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Separation and Determination of Free Fatty Acids in Corn Oil and Palm Oil by Liquid-Liquid Extraction and Acidi-Alkalimetric Titration

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Abstract

Free Fatty Acids (FFA), which include unsaturated fatty acids like oleic and linoleic acid and saturated fatty acids like palmitic and stearic, are present in most oil compositions. The various negative impacts of FFA require these compounds to be eliminated. This research aims to separate FFA using Liquid-Liquid Extraction (LLE) and determine the levels of FFA using acid-alkalimetric titration in corn oil and palm oil. FFA extraction was carried out for 4 minutes with a solvent ratio of 9,5 ml diethyl ether + 40 ml ethanol + 0,5 ml water. By adding 1M Na_2SO_4, re-extraction was carried out for 3 minutes. The results obtained were 0.95% FFA levels in corn oil and 1.26% FFA levels in palm oil. FFA levels of both oils are still below the SNI FFA percentage of 3%; besides that, it is found that the FFA levels in palm oil are higher than in corn oil.

Keywords: *Free fatty acids; liquid-liquid extraction; acidi-alkalimetric; corn oil; palm oil*

Introduction

Oil is one of the nine human staples used for food processing. Oil is sourced from vegetable and animal sources and can usually be classified into (i) edible oil (such as oil sourced from corn, palm, coconut, sunflower, olive, etc.), (ii) non-edible oil (such as oil sourced from castor, malapari, nyamplung seeds, etc.), (iii) used cooking oil *(Aitlaalim et al.,* 2020), (iv) animal fat, or (v) oil formed by microalgae, such as algae and cyanobacteria (Abomohra *et al.,* 2020). Oils/fats typically contain about 98% triglycerides (which are chemically esters of glycerol with three fatty acids) (Fasciotti *et al.,* 2020). Other chemical compounds in the oil are mono and diglyceride (DAG), Free Fatty Acids (FFA), water, and colourants. The higher fatty acid levels bound in triglycerides will determine the physicochemical properties, such as iodine number, viscosity, and oxidative stability (Hájek *et al.,* 2021)Fatty acids can be divided into (i) saturated, which do not have double bonds on carbon atoms (such as palmitic and stearic acids), and (ii) unsaturated, which have double bonds on carbon atoms (such as oleic, linoleic, and linolenic acids).

Saturated and unsaturated fatty acids can be a source of FFA. FFA in oil is long-chain fatty acids that are not esterified, which means they are not bound to glycerol to form triglyceride molecules. FFA cause unpleasant flavours such as sourness or bitterness and reduces the smoke point, which makes the oil more susceptible to oxidative rancidity (Ferreira *et al.,* 2021). Moreover, during

heating or frying, oil is damaged or deteriorated, characterised by the formation of FFA and peroxide compounds as a result of oxidation reactions (Budiyanto *et al.,* 2010). As a result, some FFA can increase the levels of Low-Density Lipoprotein (LDL), often referred to as bad cholesterol, and be detrimental to health (Mariana *et al.,* 2020). The various negative impacts of FFA require these compounds to be eliminated.

A method that can be applied to purify and remove FFA is Liquid-Liquid Extraction (LLE) (Bou Orm *et al.,* 2020; Ferreira *et al.,* 2021; Homrich *et al.,* 2019; Tiritan *et al.,* 2020). This method utilises the difference in solubility of the components in a mixture of two immiscible solvents. The use of water, a highly polar substance in organic solvents such as anhydrous ethanol, tends to reduce the mutual solubility of the system. When a mixture of water and ethanol is used as a solvent for vegetable oil extraction, water can help extract polar FFA. In contrast, ethanol is more effective in extracting nonpolar triglycerides. LLE with ethanol results better than other polar solvents in the deacidification process (Homrich *et al.,* 2019; Bou Orm *et al.,* 2020; Tiritan *et al.,* 2020; Ferreira *et al.,* 2021). In addition, ethanol can also remove secondary compounds resulting from oxidation, giving vegetable oils an unpleasant odour and taste, accelerating degradation and shortening shelf life (Gonçalves *et al.,* 2016).

The following process to determine the level of FFA is to use the acidi-alkalimetric titration method (Mariana *et al.,* 2020; Nurulain *et al.,* 2021; Suriaini *et al.,* 2021). The acidi-alkalimetric method is a titration based on the nature of the solution to be titrated. Acidimetry is a way of determining the concentration of a quantitatively basic solution using an acidic standard solution. Alkalimetry is a way of determining the concentration of acidic solutions quantitatively using essential standard solutions (Nag *et al.,* 2023). The Indonesian National Standard (SNI) sets the limit of FFA levels in oil, such as 2% for coconut oil (SNI No. 01-7381-2008), 3% for palm oil (SNI No. 01-2901-2006), and 3% for corn oil (SNI No. 01-3555-1998). The amount of FFA levels in oil is indicated by the %FFA value. A high %FFA indicates that the free fatty acids present in an oil are also high, resulting in lower oil quality.

Based on this description, this research carried out experiments to separate FFA using Liquid-Liquid Extraction (LLE) and determine the levels of FFA using acidi-alkalimetric titration in corn oil and palm oil.

Methodology

Place of Research

This research was conducted at the Analytical Laboratory of the Chemistry Department, Faculty of Mathematics and Natural Sciences, Universitas Negeri Malang.

Materials

The tools used in this research are a burette, 100 mL separatory funnel, stative, clamp, erlenmeyer, beaker, measuring cup, drop pipette, column, hot plate, volume pipette, suction ball, and analytical balance. The materials used were corn oil, palm oil, aluminium foil, diethyl ether, anhydrous Na_2SO_4, ethanol solution, 0.1 M NaOH solution, and phenolphthalein (PP) indicator.

Research Procedures

Sample Preparation

Take each 30 g of corn oil and palm oil samples and put them into an Erlenmeyer. The Erlenmeyer filled with the sample is then covered with aluminium foil. The samples were then heated on a hotplate by measuring the sample temperature using a thermometer until the sample temperature was 200^0C.

Separation by Liquid-Liquid Extraction

Corn and palm oil samples were taken as much as 10 mL each. Then, it is put into a separatory funnel containing a mixture of solvents, such as 9.5 mL diethyl ether, 40 mL ethanol, and 0.5 mL water. Then, shaking was carried out for 4 minutes. Let stand for a few minutes until three layers are formed.

Prepared a chromatography column that contains cotton at the bottom end. Then, fill the column with 5 grams of anhydrous Na_2SO_4 as an adsorbent. Then, the bottom two layers flowed into the column, and the eluent was collected as ethanol containing FFA to determine their levels.

Qualitative Test of Fatty Acids in Samples

A mixture of NaOH and PP indicator was prepared in a ratio of 1-1 to produce a concentrated pink solution. Three drops of sample were taken and put into different test tubes. Next, 1 drop of pink testing solution was added, then shaken until a white precipitate formed, indicating that the test was positive for fatty acids.

Determination of Free Fatty Acid Levels

The alcohol layer solution obtained in the previous extraction process was weighed. Then, 10 mL of ethanol was put into a beaker and heated to 40^0C. Hot ethanol was added to the alcohol layer and allowed to stand for 15 minutes. After that, 3 drops of PP indicator were added. Titration was then carried out with 0.1 M NaOH solution until the solution's colour changed to pink, and the volume was recorded. Free Fatty Acid levels were calculated and expressed as %FFA.

Data Analysis Technique

The research method used is acid-alkalimetric titration, which calculates Free Fatty Acid levels using the percentage FFA.

$$\%FFA = \frac{ml\ NaoH\ x\ Molecular\ Weight\ Fatty\ Acids}{sample\ weight\ (g)\ x\ 100}\ x\ 100$$

Result and Discussion

Sample Preparation

In this research, samples of corn oil and palm oil were used. At first, 30 mL of each sample was taken and put into an Erlenmeyer. Erlenmeyer was covered with aluminium foil and then heated on a hot plate for 15 minutes at 200^0C. The heating process aims to break triglycerides into FFA (Untari, 2020). FFA contained in cooking oil is obtained through oxidation and hydrolysis processes during the heating or frying (Marlina et al., 2017). The preparation process of palm oil and corn oil samples is shown in Figure 1.

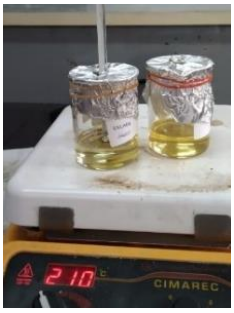

Figure 1. Sample preparation process

Separation by Liquid-Liquid Extraction

The method used in this separation experiment is the Liquid-Liquid Extraction (LLE) method with a separatory funnel because the compounds to be separated in the mixture are liquids. LLE can run in room conditions that require relatively little energy. The purpose of the extraction process is to separate FFA contained in oil based on their solubility in two or more immiscible solvents (Homrich *et al.*, 2019; Bou Orm *et al.*, 2020; Tiritan *et al.*, 2020; Ferreira *et al.*, 2021). The solvent was a mixture of polar and nonpolar solvents with 9.5 mL diethyl ether, 40 mL ethanol, and 0.5 mL water. Diethyl ether acts as a nonpolar solvent with a lower level of polarity than ethanol, isopropanol, and methanol (Rizkita *et al.*, 2021). In palm oil samples, there are triglyceride compounds that can dissolve in diethyl ether solvents because they are nonpolar (Wulandari *et al.,* 2017).Ethanol solvents can dissolve polar and nonpolar compounds because ethanol solvents are called semipolar volatile solvents (Arsa and Achmad, 2020). While water is used as a polar solvent. The LLE process is shown in Figure 2.

Figure 2. Extraction process

The choice of solvent type determines the success rate in the extraction process based on the solubility of the compound with the solvent. Based on Verdiana (2018), the like-dissolve-like principle states that a compound will dissolve in a solvent that has the same properties as the compound. A mixture of polar and nonpolar solvents can accelerate the separation process by breaking the combination of oil and FFA with lipoproteins or cell membranes and reducing surface tension (Verdiana, 2018). The oil sample used was 10 mL, and after all the solvents were added, shaking for 4 minutes began to form 3 layers, namely the top layer of diethyl ether, slightly cloudy white, the middle layer of ethanol rich in yellowish FFA, and the bottom layer of water shown in Figure 3.

Figure 3. Three layers formed after LLE

This happens because the density of water is more significant than ethanol and diethyl ether, namely water has a density of 997 kg/m^3, ethanol has a density of 789 kg/m^3, and diethyl ether has a density of 713 kg/m^3. A liquid that has a small density will be at the top, while a liquid with a more significant density will be at the bottom (Gumilar *et al.,* 2016).

FFA are polar compounds (Siratantri Mastuti, Fardiaz and Nur Faridah, 2019)The FFA distributed in ethanol is more than diethyl ether because the two solvents have different polarity indices. The polarity index of ethanol is 4.3, while that of diethyl ether is 2.8. This polarity index measures the polarity of a solvent.

The bottom layer is yellow, and the top layer is cloudy white. The bottom layer of corn oil is paler yellow than palm oil due to differences in the processing and storage of vegetable cooking oil. The oil extracts Natural colour pigments, where colours such as alpha and xanthophyll, beta carotene, chlorophyll, and anthocyanins are dissolved in the oil during processing. The darker the colour of the oil, the lower the quality of the oil.

Then, the bottom two layers are taken in the form of ethanol containing FFA and water by holding it in Erlenmeyer; the top layer of diethyl ether can be discarded. Next, the chromatography column shown in Figure 5 was prepared to remove the water content in the sample by using 5 grams of anhydrous Na_2SO_4 as an adsorbent on the column. The water previously used in the extraction process is only used to hydrolyse the oil (triglycerides) into glycerol and fatty acids, as shown in Figure 4. The oil hydrolysis reaction can occur at high temperatures through heating because it will produce high energy to break down the oil structure. In the oil hydrolysis reaction, glycerol and FFA will be formed, which will undergo further reactions to break down the glycerol molecules and FFA (Aziz *et al.,* 2019).

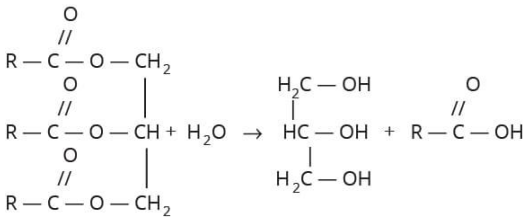

Figure 4. Oil hydrolysis reaction

In this process, anhydrous Na_2SO_4 plays a role in binding water that is still present in the oil (Apriza Marfina, 2019)In anhydrous Na_2SO_4, the molecule is released, and the O atom in SO_4^{2-}binds to H_2O to form a hydrogen

bond. As a result, there is a reaction between the two, so the water in the mixed layer is bound.

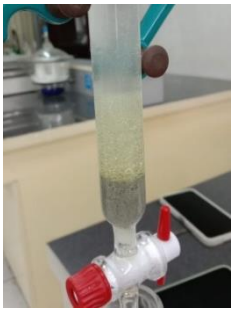

Figure 5. Chromatography column

Removing this moisture content causes the sample from two layers to remain in one layer, namely ethanol containing FFA.

Qualitative Test of Fatty Acids in Samples

This experiment aims to test the FFA levels in the samples used. FFA are free acids that do not have bonds as triglycerides (Irawan, Awalia and W.P.H, 2013) which are produced through hydrolysis and oxidation processes by joining neutral fats (Ati, Mauboy and Keneng, 2020). When FFA is reacted with a base, like NaOH, it will produce soap and water (Setianingsih and Riyani, 2019). The resulting soap is alkaline, which is detected through a change in the colour of the solution from colourless to pink through the addition of a phenolphthalein indicator to the NaOH solution. NaOH solution will react with free fatty acids as below:

$RCOOH\ (aq) + NaOH\ (aq) \rightarrow RCOONa\ (aq) + H_2O\ (l)$

The presence of FFA levels in both oil samples is evidenced by the disappearance of pink colour in corn and palm oil samples and the formation of saponification reaction. However, the saponification reaction of palm oil is more concentrated than that of corn oil because the FFA levels in palm oil are higher. Figure 6 shows the results of the FFA qualitative test.

Figure 6. FFA qualitative test results (right: corn oil, left: palm oil)

Determination of Free Fatty Acid Levels by Acidi-Alkalimetric Titration

In this experiment, the FFA levels of the ethanol layer obtained from the extraction process were determined using the acidi-alkalimetric titration method. FFA can be calculated with a sodium hydroxide (NaOH) standard solution after titration. FFA levels are the percentage of the amount of FFA in oil that NaOH can neutralise (Untari, Miksusanti and Al, 2020). NaOH is an alkaline solution that can determine FFA levels in oil because the use of NaOH can hydrolyze oil into glycerol and fatty acids (Mardiana, Andriani and Ridha, 2020).

The first step is the ethanol layer of the extraction results taken and obtained as much as 18 mL. The layer was then put into an Erlenmeyer. Next, the layer was added with 10 mL of warm ethanol. The addition of warm ethanol aims to dissolve the origin of fat so that the titration process is more straightforward because the oil cannot be dissolved in water (Faiqah, Imranah and Yusaerah, 2022). Then, the ethanol layer was allowed to stand for 15 minutes so that the dissolution of FFA in ethanol was perfect. After 15 minutes, the ethanol layer is tested with a phenolphthalein (PP) indicator, which will turn pink in an alkaline atmosphere (Ulfa, Retnaningsih and Aufa, 2017). The colour change from colourless to pink marks the titration's end point.

Furthermore, the ethanol layer was titrated using NaOH until its color changed from milky white to pink, as shown in Figure 7.

Figure 7. Titration result and top phase (right: titration result, left: top phase)

The required volume of NaOH was recorded, and the NaOH required for the sample was obtained, as shown in Table 1.

Table 1. FFA titration in 10 ml ethanol solvent

Sample	Variables observed	Average
Palm oil	The volume of the water phase	35 mL
	The volume of the organic phase	18 mL
	Organic phase titration	7,4 mL
Corn oil	The volume of the water phase	35 mL
	The volume of the organic phase	15 mL
	Organic phase titration	4,3 mL

Quantitative Analysis of Saturated Fatty Acids in Samples

After quantitative analysis of the FFA levels of cooking oil samples of corn and palm oil, the FFA levels of the samples can be determined. Therefore, the data on the FFA levels of the oils are presented in Table 2.

Table 2. Determination data of FFA levels in samples

Sample	FFA levels of the sample after treatment	FFA levels of the sample before treatment
Corn oil	0,95%	0,09%
Palm oil	1,26%	0,17%

Based on Table 2, it is known that FFA levels in the two samples are different. In palm oil, there was an increase in FFA levels after heating by 109%. Palm oil has saturated fatty acids with the most double bonds, so the more double bonds in the oil, the less resistant to heat (Rahmawati and Utami, 2022).

The increase in FFA levels before and after heating in corn oil was 9%. This is influenced by the nature of the oil, where the base material used is corn, which is resistant to high heating. This results in relatively low levels of FFA. Corn oil contains saturated fatty acids with double bonds that tend to be less than palm oil (Ula and Trisnawan, 2016).

The increase in FFA levels in the samples after heating shows that the temperature and duration of heating significantly affect the FFA levels of various types of oil. This is because the triglyceride bonds in glycerol and FFA are broken (Mardiana, Andriani and Ridha, 2020). The cause of cooking oil damage, especially vegetable oil, is the hydrolysis process. This is in accordance with Ketaren's research (2008), which states that FFA is produced from triglyceride hydrolysis by all enzymes belonging to the lipase group contained in animal and vegetable fats in the tissue. The longer this reaction lasts, the more FFA are formed.

Hydrolysis reactions that occur due to the heating of oils change their physical properties and result in chemical changes. As a result, the oil becomes rancid and produces trans fatty acids. These changes significantly affect the quality of the oil and potentially cause serious health problems. The effects include changes in cholesterol metabolism, increased risk of hypertensive disease, coronary heart disease, and even increased carcinogenic potential that can cause cancer (Karis *et al.,* 2022).

The quality of corn oil and palm oil can be related to the results of the qualitative tests conducted, which showed differences in the saponification reaction. Although both oil samples showed the presence of fatty acids based on the loss of pink colour, the intensity of the saponification reaction was higher in palm oil than in corn oil. This indicates that

palm oil's FFA levels are higher than corn oil's. Previous researchers validated this in determining FFA levels and found that palm oil has higher FFA levels than corn oil.

Conclusion

Separation of Free Fatty Acids (FFA) by Liquid-Liquid Extraction (LLE) using a polar-nonpolar solvent mixture of diethyl ether + ethanol + water showed that FFA can be distributed or dissolved in ethanol. In addition, the FFA levels in palm oil were 1.26% and 0.95% in corn oil. FFA levels of both oils are still below the SNI FFA percentage of 3%; besides that, it is found that the FFA levels in palm oil are higher than in corn oil. This research can be the basis for further development of free fatty acid analysis methods.

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