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Purification of Used Cooking Oil with Avocado Seed Powder as Biosorbent

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Abstract

Cooking oil is a basic ingredient in food preparation. Repeated use of cooking oil at high temperatures produces free fatty acids (FFA), which indicate oil degradation. The initial free fatty acid content of the used cooking oil in this study was 0.95%. This study aimed to prepare, activate, and evaluate avocado seed powder as a biosorbent for reducing free fatty acids in used cooking oil and to determine the optimal adsorption conditions with respect to temperature, contact time, and adsorbent mass. The activated avocado seed biosorbent showed a moisture content of 5.40%, ash content of 0.68%, iodine adsorption capacity of 768 mg/g, methylene blue adsorption capacity of 22.57 mg/g, and a surface area of 84.69 m²/g. The optimum adsorption condition was achieved at 90°C, 120 minutes contact time, and 20 g adsorbent mass. Under these conditions, the free fatty acid content decreased from 0.95% to 0.40%, corresponding to a reduction in efficiency of 58.05%. The peroxide value and water content were also reduced significantly. The adsorption capacity of avocado seed biosorbent was 27.96 mg/g for free fatty acids, 3.28 mg/g for peroxide value, and 6.16 mg/g for water content.

Keywords: avocado seed, biosorbent, used cooking oil

1 Introduction

Cooking oil will encounter a complex degradation reaction if it is frequently used at high temperatures (160-180 °C) and in contact with water and air. Oil quality decreases due to this degradation reaction [1]. The decrease in oil quality can be seen from the dark color of the oil, thick, smelly, increased water content, free fatty acids, and peroxide numbers. Poor oil conditions are often used as cooking oil [2].

Used cooking oil can be used as a raw material for biodiesel and soap [3]. However, the biodiesel formation reaction will be inhibited if the oil contains too high levels of free fatty acids [4]. In addition, the color of the soap and biodiesel produced will be affected if the oil color is too dark and cloudy. The method to reduce free fatty acid levels is adsorption on used cooking oil [5]. Several previous studies have clarified the use of cooking oil and biosorbents in agricultural waste, such as snake fruit and papaya seeds [6].

Avocado seeds were chosen as biosorbent because of their relatively high cellulose content of 16.36% [7]. In addition, the 80.1% starch content of avocado seeds, consisting of 43.3% amylose and 36.8% amylopectin, makes avocado seeds effective as biosorbents in used cooking oil [8]. Several studies have been conducted on avocado seeds used as biosorbents, namely to remove crystal violet dyes in liquid waste [9] and to absorb lead (II) ions in a solution [10].

This study's background is to purify used cooking oil with avocado seed powder as a biosorbent under varying conditions, including variations in temperature, contact time, and biosorbent mass, so that optimum conditions are obtained to purify used cooking oil.

2 Method

The materials used in this study were avocado seeds from fruit juice shop waste in the Citeureup area, Bogor, and used cooking oil obtained from household waste. The chemicals used were NaOH 0.1 N pa (Merck), KI pa (Merck), HCl 5% pa (Merck), glacial acetic acid-chloroform solution (3:2) pa (Merck), PP indicator pa (Merck), Ethanol 95% pa (Merck), $Na_2S_2O_3$ 0.1 N pa (Merck), and Starch.

The tools used are 250 mL glass beaker, 250 mL Erlenmeyer flask, 250 mL sharpened Erlenmeyer flask, hot plate, magnetic stirrer, analytical balance, thermometer, 25 mL burette, 100 mesh sieve, oven, blender, burette, desiccator, UV-VIS Spectrophotometer (Shimadzu UV-1800).

2.1 Making of Activated Avocado Seed Powder Biosorbent

Making biosorbents is based on research by Kartika et al. (2017). The avocado seeds are dried in the sun for 5 days and crushed with a blender until they form powder. The avocado seed powder is sieved with a 100-mesh sieve. Then, it is dried in an oven at 115 ºC. The avocado seed powder is stored in a desiccator.

Avocado seed powder was activated by immersion in a 5% HCl solution (1:10, w/v), stirred for 15 min with a magnetic stirrer, and subsequently left to soak at room temperature for 24 h. Then, it was dried in an oven at 115°C, filtered, and rinsed with distilled water until the pH of the filtrate was neutral and cooled in a desiccator.

2.2 Quality Testing of Activated Avocado Seed Powder

The biosorbent was tested for quality before and after activation on avocado seed powder. The tests included measuring water content, ash content, iodine adsorption capacity, and methylene blue adsorption capacity.

2.2.1 Water Content

Approximately 1 g of biosorbent was weighed as the initial sample weight and dried in an oven at 115 °C for 3 hours. The dried sample was cooled in a desiccator and weighed repeatedly until a constant weight was obtained. Moisture content was calculated using **Eq. 1**.

$$\text{Water content (\%)} = \frac{W_i - W_d}{W_i} \times 100 \quad \text{(Equation 1)}$$

where:
W_i = initial sample weight (g)
W_d = dry sample weight (g)

2.2.2 Ash Content

Approximately 1 g of biosorbent was weighed and incinerated at 550 °C for 2 hours. The resulting ash was cooled in a desiccator and weighed until a constant weight was achieved. Ash content was calculated using **Eq. 2**.

$$\text{Ash content (\%)} = \frac{W_a}{W_s} \times 100 \quad \text{(Equation 2)}$$

where:
Wa = weight of ash (g)
Ws = initial sample weight (g)

2.2.3 Iodine Adsorption Capacity

Avocado seed powder was preheated at 115 °C for 1 hour and cooled in a desiccator for 30 minutes. Then, 0.5 g of powder was added to 50 mL of 0.1 N iodine solution, stirred for 15 minutes, and left to stand for 1 hour. A 5 mL aliquot of the clear solution was pipetted and titrated with 0.1 N standardized sodium thiosulfate ($Na_2S_2O_3$) until the yellow color faded. 1% starch solution was added as an indicator, and titration continued until the blue color disappeared. A blank titration was also performed. Iodine adsorption capacity was calculated using **Eq. 3**.

$$Iodine\ adsorption\left(\frac{mg}{g}\right) = \frac{\text{(Vb} - \text{Vp) x fp x Np x 126}}{\text{W sampel}}$$

(Equation 3)

where:
W = weight of activated carbon (g)
Fp = dilution factor
Vp = volume of $Na_2S_2O_3$ used for the sample (mL)
Vb = volume of $Na_2S_2O_3$ used for the blank (mL)
N = normality of $Na_2S_2O_3$

2.2.4 Methylene Blue Adsorption Capacity

Avocado seed powder was preheated at 115 °C for 1 hour and cooled in a desiccator for 30 minutes. Then, 0.5 g of powder was added to 50 mL of 250 ppm methylene blue solution, stirred for 15 minutes, and left to stand for 5 minutes. The suspension was centrifuged to obtain a clear solution, and the absorbance of the supernatant was measured at 665 nm. A standard calibration curve was prepared using methylene blue concentrations from 1 to 6 mg/L. Methylene blue adsorption capacity was calculated using **Eq. 4**.

$$X_m = \frac{(\text{Co} - \text{Ce})/1000}{\text{W (g)}} \times \text{V (L)} \quad \text{(Equation 4)}$$

where:
Xm = amount of methylene blue adsorbed per gram (mg/g)
V = volume of methylene blue solution (L)
Co = initial concentration (mg/L)
Ce = equilibrium concentration (mg/L)
W = sample weight (g)

The adsorbent surface area was estimated using **Eq. 5**.

$$S = \frac{X_m \times N_A \times A}{Mr} \qquad \text{(Equation 5)}$$

where:
S = surface area of biosorbent (m²/g)
N_A = Avogadro's number (6.022×10^{23} mol^{-1})
X_m = methylene blue adsorbed per gram (mg/g)
A = surface area of a methylene blue molecule (1.97197×10^{-18} m²)
Mr = molecular weight of methylene blue (319.86 g/mol)

2.3 Determination of Optimum Adsorption Conditions

2.3.1 Optimum Temperature

In 100 grams of used cooking oil, 5 grams of avocado seed biosorbent were added and then heated at 30, 60, 90, and 120 °C for 30 minutes with stirring at 500 rpm. The free fatty acid (FFA) value was analyzed to determine the highest % reduction efficiency value.

2.3.2 Optimum Contact Time

To 100 grams of used cooking oil, 5 grams of avocado seed biosorbent was added and then heated at various optimum temperatures with contact time variations of 30, 60, 90, 120, and 150 minutes while stirring at a stirring speed of 500 rpm. The free fatty acid (FFA) value was analyzed to determine the highest % reduction efficiency value.

2.3.3 Optimum Biosorbent Mass

100 grams of used cooking oil were added to the biosorbent with variations in the mass of 5, 10, 15, 20, and 25 grams of activated avocado seed biosorbent, then heated at the optimum temperature and contact time while stirring at a stirring speed of 500 rpm. The free fatty acid (FFA) value was analyzed to determine the highest % reduction efficiency value.

2.4 Oil Quality Testing After Purification at Optimum Conditions

Oil quality testing was conducted before and after purification. The tests included water content, free fatty acid, and peroxide value. The reduction efficiency and adsorption capacity obtained were then calculated.

2.4.1 Water Content

The moisture content of the biosorbent was determined using the oven-drying method according to ISO 18134. 1 g of biosorbent was accurately weighed as the initial sample weight**.** The sample was then dried in an oven at 115 °C for 3 h**.** After drying, the biosorbent was cooled in a desiccator to room temperature and weighed. This process was repeated until a constant weight was obtained. The moisture content of the sample was calculated using **Eq. 6**.

$$\text{Water content (\%)} = \frac{W_I - W_d}{W_i} \times 100$$

(Equation 6)

where:
Wi = initial sample weight (g)
Wd = dry sample weight (g)

2.4.2 Free Fatty Acid (FFA)

FFA content was determined using the alkali titration method in accordance with the AOS Ca 5a-40 standard. Oil weighing 10 grams was added to 50 mL ethanol 95% p.a, and then three drops of phenolphthalein indicator were added, and then titrated using 0.1 N Sodium hydroxide. FFA's content was calculated from **Eq. 7** and expressed as a percentage of palmitic acid (factor 25,6).

$$\text{FFA (\%)} = \frac{25{,}6 \times V\ NaOH \times C\ NaOH}{W} \qquad \text{(Equation 7)}$$

where:
C NaOH (N) = concentration of NaOH
V NaOH (mL) = volume of NaOH used by oil sample,
W oil (g) = weight of test oil.

2.4.3 Peroxide Value

The peroxide value (PV) was determined using the iodometric titration method according to AOCS Official Method Cd 8b-90 of the American Oil Chemists' Society. Approximately 5 g of sample was placed in an Erlenmeyer flask and mixed with 50 mL of glacial acetic acid–chloroform solution (3:2**, v/v)**. The mixture was shaken until homogeneous, followed by the addition of 0.5 mL saturated potassium iodide (KI) solution. Subsequently, 30 mL of distilled water was added, and the liberated iodine was titrated with 0.1 N sodium thiosulfate ($Na_2S_2O_3$) until the yellow color nearly disappeared. Then, 0.5 mL starch indicator was added, and titration was continued until the blue color disappeared. A blank determination was conducted under the same conditions.

The peroxide value was expressed as milliequivalents of peroxide per kilogram of sample (meq O_2/kg) and calculated using **Eq. 8**.

$$PV\ (\text{mek } O_2/\text{kg}) = \frac{N \times (Vp - Vb)}{W} \quad \text{(Equation 8)}$$

where:
N = normality of sodium thiosulfate solution (N),
Vp = volume of $Na_2S_2O_3$ used for the sample (mL)
Vb = volume of $Na_2S_2O_3$ used for the blank (mL
W = the sample weight (g).

3 Result and Discussion

3.1 Activated Avocado Seed Quality Testing

Activated avocado seed powder is made by cleaning and washing avocado seeds with water until clean, then drying and grinding them into a 100-mesh powder. Avocado seed powder is activated by adding 5% (v/v) HCl solution. HCl is used as an activator because it is a strong acid that can degrade and contains H^+ groups. These ions can dissolve impurities in the surface pores, such as tar, hemicellulose, and lignin, thereby enlarging the surface area and activating the pores to maximize adsorption. **Table 1** shows the quality characteristics of the avocado seed biosorbent.

Overall, the water content, ash content, and iodine absorption analysis results meet SNI standards. In this study, activation increased the water content of the avocado seed biosorbent. The water content of the avocado seed biosorbent before activation was 1.33%, which increased to 5.40% after activation. This is because the pores formed in the biosorbent increase due to activation, so the biosorbent's hygroscopic properties are higher after activation than before.

The ash content of avocado seed powder before activation was 2.33%, while the ash content obtained was 0.68% after activation. The decrease in ash content after activation proved that the ability of avocado seed powder as a biosorbent increased because minerals and its surface area no longer covered the pores on the surface of the biosorbent.

The iodine absorption before activation was 482.01 mg/g, and after activation, it increased to 768.20 mg/g. When compared to other biosorbents derived from papaya seeds, the iodine absorption of avocado seed powder is higher than that of papaya seed powder, which has an iodine absorption of 482.22 mg/g [11].

However, the absorption parameter for methylene blue still does not meet the SNI standard. This may be due to the activation process, which is still not perfect enough or large enough to open pores with a molecular size like methylene blue, so the adsorption power of methylene blue is still relatively small.

Table 1. Quality Characteristics of Avocado Seed Biosorbent

Parameter	Standar SNI 06-3739-1995	Result (Mean ± SD)	
		Before Activation	After Activation
Water Content (%)	Maks. 15	1,33	5,40 ± 0,13
Ash Content (%)	Maks. 10	2,33	0,68 ± 0,04
Iodine Adsorption (mg/g)	Min. 750	482,01	768,20 ± 6,31
MB Adsorption (mg/g)	Min. 120	19,60	22,80 ± 0,09
Surface Area (m^2/g)	-	72,75	84,69 ± 0,34

Values are expressed as mean± standard deviation (n=3)

3.2 Results of Determination of Optimum Adsorption Conditions

Free fatty acid parameters are testing parameters used to determine the optimum conditions for biosorbent adsorption on used cooking oil. Free fatty acid testing indicates oil quality because the factor that causes oil damage is free fatty acids (FFA). The optimum conditions for free fatty acid adsorption from used cooking oil with the avocado seed biosorbent are determined using three parameters: temperature, contact time, and biosorbent mass.

In this study, the temperature of the adsorption process was varied to 30, 60, 90, and 120 ºC. The higher the temperature, the Free Fatty Acid (FFA) levels after initial adsorption decreased to a temperature of 90 °C, but at a temperature of 120 ºC, the FFA levels increased again. This shows that temperature affects the adsorption of free fatty acids to the activated avocado seed biosorbent. The higher the temperature, the lower the FFA levels. This occurs because the kinetic energy of the molecules for collisions will be greater at high temperatures, so the biosorbent's ability to adsorb free fatty acids is higher. Temperatures that are too high can also cause the FFA value to increase, as shown by the

FFA value, which increases at heating above 90 ºC. This can be caused by re-damage to the oil, leading to the formation of free fatty acids and an increase in FFA levels. **Table 2** shows the optimum conditions for the adsorption of activated avocado seed biosorbent

Table 2. Optimum Conditions for Adsorption of Activated Avocado Seed Biosorbent

Parameter			FFA (%)	%E
Temp (˚C)	Contact Time (minutes)	Mass (gram)		
30	30	5	0,8796	8,32
60	30	5	0,8079	15,79
90	30	5	0,7777	18,94
120	30	5	0,8861	7.64
90	30	5	0,7777	18,94
90	60	5	0,7040	26,62
90	90	5	0,6798	29,14
90	120	5	0,6305	34,28
90	150	5	0,7386	23,02
90	120	5	0,6305	34,28
90	120	10	0,5947	38,01
90	120	15	0,4840	49,55
90	120	20	0,3999	58,32
90	120	25	0,4969	48,21

%E = Percentage of Efficiency

The results obtained in optimizing contact time are at a contact time of 120 minutes. The longer the contact time, the lower the oil's FFA levels. This is because the opportunity between used cooking oil and biosorbent is getting longer, so the FFA levels in used cooking oil continue to decrease until the biosorbent reaches saturation point and experiences desorption, as indicated by the FFA levels increasing again.

The result of the optimum biosorbent mass optimization to purify used cooking oil is 20 grams of activated avocado seed powder biosorbent. This shows that the greater the addition of biosorbent, the lower the FFA content. The saturation point of avocado seed biosorbent to purify oil is achieved with the addition of 25 grams of biosorbent mass, as seen from the increase in the FFA content of used cooking oil. Biosorbent particles in a solution that are too dense result in the obstruction of access for adsorbate molecules to enter the biosorbent surface, resulting in desorption and the FFA value increasing again.

3.3 Analysis of Used Cooking Oil Quality After Optimization Process with Activated Avocado Seed Biosorbent

The quality of cooking oil, in general, can be determined from the water content, free fatty acids, and peroxide value. Then, the quality of used cooking oil before and after optimization was analyzed based on the analysis of water content, free fatty acids, and peroxide value. The higher the water content, free fatty acids, and peroxide value, the worse the quality of cooking oil. The results of the analysis are shown in **Table 3**. After the purification of used cooking oil with activated avocado seed biosorbent, the water content, free fatty acids, and peroxide value showed a significant decrease. Table 3 shows the results of analysis of the adsorption process of avocado seed biosorbent on used cooking oil.

Table 3. Results of Analysis of the Adsorption Process of Avocado Seed Biosorbent on Used Cooking Oil

Parameter	SNI 7709:2019	Before Adsorption	After Adsorption
Water Content (%)	Max 0.1	0,16	0,04
FFA Content (%)	Max 0.3	0,96	0,40
Peroxide Number (mEq O_2/Kg)	Max 10	17,71	9,49

The results shown in **Table 3** indicate a significant improvement in the quality of waste cooking oil after adsorption with activated avocado seed biosorbent. The peroxide value (PV) decreased substantially from 17.71 to 9.49 meq O_2/kg, which meets the SNI 7709:2019 standard (maximum 10 meq O_2/kg). This confirms effective reduction of primary oxidation products and suggests the oil’s stability improved after treatment.

However, the free fatty acid (FFA) content, although reduced from 0.96% to 0.40%, still remains above the SNI maximum limit of 0.3%. This indicates that while the adsorption process reduces FFA, it may not be sufficient alone to fully purify or “refine” the oil according to SNI standards. The residual FFA content suggests that additional processing steps, such as a second adsorption stage or other purification methods, might be required to meet stricter FFA standards.

The hydroxyl groups (-OH) in cellulose of avocado seeds possibly contribute to binding free fatty acids, as supported by the observed FFA reduction and antioxidant activities discussed elsewhere. This mechanism is plausible and well-supported by the literature.

Comparison of the clarity of used cooking oil after adsorption with biosorbent is shown in **Fig. 1**. Differences are seen in the used cooking oil samples before and after optimization. The sample before optimization was brown, cloudy and foamy, while the oil sample after adsorption was orange, clear, and non-foamy.

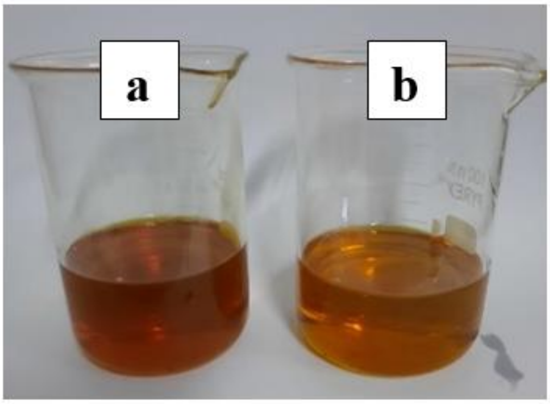

Figure 1. Clarity of Used Cooking Oil Samples (a) Before Optimization and (b) After Optimization

The water content in cooking oil used before optimization was 0.16%, and the water content decreased after the optimization of the conditions to 0.05%. Chemical components in avocado seeds, such as lignin and cellulose, can interact with water molecules in the oil and form adsorption bonds. The water content of used cooking oil after adsorption with activated avocado seed powder under optimum conditions meets the standard limit for palm cooking oil according to SNI 7709:2019, which is a maximum of 0.1%.

The free fatty acid content in used cooking oil after adsorption with activated avocado seed biosorbent before optimization was 0.96%, and the free fatty acid content decreased after optimization to 0.40%. These results indicate that adsorption with an activated avocado seed biosorbent effectively improved the quality of used cooking oil. The hydroxyl group (-OH) in cellulose in avocado seeds is electronegative (basic) and polar, so this group can interact with the carboxylic acid group (-COOH) of free fatty acids, which are electropositive (acidic) and polar, which causes free fatty acid molecules to bind to the biosorbent [12].

The peroxide value in used cooking oil after adsorption of activated avocado seed biosorbent before optimization was 17.71 mEq O_2/Kg, and the peroxide level decreased after optimization to 9.49 mEq O_2/Kg. According to research by Malangngi [13], avocado seeds contain many flavonoids. One group of phenolic compounds commonly found in plant tissue that can act as antioxidants by inhibiting the oxidation process is the chemical compound flavonoids. Because antioxidants are more reactive than oxygen, antioxidants can inhibit the rancidity process. Active antioxidant molecules bind oxygen to prevent the formation of peroxides. So, with the adsorption process with avocado seed powder, the peroxide value in used cooking oil can decrease.

3.4 Adsorption Efficiency and Capacity

Adsorption capacity is the amount of adsorbate that can accumulate on the surface of the adsorbent. After the adsorption parameters are optimized, the maximum adsorption capacity can be achieved [14].

3.4.1 FFA

The adsorption efficiency (%) of free fatty acids (FFA) was calculated using **Eq. 9**.

$$Adsorption\ Efficiency\ (\%) = \frac{C_0 - C_t}{C_0} \times 100 \quad \text{(Equation 9)}$$

where:
C_0 = initial FFA concentration (%)
Ct = FFA concentration after adsorption (%)

The adsorption capacity (q, mg/g) was calculated using **Eq. 10**.

$$q\ (mg/g) = \frac{V(C_0 - C_t)}{m} \quad \text{(Equation 10)}$$

where:
q = adsorption capacity (mg/g)
V = volume of oil sample (L)
C_0 = initial concentration (mg/L)
Ct = final concentration (mg/L)
m = mass of adsorbent (g)

3.4.2 Peroxide Value

The peroxide value (PV) is commonly expressed in milliequivalents of active oxygen per kilogram of oil (meq O_2/kg). For comparison with adsorption capacity calculations, the unit was converted to milligrams of oxygen per gram of oil (mg O_2/g).

The conversion is based on the equivalent weight of oxygen. Since oxygen (O_2) has a molecular weight of 32 g/mol and a valence of 2, its equivalent weight is calculated using **Eq. 11**.

$$Equivalent\ weight\ of\ O_2 = \frac{32}{2} = 16\ g/eq$$

(Equation 11)

Because 1 equivalent equal 1000 milliequivalents (meq), the mass corresponding to 1 meq of O_2 is 1 meq O_2 = 16 mg O_2. Therefore, the general conversion equation becomes **Eq. 12**.

$$mg\ O_2/g = \frac{meq\frac{O_2}{Kg} \times 16}{1000} \qquad \text{(Equation 12)}$$

The factor 1000 is used to convert from kilogram to gram.

Table 4. Efficiency and Adsorption Capacity of Avocado Seed Biosorbent on Used Cooking Oil

Parameter	Efficiency (%)	Capacity (mg/g)
Water	12.31	6.16
Free Fatty Acid	58.05	27.96
Peroxide Number	46.39	3.28

Table 4 shows the efficiency and adsorption capacity of avocado seed biosorbent on used cooking oil. The reduction efficiency and adsorption capacity for the purification of used cooking oil with the activated avocado seed biosorbent were greatest for free fatty acids. The results of the adsorption efficiency in this study were higher than those of the study of oil purification with avocado seeds conducted by Fitriani [15]. In this study, the adsorption efficiency of free fatty acids was 58.05%, and the peroxide number was 46.36%. In comparison, the study of oil purification with avocado seed powder showed that the adsorption efficiency of fatty acids was 43.48%, and the peroxide was 16.67%. It can be concluded that by optimizing the adsorption conditions, the adsorption efficiency of activated avocado seed biosorbent can be increased to purify used cooking oil.

4 Conclusion

An activated avocado seed biosorbent with a water content of 5.40%, ash content of 0.68%, iodine absorption capacity of 768.20 mg/g, methylene blue absorption capacity of 22.80 mg/g, and surface area of 84.69 m^2/g was successfully made.

The optimum conditions for the adsorption of free fatty acids on activated avocado seed biosorbent were obtained at 90°C, a contact time of 120 minutes, and a biosorbent mass of 20 grams in 100 grams of used cooking oil.

The efficiency and adsorption capacity of avocado seed biosorbent on the water content in used cooking oil was 12.31% and 6.16 mg/g, free fatty acid content was 58.05% and 27.96 mg/g, and peroxide content was 46.36% and 3.28 mg/g.

The relatively high adsorbent dose (20 wt%) and heating requirement (90°C for 120 minutes) suggest considerable material and energy inputs, which may limit direct large-scale application. Therefore, future studies should focus on process optimization to reduce adsorbent loading, evaluate regeneration and reuse potential, assess oil retention losses, and conduct cost-benefit and scalability analyses to support practical implementation.

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