e-ISSN xxxx-xxxx
p-ISSN xxxx-xxxx

URANIUM
Journal of Sciences Research

Publisher: Fermora Education
Available online at https://fermoraedu.com/index.php/ujsr/

Optimization of Mixing Activated Carbon and Fly Ash as Adsorbents for Reducing Free Fatty Acid Content and Acid Value in Used Cooking Oil

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Article Information:

Check for updates

Submited: 08 August 2025
Revised: 09 August 2025
Accepted: 09 August 2025

Keywords:
acid value; adsroben; FFA

Abstract

Used cooking oil is a waste product from frying oil that has been reused multiple times, leading to a decline in quality, characterized by a rancid odor and a darker color. This condition can pose health risks if the oil is reused without proper treatment. One of the purification methods for used cooking oil is adsorption using adsorbent materials. This study aims to determine the optimum conditions for the addition of activated carbon and fly ash to reduce the levels of free fatty acids and acid value in used cooking oil, as well as to calculate the percentage reduction of these two parameters. The used cooking oil treated through adsorption was tested for free fatty acid content and acid value. The results showed that the combination of activated carbon and fly ash was effective in reducing free fatty acid content and acid value. Optimum conditions were achieved with the addition of 20% adsorbent by sample weight, at a heating temperature of 50 °C for 30 minutes. The percentage reductions in free fatty acid content, acid value, and water content were 14.0%, 21.8%, and 0.27%, respectively.

1. INTRODUCTION

In food technology, cooking oil plays a vital role as a heat transfer medium, flavor enhancer, and a source of energy and calories in food. The frying process causes food to lose most of its moisture content and become dry (Febrianto et al., 2020). However, repeated use of cooking oil can lead to oil degradation, resulting in decreased food quality and nutritional value. The common practice of reusing oil, especially among food vendors and households, produces used oil that turns dark brown to black, commonly known as waste cooking oil. This oil poses potential health risks, such as fat accumulation in blood vessels, and increases the risk of heart disease and stroke (Silalahi et al., 2021).

The degradation of oil during the frying process is primarily caused by oxidation and hydrolysis reactions, which lead to the breakdown of triglycerides into various compounds, including free fatty acids. To enable the reuse of waste cooking oil, it must undergo a purification process (Musa et al., 2021). One effective method is adsorption, wherein adsorbent materials are employed to bind and remove color pigments, colloidal substances, and degradation products from the oil.

Several types of adsorbents commonly used in the purification of waste cooking oil include activated carbon, fly ash, activated clay, and bentonite (Ardi et al., 2021). Activated carbon is a porous material with a high carbon content (85–95%) and a large surface area (300–3500 m^2/g), making it highly effective as an adsorbent. Meanwhile, fly ash is a residue from coal combustion that is rich in silica, alumina, calcium oxide, and carbon. Its porous physical and chemical characteristics, along with its active carbon content, make fly ash a promising adsorbent material for the removal of organic contaminants (Wijaya et al., 2021).

Several previous studies have examined the effectiveness of various adsorbents in the purification of waste cooking oil. Denie & Firdaus, (2023) demonstrated that activated carbon significantly reduced free fatty acid (FFA) content under optimal conditions of 50 °C, 50% activated carbon addition, and a contact time of 50 minutes (Denie & Firdaus, 2023). Ardi et al. (2021) reported that a mixture of activated carbon and bentonite improved the quality of waste cooking oil to meet the Indonesian National Standard (SNI 3741:2013), with an acid value reduction efficiency of 73.81% and a moisture content reduction of 94% (Ardi et al., 2021). Another study by Rahmayanti et al, (2023) utilized activated carbon derived from sugarcane bagasse activated with H_3PO_4, showing a reduction in FFA content to 0.336% and a peroxide value of 6.99 meq/kg, making the purified oil suitable as a raw material for biodiesel production (Rahmayanti et al., 2023).

Based on these findings, the combination of activated carbon and fly ash as adsorbents is considered to have promising potential in enhancing the effectiveness of waste cooking oil purification. Therefore, this study aims to optimize the mixture of activated carbon and fly ash in reducing the free fatty acid content and acid value of waste cooking oil through an adsorption process.

2. METHOD

Materials and equipment

The equipment used in this study included a heater, magnetic stirrer, burette, stand, clamp, thermometer, 100 mL and 250 mL beaker glasses, 100 mL volumetric flask, 250 mL Erlenmeyer flask, funnel, 50 mL measuring cylinder, dropper pipette, digital balance, porcelain crucible, glass stirrer, and oven. The materials used in this study included waste cooking oil (sourced from street food vendors and household activities), activated carbon, fly ash, 0.1 N NaOH, distilled water (aquadest), 95% neutral alcohol, and phenolphthalein (pp) indicator.

Free Fatty Acid (FFA) Analysis

A total of 110 g of waste cooking oil sample was weighed and transferred into an Erlenmeyer flask, which was then covered with plastic. Next, 50 mL of 95% alcohol was added to the flask and homogenized. The cover was removed, and the mixture was heated to boiling, then allowed to cool. After cooling, the solution was titrated with 0.1 N NaOH standard solution, and phenolphthalein (pp) indicator was added until a color change from colorless to a stable pink (lasting for at least 30 seconds) was observed (Susanti & Husin, 2023). The percentage of free fatty acids was calculated using the following equation.

$$ree\ fatty\ acid\ (\%) = \frac{Volume\ NaOH\ \text{ x N NaOH x BM fatty acid}}{mass\ of\ sample}$$

Acid Value Analysis

The free fatty acid content of the waste cooking oil obtained from the previous step can be used to calculate the acid value by converting the molecular weight (MW) of the fatty acid to the molecular weight of NaOH (Ilyas & Husin, 2023). The acid value can be calculated using the following equation:

$$Acid\ value\ (\%) = \frac{V\ NaOH\ \text{ x N NaOH x BM NaOH}}{mass\ of\ sample}$$

Adsorption Process

The adsorption process was carried out by placing 110 g of waste cooking oil into a beaker glass and heating it at two different temperature variations (50°C and 60°C). Once the desired reaction temperature was reached, the adsorbent was added in specific compositions (10% and 20% of the sample weight), followed by stirring. The mixture of waste cooking oil and adsorbent was then separated, and the filtrate was collected for further analysis (Jondra et al., 2022).

Research Variations

This study involved both fixed and variable parameters. The fixed variable was the stirring speed, which was maintained at level 6 (500 rpm). The variable parameters included the adsorbent percentage (10% and 20% of the sample weight), heating temperature (50°C and 60°C), and contact time (30 and 40 minutes). The experimental combinations were as follows: A1B1C1 (Adsorbent 10%, Heating Temperature 50°C, Time 30 Minutes), A1B1C2 (Adsorbent 10%, Heating Temperature 50°C, Time 40 Minutes), A1B2C1 (Adsorbent 10%, Heating Temperature 60°C, Time 30 Minutes), A1B2C2 (Adsorbent 10%, Heating Temperature 60°C, Time 40 Minutes), A2B1C1(Adsorbent 20%, Heating Temperature 50°C, Time 30 Minutes), A2B1C2 (Adsorbent 20%, Heating Temperature 50°C, Time 40 Minutes), A2B2C1 (Adsorbent 20%, Heating Temperature 60°C, Time 30 Minutes), A2B2C2 (Adsorbent 20%, Heating Temperature 60°C, Time 40 Minutes).

3. RESULT AND DISCUSSION

In this study, the quality of waste cooking oil was improved through an adsorption process using activated carbon and fly ash to reduce the free fatty acid (FFA) content and acid value. The waste cooking oil sample used in this study was obtained from leftover oil used for frying chicken. The waste oil was filtered to remove residual food particles before undergoing the adsorption process and subsequent analysis. The use of cooking oil at high temperatures and repeated usage leads to a decline in oil quality, as indicated by a darker color and the development of rancid odors. This deterioration negatively affects the sensory and nutritional qualities of fried foods, including taste, appearance, and healthiness. To allow the reuse of waste cooking oil, regeneration efforts were carried out by reducing its free fatty acid content and acid value (Rini Rahmayanti et al., 2023).

The quality of waste cooking oil can be improved through the adsorption process using adsorbents to preserve the oil's quality for reuse. The adsorption process involves adding the adsorbent to the oil and stirring the mixture. According to Adam (2017), to reduce the risks associated with the use of waste cooking oil, recovery methods can be applied. One such method is adsorption, which enables the reuse of oil without compromising its quality. Adsorption is considered an economical and effective method due to its relatively low cost, regenerability, and operational simplicity.

In this study, activated carbon and fly ash were used as adsorbents. Activated carbon possesses a high adsorption capacity, making it effective in absorbing gases, odors, and color compounds present in used cooking oil (Irawan et al., 2013). It can be produced from various materials such as coconut shells, plantain peels, cashew nut shells, and salak seeds. This is supported by Adam et al. (2017), who reported the use of activated carbon as an adsorbent to reduce free fatty acid content and acid value in used cooking oil (Adam, 2017). Activated carbon can adsorb waste cooking oil components to restore the quality of the oil by removing the dark color and turbidity. A small particle size of activated carbon is preferred to accelerate the adsorption process by increasing the surface area of the adsorbent. In this study, activated carbon derived from coconut fiber was used due to its effectiveness as an adsorbent. Moreover, a higher amount of activated carbon in the adsorbent mixture enhances the efficiency of the adsorption process.

Coal fly ash is an industrial waste product that is typically disposed of in landfills, which poses potential environmental and public health risks. These include the leaching of heavy metals into water bodies and the dispersion of fine fly ash particles by wind, which can cause respiratory issues and contribute to air pollution. Although fly ash has many potential applications, its use has so far been largely limited to being an additive in concrete production. The use of fly ash as an adsorbent is justified by its abundant availability and low cost.

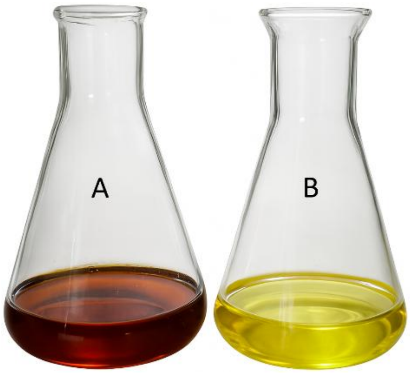

Figure 1. Used cooking oil sample before (A) and after (B) adsorbent treatment

Free Fatty Acid (FFA) Analysis

Free fatty acid (FFA) content serves as a fundamental indicator of the quality of cooking oil. The FFA content in waste cooking oil was analyzed using an acid-base titration method. This method is based on the principle of neutralizing the hydrogen ions (H^+) of free fatty acids with hydroxide ions (OH^-) from sodium hydroxide (NaOH) until the equivalence point is reached, which is indicated by a color change in the sample. The analysis was conducted on both treated and untreated samples. The untreated sample represents the initial condition of the waste cooking oil before undergoing the adsorption process, while the treated samples refer to oil that had been subjected to various adsorption treatments using activated carbon and fly ash (Susparini et al., 2022). The following table presents the results of FFA analysis for both treated and untreated waste cooking oil samples.

Table 1. Free fatty acid of analysis for both treated and untreated waste cooking oil samples

Variations	FFA before adsorption (%)	FFA after adsorption (%)	FFA lowering (%)
$A_1B_1C_1$		0.282±0.001	9.5
$A_1B_1C_2$		0.273±0.001	10.4
$A_1B_2C_1$		0.272±0.001	10.5
$A_1B_2C_2$	0.377±0.0005	0.268±0.001	10.9
$A_2B_1C_1$		0.237±0.0005	14.0
$A_2B_1C_2$		0.263±0.001	11.4
$A_2B_2C_1$		0.249±0.001	12.8
$A_2B_2C_2$		0.250±0.0005	12.6

The addition of activated carbon and fly ash as adsorbents significantly affected the free fatty acid (FFA) content in waste cooking oil. The quality of the used cooking oil improved after undergoing the adsorption process with activated carbon and fly ash, as indicated by a reduction in FFA content ranging from 9% to 14%. The hydrolysis reaction between oil and water contributes to the reduction in FFA levels. Activated carbon and fly ash adsorbents possess short carbon chains that react with the hydroxyl groups present in FFAs due to their polar nature, which allows them to dissolve in water. As a result, FFAs are adsorbed by the combined activated carbon and fly ash adsorbents.

These adsorbents contain cellulose, which can adsorb FFAs as well as color compounds in the oil. Cellulose has hydroxyl (–OH) groups, while FFAs contain functional groups that can bind to the –OH groups of the adsorbent. Coconut fiber, in particular, has a relatively high cellulose content, leading to a greater reduction in FFA levels. According to Irawan et al. (2013), the cellulose content in coconut fiber ranges from 32% to 43%. The cellulose content in the adsorbent also plays a role in absorbing color pigments from the oil (Murjana & Handayani, 2022).

Based on Table 1, the most effective adsorption treatment was the addition of 20% adsorbent (by sample weight), heated at 50°C for 30 minutes, resulting in a 14% reduction in FFA content. This finding confirms that increasing the adsorbent dosage (up to 20% of sample weight) increases the available surface area of the adsorbent, thereby providing more active sites for adsorption. A larger surface area corresponds to a higher number of active contact points with the compounds present in the waste cooking oil.

Analysis of acid value

The quality of oil can also be assessed through acid value analysis. The breakdown of oil due to complex reactions such as hydrolysis can produce acids that affect the flavor and aroma of the oil, leading to rancidity. The acid value is defined as the number of milligrams of base required to neutralize one gram of the waste cooking oil sample (Jondra et al., 2022). A higher acid value indicates a greater amount of free fatty acids resulting from the hydrolysis of oil. The higher the acid value, the lower the oil quality; conversely, a lower acid value reflects better quality and oil that is more suitable for consumption. The acid value can be calculated based on the free fatty acid content.

Table 2. Acid value of analysis for both treated and untreated waste cooking oil samples

Perlakuan	Acid value before adsorption (%)	Acid value after adsorption (%)	Acid value lowering (%)
$A_1B_1C_1$		0.440±0.0005	14.9
$A_1B_1C_2$		0.427±0.001	16.2
$A_1B_2C_1$		0.425±0.001	16.4
$A_1B_2C_2$	0,589±0.001	0.418±0.0005	17.1
$A_2B_1C_1$		0.371±0.0005	21.8
$A_2B_1C_2$		0.411±0.001	17.8
$A_2B_2C_1$		0.389±0.001	20.0
$A_2B_2C_2$		0.393±0.0005	19.6

According to the Indonesian National Standard (SNI 3741:2013), the maximum permissible acid value in cooking oil is 1 mg NaOH/g. Based on Table 5, the acid value analysis of waste cooking oil treated with activated carbon and fly ash as adsorbents shows notable differences among the samples. The highest acid value after the adsorption process was found in sample A1B1C1 (10% adsorbent, 50°C, 30 minutes) at 0.440 mg NaOH/g, while the lowest was recorded in sample A2B1C1 (20% adsorbent, 50°C, 30 minutes) at 0.371 mg NaOH/g. However, all acid value results following the adsorption treatment were within the acceptable limit set by the Indonesian National Standard, which is a maximum of 1 mg NaOH/g.

The addition of activated carbon and fly ash as adsorbents affected the acid value of the waste cooking oil. The quality of used cooking oil improved after the adsorption process, as indicated by a reduction in acid value ranging from 14% to 21%. This decrease suggests that the use of these adsorbents helps inhibit oxidation processes in the oil, thereby preventing the formation of aldehydes. The absence of aldehydes further prevents the formation of carboxylic acids, which are responsible for increasing the acid value in waste cooking oil (Harahap et al., 2021).

Based on Table 2, the most effective adsorption treatment was the addition of 20% adsorbent (by sample weight) at 50°C for 30 minutes, resulting in an acid value reduction of 21.8%. This confirms that increasing the amount of adsorbent increases the surface area available, thereby providing more active sites for interaction with the compounds in the waste cooking oil. The surface area reflects the number of active contact points available for adsorption (Nadir et al., 2013).

The acid value analysis results in Table 2 demonstrate a significant improvement in the quality of waste cooking oil after the adsorption process. However, despite these improvements, it is still recommended to limit the repeated use of cooking oil (waste oil) to maintain health and safety.

4. CONCLUTION

The optimum condition for reducing free fatty acid content and acid value in used cooking oil is achieved by adding 20% adsorbent (based on sample weight) with heating at 50°C for 30 minutes. The percentage reductions in free fatty acid content, acid value, and water content of the used cooking oil with the addition of activated carbon and fly ash were 14.0%, 21.8%, and 0.27%, respectively."

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