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ISOLATION AND CHARACTERIZATION OF GLYCEROL BY TRANSESTERIFICATION OF USED COOKING OIL

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ABSTRACT
Used cooking oil is oil that comes from two or more frying food ingredients. This used cooking oil produces methyl esters with glycerol as a by-product through a transesterification reaction. The goal of this study was to convert and utilize used cooking oil into glycerol, as well as to demonstrate the similarity of functional groups and components found in commercial glycerol using IR and GC-MS spectroscopy. The results of the study found that using cooking oil glycerol and commercial glycerol showed that there were similarities in the 1,2,3 propanetriol groups with retention times that were close to 9.6-10.3 minutes and 9.6-10.13 minutes. The IR spectrum showed the presence of OH, CH, C=O, and C-O groups as well as those found in commercial glycerol, and the glycerol content obtained was 90.17%.
Keywords: Used Cooking Oil, Transesterification, Glycerol, FT-IR, GC-MS Spectroscopy.
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INTRODUCTION

Used cooking oil is oil that has been used two or more times to fry food ingredients.[1] Cooking oil can be produced from the coconut plant and can also come from the oil palm plant (Elaeis quineensis), which belongs to the Palmae family. Cooking oil derived from palm oil is typically preferred over cooking oil derived from coconut oil due to a number of advantages, including a lower tendency to smoke and less rusting in the cauldron. Palm oil is made up of glycerol compounds as well as fatty acids in the form of triglycerides.[2] Used cooking oil can be used as a more valuable product, such as glycerol. Glycerol is an alcohol compound that has 3 hydroxyl groups. This compound is a colorless liquid with a boiling point of 290°C.[3] This very strong boiling point is due to the presence of very strong hydrogen bonds between glycerol molecules. Glycerol is an ingredient needed in various industries, such as pharmaceuticals, foodstuffs, cosmetics, toothpaste, and the chemical industry.[4] Some people still consider used cooking oil to be waste. Because of the number of searches for alternative energy, used cooking oil is being sought for its use.[5] The manufacture of biodiesel from used cooking oil produces a by-product in the form of glycerol with an amount of about 10% of the total volume of biodiesel obtained.[6] If biodiesel continues to be produced, more by-products in the form of glycerol will be obtained, resulting in a buildup of crude glycerol. One solution to overcome this buildup of crude glycerol is to convert it into useful and economical products like glycerol. Glycerol is a common by-product of biodiesel production that results from the transesterification reaction.[7] In general, the transesterification reaction uses a base catalyst to react alcohol with triglycerides to produce methyl esters and glycerol. Transesterification can be carried out using methanol and an alkaline catalyst. Base catalysts can be homogeneous, heterogeneous, or enzyme catalysts.[8] Homogeneous catalysts that are often used are KOH and NaOH. Research using a NaOH catalyst produces glycerol with a purity level of 93.34%.[9] This transesterification reaction produces glycerol using a KOH catalyst, which has a similar spectrum to commercial glycerol and glycerol produced from waste cooking oil.[10] In 2020, the Association of Indonesian Biofuel Producers (Aprobi) recorded biodiesel production to reach 9.5 million, and in 2021, biodiesel production is estimated to reach 10 million. Because of the numerous impurities found in crude glycerol, the biodiesel

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industry has not used it thus far.[11] Several studies on the purification of glycerol residue obtained from the transesterification of used cooking oil and soybean oil were developed with the aim of adding economic value. The purity of the glycerol obtained is 97.85%. Because glycerol is insoluble in methyl esters, it can be separated by chemically converting oils and fats into methyl esters.[12] The transesterification process's glycerol is expected to be used as an alternative to meet glycerol requirements. This glycerol purification can provide economic benefits and can reduce the impact on the environment.[13] Pure glycerol plays a major role in the production of various industrial products, for example in the cosmetics, pharmaceuticals, and food industries.[14] The aim of this research is to produce glycerol from used cooking oil so that it can be used in various industries, for example, the cosmetic industry which can use glycerol as a cosurfactant or to moisturize the skin.

EXPERIMENTAL

The material used is cooking oil from the remainder of the brand X frying pan, which is used for 2x frying pans obtained from Medan's Sisingamaraja area. The chemicals used in this study were KOH (Merck), methanol (Merck), phosphoric acid (Merck), activated carbon, commercial glycerol, glycerol (a by-product of used cooking oil), and aqua dest. The instruments used are a magnetic stirrer, desiccator, analytical balance, furnace, FT-IR KBr spectrophotometry (Shimadzu IR Prestige-21), and GC-MS (Shimadzu QP2010).

Making Glycerol from Used Cooking Oil

Glycerol Crude Manufacturing

After filtering the used cooking oil, 500 g is taken and heated to a temperature of 110°C. In 250 mL of methanol, dissolve 5 g of potassium hydroxide. The sample was heated to 60°C, then a mixture of methanol and potassium hydroxide was added and stirred for 1 hour with a magnetic stirrer before being left to separate the biodiesel and crude glycerol for approximately 8 hours.[2]

Glycerol Crude Refining

The crude glycerol obtained was then added with 3 ml of phosphoric acid (H_3PO_4) to the desired pH of 6. A pH meter was used to take the measurement. The glycerol layer was separated from the other layers after three layers were formed, and the glycerol content was determined. 70 ml of distilled water and 4% activated carbon was added to the separated crude glycerol. First, the previously used activated carbon was washed. After stirring for 30 minutes, the mixture was set aside for 24 hours. To remove any remaining methanol and water, it was filtered and evaporated with a rotary evaporator. The sample was placed in a rotary evaporator pre-set to vacuum pressure and 60°C.[9] FT-IR and GC-MS spectrophotometry was used to examine the bottom product of glycerol.

Analysis of Gas Chromatography-Mass Spectrometry (GC-MS)

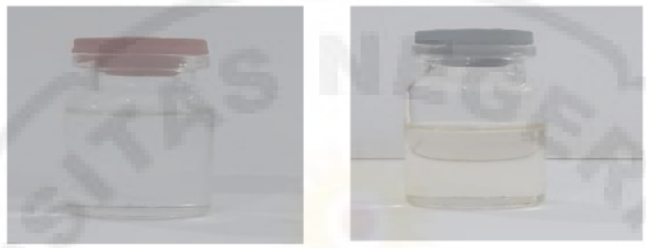
The GC-MS was turned on, and all associated components were fine-tuned until 1 μl sample was ready to be injected and run. The analysis view has been established. Taken sample login while waiting for the GC and MS to appear on the monitor. When the Start button on the monitor is pressed, 1 μl of the sample is injected into the autoinjector, and the automatic injector cleans the syringe according to the settings. If the graph is already looking flat, you can stop the GC analysis by clicking stop on the monitor. Using the similarity function, the graph peaks are identified at each retention time from the initial peak to the final peak and matched with references in the GC-MS program.

RESULTS AND DISCUSSION

This transesterification process produces biodiesel, with the by-product being crude glycerol. The crude glycerol yield is 200.35 g from 500 g of used cooking oil, and the purification yield after being evaporated with a rotary evaporator is 57.27 g. The resulting glycerol can be seen in Fig.-1. This research's glycerol by-product can be proven using FT-IR analysis. Figures-2 and 3 show the results of as a comparison, the FT-IR analysis of commercial glycerol and the glycerol obtained from the transesterification reaction of used cooking oil were performed.

The isolation results using infrared spectrophotometry revealed a change from used cooking oil to glycerol. Glycerol has the molecular formula C_3H_8O and the chemical formula 1,2,3 Propanetriol.[10] The

OH group was revealed by analyzing glycerol samples from used cooking oil,which had a wave number of 3263cm^{-1}. An aliphatic (alkyl) CH group absorbs at wave numbers 2931cm^{-1} and 2877cm^{-1}, a C=O group absorbs at wave number 1653cm^{-1}, and a C-O group absorbs at wave number 1300-1000 cm^{-1}. Based on the FTIR spectrum data, the compounds studied contain hydroxyl OH groups, aliphatic CH, C=O, and C-O esters, indicating the presence of glycerol.



(a) (b)

Fig.-1: (a) Commercial Glycerol and (b) Used Cooking Oil By-Product Glycerol

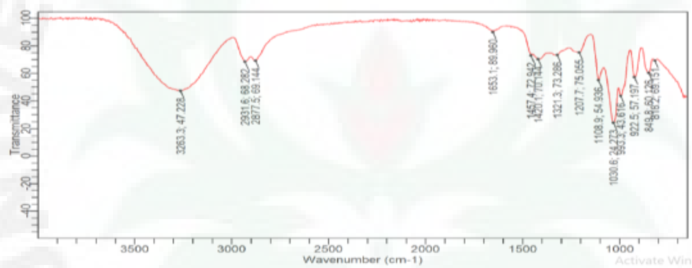


Fig.-2: FTIR Spectrum of Commercial Glycerol

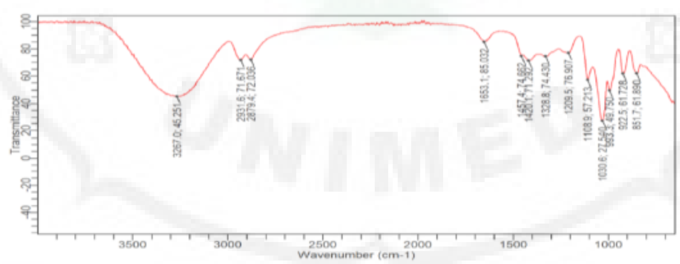


Fig.-3: FTIR Spectrum of Glycerol By-Product from Used Cooking Oil

Commercial glycerol FT-IR analysis revealed a broadband with a wave number of 3267 cm^{-1}, indicating an OH (hydroxyl) group. The aliphatic CH group absorption band appears at wave numbers 2931 cm^{-1} and 2879 cm^{-1}, C=O absorption appears at wave number 1653 cm^{-1}, and C-O functional group absorption appears around 1300-1000 cm^{-1}. The FT-IR spectrum of glycerol from used cooking oil and commercial glycerol by-products revealed that the glycerol compound obtained from used cooking oil contains OH (hydroxyl), aliphatic CH, C=O, and C-O groups, indicating the presence of glycerol. Glycerol produced during the transesterification process is expected to be used as a glycerol substitute. Glycerol derivative products are widely used to manufacture drugs, cosmetics, and cosurfactants.[1]

Analysis of Gas Chromatography-Mass Spectrometry (GC-MS)

Figures-4 and 5 show the results of the GC-MS analysis on glycerol by-products from used cooking oil and commercial glycerol. The chromatograms obtained show the similarity of 1,2,3-Propanetriol between glycerol produced by used cooking oil and commercial glycerol with time. The retention of glycerol as a by-product of used cooking oil is 9.6-10.3 minutes and that of commercial glycerol is 9.6-10.16 minutes. The data from this study shows the similarity between the by-product samples of used cooking oil and commercial glycerol with a retention time that is close to that of commercial glycerol. A GC-MS analysis was carried out to re-confirm that the purified compound was a glycerol compound. The GC-MS analysis found the following compounds in glycerol as a by-product of used cooking oil: 2,4 pentanediol, 2-methyl-, butanoic acid, 4-(2,4,5-trichloro phenoxy) methyl-, and 1,2,3-Propanetriol (CAS). The results of the chromatogram analysis using mass spectroscopy resulted in a fragmentation pattern identical to the mass spectra of commercial glycerol, which showed the base peak was at fragment mass 59. A quantitative analysis of glycerol after purification was carried out using the determination of glycerol content according to SNI 1995. This glycerol content needs to be determined to see the level of glycerol purity. The results of the study obtained a glycerol content of 90.67%. The requirement for glycerol content that is allowed to be commercialized according to SNI is a minimum of 80%.

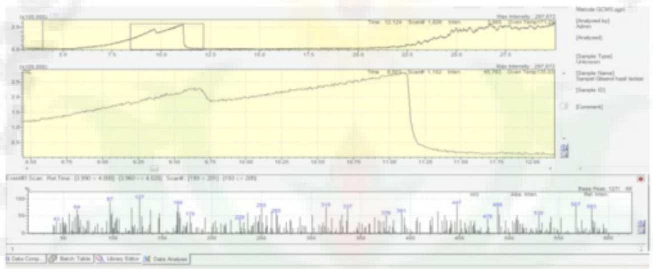


Fig.-4: Glycerol By-Product from Used Cooking Oil GC-MS Chromatogram

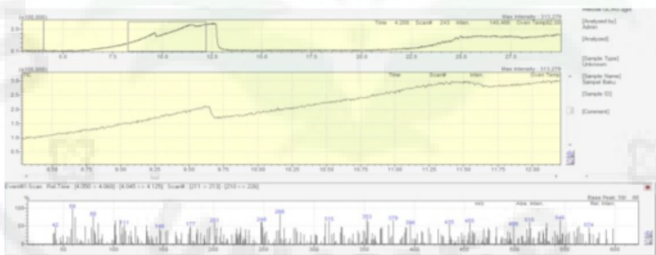


Fig.-5: Commercial Glycerol GC-MS Chromatogram

CONCLUSION

The functional groups of glycerol derived from used cooking oil are similar to those of commercial glycerol. The IR spectrum of glycerol compound, a by-product of used cooking oil, shows the presence of hydroxyl OH groups, aliphatic CH, C=O, and C-O esters. The result of this research is that the glycerol content is 90.67%, and it has met the requirements of SNI.

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AUTHOR CONTRIBUTIONS

All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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