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Quality Improvement of Kesambi-Seed Oil using Free Fatty Acids and Hydrogen Cyanide Adsorption

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Abstract. Kesambi-seed (*Schleichera oleosa*) is an Indonesian potential feedstock to produce vegetable oil for energy resources and oleochemicals. Crude Kesambi-seed oil (KSO) contains 8.43% free fatty acids (FFA) and 128 ppm hydrogen cyanide (HCN), which exceeds the Indonesian National Standard (SNI) for refined oil. Purification process is needed for crude KSO to be high quality vegetable oil. This study aimed to improve the quality of KSO by reducing the FFA and HCN contents using adsorption. The adsorption equilibrium is also determined to obtain the equilibrium constants. The purification process was developed in 2 steps, i.e., degumming and adsorption. Degumming was developed using 2.5% v/v H_3PO_4 to separate phospholipids/phosphatides in the crude oil, while adsorption using activated carbon was introduced to reduce the FFA and HCN contents. Kesambi-seed oil purification using activated carbon adsorption could be used as one-step refining for bleaching and reducing FFA and HCN. More adsorbent is needed to reach the FFA standard for the shorter adsorption time. Adsorption using 10% activated carbon for 24 hours reduced hydrogen cyanide up to 36 mg/kg oil, lightened the oil color, and achieved the highest yield (74.62%). Meanwhile, the activated carbon concentration of 40% w/w for 96 hours obtained 0.16% FFA. All three parameters have fulfilled the Indonesian National Standard for refined vegetable oil. Furthermore, the Freundlich model best fits equilibrium adsorption of FFA in the KSO.

Keywords: Adsorption, Activated Carbon, Degumming, Free Fatty Acid, Kesambi-Seed Oil

INTRODUCTION

Oils and fats are very important materials that used in the production of oleochemicals. They are used in various chemical products, lubricants, cosmetics, biofuel, and food products. Food and Agriculture Organization (FAO) reported that the average growth rate of world vegetable oil production from 2015 to 2020 is 3.22% per year, while the increasing consumption has a greater rate (3.55% per year). This fact indicates excess requests in the global vegetable oil market. Due to the rising demand for oleochemical products, there should be an increase in oilseed production. The main feedstock for the oleochemical industry is palm oil. However, other resources are now needed to supply the oilseed requirement.

Oleochemical, which are fatty acids, are consumed for the production of surfactants, detergents, soaps, and lubricants. They are

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also used in the production of varnishes and pharmaceuticals. This is all expected to provide a growth opportunity for the oleochemicals market.

Along with the increasing need for raw materials for the oleochemical industry, it is necessary to use other vegetable oils that potential for feedstocks. One of this potential feedstocks in Indonesia is Kesambi (*Schleichera oleosa*) seed, which contains about 70% oil with 60% unsaturated fatty acids as a major component. Kesambi-seed oil (KSO) has so far been used for non-edible products such as biodiesel feedstock. However, with proper purification, non-edible oils may be used as food ingredients, such as cooking oil, margarine, and others.

Crude KSO contains hydrogen cyanide (HCN) and also high free fatty acids (FFA), which have to be reduced (Tamara *et al*., 2020). High FFA indicates oil deterioration or lower quality of oil. Free fatty acids are caused by oil hydrolysis and rancidity, which produce an unpleasant odor. Kesambi-seeds were extracted into oil and produced 64% yield with 0.1% water content (Tamara *et al*., 2020). KSO refining also has been developed in three steps (degumming, adsorption using zeolite and neutralization). Degumming using H_3PO_4 and adsorption using zeolite 25% w/w in oil for 14 hours only reduce FFA to 6.46%. It did not fulfill the Indonesian National Standard and still needed neutralization using NaOH 17% to achieve the FFA standard (Putri *et al*., 2022).

Previous research has been developed to reduce FFA from crude or waste vegetable oil using various adsorbents. Bentonite (Ifa *et al*., 2021), zeolite (Putranti *et al*., 2018), activated clay (Baptiste *et al*., 2020), activated carbon (Rengga *et al*., 2021), and anionic resins (Mhadmhan *et al*., 2023; Wirawan *et al*., 2022) are usually used as FFA adsorbent. Activated carbon from many bioresources called biosorbents were also utilized (Rahayu *et al*., 2018). Silicate compounds from organic resources were also used (Clowutimon *et al*., 2011). The urea inclusion technique was also developed for saturated FFA to be adsorbed into urea molecules. It was addressed to separate it from unsaturated ones (Setyawardhani *et al*., 2015, 2018, 2019).

Crude vegetable oil purification is developed in two ways: chemical and physical refining. Degumming, bleaching, dewaxing, and deodorization could be done in chemical and also physical refining, but neutralization is chemical only. Both of them have their advantages and have disadvantages, including investment cost, the amount of energy consumed, product yield, and many others (Gharby, 2022; Ma *et al*., 2017).

Typically, each refining process serves the only purpose of purification. For instance, degumming aims to eliminate gum, bleaching is employed to enhance the color brightness, and neutralization is carried out to eliminate Free Fatty Acids (FFA), among other processes. This research implemented only two refining steps, i.e., degumming and adsorption, which were expected to eliminate gum, enhance the color brightness and reduce FFA and HCN for KSO. It was done to improve the quality of KSO for fulfilling the Indonesian National Standard for refined oil (Standar Nasional Indonesia, 2013). Especially for the adsorption step, it was done in an equilibrium stage to determine the equilibrium models and predict the adsorbent dose for certain adsorption times.

MATERIALS AND METHODS

Materials

Kesambi-seed crude oil was obtained from Probolinggo, East Java, Indonesia. Palm

kernel-shell activated carbon from the local market was used as an adsorbent. The activated carbon surface area was 530-1605 cm^2/g, and the particle size was 325 mesh. Crude KSO was degummed using 20% H_3PO_4 solution. The H_3PO_4 was from Merck with 85% purity. FFA content was determined by titrimetric analysis using food grade ethanol (95% from local market), NaOH (99% purity Merck, Germany), and phenolphthalein indicator (Merck, Germany). Sodium carbonate (Na_2CO_3) anhydrous (99% purity Merck, Germany); tartaric acid ($C_4H_6O_6$) (99.5% purity, Sigma Aldrich), and picric acid (98% purity, Sigma Aldrich) were used for HCN qualitative analysis, while Oxalic acid ($C_2H_2O_4$) (99% purity, Merck, Germany); NH_4OH (Sigma Aldrich); KI (99% purity Merck, Germany); and $AgNO_3$ (99% purity, Sigma Aldrich) were used for the HCN content analysis.

Apparatus

Degumming was held in a three-necked flask equipped with a heater, stirrer, and cooler, as described in Figure 1. The oil and gum mixture was separated using a separator funnel. Erlenmeyer was used to develop the adsorption and a Buchner funnel and filter paper to filtrate the oil from adsorbent. Analytical balance, oven, desiccator, beaker glass, porcelain cup, erlenmeyer, burette, stative, clamp, thermometer, pipettes, weighing bottles, centrifuges, pipettes, Kjeldahl flasks, and distillation flasks were used for analysis.

Experimental Procedures

Crude KSO was refined in this research in two steps, i.e., degumming using H_3PO_4 solution and adsorption using activated carbon. The second step was completely for bleaching, removed HCN, and reduced FFA.

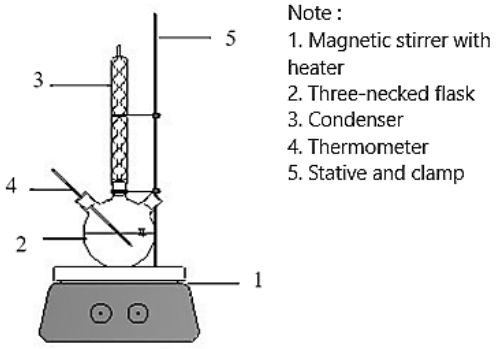

Fig. 1: Degumming aparatus

The first step of the refining process was crude Kesambi-seed oil degumming, intended to remove gum and impurities in the oil. Degumming was conducted by heating 100 mL of oil until 70°C then cooling to 50°C for 30 and 10 minutes, respectively. Twenty percent of the H_3PO_4 solution was added to the oil in a 2.5 v/v acid/oil ratio, with 500 rpm stirring for 20 minutes. The gum was separated from the oil by filter paper and then dried in an oven at 75°C for 1 hour. The gum was weighed to measure the yield of degumming. Meanwhile, the oil was washed with distilled water at 50°C until the pH of the distilled water was 6.5-7.

The following step, known as the adsorption process, was conducted to decrease the levels of FFA and HCN and enhance the color of the oil. The adsorbent used in this research was activated carbon from palm shells. Activated carbon was added to the oil in various concentrations (10%, 20%, 30%, 40%) w/w. The adsorption was conducted without stirring in various immersion times (24, 48, 72, 96 hours) at room temperature. At each step's end, the oil and adsorbent mixtures were filtered until the activated carbon was separated from the oil.

The free fatty acid content in the oil was analyzed using the titration method according to AOCS Official Method Ca 5a-40. The volume and concentration of NaOH (V

NaOH, L and M NaOH, mole/L) of NaOH were used to calculate the FFA contents (% FFA) using Eq. (1), where the MW_{FFA} and W_{oil} were the molecular weight of free fatty acid (g/mol) and weight of Kesambi-seed oil (g), respectively.

$$\% FFA = \frac{V_{NaOH} M_{NaOH} MW_{FFA}}{W_{oil}} x\ 100\% \quad (1)$$

The oil which was obtained from the whole purification then weighted and calculated as the yield of the KSO, as Eq. (2). $Woil_{(c)}$ and $Woil_{(r)}$ are the weights of crude KSO and refined KSO, respectively.

$$Oil\ yield\ (\%) = \frac{W\ oil_{(r)}}{W\ oil_{(c)}} x\ 100\% \quad (2)$$

Hydrogen cyanide was analyzed both in the qualitative and quantitative tests. They referred to AOAC Official Method for Cyanogenetic Glycosides in Feeds (AOAC, 2000). A simple and qualitative technique was used for visual inspection; that is, picric acid test strips form a red color in the presence of hydrocyanic acid (Oshima *et al.*, 2003). Afterwards, a quantitative analysis was developed for adsorption of oil with 10% w/w activated carbon. Hydrogen cyanide content was calculated by Eq. (3), while V_{AgNO3} was silver nitrate volume in mL.

$$HCN = \frac{V\ AgNO_3 x\ 0.54}{W\ oil_{(r)}} x\ 1000\ mg/kg \quad (3)$$

Data Analysis

Equation (1), (2) and (3) were used to determine the FFA, the oil yield and HCN content. Percentage of FFA then was used to predict the adsorbent dose to achieve the standard for certain adsorption time. Moreover, they were also used to study the equilibrium condition.

Hydrogen cyanide in KSO was analysed using qualitative and quantitative methods. Both results were then checked for the compatibility with the standard.

Free fatty adsorption was conducted in an equilibrium state. Linear model, Langmuir, Freundlich and Temkin equation were used to represent the adsorption equilibrium in Eq. (4), Eq. (5), Eq. (6) and Eq. (7), respectively. Free fatty acid concentrations in the liquid phase (oil) and solid phase (activated carbon) are presented as C_e and Q_e. Then a plot of Q_e versus C_e generates K constant for the Linear model, which is the slope of the graph. Linearization of Eq. (5) and plotted ln Q_e versus ln C_e resulted in k_f and n as Freundlich constants. Likewise with Eq. (6) for Langmuir equation, plotting C_e/Q_e versus C_e obtained maximum FFA concentration in activated carbon (Q_m) and Langmuir constant (K_L) (Setyawardhani *et al.*, 2019). Temkin isotherm was represented by plotting Q_e versus ln C_e (Eq. (7)) to obtain A and B parameters.

$$Q_e = KC_e \quad (4)$$

$$Q_e = k_f C_e^n \quad (5)$$

$$Q_e = Q_m \frac{K_L C_e}{1 + K_L C_e} \quad (6)$$

$$Q_e = B \ln C_e + B \ln A \quad (7)$$

RESULTS AND DISCUSSION

During the degumming process, 48.32 g of gum was separated from 100 mL (78 g) of KSO (equivalent to 38% yield). The FFA content in the oil before degumming was 8.43%, while after degumming, it decreased to 5.6%. Degumming was able to reduce FFA content in KSO by 33.6%. However, this value still not meet Indonesian National Standard

(SNI) for FFA content in refined, bleached, and deodorized (RBD) oil (which is stated at 0.3%). Then, it still needs further steps of purification.

Neutralization using alkaline solution is usually used to remove FFA from the oil, but this tends to form soap due to the reaction between alkaline and triglycerides, which reduces the oil yield (Ma *et al*., 2017). Moreover, oil losses are an important risk (Chumsantea *et al*., 2012). Free fatty acids may be removed from the oil with physical treatment as adsorption. It has more advantages because of the lower cost and no harm risk due to the chemicals involved in the product.

Some previous research determined that adsorption could reduce both FFA and HCN contained in vegetable oil. In this research, activated carbon from palm shells was used as an adsorbent. Activated carbon was immersed in oil with variations of time for 24, 48, 72, and 96 hours and variations in the concentration of 10% w/w, 20% w/w, 30% w/w, and 40% w/w.

Hydrogen Cyanide Purification

Hydrogen cyanide (HCN) content in crude KSO was 128 ppm. HCN is toxic for humans when it is consumed more than 40 mg/kg of human body weight. So HCN must be removed from vegetable oil. The HCN adsorption was conducted in 10% w/w adsorbent in the oil for 24 hours. Hydrogen cyanide analysis used both qualitative and quantitative tests. The qualitative test showed that the picrate paper remained yellow after the adsorption, which indicated that the HCN in the oil was only very little or even absent. Moreover, the quantitative test showed that HCN content decreased to 36 mg/kg, which fulfilled the Indonesian National Standard (SNI) no. 7709: 2019 for RBD oil. Due to this result, no more adsorbent dose or longer adsorption time was needed to achieve the HCN standard.

Kesambi-Seed Oil Yield in Adsorption

Oil yield is the oil weight obtained from the adsorption. The yield from the immersion of activated carbon adsorbent at 10% w/w, 20% w/w, 30% w/w, and 40% w/w from 50 grams of oil are shown in Table 1. It showed that a higher concentration of the adsorbent obtained a lower yield. A higher concentration of the adsorbent means more sites to accommodate the FFA molecules to adsorbed, so the weight of the oil yield will be reduced. Lower FFA means an advantage for the oil quality, but lower yield is considered as a disadvantage.

Table 1. Kesambi-seed oil yield

Adsorbent Concentration (%w/w)	Oil yield (%)
10	74.62
20	59.04
30	42.08
40	22.11

Kesambi-Seed Oil Bleaching using Adsorption

Kesambi seed oil, which was degummed and adsorbed for 24 hours using 10% activated carbon from palm shells, has lower FFA content and 36 mg/kg HCN, which fulfilled the oil quality standard of SNI No. 7709 : 2019. After the adsorption, Kesambi-seed oil also had a bright-yellow color, which met the quality standard according to SNI. Figure 2 shows the color change from crude oil to refined oil using adsorption. As well to the HCN result, no more adsorbent dose and no longer adsorption time were needed to brighten the oil color.

Adsorption Equilibrium Studies

Many studies on FFA adsorption from vegetable oil have been studied the kinetic models. The adsorption was usually held at various temperatures with mixing. They determined that equilibrium conditions were achieved in several hours of adsorption. In this research, batch adsorption was conducted in an environmental condition, room temperature, without stirring, but it took longer than several hours to ensure that equilibrium was achieved.

Fig. 2: Crude and bleached Kesambi-seed oil using adsorption

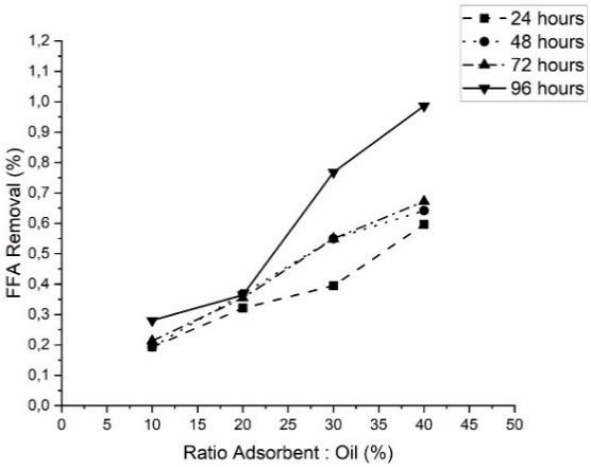

Fig. 3: FFA removal (%) in various adsorbent concentrations

According to the Indonesian National Standard (SNI), the maximum FFA content in the oil is 0.3%. Figure 3 shows that to achieve the condition, 10-30% activated carbon still does not fulfill the standard, even for 96 hours of adsorption. However, 40% w/w of activated carbon was enough to reduce the FFA content to 0.16% after 96 hours of immersion.

A linearization technique was used to predict the activated carbon concentration that could fulfill the standards. Figure 4 (a-d) shows the equation to evaluate the amount of adsorbent needed at certain adsorption times.

Linearization of each graph in Figure 4 resulted in equations to predict adsorbent: oil ratio to achieve the FFA concentration, which fulfilled the SNI. Table 2 indicates the activated carbon: oil ratio needed to meet the SNI for FFA content.

Table 2. Predicted activated carbon : oil ratio for various adsorption times

Adsorption time (hours)	Activated carbon : oil ratio (%)
24	69.50
48	61.75
72	56.80
96	39.20

This research focuses on the equilibrium condition of the adsorption. The equilibrium behavior of FFA adsorption in the KSO was studied by varying adsorbent (activated carbon) : oil ratio. The equilibrium study measured FFA levels in the oil after being adsorbed for 24-96 hours at room temperature. Increasing the adsorbent amount affected the rising of the % removal of FFA, as shown in Figure 4. However, during the adsorption process, the amount of active site increases and turns to be unsaturated. So, the density of FFA in the adsorbent is decreased, as presented in Figure 5.

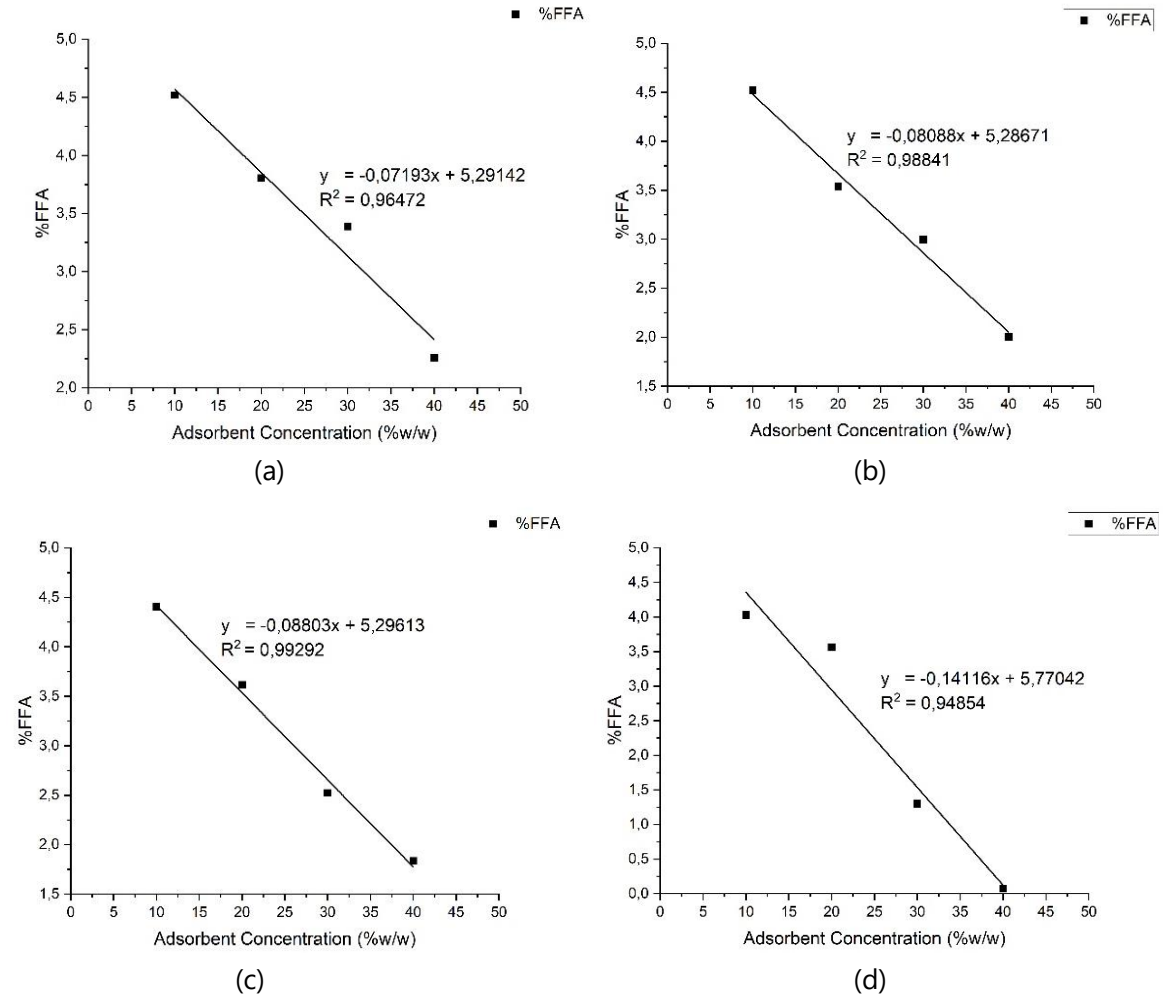

Fig. 4: Predicted activated carbon concentration needed for (a) 24; (b) 48; (c) 72 and (d) 96 hours of adsorption

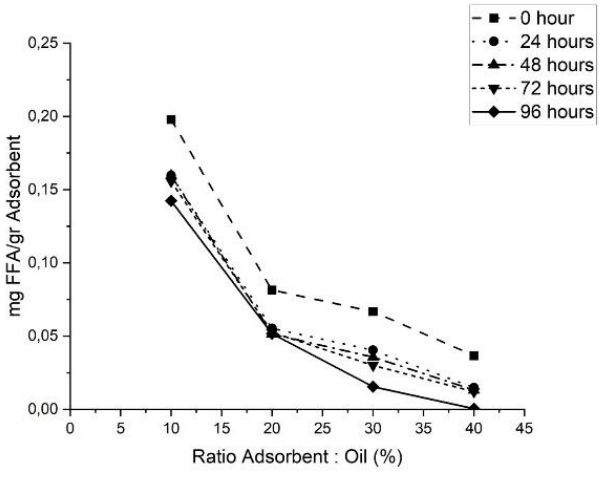

Fig. 5: FFA density in the adsorbent concentration

Figure 6 shows that after one day (24 hours), the FFA content was almost constant until 72 hours. However, after 72 hours, the FFA content was increased. It may be due to the decreasing of the active sites of the carbon. As adsorption using activated carbon tends to be chemisorption, it is probably a reversible process that lead to the adsorbed FFA to release from the adsorbent (Wirawan *et al*., 2022). So, free fatty acids concentration on the 3rd day (72 hours) was used to define the equilibrium data.

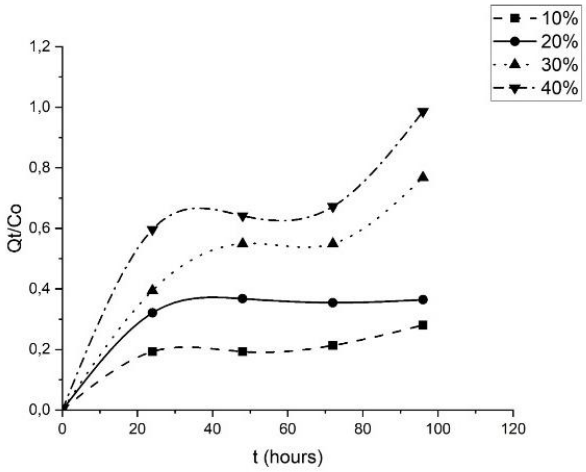

Fig. 6: FFA content (%) in various concentrations of activated carbon

The correlation between FFA residue in the oil ($mg\ FFA/g$ KSO) and FFA content on the adsorbent is evaluated using Linear isotherm (Eq. (4)), Freundlich correlation (Eq. (5)), Langmuir equation (Eq. (6)) and Temkin equation (Eq. (7)). The adsorption equilibrium was evaluated by fitting the data on adsorption isotherm using Linear, Freundlich, Langmuir and Temkin model. They were determined to provide the most suitable isotherm, defined by the highest R-squared value (Setyawardhani *et al*., 2019). C_e and Q_e presented free fatty acid concentrations in the oil and adsorbent, respectively. Q_m was the maximum concentration of FFA in the adsorbent. Then K, K_L, K_f, n, A and B indicate the Linear, Langmuir, Freundlich and Temkin constants.

Based on the monolayer's adsorption, the Langmuir adsorption isotherm is calculated assuming that the energy in the adsorption system was constant. The Langmuir model's validation points to a uniform distribution of active sites on the carbon surface. Eq. (8) was used to calculate the Langmuir model (R_L) dimensionless separation factor. This research results are 0.414 (between 0 and 1), indicating that the adsorption is advantageous, and that the Langmuir model is suitable for a homogeneous system (Ayawei et al., 20 17).

$$R_L = \frac{1}{1+K_L C_0} \quad (8)$$

The Freundlich adsorption isotherm indicates heterogeneous systems and provides multilayer adsorption on the active surface. Freundlich isotherm model recommends a heterogeneity of the surface of the adsorbents. The surface of the adsorbent is not uniform. Then, it is associated with multi-molecular layer adsorption. Adsorption takes place from the strongest energy sites to the lowest energy sites. FFAs are adsorbed on different sites which have low affinity. It is indicated by the low value of the n coefficients resulting in this research (Baptiste *et al*., 2020).

The Temkin adsorption isotherm takes into account how indirect adsorbate/ adsorbate interactions impact the adsorption process. Additionally, it is anticipated that as surface coverage increases, the heat of adsorption (ΔH ads) of all molecules in the layer will fall linearly rather than staying constant. The primary benefit of the Temkin isotherm is that it makes it possible to calculate the adsorption heat. An exothermic adsorption process would occur if the value of is positive (Benjelloun *et al*., 2021). This research was developed at room temperature and exhibited the B value of 0.0166, which denoted the exothermic condition.

Figure 7 – Figure 10 shows the adsorption equilibrium isotherm. Freundlich isotherm model showed the best fitting of all, represented by the highest R-squared value on each chart. This was in accordance with similar previous research about FFA adsorption (Putranti *et al*., 2018; Rengga *et al*., 2021; Wirawan *et al*., 2022). Table 3

summarizes the equilibrium parameters of the four models.

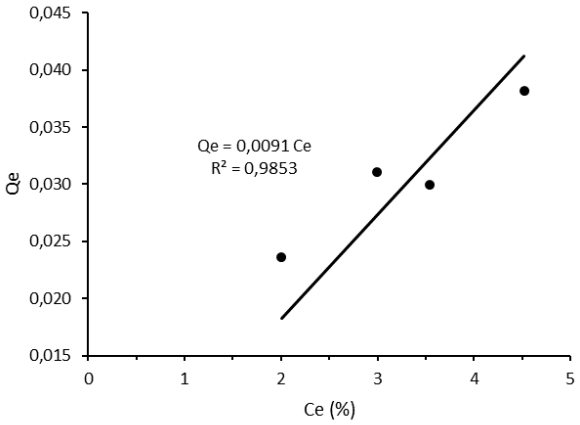

Fig. 7: Linear isotherm for FFA adsorption in activated carbon

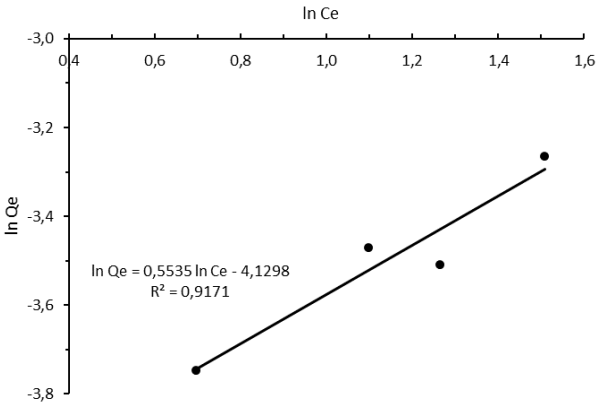

Fig. 8: Freundlich isotherm for FFA adsorption in activated carbon

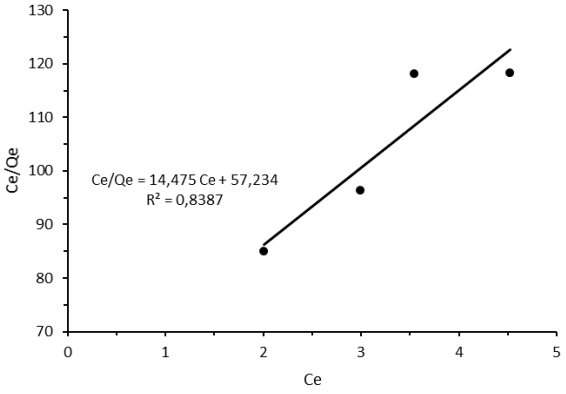

Fig. 9: Langmuir isotherm for FFA adsorption in activated carbon

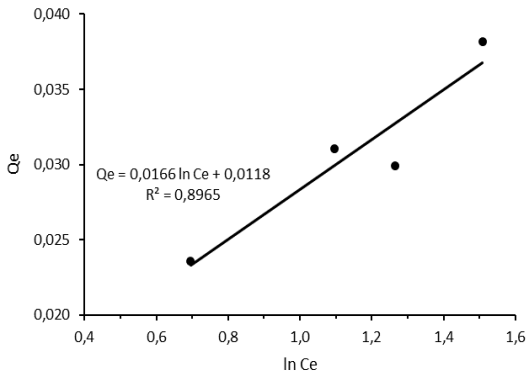

Fig. 10: Temkin isotherm for FFA adsorption in activated carbon

Table 3. Equilibrium adsorption constants

Const.	Linear	Freundlich	Langmuir	Temkin
K	0.0091			
K_f		0.01609		
n		0.5535		
Q_m			0.0691	
K_L			0.2529	
A				2.0357
B				0.0166

Comparison with Previous Research

Many studies have been carried out on the adsorption of FFA from vegetable oils using various adsorbents. Some of them use activated carbon from various organic materials. This study uses activated carbon from palm shells. Table 4 compares the results of reducing FFA from various oils using activated carbon.

Free fatty acid adsorption using activated carbon from pineapple dreg, coconut husk, and baggase succesfully removed 57-65% FFA from waste cooking oil (Rahayu *et al*., 2018). Meanwhile, activated carbon from Durian outer skin, cassava peel, and corncobs removed 17.5%, 40% and 25% FFA, respectively (Simatupang *et al*., 2020). Both of studies conducted adsorption with continuous stirring. This research successfully removed 46.4 % -78.65 % FFA only in an immersion process (without stirring) for the

same adsorption times. It also needed a lower ratio of the adsorbent : oil to achieve higher FFA removal. It showed that palm shell-activated carbon could adsorb FFA better.

Table 4. Percentage of FFA removal after adsorption

Activated carbon raw material	Adsorption time (hour)	Ratio carbon: oil	FFA removal (%)
Pineapple dreg	72	1 : 15	57.0
Coconut husk	72	1 : 15	60.0
Baggase	72	1 : 15	65.0
Durian peel	24	1 : 15	17.5
Cassava peel	24	1 : 15	40.0
Corn cobs	24	1 : 15	25.0
Palm shell	24	1 : 10	46.4
Palm shell	24	1 : 5	54.9
Palm shell	24	1 : 3.3	59.7
Palm shell	24	1 : 2.5	73.9
Palm shell	72	1 : 10	47.0
Palm shell	72	1 : 5	57.3
Palm shell	72	1 : 3.3	70.3
Palm shell	72	1 : 2.5	78.7

CONCLUSION

Kesambi-seed oil purification using activated carbon adsorption could be utilized as one-step refining for bleaching and reducing FFA and HCN. More adsorbent is needed to reach the FFA standard for the shorter adsorption time. Twenty four hours of adsorption without stirring using activated carbon of 10% concentration is enough to reduce HCN and bleach the oil color. The highest yield was 74.62% for 10% activated carbon, but the lowest FFA resulted from 40% activated carbon. Freundlich model is the best fit for equilibrium adsorption.

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NOMENCLATURE

A	:	Temkin constant [L.g^{-1}]
B	:	Temkin constant [J.mol^{-1}]
C_e	:	FFA content in the oil [mg. L^{-1}]
n	:	Freundlich constant
K	:	Linear model constant
k_f	:	Freundlich adsorption capacity [mg.g^{-1}]
K_L	:	Langmuir constant [L.mg^{-1}]
Q_e	:	FFA content in carbon [mg.g^{-1}]
Q_m	:	Maximum FFA adsorbed [mg.g^{-1}]

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