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Free Fatty Acid Reduction Using Cacao (*Theobroma cacao L*) Bean Husk Charcoal In Used Cooking Oil

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Abstract: Used cooking oil if not well-maintained will give some problems to drainage system or surface water. Used cooking oil has the potential to be processed into energy. However, the high free fatty acid (FFA) as a result of the use of cooking oil makes the process into energy more difficult. Because the high free fatty acid content will cause the saponification reakction in making used cooking oil into energy. Cocoa bean husk charcoal (Theobroma cacao L) has a K_2CO_3 content that similar to its commercial product, and also contains some traces of silicates and sulfates so that it can increase the efficiency of free fatty acids removal in used cooking oil. In this study, tests were carried out to reduce FFA levels in used cooking oil using charcoal from cocoa bean husks activated with 10M of hydrochloric acid, varying the concentrations of 5, 7.5, and 10%-m/v, as well as the temperature treatment during the process of mixing used cooking oil with activated charcoal adsorbent, which was 75, 100, and 150°C. The best FFA reduction results were found in the C2T2 reactor which reached 75% of FFA removal, where the best concentration of cocoa bean husk charcoal as an adsorbent were 7.5%m/v and the temperature of the mixing process was 100°C which can remove FFA content from 2.72 to 0.64

Keywords: Cacao bean husk charcoal, Free fatty acid, Used cooking oil.

1. Introduction

Energy is a basic human need in carrying out daily activities, starting from food consumption, cooking, transportation, electricity, to running industry to process the necessary products [1]. This is proven by the energy demand in Indonesia in 2021 which was 60,452 petajoules [2]. So, in achieving these energy needs, the Indonesian government through the directorate general of the national energy council is making various efforts to meet energy needs, including planning and allocating energy in various sectors using 3 scenarios, namely BaU (Business as Usual), PB (Sustainable Development), and RK (Low Carbon) [3].

One of the efforts made by the Indonesian government is to increase the renewable energy mix, namely by creating B30 biodiesel. However, the production of B30 biodiesel often clashes with the community's main need, namely cooking oil which is made from the same raw material, namely crude palm oil (CPO). The use of raw materials from vegetable oils as raw materials for biodiesel competes with food needs, so research efforts are needed to make biodiesel from non-food materials [4]. The Palm Oil Farmers Union in Suara.com said that the scarcity of cooking oil occurred

because the raw material for cooking oil, namely palm oil, was used to make biodiesel in the government's B30 program [5].

In fact, there are still around 3.46-6.72 million KL of used cooking oil that remains unmanaged and has the potential to be utilized as a raw material for biodiesel production [6]. However, current government policies tend to prioritize biodiesel production from virgin feedstocks such as fresh cooking oil to achieve higher quality fuel, which has contributed to cooking oil scarcity and price increases in 2021 [7]. Therefore, the utilization of waste cooking oil as an alternative feedstock is both environmentally and economically important.

Alternative biodiesel processing using waste has been widely used, especially using waste as raw material. Various studies have explored biodiesel production from waste cooking oil using different approaches. For instance, transesterification using a heterogeneous base catalyst made from egg shells of 15% by mass, a molar mass ratio of methanol:oil of 18:1, with microwave radiation for 4 minutes producing a biodiesel yield of 96.7% [8]. In addition, activated carbon derived from cocoa bean husks has been shown to effectively reduce free fatty acid (FFA) levels by 66.7–86.7% and produce biodiesel that meets SNI and ASTM standards [9]. Similarly, the use of a heterogeneous potassium oxide (K_2O) catalyst derived from cocoa shell waste demonstrated a reduction in FFA from 3.13% to 0.82% (73.80% reduction) under pre-treatment conditions of 1% (w/w) activated carbon at 100°C for 80 minutes. Optimal biodiesel production was achieved with 6% (w/w) catalyst calcined at 650°C, a reaction time of 180 minutes, a methanol-to-oil molar ratio of 12:1, and a temperature of 60–65°C, resulting in a biodiesel purity of 99.58% [10].

Despite these promising results, the research gap lies in the lack of systematic optimization of key parameters, particularly the adsorbent concentration and activation temperature of cocoa shell-derived materials, which significantly influence FFA reduction efficiency and overall biodiesel yield. Previous studies have demonstrated the effectiveness of cocoa shell-based adsorbents; however, they have not comprehensively investigated the interaction between activation conditions and adsorbent dosage. Therefore, this study aims to address this gap by focusing on the optimization of adsorbent concentration and activation temperature of cocoa shell-derived activated carbon, in order to maximize FFA reduction and improve biodiesel yield. This approach represents the novelty of the research, as it provides a more in-depth evaluation of process parameters that have not been thoroughly explored in previous studies.

2. Research Method

2.1 Sample Preparation

The raw material for used cooking oil is obtained from several fried food traders and cracker sellers that have different qualities of used cooking oil. Therefore, the used cooking oil obtained must be homogenized so that the quality of the used cooking oil used in the research is of the same quality. The homogenization method used is by mixing the used cooking oil and collecting it in the same large container (sufficient to hold ± 1.5 liters) and stirring until evenly mixed. After stirring, the undissolved solids in the used cooking oil are filtered using filter paper so as not to damage the quality of the used cooking oil when it is processed into biodiesel.

The raw material needed for used cooking oil is 1.5 liters because it has to meet the 50 ml requirement for 27 reactors, so 1,350 ml of used cooking oil is needed while the remaining 150 ml will be used as a sample to be tested for quality. After that, the used cooking oil is heated in an oven at 105-110°C for ± 1 hour to remove the water content because the presence of water will reduce the water quality. Water and oil can react and form free fatty acids [11]. Used cooking oil that has had its water content removed is then placed in a desiccator for 15 minutes so that the cooling process can take place without increasing the water content of the used cooking oil sample. After that, some samples were taken to test their quality, such as moisture and volatile matter, insoluble impurities, iodine value, free fatty acids and density of used cooking oil. The complete process of preparing the used cooking oil can be seen in **Figure 1.**

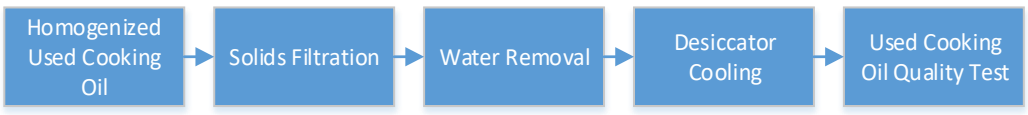

Figure 1. Used Cooking Oil Preparation

Figure 2. Take 1.5 liters used cooking oil to homogenized in flask.

Figure 3. Homogenized 1.5 liters used cooking oil with magnetic stirrer on 500 rpm

Figure 4. Water content removal using oven at 105-110°C for ± 1 hour.

Figure 5. Insoluble impurities removal using filter paper.

Figure 6. Free Fatty Acid measurement of used cooking oil.

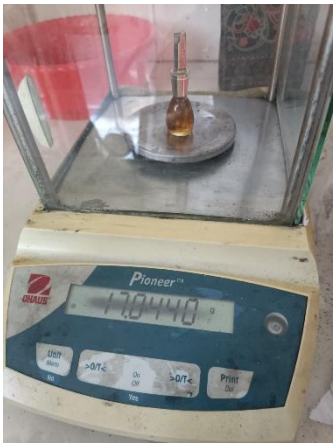

Figure 7. Density measurement using pycnometer method.

2.2 The Making of Adsorbent

About 50 kg of cocoa bean husks purchased from the Coffee and Cocoa Research Center (Pusat Penelitian Kopi dan Kakao), Jember Regency were first dried in the sun for 3-4 hours. During the drying process with the help of sunlight it only shrank to 45 kg or around 10%. The dried seed coats are then roasted using the slow pyrolysis method at a temperature of 300°C for ± 6 hours using the conventional method with the help of a simple biomass stove. During the pyrolysis process, cocoa bean shell biomass waste was reduced by 33% to 30.15 kg. The charcoal is then crushed with a mortar and pestle until it is quite fine. Then the finely ground charcoal is sieved using a 40 mesh and 60 mesh sieve.

The charcoal grains that pass through the 40 mesh sieve and are retained on the 60 mesh sieve are then used as charcoal to be activated. The activation process is carried out by soaking 150 grams of activated charcoal in 1 liter of 10M HCl then stirring at a speed of 500 rpm (scale 4 on the IKA © C-MAG HS 10 magnetic stirrer) at a temperature of 100°C for 1 hour. After that, the charcoal is separated from the HCl solution and neutralized by washing with distilled water until pH = 7. The charcoal which already has a pH of 7 is then placed in the oven at a temperature of 110°C for 3 hours (until dry) and after that it is cooled in a desiccator for ± 15 minute. The complete process of adsorbent making can be seen in **Figure 8.**

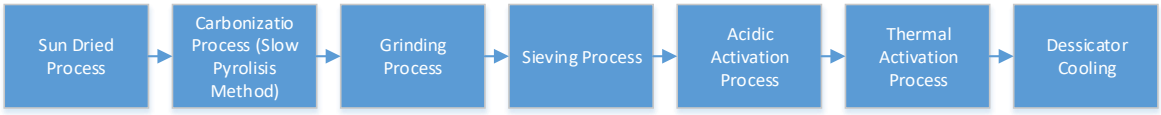

Figure 8. Adsorben Making Process

Figure 9. Carbonization process with slow pyrolysis at 300°C for 3-4 hours using biomass stove.

Figure 10. Grinding process of cocoa bean husks charcoal using mortar and pestle.

Figure 11. Sieving process of fine cocoa bean charcoal using 40, 60, and 100 mesh.

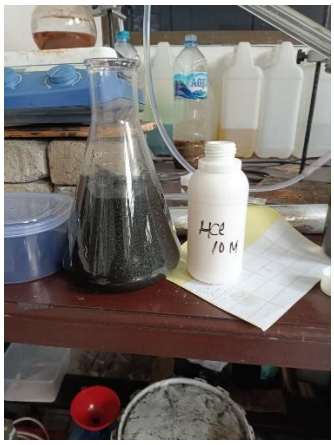

Figure 12. Soaking cocoa bean shell charcoal with 1 liter 10 M HCl.

Figure 13. Mix, stir, and heat cocoa bean shell charcoal + HCl on a hot plate.

Figure 14. Heat the cocoa bean husks charcoal in the oven at 110°C for ± 3 hours.

2.3 Adsorbtion Process

The adsorption process was conducted by adding 5%, 7.5%, and 10% (w/w) activated cocoa husk charcoal into 50 mL of used cooking oil according to the reactor division in **Table 1**. The selected adsorbent concentrations were based on previous studies reporting effective FFA reduction within the 1–10% (w/w) range, indicating improved adsorption performance with increasing dosage until equilibrium is reached [9]. The mixture was stirred at 500 rpm for 90 minutes under temperature variations as shown in **Table 1**. The selected temperature range was adopted from prior research demonstrating that moderate temperatures (60–100°C) enhance adsorption kinetics and mass transfer without causing thermal degradation of the oil [10]. After stirring, the used cooking oil is then separated from the cocoa shell charcoal using filter paper and then tested for FFA levels using the AOCS Ca 5a-40 1989 test method [12], and density with test standard AOAC 920.212 [13] and AOAC 920.213 [14].

Table 1. Treatment Variations and Reactor Naming

Adsorption Process Temperature (T)	Activated Charcoal Adding Concentration (C)		
	C1 (5%)	C2 (7,5%)	C3 (10%)
T1 (75°C)	C1T1	C2T1	C3T1
T2 (100°C)	C1T2	C2T2	C3T2
T3 (125°C)	C1T3	C2T3	C3T3

Figure 15. Taking 50 ml of used cooking oil for the FFA level reduction test process.

Figure 16. The process of adsorption of FFA levels using cocoa bean husk by heating and stirring.

Figure 17. The process of separating adsorbents from used cooking oil.

Figure 18. Used cooking oil results from the adsorption process.

Figure 19. The FFA level test of used cooking oil after the adsorption process.

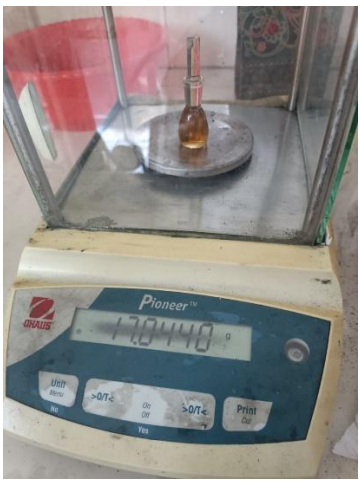

Figure 20. The density test of used cooking oil after the adsorption process.

2.4 Experimental Design dnd Statistical Analysis

This study employed a completely randomized design (CRD) with two main factors: adsorbent concentration (5%, 7.5%, and 10% w/w) and adsorption temperature (as presented in Table 1). Each experimental condition was conducted in triplicate to ensure data reliability and reproducibility.

The experimental data were statistically analyzed using Analysis of Variance (ANOVA) to evaluate the significance of the effects of adsorbent concentration and temperature on free fatty acid (FFA) reduction and oil density. ANOVA was selected because it is a robust statistical method for comparing the means of multiple treatment groups

and determining whether observed differences are statistically significant. It is widely used in experimental studies involving multiple factors and levels, as it minimizes Type I error compared to multiple pairwise comparisons [15], [16].

When significant differences were identified by ANOVA ($p < 0.05$), Duncan's Multiple Range Test (DMRT) was applied as a post hoc analysis to compare treatment means. DMRT was chosen due to its effectiveness in detecting differences among group means while maintaining a balance between Type I and Type II errors. It is commonly used in agricultural, chemical, and environmental studies for multiple comparison procedures, particularly when treatments are relatively homogeneous [17], [18].

3. Result

3.1 Quality of Used Cooking Oil

The used cooking oil that used in this research was obtained from the fried food traders and cracker sellers in Jember Regency, where the total used cooking oil collected was around ± 1.5 liters in accordance with the required number of variations and repetitions (9 sample variations and 3 experimental repetitions, @ 50 milliliters). This cooking oil is used more than 4 times for frying where there is a change in the color of the oil, so that you first know the characteristics of the used cooking oil starting from the FFA content, density and acid number. Table 2 is test data on the characterization of homogenized used cooking oil.

Tabel 2. The Quality of Used Cooking Oil

Quality parameters	Units	Value	Standard test method
Free Fatty Acid	%	2.72	AOCS Ca 5a-40 [12]
Density	g/cm^3	0.916	AOAC 920.212 [13], AOAC 920.213 [14]
Moisture and volatile matter	%	2.6	AOCS Ca 2c-25 [15]
Insoluble impurities	%	1.2	AOCS Ca 3a-46 [16]
Iodine value	g I_2/100g	15	AOCS Tg 1-64 [17]

3.2 The Free Fatty Acid Removal

The cause of the increase in FFA levels in used cooking oil is due to the oxidation and hydrolysis processes combined with neutral fats [11]. The oxidation process can take place when there is contact between a certain amount of oxygen and oil or fat [18]. The occurrence of this oxidation reaction will result in a rancid odor in oils and fats. Oxidation usually begins with the formation of peroxides and hydroperoxides, this causes FFA levels to increase by more than 2% which will inhibit the transesterification process. Data for measuring FFA levels in used cooking oil were tested using the AOCS-5a-40 method (American Oil Chemists Society, 1997). Data from FFA measurements with 3 repetitions resulted in an average decrease in FFA in Table 3.

Table 3. Data on the Results of Reducing FFA Levels for Each Variation

Variation	Initial FFA Level (%)	Final FFA Level (%)	FFA Removal (%)
C1T1		0.77	71.82
C2T1		0.67	75.49
C3T1		0.65	76.10
C1T2		0.7	74.37
C2T2	2.72	0.64	76.41
C3T2		0.67	75.49
C1T3		0.75	72.43
C2T3		0.73	73.04
C3T3		0.65	76.10

4. Discussion

Based on the test results in the laboratory and continued with the analysis of variance (ANOVA) test and further Duncan multiple range test (DMRT), it was found that the C2T2 reactor was the best variation in reducing FFA levels. Used cooking oil which underwent an adsorption process with the addition of 7.5% m/v adsorbent and the adsorption process at a temperature of 100°C in the C2T2 reactor FFA content was able to be reduced to 76.41%. There were increases and decreases in sample treatment data regarding FFA levels, due to the different interaction results for each treatment. A graphic comparison of FFA levels based on the interaction between temperature and acid concentration is also presented in **Figure 21.**

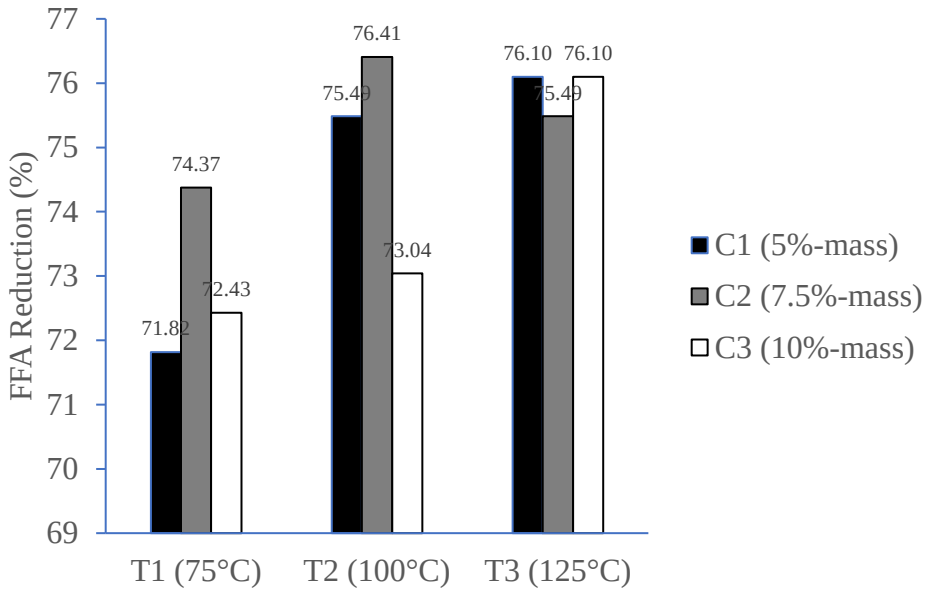

Figure 21. Reduction of FFA Levels (%) Based on Adsorbent Concentration and Temperature

The reduction of FFA levels in this study was strongly influenced by both adsorbent concentration and adsorption temperature. Increasing the concentration of activated cocoa shell charcoal improved FFA removal efficiency, as shown by reactor C1 (5% w/v), which achieved an average reduction of 72%, compared to reactor C2 (7.5% w/v) with 75%, and reactor C3 (10% w/v) with 76%. This trend indicates that higher adsorbent dosage provides more active sites for adsorption, thereby enhancing FFA removal. These results are consistent with previous studies, which reported FFA reduction efficiencies in the range of 66.7–86.7% using cocoa shell-based activated carbon [9], and up to 73.80% reduction under similar adsorption conditions [10]. Compared to these studies, the present work demonstrates comparable performance, particularly at higher adsorbent concentrations, confirming the effectiveness of cocoa shell-derived adsorbents.

The activation of charcoal using HCl plays an important role in enhancing adsorption performance. As a strong acid, HCl facilitates the removal of impurities and opens the pore structure of the adsorbent, thereby increasing surface area and adsorption capacity [19]. This improved pore structure enhances the interaction between the adsorbent and FFA molecules in used cooking oil.

Temperature also significantly affects the adsorption process. An increase in temperature generally enhances adsorption capacity and rate due to improved mass transfer and stronger interactions between adsorbent active sites and adsorbate molecules [20], [25]. In this study, higher temperatures increased FFA removal efficiency up to an optimum condition. However, at 125°C, the FFA reduction percentage decreased, indicating that excessive temperature negatively affects the process. This phenomenon can be attributed to thermal degradation and oxidation of the oil, which leads to the formation of additional free fatty acids and oxidation products such as aldehydes and ketones [26], [27]. These findings are in agreement with previous studies reporting that temperatures above 100°C may accelerate oil degradation and reduce overall adsorption effectiveness.

However, at a temperature of 125°C the percentage reduction in FFA levels actually decreased, this shows that the adsorption temperature has an effect on FFA levels. The results of the adsorption process that have been absorbed or bound by the cocoa bean shell charcoal adsorbent undergo a dissolution reaction. Temperatures above 100°C heating used cooking oil will cause damage to the used cooking oil and increase the levels of free fatty acids [22]. A temperature of 125°C also allows the oxidation reaction of used cooking oil to occur. The oxidation reaction in oil produces organic

compounds, namely aldehydes and ketones. This compound will cause rancidity in used cooking oil, apart from that, other consequences caused by the oxidation process are color changes, decreased vitamin content, and can result in poisoning [23].

Despite these promising results, several limitations should be acknowledged. This study did not include detailed characterization of the adsorbent, such as surface area analysis (BET), pore size distribution, or surface morphology (SEM), which are important parameters for understanding adsorption mechanisms. In addition, the regeneration and reusability of the adsorbent were not evaluated, limiting the assessment of its economic and practical feasibility.

Therefore, future research should focus on comprehensive characterization of cocoa shell-derived activated carbon using techniques such as BET and SEM to better understand its physicochemical properties. Furthermore, studies on adsorbent regeneration and reuse are necessary to evaluate long-term performance and cost efficiency. Scaling up the process to a pilot-scale biodiesel production system is also recommended to assess its industrial applicability.

5. Conclussion

The results of this study indicate that the optimal condition for reducing free fatty acid (FFA) levels was achieved in the C2T2 variation, with a reduction of up to 76.41%, using 7.5% (w/v) activated cocoa bean shell charcoal at an adsorption temperature of 100°C. These findings demonstrate the effectiveness of cocoa shell-derived activated carbon as a low-cost and environmentally friendly adsorbent for improving the quality of used cooking oil. This study contributes to the valorization of agricultural waste, particularly cocoa bean shells, as a functional material in biodiesel pre-treatment processes. Furthermore, this method has strong potential to be developed as a cost-effective and sustainable pretreatment technology for biodiesel production from used cooking oil, thereby enhancing waste utilization and supporting circular economy practices.

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