BIO Web of Conferences **99**, 02013 (2024) https://doi.org/10.1051/bioconf/20249902013
ICAFES 2023

Reactivation of used bentonite (Spent Bleaching Earth) and its reuse as adsorbent for CPO (Crude Palm Oil) bleaching process

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Abstract. Used bentonite or spent bleaching earth (SBE) is one of agro-industrial solid wastes in palm oil bleaching process. SBE has potential to be reactivated and reuse as adsorbent of crude palm oil (CPO). Reusing reactivated SBE is an effort to utilize waste so that it can increase added value while also preventing environmental damage due to waste accumulation. This study aimed to determine the effect of repeated use of reactivated SBE as adsorbent in the CPO bleaching process. The research methods consisted of 4 stages: characterization, reactivation of SBE and CPO bleaching process, then analysis of bleached oil, and cost analysis of the process. This study used two different types of SBE whose oil content had previously been recovered. Repetitive use of both SBE samples affected the quality of bleached oil. Maximum repetitive use that could be received by using SBE samples A was at fourth level and B was at the second level. Costs analysis of reactivation and two times repeated use of SBE was higher than fresh bleaching earth (FBE) purchase with equivalent amount for bleaching process. However, reactivation and reuse of SBE could increase the added value of the waste and prevent negative impacts on the environment.

1 Introduction

The largest use of bentonite is in the oil refining industry sector, especially crude palm oil or CPO (Crude Palm Oil). Almost all refining industries in Indonesia use bleaching earth in the form of activated bentonite. This has an impact, namely the emergence of solid waste in the form of used bentonite with a mixed composition between clay and hydrocarbon compounds from CPO. In 2022 Indonesia's CPO production was 45.58 million tons [1], while the national consumption was 20.97 million tons [2]. In the CPO refining process, one of the important processes is the bleaching process. The CPO bleaching process uses bleaching earth between 0.5-2.0% of the CPO mass. Assuming that in 2022 the CPO of 20.97 million tons was

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processed for the domestic market, then refining process required bleaching earth as an adsorbent of 104 to 420 thousand tons.

The main composition of bentonite is montmorillonite, which consists of crystalline aluminium silicate (SiO_2, Al_2O_3), water, alkali metals (calcium oxide, CaO, magnesium oxide, MgO) and other transition metals such as iron oxide, Fe_2O_3 [3]. One of the stages of the palm oil production process is the bleaching process. The bleaching process aims to remove unwanted colour and impurities in the oil by using adsorbents (bleaching earth, activated clay or charcoal). The most widely used bleaching earth in Indonesia is bentonite. Bentonite is a type of clay with a main composition consisting of SiO_2, Al_2O_3, water and Ca^{2+} ions, magnesium oxide and iron oxide. The bleaching power of bentonite is due to the presence of Al^{3+} ions on the surface of the absorbent particles so that they can adsorb colours and depends on the ratio of Al_2O and SiO_2 [4].

Used bleaching earth (spent bleaching earth or SBE) will be deactivated over time because its surface has been covered by impurities contained in the CPO refining process including phosphatides, gums, metals, fatty acids and colours in CPO, so that it cannot be reused. If SBE interacts with water, the compound will easily decompose, causing a foul odour that disturbs the environment [5]. Most of the SBE is resold to other parties, burned, or dumped as waste on vacant land, which can cause fires and harm the environment due to its oil content. According to Indonesian Government Regulation No. 101 of 2014 concerning Hazardous and Toxic Waste Management, SBE is declared as B3 waste category 2, which contains B3, has a delay effect, has an indirect impact on humans and the environment and also has sub-chronic or chronic toxicity. As is the case with FFB processing waste at palm oil mills that have been handled so that they are referred to as Zero Waste Factories, SBE also has many possibilities to be reused, reutilized or processed for various other uses [6].

Bentonite is a non-renewable material that needs to be used efficiently. In addition, the reuse of SBE is one of the efforts to utilize waste so as to increase its added value. Based on the Indonesian Government Regulation No. 22 of 2021, SBE is already classified as non-hazardous waste (B3) if the oil content is less than or equal to 3%. SBE reactivation process is carried out by restoring its absorption ability. According to Soetaredjo *et. al.* [7], the surface and acidic properties of bleaching earth such as bentonite can be optimized by controlling activation conditions such as the type and amount of acid, temperature, and activation time. Repeated use can also be done to reduce the amount of waste generated. This can reduce the use of bentonite.

SBE reactivation has been done in many previous studies with various methods. Boukerroui *et. al.* [8] reactivated bleaching earth with the addition of hydrochloric acid, optimal values of treatment (temperature 350°C, carbonization time 01 h, and HCl concentration 1M) gave a very efficient material and using the reactivated bleaching earth to bleached soybean oil. While Suryani *et. al.* [9] determined the type of acid and the best ratio for reactivation using phosphoric acid, nitric acid, and sulphuric acid, the best results produced were using nitric acid with ratio of 1:2. In addition, Bachmann *et. al.* [10] has also conducted a thermal analysis of the temperature of the active thermal decomposition process of bleaching earth which produces optimal conditions for the thermal regeneration were 108 min at 587 °C to produce regenerated spent bleaching earth characteristics which were similar to the activated bleaching earth.

The potential for repeated use of used bentonite needs to go through further research stages so that the effect of reuse of bentonite as an adsorbent on the quality of the refined oil can be known. This study aims to determine the effect of reusing used bentonite that has been reactivated as an adsorbent for the CPO bleaching process. Then the purpose of this study is to determine the maximum limit of repeated use of reactivated bentonite with acceptable performance parameters. In addition, this study aims to compare the cost of the reactivation process and the reuse of reactivated SBE with the purchase of fresh bleaching earth.

2 Material and methods

2.1 Tools and materials

The equipment used were glass wares, furnace, hot plate, pH-meter, porcelain dish, aluminium dish, analytical balance, oven, 150 mesh sieve, thermometer, HACH spectrophotometer, HITACHI SU-3500 SEM (Scanning Electron Microscope) and HORIBA EDX detector.

The main materials used in this study were 2 different types of SBE, referred to as sample A, that was used in the CPO (Crude Palm Oil) bleaching process and sample B was that was used for the RBDPO (Refined Bleached Deodorized Palm Oil) bleaching process. The oil content of both SBE samples have been extracted using the reflux method with a material/solvent ratio of 1:8. In addition, FBE (fresh bleaching earth) was also used as a comparison and CPO. The chemicals used were 15% HNO3, 0.1 N KOH, PP indicator, and distilled water.

2.2 Methods

Sample A and sample B were characterized before and after the reactivation process. The characteristics tested include pH value, water content, ash content [11]. Reactivation is carried out by activation using an acid solution, namely 5% HNO_3 and then 2-stage of heating. The reactivation process of SBE is based on Suryani *et. al.* [9] with modification as seen in Figure 1.

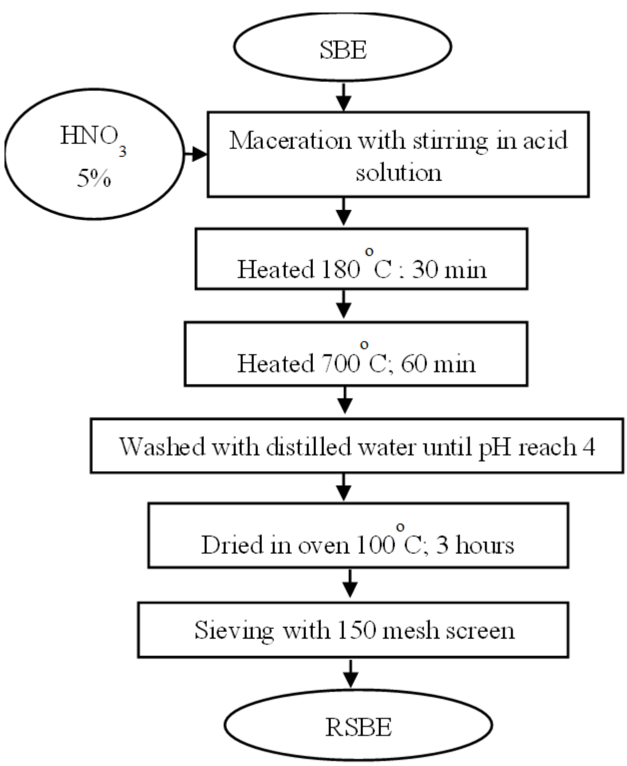


Fig. 1. Reactivation process flow diagram.

After producing reactivated SBE, the product was then used as an adsorbent in the CPO bleaching process. The reuse of reactivated SBE as an adsorbent in CPO bleaching was done repeatedly as seen in Figure 2. Each bleached oil or BPO (Bleached Palm Oil) using reactivated SBE adsorbent was analysed. The quality parameters of BPO analysed included clarity, bleaching efficiency, and acid number.

The data analysis of this study was conducted using a single-factor completely randomized design with two replications. The treatment used was the re-usage time(s) of reactivated SBE as a CPO bleaching adsorbent, namely 1 time, 2 times, 3 times, 4 times, and 5 times. The treatment was given to both samples, A and B so that data were obtained for each samples. The data obtained were analysed by analysis of variance (ANOVA) and if it showed a significant effect, then continued with the Duncan New Multiple Range Test (DNMRT) at 5% level of confidence.

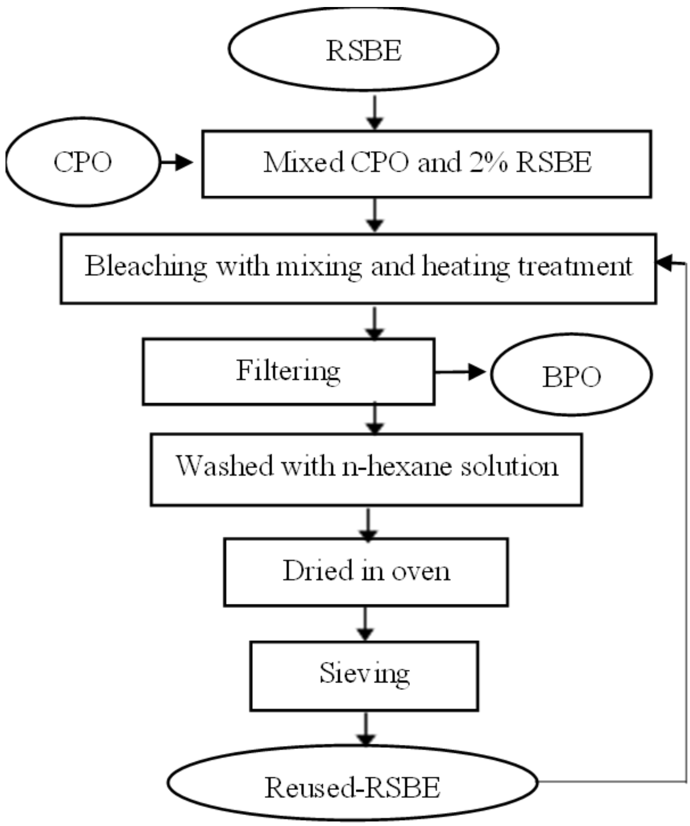


Fig. 2. Bleaching process flow diagram.

Cost analysis was conducted to determine the cost required to carry out the CPO bleaching process using reactivated SBE with the recommended amount of reuse from this study. The results of the cost calculation were compared with the cost of purchasing FBE.

3 Results and discussion

3.1 Characteristics of samples

Table 1. Samples characterization results

Sam-ple	Parameters	Value	
		Before reactivation	After reactivation
A	Water content (% w/w)	1.74	1.56
	Ash content (% w/w)	0.23	0.09
	pH	3.17	5.95
B	Water content (% w/w)	1.87	1.58
	Ash content (% w/w)	0.25	0.14
	pH	3.16	7.92
FBE	Water content (% w/w)	0.13	
	Ash content (% w/w)	0.07	
	pH	4.35	

Table 2. Physical characteristics of bleaching earth according to SNI

Parameters	Unit	Value
Water content	%	Max 15
pH suspense (10% solid)	-	6.5-8.5
Density	g/mL	2.0-2.7
Bleaching efficiency	%	Min 40%

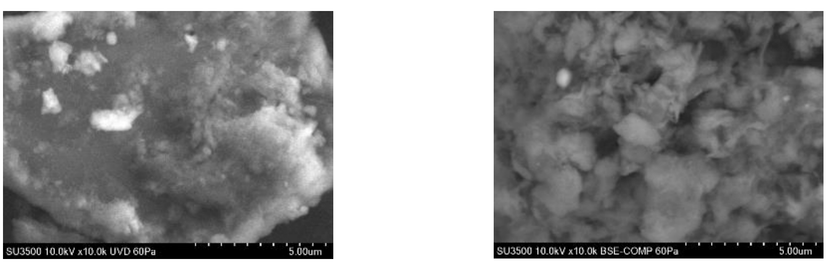
The moisture content of both samples A and B before and after reactivation has met SNI standards as bleaching earth. The results in Table 1 show that the moisture content of the samples after reactivation decreased than before for both samples A and B. The water content after reactivation is still higher than FBE. The decrease in moisture content after reactivation can be caused by the heating process in the activator furnace which makes the water trapped in the samples escape. In addition, the water content is also related to the hygroscopic properties of the acid activator used, HNO_3. The reactivation process using acid can cause the water content in bentonite crystals to be carried away by HNO_3 when direct contact occurs. Water content has a relationship with the ability of bentonite to absorb impurities, the lower the water content, the wider the available pores on the surface.

While the ash content shows the amount of non-organic material contained in the material. The ash content of bentonite consists mostly of SiO_2 and Al_2O_3. The ash content of bentonite after reactivation is lower than before reactivation. This decrease can be caused by the reduction of mineral residues in the bentonite that were carried away in the purification process on the previous use of bentonite. According to Abukhadra *et. al.* [12], adsorbents can adsorb dissolved minerals and macromolecules such as organic acids that contain many atoms of Ca, Fe, Mg and Al which are impurities (Table 2).

The pH value of the reactivated bentonite has increased from a tendency to be acidic to getting closer to neutral. Ketaren [4] states that the adsorption capacity of the oil's colour will be more effective if the pH of the adsorbent is close to neutral. Based on the results obtained, it is expected that the reactivated SBE can absorb impurities in CPO better. Bentonite is an aluminosilicate mineral with a 2:1 layer structure (SiO_2 and Al_2O_3). The active side of bentonite can be divided into surface and edge. Both of these sides are external surfaces. The surface always has a negative charge due to the isomorphic substitution of Si^{4+} by Al^{3+} [13,14]. Otherwise, the edge side of clay minerals varies in charge depending on pH, being positively charged at low pH and negatively charged at high pH as a result of protonation and deprotonation of surface hydroxyl groups (SiOH) [15].

As support for the allegation of changes in the pores on the bentonite surface after reactivation, an analysis of the morphological conditions of bentonite was carried out. The analysis was carried out using SEM (*Scanning Electron Microscope*), which is one type of electron microscope that uses electron beams to describe the surface contours of a material. Testing using SEM was carried out at magnifications of 500x, 1000x, 2500x, 5000x, and 10 000x. Magnification of 10 000x produces the clearest morphological image of the sample surface.

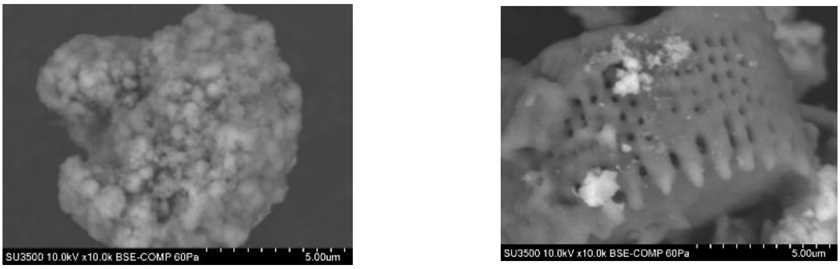
The results of sample A before reactivation can be seen in Figure 3 (a). The pores on the surface of sample A cannot be observed because sample A is SBE that has been used for the CPO bleaching process so that impurities such as phosphatides, and short-chain fatty acids and free fatty acids from the previous process were trapped in the pores. Figure 3 (b) shows the surface of sample A after reactivation. The surface of sample A shows the surface contours after reactivation, but there are still visible impurities around the surface of the sample. This can occur due to the imperfect reactivation process. The incomplete process can be caused because the sample contact with the activator acid solution, HNO_3, does not occur properly. The sample cannot dissolve and mix with the acid solution. This can be caused by the large interfacial tension of the two materials, the bentonite and acid, so that contact is difficult to occur. Therefore, impurities that stick to the surface and close the pores of sample A cannot be removed optimally.



(a) (b)

Fig. 3. Surface morphology of sample A before (a) and after (b) reactivation at 10000x magnification.

Figure 4 (a) shows that the surface of sample B is mostly covered by thick impurities. The impurities that appear thick and stick to the outside of the sample surface may indicate that the adsorbent pores on the surface of sample B have been closed so that the ability of bentonite sample B as an adsorbent is poor. The reactivation of sample B results in characteristic changes that indicate an increase in adsorbing ability.



(a) (b)

Fig. 4. Surface morphology of sample B before (a) and after (b) reactivation at 10000x magnification.

Meanwhile, Figure 4 (b) shows the surface of sample B with more number of open pores, which may be the result of reactivated material. Impurities are still visible attached to some parts of the RSBE exterior but the number of open pores can be seen clearly.

3.2 Bleaching process

3.2.1 Clarity

The treatment of re-usage times of RSBE using both samples A and B affected the clarity of the bleached palm oil (BPO). Clarity is measured based on the transmittance value read from the spectrophotometer. The higher the transmittance value indicates the clearer the oil. The clarity of BPO from bleaching using RSBE as adsorbent decreased the more re-usage repeated times of RSBE. Based on Figure 5, the highest transmittance value for BPO using sample A was obtained on the first use bentonite of 24.1%. These results are still below the transmittance value of BPO bleached using FBE (Fresh Bleaching Earth) as adsorbent which is 46.70.

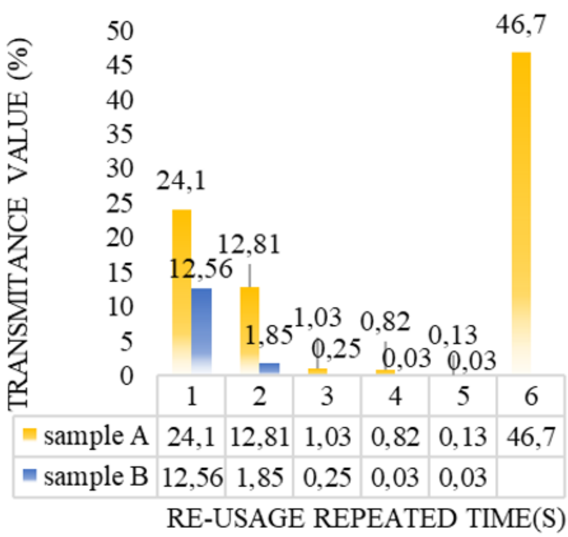


Fig. 5. Effect of re-usage repeated time treatment of sample A on the transmittance value of BPO.

The results show that the highest transmittance value of BPO using sample B is at 1 time reuse which is 12.56%. These results are below the transmittance value of BPO that was bleached using FBE. It can be interpreted that the ability to absorb color and impurities from RSBE sample A and B is below the capability of FBE. These results can occur because after the reactivation process bentonite sample A and B still contains impurities so that its adsorption capability is not as good as FBE. The longer RSBE is used, the less amount of impurities that can be absorbed from the CPO due to the pores of the adsorbent being increasingly covered by impurities.

3.2.2 Bleaching efficiency

A parameter that can indicate the quality of a bleaching process is the bleaching efficiency. Based on the result, it was found that the repeated use of RSBE samples A and B as adsorbent affected the bleaching efficiency. The CPO bleaching efficiency using sample A and B can be seen in Figure 6. It can be observed that the efficiency had a tendency to decrease in line with the more time RSBE samples were reuse as adsorbent. The decrease in bleaching efficiency can be caused by the more RSBE is reused, the more pores are covered by impurities so that the adsorption ability decreases. The result that given the highest bleaching efficiency of 82.34% is sample A with the 1 time reuse. Then at the 2nd reuse, it was resulting efficiency at 74.50%, 3rd 43.21%, 4th 40.26%. The four results still meet the requirements of bleaching earth standard which is at least 40% while the 5th level does not meet SNI standard. The best result of 82.34% is still below the efficiency result of using new adsorbent or FBE which is 90.53%.

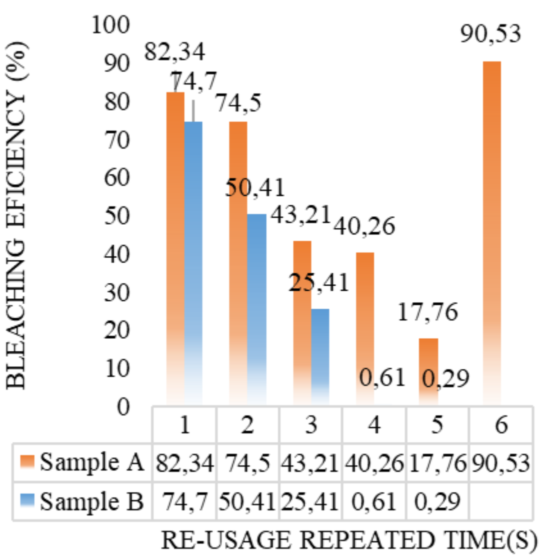


Fig. 6. Effect of re-usage repeated time treatment on the bleaching efficiency of the adsorbents sample A and B.

Effect of reusage repeated time on bleaching efficiency of sample B, which is 74.70%, is lower than the efficiency using FBE. This can be caused because after reactivation, sample B still contains impurities as can be seen from the results of morphological testing using SEM in the previous section. Duncan's test showed significant differences between the 1st and 2nd reusage times, while the 3rd, 4th, and 5th r eusage times did not show significant differences.

https://doi.org/10.1051/bioconf/20249902013

Similar results were also obtained by Suryani *et. al.* [9] where the bleaching efficiency ranged from 95.27±0.03%-98.77±0.03% and FBE had the highest bleaching efficiency compared to all adsorbents.

3.2.3 Free fatty acids

Free fatty acids can be one of the indicators of oil quality. Free fatty acids can be formed in every process of oil processing. The amount of free fatty acids is a factor that can reduce oil quality because these fatty acids are easily oxidized and cause odour and damaging the oil. In addition to colour bleaching and absorbing impurities, the use of adsorbents can also reduce the amount of free fatty acids contained in oils such as CPO. The ability of bleaching earth to absorb free fatty acids is due to the presence of silanol groups (Si-O-H). The groups are reactive groups formed from SiO_2 compounds in bleaching earth during reactivation with acid. The more amount of SiO_2 in the adsorbent will increase the number of Si-O-H (silanol) groups on the adsorbent surface. These silanol groups will absorb free fatty acids, organic substances and other polar substances such as peroxide compounds [16].

The use of RSBE as an adsorbent is expected to reduce the presence of free fatty acids in CPO. The results showed that the re-usage repeated times of sample A and B as an adsorbent affected the acid number of BPO. The acid number of BPO using sample A as adsorbent can be seen in Figure 7. The initial acid number of CPO before purification was 5.61. The lowest BPO acid number resulted from the 1-time re-usage of sample A is 3.41. This value has a lower value compared to bleached BPO using FBE. The tendency of the acid value of BPO increased with the increasing number of re-usage time of RSBE as adsorbent. The acid number values at the 3rd, 4th, and 5th stages have higher values compared to the initial acid number value of CPO before refining. This can be caused by the addition of fatty acids to the refined oil carried by RSBE during previous usage as adsorbent.

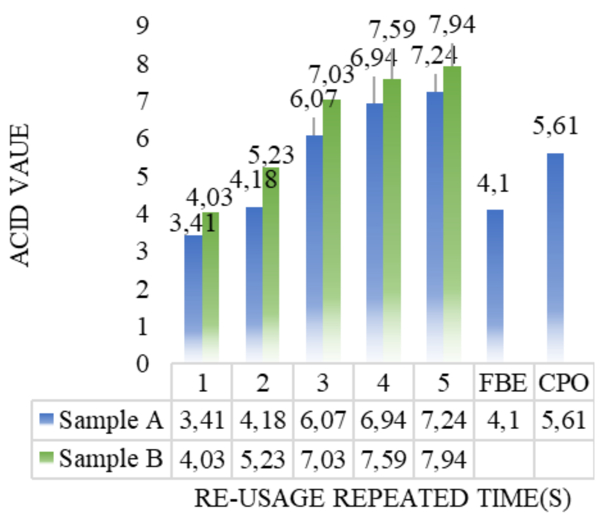


Fig. 7. Effect of re-usage repeated time treatment on the acid value of bleached oil with sample A and B.

The results showed the lowest acid value of BPO using sample B was 4.03 at 1-time re-usage RSBE. This value has a higher value compared to the BPO bleached using FBE. The bleaching process accompanied by heating can also be the cause of the increase in free fatty acids in oil.

3.3 Cost analysis and environmental benefit

The reuse of RSBE as an adsorbent in the CPO bleaching process needs to be analysed regarding costs. The simple cost analysis conducted in this study was carried out by comparing the cost of purchasing FBE compared to the cost of re-usage and reactivation of SBE. Table 3 is the result of the comparison of both purchasing cost of FBE and the re-usage of RSBE. The results showed that the cost of reactivation and reuse of RSBE is cheaper than the purchase of FBE. Table 3 showed the cost of buying FBE assumed that the price is IDR 7.153/kg. So that for buying 2 tons of FBE the cost needed is IDR 14.305.724.

Table 3. Cost for buying FBE

Mate-rial	A-mount	Unit	Price/Unit	Cost
FBE	IDR 2.000	kg	IDR 7.153	IDR 14.305.724
Total				**IDR 14.305.724**

Based on the results showed in Table 4, the cost of reactivation and reuse of bentonite sample A or B with maximum re-usage repeated 2 times on a 2000kg/batch usage basis is IDR 5.894.771. The cost is cheaper than the purchase of FBE. The comparison results showed that economically, the method of reactivation and repeated re-usage of RSBE as a CPO refining adsorbent has cheaper cost than the conventional method of using FBE. This method has promising potential for upscaling to industrial scale.

Table 4. Cost of SBE reactivation and re-usage as adsorbent.

Material	A-mount	Unit	Price/ Unit	Cost
SBE	2000	kg		IDR -
HNO3	5	unit (35 kg)	IDR 1.000.000	IDR 5.000.000
Energy for activation	1550	watt/ unit device (capasity 5.8 kg)		IDR 799.111
	90	minutes		
	345	unit device for 2000kg		
	996,74	Rp/ kWh		
Energy for drying	1000	watt/ unit device (capasity 1000 L)		IDR 83.700
	180	minutes		
	4	unit device for 2000kg		IDR 11.961
Total				**IDR 5.894.771**

However, other benefits besides economic factors can be obtained by applying this method is also could gain environmental benefits. Reusing RSBE by reactivation process can increase the added value of SBE waste. In addition, it can reduce the need for the use of FBE and prevent the emergence of more waste from the use of bentonite in the palm oil refining industry.

In addition, an important benefit to be gained is the positive impact on the environment. The utilization of solid waste in the form of RSBE and its repeated re-usage allows the industry to consume less FBE than using it continuously. One of FBE source is bentonite, which is a type of mining earth that is one of the non-renewable natural resources. Bentonite used as an adsorbent in the oil refining industry is Ca-bentonite or in the trade known as tonsil, NKH, galleon and others. The main users of bentonite are the palm oil and coconut oil industries, followed by margarine, metals for building, and the cast machine industry. The palm oil industry consumes bentonite about 70% (68 910.6 tons), then the coconut oil industry about 16% (15 751.1 tons) and the remaining 14% (13 782 tons) is consumed by the margarine, foundry, machinery, soap, cosmetics and paint industries. Indonesia's palm oil production has increased significantly at an annual rate of 17.51% since 1999. Some of the other major bentonite-using industries are coconut cooking oil and margarine with growth rates of 8.22% and 25.3% respectively. This indicates that the need for bentonite in the country is also increasing [17].

After SBE is being used as an adsorbent in the oil refining process, it is categorized as category 2 B3 waste, which contains B3, has a delay effect, and has an indirect impact on humans and the environment and has sub-chronic or chronic toxicity. Therefore, special handling is required before disposal into the environment. In addition, hazards can arise from the disposal of this waste if done carelessly. It is not uncommon to find misuse of SBE waste that still high in oil contained, for example for additional fuel for boiler furnaces in industry or disposal on vacant land without treatment of waste first. Indiscriminate disposal of waste can cause problems if SBE interacts with water, the compound will easily decompose, causes a distressing unpleasant odour that pollutes the environment [5]. In addition, the oil content that is still contained in the used bentonite as an oil refining adsorbent can also cause land fires if left alone in contact with oxygen and fire sources. These things can be avoided if reutilization is carried out as well as management of this solid waste before disposal into the environment.

4 Conclusion

The reuse of used bentonite (samples A and B) as a result of reactivation affects the efficiency of bentonite as an adsorbent and the clarity and acid number of the blanched oil. The treatment of repeated use of reactivated bentonite in both samples A and B affects the quality parameters of bentonite as an adsorbent and blanching oil, namely oil clarity parameters, blanching efficiency, and blanching oil acid number. Based on the results of the analysis of the tested parameters, the maximum acceptable reuse using bentonite sample A is up to the 4th time of reuse. As for sample B, the maximum reuse is up to the 2nd reuse. The cost of reactivation and repeated use of 2 times of used bentonite as CPO adsorbent is higher than the purchase of FBE with an equivalent amount requirement. However, reactivation and reutilization of used bentonite can increase the added value of the waste and prevent negative impacts on the environment.

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