Metana: Media Komunikasi Rekayasa Proses dan Teknologi Tepat Guna ISSN: 1858-2907
Juni 2023 Vol. 19(1):44-52 EISSN: 2549-9130

Pretreatment of Used Cooking Oil Using Avocado Seed Adsorbent for Biodiesel Production Preparation

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Abstract

Used cooking oil (UCO) can be used as raw material for biodiesel production, but its free fatty acid (FFA) content is still quite high, so it is necessary to do pretreatment in the form of an adsorption process to reduce FFA levels. This study aims to determine the optimum conditions for the adsorption process and determine FFA levels of UCO before and after pretreatment. The adsorbent used is avocado seed activated charcoal, because it has a surface area of 19.62 m^2/g. The larger the surface area of the adsorbent material, the greater the adsorption capacity of the adsorbent. Optimization of the UCO adsorption process includes variations in adsorbent mass (6, 8, 10 g), adsorbent particle size (60, 100, 140 mesh), contact time (2, 6, 10 hours), and temperature (60, 80, 100^{o}C). The results showed that the optimum conditions for adsorption of UCO were obtained at mass (10 g), particle size (100 mesh), contact time (6 h), and temperature (80^{o}C). This condition can reduce the FFA content of UCO by 71.64% (w/w), from 5.29% (w/w) to 1.50% (w/w). The FFA content of UCO [1.50% (w/w)] produced after pretreatment was qualified as a raw material for the preparation of biodiesel production through the transesterification stage.

Keywords: *adsorption, avocado seed activated charcoal, free fatty acids (FFA), biodiesel, used cooking oil (UCO)*

INTRODUCTION

Used cooking oil (UCO) can be used as raw material for making biodiesel because it contains triglycerides. Triglycerides are triesters of glycerol and fatty acids. UCO contains 84% (w/w) oleic acid as free fatty acid (FFA) (Dali *et al*., 2021). This FFA level is still quite high because the minimum requirement for FFA levels in UCO is lower than 2.00% (w/w) (Hashatan *et al*., 2012). If UCO with FFA content > 2% (w/w) is directly transesterified with a base catalyst, triolein will be hydrolyzed by base (saponification) to form soap and glycerol. The formation of soap in large enough quantities can inhibit the separation of glycerol from biodiesel due to the formation of an emulsion during washing (Dali and Dali, 2019).

FFA levels of UCO can be reduced in two ways. First, FFA of UCO was adsorbed using activated charcoal as adsorbent. One of the activated charcoal that can be used as an adsorbent is avocado seed. According to Taufiq (2007) activated charcoal from avocado seeds can reduce FFA levels of UCO by 74.6% (w/w), from

*)Correponding author
DOI : 10.14710/metana.v19i1.51797
Diterima: 15-01-2023
Disetujui: 19-05-2023

0.50 - 0.127% (w/w). Second, FFA of UCO was esterified with methanol using a 98% (v/v) H_2SO_4 catalyst. The esterification method can reduce the FFA levels of UCO by 78.34% (w/w) from 1.57 - 0.34% (w/w) (Dali and Dali, 2019).

One way that can be used to reduce FFA levels of UCO is by adsorption. Adsorption is a process of adsorption of adsorbate (substance that is adsorbed) on the surface of the adsorbent (substance that is adsorbed). One of the factors that affect the adsorption process is the surface area of the adsorbent, because the adsorption process involves the interaction between the adsorbent surface and the adsorbate molecules. The larger the surface area of the adsorbent material, the greater the adsorption capacity of the adsorbent.

One of the activated charcoal that can be used as an adsorbent is avocado seed. The use of avocado seeds as an adsorbent has been widely carried out. The results of the research of Munawarah *et al*. (2022) and Putra *et al*. (2022) showed that avocado seed activated charcoal has the potential as an adsorbent of Cd^{2+} and Pb^{2+} ions. The adsorption potential of avocado seed activated charcoal on Cd^{2+} and Pb^{2+} ions is optimum at concentrations of 0.38 ppm and 4.83 ppm, respectively, with adsorption efficiency of 97.11% and 97.92%, respectively (Munawarah *et al*., 2022) and 99.34% for Cd^{2+} ions and 98.86% for Pb^{2+} ions (Rahayu, 2014). Characterization of avocado seed activated charcoal showed that activation using 5% H_2SO_4 activator had good characteristics with a water content of 3.68%, ash content of 0.84%, iodine adsorption 385.22 mg/g, and a surface area of 19.62 m^2/g. The results of the quality test of avocado seed activated charcoal that was carried out by Rahayu (2015) showed that activation using 5% HCl activator had characteristics that met SNI standard No. 06-3730-1995, namely water content of 4.55%, ash content of 3.05%, volatile matter content of 13.90%, carbon content of 78.51%, and iodine absorption of 888.07 mg/g. The combination of electrocoagulation and adsorption of activated carbon from avocado seeds can reduce the concentration of Pb^{2+} ions in wastewater by 96.01% (Setiawan *et al*., 2020) (Kartika *et al*., 2017). The results of the test of peroxide number, acid number, FFA content, and water content of black UCO after purification using activated carbon of avocado seeds were 7.89 O_2/100 g, 1.46 mg KOH/g, 0.64 % (w/w), and 0.61% (w/w), while brown UCO was 5.15 O_2/100 g, 0.94 mg KOH/g, 0.41% (w/w), and 0.79% (w/w) (Suroso, 2013). The results of this study indicate that avocado seed activated charcoal has the potential to be used as an adsorbent. Therefore, the adsorbent used in the pretreatment of this study was activated charcoal from avocado seeds.

This study aims to determine the optimum conditions for the adsorption process and determine FFA levels of UCO before and after pretreatment. Optimization of the UCO adsorption process includes variations in adsorbent mass (6, 8, 10 g), adsorbent particle size (60, 100, 140 mesh), contact time (2, 6, 10 hours), and temperature (60, 80, 100°C).

The results showed that the optimum conditions for adsorption of UCO were obtained at mass (10 g), particle size (100 mesh), contact time (6 h), and temperature (80°C). This condition can reduce the FFA content of UCO by 71.64% (w/w), from 5.29% (w/w) to 1.50% (w/w). The FFA content of UCO [1.50% (w/w)] produced after pretreatment was qualified as a raw material for the preparation of biodiesel production through the transesterification stage.

METHODOLOGY

The tools used are blender, analytical balance, oven, furnace, magnetic stirer, spatula, filler, clamp, centrifuge tube, centrifugal, (60,100, 140 mesh) sieve, stative and clamp, hot plate, three-neck round bottom flask, desiccator, dark bottle, stirring rods, measuring cups, beaker, erlenmeyer, funnel, separating funnel, mortar and pestle porcelain, and Buck M500 FTIR spectrometer (Scientific).

The materials used are UCO, avocado seeds, methanol p.a., potassium hydroxide p.a., phenolphtalein (PP) indicator, chloroform p.a., concentrated hydrochloric acid p.a., concentrated sulfuric acid p.a., concentrated nitric acid p.a., amylum indicator, potassium iodide p.a., $Na_2S_2O_3.5H_2O$ p.a., hexane p.a., paper filter Whatman No.42, universal indicator, aluminum foil, tissue, and aquabidest (Onelab Waterone).

Production of Avocado Seed Activated Charcoal

Avocado seeds are separated from the skin. Avocado seeds are cut into small pieces and then washed with water. Avocado seeds are dried in the sun for 3 days. Dried avocado seeds were carbonated at 600ºC for 2 hours. Avocado seed charcoal was cooled in a desiccator for 6 hours. Avocado seed charcoal is ground in porcelain. Avocado seed charcoal powder was sieved into 60, 100, and 140 mesh sizes. These avocado seed charcoal powders with different particle sizes were each dissolved in 5% (v/v) HCl solution. The mixture was stirred with a magnetic stirrer for 24 hours. Avocado seed activated charcoal powder was filtered and washed with aquabidest until neutral. Avocado seed activated charcoal powder was dried in an oven at 110ºC for 2 hours (Maulinda *et al*., 2015), (Rizqi *et al*. 2014), (Nasir *et al*., 2014), (Fitriani and Nurulhuda, 2018), (Dahlan, 2013). Avocado seed activated charcoal powder was characterized by FTIR spectrometer.

Pretreatment of UCO

UCO (150 mL) and avocado seed activated charcoal powder (6, 8, 10 g) with sizes (60, 100, 140 mesh) were put into 3 beakers. The mixture was stirred with a magnetic stirrer for (2, 6, 10 hours) at a temperature of (60, 80, 100ºC). The mixture was allowed to stand for 12 hours. The mixture was filtered through Whatman No. filter paper. 42. Pure oil is collected in a beaker and the residue of activated charcoal powder from avocado seeds is removed (Marlinda, 2012).

Determination of FFA Levels (ASTM D 664)

The oil sample (3 g) was put into the erlenmeyer flask. Next to the erlenmeyer flask was added hexane (15 mL), 95% (v/v) alcohol (20 mL), and PP indicator (3 drops). The mixture is titrated with 0.1 N KOH until the mixture changes color to pink. Then the oil FFA level is calculated using Equation (1) (Bouta *et al*., 2020).

$$\% \text{ FFA} = \frac{\text{V KOH (mL)}\left(\frac{\text{L}}{10^3\ \text{mL}}\right) \text{x N KOH}\left(\frac{\text{mole equivalent}}{\text{L solution}}\right) \text{x Mr FFA}\left(\frac{\text{g}}{\text{mole}}\right)}{\text{Sample Mass (g) x 1000}} \text{ x } 100\%$$

If the oil FFA level is < 2% (w/w), the synthesis of biodiesel can proceed to the transesterification stage. Conversely, if the oil FFA level is > 2% (w/w), the synthesis of biodiesel starts from the esterification stage.

RESULTS AND DISCUSSION

Characterization of Avocado Seed Activated Charcoal

Table 1 shows the results of the interpretation of the FTIR spectrum of avocado seed activated charcoal. Table 1 shows that avocado seed activated charcoal contains three main functional groups, namely OH, CO, and CN groups. The OH group appears as a strong and wide absorption band at 3414.00 cm^{-1}, the CO group appears as a strong and sharp absorption band at 1695.43 cm^{-1}, and the CN group appears as a strong and sharp absorption band at 2360.04 cm^{-1}.

Pretreatment of UCO

Discoloration of UCO before and after purification using Moringa oleifera activated charcoal is shown in Figure 2. Figure 2 shows that the color of the UCO before and after refining has changed from brownish red to brownish yellow. This indicates that the particles causing the turbidity and reddish brown color of the UCO have been absorbed by the activated charcoal adsorbent of avocado seeds. This adsorption process occurs because the activated charcoal adsorbent of avocado seeds has a large surface area. The larger the surface area of adsorbent, the greater the capacity of the adsorbent to adsorb an adsorbate.

According to Langmuir-Hinshelwood in Laidler and Meiser (1995), when a solid surface catalyzes a process that involves a reaction between three adsorbate molecules at the adsorbent surface site, one of the possible mechanisms

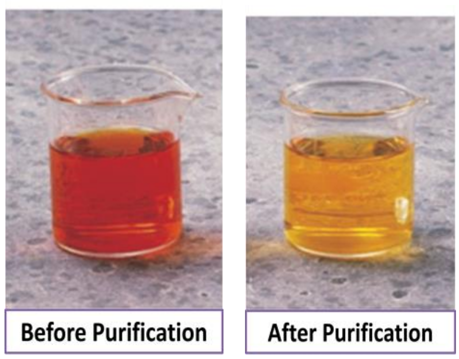

Figure 2. Changing color of UCO before and after purification using avocado seed activated charcoal

is that the three adsorbate molecules A, B, and C become adsorbed. at the adjacent adsorbent surface site (S). Furthermore, the reaction occurs through an activated complex to produce reaction products X, Y, and Z (Figure 3) (Dali and Dali, 2019).

Determination of FFA Levels

The results of determining FFA levels of UCO before and after pretreatment using avocado seed adsorbent are shown in Table 2. Table 2 shows that the avocado seed adsorbent can reduce the FFA content of UCO by 71.64% (w/w). Table 2 also shows that the FFA content of UCO decreased to 1.50% after pretreatment. If the FFA content of UCO is < 2.00% (w/w), then the UCO can be transesterified directly into biodiesel without having to go through the esterification stage (Hashatan *et al.*, 2012), (Dali and Dali, 2019), (Anisah *et al.*, 2018). This shows that the avocado seed adsorbent can be used as a pretreatment of UCO to produce biodiesel through the transesterification stage.

Table 1. The results of the FTIR spectrum interpretation of avocado seed activated charcoal

No.	Frequency (cm^{-1}) and Intensities	Frequency Ranges (cm^{-1})*	Group or Class	Type of Vibrations
			Alcohols, R-OH	
1	3414.00 (s)	3600 - 3200 (s)	Bonded -OH	OH stretch
2	1438.90 (s)	1440-1260 (s)		C-OH deformation
3	1042.20 (s)	1150 - 1040 (s)	Primary $-CH_2OH$	C-OH stretch
4	1101.60 (m)	1150 - 1050 (m)		C-O antisym stretch
5	809.00 (m) 800.46 (m)	970 - 800 (s)		C-C-O sym stretch
6	1375.25 (m)	1450 - 1350 (m)		C-OH in-plane bend
7	600.30 (m)	700 - 600 (m)		C-OH out-of-plane deformation
			Carboxylic acids, $R-CO_2H$	
8	3630.30 (m)	3650 - 3590 (s)	Free -OH	OH stretch
9	1695.43 (s)	1710 - 1680 (vs)		C=O stretch of dimer
10	1704.16 (m)	1705 - 1680 (m)		Aryl
11	1600.02 (s)	1610 - 1550 (s)		Antisymmetrical stretch
			Aliphatic, RH	
12	2922.10 (s)	2940 - 2920 (s)	Methylene, $-CH_2-$	C-H stretches in alkanes
13	2868.61 (s)	2970 - 2850 (s)	Methyl, $-CH_3$	C-H stretch in $C-CH_3$
14	1901.01 (m)	2000 - 1660 (w)	Aromatic, ArH	Overtone and combination bands
15	762.24 (m)	900 - 650 (s)		Out-of-plane CH deformations
16	2360.04 (s)	2400 - 2000 (s)	Nitriles, -C-C≡N	C≡N stretch
17	368.40 (s)	380 - 280 (s-vs)		C-C≡N bend
18	1234.44 (m)	1280 - 1220 (s)	Ethers, ROR’	C-O-C stretch in alkyl aryl ethers
19	570.03 (m)	675 - 570 (s)		O-C-O bend

◦Notes: vbr = very broad; vs = very strong; v = variable; s = strong; m = medium; w = weak.
*Sources: (Kemp, 1991), (Lambert *et al.*, 2011), (Sastrohamidjojo, 1992).

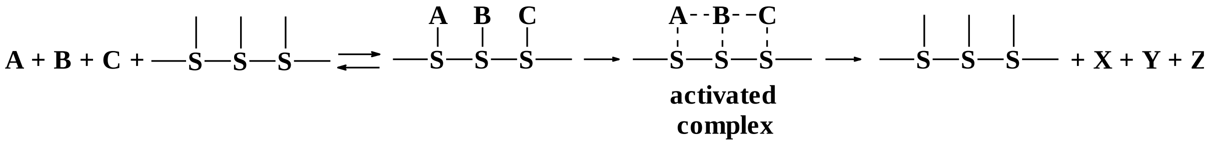

Figure 3. Mechanism of absorption of adsorbate molecules on the surface site of the adsorbent through the formation of activated complexes to produce products (Dali dan Dali, 2019)

Table 2. The results of determining FFA levels of UCO before and after pretreatment using avocado seed adsorbent

Treatment	Volume of Titrant KOH (mL)				Sample Mass (g)	FFA Level (% w/w)
	1	2	3	Average		
Before pretreatment	6.1	6.3	6.2	6.2	3	5,29
After pretreatment	1.8	1.7	1.9	1.8	3	1,50

The results of determining FFA levels of UCO after pretreatment using avocado seed adsorbent under various adsorption conditions are shown in Table 3. Table 3 shows the results of determining FFA levels of UCO after pretreatment using avocado seed adsorbent under various adsorption conditions. Three observations can be made from Table 3 regarding the FFA content of UCO after pretreatment using avocado seed adsorbent. First, the heavier the adsorbent mass, the smaller the particle size of the adsorbent, the longer the contact time of the avocado seed adsorbent, and the higher the temperature the lower the FFA content of the UCO. Second, the interaction between each mass (6, 8, 10 g) and particle size (100 mesh) with contact time (2, 6, 10 h) of avocado seed adsorbent and temperature (80°C) resulted in low FFA levels of UCO. Third, the lowest FFA content of UCO (1.50%) was obtained from the interaction between mass (10 g) and particle size (100 mesh) with contact time (6 h) and temperature (80°C).

Table 3 shows that the lighter the adsorbent mass and the larger the particle size of the avocado seed adsorbent, the higher the FFA content of UCO (curves 1, 4, 7). On the other hand, the heavier the adsorbent mass and the smaller the particle size of the avocado seed adsorbent, the lower the FFA content of UCO (curves 2, 5, 8). This is because the heavier and smaller the particle size of the adsorbent, the larger the surface area of the adsorbent, so that the more FFA of UCO can be absorbed by the avocado seed adsorbent. According to Nufida *et al.* (2014), and Erawati and Fernando (2018), the heavier and smaller the particle size of the adsorbent in the purification of UCO, the more the adsorption capacity of the adsorbent will increase.

This is because the smaller the particle size, the larger the surface area of the adsorbent. The large surface area of the adsorbent can absorb impurities and other compounds, so that the obtained oil becomes clearer. According to Syauqiah *et al.* (2011), one of the factors that influence the adsorption process is the surface area, where the larger the surface area of the adsorbent the more substances are adsorbed. The results of the research of Barau *et al.* (2015), stated that the adsorption results on the clarity parameter, a larger particle size obtained clearer UCO compared to a smaller particle size.

Table 3 shows that the higher the temperature and the longer the contact time of the avocado seed adsorbent, the lower the FFA content of the UCO. This is because the avocado seed adsorbent can interact with UCO optimally in absorbing FFA. These results are in accordance with the research results of Miskah *et al.* (2018), Wenti (2009), and Lestari (2010) which stated that the higher the temperature and the longer the

Table 3. The results of determining FFA levels of UCO after pretreatment using avocado seed adsorbent at various adsorption conditions

Adsorbent Mass (g)	Adsorbent Particle Size (mesh)	Avocado Seed Adsorbent Contact Time (hours)								
		2			6			10		
		Temperature (°C)			Temperature (°C)			Temperature (°C)		
		60	80	100	60	80	100	60	80	100
6	60	4,53	4,15	4,39	3,99	3,67	3,85	3,95	3,28	3,45
	100	4,51	4,13	4,37	3,95	3,44	3,72	3,60	3,17	3,29
	140	4,85	4,49	4,77	4,11	3,78	3,98	3,98	3,30	3,74
8	60	3,99	3,17	3,40	3,86	3,40	3,62	3,66	3,09	3,33
	100	3,62	3,03	3,32	2,97	2,21	2,40	2,80	2,33	2,46
	140	4,35	4,09	4,28	3,87	3,52	3,68	3,85	3,26	3,46
10	60	2,85	2,38	2,57	2,51	1,91	2,43	2,80	2,34	2,66
	100	2,55	2,28	2,39	2,17	1,50	2,02	2,74	2,28	2,41
	140	3,59	3,09	3,29	2,73	2,09	2,25	2,95	2,55	2,74

contact of the adsorbent with UCO, the lower the FFA levels obtained because the more amount of FFA bound to the adsorbent surface.

Table 3 shows that the interaction between each mass (6, 8, 10 g) and particle size (100 mesh) of avocado seed adsorbent resulted in low FFA levels of UCO (curves 3, 6, 9). This indicates that the adsorbent absorption of the FFA in UCO increases with the increase in the mass of the avocado seed adsorbent. This is because the heavier the mass of the adsorbent, the greater the number of particles that can absorb FFA so that the FFA content of UCO is lower. On the other hand, the larger the particle size of the adsorbent, the higher the FFA content of the UCO obtained. This indicates that the adsorption capacity of the adsorbent on FFA decreases with the increase in the particle size of the avocado seed adsorbent. This is because the larger the particle size, the narrower the surface of the adsorbent, so that the absorption capacity of the adsorbent decreases. However, the maximum adsorbent absorption occurs at a particle size of 100 mesh. This shows that although the surface area of the adsorbent is large at the particle size of 60 mesh, the adsorbate density is high and the number of adsorbent particles is small, causing the adsorbent absorption to decrease. On the other hand, at the particle size of 140 mesh, although the number of adsorbent particles is large, because the surface area of the adsorbent is small, it causes the absorption of the adsorbent to decrease. According to Wenti (2009), the heavier and wider the area where the adsorption process occurs, the greater the amount of FFA bound by the adsorbent surface, so that the FFA content of UCO decreases.

Table 3 shows that the interaction between each contact time (2, 6, 10 h) of avocado seed adsorbent at a temperature (80°C) resulted in low FFA levels of UCO. This indicates that the adsorbent absorption of the FFA in UCO increases with the increase in contact time of the avocado seed adsorbent. This is because the longer the adsorbent interacts with the UCO, the more adsorbate fills the pores of the avocado seed adsorbent, so that the FFA content of the UCO decreases. The results of this study are in line with the results of research by Zunifer et al. (2020) which states that the longer the contact time with activated charcoal, the more adsorbate fills the pores of the activated carbon. However, at 80°C the maximum adsorption capacity of the FFA in UCO occurred at each contact time (2, 6, 10 h) of the avocado seed adsorbent. This indicates that at a temperature of 80°C the adsorbent absorption of FFA in UCO has reached the saturation point, which is a condition where all the avocado seed adsorbent pores are fully filled with FFA in UCO, so that the adsorption system has reached an equilibrium condition. At low temperature (60°C), the adsorbent's adsorption capacity to the FFA in UCO is low. This is because at low temperatures the ratio of the initial FFA concentration of UCO to the pores of the adsorbent is very large, so that the adsorption system is still in an unsaturated condition, which is a condition where the concentration

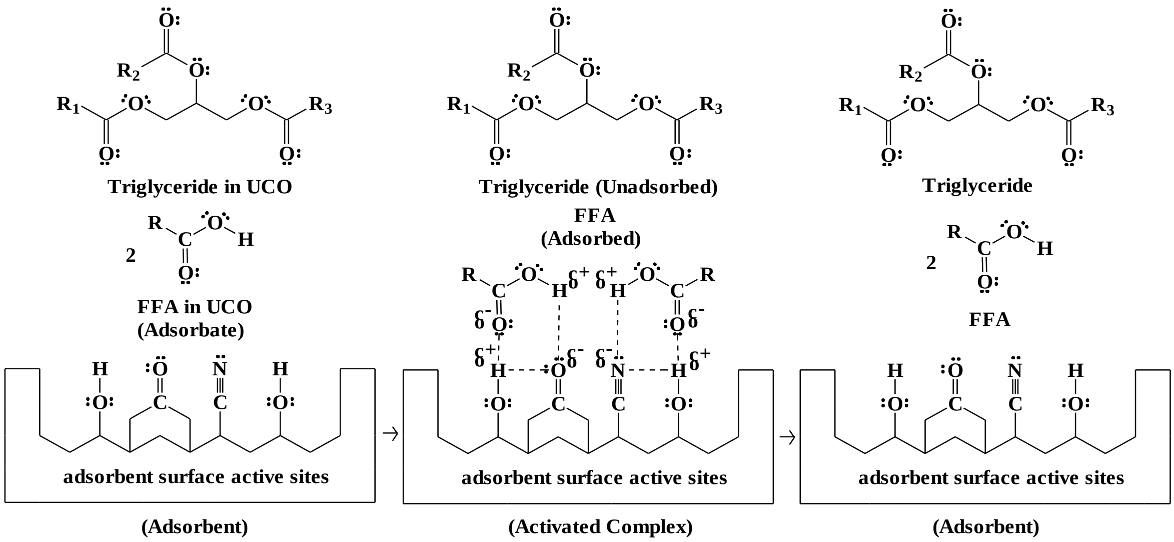

Figure 3. The mechanism of interaction between the surface active sites of avocado seed adsorbent and adsorbate molecules (FFA) in UCO (Laidler and Meiser, 1995)

of adsorbate which has the capacity to be adsorbed at a certain temperature is less than the concentration of adsorbent (Sandi and Astuti, 2014). At high temperature (100°C), the adsorbent's absorption of the FFA in UCO decreased. This is because at high temperatures the ratio of the initial FFA concentration of UCO to the pores of the adsorbent is very small, so that the adsorption system has reached a state of supersaturation, which is a condition where the concentration of adsorbate which has the capacity to be adsorbed at a certain temperature is higher than the concentration of adsorbent (Fadilah and Utami, 2021).

Table 3 shows that the lowest FFA content of UCO (1.50%) was obtained from the interaction between mass (10 g) and particle size (100 mesh) with contact time (6 h) and temperature (80°C). This indicates that the optimum conditions for adsorption of UCO were obtained at mass (10 g), particle size (100 mesh), contact time (6 h), and temperature (80°C). This condition can reduce the FFA content of UCO by 71.64% (w/w), from 5.29% (w/w) to 1.50% (w/w) (Table 2). The decrease in FFA levels in UCO occurred because FFA had been absorbed by the avocado seed adsorbent. Avocado seed adsorbent can absorb FFA because it has three functional groups, namely OH, CO, and CN groups (Table 1). These three groups function as active sites on the surface of the avocado seed adsorbent.

The mechanism of interaction between the active sites on the surface of the adsorbent and the adsorbate can be described as follows (Figure 3). According to Langmuir-Rideal in Laidler and Meiser (1995), the mechanism for the adsorption process on the bimolecular surface is the reaction that occurs between the unadsorbed molecule (triglycerides) and the adsorbed molecule (FFA) (Figure 4). In the initial stage, the triglyceride molecules are not adsorbed and the FFA molecules become adsorbed on the adjacent adsorbent surface active sites. Subsequently, the reaction occurs via an activated complex to give the reaction products of triglycerides and FFA.

CONCLUSION

The optimum conditions for adsorption of UCO were found in mass (10 g), particle size (100 mesh), contact time (6 h), and temperature (80°C). This condition can reduce the FFA content of UCO by 71.64% (w/w), from 5.29% (w/w) to 1.50% (w/w). The FFA content of UCO [1.50% (w/w)] produced after pretreatment was qualified as a raw material for the preparation of biodiesel production through the transesterification stage. The optimum adsorption conditions obtained in this study can be used for the preparation of biodiesel production from UCO through the transesterification stage.

ACKNOWLEDGEMENTS

Thank you to the Head of the UHO FMIPA Chemical Laboratory for permission to use the laboratory facilities and the Head of the UGM FMIPA Chemical Instrument Laboratory Yogyakarta for your assistance in analyzing our sample with FTIR.

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