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Effectiveness of Purification Used Cooking Oil Using Adsorbents: Activated Charcoal Seeds Salak *(Salacca zalacca)*

***Sufia, Tri Santoso, Sitti Aminah, & Sitti Rahmawati**
Program Studi Pendidikan Kimia/FKIP – Universitas Tadulako, Palu – Indonesia 94119

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Abstract

Salak seeds have a reasonably high cellulose content. This suggests that salak seeds can be used as a raw material for producing activated charcoal to refine used cooking oil. This study aims to determine the effectiveness of refining used cooking oil using active charcoal adsorbents derived from Salacca zalacca seeds. The methods used to determine free fatty acid levels and peroxide values are the acid-base and iodometric titration methods. This study will observe several variables that affect the adsorption process, namely the mass of activated charcoal and the contact time of used cooking oil. The results showed a decrease in free fatty acids and peroxide number, and the colour of the oil became brighter with increasing adsorbent and contact time. The best results were obtained at a mass of 15 grams and an adsorption time of 120 minutes. Based on the quality requirements for cooking oil as specified in SNI 3741-1995.

Keywords: Used cooking oil, salak seeds, activated charcoal

Introduction

Cooking oil is essential as a medium for food processing, making it difficult to separate it from human life (Sari et al., 2021). Cooking oil is critical for creating colour and aroma, enhancing the taste of food, extending shelf life, increasing the nutritional value of products, and improving the savoury flavour of food (Ihwan et al., 2019).

The repeated use of cooking oil without limitation and at high temperatures decreases oil quality, characterised by a dark colour change, a less pleasant aroma, and increased peroxide value and free fatty acid levels (Ihwan et al., 2019). Repeated cooking is commonly called used cooking oil (Bakhri et al., 2021). Used cooking oil can increase the potential for cancer in the body if you consume it too often (Yuniati et al., 2022). According to health experts, cooking oil can only be used two to four times for frying because it has a toxic effect on humans (Zulkifli, 2019). Used cooking oil that has been damaged can still be utilised. One alternative to improve its quality is adsorption using salak seed adsorbents, which helps maintain the oil's quality. This absorbent can remove some free fatty acids and peroxides (Bakhri et al., 2021).

Salak fruit is one of the fruit crop commodities cultivated in the Central Sulawesi Agency (BPS). In 2020, salak fruit production was around 963.00 tons. Salak fruit will also affect the amount of salak seed waste produced. Most of Indonesia, especially Central Sulawesi, has waste of salak seeds that are just thrown away and not utilised properly. Salak seeds contain the main chemical components of carbohydrates, consisting of 28.98 % cellulose and 59.37 % hemicellulose, specifically glucomannan, which makes them suitable for use as active charcoal adsorbents.

Activated charcoal is a material with a carbon composition (85 – 95 %), a large surface area, and a complex porous structure, making it easier for the adsorption process. Therefore, it is widely used as an adsorbent in industries, such as the food processing industry, one of which is to purify charcoal-used cooking oil (Wardani et al., 2022).

Observing this, researchers are interested in recycling used cooking oil using activated charcoal derived from salak seeds. Adsorbents are an alternative method for purifying used cooking oil, which examines the parameters of peroxide number and free fatty acid levels. The research showed that activated charcoal from snake fruit seeds could reduce the peroxide value by 56.18 % and provide a 76.04 % reduction in the free fatty acid levels found in used cooking oil.

Based on the above, the researchers continued the research conducted by Ihwan et al.

*Correspondence:
Sufia
e-mail: fiasufia13@gmail.com

(2019) by testing the feasibility of the oil in terms of the parameters of peroxide value and free fatty acids. This study also reviewed the parameters of peroxide value and free fatty acids. The oil's acid, specific gravity, and colour are considered to determine if used cooking oil can be reused. Refining used cooking oil is expected to extend the shelf life of cooking oil without compromising consumer health and meeting the Indonesian National Standard (SNI) requirements.

Methods

Tools and materials

The tools used in this study include 250 mL and 1000 mL beakers, magnetic stirrer, 50 mL burette, stand and clamps, 80 mesh sieve, electric furnace, desiccator, oven, dropping pipette, measuring cup, Erlenmeyer, hot plate, analytical balance, blender, thermometer, funnel, stopwatch, pH meter, separating funnel, tongs, 10 mL pycnometer, centrifuge, and glassware commonly used in laboratories.

The ingredients used include: cooking oil for frying fresh fish, salak seeds, 96 % ethanol, phenolphthalein indicator, 0.1 N NaOH, 15 % NaOH, $ZnCl_2$ 10 %, mixed CH_3COOH dan $CHCl_3$ (3:2), solution $Na_2S_2O_3$ 0.01 N, saturated potassium iodide, 1 % starch indicator solution and distilled water.

Procedure

Production of activated charcoal from salak seeds

Salak seeds are washed and cut into small pieces to optimise drying. They are dried in the sun for ± 7 days to remove the water content. The dried malacca seeds are carbonised using a furnace at 400 °C for 2 hours until the seeds turn into charcoal. Afterwards, the charcoal is left to cool down, and then it is ground using a blender and sifted through an 80-mesh sieve (Ihwan et al., 2019).

Activation of salak seed charcoal

Salak seed charcoal, which has been mashed, is then subjected to the activation process. Charcoal activation begins with weighing 50 grams of charcoal and placing it into a 500 ml beaker. Next, 250 ml of 10 % ZnCl2 solution is added, and the mixture is shaken with a magnetic stirrer for 10 minutes. The charcoal is then washed with distilled water until the pH is neutral, as measured using a pH meter. Then, the activated charcoal is dried in an oven at 105 °C for 3 hours (Nusratullah & Aminah, 2020).

Characterisation of activated charcoal

Water level

Activated charcoal was weighed carefully, up to 1 gram, in a porcelain cup whose weight was known. Then, it is dried in an oven at 105 °C for approximately 3 hours. After that, it was cooled in a desiccator and weighed. The same treatment was repeated until a constant weight was obtained (Lestari et al., 2016). The following formula can be used to determine the water content.

$$\text{Water content} = \frac{B-F}{B-G} \times 100\% \qquad (1)$$

(Lestari et al., 2016)

Where:
B = mass of the cup with initial charcoal (grams);
F = mass of the cup with dried charcoal (grams);
G = mass of the cup (grams).

Ash rate

Activated charcoal is weighed carefully, typically up to 1 gram, in a cup whose weight is known. Then, it was incubated in the furnace at 600 °C for 2 hours. After that, cool it in a desiccator and weigh it until a constant weight is obtained (Lestari et al., 2016). The following formula can be used to determine ash content.

$$\text{Ash Content} = \frac{F-G}{B-G} \times 100\% \qquad (2)$$

(Lestari et al., 2016).

Where,
B = mass of the cup with initial charcoal (grams);
F = mass of cup with ash (grams);
G = mass of the cup (grams).

Proses despicing

Weigh as much as 500 grams of used cooking oil then add water with the composition of oil: water (1:1), put it in a 1000 mL beaker, then heat it until the water in the beaker is reduced by half, then the oil layer is deposited in a separatory funnel for 1 hour then water fraction at the bottom is separated to obtain water - free oil, after which it is filtered to separated the remaining impurities. This process was followed by neutralisation: 400 grams of disputed oil was heated to 40 °C, and 20 mL of 15 % NaOH was added to the oil while stirring for 10 minutes. The mixture is allowed to stand for 10 minutes to cool, and then it is filtered through a centrifuge to separate the filtrate and residue (Miskah et al., 2018).

Used cooking oil adsorption

Up to 100 mL of used cooking oil is placed into a 250 mL beaker and then heated to 90 °C. After that, 5 grams, 10 grams, and 15 grams of activated charcoal from Zalacca seeds are added and stirred at 100 rpm for 60 minutes, 90 minutes, and 120 minutes. The purified cooking oil is filtered using a centrifuge and then filtered using Whatman No. 42 (Ubaidah et al., 2018).

Oil quality analysis

Free fatty acid determination

Up to 5 grams of used cooking oil was added to a 125 ml Erlenmeyer flask, followed by 25 ml of 96% ethanol. The mixture was then heated at 75 °C for 10 minutes. After cooling, three drops of PP indicator were added. The sample is stirred for 30 seconds and titrated with 0,1 N NaOH solution. The titration is stopped if the titrant solution turns pink and at least 10 seconds have passed. This treatment was carried out twice. The following formula can determine Free fatty acids (Nusratullah & Aminah, 2020).

$$\text{Free fatty acids} = \frac{V \times N \times A}{m \times 1000} \times 100\% \qquad (3)$$

Where,
V = Volume of NaOH used for titration (mL);
N = NaOH Normality;
A = molecular weight of lauric acid;
m = sample weight.

Peroxide number determination

Weigh approximately 3 grams of used cooking oil and place it into a 125 mL Erlenmeyer flask. Add 15 mL of a 3:2 acetic acid–chloroform solution and shake until the mixture is homogeneous. Then, add 0.5 mL of a saturated KI solution to a closed Erlenmeyer flask. Then, stand for 1 minute while shaking and add 15 mL of distilled water. The mixture is titrated with 0.01 N $Na_2S_2O_3$ until the yellow color almost disappears. Then, 0.5 ml of the titrant, a 1% starch solution, is added, and the mixture is titrated again until the blue colour begins to fade. This treatment was carried out 2 times. The following formula can determine the peroxide number (Mardiana & Santoso, 2020).

$$\text{Peroxide number} = \frac{V \times N \times 1000}{sample\ waight} \times 100\% \qquad (4)$$

Specific weight

The pycnometer is washed and dried, then the oil sample is weighed carefully in a 10 mL pycnometer whose weight is known. This treatment was carried out 2 times. The following formula can determine this specific gravity

$$\text{Specific gravity} = \frac{b-a}{v} \qquad (5)$$

(Miskah et al., 2018).

The clarity or colour of the oil

The clarity or colour of the oil is measured using a Spectronic 20. The cooking oil sample is put into the cuvette. Then, the absorbance is measured at a wavelength of 470 nm using pure oil as a blank.. (Fauziah, 2013). This treatment was carried out 2 times.

Results and Discussion

Characterisation of activated charcoal

This research began by producing active charcoal from Zalacca seeds as an adsorbent for refining used cooking oil for frying fresh fish collected from restaurants in Palu City and households. Making charcoal from salak seeds involves dehydration, carbonisation, and activation. In the dehydration process the salak seed sample has been cleaned and cut into small pieces and then dried in the sun for ± 7 days where this drying aims to remove the water content contained in the salak seeds, the following process is carbonisation, the salak seeds are put into the furnace to carry out the coagulation process at temperature of 400 °C for 2 hours until the zalacca seeds turn into charcoal.

This carbonisation process produces charcoal with absorption properties and a neat structure (Mantong et al., 2018). The charcoal was then crushed using a blender and sieved through an 80-mesh screen. Sieving aims to get a uniform particle size of charcoal. The final process is activation, where the charcoal is weighed at least 50 grams. Then, 250 ml of 10% $ZnCl_2$ is added, and the charcoal is activated by soaking for 24 hours.

Activation is a treatment for charcoal that aims to increase its porosity, primarily by breaking hydrocarbon bonds or oxidising surface molecules, thereby altering its physical and chemical properties. Specifically, the surface area increases, enhancing its adsorption power (Anggriawan et al., 2019). After being soaked with 10 % $ZnCl_2$ solution, filtered using filter paper which aims to separate the filtrate and residue in the form of charcoal, then washed using distilled water until the pH is neutral, the washing process with purified water aims to remove impurities left on the activated charcoal when activated with 10 % $ZnCl_2$ so that does not affect the adsorption power of the activated charcoal. Then, the activated charcoal is dried in an oven at 105 °C for 3 hours. This drying aims to remove the water content from activated charcoal. The research results for the characteristics of activated charcoal from salak seeds, including water and ash content, are presented in **Table 1.**

Table 1. Characterisation of activated charcoal from salak seeds

Sample	Average moisture content (%)	Average ash content (%)
Activated charcoal of salak seeds	1.13	2.48

Water content

Moisture content is one of the key requirements for activated charcoal, significantly affecting its quality. The purpose of determining the water content is to determine the hygroscopic nature of activated charcoal (Batu et al., 2022). The remaining water content will fill the pores of the charcoal, thereby reducing the adsorption power of activated charcoal. The moisture content was determined by heating the activated charcoal sample in an oven at 105°C. This aims to fully evaporate the water content in the activated charcoal of the Zalacca seeds. The average water content from activated charcoal made from Zalacca seeds is 1.13 %. Based on the results, the water content is relatively low, below the requirements for good activated charcoal (SNI No. 06-3730-1995), which stipulates a maximum of 15 %.

Ash content

Another parameter that affects the quality of activated charcoal is ash content. The determination of ash content aims to quantify the metal oxides that remain in the activated charcoal of salak seeds after the activation process. The ash content was tested by placing the Zalacca seeds in a furnace at 600 °C for 2 hours (Lestari et al., 2016). Ash content will affect the quality of activated charcoal as an adsorbent. Excessive ash will cause pore blockage, thereby decreasing the surface area of activated charcoal. The average ash content of activated charcoal in Zalacca seeds is 2.49 %. Based on the results, the ash content of activated charcoal in salak seeds is relatively low, meeting the requirements for activated charcoal as specified in SNI No. 06-3730-1995, which stipulates a maximum ash content of 10 %.

Analysis of used cooking oil quality

This study's refining of used cooking oil was conducted through three processes: desolventising, neutralisation, and bleaching. Removing spices removes fine suspended particles, such as proteins, carbohydrates, salt, sugar, and spices used for frying food, without reducing the amount of free fatty acids in the oil. A neutralisation process follows this process. The neutralisation process is a method to separate free fatty acids from oil by reacting them with bases to form soap. The following process is the bleaching process. Bleaching is a purification process to remove unwanted dyes in oil using an adsorbent (Dewi et al., 2022). The adsorbent used in this study was charcoal derived from snake fruit. The results of analysing the quality of used cooking oil, including free fatty acids, peroxide value, specific gravity, and oil colour, are presented in Tables 2 and 3.

Table 2. Comparison of time variations in the determination of free fatty acids and peroxide value

No	Oil type	Adsorbent weight (gram)	Free fatty acid (%)				Peroxide Number (meq/Kg)			
			Adsorption time							
			0	60	90	120	0	60	90	120
1.	MP	0	0.14	-	1.17	-	0.14	-	1.17	-
2.	MJRM 1	0								
		5	1.32	0.75	0.71	0.63	10.83	6.17	5.50	4.67
		10		0.69	0.59	0.51		5.33	4.67	3.17
		15		0.67	0.49	0.30		5.17	3.17	1.83
3.	MJRM 2	0								
		5	1.26	0.69	0.67	0.55	10.17	5.17	4.83	4.33
		10		0.63	0.51	0.30		4.17	3.50	2.83
		15		0.59	0.47	0.26		3.67	2.17	1.50

Table 3. Comparison of time variation in determining the specific gravity and colour of the oil

No	Oil type	Adsorbent weight (gram)	Specific gravity g/ml				Colour (Absorbance)			
			Adsorption time							
			0	60	90	120	0	60	90	120
1.	MP	0	0.8419	-	0.04	-	0.8419	-	0.04	-
2.	MJRM 1	0								
		5	0.9536	0.9356	0.9355	0.9341	0.39	0.17	0.15	0.12
		10		0.9347	0.9346	0.9339		0.14	0.12	0.09
		15		0.9342	0.9333	0.9329		0.12	0.10	0.06
3.	MJRM 2	0								
		5	0.9425	0.9289	0.9279	0.9268	0.35	0.15	0.14	0.11
		10		0.9281	0.9271	0.9266		0.11	0.10	0.06
		15		0.9275	0.9267	0.9263		0.09	0.07	0.05

Information :
Comparison Oil (MP)
Restaurant Cooking Oil before adsorption (MJRM 1)
Household Used Cooking Oil before adsorption (MJRT 2)

Free fatty acids

Free fatty acids are formed through the hydrolysis reaction of oils or fats. The content of free fatty acids in an oil is one of the parameters determining the quality of cooking oil (Nasir et al., 2014). Data on the research results regarding free fatty acid levels are presented in Table 2.

Based on the measurement results, it is evident that the lowest free fatty acid content is observed in the adsorbent mass of 15 grams and a contact time of 120 minutes. The free fatty acid content in restaurant cooking oil after adsorption and household cooking oil after adsorption is 0.30 % and 0.26 %, respectively, indicating that the results obtained are below the SNI standard, which is a maximum of 0.3 %. The data is relatively low and does not meet the quality requirements for cooking oil, as specified in SNI 01-3741-1995. The contact time and mass of the adsorbent are two key factors that influence the adsorption process, as absorption occurs through physical interactions between the adsorbate and the adsorbent during the contact process. This indicates that activated charcoal from Zalacca seeds is effectively used as an adsorbent for refining used cooking oil.

This study was conducted by Qory et al. (2021). Refining used cooking oil using activated carbon from salak seeds, with a 90-minute processing time and an adsorbent size of 100 mesh, yielded a fatty acid content of 0.108 %. The longer the absorption time of free fatty acids, the more significant the decrease. Particle size also affects the reduction of free fatty acids because particle size is related to surface area; in this study, the amount obtained was lower.

Peroxide number

The peroxide number indicates how much fat or oil has undergone oxidation. It is significant in identifying the oil's oxidation states. Oxygen can oxidise oil containing unsaturated fatty acids to produce a peroxide compound. The lower the Peroxide Number value, the higher the oil quality (Sari et al., 2021).

Based on the measurement results, the lowest peroxide number is found at an adsorbent mass of 15 grams. A contact time of 120 minutes, the peroxide number in restaurant used cooking oil after adsorption and household used cooking oil after adsorption is 1.83 meq / Kg and 1.50 meq / Kg This means that the data is relatively low and is following the quality requirements for cooking oil according to SNI 01-3471-1995, where the quality requirement is a maximum peroxide value of 2 meq / Kg. This shows that activated charcoal from salak seeds is effective as an adsorbent for purifying used cooking oil.

In this study conducted by Qory et al. (2021), refining used cooking oil using activated carbon from salak seeds, a 90-minute process with an adsorbent size of 100 mesh, resulted in a peroxide value of 2.5 meq O_2/kg. It can be seen that the longer the peroxide absorption time, the more significant the decrease in peroxide number. In this study, the amount obtained was higher.

Specific gravity

Specific gravity is one of the key parameters determining oil quality. Its value is determined by comparing the weight of oil and water in the same volume. The value of particular gravity is related to each component's weight fraction. The value of the specific gravity fraction is determined using a pycnometer; the standard particular gravity of oil is 0.900 g/cm^3 (Mardiana & Santoso, 2020).

Based on the results of specific gravity measurements, it was shown that for household used cooking oil after adsorption and household cooking oil after adsorption, namely 0.9329 g/mL and 0.9263 g/mL, it was close to the quality requirements for cooking oil according to SNI 01-3741-1995, where the requirements the quality of the specific gravity of cooking oil is a maximum of 0.900 g / mL. The use of activated charcoal from salak seeds results in a significant reduction in the specific gravity of used cooking oil.

In another study conducted by Miskah et al. (2018) on the purification of used cooking oil using activated carbon from durian as an adsorbent, the contact time was 150 minutes, and the adsorbent weight was 6 grams. Obtained a specific gravity value of 0.909 g/ml; in this study, the amount received was higher. It can be seen that the longer the absorption time, the more significant the decrease in oil density. This decrease is due to the adsorbent having adsorbed many impurities from used cooking oil. Consequently, the oil molecules are reduced, eliminating the odour and dark colour. The determination of density is influenced by the water content and the level of impurities attached to the oil.

Color clarity

The colour has been used as an index of the quality of cooking oil for many years. The testing method can be colour, which has been used as an index of cooking oil quality for many years. The test method can be done using a spectrophotometer. The higher absorbance value shows the darker colour of the oil. More products are derived from oil degradation (Paramitha, 2012).

Based on the results of measuring the colour of the oil at a mass of 15 grams and a contact time of 120 minutes, the lowest absorbance values were obtained for restaurant cooking oil after adsorption and household cooking oil after adsorption, precisely 0.06 A and 0.05 A, respectively. The high clarity of the oil indicates that the impurities contained in it are decreasing. The lower the absorbance value of the

oil, the more precise the colour of the oil will be. The decrease in the absorbance value of the oil is due to the physical adsorption process by the Zalacca seed charcoal, which is composed of pores with a surface area capable of absorbing compounds in the oil.

Conclusions

Based on the research results, it can be concluded that the process of refining used cooking oil using snake fruit-activated charcoal is influenced by the adsorbent mass and the contact time of the cooking oil with the snake fruit-activated charcoal. The greater the mass of the adsorbent and the longer the contact time, the higher the quality of the used cooking oil obtained, as indicated by a decrease in free fatty acids, peroxide value, specific gravity, and oil colour. The effectiveness of purifying used cooking oil using activated charcoal adsorbent from salak seeds was achieved at an adsorbent mass of 15 grams and a contact time of 120 minutes, with various oil quality analyses namely free fatty acids, peroxide value and oil color, which met the cooking oil quality requirements according to SNI 3741-1995, but the specific gravity is close to the quality requirements for cooking oil according to SNI 3741-1995.

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