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THE USE OF SPENT BLEACHING EARTH (SBE) AS AN ADSORBENT TO REDUCE FREE FATTY ACIDS IN WASTE COOKING OIL

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ABSTRACT

High free fatty acids in waste cooking oil can interfere with the re-use of the cooking oil. Therefore, the absorption technique is one way that can reduce the free fatty acids contained in waste cooking oil. Aim: The purpose of this study is to find out the ability of Reactivated Spent Bleaching Earth (RSBE) to adsorb free fatty acids contained in waste cooking oil to be biodiesel products. In this study, waste cooking oil was collected from fried food sellers in Bekasi, while spent bleaching earth (SBE)-one of the solid wastes produced by refinery oil palm industry as an adsorbent-was from the palm oil industry PT X. For Reactivated Spent Bleaching Earth (RSBE), the redundant used 0.7 M HNO3. To obtain the optimum products, The redundant applied the variation of time (i.e., 30, 45, 60, 75, and 90 minutes) and concentration (6%, 9%, 12%, 15%, and 18%). Methodology and results: Results indicated that the time variation of temperature 90 minutes were the maximum and the concentration of 15% were the optimal combination to reduce free fatty acids 0.65%. Conclusion, significance, and impact of study: The values of water and ash contents of RSBE also meet the quality standard as activated carbon and can be used as an adsorbent for the purification of waste cooking oil. The oil content in SBE is less than 3% and is considered not to be a toxic hazardous waste. Therefore, it can be reused without pre-treatment. In addition, the redundant found that there were changes pore on the surface of the SBE, both activated and not activated. To conclude, SBE can be used as an absorbent to absorb the fatty acids in waste cooking oil.

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- Spent Bleaching Earth
- Waste Cooking oil

1. INTRODUCTION

The total area of oil palm plantations in Indonesia in 2019 reached 14,456,611 hectares. In addition, Indonesia was able to export palm oil with a total volume of 28,279,350 tons. The high value of exports and product demands also increases waste generated. One of the steps in dealing with the accumulation of waste is that all palm oil entrepreneurs apply the principle of sustainability in their business activities through the program of Indonesian Sustainability Palm Oil (ISPO) (Arifandy *et al.*, 2021). Biodiversity, environmental management, and natural resources as waste utilization criteria are some of the principles contained by ISPO (Schoneveld *et al.*, 2019).

Spent Bleaching Earth (SBE) is solid waste like sand that has color. Based on Government Regulation No. 22/2021 regarding the implementation of environmental protection and management, SBE is included in 8 types of waste that are excluded from toxic hazardous waste. Attachment IX of the regulation explains that SBE with waste code B413 can be used in oleochemical industrial processes and/or animal or vegetable oil processing with an oil content of less than 3%. SBE is categorized as waste that requires management through 3R activities, namely recovery, reuse, and recycle (Paspi, 2020). According to Government Regulation No. 22/2021, determining whether the waste is included in the toxic hazardous waste category can be carried out through TCLP testing to find out the heavy metals contained in it.

Palm oil waste is the remains of oil palm plantations that are not included in the main product or are a by-product of the palm oil processing process, either in the form of solid waste or liquid waste. Palm oil waste generally contains high organic matter which can harm the environment. The more production is carried out, the more waste is generated by the palm oil industry. In addition, there are several types of waste generated by the palm oil industry in the 2019-2020 period, as seen in Table 1.

Table 1 Quantity of palm oil industrial waste from PT. X in 2019-2020

No	Type of Waste	Amount
1.	Spent Bleaching Earth (SPE)	5,962,489 Ton
2.	Bottom Ash	664,460 Ton
3.	Fly Ash	1,436,310 Ton
4.	Sludge from Wastewater Treatment Plant (WWTP)	99,300 Ton

SBE can be regenerated for reuse to reduce waste problems. SBE regeneration can be carried out by thermal, chemical, and physical activation methods. In this study, SBE had a low adsorption capacity. However, if SBE was regenerated by heating and adding materials, the adsorption power would increase. The regeneration process can be carried out by several methods, including heating and recycling processes physically we can activate SBE by the heating method and also chemically we can perform the same by using activators. The activation process in acidification is usually carried out with an acid solution. The acidic nature of SBE can form an acid center that functions as the active site of the adsorbent.

The process of reactivation of SBE is carried out by restoring its absorption ability. To increase the adsorption power of SBE, an acid solution can be used to change the structure of the compound in montmorillonite and to dissolve magnesium, aluminum, and iron ions from the octahedral layer, causing the porosity on the surface of the material. After that, the interlayer cleavage process can occur to open the previously inaccessible parts of the mineral particle structure (Almeida *et al.*, 2019; Hussin *et al.*, 2011; Pickard, 2005; Simone M. Silva *et al.*, 2014).

Waste cooking oil is waste from vegetable cooking oil that comes from industrial and household needs. In its creation process, the good and new cooking oil is used for culinary purposes more than 3-4 times at high temperatures (160-180°C). At the time when waste cooking oil had contact with air or water in the heating process can cause a decrease in the quality of the oil so that it can no longer be used for frying and must be discarded (Sulung, 2019).

Cooking oil used repeatedly will affect the nutritional value of fried foods and the quality of the oil. The good cooking oil must be odorless and neutral in taste. The higher the glycerol content is, the lower the smoke point will be. It means that the oil smokes faster. Likewise, the higher the smoke point is, the better the quality of the oil will be. The main damage to the oil is the emergence of a rancid taste and smell. In addition, other damages include an increase in free fatty acids, the rise of peroxide value, and changes in the color to be darker (Kusumastuti, 2004; Wijana *et al.*, 2005).

The use of waste cooking oil can be found in several restaurants, household kitchens, and cadger. The high fatty acids contained in waste cooking oil can speed up some unwanted

reactions. Therefore, the use of waste cooking oil should be pretreated to reduce free fatty acids by conducting adsorption using SBE.

2. RESEARCH METHODOLOGY

2.1 Preparation Stage

This study was conducted from December 12, 2020, to August 27, 2021, at the Environmental Laboratory, Department of Environmental Engineering, Campus A, Universitas Trisakti, Grogol, West Jakarta. The sample of SBE waste was collected from PT. X, which is located in North Jakarta. Meanwhile, the sample of the waste cooking oil was obtained from one of the fried food sellers in Rawalumbu, Bekasi.

2.2 Implementation Stage

The research implementation stage began from the preparation of raw materials to the analysis and determination of research results. The stages of this research can be seen in the diagram below (Figure 1).

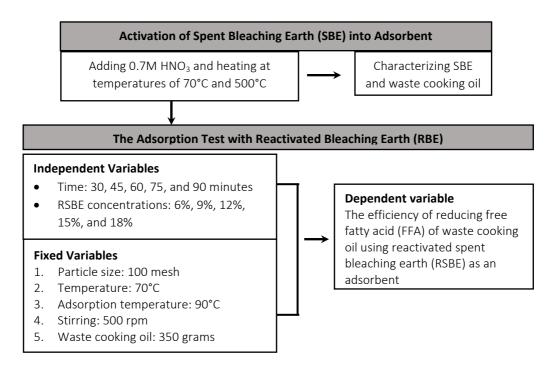


Figure 1 The flowchart of research stages

The SBE reactivation process was carried out with the addition of HNO₃ acid and SBE in a ratio of 1:10 (mg/L). Referring to a study conducted by Fajrudin (2016), the researchers carried out sieving with mesh 100, heating at 70°C for 1 hour while stirring at 300 rpm, and then reheating at a temperature of 105°C for 24 hours. During the adsorption test with Reactivated Spent Bleaching Earth (RSBE), the researchers recorded RSBE particle size (mesh), stirring time (minutes), RSBE concentration (% w/w), and adsorption temperature (°C). The RSBE and SBE samples were respectively tested for topographic analysis using SEM (Scanning Electron Microscope).

The optimization of stirring time in this study was 30, 45, 60, 75, and 90 minutes. Waste cooking oil was put as much as 350 grams into a 500 ml Erlenmeyer series, added with RSBE as much as 12% (w/w), and heated at a temperature of 70°C. After that, each sample was tested for water content and free fatty acids at the Environmental Laboratory of Universitas Trisakti, Jakarta.

In the RSBE concentration optimization test, 350 grams of waste cooking oil was filtered with filter paper and added adsorbents (RSBE) as much as 6%, 9%, 12%, 15%, and 18% (%w/w). After that, the researchers stirred the mixture of waste cooking oil and SBE at a speed of 500 rpm with a temperature of 90°C. The test samples for each variation were analyzed for water content and free fatty acids.

2.3 Data Analysis

The characteristic parameters of SBE tested in this study were water content, ash content, and oil content. The quality test of waste cooking oil was carried out by measuring the water content and free fatty acids according to SNI 01-3741-2013. The results of the adsorption test of waste cooking oil and activated SBE were calculated using the titration method to determine the percentage of free fatty acids that were adsorbed. In addition, its adsorption isotherm was also calculated using the Freundlich and Langmuir methods. The following are parameters, methods, tools, and materials used in this study.

2.3.1 The Characterization Test

The tools and materials used in characterization test for SBE from PT. X and waste cooking oil fried food sellers in Rawalumbu, Bekasi according to the stages of research, can be seen in Table 2 and 3.

No	Types of Analysis	Method	Tools	Materials
1.	Water content	Based on ASTM-1959	Cup, Oven, Analytical balance	SBE
2.	Ash content	Based on ASTM-1970	Cup, Furnace, Analytical balance	SBE
3.	Oil content	Based on EPA 9071 B	Soxhlet, Analytical balance, Vacuum pump, Furnace, Water bath, Erlenmeyer	SBE, Filter paper, pH paper

Table 2 Tools and materials in characterization test for SBE from PT.	. Х
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Table 3 Tools and materials in characterization test forwaste cooking oil fried food sellers in Rawalumbu, Bekasi

No	Types of Analysis	Method	Tools	Materials
1.	Water content	Based on SNI 01-3741-2013	Cup, Oven, Analytical balance	Waste cooking oil
2.	Free fatty acids	Based on SNI 01-3741-2013	Erlenmeyer, Oven, Analytical balance	Methanol, Waste cooking oil, 0.013 N NaOH, Phenolphthalein,

2.3.2 The Adsorption Test with RSBE

The tools and materials used in the adsorption test SBE from PT. X which has been activated with $HNO_3 0.7$ M becomes Reactivated SBE (RSBE) according to the stages of research, can be seen in Table 4.

No	Types of Analysis	Method	Tools	Materials
1	Making of Reactivated Spent Bleaching Earth (RSBE)	Activation, acidification, and titration	Sieve, Furnace, Oven, Analytical balance, UV-vis spectrophotometer	Distilled Water/Aquadest, HNO ₃ 5%, Filter paper, SBE, Waste cooking oil
2	The topography test on RSBE surface	SEM (Scanning Electron Microscope)	SEM with Bruker brand	SBE, RSBE
3	The adsorption test	Filtering	Cup, Erlenmeyer Cap, Erlenmeyer, Stirrer, Pipette 10 ml, Burette Pipette 25 ml, Analytical balance, Oven, Spatula, Filter paper	Waste cooking oil, RSBE

Table 4 Tools and materials in the adsorption test with SBE from PT. X which has been activated with $HNO_3 0.7$ m becomes reactivated SBE (RSBE)

3. RESULTS AND DISCUSSION

3.1 Characteristics of Waste Cooking Oil

The quality of wasted cooking oil that will be used from a fried food store in Rawalumbu, Bekasi with the use of more than 5 frying. Characteristics of jelantah oil can be seen in Table 5.

No.	Parameters	Unit	Results of Analysis	Cooking Oil Standard*
1	Free Fatty Acid Content	%	1.06	Max 0.3
2	Acid Number	mgKOH/g	2.33	Max 0.6
3	Water content	%	1.6	Max 0.1

Table 5 Initial characteristics of waste cooking oil

Note: *) SNI 3741:2013 regarding cooking oil

Waste cooking oil contains a water content of 1.6%. The water content is a determinant of the damage in waste cooking oil because the high value of water content can undergo a hydrolysis process at the stage of oil purification. The high value of water content in waste cooking oil is caused by fried food ingredients and the humidity of the air during being stored (Sulistijowati, 2013). Water in waste cooking oil can undergo hydrolysis, resulting in fatty acids (Eva, 2017). When considering SNI 3741:2013 regarding cooking oil, the water content found in this study is greater than the quality standard. Therefore, it has been damaged and is not suitable for use so it can be called waste cooking oil or used cooking oil. If this waste cooking oil is reused for food consumption, it can damage the health of those who consume it.

The quality of cooking oil is also determined by the fatty acid components and their constituents. Therefore, the researchers used the alkalimeter method to minimize errors and obtain accurate data in the titration process. Examples of errors are tools that are not clean and human error. The value of free fatty acids of waste cooking oil in this study was 1.06%. For this reason, the waste cooking oil must be treated to reduce free fatty acids.

3.2 Characteristic of SBE

The characterization test SBE aims to find out how much water content, ash content, oil content contained in spent bleaching earth before reactivating to be further mandated as adsorben in the process of refining wasted cooking. Characteristic SBE can be seen in Table 6.

No.	Characteristics	Score		SNI
		SBE	RSBE	
1	Water content (%)	2.32	1.39	Max 15%*
2	Ash content (%)	71.515	61.943	Max 10%*
3	Oil content (%)	0.033	-	Max 15% **

Table 6 Characteristics of SBE

Note: *SNI No. 06-3730-1995, **Minister of Environment and Forestry Regulation No. 6/2021

The characteristic values of Spent Bleaching Earth (SBE) are 2.32% for water content and 71.515% for ash content, while the characteristic values of Reactivate Spent Bleaching Earth (RSBE) are 1.39% for water content and 61.943% for ash content. Meanwhile, the characteristic values of waste cooking oil are 1.06% for free fatty acid and 1.6% for water content.

The results of the proximate analysis indicate that SBE has a water content of 2.32%. Low water content in SBE is due to the presence of oil in the pores of SBE. In a study conducted by

Muslich (2020), the obtained water content was 1%. In his study, the SBE used was stored for 3 months. However, the storage time of SBE did not affect because SBE contains oil, thereby reducing the ability of SBE to absorb surrounding water molecules. After conducting the Reactivation of SBE (RSBE), the water content decreased to 1.39%. This is caused by the heat during the activation process so that water trapped in the sample disappears with time (Aulia, 2018). The water content is related to the ability of SBE to absorb impurities. The lower the water content contained in the SBE is, the wider the pores on the surface will be, increasing its adsorption ability.

The ash content produced by SBE has a value of 71.515%. When compared with SNI No. 06-3730-1995 regarding activated carbon with a maximum value of 10%, the SBE in this study has a higher value than what has been predetermined. The SBE cannot be burned because, according to Manik (2010), the montmorillonite minerals contain high silica. However, the ash content of RSBE has a lower value than SBE, which is 61.943%. This is because RSBE the mineral residues are also reduced due to being carried away in the reactivation process using acid and removed by the washing process (Latisya, 2018). When compared with SNI No. 06-3730-1995, the water and ash content of RSBE meets the quality standard as activated carbon and can be used as an adsorbent for the purification of waste cooking oil.

The oil content contained in SBE is the residual oil remaining in the bleaching process. According to the Minister of the Environment Regulation and Forestry No. 6/2021 concerning technical approvals in the field of toxic hazardous waste management, waste disposal activities are carried out by testing TCLP, TPH, and heavy metal concentrations. The results of the TPH test indicate that the value of oil content in SBE is 0.03%. Based on a study conducted by Siami *et al.*, (2021), SBE in this study is categorized in the TCLP A with a value metal concentration contained in SBE is mercury (Hg) (< 0.0005 mg/l), silver (Ag) (< 0.2 mg/l), arsenic (As) (< 0.02 mg/l), barium (Ba) (1m46 mg/l), cadmium (Cd) (< 0.02 mg/l), copper (Cu) (< 0.02 mg/l), tin (Sn) (< 0.02 mg/l), nickel (Ni) (< 0.02 mg/l), and zinc (Zn) (0.08 mg/l). When compared with the Minister of Environment Regulation and Forestry No. 6/2021, the value of oil and metal content contained in SBE is less than the applicable quality standard. Furthermore, according to Government Regulation (PP) No. 22/2021, the oil content in SBE which is less than 3% is considered not to toxic hazardous waste. Therefore, SBE in this study is included in non-toxic hazardous waste and can be reused without pre-treatment.

3.3 Morphological Analysis Using SEM

SEM is an electron microscope utilized to describe the surface shape of the material being analyzed. According to Zunifer (2020), the size of the surface area of the adsorbent can affect the absorption of impurities and other compounds. In other words, the larger the surface area is, the more substances are adsorbed will be. In this study, the researchers carried out 1000x magnification on SBE which had been sifted with a sieve of mesh 100, either SBE that had not been activated or SBE that had been activated. Compounds were most clearly visible at 1000x magnification.

In the magnification, we found that SBE that had not been activated had closed pores. This is possible because SBE has undergone the Crude Palm Oil (CPO) bleaching process so that impurities are trapped in the pores. Meanwhile, SBE that had been activated showed voids on the surface but impurities were still visible. According to Latisya (2018), the reactivation process in SBE is there's not too much change in the surface because the contact between acid and SBE does not occur properly. In other words, the sample is insoluble and miscible with acid. Moreover, impurities that stick to the surface and close the pores on RSBE cannot be completely removed. This study proves that there is a change in the surface of the SBE that has not been activated and SBE that has been activated even though there's not too much change. However, SBE can be used as an adsorbent to adsorb free fatty acids contained in waste cooking oil because the larger pores allow for better adsorption compared to those that have not been activated.

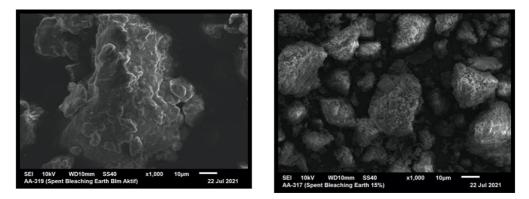


Figure 2 a) 1000x magnification of SBE that has not been activated, b) 1000x magnification of SBE that has been activated with 0.7 M HNO_3

3.4 The Effect of Adsorption Time on Free Fatty Acid (FFA) Content

In this study spent bleaching earth that was activated applied to waste cooking oil with variations of time 30, 45, 60, 75, and 90 minutes. Fatty acid analysis levels can be seen in Table 8.

Variation of adsorption time (minutes)	Initial fatty acid content (%)	Fatty acid content (%)
30	1.06	0.906
45	1.06	0.867
60	1.06	0.818
75	1.06	0.816
90	1.06	0.690

Table 8 Results of the analysis of free fatty acids against time

Data presented in Figure 3 the longer the contact time is, the more the adsorbate will be adsorbed because more will stick to the pores of SBE.

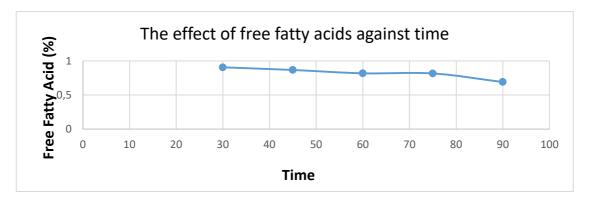


Figure 3 Graph of the effect of free fatty acids against time

From the graph, it can be seen that waste cooking oil, before treatment, had a free fatty acid value of 1.06%. In the first 30 minutes, the amount of free fatty acid content produced was 0.906%. At minutes 45, 60, and 75 minutes, the levels of free fatty acids were 0.867%, 0.818%, and 0.816%, respectively. Furthermore, at minute 90, the free fatty acid content was 0.690%. In this study, it was proven that 90 minutes generated the lowest free fatty acids. Therefore, it could be set as the maximum time. This occurs because the longer the adsorption time is, the longer the contact between the adsorbent and waste cooking oil will be in binding dirt into the

pores of the adsorbent. However, there will be a decrease in absorption if the adsorbent condition is saturated (Hasyim, 2019).

Based on a study conducted by Adhani (2016) examining the adsorption time of 30,45, 60, 75, and 90 minutes using an adsorbent from zeolite, the optimum time was 90 minutes with a value of 1.3%. When those results are compared with the results of this study, we found that RSBE can absorb higher free fatty acids than zeolite.

3.5 The Effect of RSBE Concentration and Waste Cooking Oil

After finding the optimum time to do adsorspi which is for 90 minutes, in this study continued by mixing RSBE and 350 ml of jelantah oil with variations of 6%, 9%, 12%, 15%, and 18% with a stirring speed of 500 rpm. Free fatty acid adsorption levels can be seen in Table 9.

RSBE variation (%)	Initial fatty acid content (%)	Fatty acid content (%)	Acid number (mg KOH/g)
6	1.06	0.848	1.843
9	1.06	0.780	1.745
12	1.06	0.700	1.732
15	1.06	0.650	1.629
18	1.06	0.720	1.662

Table 9 Adsorption of free fatty acids with variations of RSBE concentrations

According to Pardede (2020), the more adsorbent and contact time are the faster the purification process will be. Table 7 shows a decrease in free fatty acids. The 6% concentration had a fatty acid content of 0.848%, the 9% concentration was 0.780%, the 12% and 15% concentrations were 0.700% and 0.650% respectively, and the 18% concentration had a value of 0.720%. The table shows a decrease in free fatty acid contents as the adsorbent concentration increases. In this study, the best concentration to be used as raw material for biodiesel is a concentration of 15%. Furthermore, the best concentration for the transesterification process is 15% w/v.

3.6 Adsorption Isotherms

The adsorption mechanism can show an interaction between the adsorbent and the adsorbate. Interactions in physics occur if a van deer walls force that binds weak molecules can react back and forth (reversible), form multiple layers, and take place quickly. In chemical adsorption, the formation and breaking of bonds occur due to the reaction between the adsorbent and the adsorbate molecules which form covalent bonds with ions, forming a single layer (monolayer), which is unidirectional (irreversible).

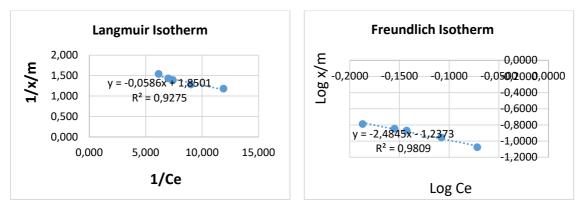


Figure 4 Langmuir isotherm



Figures 3 and 4 shows with 1/ce and 1(x/m) to get Langmuir value and plotting log(x/m) and log ce to get the Freundlich equation. The constant in this approach (K) is obtained by determining the intercept and slope respectively. The constant K can indicate the overall adsorption value and the constant n can indicate the exponent of adsorption, which is related to the adsorption efficiency at the solvent concentration (Mardina, 2012). In this study, the Langmuir equation indicates that the value a is 5.76 and the value b is 0.31. Meanwhile, the Freundlich equation indicates that the value K_f is 1.301 and the value n is 0.414. The results of the tests of the Freundlich and Langmuir equations indicate that the coefficient of determination (R^2) is 0.9, meaning that the adsorption of free fatty acids by the RSBE adsorbent fulfills the Langmuir adsorption equation with R^2 of 0.9275 and Freundlich adsorption equation with R^2 of 0.9809. This shows that the free fatty acid absorption using RSBE as the most dominant adsorbent is physical absorption (physics adsorbtion). The comparison of the calculation results of regression values and constants (a), (b), (n), and (K_f) of the Langmuir and Freundlich isotherms can be seen in Table 10 below.

Isotherms	R^2	Constants	Value
Langmuir	0.9275	а	5.76
		b	0.31
Freundlich	0.9809	n	0.414
		K _f	1.301

Table 10 Values of R^2 and constants of Langmuir and Freundlich isotherms

4. CONCLUSION

The value of water and ash contents of RSBE meets the quality standard as activated carbon and can be used as an adsorbent for the purification of waste cooking oil. The oil content in SBE which is less than 3% is considered nontoxic hazardous waste and can be reused without pretreatment. There was a change on the surface of the SBE which had not been activated and SBE which had been activated although it was not fully perfect because there's no too much change of pores on the surface. Therefore, SBE can be used as an adsorbent to adsorb free fatty acids contained in waste cooking oil. From the results obtained in this study, it is necessary to study further, among others, the use of different samples of adsorbents to reduce free fatty acids in waste cooking oil.

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