Reaktor

p-ISSN 0852 – 0798
e-ISSN 2407 – 5973

Website: http://ejournal.undip.ac.id/index.php/reaktor/

Reaktor, Vol. 23 No. 3, December Year 2023, pp. 92-100

Reduction of Free Fatty Acid Content of Tengkawang Oil through Adsorption Process Employing Palm Frond as An Adsorbent: Effect of Adsorbent Concentration

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(Received: 16 June 2022; Published: 31 January 2024)

Abstract

Crude tengkawang oil contains impurities such as free fatty acids that can significantly affect the oil's quality. These impurities have to be be removed as it can lead to a rancid odor. To solve this matter, we employed an adsorption process utilizing palm fronds adsorbent by using the cellulose content. Palm fronds are delignified by 10% NaOH. The adsorption process was conducted with various adsorbent concentration:5, 10, 15, 20, and 25%. The characterization of adsorbent was performed through FTIR and SAA. The FTIR result revealed that palm fronds contain some functional groups associated with lignocellulose. After delignification, the C=C lignin functional groups were still found in FTIR result. However, after adsorption process, there was a reduction of the O-H intensity, indicating that cellulose content and hydroxyl (-OH) group reacted with adsorbate. Furthermore, the presence of the C-H functional group indicates successful adsorption of the impurities such as free fatty acid by palm fronds adsorbent. The characterization of the palm frond adsorbent post-delignification revealed that the adsorbent falls into microporous range with surface area of 6.480 m^2/g, pore volume of 0.01138 cc/g, and pore radius of 12.4 Å. The research results demonstrated a decrease in the percentage of free fatty acids, along with increased capacity values and adsorption efficiency of Tengkawang oil. Notably, the highest performance was achieved with a 25% adsorbent concentration, resulting in a reduction of free fatty acids by 4.68%, an adsorption capacity of 207.19 mg/g, and an adsorption efficiency of 36.28%. This promising outcome potentially leads into further exploration of palm frond adsorbents in the purification of Tengkawang oil. Furthermore, the purified Tengkawang oil, with its improved quality, can be a potential raw material for many industries, including cosmetics industry.

Keywords: *adsorption; delignification; free fatty acid; tengkawang oil*

How to Cite This Article: Puspita, F.A., Sitanggang, E.P.O., Rakasiwi, R.R., Ivontianti, W.I., (2023), Reduction of Free Fatty Acid Content of Tengkawang Oil through Adsorption Process Employing Palm Frond as An Adsorbent: Effect of Adsorbent Concentration, Reaktor, 23 (3), 92 - 100, https://doi.org/10.14710/reaktor. 23. 3. 92 - 100

INTRODUCTION

Tengkawang is a very unique plant in Indonesia as it has multiple uses from its trunk, fruit, seeds, and bark, which can be used to produce a product with economic value (Fambayun, 2014). Tengkawang belongs to the Dipterocarpaceae family and grows in West Kalimantan, Central Kalimantan, East Kalimantan, and Sumatra. Tengkawang is the mascot of West Kalimantan, especially the Dayak tribe. To the Dayaks, Tengkawang is considered the tree of life because almost every part of it can be used on a daily basis, especially its fruits. Tengkawang fruits can be pressed to produce vegetable oil, which has a marketable value (Leksono dan Hakim, 2018). Tengkawang oil can be used as an ingredient in producing sweets, butter, chocolate, soap, medicine, and fodder (Kusumaningtyas et al., 2012). Tengkawang oil has many remarkable chemical properties, including a higher melting point than any other vegetable oil. Tengkawang oil can also be used to produce cosmetics such as lipstick, body lotion, sunscreen, and face masks, and it can also be used to heal a scar (Verschoor, 2019).

Tengkawang oil that has not been purified contains properties that could affect its quality, such as free fatty acid (FFA), color pigment, scent, etc. It is necessary to purify and separate the impurities in order to improve the quality of Tengkawang oil. It is necessary to purify and separate the impurities to improve the quality of Tengkawang oil. FFA content is one of the parameters used as a quality standard for Tengkawang oil. The amount of FFA in Tengkawang oil is very alarming. According to Minnifie (1999), Tengkawang oil contains about 5-25% FFA. The FFA can be considered an impurity because it makes the oil less stable compared to the other triglycerides, which means that the oil will evaporate at a high rate. It is also possible for the oil to become rancid due to high levels of FFA. Free fatty acid levels in vegetable oils are indicated by their acid number, and a high acid number indicates more FFA present in an oil, but it also indicates poorer oil quality and leading to a lower selling price (Sopianti et al., 2017). An alternative method for reducing FFA in tengkawang oil is absorption using a specific adsorbent (Syahwandi et al., 2019).

Palm fronds offer a compelling choice as an adsorbent due to their abundant availability as an agricultural waste product from palm plantations. This addresses the challenge of disposing of or managing such waste, contributing to sustainability by repurposing a material that might otherwise go unused. Utilizing palm fronds as an adsorbent presents an eco-friendly and cost-effective solution. Palm fronds have the most significant cellulose component, where the hydroxyl (OH) group within the component can be used for the absorption of free fatty acids in tengkawang oil. The cellulose in palm fronds is coated with lignin, a phenolic polymer network that strongly binds cellulose fibers (Sari et al., 2018). The presence of lignin reduces adsorption capacity in the absorption process due to lignin's ability to block the cellulose -OH group in the adsorbent by forming a covalent bond with lignin chromophores (Royana et al., 2016). Delignification, which destroys the lignocellulose structure of lignin, can be used to remove lignin content. This process breaks the lignin structure, allowing lignin-bound cellulose to detach (Lismeri et al., 2018). Alkaline solutions such as NaOH are commonly used for delignification. NaOH solution is used in this process because it breaks down lignin structure into crystalline and amorphous parts, allowing hemicelluloses to be separated (Pasue et al., 2014). OH- ion from NaOH breaks the basic lignin structure, so lignin dissolves easily, while Na^+ binds forming phenolic to lignin resulting in phenolic sodium (Safrianti et al., 2012).

This research aims to investigate the feasibility of using palm frond as an adsorbent and to determine the optimum adsorbent concentration, subsequently quantifying the free fatty acid (FFA) reduction efficiency of this approach in reducing FFA content in tengkawang oil. This research aims to bridge this gap by examining the efficacy of palm frond as a sustainable and locally available adsorbent, potentially offering a cost-effective solution for improving the quality of tengkawang oil.

METHODOLOGY

Materials

The materials used are palm fronds, tengkawang oil, aquadest, phenolphthalein indicator and sodium hydroxide. The palm fronds used in this research were collected from the palm plantation in 2nd Wonodadi Alley, 2nd Ayani Street, Desa Sungai Raya, Kecamatan Sungai Raya, Kabupaten Kuburaya.

The Preparation of the Adsorbent

To prepare the palm fronds adsorbent, the fronds were cleaned and then cut into small pieces of about 2-4 cm in size, so that the process of heating, storage, and crushing was much easier to be done (Putra et al., 2014). After cleaning the palm fronds, the palm fronds were placed in an oven at a temperature of 100 °C for one hour; the purpose of heating the fronds in the oven was to reduce the water content in the palm fronds so that microorganisms that could damage the adsorbent would not be present (Sulaiman et al., 2017).

To delignify the palm fronds powder that passed through the 100 mesh sieve, about 200g of palm fronds were weighed and put in a beaker glass with sodium hydroxide (NaOH) in a 1:10 ratio and 10% solution concentration. The mixture was stirred for two hours, then filtered using a filter paper to separate the solids from the solvent. Later, the palm fronds were washed with aquadest to eliminate the dissolved lignin and NaOH within the filtration until the pH neutral. As a consequence of washing, a brown-blackish solution was obtained from the palm fronds adsorbent, indicating the dissolution of lignin in NaOH (Safaria, 2013). The NaOH solution disperses the lignocellulose in palm fronds, whereby the OH^- ions from NaOH sever the bonds from the basic lignin structure. The Na+ ions bind themselves with lignin and form natrium phenolic. This sodium phenolate has a polar quality. Therefore, during the washing process, the sodium phenolate will be removed along with the washing water. The lignin dissolved in the solvent is marked Palm fronds powder that were obtained by using 100 mesh sieve got delignification by weighing it approximately 200 gr of palm fronds and then put it in the beaker glass to do delignification with natrium hydroxy (NaOH) in 1:10 ratio (b/v) and 10% solution concentration. The mixture was stirred for about 2 hours, then the ingredient separated from its solvent with filter paper and then washed with aquadest and put into the oven with temperature of 105°C for about 4 hours.

Adsorption process of free fatty acid Tengkawang oil was cariied out by varying its initial concentration at 5, 10, 15, 20 and 25%, then Tengkawang oil was weighed approximately 50 grams and heated till it reached the temperature of 70°C. Accordingly, palm fronds adsorbent was added into Tengkawang oil with black colour,The adsorption process for removing free fatty acid from Tengkawang oil was carried out using concentration levels of 5%, 10%, 15%, 20%, and 25%. Approximately 50 grams of Tengkawang oil was heated to a temperature of 70°C. After heating, palm fronds adsorbent was added to the Tengkawang oil. The mixture was then stirred with a magnetic stirrer at 300 rpm for about 60 minutes. The resulting mixture of the palm fronds adsorbent and the Tengkawang oil was then separated with a filter paper.

The adsorption process was conducted using different adsorbent concentrations, which was the variable of this research, in 5%, 10%, 15%, 20%, and 25% (w/w). This range of adsorbent concentrations was used to examine the relationship between the increase in adsorbent concentration and the free fatty acid content in Tengkawang oil. Setyawati et al. (2015) established that the adsorption process was affected by the increase in adsorbent concentration, as the greater the adsorbent concentration applied, the more effective the adsorption process becomes. Stirring during the adsorption process causes the molecules in the adsorbat and adsorbent to collide, which further accelerates the adsorption process (Widayatno et al., 2017). Subsequently, the adsorbent made of palm fronds and Tengkawang oil were separated using filter paper in order to analyze both the adsorbent and the oil separately.

Adsorbent and Tengkawang Oil Analysis

Analysis of palm fronds was done by using FTIR (Fourier Transform Infra Red), where this instrument used to identify cellulose function cluster and other function cluster which can be found in the sample, so that the produced adsorbance can be compared with correlation table and compoud spectrum that just found (Anam *et al*., 2007). In addition, the analysis of Tengkawang oil was carried out by calculating the free fatty acid content. The percentage of free fatty acid content, as per SNI 3748-2009, can be calculated using equation (1):

$$\%ALB = \frac{V\ NaOH \times N\ NaOH \times 28.2}{m} \qquad (1)$$

where:

% FFA	: Free Fatty Acid (%)
ml NaOH	: NaOH titration volume
N NaOH	: NaOH normality solution
M	: Sample weight
28.2	: Fatty Acid constant

Determination of the adsorption capacity can be calculated by the following equation (Agustrya et al., 2015):

$$Q = \frac{ma(Co-C)}{m} \qquad (2)$$

where:

Q	= Adsorption capacity (mg/g)
ma	= Tengkawang Oil mass (g)
m	= Adsorbent mass (g)
Co	= Initial concetration (mg/L)
C	= Last concentration (mg/L)

The efficiency of free fatty acids reduction in Tengkawang oil can be determined using the following equation (Agustrya et al., 2015):

$$EA(\%) = \frac{Co-Ca}{Co} \times 100\% \qquad (3)$$

where:

Co	= Initial concentration (ppm)
Ca	= Last concentration (ppm)

RESULTS AND DISCUSSION

This study shows that the delignification process leads to a difference in the weight of the palm fronds before and after the process, as shown in the following table.

Table 1. Weight Loss Percentage

Phase	Weight
Before Delignification	200 gram
After Delignification	91.5 gram
Weight Loss Percentage	54.25%

According to the table shown above, it can be observed that the palm fronds lost weight, indicating the dissolution of lignin during the delignification process (Safrianti et al., 2012). The phenomenon of weight loss on palm fronds after delignification process is caused by the characteristic of NaOH solution that can dissolve lignin component decently, so that half of the lignocellulose component fraction dissolved and caused the weight of palm fronds fraction decreased (Sudiyarmanto, 2018).

Results of FTIR analysis

Fig. 2 shows that the black line resulting from FTIR has Analysis of palm fronds was performed using Fourier Transform Infrared (FTIR) spectroscopy. The FTIR analysis was conducted in the wavelength range of 400 – 4000 cm^{-1}. A wave length of 3411.71 cm^{-1}, indicating the presence of O-H stretching function group absorption on hydroxy cellulose and an absorption area of 2922.56 cm^{-1}, which shows C-H stretching group on CH_2 from CH_2-OH cellulose group (Kumar et al., 2014). Additionally, the cellulose compound's C-H function group absorption is also shown at wave length of

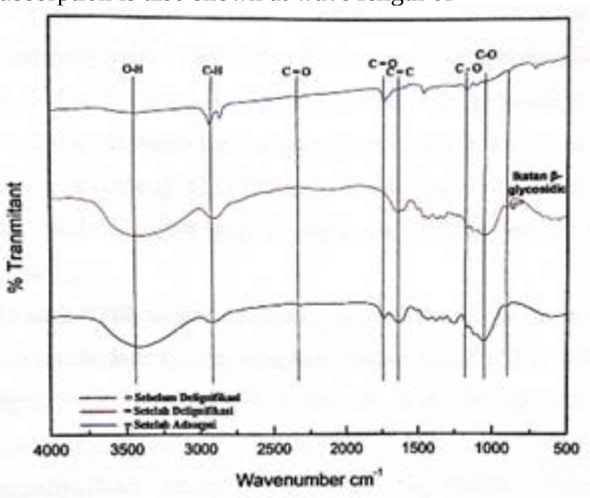

Fig. 2. FTIR Spectrum

1428.33 cm^{-1} (Schwanninger et al., 2011) and 1375.40 cm^{-1} (Wiwin et al., 2018). A peak absorption at a wave length of 1052.51 cm^{-1} indicates the presence of C-O-C function group on pyranous ring, which is a typical absorption from cellulose compound (Morain et al., 2008). The FTIR spectrum shows that the peak of absorption with wave number of 1734.26 cm^{-1} indicates the existence of C=O function group (1760-1665 cm^{-1}), which refers to C=O carbonyl. The group is a carboxylic acid group chain in the ferulic group and p-koumaril in lignin or is considered typical group found on lignin compound. The peak absorption at a wave number of 1250.22 cm^{-1} indicates the presence of a C-O-C group (1300-1000 cm-1), revealing the existence of lignin fiber compound in palm fronds (Kumar et al., 2014). Another lignin presence is shown by the peak at a wave length of 1635.49 cm^{-1}, which is the C=C functional group on the aromatic ring with wave numbers ranging from 1570-1700 cm^{-1} (Grube et al., 1999). Additionally, the C-H alkyl functional group on lignin is detected at a wave number of 607.94 cm^{-1} (Setiati et al., 2016). The peak absorption at a wave length of 2332.25 cm^{-1} reveals the presence of hemicellulose compounds, which is a typical absorption peak in the acetyl group and ester group on hemicellulose (Nafi'ah and Fahmi, 2021). The results of the FTIR analysis carried out after the delignification process showed absorption shifts indicating that a characteristic transformation had taken place, although the difference observed in comparison with the results obtained prior to delignification was not very far. The absorption of the hydroxyl cellulose O-H functional group showed a wave number shift from 3411.71 to 3416.04 cm^{-1} with decreasing intensity. A shift in wave number towards a longer wavelength indicated an increase in energy and strength of the bond, while a decrease in intensity suggested an increase in the amount of hydrogen bond caused by the detachment of lignin and hemicellulose compounds (Rahmiyani et al., 2021). The absorption of the carbonyl C=O functional group before delignification showed a typical absorption peak of hemicellulose compound at a wavelength of 2332.25 cm^{-1}, which underwent a decrease in intensity and a shift in wavelength to 2130.90 cm^{-1}, indicating a decrease in hemicellulose content (Tajalla et al., 2018). The C-O bending group is indicated by the wave number of 1163.71 cm^{-1}, where the absorption group is located between 1050-1300 cm^{-1}. This suggests the presence of C-O stretching on the structure from the cellulose component (Putera, 2012). The presence of the β-glycosidic bond absorption area can be observed at a wave length between 666-900 cm^{-1} with a peak absorption at a wave length of 897.13 cm^{-1}. This is a typical functional group from the cellulose compound, indicating the presence of CH_2 that bound with and formed pyranose on the cellulose compound (Pangau et al., 2017). The FTIR result after delignification showed that peak absorption indicating the existence of the lignin compound, which is normally found at wave lengths of 1734.26 and 1250.22 cm^{-1}, was not detected. However, there is still a wave number of 1635.49 cm^{-1} indicating the presence of the C=C functional group of aromatic ring from the lignin compound, left within the palm frond adsorbent as a result of the delignification process.

The FTIR spectrum of palm frond adsorbent used in the adsorption process to decrease free fatty acid in Tengkawang oil is displayed in Figure 2. Aziz et al. (2018) stated that free fatty acids have functional groups in the form of C=O, C-O, and C-H. As evidenced in Figure 2, after the adsorption process, a reduction in intensity of O-H occurred, and the occurrence of the reduction in intensity was caused by the adsorbent with cellulose content and hydroxyl (-OH) functional group, such as palm fronds, which reacted with the adsorbate found in Tengkawang oil. Furthermore, there were peak absorptions at wavelengths of 2922.23, 2852.13, 1465.78, and 720.30 cm^{-1}, which are absorption bands of the C-H functional group. This functional group indicates the presence of free fatty acid compounds and other pollutants that were adsorbed by the palm fronds adsorbent. Free fatty acid has a carboxylic acid C-O functional group, which appeared as a peak with a wavelength between 1050-1300 cm^{-1}, and can be found at a wavelength of 1173.60 cm^{-1}. The difference between the FTIR spectra before and after the delignification process and those after the adsorption process is the appearance of the carbonyl functional group of free fatty acids in Tengkawang oil, which can be found within the palm fronds adsorbent. This functional group consists of a carbon atom bonded with an oxygen atom (C=O) and appears at wavelengths between 1690-1760 cm^{-1}, and it can be found in the FTIR result at a wavelength of 1739.99 cm^{-1} (Fatarina et al., 2016).

Results of SAA analysis

The analysis of adsorbents was performed by observing its pore characteristic on palm fronds that had undergone delignification process using Surface Area Analyzer (SAA) instrument. This instrument is based on the principle of adsorption and desorption cycles of isothermal nitrogen gas on powder samples at liquid nitrogen temperature, and after the addition of nitrogen gas inside the sample tube, the pressure sensor records variable pressure data. Gas volume data, the amount of which is known, along with the result data from pressure increases have been made as BET equation, which is used as the basis of surface area calculation on samples (Rosyid et al., 2012).

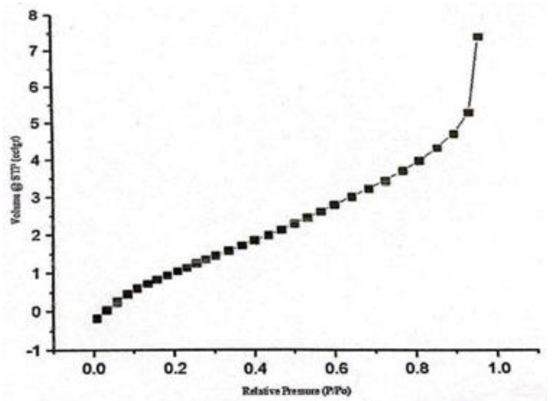

Fig. 3. Isotherm Adsorption Graph

Figure 3 is a chart depicting isothermal adsorption. It displays the quantity of nitrogen adsorption (N_2) relative to pressure (P/Po). Isothermal adsorption is an equilibrium relationship between the concentration of an adsorbate in a particular fluid and surface adsorbent at a constant temperature. The classification of isothermal adsorption by IUPAC shows that Fig. 3 belongs to type II of isothermal adsorption.This type is very common in physical adsorption and implies the formation of several layers on solid materials without pores, as well as on materials with micropores and mesopores (Astuti et al., 2018).The linear line on the graph represents the transition from monolayer to multilayer adsorption.The turning point of this type of isotherm usually appears close to the end of monolayer adsorption.The increase in relative pressure causes the second and subsequent layers to reach a saturation point, where the amount of adsorbate after adsorption becomes infinite (Rengga, 2020). The analysis of palm fronds after delignification yielded results indicating that they contain pores. This was proven by the SAA test, which revealed that the palm fronds have a surface area of 6.480 m2/g, pore volume of 0.01138 cc/g and pore radius of 12.4 A. The data obtained indicates that the pores in palm fronds fall within the micropore range, with a pore radius of 12.4 A or 1.24 nm found in palm fronds after the delignification process. This classification is based on the IUPAC, which defines micropores as materials with a radius less than 2 nm, mesopores as having a radius of 2-50 nm and macropores having a radius greater than 50 nm (Sukir, 2008).

Furthermore, the SAA analysis result indicates that the adsorption process has occurred in addition to the obtained adsorbent characteristics such as surface area, pore volume and pore radius. Adsoption is divided into two types: physical adsorption and chemical adsorption. Physical adsorption is a process that involves intermolecular forces such as Van der Waals or hydrogen bonds, in which the bound molecules are relatively weak and the released energy in the physical adsorption process is relatively low, i.e., less than 20 kJ/mol. In contrast, chemical adsorption occurs due to the formation of covalent bonds and ions between adsorbate molecules and adsorbent. Moreover, the amount of adsorption energy produced in chemical adsorption is more than 20 kJ/mol (Syauqia et al., 2011). The results show that the adsorption process between palm fronds adsorbent and free fatty acid on Tengkawang oil is physical adsorption, producing adsorption energy of 2.085 kJ/mol.

The free fatty acid content of Tengkawang oil was analyzed by calculating it. This was aimed to investigate the effect of adsorbent concentration on adsorbing free fatty acid on Tengkawang oil with different adsorbent concentration variety that had been decided, the concentration variety are one of the factor that could affect the adsorption process (Iffah et al., 2019). The research results showed the percentage of free fatty acid content in Tengkawang oil which was obtained through calculations and can be seen in Table 2.

Table 2 shows the percentage of free fatty acid content from Tengkawan oil treated with adsorbent created from palm fronds. As indicated in the table, the reduction of free fatty acid content in the Tengkawang oil was due to the increase in adsorbent concentration. The lowest level of free fatty acid content was observed at a 25% adsorbent concentration, which was reduced as much as 4.68% from 12.9% to 8.22% of free fatty acid content, this shows that the more adsorbent concentration is added, the more interaction occurs between adsorbent and adsorbat, where the concentration increase will expand the surface area and availability of active site on adsorbent to increase the free fatty acid on Tengkawang oil (Aziz et al., 2018).

Table 2. The Reduction of FFA Content

Adsorbent Concentration (%)	% FFA
0	12.9
5	12.4
10	11.94
15	10.98
20	10.46
25	8.22

The adsorption process occurs between palm fronds adsorbent and free fatty acid found in Tengkawang oil is a physical adsorbeption. This is caused by the interaction between palm fronds and free fatty acid that involves hydrogen bonding. According to Adhani et al. (2016), the ability of palm fronds adsorbent to adsorb free fatty acid of Tengkawang oil is caused by the presence of hydroxyl group on the adsorbent surface, which can bond with carbonyl group of free fatty acid on the oil.Through this interaction, some of the O atom on free fatty acid will undergo hydrogen bonding with H atom on hydroxyl cellulose, which causes free fatty acid to be adsorbed by palm fronds adsorbent. Furthermore, the reduction mechanism of free fatty acid on the oil went through adsorption process that can occur because of -OH group reacted with -COOH group (carboxylic acid) caused H atom from the compound will react and produce RCOO and H2O compound (Ratno et al., 2013). The following is the reaction mechanism between cellulose and free fatty acid (Royana et al., 2016):

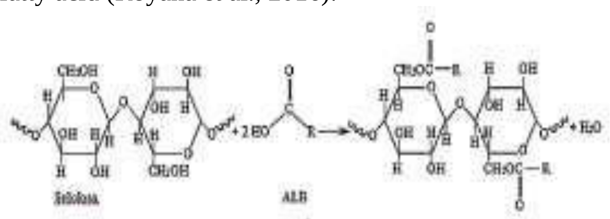

Fig. 4. Reaction Mechanism between cellulose and free fatty acid

Apart from comprehending how adsorbent concentration affects the absorption of free fatty acids in oil, adsorption capacity is another factor that can influence the adsorption process. Several conditions can have an effect on the adsorption capacity in the adsorption of the adsorbate, and one of them is the concentration of the adsorbent. Based on the results of the analysis, an increase in the concentration of the adsorbent can affect the adsorption capacity, where a higher adsorption capacity indicates that a larger amount of free fatty acids can be adsorbed by the palm fronds adsorbent. The adsorption capacity of palm fronds adsorbent can be seen in the Table 3:

Table 3. FFA Adsorption Capacity

Adsorbent Concentration (%)	Q (mg/L)
5	110.68
10	106.25
15	141.67
20	135.03
25	207.19

The data from the research show that the adsorption capacity value does not directly increase with the concentration of the adsorbent. As shown in Table 3, the adsorption capacity varied based on the concentration of the adsorbent. For concentrations of 10% and 20%, the adsorption capacity reduced to 104.99 and 135.03 mg/g respectively, whereas for concentrations of 15% and 25%, the adsorption capacity increased to 141.67 and 207.19 mg/g respectively. In theory, the concentration of the adsorbent determines its adsorption capacity in the adsorption process. The decrement of the free fatty acid percentage increases with the increase in adsorption concentration because a large amount of adsorbent concentration can expand the surface area and availability of the active site found within the palm fronds adsorbent, which binds free fatty acid on Tengkawang oil during adsorption. (Yenti et al., 2018). The fluctuation of adsorption capacity in this study is due to the lack of an optimum adsorption capacity caused by the inactive adsorbent sites which are not yet occupied by the adsorbate, resulting in an unstable adsorption capacity at different adsorbent concentrations (Aisyah et al., 2019). In addition to the adsorption capacity, the influence of the adsorbent concentration on the adsorption process can be described using the adsorbent effectiveness unit. The findings of the analysis of the effectiveness of adsorption for Tengkawang oil using palm fronds as an adsorbent are presented below:

Table 4. FFA Adsorption Effectiveness

Adsorbent Concentration (%)	EA (mg/L)
5	3.88
10	7.44
15	14.88
20	18.91
25	36.28

Adsorbent effectiveness is reported in percentage that was obtained from the comparison value between the absorbed adsorbate concentration in comparison with the initial adsorbate concentration value. This study indicates that the effectiveness of adsorption increases proportionally with the concentration of palm fronds adsorbent. According to Nuhasni et al. (2012), an increase in adsorbent concentration leads to an increase in adsorbent amount, which results in more adsorbate being adsorbed. The increased amount of adsorbent is related to the increased number of adsorbent particles and subsequenly its surface area, which increases the amount of free fatty acid spots that are bound, which is why the adsorption efficiency value increased (Alifaturrahma and Hendriyanto, 2017).

CONCLUSION

In conclusion, the research results indicate that the concentration of adsorbent plays a significant role in reducing the amount of free fatty acids in Tengkawang oil. The most effective concentration of adsorbent for this purpose was found to be 25%, which resulted in a significant reduction in free fatty acid content from 12.9% to 8.22%, a reduction percentage of 4.68%. The optimum concentration of 25% with the highest value of absorption capacity was 207.19 mg/g and the absorption efficiency value was 36.28%. These results emphasize the potential of the palm leaf adsorbent to improve the quality of the Tengkawang oil by reducing the content of free fatty acids.

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