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Preparation of Activated Carbon From Kluwek (Pangium Edule) Shells as An Adsorbent for Effective Removal of Free Faty Acid (FFA) from Used Cooking Oil

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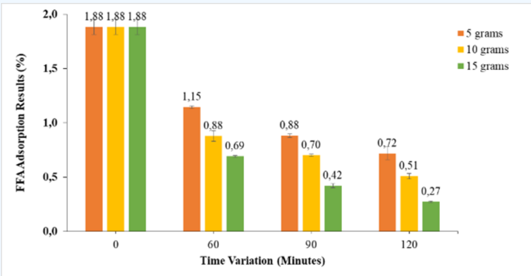
ABSTRACT

Used cooking oil from repeated use exhibits characteristics such as high free fatty acid (FFA) content, brownish color, and rancid odor, which pose risks to both health and the environment. This study aims to analyze the effectiveness of activated carbon derived from Pangium edule shells in adsorbing FFAs, color, and odor from oil samples, as well as to identify the functional groups present in the adsorbent using FTIR analysis. The adsorption process was conducted with varying adsorbent masses (5, 10, and 15 grams) and contact times (60, 90, and 120 minutes) to determine the optimal removal conditions. The results indicate that NaOH-activated Pangium edule shell carbon contains hydroxyl (-OH) and carbonyl (C=O) functional groups, which play a crucial role in adsorption. After the adsorption process, an efficiency of 85.57% was achieved, reducing the FFA content from 1.88% to 0.27% under optimal conditions (adsorbent mass of 15 grams and contact time of 120 minutes), along with a significant color change. The shift in FTIR spectrum intensity after adsorption suggests that polar compounds such as FFAs interacted with active sites on the adsorbent surface. Thus, activated carbon from Pangium edule shells is effective for treating used cooking oil in an environmentally friendly manner, meeting the quality standards of SNI 7709:2019, and offering a promising low-cost and sustainable solution that can be applied in household settings, small-scale food businesses, and community-based waste oil treatment initiatives



Keywords: *Used Cooking Oil, Kluwek Shells, Adsorption, Free Fatty Acids, FTIR*

1. INTRODUCTION

Household consumption of used cooking oil in 2023, as recorded by the National Food Agency (Badan Pangan Nasional—BAPANAS), reached 2.66 million tons per year, reflecting a 2% increase from the previous year [1]. This condition can be attributed to the repeated use of cooking oil, resulting in waste in the form of used cooking oil [2]. The more frequently the oil is reused, the greater its degradation, posing significant risks to both health and the environment [3]. Oil degradation can be observed through its free fatty acid (FFA) content and physical changes such as a brownish color and an unpleasant odor.

Unused cooking oil is often disposed of indiscriminately into water bodies or soil. This practice can cause water pollution, leading to foul odors and inhibiting the absorption of nutrients in the soil [4]. Additionally, from a health perspective, consuming degraded cooking oil has been linked to severe conditions such as cancer, digestive disorders, and metabolic imbalances. FFAs in used cooking oil result from hydrolysis reactions that occur during the frying process, particularly at high temperatures ranging from 160 to 200°C. The higher the FFA content, the lower the quality of the used cooking oil [5]. According to the Indonesian National Standard (SNI 7709:2019) for palm cooking oil, the acceptable FFA content limit is 0.3% [6]. Therefore, effective treatment of used cooking oil is necessary to improve its quality and enable its reuse.

One of the most effective methods for treating used cooking oil is adsorption using activated carbon as an adsorbent. The adsorption process facilitates the reduction of FFAs, color clarification, and odor removal in used cooking oil. Adsorbents used in this treatment are typically derived from organic waste materials containing cellulose, hemicellulose, and lignin, which are known for their excellent adsorption capacity [7]. In this study, the researchers utilized Pangium edule shell waste as the raw material for adsorbent production, sourced from local food vendors.

Pangium edule shells are an organic waste product, with the fruit's flesh commonly used as a food seasoning while the shells are discarded with minimal utilization. The primary composition of Pangium edule shells includes 40.99% hemicellulose, 70.52% cellulose, and 27.88% lignin [8]. Due to its high cellulose content, the shell has a brown color and a hard texture. Moreover, activated carbon derived from cellulose-rich materials has been shown to enhance adsorption efficiency due to its porous structure, making it a promising adsorbent.

Numerous studies have explored different methods for processing used cooking oil. For instance, a study [9] utilized activated carbon from coffee grounds, achieving optimal removal at an adsorbent mass of 10 g within 120 minutes,

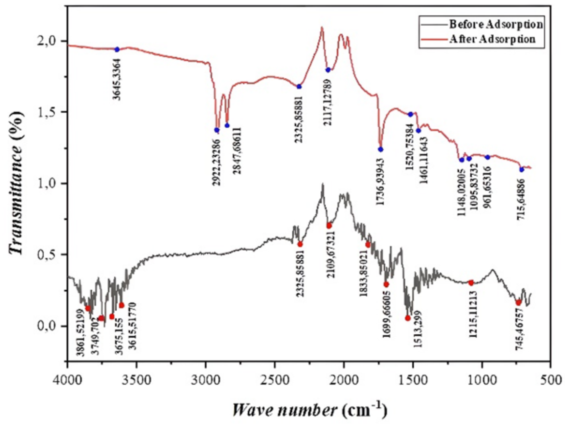


Figure 1. FTIR Spectrum Before and After Adsorption.

reducing FFA levels from 0.972% to 0.383%, while also clarifying the oil's color. Another study [10] experimented with NaOH-activated eggshell-based activated carbon, achieving effective results at a 7.5 g adsorbent dosage, reducing FFA content from 10.7% to 0.33%. Previous research indicates that increasing the adsorbent mass generally enhances adsorption efficiency [11].

Therefore, this study aims to utilize Pangium edule shell waste as an adsorbent for used cooking oil treatment. The primary objectives are to analyze the characterization of the adsorbent before and after adsorption using FTIR and to evaluate the effectiveness of activated carbon in removing FFAs, color, and odor from used cooking oil.

2. METHODS

In this experiment, activated carbon from Pangium edule shells was produced and activated using NaOH as an adsorbent for the adsorption process of used cooking oil. The research was conducted from September to November 2024 at the Environmental Engineering Laboratory, Institut Teknologi Sumatera.

2.1 Research Equipment

The equipment used in this experiment includes a beaker glass, Erlenmeyer flask, burette, stand, bulb, spatula, 10-mesh sieve, oven, desiccator, funnel, hotplate, magnetic stirrer, and orbital shaker.

2.2 Research Materials

The materials used in this study include used cooking oil, Pangium edule shells, 0.1 N and 1 N NaOH solution, distilled water, 95% ethanol, and 1% phenolphthalein indicator.

2.3 Used Cooking Oil Sample Collection

The used cooking oil samples were collected from food vendors in Way Hui Village, Jati Agung District. The samples were obtained from repeatedly used cooking oil (3–10 times) that had undergone quality degradation. The sampling process was carried out using the grab sampling method to ensure uniform sample characteristics for testing.

2.4 Adsorbent Preparation

In the adsorbent preparation process, Pangium edule shells were first cleaned and then sun-dried. Next, the shells underwent a carbonization process to convert them into charcoal. The resulting charcoal was ground and sieved using a 10-mesh sieve. The activation process was then carried out using NaOH solution to enhance adsorption efficiency. The final stage involved FTIR analysis to identify the functional groups present in the adsorbent.

2.5 Adsorption Process

The adsorption process was conducted by adding the adsorbent to 200 mL of used cooking oil sample. Adsorption was performed using varying adsorbent masses of 5 g, 10 g, and 15 g, with contact times of 60 minutes, 90 minutes, and 120 minutes. The adsorbent and used cooking oil sample were homogenized by stirring at 200 rpm. After adsorption, the sample was allowed to settle before being filtered to separate the oil from the adsorbent. The adsorbent was then dried in an oven at 105°C for 1 hour, while the treated oil underwent titration analysis to measure the free fatty acid (FFA) content after adsorption.

2.6 Titration Test

A chemical titration method was used to measure the free fatty acid content in the used cooking oil samples before and after adsorption. In this test, 30 g of used cooking oil was prepared, followed by the addition of 5 drops of phenolphthalein indicator and 50 mL of preheated 95% ethanol. The mixture was then titrated with NaOH, and the volume required to change the solution's color to pink was recorded.

2.7 FTIR (Fourier Transform Infrared Spectroscopy) Analysis

FTIR analysis was conducted to identify the functional groups present in the Pangium edule shell-based activated carbon before and after adsorption. The FTIR results were presented in the form of a spectral wavelength range of 500–4000 cm^{-1}. The working principle of FTIR involves infrared energy being emitted from a source, passing through a slit to control the amount of energy received by the sample. The sample beam then enters the sample chamber and is either transmitted or reflected by the sample surface, depending on the characteristic energy absorption of the sample.

2.8 Organoleptic Test

The color and odor of the oil samples were evaluated through an organoleptic test using visual and olfactory senses. In this test, 10 panelists assessed the color and odor of the samples, referring to the standards set in SNI 7709:2019.

3. RESULTS AND DISCUSSION

3.1 Adsorbent Characterization Using FTIR

The functional group analysis reveals the presence of elements or synthesized compounds, as indicated by the FTIR wavenumbers, which represent the vibrational structure of the chemical constituents in the sample. Meanwhile, transmittance exhibits a linear correlation with the compositional variations of the sample [12]. The characterization of functional groups in the adsorbent before and after adsorption is presented in Figure 1.

FTIR analysis was conducted on activated carbon derived from Pangium edule shells that had been chemically activated using 1 N NaOH. This activation aimed to introduce polar functional groups and increase the adsorbent's surface area to enhance adsorption efficiency for removing free fatty acids (FFAs) from used cooking oil. The FTIR spectrum prior to adsorption displayed several distinct absorption peaks, notably:

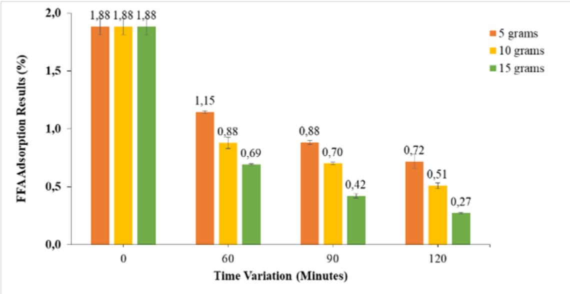


Figure 2. Adsorption Results of Free Fatty Acids.

hydroxyl (-OH) stretching at 3615–3861 cm^{-1}, indicating the presence of polar groups essential for adsorption; alkyne or nitrile groups (C≡C/C≡N) at 2109 cm^{-1}; carbonyl (C=O) stretching at 1699–1833 cm^{-1}; aromatic C=C at 1513 cm^{-1}; and C–O vibrations at 1125 cm^{-1}. According to [13][13], the presence of O–H, C–O, and C=C groups contributes significantly to the reduction of FFAs, supporting the potential of Pangium edule-based activated carbon in enhancing the adsorption process.

Following adsorption under optimal conditions (15 g adsorbent and 120 minutes of contact time), a notable reduction in the intensity of the -OH peak at around 3645 cm^{-1} was observed, suggesting that polar compounds such as FFAs had successfully interacted with and been adsorbed onto the activated carbon surface[14, 15, 16]. In addition, new peaks emerged: aliphatic C–H stretching at 2922 and 2847 cm^{-1}, ester C=O at 1736 cm^{-1}, and modified C–O vibrations at 1148 cm^{-1}. Aromatic C–H bending was also observed at 715 and 961 cm^{-1}. These shifts in functional groups indicate that various organic compounds in the oil, especially FFAs, were chemically adsorbed onto the adsorbent.

A previous study [17] reported similar findings, with the emergence of methylene C–H (2855 cm^{-1}) and ester C=O (1742 cm^{-1}) groups, associated with peroxides and adsorbed FFAs on biosorbents. The observed decrease in the intensity of hydroxyl (O–H), carbonyl (C=O), and aliphatic C–H functional groups further confirms strong adsorbate–adsorbent interactions. Moreover, a lower transmittance percentage is generally indicative of more effective adsorption due to the increased presence of surface-bound functional groups[18].

3.2 Initial Characteristics of Used Cooking Oil

The consumption of used cooking oil poses significant health risks, including cancer, the accumulation of Trans Fatty Acids (TFA) in blood vessels, and reduced fat and oil digestibility, which may impact cognitive function. Therefore, preliminary treatment is necessary to improve the quality of used cooking oil before further utilization. The initial characterization of used cooking oil samples is essential to assess key parameters before and after adsorption. The results of this analysis are presented in Table 1.

Table 1. Initial Characteristics of Used Cooking Oil

No	Information	Initial Characteristic	Quality Standards
1	Free Fatty Acid	1,88%	0,3%
2	Color	Dark Brown	Clear Yellow
3	Smell	Rancid	Normal

The used cooking oil samples utilized in this study had undergone approximately 5–7 frying cycles. The characterization process for determining the free fatty acid (FFA) parameter was conducted using titration. The initial analysis of the samples revealed an FFA content of 1.88%, which exceeds the quality standard limit set by SNI 7709:2019, which is 0.3%.

The free fatty acid content in cooking oil tends to increase with prolonged hydrolysis, a process accelerated by heat and moisture, leading to changes in color and odor. The darkening of cooking oil is primarily caused by repeated exposure to high temperatures, which induces oxidation and results in the formation of peroxides. These peroxides, which are inherently unstable, eventually decompose into aldehydes and ketones.

According to research [19], the initial free fatty acid (FFA) content was 0.45%, with a dark brown color and an intense, unpleasant odor, making it unsuitable for reuse or disposal into the environment. Consistent with this research, the initial FFA levels presented in Table 4.1 also indicate significant degradation, highlighting the need for further effective treatment to improve the quality of used cooking oil.

3.3 Free Fatty Acid Analysis After Adsorption

The treatment of used cooking oil samples in this study was conducted using activated carbon derived from Pangium edule shells, which had been chemically activated for the adsorption process. The adsorption experiments were performed using a batch method with varying adsorbent dosages of 5 g, 10 g, and 15 g, and adsorption durations of 60 minutes, 90 minutes, and 120 minutes. The results of the adsorption process are presented in Figure 2.

The adsorption process using activated carbon derived from Pangium edule shells, activated with NaOH, resulted in a significant reduction of free fatty acid (FFA) levels in used cooking oil. A batch adsorption approach was employed to optimize FFA reduction, utilizing Pangium edule shell-derived activated carbon with NaOH activation as the adsorbent. The experiment involved treating 200 mL of used cooking oil with varying adsorbent dosages and contact times, followed by titration to determine the most effective conditions for reducing FFA content.

For an adsorbent dosage of 5 grams, the FFA content decreased to 1.15% after 60 minutes, 0.88% after 90 minutes, and 0.72% after 120 minutes. With a 10-gram dosage, the FFA content reduced to 0.88% at 60 minutes, 0.70% at 90 minutes, and 0.51% at 120 minutes, approaching the quality standard of 0.3% set by SNI 7709:2019. These results indicate that Pangium edule shell-derived activated carbon effectively adsorbed FFA from used cooking oil.

The best adsorption performance was observed at a dosage of 15 grams, with FFA levels of 0.69% at 60 minutes, 0.42% at 90 minutes, and 0.27% at 120 minutes—falling below the regulatory threshold of 0.3%. This indicates that both adsorbent mass and contact time significantly influence adsorption efficiency. The results suggest that increasing adsorbent mass and extending contact time enhances FFA removal, improving used cooking oil quality. The most effective adsorption condition was 15 grams of activated carbon with a 120-minute contact time, achieving an FFA level of 0.27%, demonstrating the substantial impact of adsorption treatment in meeting quality standards.

Another researcher[20] conducted a similar experiment, achieving a reduction in free fatty acids (FFA) from 3.51% to 0.55% using a mass variation of 5.5 grams and a contact time of 80 minutes, resulting in an adsorption efficiency of

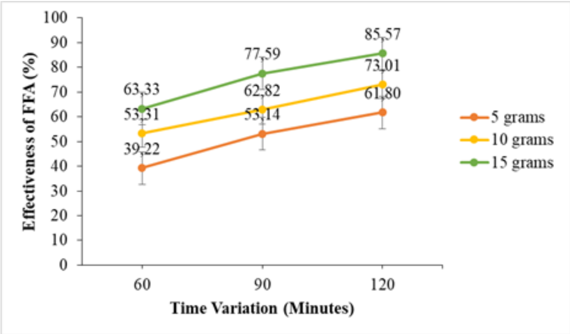


Figure 3. Effectiveness of Free Fatty Acid Adsorption.

84.15%. This indicates that natural adsorbents have potential for reducing FFA levels in used cooking oil. The reduction of FFA can be influenced by several factors, including the type of adsorbent, activator, mass addition, and adsorption time [21].

3.4 Organoleptic Analysis

The color and odor analysis of used cooking oil was conducted before and after adsorption. The organoleptic assessment followed the testing procedures outlined in SNI 7709:2019 for palm cooking oil. Observations of color and odor were performed on the oil samples both before and after treatment, involving a panel of 10 evaluators. The results of the color and odor analysis for used cooking oil are presented in Table 2.

Table 2. Results of Organoleptic Testing for Color and Odor

Organoleptic Test of Used Cooking Oil Samples											
Sample	Color										Smell
	1	2	3	4	5	6	7	8	9	10	
M0T0	A	A	A	A	A	A	A	A	A	A	A
M1T1	A	A	A	A	A	A	A	A	A	A	A
M1T2	A	A	A	A	A	A	A	A	A	A	A
M1T3	A	A	A	A	A	A	A	A	A	A	A
M2T1	A	A	A	A	A	A	A	A	A	A	A
M2T2	A	A	A	A	A	A	A	A	A	A	A
M2T3	A	A	A	A	A	A	A	A	A	A	A
M3T1	A	A	A	A	N	A	A	A	N	A	A
M3T2	A	N	N	N	A	N	N	N	A	N	A
M3T3	A	N	N	N	A	N	A	N	A	N	A

Description:
A = Abnormal (Dark brown until Black)
N = Normal (Yellow until Orange)

According to the Indonesian National Standard for palm cooking oil (SNI 7709:2019), "normal" cooking oil color is defined as pale yellow/clear to yellow-orange, which aligns with the standard requirements. Meanwhile, "abnormal" color is characterized by a brownish to dark brown-black appearance. Furthermore, cooking oil with a "normal" odor emits the typical aroma of cooking oil, while an "abnormal" odor is identified by a rancid or unpleasant smell.

Based on the presented data, the changes in color and odor of the cooking oil were not significantly different across the tested variations. The treatments using 5 grams and 10 grams of adsorbent did not exhibit substantial color improvement, remaining classified as abnormal. However, at 15 grams, a noticeable change occurred, with the oil appearing yellow, allowing it to be categorized as normal. Regarding the odor parameter, the adsorption process was not yet optimal in removing the compounds responsible for unpleasant smells. As a result, the undesirable odor persisted, indicating that the treatment was not entirely effective in eliminating unwanted aromas.

According to a previous study [22], the adsorption of used cooking oil resulted in a color approaching yellow; however, the odor level was still not effectively reduced. The use of activated charcoal as an adsorbent may have varying impacts on each targeted parameter. While the addition of activated charcoal improved the color quality, it did not show significant effectiveness in reducing odor in the treatment of used cooking oil.

3.5 Analysis of Adsorption Effectiveness

The effectiveness analysis of free fatty acid (FFA) reduction was conducted to determine the extent of adsorption achieved. This calculation aims to evaluate the adsorption efficiency for each sample variation tested. The obtained test results, recorded in tabular form, were further analyzed using Excel, applying the adsorption effectiveness percentage formula. The effectiveness values are presented in percentage form, ranging from 0% to 100%. The adsorption results for used cooking oil are illustrated in Figure 3.

Based on the calculation of the effectiveness of free fatty acid adsorption, each variation in adsorbent mass and contact time shows a parallel trend in effectiveness. Each variation exhibits an increasing effectiveness, indicating that the adsorption process yields relatively good results. The findings suggest that a higher adsorbent mass and a longer adsorption time result in a more optimal adsorption process.

In this study, the optimal adsorption condition was achiev -ed with a 15-gram adsorbent dose and a contact time of 120 minutes, yielding an effectiveness of 85.57%. According to the test data and calculations, the reduction in free fatty acid levels to 0.27% successfully meets the quality standard of <0.3%, as specified in SNI 7709:2019 (Palm Cooking Oil). Thus, activated carbon derived from kluwek shells exhibits strong interaction in adsorbing free fatty acid compounds from the oil sample. This interaction is attributed to the presence of functional groups within the adsorbent, which are capable of binding the carbon in free fatty acids, leading to their reduction.

4. CONCLUSION

The adsorption process using activated carbon from kluwek shells demonstrated effective removal of free fatty acids from used cooking oil. The presence of polar functional groups such as -OH, C=O, and aromatic C=C on the adsorbent surface played a crucial role in adsorption. Post-adsorption analysis showed a reduction in hydroxyl (-OH), carbonyl (C=O), C-H, and C-O functional group intensities, indicating strong adsorbate–adsorbent interactions and effective organic compound removal. The initial characteristics of used cooking oil included a free fatty acid (FFA) content of 1.88%, a dark brown color, and a strong rancid odor. Adsorption at an optimal condition of 15 grams of adsorbent for 120 minutes resulted in an 85.57% reduction in FFA, lowering the concentration to 0.27%, which meets the quality standard (<0.3%) set by SNI 7709:2019. While the oil color improved to pale yellow, the odor remained strong, indicating that adsorption effectively reduced FFA and improved color but was less effective in odor removal. Therefore, future research is recommended to explore the regeneration and reuse potential of the adsorbent, as well as to develop complementary treatment methods or combined processes that can further enhance the removal of undesirable odors from used cooking oil.

DATA AVAILABILITY STATEMENT

The datasets generated during and/or analyzed during the current study are available from the corresponding author on reasonable request.

CONFLICT OF INTEREST

The authors declare that there are no conflicts of interest.

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