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REDUCING FREE FATTY ACIDS AND PEROXIDE VALUE IN USED COOKING OIL USING ACTIVATED COCONUT SHELL CHARCOAL COMBINED WITH LEMONGRASS STEMS

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Abstract: Used cooking oil is obtained from the residue of the frying process, which has been used repeatedly where the fatty acids are increasingly saturated and the triglyceride content breaks down into volatile and non-volatile components that dissolve in oil. Used cooking oil waste can cause an increase in COD and BOD levels in waters, resulting in the death of aquatic biota. The aims of this study were (1) to determine the optimum contact time of activated charcoal d combination of citronella sticks on decreasing levels of free fatty acids, (2) to determine the optimum contact time of activated charcoal d combination of citronella sticks on decreasing peroxide numbers, (3) to determine the average percentage of free fatty acid reduction and peroxide value in the clarification of used cooking oil using activated coconut shell charcoal combined with lemongrass stems. This research method is a laboratory experiment with despising, neutralization, and bleaching stages. The results showed that the average decrease in optimum free fatty acid levels occurred at 60 minutes of contact time, namely A3 at 73.86%, B3 at 66.67%, and C3 at 82.74%. The average decrease in the optimum peroxide number occurred at a contact time of 60 minutes, namely A3 of 57.16%, B3 of 52.85%, and C3 of 49.68%. So, the coconut shell-activated charcoal combined with lemongrass stems effectively clarifies the used cooking oil. In the future, it is necessary to vary the absorbent particle size to improve the quality of the waste cooking oil so that it can be reused.

Keywords: *Activated Charcoal, Coconut Shell, Lemongrass Stalks, Used Cooking Oil, Clearing*

INTRODUCTION

Cooking oil is one of the basic human needs that functions as a very important frying medium, and its needs are increasing. The increase in cooking oil prices occurred in November and December 2021, namely by 0.08 percent each, from 107.10% in September 2021 to 112.34% in March 2022 [1]. This encourages consumers, namely homemakers and MSMEs, to switch to using used cooking oil or even using oil repeatedly. Used cooking oil is cooking oil obtained from the residue of the frying process, which has been used repeatedly where the fatty acids have become increasingly saturated and the triglyceride content breaks down into volatile and non-volatile components that dissolve in the oil [2]. The high-heating process in cooking oil will produce free fatty acids, carbonyl compounds, and peroxides, which can cause chronic poisoning in humans [3]. Using cooking oil repeatedly in frying pans will damage some of the vitamins and essential fatty acids in cooking oil, causing poisoning in the body [4].

In addition, used cooking oil is also household waste, which is sometimes disposed of carelessly into the environment so that it can cause environmental pollution [5]. Used cooking oil waste can cause an increase in COD and BOD levels in the waters, resulting in the death of aquatic biota [6]. Used cooking oil continuously dumped into the ground or ditches will cause soil pollution in the form of soil pores being closed by used cooking oil, the soil becomes infertile, and the mineral content in clean water becomes polluted [2]. For this reason, it is necessary to manage used cooking oil waste into products that have economic value to reduce its repeated use and indiscriminate disposal of used oil waste.

One way to manage used cooking oil is to purify it before it is made into an economically valuable product such as bar soap from used cooking oil [7], process waste used cooking oil into balsam [8], increase the value of used cooking oil is economical by processing it into laundry soap [9], and processing used cooking oil into aromatherapy candles [10]. Before processing it into a product with economic value, waste cooking oil needs to be purified and clarified to reduce the acid number and peroxide value, decrease concentration, and eliminate rancidity. Refining used cooking oil is one way to approach the characteristics of fresh cooking oil, extend the cooking oil's life, and ensure its use safety. Several studies that have been conducted on the refining and Clarification of used cooking oil include; Hartono and Suhendi purified used cooking oil using a natural zeolite catalyst to produce the best color absorbance at a 15% NaOH concentration of 4.03, for bleaching using 90% zeolite (15% NaOH concentration) of 3.08 [11]; Muhammad clarification of used cooking oil using activated charcoal Leucaeba leucocephala wood with activated charcoal Leucaena leucocephala wood can improve the quality of used cooking oil by reducing levels of fatty acids so that the color of used cooking oil becomes clearer [4]; Pardede, et. al. purified used cooking oil using an eggshell adsorbent with the results of the acid number, peroxide value and water content obtained at optimum conditions that fulfilled the SNI 3741-2013 standard for cooking oil which was 0.6 milligram KOH/ gram, 10 meq O2/ kilogram as well as 0.15% [12]; Tuslinah, et. al. purified used cooking oil using activated charcoal from ironwood sawdust with an FFA content of 0.5576% with a decrease of 84.15% and a peroxide number of 2.4617 mek/kg with a decline of 89.15% [13].

In this study, the clarification of used cooking oil used activated coconut shell charcoal combined with lemon grass powder with variations in contact length with depicting, neutralization, and bleaching methods. Activated charcoal is the most popular adsorbent used as an adsorbent in the adsorption process of liquid waste[14]. Activated charcoal with coconut shell as raw material was chosen in this study because it is easy to obtain and economical, and the resulting surface is also quite large, ranging from 524-704 m2/gr. Surface area is a material selection parameter because, in gas adsorption applications, the greater the surface area, the greater the adsorption capacity of activated carbon. The coconut shell is a hard layer of lignin, cellulose hemicellulose methoxyl, and various minerals. The content of these materials varies according to the type of coconut. The hard structure is caused by silicate (SiO2), which is quite high in the coconut shell. The shell weight is around 15-19% of the total weight of the coconut. Coconut shell belongs to the hardwood group with a water content of approximately 6-9% [15]. The purpose of this study was to determine the optimum contact time of activated charcoal with a combination of citronella stems in the clarification of used cooking oil based on FFA content and peroxide value; to determine the effectiveness of coconut shell activated charcoal combined with citronella containsins in clarification of used cooking oil.

RESEARCH METHODS

Production of activated charcoal

Clean the coconut shell from the fibers and the remaining fruit flesh that sticks,, then heat it in the oven at 110ºC for one hour. Put several coconut shells in the furnace at 300ºC for 1 hour. Then, the charcoal is cooled in the desiccator. Grind the charcoal to a size of 35 mesh, then soak it in 2% HCl for 2 hours. After that, the charcoal is separated from the activator solution by filtering and drying it.

Research Sample

The sample in this study consisted of three samples, namely sample A (used oil for frying fish), sample B (used oil for frying dried fish), and sample C (used cooking oil for skin crackers). Each sample will be given a long contact treatment with activated coconut shell charcoal combined with lemongrass stems with time variations of 15 minutes (1), 30 minutes (2), and 60 minutes (3).

Cleaning of used cooking oil (Despicing)

A total of 250 mL of used cooking oil is weighed, and then water is added with a ratio of 250 mL oil, and 250 mL water 1:1. The mixture is put into a 500 mL glass beaker. Next, the crucible and the sample are heated until the water in the glass beaker remains a portion of the initial volume. The mixture was put into a separatory funnel and stood for 1 hour. The water fraction at the bottom is separated to obtain water-free oil. After that, it is filtered with filter paper to separate the remaining impurities. The filtering results are used in the neutralization process.

Cleaning of used cooking oil (Neutralizing)

190 mL of despised cooking oil was put in an Erlenmeyer, heated to a temperature of 35°C, and added 6 mL of 16% NaOH solution. The mixture was stirred for 10 minutes at 40°C, then cooled for 10 minutes and filtered

Cleaning of used cooking oil (Bleaching)

From the results of neutralization, we then added an activated charcoal combination of lemongrass with certain time variations for the clarification or bleaching of used cooking oil.

Process of cleaning/bleaching

A total of 39 grams of coconut shell activated carbon combined with lemon grass has interacted with 100 mL of neutralized used cooking oil with variations in contact time of 10, 30, and 60 minutes. The results of this Clarification are used to determine the number of peroxides and free fatty acids.

Determination of Free Fatty Acid Content

Each 5 mL of clarified used cooking oil was measured and put into a 250 mL Erlenmeyer, then 25 mL of ethanol and heated at 40°C. After that, 2 mL of PP indicator was added to the mixture titrated with 0.05 M NaOH solution until a pink color appeared and did not disappear for 30 seconds. Determination of free fatty acids in this study will use the following formula:

$$\%\ \text{FFA} = \frac{\text{Ml NaOH x N. NaOH x BM. Palmitic acid}}{\text{Sample weight x 1000}} \text{ x } 100$$

Keterangan :
% FFA = Free fatty acid content
mL NaOH = Titrant volume NaOH
N. NaOH = Solution Normality NaOH (0,1)
BM = Palmitic acid molecular weight (256,42 gr/mol)

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Determination of Peroxide Number

Each 8 mL of refined cooking oil was put into a 250 mL Erlenmeyer, then 15 mL of 95% acetic acid solution and 97% chloroform (3:2), shaken until homogeneous. Then, the mixture was added with 0.5% KI solution and closed tightly. Let stand for 2 minutes while shaking, then add 25 ml of distilled water. The mixture was titrated with 0.1 N Na2S2O3 until the yellow color almost disappeared, and then 0.5 mL of 1% starch solution was added and titrated again until the blue color disappeared.

Calculate the peroxide number expressed in milliequivalents in every 1000 grams of sample. The determination of the peroxide number uses the following formula:

$$\text{Peroxide Number} = \frac{\text{mL } Na_2S_2O_3 \text{ x N}.Na_2S_2O_3 \text{ x } 1000}{\text{sample (gr)}}$$

Keterangan:
mL Na2S2O3 = Titrant volume Na2S2O3
N. Na2S2O3 = Solution Normality Na2S2O3 (=0,1)

RESULTS AND DISCUSSION

Quality Test of Coconut Shell Activated Charcoal

The activated charcoal obtained is tested for quality standards concerning the Indonesian National Standard (SNI) 06-3730-1995 concerning quality requirements and testing of activated charcoal. The results of the activated charcoal quality test can be seen in Table 1.

Table 1. Quality Test of Coconut Shell Activated Charcoal

No	Type of Analysis	Test Results after activation
1	yield	95.15 %
2	Water content	1.92 %
3	Ash Content	6.07 %
4	Volatile Substances	24.19 %
5	Iodine Absorption	369.028 mg/gr

(Research Primner Resources: 2023)

Activated charcoal is a carbon compound whose adsorption power has been increased by carrying out carbonation and activation processes. Before using activated charcoal, it is necessary to carry out a quality test to prepare the best quality of activated charcoal before application. Based on Table 1, the yield information obtained from activated charcoal, which has been activated with 2% HCl for 24 hours, yields 95.15% and a water content of 1.92%. The results of testing the water content in this study have met the quality standards of activated charcoal based on SNI No. 06-3730-1995, which is a maximum of 15%. This water content test was carried out to analyze the hygroscopic properties of activated coconut shell charcoal. The low water content in activated coconut shell charcoal in this study indicated that the free water content and bound water in the activated charcoal had evaporated during the carbonization process. High water content in activated charcoal can reduce the quality of its adsorption power. High water content will reduce the absorption of activated charcoal [16]. Therefore, activated carbon is expected to have a low water content so that the quality of activated charcoal becomes high.

Percentage of ash content identifies the content of metal oxides that may still be present in activated carbon. These metal oxides can be small amounts of silicon, sulfur, calcium, or other metals [17]. Based on Table 1, the ash content of activated charcoal after activation is 6.07. The ash content of coconut shell activated charcoal in this study met the standard quality of activated charcoal set by SNI 06-3730-95, which is a maximum of 10%. High ash content affects the quality of activated carbon when applied as an adsorbent [18].

The percentage of volatile matter content indicates the amount of compound that has not evaporated during the carbonization and activation processes but evaporates at 900°C. The absorption ability of activated carbon is affected by the high levels of volatile matter, which, if the value is too high, will reduce its absorption capacity [18]. Based on Table 1, it is known that the level of active charcoal vapor after activation is 24.19%. This value meets the quality standard requirements for activated charcoal according to SNI 06-3730-1995, a maximum of 25%.

In this study, iodine absorption by activated charcoal showed a value of 369.028 mg/gr. This meets the standard for activated carbon. According to SNI 06-3730-1995, the iodine number has at least a minimum value of 200 mg/g. Iodine absorption describes the capacity of activated carbon to adopt components with low molecular weight [17]. The number of mg of iodine that can be absorbed by 1 gram of activated carbon is called the iodine number. Referring to activated carbon with a high iodine number it identifies a larger surface area and microstructure [19].

Results of Preliminary Measurement of Free Fatty Acid Levels and Peroxide Numbers before being given treatment

Free fatty acids are one of the indicators to determine or control the quality of cooking oil. At the same time, the peroxide value is used as a determinant to identify the level of oxidation of cooking oil. The use of cooking oil repeatedly can not immediately determine whether, sooner or later the cooking oil is no longer suitable for use, besides that, the type of food that is fried also greatly affects the speed of damage to the quality of the cooking oil. Following are the results of initial measurements of free fatty acid levels and peroxide numbers in several used cooking oils for several types of foodstuffs:

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Table 2. Measurement of FFA Initial Levels and Peroxide Numbers

Sample	initial free fatty acid levels (FFA)	Peroxide Number Initial Levels
A	7.23%	76.91 mek/kg
B	3.84%	66.15 mek/kg
C	5.97%	73.76 mek/kg

(Research Primner Resources: 2023)

The high free fatty acids can affect the taste and smell of the oil, causing a decrease in the quality of the oil. The higher the FFA value, the more free fatty acids contained in the oil, so that these free fatty acids will affect the oil's chemical properties, physical properties, and stability during the frying process [20]. Based on Table 1, the highest levels of free fatty acids were found in sample A, where the cooking oil used came from the former frying of wet fish. The higher the water content in cooking oil, the higher the free fatty acid levels are due to the higher hydrolysis reactions in the cooking oil used [21]. Unsaturated fatty acids can increase the oxygen in the double bond to form peroxides. Peroxide is formed due to heating, which causes damage to the oil or fat. The higher the water content in the oil, the higher the peroxide value.

Effect of Length of Contact on Reducing Free Fatty Acid Levels

During frying, cooking oil will experience heating at high temperatures of 170^{O}C – 180^{O}C for quite a long time, which will cause oxidation, hydrolysis, and polymerization processes that produce compounds resulting from oil degradation, such as ketones, aldehydes, and polymers, which are detrimental to human health. These processes cause oil damage, including increased free fatty acids (FFA) levels. The degree of unsaturation of the oil decreases along with the increase in temperature in the heating process; it can cause the fatty acid chains to break up into free radicals, which are harmful to health. For this reason, cooking oil is not recommended to be used repeatedly or disposed of carelessly in the environment before cleaning. The following is the result of the clarification of used cooking oil using activated coconut shell charcoal combined with lemongrass:

Based on Table 4 above, it can be seen that the tendency for free fatty acids in used cooking oil decreased after absorption treatment with active coconut shell charcoal combined with lemongrass stems. In each sample with a contact time of 60 minutes, there was a decrease in free fatty acid levels; this could be caused by free fatty acid compounds in used cooking oil with polar carboxyl ends being absorbed by activated coconut shell charcoal adsorbents combined with lemongrass. The decrease in free fatty acid levels is affected by the length of contact between the used cooking oil and the adsorbent, where the longer the contact time, the faster the purification process will be [22]. It can be said that more free fatty acids are adsorbed into the pores of the coconut shell-activated charcoal. This is in line with research conducted by Ritonga, et. al. (2020), which states that the longer the adsorption time, the greater the contact time between activated charcoal and used cooking oil so that the absorption of FFA takes place more optimally [23]. As an adsorbent for activated charcoal, the combination of lemongrass coconut shells can reduce levels of free fatty acids beyond the levels of free fatty acids contained in used cooking oil that have not been adsorbed; this is due to the presence of compounds contained in activated charcoal and can neutralize or stabilize the free fatty acid compounds contained in cooking oil.

Table 3. Measuring FFA Levels for Purifying Used Cooking Oil

Treatment	initial free fatty acid levels (FFA)	final free fatty acid content (FFA)	Decrease Percentage FFA
A1	7.23%	4.82%	33.33%
A2	7.23%	3.23%	55.32%
A3	7.23%	1.89%	73.86%
B1	3.84%	3.18%	17.18%
B2	3.84%	1.59%	58.59%
B3	3.84%	1.28%	66.67%
C1	5.97%	4.62%	22.61%
C2	5.97%	2.56%	57.12%
C3	5.97%	1.03%	82.74%

(Research Primner Resources: 2023)

The Effect of Long Contact on Peroxide Number Decreasing

The peroxide value is the amount of meq peroxide in every 1000 grams (1 kg) of oil or fat. The peroxide value indicates the degree of damage to the oil or fat. The following is a decrease in the peroxide value resulting from the clarification of used cooking oil using activated charcoal in a combination of lemongrass:

Table 4. Measurement of Peroxide Number after Treatment

Treatment	Initial Peroxide Number	final concentration of Peroxide Number	Percentage of Decrease in Peroxide Numbers
A1	76.91 mek/kg	60.08 mek/kg	21.88%
A2	76.91 mek/kg	46.51 mek/kg	39.53%
A3	76.91 mek/kg	32.95 mek/kg	57.16%
B1	66.15 mek/kg	52.63 mek/kg	20.44%
B2	66.15 mek/kg	44.83 mek/kg	32.23%
B3	66.15 mek/kg	31.19 mek/kg	52.85%
C1	73.76 mek/kg	56.64 mek/kg	23.21%
C2	73.76 mek/kg	50.78 mek/kg	31.15%
C3	73.76 mek/kg	37.11 mek/kg	49.68%

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Based on Table 4 above, it was found that the decrease in peroxide value in the clarification of used cooking oil using activated coconut shell charcoal combined with citronella stems increased with increasing contact time. The lowest peroxide number was found in the variation of contact time of 60 minutes for each sample, which decreased by an average of 53.23%. This is in line with research conducted by Tumagor and Ayu, which stated that the contact time of activated carbon significantly affected the water content, peroxide value, free fatty acids, specific gravity, color sensory test, and the aroma of used cooking oil [24]. In this study, the highest decrease in peroxide occurred at 60 minutes, where the adsorbent may not reach a saturation level, so the possibility of absorption can occur if the contact time is added. The optimum reaction time is effective and efficient for reducing the peroxide value in used cooking oil because the adsorbent is saturated. Hence, the absorption is not optimal [25].

The adsorption process between peroxide and activated charcoal from coconut shells combined with lemongrass stems is due to the difference in potential energy between the surface of the adsorbent and the adsorbed substance, involving either physical or chemical forces. The interaction between peroxide and activated charcoal of coconut shells combined with citronella stems in this study is chemical adsorption, namely, the interaction between the adsorbate and the adsorbent involves the formation of chemical bonds such as covalent bonds [26].

The decrease in peroxide may occur due to absorption by the adsorbent through its pores. Coconut shell-activated charcoal has a large surface area of 500 m2/g, so it has a high potential for absorption of heavy metals in water [27]. Activated charcoal is a material that has changed its physical and chemical properties due to activation of the charcoal. The physical and chemical activation of charcoal makes the particles' absorption and surface area higher, so the ability to absorb the material is expected to be higher [28].

The adsorption process between peroxide and activated charcoal from coconut shells combined with lemongrass stems is due to the difference in potential energy between the surface of the adsorbent and the adsorbed substance, involving either physical or chemical forces. The interaction between peroxide and activated charcoal from coconut shells combined with citronella stems in this study is chemical adsorption, namely, the interaction between the adsorbate and the adsorbent involves the formation of chemical bonds such as covalent bonds [29].

The chemical content of lemongrass is essential oil, saponins, polyphenols, and flavonoids; these active compounds indicate that lemongrass has considerable antibacterial activity [30]. Compounds that dominate the antibacterial effect of lemongrass are polyphenol compounds, which can cause protein denaturation; flavonoid compounds form complex compounds with extracellular proteins as antibacterial by denaturing bacterial cell proteins and damaging cell membranes, and saponin compounds effectively inhibit the growth of Gram-positive bacteria [31]. The combination of lemongrass stalks can increase the clear color, improve the smell, and inhibit the growth of bacteria in used cooking oil after cleaning.

CONCLUSION

Based on the description of the discussion above, from the results of this study, it can be concluded that several things are by the objectives of conducting this research, namely: The optimum contact time for coconut shell activated charcoal combined with citronella stems in the clarification of used cooking oil in reducing free fatty acid levels and reducing peroxide value is 60 minutes, and clarification of used cooking oil using activated coconut shell charcoal combined with lemongrass stems is effective in reducing levels of free fatty acids and peroxide value in used cooking oil.

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