonly found in synthetic glycerol.

Proof that the secondary product obtained is glycerol is done with Dunstan's test, a qualitative test to prove the presence of glycerol. This test is done by adding a solution of borax and phenolphthalein, then utilizing the reversible color change on heating/cooling in the presence of glycerol. A positive result in this test supports the presence of poly-hydroxyl compounds such as glycerol before instrumental analysis (35). The results of Dunstan's test can be seen in Figure 3. Figure 3a shows the color of borax after the addition of phenolphthalein. Figure 3b shows the color fading after heating and returning to a sharp color after cooling as shown in Figure 3c. Dunstan is a rapid and non-specific examination of poly-ol, the results are quite reliable as preliminary evidence. However, to prove the presence of glycerol in this study quantitatively, FTIR and GCMS analyses were performed. The results of the FTIR analysis of glycerol synthesized from peanut oil are shown in Figure 5, which differ from the GCMS results of peanut oil as shown in Figure 4.

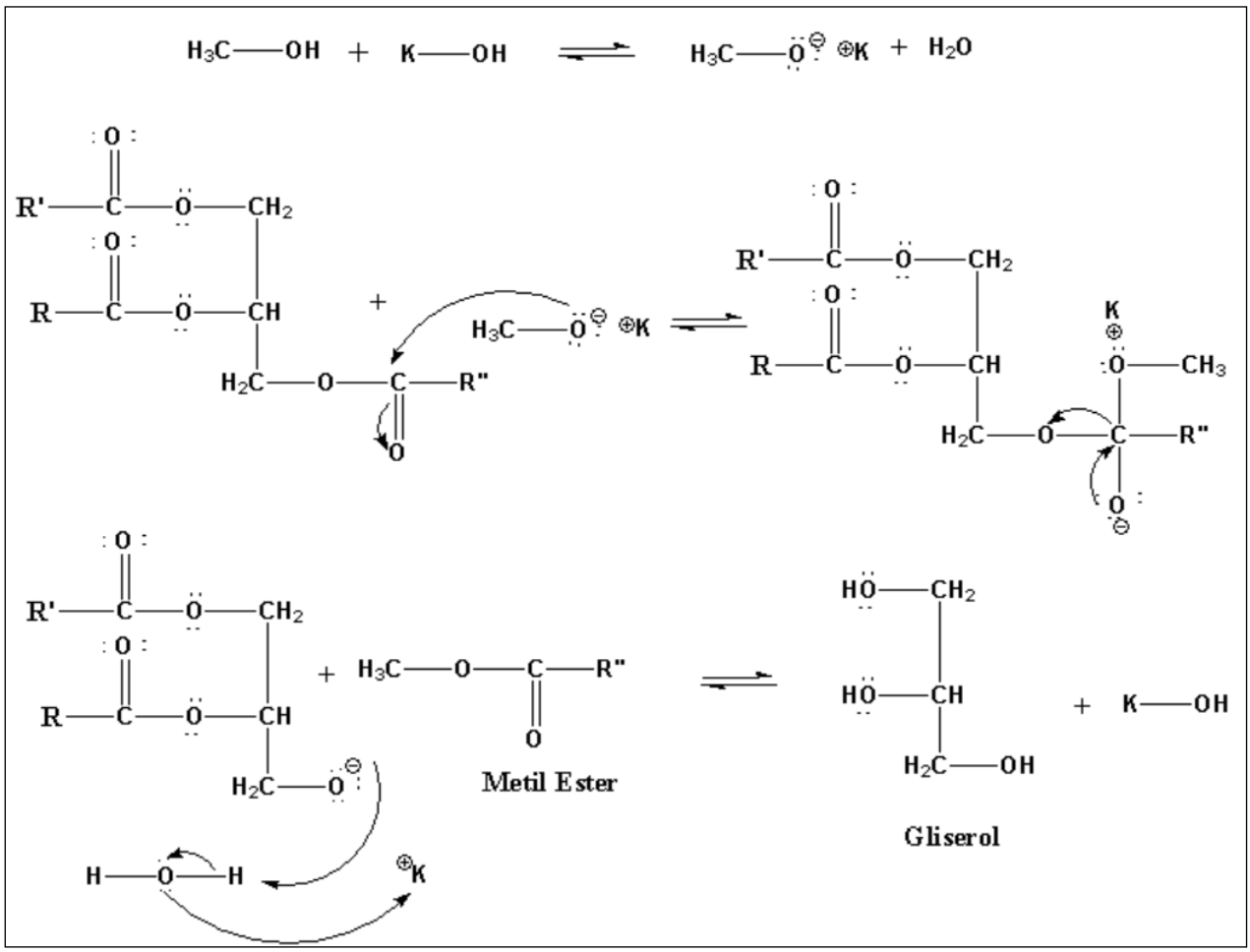

Fig 1 Transesterification Reaction Mechanism.

Analysis using an FTIR spectrophotometer aims to determine the presence of functional groups in various synthetic products. The FTIR spectrum of peanut oil shows that there are absorptions of functional groups, including the absorption of the C-CH group at a wave number of 2924.09 cm-1 and 2854.65 cm-1, absorption of the C=O group at wave number 1751.36 cm-1, absorption of the C=C group at wave number 1651.07 cm-1. C-H group absorption at a wave number of 1458.18 cm-1, at wave numbers 1373.32 cm-1 indicates the presence of O-C-O bond vibrations. The

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interpretation results indicate that there are functional group absorptions that are characteristic of the unsaturated fatty acid compounds that make up mahogany seed oil. The absorption of the O-H group (3471.87 cm-1) and CO (1033.85 cm-1) has a very weak intensity because it comes from free fatty acids contained in peanut oil in very small amounts of around 1% (36).

Fig 2 Results of Trans-esterification of Peanut Oil with Methanol

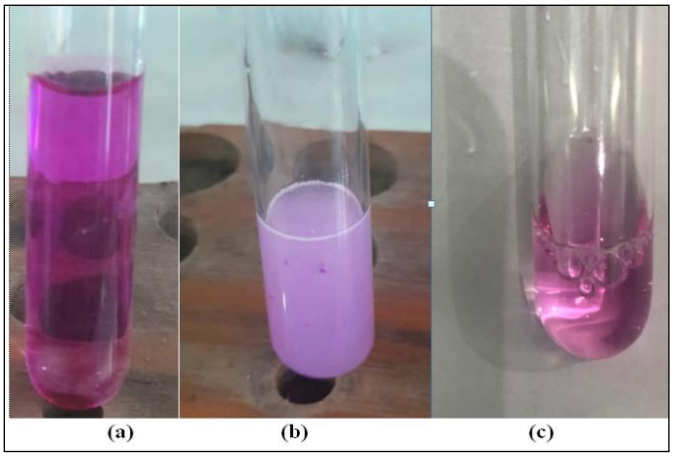

Fig 2 Results of Dunstan's Test Glycerol and Peanut Oil

The FTIR spectrum of glycerol in Figure 5 shows the presence of absorption. functional group absorption includes absorption of the OH group at wave number 3340.71 cm-1. Absorption of C-CH groups at a wave number of 2939.52 cm-1 and 2831.50 cm-1, absorption of CH groups at a wave number of 1450.47 cm-1 and 1404.18 cm-1, and absorption of the C-OH group at wave number 1028.13 cm-1. The absorption of the OH and C-OH groups in this IR spectrum has a very sharp intensity, indicating that glycerol has been formed (31). More details regarding the comparison of the spectra of peanut oil and glycerol can be seen in Table 1.

The results of the GCMS analysis as shown in Figure 7 show that at a retention time of 15.781, glycerol was obtained with a content of 79.71%. The peak obtained at this retention time also indicates that glycerol is a very polar and non-volatile molecule, therefore derivatization is required to produce a separate and strong volatile peak (37), indicated by a peak that is not as sharp as the others. The content of 79.71% also shows that there is still around 20% of the other fractions which are impurities, a common phenomenon indicating that glycerol, the product tested was crude glycerol from the trans-esterification process. This can be seen through a comparison with the GCMS results of peanut oil in Figure 6, where the GC results did not show any glycerol peak at a retention time of 15,781. The six peaks found in the GC results of peanut oil identified by MS resulted in an interpretation in the form of fatty acids that make up peanut oil.

A positive Dunstan test, accompanied by GCMS results indicating the presence of glycerol at a retention time of 15.781, showed the presence of glycerol at a concentration of 79.71%, proving the success of the synthesis through the esterification-transesterification process. The 79.71% concentration obtained also indicates that optimization of the recovery and purification of the synthesized glycerol is still needed. These impurities can be residual methanol, mono- and diglycerides, residual esters, water, or residual catalyst. Purifying crude glycerol as produced in this study is

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challenging and various techniques (precipitation, extraction, vacuum distillation, ion exchange, activated carbon) have been developed to overcome this problem (38).

Table 1 FTIR Spectrum Interpretation Results Data from Peanut Oil and Glycerol

No.	Wave number (cm^{-1})				Vibration Type
	Peanut Oil	Library	Glycerol	Library	
1	-	-	3340,71	3550-3200	O-H
2	2924.04 and 2854.65	3000-2800	2939,52 and 2831,50-	3000-2800	C-CH_3 and C-CH2
3	1751.36	1740-1720	-	-	C=O
4	1651.07	1680-1620			C=C
5	1458.18	1470-1458	1450.47 and 1404.18	1470-1458	CH_2
6	1373.32	1380-1210	-	-	O-CO
7	-	-	1028.13	1300-1000	C-O atau C-OH

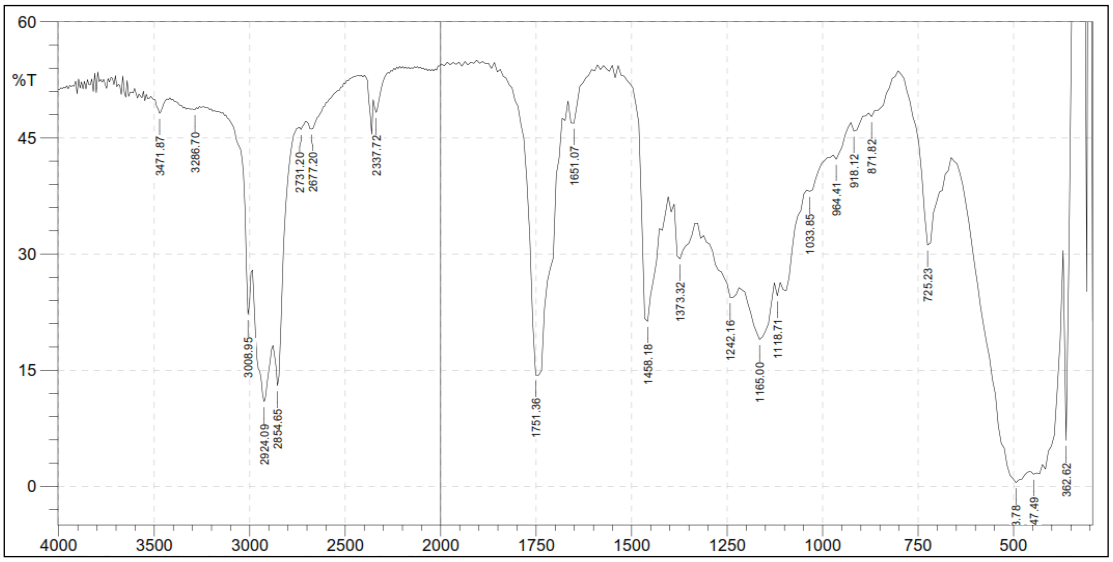

Fig 4. FTIR Spectrum of Peanut Oil.

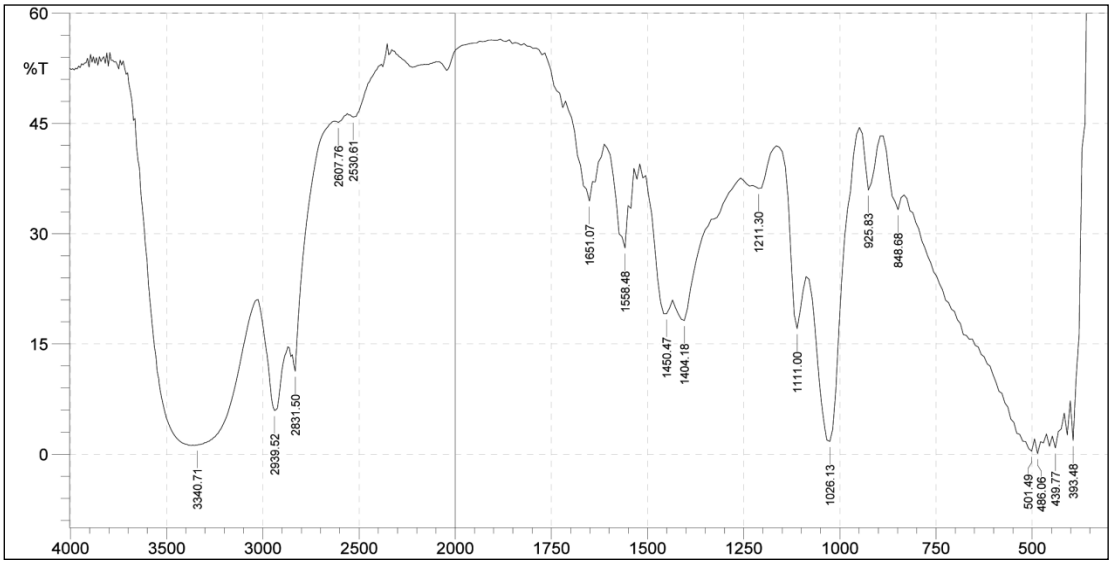

Fig 5. FTIR Spectrum of Glycerol Synthesized from Peanut Oil

Furthermore, several strategies such as (i) optimization of the methanol: oil ratio and catalyst type/quantity to minimize the formation of mono/di-glycerides; (ii) removal of methanol before phase separation (light evaporation) to reduce the solubility of glycerol in the ester phase; (iii) increasing the efficiency of phase separation (repeated centrifugation or decantation) to reduce the glycerol carrier to the ester phase; and (iv) post-isolation purification (vacuum distillation, methanol extraction, acid/alkali precipitation, activated carbon adsorption or ion exchange) to increase the purity of the synthesized glycerol (38).

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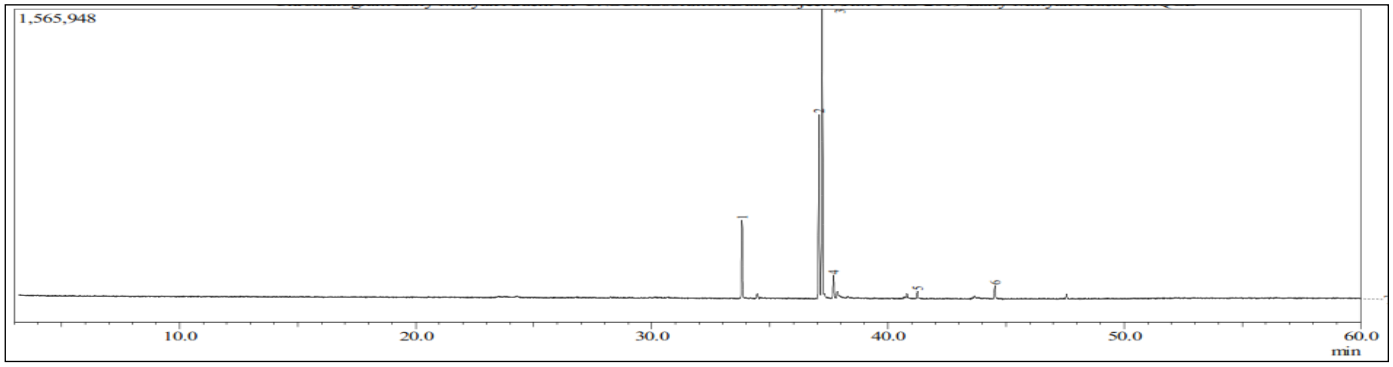

Fig. 6. GCMS Spectrum of Peanut Oil

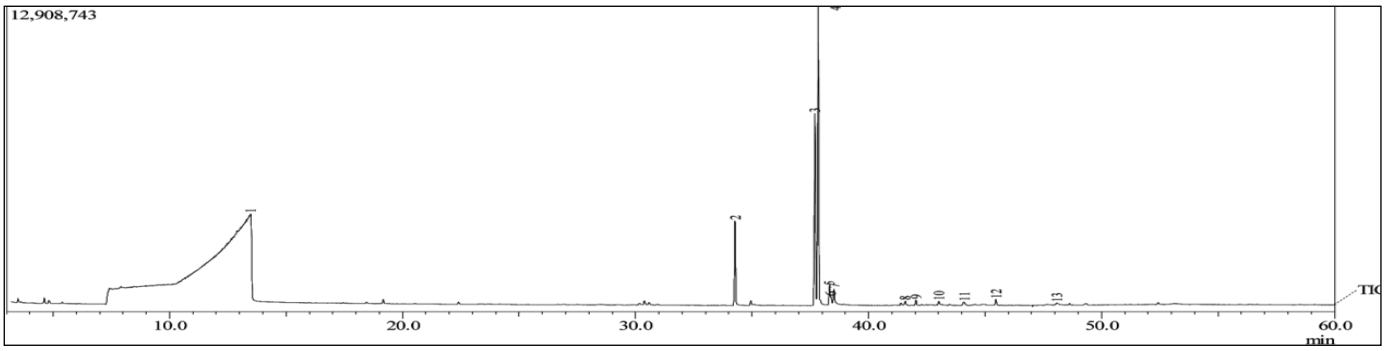

Fig. 7. GCMS Spectrum of Glycerol Synthesized from Peanut Oil

IV. CONCLUSION

The results of the study showed that glycerol as a by-product of esterification- Transesterification can be synthesized from peanut oil and methanol. The resulting glycerol exhibits unique physical characteristics with a clear color (not blackish brown) that responds positively to the Dunstan test for qualitative testing in the form of a faded purple color when heated and a color that returns to light when cooled. The FTIR analysis results show OH absorption at a wavelength of 3340.71 cm-1and the CO group at 1028.13 cm-1, two specific functional groups that did not appear in the FTIR analysis results for peanut oil. The GCMS analysis results confirmed the presence of glycerol as indicated by a peak at a retention time of 15.781 minutes with a content of 79.71%.

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