

## Determination of Pork Fatty Acid in Bulk Cooking Oil

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### Abstract

The increase in cooking oil prices in early 2022 will force people to switch from packaged cooking oil to bulk cooking oil, which does not have a brand or label. People cannot differentiate between bulk cooking oil produced by factories and recycled used cooking oil. Currently, people not only use vegetable cooking oil in their daily lives, but many people mix it with animal cooking oil to produce a more savoury taste in their dishes. This research aims to determine the fat content of pork based on the fatty acid composition and fatty acid levels in bulk cooking oil circulating in the community. The method used to determine the fat content of pork in bulk cooking oil samples is using GC-FID. The level of the fatty acid 11,14-Eicosadienoic Acid (C20:2) in pork fat, which is a marker for the presence of pork fat in the sample, is 0.57%, whereas it was not detected in the bulk oil sample.

**Keywords** *Bulk Cooking Oil, GC-FID Instrument, Pork Fatty Acid, Lard*

### INTRODUCTION

Cooking oil is one of the basic needs of the Indonesian people. Cooking oil consumption in Indonesia is increasing every year because almost all dishes use cooking oil. Indonesia is the largest cooking oil-producing country in the world after Malaysia. Cooking oil production in Indonesia continues to increase every year. Cooking oil is usually cooked with food or used as a medium of heat conducting in cooking food ingredients (Ketaren, 2012). Raw materials used to make cooking oil are palm oil, soybeans, canola, olives, pork fats and others.

The Ministry of Energy and Mineral Resources (ESDM) set the Indonesian Crude Price (ICP) price for December 2020 at USD 47.78 per barrel. An increase of USD 7.11 per barrel from USD 40.67 per barrel in November 2020 (Merdeka.com, 2021). Based on World Bank data (2022), the price of cooking oil increased from USD 748 in January 2019 to USD 1,957 per ton in March 2022 (Liputan6.com, 2023). The increase in cooking oil prices made people switch from packaged cooking oil to bulk cooking oil that did not have brands and labels. Many naughty parties sell used oils collected from housing, hotels, and fast food to restaurants, which is not clear where they come from and are questioned halal, then the oil is cleaned and resold to retail traders as bulk oil (Detikfinance, 2019). The community cannot distinguish bulk oil from the factory, with used cooking oils again (BBC News Indonesia, 2022). At present, bulk cooking oil is still in demand by the public, where bulk cooking oil is usually sold at a cheaper price of Rp 13.000 per litre than cooking oil packaged which is sold at Rp. 19.000 per 1 litre. This is one of the reasons for some people to prefer bulk oil to fry (Dewi, 2022; CNN, 2019). The use of bulk oil is increasing in the community, this is due to the increase in prices so that people switch from packaged cooking oil to bulk cooking oil that does not have a brand and label on the packaging. Research on bulk oil fatty

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acids must be done because it allows the contamination of pork fat or its derivatives.

One of the concepts of halal in Islam, namely consuming food that is halal, pure, and does not contain the slightest pork content, is a religious command and is obligatory by law. Halal is everything free from haram elements that cannot be consumed by Muslims (Rahmadani, 2015). Food and ingredients that are considered haram for consumption by Muslims can be classified into four types, namely carrion, pork and its derivatives, alcohol and its derivatives and blood and its derivatives. Apart from that, food contaminated with these products is also haram for consumption. Based on this background, the researchers wanted to know how the equation's results for the fatty acid composition of bulk oil and pork fat acids compare.

## LITERATURE REVIEW

The existence of food forgery, especially in oil, fat and alcohol, has become a problem for Muslim consumers (Andriyani et al., 2019). To detect pork fats in bulk oil sold by traders requires a method of pig fat analysis that can provide accurate and fast analysis results. Several chemical analysis methods have been available to detect animal fat content in foods with different levels of accuracy and sensitivity (Rohman, 2020). Chromatography (GC) gas is one of the chromatography that can be used to separate and analyze a compound. GC can be used to test certain materials' purity or separate various components from the mixture (Hilda, 2014). Based on the research of Prabawati and Fajriati (2019) shows, the results that with GC obtained acid content of saturated fat (palmitic acid and oocate-denganoic acid) in cow fats more than saturated fatty acids in pork fats. The content of unsaturated fatty acids (oleic, linoleic, and linolenic acid) in pork fats is higher than in cow fatty acids.

### Halal food

Food is a primary need that must be met as a supporter of human survival. Besides as a necessity for life, food or food will affect health, the good and bad growth and behavior of humans. Consuming halal and good food and beverages is an obligation and an order in Islamic teachings. As the word of Allah SWT in the Qur'an Surat al-Baqarah verse 173:

إِنَّمَا حَرَّمَ عَلَيْكُمُ الْمَيْتَةَ وَالدَّمَ وَلَحْمَ الْخَنزِيرِ وَمَا أُهِلَّ بِهِ لِغَيْرِ اللَّهِ فَمَنْ اضْطُرَّ غَيْرَ بَاغٍ وَلَا  
عَادٍ فَلَا إِثْمَ عَلَيْهِ إِنَّ اللَّهَ غَفُورٌ رَحِيمٌ

Meaning: "Indeed, he only forbids you carcasses, blood, pork, and (meat) animals that are slaughtered by (mentioning names) besides Allah. But whoever is forced (to eat it), not because he wants it and not (also) beyond the limits, then there is no sin for him. Really, Allah is forgiven, Most Mirror" (Al-Baqarah: 173).

Allah SWT has ordered Muslims to eat something good from the sustenance passed down to them so that they carry out obligations for the blessings by being grateful to Him as the giver of blessings. The Ministry of Religion of the Republic of Indonesia (Indonesia. Ministry of Religion, 2021) provides instructions and conditions regarding halal guarantees, including:

1. It does not contain parts or objects of animals that are forbidden for consumption by Muslims.
2. Does not contain something unclean.
3. Not processed using tools that are not free of unclean.
4. The storage process is not in contact and not close to the object that unclean punishes.

The halal requirements for a product consumed include Halal Essence, Halal how to obtain it, Halal in processing it, Halal in storage, Halal in transportation, and Halal in its presentation.

Guarantee that a food product is halal with a halal certificate listed on the packaging. A halal certificate is a written fatwa from the MUI that states the halal of a product in accordance with Islamic sharia. Foods that are not halal are grouped into nine categories, namely (1) carcasses, (2) flowing blood or which have frozen, (3) pork derivatives such as pork, pork fats, and products derived from pork such as gelatin pork and So, (4) Animals that are slaughtered do not mention the name of God, animals that are slaughtered by mentioning names other than God are not allowed to be consumed, (5) animals that are slaughtered in such a way as to prevent their blood from flowing out perfectly from their bodies, (6) all types Such as alcohol and narcotics, (7) carnivorous animals such as lions and tigers, (8) birds with fanfare, (9) and ground animals such as frogs and snakes (Rohman, 2020).

### **Pork Fatty Acid and Lard**

Pork fat is one of the animal fats usually used together with fats from plants such as olive and palm oil for margarine production or oil in other foods (Rohman, 2020). The main composition of fatty acids in pigs is oleic acid (40%), palmitic acid (25%), stearate acid (13%), and linoleic acid (13%). Pork fat contains triacylglycerol (TGA) and fatty acids, with a small amount containing tocopherol, carotene, sterols, and fat-free acids. TGA is the main fat component correlated with the cooling and melting process. Pork fat has a lower triglycerol content than triglycerol in beef fat. Therefore, pork fat melts at lower temperatures (Aminullah, 2018).

Three main types of pork fats are used for different purposes, namely back fat or fatback) derived from the back of the shoulder and buttocks and is located right under the pig skin, used to saute and fry. Pork belly fat is used to fry, and deep fat (leaf lard) is fat located around the pig kidney. Inner fat is the cleanest pork fat, and the oil produced is suitable for making cakes and roasted foods. Pork fats are obtained from any part of the pig body as long as there is fat tissue with high concentrations. The best quality pork fat is obtained from the parts around the kidneys and the pig's stomach. The best quality fats are also obtained from the back, in the part between pork's muscles and hard fat. The lowest quality pork fat is obtained from fats around the digestive organs (Lidansyah, 2015).

### **Bulk Cooking Oil**

Bulk cooking oil is palm cooking oil that is sold to consumers in a condition that is not packaged and does not have a label or brand (Indonesia. Ministry of Trade, 2022). Low-quality cooking oil (bulk) usually comes from low-quality raw materials (CPO), to be produced into high-quality cooking oil will require expensive production costs, so this oil is produced into bulk cooking oil. Bulk cooking oil contains more saturated fat, so it is not healthy. In addition, the distribution of bulk cooking oil from the factory to retail through a long distribution chain is feared that the aspects of bulk oil hygiene are less feasible for consumers. Bulk cooking oil is sold to the market without using brands and product labels that are usually placed in large jerry cans or drums and then sold to consumers in retail (Fitriana, 2015).

Bulk cooking oil is a one-stage fractionation product of palm oil, generally sold in traditional markets whose handling is relatively less hygienic than packed cooking oil. The distribution of cooking oil to consumers is generally distinguished based on the packaging of cooking oil, which is packaged and not packaged. Generally, the cooking oil that is packaged is Branded cooking oil, while bulk cooking oil is sold retail by traders and then packaged according to the wishes of the buyer. The distribution of bulk cooking oil is two ways: first, the factory to the distributor, distributor to sub-distributors, sub-distributors to retailers or consumers and second, factories to distributors and distributors directly to sub-distributors, retailers and consumers. Meanwhile, in the distribution of Branded cooking oil, producers pack it and then sell it to distributors to be

forwarded to consumers. The distribution process, ranging from the factory to consumers, shows that the packaged cooking oil is more hygienic than the bulk cooking oil. The potential contamination of bulk cooking oil is greater than that of Branded cooking oil (Bukhori & Tutik, 2017).

### **Gas chromatography**

Gas chromatography is an analytical technique used in fields such as industry, environment, pharmacy, oil, chemistry, clinics, forensics, food, and so on (Gandjar & Rohman, 2007). The general use of gas chromatography is to do dynamic separation, identify all types of volatile organic compounds, and conduct qualitative and quantitative analysis of compounds in the mixture. Gas chromatography can be destructive and non-destructive depending on the detector used. Gas chromatography can be automated to analyze solid, liquid and gas samples. Solid samples can be extracted or dissolved in a solvent so that they can be injected into a gas chromatography system (Gandjar & Rohman, 2007).

In gas chromatography, the movement phase is gas, and the solute is separated as steam. Separation is reached with a sample partition between the moving and stationary phases in the form of a liquid with a high boiling point (not volatile) bound to the substance, determining the absorbent solid substance. The use of liquid as a stationary phase is more widespread than a solid, so this technique is sometimes known as gas-liquid chromatography (Gandjar & Rohman, 2007). The principle of gas chromatography, the separation of gas chromatography, is based on the boiling point of a compound reduced by all the interactions that may occur between the solute and the stationary phase. The gas phase will escape the solute from the tip of the column and then deliver it to the detector. Temperature increases (usually in the range of 50-350 ° C) aim to ensure that the solute will evaporate and, therefore, be quickly eliminated.

## **RESEARCH METHOD**

### **Research design**

The sample collection technique was carried out by the purposive sampling method. The selection of samples was based on the specified inclusion criteria that had been determined were bulk oil sold in the market, with a price interval of fourteen thousand rupiahs up to fifteen thousand five hundred rupiah (Rp. 14,000 - Rp. 15,500) Per litre, does not have a brand, and does not have a label. Samples are three samples (MC1-MC3).

### **Tools and materials**

This study uses a closed weighing bottle (pyrex), desiccator (duran), filter paper (whattman), 100 ml fat flask (pyrex), soxhlet (duran), water bath (corning), oven (memmert), a set of gas chromatography tools (GC 2010 Plus), Syringe 10 µl, Water Bangs (Grant), Teflon (Pyrex) Closed Tubes, Analytical Balance (Pioneer), Micro Pipette (DLAB).

This study uses bulk cooking oil, three bulk oil samples, packaged cooking oil, pork fat, petroleum ether (smart-lab), NaOH 0.5 N (Merck), BF<sub>3</sub> 20% (Merck), NaCl (Merck), Hexan (Merck), Na<sub>2</sub>SO<sub>4</sub> anhydrous (Merck).

### **Determination of fat levels of pork fat samples**

#### *Extraction of pork fat using the Soxhlet method*

Weigh 7-10 grams of dried pork fat-free of water and put it in a paper shell coated with cotton. The paper plug contains pork fat with cotton, then put in a fat flask with boiling stone that has been dried and weighed. For about six hours, extracts with 100 millilitres of Petroleum Benzene solvents. Soxhletation results are then distilled to distinguish between fat and petroleum ether

solvents. For thirty minutes, dry the fat extract in the oven at 105 ° C. Then cool and weigh the sample to find the yield.

### **Analysis of fatty acids with gc-fids in pork fat samples and cooking oil**

#### *Making 0.5 N NaOH reagents in methanol*

Weigh 2 grams of NaOH into a watch glass. Move the NaOH that has been weighed into a 100 ml measuring flask. Rinse the watch glass with methanol, and add the rinse. After dissolving, impact with methanol until the limit is homogeneous.

#### *BF3 reagent making 20% in methanol*

20 ml BF3 stock solution with a pipette to a 100 ml measuring flask. Add methanol to the 100 ml Takar Pumpkin with a 20 ml BF3 until the pumpkin appears. After shaking a 20% BF3 solution, keep the solution ready to use in a dark glass bottle.

#### *Sample preparation (hydrolysis and esterification)*

Weigh twenty to thirty milligrams of sample fat or oil obtained previously. After adding 1 ml of 0.5 N NaOH to the methanol and pushing it with nitrogen, heat it in a water bath for 20 minutes. After cold, add 1 ml of isooctane or hexane and shake well. Move the hexane layer into a tube containing about 0.1 g Na<sub>2</sub>SO<sub>4</sub> anhydrous with a dropper. Leave for 15 minutes. The liquid phase is separated, and the organic phase is inserted into the gas chromatography.

### **Analysis of fatty acid components, as a fame**

To analyze hydrolyzed fat samples and esterification, analyzed using GC 2010 plus set first. The column consists of methyl cyanopropyl (capillary column) with dimensions P = 60 m, inner diameter = 0.25 mm, and film thickness 0.25 mm. Flow rate N<sub>2</sub> = 30 ml/minute, flow rate he = 30 ml/minute, flow rate H<sub>2</sub> = 40 ml/minute, and airflow rate = 400 ml/minute. Injector temperature = 220 ° C, detector temperature = 240 ° C, and column temperature = temperature program. Temperature column, average speed (° C/minute) = -3, 5, 10, 5, 3 Temp (° C) = 125, 185, 205, 225 (Time (° C/Min) = 5, 5, 20, 7 split ratio = 1: 80, inject volume = 1 µl, linear velocity = 23.6 cm/sec. After all the peaks come out, injection 1 µl of ordinary fame mixture. Measure the peak time and retention for each component. Retention time is compared to the standard to determine the type of fatty acid component in the sample.

### **Data analysis**

In identifying this fatty acid, using the instrumental method with gas chromatography used. The sample will be identified in advance with gas chromatography. The results obtained on gas chromatography are chromatograms. The results obtained were then identified the profile of fatty acids and a ratio of levels between bulk oil fatty acids and pork fatty acids.

### **FINDINGS AND DISCUSSION**

Samples of pork fats must be extracted using the soxhlet method to separate fat in the sample; this method can produce fat extracts that are high enough with less solvents and shorter extraction time. Before extracting, pork fat samples are done first to reduce the water content during extraction. The solvent used in the extraction process is petroleum ether, an organic solvent that can dissolve non-polar compounds such as fat. The result of fat extraction is shown in Table 1.

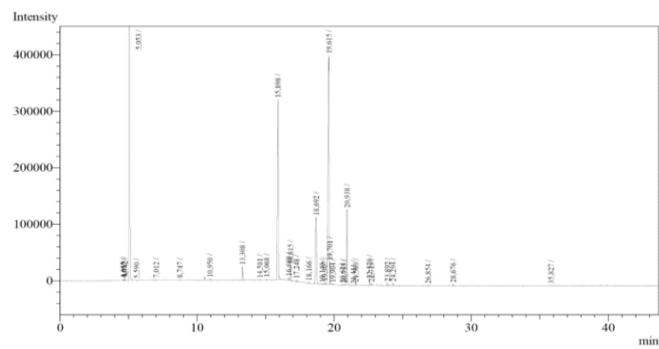
The yield of fat levels obtained in pure pork fat is 42.3912%; the size or small yield produced depends on the fat in the sample extracted using the soxhlet method. After extracting fat samples obtained from pork fat are then tested using GC-Fid. The method known as gas

chromatography is used to separate parts of the chemical mixture contained in a material. A gas referred to as carrier gas carries components to be separated through columns, and the sample mixture is divided between the carrier gas and the stationary phase. The carrier gas holds components based on the distribution coefficient so that a number of different bands are formed in the carrier gas. These ribbons leave the column with the carrier gas flow and are recorded by the detector as a time function. In the sample injection, the sample is put into the injector room and then carried by the gas into the column, which produces a chromatogram.

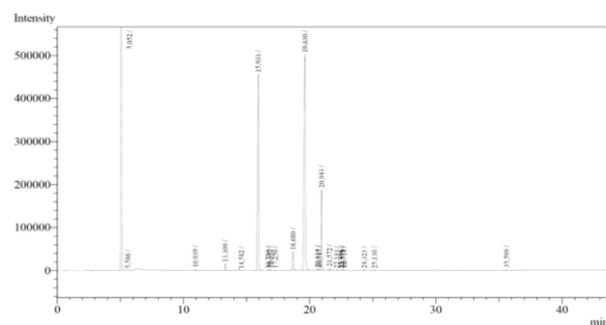
**Table 1.** The Results of The Yield of Pork Fat

Sample Code	Weight Sample (g)	Empty fat volumetric weight (g)	volumetric + fat (g)	fat weight (g)	Fat Yield (%)
FA	8.2826	75.2359	78.748	3.5111	42.39%

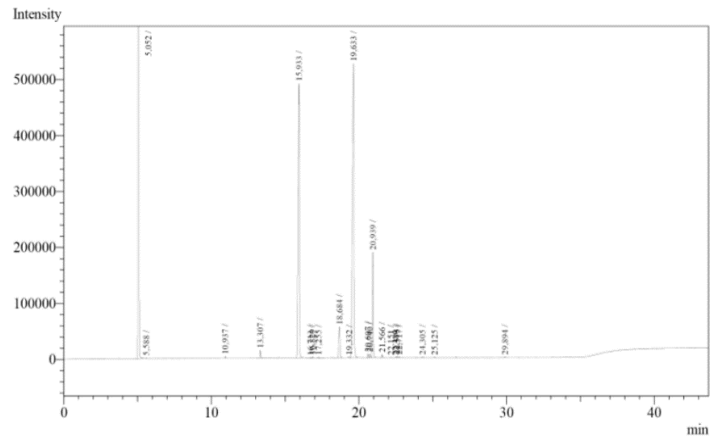
Methyl ester fatty acids are a form of fatty acids found, and their separation is done with gas chromatography. Qualitative analysis of this GC-FID aims to identify a component of each sample using a retention time parameter, using the same analysis condition to compare the retention time of unknown components with standard compounds, retention time, which is the time required by acids fat to appear in the detector, shows that the two compounds are the same calculated from the time of the sample injected from the injection point to the peak of the fatty acid (Khopkar, 2007). The results of qualitative analysis in the form of GC chromatograms show the peak of the separation results and the area used for quantitative analysis data. The results obtained in bulk oil samples and pork fat are served in Figures A, B, C, and D.



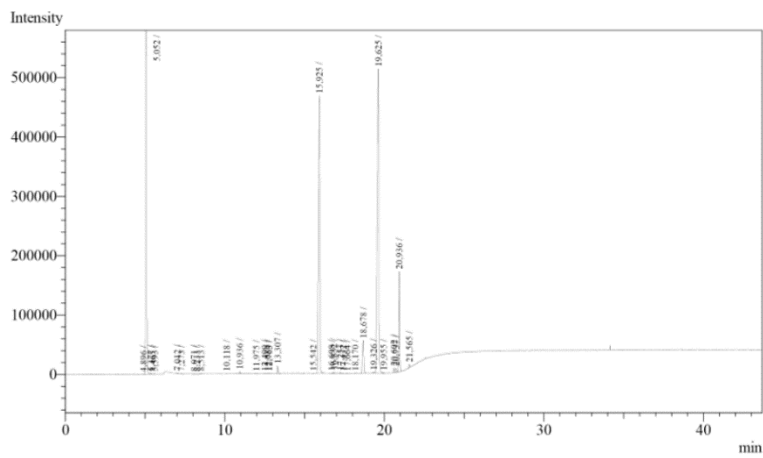
(A)



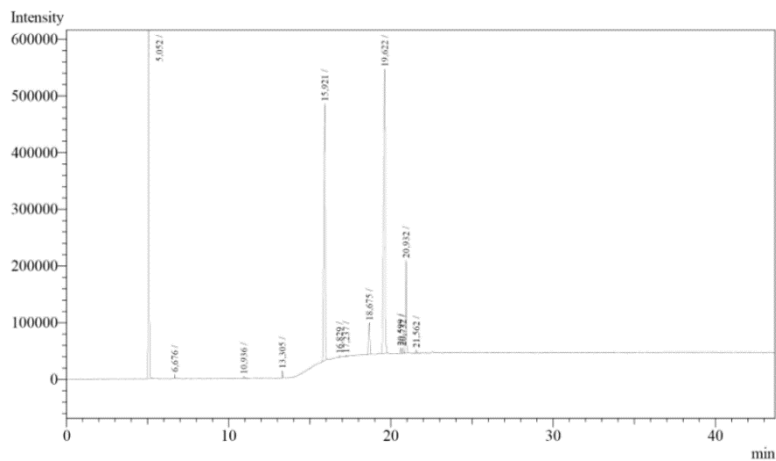
(B)



(C)



(D)



(E)

**Figure 1.** Chromatogram results (A) pork fat chromatogram (B) packaged cooking oil chromatogram (C) bulk oil chromatogram 1 (D) bulk oil chromatogram 2 (E) bulk oil chromatogram 3

Based on the chromatograms of the five samples (images A, B, C, D, and E), the analysis results were obtained, which showed the peak separation results along with the % area used for quantitative data to determine the fatty acid composition and the calculated levels of each fatty acid. The peak of the fatty acid chromatogram shows different fatty acid profiles in lard, packaged cooking oil, bulk oil 1, bulk oil 2, and bulk oil 3. The results of the fatty acid chromatogram are then interpreted to determine the fatty acid composition and levels of fatty acids present in the sample.

One way to carry out fatty acid analysis with GC is through esterification. This is achieved by adding methanol to the BF<sub>3</sub> base catalyst, which changes the structure of the fat that will be used as a substrate for the esterification reaction into an ester-formed fatty acid known as fatty acid methyl ester (FAME). FAME is usually used as a raw material or standard for qualitative and quantitative analysis in sample analysis because it is an acid derivative of fatty acids with an ester functional group (Zaid et al., 2016). Fatty acids are organic acids with a straight hydrocarbon chain with a hydroxyl group (COOH) at one end and a methyl group (CH<sub>3</sub>) at the other end (Almatsier, 2006). Fatty acid analysis results samples of lard, packaged cooking oil and bulk cooking oil are presented in Table 2.

**Table 2.** Fatty Acid Composition in Each Sample

Component Type	Fatty Acid Levels in Samples				
	Lard	Packaged Cooking Oil	Sample 1	Sample 2	Sample 3
Caprylic acid, C8:0	0.14	Ttd	Ttd	Ttd	Ttd
Undecanoic acid, C11:0	0.09	0.04	0.035	0.035	0.035
Myristic acid, C14:0	1.86	0.735	0.60	0.62	0.535
Pentadecanoic acid, C15:0	0.06	0.01	Ttd	Ttd	Ttd
Palmitic Acid, C16:0	34.25	48.96	49.835	48.475	47.57
Heptadecanoic acid, C17:0	0.24	0.03	0.035	0.035	Ttd
Cis-10-Heptadecanoic acid, C17:1	0.12	Ttd	Ttd	Ttd	Ttd
Stearic acid, C18:0	15.92	3.01	3.71	4.33	3.935
Palmitoleic acid, C16:1	2.43	0.01	0.01	0.01	0.05
Elaidic acid, C18:1n9t1	0.09	Ttd	Ttd	Ttd	Ttd
Oleic acid, C18:1n9c	49.88	39.36	38.595	39.40	39.08
Linolelaidic acid, C18:2n9t	0.07	Ttd	Ttd	0.025	Ttd
Linoleic acid, C18:2n6c	0.06	0.03	0.06	1.89	0.10
Arachidic acid, C20:0	0.04	0.25	0.27	0.28	0.31
Cis-11-Eircosenoic acid, C20:2	0.95	Ttd	Ttd	Ttd	Ttd
Linolenic acid, C18:3n3	0.32	0.05	0.05	Ttd	Ttd
Cis-11,14-Eicosadienoic acid, C20:2	0.57	Ttd	Ttd	Ttd	Ttd
Behenic acid C22:0	Ttd	0.04	Ttd	Ttd	Ttd
Arachidonic acid C20:4n6	0.14	Ttd	Ttd	Ttd	Ttd
Docosadienoic acid, C22:2	0.08	Ttd	Ttd	Ttd	Ttd
Total Fatty Acid	107.3 1	92.525	93.20	95.10	91.615



Based on the data in Table 2. It can be seen that the fatty acid content found in lard, packaged cooking oil, and bulk cooking oil samples had almost the same fatty acid composition detected. Such as several fatty acids, namely palmitic, stearic, oleic and linoleic acids, which are the main fatty acids that makeup pork fat (Codex Alimentarius, 1999). Firmansyah (2015) stated that several researchers, namely Mosley et al. (2007), Montoya et al. (2013) and Chen et al. (2014), stated that the fatty acids that makeup palm oil, according to research that has been conducted are palmitic, myristic, oleic, linoleic and arachidic acids.

The main fatty acid compositions detected in pork fat were linoleic acid (0.06%), oleic acid (49.88%), stearic acid (15.92%), and palmitic acid (34.25%) as well as the marker compound fatty acid, which is a marker for the presence of pork fat in a sample, namely 11,14-eicosadienoic acid (0.57%). According to Indrasti et al. (2010), regarding the fatty acid profile of pork, the differences between chicken, beef and goat meat are 11,14-eicosadienoic acid, 11,14,17-eicosadienoic acid, and trans-9,12 acid. ,15-Octadecanoate. These three fatty acids are marker compounds for pork fatty acids that have been successfully detected, so these fatty acids can be used to distinguish pork fat from other animal fats in the halal authentication process for food. The results of the analysis of the fatty acid composition in packaged cooking oil consisted of linoleic acid (0.03%), oleic acid (38.48%), stearic acid (2.93%), and palmitic acid (47.43%). For bulk oil samples, the main fatty acid components consist of linoleic acid (0.06%), oleic acid (38.41%), stearic acid (3.68%) and palmitic acid (49.55%). Bulk oil 2 consists of linoleic acid (1.87%), oleic acid (38.93%), stearic acid (3.84%), and palmitic acid (48.40%). For bulk oil sample 3, it consists of linoleic acid (0.10%), oleic acid (39.68%), stearic acid (3.96%), and palmitic acid (48.03%).

From the results obtained, it can be said that the bulk oil samples were negative for containing lard, as indicated by the undetectability of 11,14-eicosadienoic acid in the bulk oil samples. This is in accordance with research conducted by Indrasti et al. (2010) regarding the fatty acid profile of pork, which differentiates it from fatty acids from chicken, beef and goat, namely trans-9,12,15-octadecanoic acid; 11,14,17-eicosatrienoate; and 11,14-eicosadienoate. This fatty acid is the third profile marker for pork fatty acids that were successfully detected using the GC-Time of Flight-MS instrument so that these fatty acids can be used as a basis for distinguishing pork fat from other animal fats in the process of halal authentication of food.

## CONCLUSIONS

The structure of the fatty acids in pork fat and the fatty acids in bulk oil are similar in several fatty acid components such as linoleic acid, oleic acid, stearic acid and palmitic acid. However, in bulk oil, there is no 11,14-Eicosadienoic acid (C20:2), which is a marker for the presence of lard in the sample, so it can be said that the sample is negative for containing lard. The fatty acid content of 11,14-Eicosadienoic Acid (C20:2) in pork fat was 0.57%, which was not detected in bulk oil samples.

## LIMITATION & FURTHER RESEARCH

The limitations of this research are that the bulk oil sample does not have a brand, does not have a label, and the price is below Rp. 15.500/litre. The sample analysis method uses gas chromatography instrumentation with Cyanopropyl methyl Column conditions (capillary column), Column dimensions (p) = 60 m, Ø inside = 0.25 mm, 025 µm Film Thickness, N2 flow rate = 30 mL/minute, Flow rate He = 30 mL/minute, H2 flow rate = 40 mL/minute, Air flow rate = 400 mL/minute, Injector temperature = 220°C, Detector temperature = 240°C, column temperature = Program temperature, Split Ratio = 1: 80, Inject Volume = 1 µL, Linear Velocity = 23.6 cm/sec. Future research is expected to carry out analysis using different methods and instruments, such as FTIR combined with chemometrics, to obtain good results.

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