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The Refining Capability of Palm Shell Activated Carbon for Waste Cooking Oil

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ABSTRACT

The high level of consumption of cooking oil will have an impact on increasing the waste of cooking oil produced. Using the waste of cooking oil needed purification, including through the adsorption process using activated carbon. This research aimed to study the ability of palm kernel shell activated carbon (PKSAC-AC260) compared to standard activated carbon (DAC or Decolorized Activated Charcoal by Sigma-Aldrich) in refining waste cooking oil at various concentrations of activated carbon. The results showed that the type of activated carbon influenced color, free fatty acid content, peroxide value, and saponification value of purified waste cooking oil, but had no effect on water content, saponification value and iodine value. Activated carbon between PKSAC-AC260 and DAC with a concentration of 2.5% was able to improve the quality of the best waste cooking oil. Purification of used cooking oil using PKSAC-AC260 and DAC was able to increase the color brightness of waste cooking oil (ΔE) by 5.44 and 4.53, reduce the free fatty acid content of waste cooking oil from 1.47% to 0.79% and 0.61%, reduce the peroxide value of waste cooking oil from 163.47 meq/kg to 116.40 meq/kg and 98.82 meq/kg, and increase the saponification value of waste cooking oil from 155.22 mg/g to 180.48 mg/g and 184.48 mg/g, respectively. The ability of PKSAC-AC260 to purify waste cooking oil is lower than that of DAC. The quality of processed oil from waste cooking oil does not meet the quality standards of cooking oil based on SNI 3741: 2013.

Keywords: Palm kernel shells, activated carbon, refining, waste cooking oil

INTRODUCTION

Based on data from the Edible Oil Market Outlook 2022-2026, Indonesia is the world's 5th largest consumer of vegetable oil after India, China, the United States and Brazil (Reportlinker, 2023). The high level of consumption of cooking oil will have an impact on increasing the waste of cooking oil produced.

Using cooking oil repeatedly at high temperatures (170^{o}C – 200^{o}C) causes a decrease in cooking oil quality. Oxidation of cooking oil will change the structure of unsaturated fatty acids (Cis) into (Trans) along with the formation of peroxide and hydroperoxide compounds which are free radicals. The characteristics of waste cooking oil can be seen from the changes in appearance and taste (Ketaren, 2008).

Heating cooking oil can cause changes in its physical and chemical structure. The change in chemical structure is the oxidation

of unsaturated fatty acids to produce peroxide groups which are free radicals and cyclic monomers. Fatty acids will be released from triglycerides so that they are oxidized to aldehydes, ketones, and alcohols resulting in the formation of a rancid flavor and a brown color (Megawati *et al*., 2019). Heating cooking oil for a long time can cause an increase in the temperature of the cooking oil. By heating the oil for 30-40 minutes, the temperature will reach over 190^{o}C and the oil will start to smoke. This results in the process of oxidation, hydrolysis, and polymerization of unsaturated fatty acids, forming form ketone compounds, aldehydes, and polymers (Manurung et al., 2015). Polymers, peroxides, aldehydes, ketones, amines, or diamines that are formed due to the oxidation of heating cooking oil repeatedly have the potential to accumulate in the human body and cause diseases such as cancer, high blood pressure, atherosclerosis, and others (Kamilah *et al*., 2015).

The health effects of repeated heating of cooking oil can cause cancer. Peroxide numbers of palm oil and soybean oil were measured after 5 times of heating, which increased significantly compared to one-time heating or with fresh oil. Fresh soybean oil with five times heating had a higher peroxide value than palm oil with the same treatment. Plasma lipid peroxidation in animals (ovariectomized male and female rats) showed significantly higher thiobarbituric acid reactive substances (TBARS) values in male rats treated orally containing 15% palm oil and soybean oil heated once and five times for four months compared to controls. The TBARS values of the mice fed the oil were five times higher than that of the one-time-heating oil group. Palm oil treatment had a much lower TBARS value than soybean oil (Deshmukh, 2019).

Purification or regeneration of waste cooking oil aims to improve the quality of waste cooking oil. The purification method of waste cooking oil can be done in several ways, including through the adsorption process using activated carbon. Adsorption is a method of purifying waste cooking oil which is considered as an uncomplicated process (Oko *et al*., 2020).

Activated carbon is a carbonaceous material that is chemically or physically activated so that the pores of the carbonaceous material become more open with a surface area ranging from 300 - 2000 m^2/g. An increase in the surface area of activated carbon will cause an increase in the adsorption of activated carbon to gas or liquid (Sudrajat & Soleh, 1994). Activated carbon is a carbon material with an amorphous structure and a large internal surface area with a high level of porosity. Activated carbon contains micropores, mesopores, and macropores in its structure. This structure has an important role in determining the performance of activated carbon as an adsorbent (Lubis *et al*., 2020). Activated carbon adsorption reaches 25-100%, so it is often used by industry.

Palm oil mill biomass waste sources of lignin, hemicellulose, and cellulose-containing carbon elements can be used as precursors of activated carbon, one of which is palm shells. The palm kernel shell contains 31.33% cellulose, 17.94% hemicellulose, and 48.83% lignin (Yanti *et al*., 2017).

The palm kernel shell of the Tenera variety contains 44.74% carbon, 1.12% ash, 0.07% total N, and 5.42% moisture content. Palm shells can be used as activated carbon because of their high carbon content (Ulfah *et al*., 2016). Palm kernel shell activated carbon (PKSAC) which is made through a chemical activation process using a 65% phosphoric acid solution by immersing the palm kernel shell particles in a solution of phosphoric acid (1 g/2 mL) for 60 minutes, has a surface area of 1,395.68 m^2/g, mesopore volume of 0.16 cc/g, with a carbon content of 67.55 ± 0.55% (Ulfah *et al*., 2019). Decolorized activated

carbon (DAC) as standard activated carbon is activated carbon with fine particles that are often used to decolorize colored solutions. According to Ulfah *et al*. (2016), DAC-activated carbon has a surface area of 1068.391 m^2/g.

The process of refining waste cooking oil using palm shell activated carbon compared to standard activated carbon at different concentrations will be studied in this research. This study aims to determine the effect of the type and concentration of activated carbon on the characteristics of waste cooking oil, and to determine the type and concentration of activated carbon capable of producing the best quality oil. The method of refining waste cooking oil to produce oil-based non-food product raw materials is expected to be obtained from this research.

MATERIALS AND METHODS

Tools and Materials

The materials used in this research include palm kernel shell activated carbon (PKSAC-AC260), standard activated carbon namely decolorized activated charcoal or DAC (Sigma-Aldrich), waste cooking oil, control cooking oil (Bimoli Special), alcohol (Merck), chloroform (Merck), CH_3COOH (Merck), ultra-pure distilled water H_2O, KOH (Merck), KI (Merck), amylum (Merck), NaOH (Merck), $Na_2S_2O_3$ (Merck), HCl (Merck), I_2 (Merck), Br (Merck). The tools used in this study include a portable colorimeter (NH310 3nh), vacuum pump (Roker 300), hotplate stirrer (Cimarec SP88857105), oven (Memmert u40), analytical balance (Ohaus CP 214), and other glassware used in this research.

Methods

This study was designed using a Complete Block Design with 2 factors. The first factor is the type of activated carbon with 2 types, namely: A1 = palm shell activated carbon (PKSAC-AC260) and A2 = decolorized activated charcoal (DAC) as standard. The second factor is the concentration of activated charcoal, which consists of: B1 = 1.5%, B2 = 2% and B3 = 2.5%. This study was repeated 2 times which was expressed as a block. The data obtained were analyzed for variance, if there is an influential treatment then it is continued with Duncan's multiple range test with a significance level of 0.05 (Gomez & Gomez, 1984).

Purification of waste cooking oil using PKSAC-AC260 and DAC with concentrations of 1.5%, 2% and 2.5%. Purification was carried out by preparing 100 mL of waste cooking oil into a 250 mL Erlenmeyer for each treatment. Furthermore, activated carbon was added as much as 1.5% (1.5 g), 2% (2 g), 2.5% (2.5 g), respectively. The Erlenmeyer containing waste cooking oil and activated carbon was coated with aluminium foil, then heated and stirred using a hot plate magnetic stirrer for 60 minutes at 70^oC. Furthermore, the filtrate is separated from the residue using a vacuum filter. The filtrate that has been obtained is then filled into bottles that have been coated with aluminium foil for further analysis. The waste cooking oil before and after purification was analyzed for the total color difference (Portable Colorimeter NH310 3nh), the free fatty acid content of the AOCS Ca 5a-40-97 method (AOSC, 2004), the moisture content and volatile compounds using the AOCS Ca 2c-25-97 method. The peroxide value for the AOCS method Cd 8-53-03 (AOCS, 2004), the saponification number for the AOCS method Cd 3-25 (AOCS, 2017), the iodine number for the AOCS method Cd 1d-92 (AOCS, 2017).

RESULTS AND DISCUSSION

Total of Color Difference

Color indicates the level of brightness of a material. PKSAC-AC260 and DAC's

ability to purify waste cooking oil can be seen from the total color difference compared to standard cooking oil. Analysis of color differences was carried out using a portable colorimeter NH310 3nh. The appearance of waste cooking oil after purification using PKSAC-AC260 as well as DAC and standard cooking oil can be seen in Figure 2. The difference of waste cooking oil color after purification between PKSAC-AC260 and DAC can be seen in Table 1.

Table 1 shows that the total color difference of waste cooking oil after being purified using DAC has a smaller value than PKSAC-AC260. This shows that the color of waste cooking oil that has been purified using DAC is similar in color to the control cooking oil, while the total color difference of the control cooking oil can be seen in Table 6. This is due to the higher ability of DAC to absorb the color of used cooking oil compared to PKSAC-AC260. The results of the study (Ulfah *et al*., 2016), showed that the total surface area of DAC was 1068.391 m^2/g, while the total surface area of PKSAC according to (Ulfah et al., 2019) was 1395.69 m^2/g. The mesoporous surface area of the DAC is 275.431 m^2/g with a mesoporous volume of 0.528 cc/g (Ulfah *et al*., 2017), while PKSAC-AC260 has a mesoporous surface area of 139.81 m^2/g with a mesoporous volume of 0.16 cc/g (20%) (Ulfah *et al*., 2019). From the textural properties between DAC and PKSAC activated carbons, it shows that higher mesoporous surface area and mesoporous volume of activated carbon will also adsorb higher dyes.

DAC has the ability to adsorb free fatty acids higher than PKSAC, this is due to the different mesoporous properties of the two types of activated carbon. According to Ulfah *et al.* (2016), palm kernel shell activated carbon (PKSAC) has a lower adsorption ability than DAC. PKSAC-AC260 has a surface area of 1680.877 m^2/g, while DAC has a surface area of 1068.391 m^2/g with a mesoporous volume/total pore volume of 55.4% (Ulfah *et al*., 2017).

DAC has the ability to adsorb free fatty acids higher than PKSAC-AC260, this is also influenced by the composition of surface functional groups of DAC which are non-polar more than polar groups. The surface functional groups of the polar DAC consist of NH (amine), P-H (phosphine), C-N (amine). While the non-polar functional groups consist of C-H (ester), N-H (amine), carbonate ion, C-H (alkyne) and C-I (aliphatic iodo compounds) (Ulfah *et al.*, 2019). Although PKSAC-AC260 has a higher surface area than DAC, the composition of non-polar surface functional groups is less than that of polar functional groups. According to Ulfah *et al.* (2019), PKSAC-AC260 contains a polar functional group consisting of -NH stretch (amine), P-H (phosphine), NO_2 antisym stretch (aromatic nitro compound), organic sulphate, C-N stretch (secondary amine), C-O stretch (alcohol) and O-H out-of-plane bend (alcohol). While the non-polar functional groups of PKSAC-AC260 consist of C-H asym/sym (methyl), C-O-C antisym stretch (aliphatic ether), C-H out-of plane bend (vinyl) and C-H bend (alkyne).

The higher the concentration of activated carbon used for refining waste cooking oil, the free fatty acid content of the purified oil decreased. According to Karabulut *et al.* (2008), increasing the concentration of activated carbon will provide more surface area for adsorption. At a certain concentration, the adsorption ability of activated carbon will slow down and eventually be constant. Activated carbon at a concentration of 1.5% to 2.5% showed an increase in free fatty acid adsorption, this was indicated by the lower free fatty acid content. When compared to the free fatty acid content of standard cooking oil, that is maximum 0.3% based on SNI 3741:2013 (BSN, 2013),

so refined waste cooking oil contains much higher free fatty acids, so the purified oil is not good if used as edible oil because it will have an impact on health.

Free Fatty Acid

Free fatty acid content is a parameter of oil quality due to the hydrolysis process. High levels of free fatty acids indicate low oil quality. The effectiveness of adsorption of waste cooking oil using PKSAC-AC260 and DAC can be seen from the decrease in free fatty acid levels after purification. The levels of free fatty acids after the purification process using PKSAC-AC260 and DAC are shown in Table 2.

Water Content

The water content in cooking oil will contribute to the hydrolysis process which produces free fatty acids, thereby reducing them. The ability of PKSAC-AC260 and DAC to adsorb water from waste cooking oil can be seen from the water content of waste cooking oil after purification which is presented in Table 3.

Based on Table 3, both PKSAC-AC260 and DAC could adsorb waste cooking oil water, which is not significantly different, this is probably due to the low water content of waste cooking oil (1.33%).

The higher concentration of activated carbon did not affect the water content of the waste cooking oil after adsorption. These data support the reason that although the surface area of activated carbon increases with increasing concentration of activated carbon, but because the polar surface functional groups of activated carbon are not strong enough to bind hydrogen ions from water, so the water adsorbed is quite low.

Based on SNI 3741:2013, good quality cooking oil has a maximum moisture content of 0.15% (BSN, 2013). Waste cooking oil after the adsorption process using PKSAC-AC260 and DAC is still in accordance with SNI standards.

Peroxide Value

Peroxide value is a parameter for reducing the quality of cooking oil caused by the oxidation process of unsaturated fatty acids that bind oxygen to the double bonds (Ketaren, 2008). Peroxide value has a positive correlation to the rate of oxidation reaction of cooking oil. The ability of PKSAC-AC 260 and DAC in purifying waste cooking oil based on peroxide value is presented in Table 4.

Table 4 shows that waste cooking oil after refining using DAC produced a lower peroxide value than PKSAC-AC260. The adsorption of waste cooking oil peroxide involves surface functional groups contained in activated carbon. Peroxide compounds are non-polar oxidation products that are soluble in cooking oil, so they are more easily bound to the non-polar surface functional groups of activated carbon.

The higher the concentration of activated carbon used for adsorption, the lower the peroxide value of waste cooking oil. The more activated carbon will increase the surface area of contact with the peroxide compound, so the chance of the peroxide bound to the functional group on the surface of the activated carbon is also higher. Increasing the concentration of activated carbon will increase the adsorption of a compound on the adsorbent particles physically and chemically.

The standard cooking oil peroxide value is 18.84 meq/kg, while based on SNI 3741:2013 the maximum is 10 meq/kg (BSN, 2013). This shows that at concentrations of activated carbon up to 2.5%, it has not been able to produce regenerated waste cooking oil that is suitable for use as edible oil. The regenerated waste cooking oil in this study would be more suitable to be used as an oil-based non-food product.

Saponification Value

The saponification value indicates the mg of KOH required to saponify one gram of oil or fat (Ketaren, 2008). The saponification value is used to estimate the molecular weight of crude oil or fat. A low saponification value indicates a high molecular weight. The ability of PKSAC-AC 260 and DAC for refining waste cooking oil based on saponification value can be seen in Table 5.

Table 5 shows that the saponification value of waste cooking oil after purification using DAC and PKSAC-AC260 is not significantly different, but the saponification value that using DAC is greater than that of PKSAC-AC260. This is because DAC has a higher ability to adsorb free fatty acids, so the oil contains lower free fatty acids and the oil has a higher purity.

The higher concentration of activated carbon for refining waste cooking oil will increase the saponification value. Due to the greater amount of activated carbon used for adsorption, the more surface area for free fatty acid adsorption, so that the purity of the oil is higher. The higher purity of refined oil, it will have properties similar to the quality standards of cooking oil. The saponification value of cooking oil standar is 190.259 mg KOH/g, while according to Hui (1996) the saponification value for palm oil is 190.1 – 201.7 mg/g (Hui, 1996).

The Quality of Refined Oil Using Activated Carbon

The quality of waste cooking oil that was purified using activated carbon PKSAC-AC260 and DAC compared to the quality of waste cooking oil before purification with fresh of cooking oil and standard of cooking oil based on SNI 3741:2013 is presented in Table 6.

From Table 6 it can be seen that activated carbon A1 (PKSAC-A260) and A2 (DAC) have the ability to increase the color brightness of waste cooking oil (ΔE) by 5.44 and 4.53, respectively. PKSAC-A260 and DAC were able to reduce the water content of waste cooking oil from 1.33% to 0.13% and 0.09%, respectively. PKSAC-A260 and DAC were able to reduce the free fatty acid content of waste cooking oil from 1.47% to 0.79% and 0.61%, respectively. PKSAC-A260 and DAC were able to reduce the peroxide value of waste cooking oil from 163.47 meq/kg to 116.40 meq/kg and 98.82 meq/kg, respectively. PKSAC-A260 and DAC were able to increase the saponification value of waste cooking oil from 155.22 mg/g to 180.48 mg/g and 184.48 mg/g, respectively. The ability of PKSAC-AC260 to purify waste cooking oil is lower than that of standard activated carbon (DAC).

CONCLUSION

Activated carbon PKSAC-AC260 or DAC with a concentration of 2.5% was able to improve the quality of the best waste cooking oil. Purification of used cooking oil using PKSAC-AC260 and DAC was able to increase the color brightness of waste cooking oil (ΔE) by 5.44 and 4.53, respectively. PKSAC-A260 and DAC were able to reduce the water content of waste cooking oil from 1.33% to 0.13% and 0.09%, respectively. PKSAC-A260 and DAC were able to reduce the free fatty acid content of waste cooking oil from 1.47% to 0.79% and 0.61%, respectively. PKSAC-A260 and DAC were able to reduce the peroxide value of waste cooking oil from 163.47 meq/kg to 116.40 meq/kg and 98.82 meq/kg, respectively. PKSAC-A260 and DAC were able to increase the saponification value of waste cooking oil from 155.22 mg/g to 180.48 mg/g and 184.48 mg/g, respectively. The ability of PKSAC-AC260 to purify waste cooking oil is lower than that of standard activated carbon (DAC). The quality of processed oil from waste cooking oil does not meet the quality standards of cooking oil based on SNI 3741: 2013. Waste

cooking oil purification products are more suitable used for oil-based non-food products, such as soap, biodiesel, or other non-food products.

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Figure 1. Waste cooking oil after purification using PKSAC-AC260 as well as DAC and control cooking oil. a (DAC 2.5%); b (DAC 2%), c (DAC 1.5%); d (waste cooking oil); e (control cooking oil); f (PKSAC-AC260 1.5%); g (PKSAC-AC260 2%); h (PKSAC-AC260 2.5%)

Table 1. Total difference color from waste cooking oil after purification

Type of Activated Carbon	Concentration of Activated Carbon			Average of A
	B1 (1.5%)	B2 (2%)	B3 (2.5%)	
A1 (PKSAC-A260)	6.35	5.15	4.82	5.44^{x}
A2 (DAC)	5.74	4.86	2.98	4.53^{y}
Average of B	6.04^{c}	$5,01^{d}$	3.90^{f}	

Note: The mean value in the column or row followed by a different letter indicates the difference between treatments at a significance level of 0.05 (Duncan's Test)

Table 2. Fatty acid contents of waste cooking oil after purification

Type of Activated Carbon	Concentration of Activated Carbon			Average of A
	B1 (1.5%)	B2 (2%)	B3 (2.5%)	
A1 (PKSAC-A260)	0.99	0.73	0.66	0.79^{x}
A2 (DAC)	0.71	0.62	0.49	0.60^{y}
Average of B	0.85^{c}	0.68^{d}	0.57^{f}	

Note: The mean value in the column or row followed by a different letter indicates the difference between treatments at a significance level of 0.05 (Duncan's Test). Free fatty acid content of control cooking oil is 0.17%, while based on SNI 3741:2013 a maximum of 0.3% (BSN, 2013)

Table 3. Water content of waste cooking oil after purification (%)

Type of Activated Carbon	Concentration of Activated Carbon			Average of A
	B1 (1.5%)	B2 (2%)	B3 (2.5%)	
A1 (PKSAC-A260)	0.10	0.13	0.16	0.13^{x}
A2 (DAC)	0.10	0.07	0.09	0.08^{x}
Average of B	0.10^{c}	0.10^{c}	0.12^{c}	

Note: The mean value in the column or row followed by a different letter indicates the difference between treatments at a significance level of 0.05 (Duncan's Test). Water content of control cooking oil is 0.02%, while based on SNI 3741:2013 a maximum of 0.15% (BSN, 2013)

Table 4. Peroxide value of waste cooking oil after purification (meq/kg)

Type of Activated Carbon	Concentration of Activated Carbon			Average of A
	B1 (1.5%)	B2 (2%)	B3 (2.5%)	
A1 (PKSAC-A260)	151.51	114.12	83.56	116.40^{x}
A2 (DAC)	120.82	95.08	53.57	89.82^{y}
Average of B	136.16^{c}	104.60^{e}	68.56^{d}	

Note: The mean value in the column or row followed by a different letter indicates the difference between treatments at a significance level of 0.05 (Duncan's Test). Peroxide value of control cooking oil is 18.84 meq/kg, while based on SNI 3741:2013 a maximum of 10 meq/kg (BSN, 2013)

Table 5. Saponification value of waste cooking oil after purification (mg KOH/g)

Type of Activated Carbon	Concentration of Activated Carbon			Average of A
	B1 (1.5%)	B2 (2%)	B3 (2.5%)	
A1 (PKSAC-A260)	174.85	182.00	184.58	180.48^{x}
A2 (DAC)	175.64	177.46	200.33	184.48^{x}
Average of B	175.25^{c}	179.73^{c}	192.45^{c}	

Note: The mean value in the column or row followed by a different letter indicates the difference between treatments at a significance level of 0.05 (Duncan's Test). Saponification value of control cooking oil is 190.259 mg KOH/g, while according to Hui (1996) the saponification value of palm oil is 190.1- 201.7 mg KOH/g (Hui, 1996)

Table 6. The quality of purified waste cooking oil compared to the fresh of cooking oil

Sample	TCD (ΔE)	W (%)	FFA (%)	PV (meq/kg)	SV (mg/g)
Refined oil using PKSAC-A260	5.44	0.13	0.79	116.40	180.48
Refined oil using DAC	4.53	0.09	0.61	89.82	184.48
Waste cooking oil	19.75	1.33	1.47	163.47	155.22
Fresh of cooking oil *	0.00	0.02	0.02	18.84	190.26
SNI 3741:2013	Normal	Max. 0.15	Max. 0.3	Max. 10	190.1 – 201.7**

Note: TCD (Total color difference); W (water content); FFA (free fatty acid content); PV (peroxide value); SV (saponification value); * (Bimoli special); ** (Hui, 1996)