Bleaching Earth Recovery from Waste to Purify Coocking Oil by Extraction–Activation Method

Caecilia Pujiastuti*, Ketut Sumada, Meriana Armidianti, and Adinda Rimarsya Bahita Ahmad
Chemical Engineering, University of Pembangunan Nasional "Veteran" East Java, Surabaya, Indonesia
***caeciliapujiastuti@gmail.com**

OPEN ACCESS

***Citation*:** Caecilia Pujiastuti, Ketut Sumada, Meriana Armidianti, and Adinda Rimarsya Bahita Ahmad. 2022. Bleaching Earth Recovery from Waste to Purify Coocking Oil by Extraction–Activation Method. *Journal of Research and Technology* Vol. 8 No. 2 Desember 2022: Page 169–177.

Abstract
Bleaching Earth is another term used for a type of clay or clay that has a montmorillonite structure. Activation by means of physics, namely heating, can remove dirt and moisture content in the bleaching soil particles. This study aims to reactivate Spent Bleaching Earth (SBE) so that it does not become waste in the surrounding environment. As well as knowing the results of the characteristics of Reactivated Bleaching Earth (RBE) obtained. The reactivation process consists of 2 stages, namely extraction on SBE with n-hexane and activation using a furnace. The method used was maceration extraction with extraction times of 60, 90, 120, 150, 180 and 210 minutes. And the activation temperature is 500, 550, 600, 650, 700 and 750 ˚C. This research gives the result that thermal activation using the furnace can reactivate SBE so that it can match the existing Bleaching Earth characteristics. The best conditions for RBE based on the National Standardization Agency (BSN) in this study were the extraction time of 120 minutes and an activation temperature of 550 °C with a water content value of 0.1625%, real density 2.2970 gr/mL, pH 7.1, and yield. SEM is smoother than others.

***Keywords*:** *Bleaching Earth, n-Hexane, Reactivated Bleaching Earth, Spent Bleaching Earth*.

1. Introduction

Indonesia is one of the countries that has a large plantation sector because it has a tropical climate. One of the plantation crops that can grow well in tropical climates is oil palm (*Elaeis guinensis*). Oil palm is a plant that is included in oil-producing plants. In the cooking oil industry, it cannot be separated from the refining process. Bleaching earth is an ingredient in cooking oil refining. Bleaching earth or bleaching clay or often called bentonite is a type of clay with the main composition consisting of SiO_2, Al_2O_3, bound water and Ca^{2+} ions, magnesium oxide and iron oxide. The bleaching earth's bleaching power is due to the presence of Al^{3+} ions on the surface of the absorbent particles so that they can adsorb dyes and depending on the ratio of Al_2O_3 and SiO_2 in the bleaching earth (Ketaren, 2008).

According to Government Regulation No. 85 Year 1999, this waste includes B3 waste (Hazardous and Toxic Materials) from specific sources. According to PP 74/2001, hazardous and toxic materials, hereinafter abbreviated as B3, are materials which due to their nature or concentration and / or quantity, either directly or indirectly, can pollute and or damage the environment, and or may endanger the environment, health, the survival of humans and other living things (Damanhuri, 2010).

Extraction is a process that separates solid and liquid materials with the help of a solvent. The solvent used in the extraction process must be able to extract the desired substance without dissolving other materials. The solvent used in an extraction process must meet several existing requirements such as the solvent must only be able to dissolve the material to be extracted and then easily separated from the solute and also the solvent does not react with the solute by all means. The maceration method is carried out at room temperature ranging from 20˚C-25˚C. Maceration is carried out at room temperature to prevent excessive solvent evaporation due to temperature factors. The n-hexane solvent is a volatile solvent so that during the maceration process, the macerator is covered with aluminium foil as tightly as possible to prevent the solvent from evaporating (Krisyanti and Sukandar, 2011).

Spent Bleaching Earth is a non-renewable material so it needs to be used efficiently by reactivating it for reuse as an adsorbent. This reactivation process is expected to reduce the dependence of the cooking oil industry on bleaching soil. SBE is a mixture of clay and hydrocarbon compounds from CPO. SBE needs special handling so as not to cause problems for the cooking oil bleaching industry. There are two ways of activation to increase the absorption of bentonite:

a. Activation by heating Activation by heating aims so that the water-bound by the molecular gap can be evaporated, so that its porosity increases. This process is very suitable for the Swelling type of bentonite.
b. Activation by acidification. Activation with acidification can increase the ratio between SiO_2: Al_2O_3. This process is carried out by dissolving bentonite into acid (HCl or H_2SO_4) at a certain concentration with a certain immersion time as well. (Herdiani, 2009)

Factors affecting bleaching earth activation process:

1. Activation temperature
   The results showed that when the temperature was set at 400˚C the cation exchange capacity decreased slightly due to reversible dehydration. Where dehydration is a physical event in which water is lost without structural loss, while dihydroxylation is a chemical event that requires decomposition. In addition, the smectite crystal structure collapsed at 900˚C. (Hussin et al., 2011).
2. Activation time
   The results showed that heating the bleaching earth at 100˚C and 200˚C for 12 hours, caused a significant reduction in the amount of exchange acidity, but did not reduce water

absorption. In contrast, the bleaching earth suffers from an almost complete loss of cation-exchange capacity and does not expand upon heating to 200 ° C. Heating at 300˚C for 12 hours causes a large loss in cation exchange capacity and results in dehydration which is not seen well from bleaching earth (Hussin et al., 2011).

Reactivated Bleaching Earth is the result of reactivation treatment of used bleaching soil. In principle, SBE has a low adsorption capacity, but if reactivation is carried out, the adsorption power will increase. The reactivation process in SBE can be carried out physically and chemically. The physical recycling process can be done by activating the SBE by heating and chemically using an activator medium, such as strong acids (HCl, H_2SO_4, H_3PO_4) and hydrogen peroxide (H_2O_2). During the heating process at 180°C, it is expected that a polymerization reaction will occur in the oil attached to the used bleaching soil. This polymerization reaction is expected to form hydrocarbon compounds that have complex chemical structures with high molecular weight. Continued heating (300-700°C) is expected to convert these hydrocarbons into coke (charcoal). With the help of high temperature, the coke is expected to be a material that has an active surface. One thing that can reduce the adsorption power of bentonite as an adsorbent is the presence of element Carbon (Wahyudi, 2000). Element Carbon has become charcoal at the time of reactivation. The charcoal structure will become a surface-active material in adsorbing dye on CPO bleaching with the addition of acid at high temperatures. The wider the soil surface of the blanchers will increase the adsorption power of the blanchers against CPO.

Reactivation of Spent Bleaching Earth (SBE) that is produced must meet the quality requirements of Fresh Bleaching Earth (FBE), so that the results of reactivation of the bleaching earth can have the ability to bleach cooking oil. The conditions that must be met include moisture content, ash content, acid number, fat content, and pH (Degree of Acidity).

Table 1. Physical Properties of Bleaching Earth

Test Components	Unit	Fresh Bleaching Earth
Water Content (bk)	(%)	Max15
Specific Gravity	(gr/ml)	2,0 – 2,7
Efficiency of bleaching color	(%)	Min 40
pH suspense (10% Solid)		6,5 – 8,5

Source: BSN 2000

2. Methodology

In this research, the 25 gram spent bleaching earth was put into a three-neck flask and 250 ml of n-hexane solvent was added and then extracted according to the extraction time (60, 90, 120, 150, and 210) (minutes). Filter to separate n-hexane solvent from bleaching earth. Furthermore, the bleaching earth is washed with aquadest to remove the remaining n-hexane solvent. Then the bleaching earth is dried in the oven at 100 ˚C for 1 hour. Bleaching earth from the extraction process is then activated by heating, by placing it in a furnace with temperatures (500, 550, 600, 650, and 750) (˚C) at 2 hours. The processing tools are shown in Figure 1.

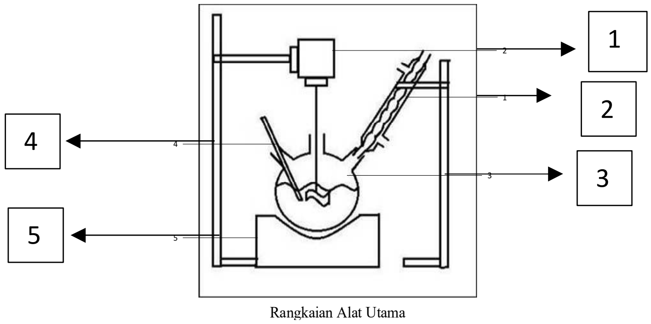

Figure 1. Processing Tools

Where:
1. Mixer
2. Condensor
3. Boiling Three Neck
4. Thermometer
5. Heating Mantel

The research flow is provided in Figure 2.

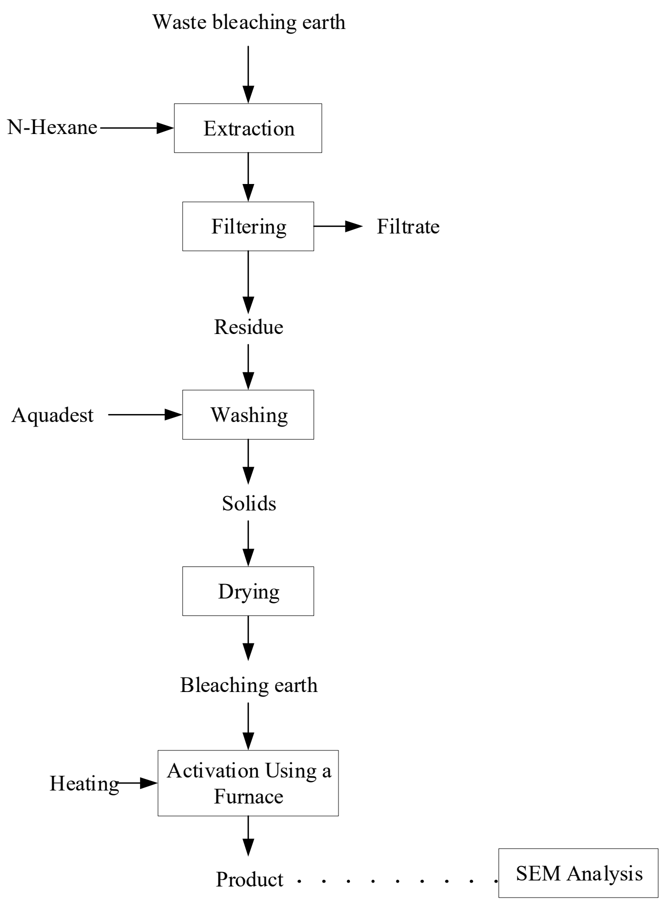

Figure 2. Research Flowchart

3. Result and Discussion

The quality of the bleaching earth produced must comply with the national standard for good bleaching earth. To see the quality of the reactivated bleaching earth, a physical and mechanical test was carried out for the reactivated bleaching earth itself. Physical tests include moisture content, specific gravity, degree of acidity (pH), bleaching efficiency and SEM analysis.

Table 2. Comparison of Spent Bleaching Earth Characteristics with SNI Fresh Bleaching Earth

Tested Components	SNI Standard	Spent Bleaching Earth	Fresh Bleaching Earth
Water Content	Max 15 %	3.4049	9.5365
Specific Gravity	2.0-2.7 gr/mL	1.5619	2.0159
Degree of Acidity (pH)	6.5-8.5	4,9	6,8

Water content

Table 3. Results of the Water Content (%) Recovery Bleaching Earth Test

Temperature (ºC)	Water content (%)				
	60 minutes	90 minutes	120 minutes	150 minutes	210 minutes
500	0,2861	0,2760	0,2630	0,2040	0,2305
550	0,2757	0,2520	0,1625	0,1561	0,1648
600	0,1376	0,1625	0,1500	0,1031	0,1640
650	0,1637	0,1087	0,0707	0,0180	0,0162
750	0,0079	0,0075	0,0091	0,0061	0,0087

$$\text{Water Content} = \frac{(W1-W2)}{(W1-W0)} \times 100 \qquad (1)$$

Information

Wo = weight of empty cup (g)

W1 = weight of cup and SBE remaining cooking oil or RBE before drying (g)

W2 = weight of cup and SBE remaining cooking oil or RBE after drying (g)

From the data on Table 3, the graph below will be obtained:

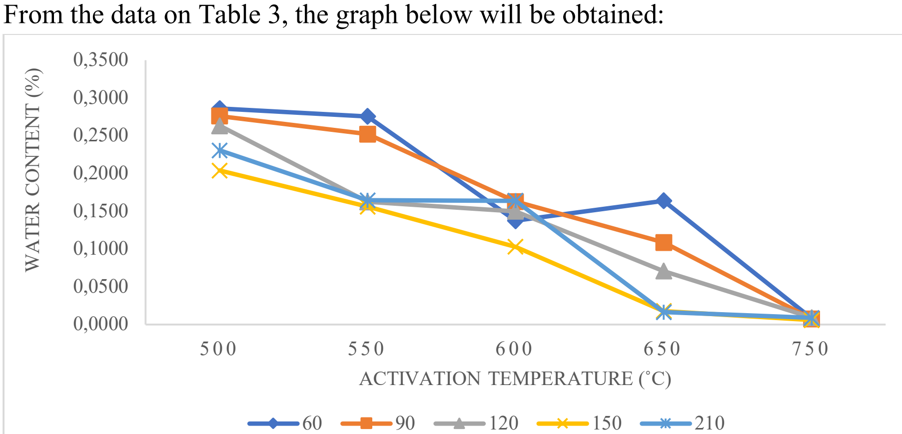

Figure 3. Relationship Between Extraction Time and Activation Temperature Against Moisture Content (%) Recovery Bleaching Earth

Based on Figure 3 shows that the results of the Recovery Bleaching Earth moisture content range from 0.0061 to 0.2861%. The highest water content test value lies in the sample with an extraction time of 60 minutes and an activation temperature of 500 ˚C, which is 0.2861%. The lowest water content test value lies in the extraction time for 150 minutes and an activation temperature of 750 ˚C, which is 0.0061%. According to the National Standardization Agency (BSN) in 2000, the standard water content of Bleaching Earth is a maximum of 15%. When compared with research results, all Recovery Bleaching Earth results obtained are in accordance with the National Standardization Agency (BSN). Thermal activation of blanching soil is a physical treatment process that involves calcining the blanching soil at high temperatures. This removes dirt and moisture content in the bleaching soil particles.

Specific Gravity

Table 4. The results of the Real Specific Gravity (gr/mL) Recovery Bleaching Earth test

Temperature (ºC)	Specific Gravity (gram/mL)				
	60 minutes	90 minutes	120 minutes	150 minutes	210 minutes
500	2.3008	2.2489	2.2704	2.3362	2.3655
550	2.2574	2.2812	2.2970	2.3740	2.2569
600	2.4364	2.3512	2.4298	2.3692	2.3597
650	2.3499	2.3025	2.2709	2.3297	2.2807
750	2.4311	2.4845	2.5033	2.3108	2.2808

From the data on Table 4, the graph below will be obtained:

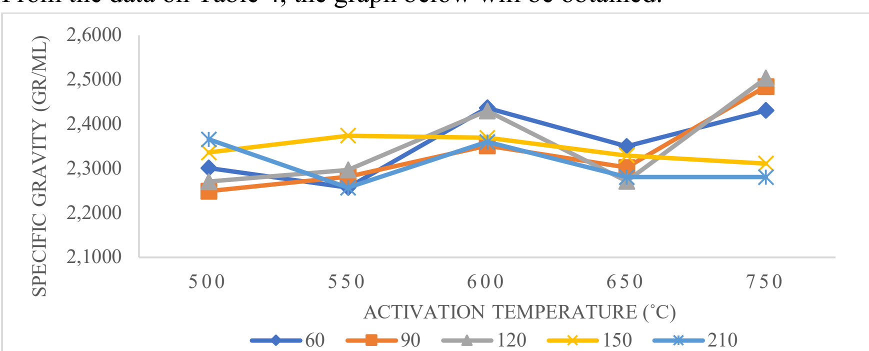

Figure 4. Relationship Between Extraction Time and Activation Temperature Against Specific Gravity (gr/mL) Recovery Bleaching Earth

Based on Figure 4 shows that the results of the Recovery Bleaching Earth's real density ranged from 2.2489-2.5033 gr/mL. The highest real density test value lies in the sample with an extraction time of 120 minutes and an activation temperature of 750˚C, which is 2.5033 gr / ml. The lowest water content test value lies in the extraction time for 90 minutes and an activation temperature of 500˚C, which is 2.2489 gr/mL. According to the National Standardization Agency for Indonesia (BSN) in 2000, the maximum density standard for Bleaching Earth is 2.0-2.7 gr/mL. When compared with research results, all Recovery Bleaching Earth results obtained are in accordance with the National Standardization Agency (BSN).

Degree of acidity (pH)

Table 5. Recovery Bleaching Earth pH Test Results

Temperature (ºC)	Degree of acidity (pH)				
	60 minutes	90 minutes	120 minutes	150 minutes	210 minutes
500	7	7	7	7	7
550	7.1	7.2	7.1	7.2	7.1
600	7.4	7.4	7.5	7.5	7.4
650	7.9	7.9	7.8	7.8	7.9
750	7.9	7.8	7.8	7.9	7.9

From the data on Table 5, the graph below will be obtained:

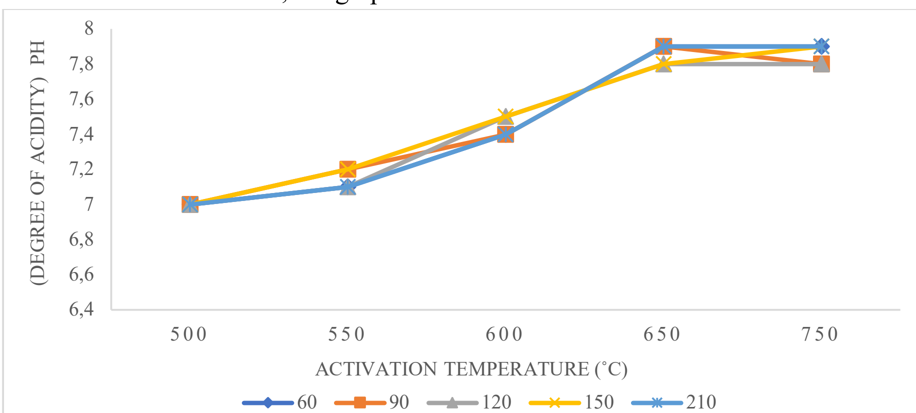

Figure 5. Removal Efficiency of Warna (%) and Stirring Time (Minute) at Various Coagulant Volume

Based on Figure 5 shows the results of Recovery Bleaching Earth pH measurements obtained ranging from 7-7.9. The highest pH measurement test value is 7.9. The lowest pH measurement test value is 7. According to the National Standardization Agency for Indonesia (BSN) in 2000, the standard for Bleaching Earth pH is around 6.5-8.5. When compared with research results, all Recovery Bleaching Earth results obtained are in accordance with the National Standardization Agency (BSN). At an activation temperature of 650 ºC and 750 ºC, the pH obtained is greater than the previous activation temperature. The pH value at an activation temperature of 500 ºC is also close to the pH value of Fresh Bleaching Earth, which is 6.9. According to Ketaren (1986), the adsorption power of color will be more effective if the pH of the adsorbent is close to neutral.

Efficiency of Bleaching Color

Based on Figure 6, shows the results of the Efficiency Analysis to bleed the resulting Recovery Bleaching Earth. The test was carried out by entering a sample in the form of RBE results into used cooking oil from a food frying pan. Samples were entered as much as 3% of used cooking oil. It can be seen from the results of the analysis that in the bleaching efficiency test at the extraction time of 150 minutes, it was found that the oil could be slightly clearer than at the extraction time of 90 minutes.

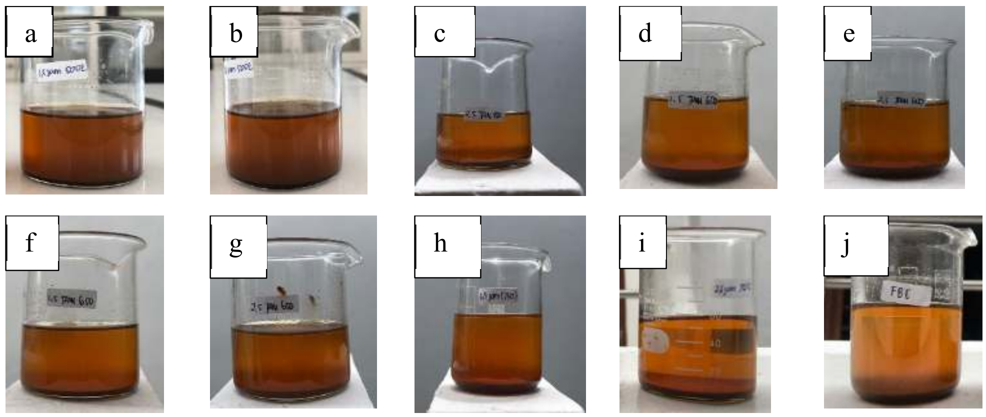

Figure 6. Result of the RBE Bleaching Effectiveness Test (a) 90 minutes 500ºC (b) 150 minutes 500ºC (c) 150 minutes 550 ºC (d) 90 minutes 600ºC (e) 150 minutes 650ºC (f) 90 minutes 650ºC (g) 150 minutes 650ºC (h) 90 minutes 750ºC (i) 150 minutes 750ºC (h) FBE

SEM Analysis

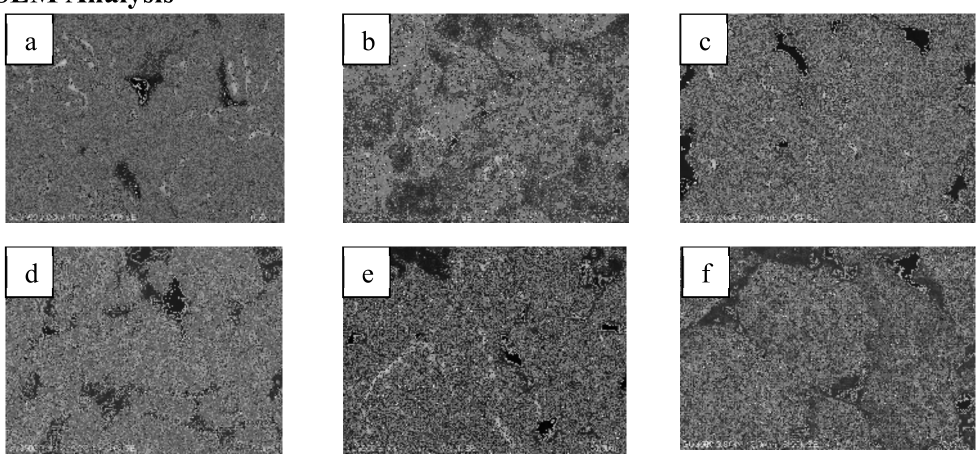

Figure 7. SEM Test Results (a) Spent Bleaching Earth (b) RBE at 500ºC (c) RBE at 550ºC (d) RBE at 600ºC (e) RBE at 650ºC (f) RBE at 750ºC at 120 Minutes

Based on Figure 7, shows the results of the SEM analysis that has been done. SBE and RBE testing using SEM was carried out at magnifications of 500x, 1000x, 1500x, and 3000x. From the four magnifications, it is still unclear the pore structure of the two types of blanching soil. In Figure 7, by using a 3000x magnification, it can be seen that overall, there is a difference in the surface structure between Spent Bleaching Earth before being reactivated and after being reactivated. Figure 7a which is the Spent Bleaching Earth left over from the bleaching process of cooking oil before being activated, seen by SEM at 3000x magnification shows that there are still a lot of dirt on the surface and it can also be seen that the remaining SBE is still clumped together because it still contains water and oil in it. The dirt that sticks to the surface of the SBE results in the closing of the pores of the bleaching soil. So that the absorption power does not work properly. Whereas in Figures 7b, 7c, 7d, 7e, and 7f which are the results of reactivation of RBE with an extraction time of 120 minutes, it shows that many of the impurities on the surface have been removed, the surface pores are also more visible. In Figure 7c, the reactivated RBE at 550ºC, it can be seen that the surface looks smoother and clearer than the unreactivated SBE. And in Figure 7e which is the result of reactivation of RBE at 650ºC, the surface pores are more visible. Larger pores on the surface of the RBE allow this blanching soil material to perform better adsorption power than that which has not been activated.

4. Conclusion

Based on the research results, from the Recovery Bleaching Earth, it has met the 2000 National Standard (BSN). The best condition is the stirring time of 120 minutes at an activation temperature of 550 ˚C, Because The selection of the best variables seen from the results of the analysis of each sample includes water content, real specific gravity, pH, effectiveness, and SEM analysis of variable results with conditions at a stirring time of 120 minutes at an activation temperature of 550 ºC which is the best condition.

Appreciation

Researchers would like to thank the Chemical Engineering, University of Pembangunan Nasional "Veteran" East Java, Indonesia for providing the opportunity to use laboratory equipment are also for the entire team in research laboratories who have contributed in this research.

REFERENCES

[BSN] National Standardization Agency. 2000. Bentonite for Vegetable Oil Bleaching. SNI 136336-2000

Damanhuri, E. 2010. Study of Management of Hazardous Waste in the Laboratories of ITB. Journal of Enviromental Engineering: Bandung Institute of Technology.

Herdiani, I. A. 2009. Adsorbent Application in Purification Process of Jatropha curcas (Jatropha curcas L.) Biodiesel Using Column Method. Essay. Bogor Agricultural Institute: Bogor.

Hussin, F., Aroua, M. K., & Daud, W. M. A. W. 2011. Textural Characteristics, Surface Chemistry and Activation of Bleaching Earth. Chemical Engineering Journal, 170(1), 90–106.

Ketaren, S. 1986. Pengantar Teknologi Minyak dan Lemak Pangan. 1st Edition. University of Indonesia: Jakarta.

Ketaren, S. 2008. Minyak dan Lemak Pangan. 1st Edition. University of Indonesia: Jakarta.

Krisyanti, S. and Sukandar. 2011. Oil Recovery From Hazardous and Toxic Waste Spent Bleaching Earth With Solvent Extraction Method. Journal of Enviromental Engineering: Bandung Institute of Technology.

Wahyudi, M.Y., 2000. *Studi Penggunaan Kembali Bleaching Earth Bekas sebagai Adsorben dalam Proses Refining CPO*. Tesis Magister. Program Studi Teknik Lingkungan, Institut Teknologi Bandung, Bandung.