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Performance Evaluation of Reactivated Spent Bleaching Earth on Color Reduction of Crude Palm Oil

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ABSTRACT
This study aims to evaluate the performance of reactivated Spent Bleaching Earth (SBE) in reducing the color of Crude Palm Oil (CPO). SBE was reactivated through chemical activation using HCl and H_2SO_4 in single, mixed, and sequential modes, followed by calcination at 600°C, producing Reactivated Spent Bleaching Earth (RSBE A–K) samples. The reactivated materials were characterized using Scanning Electron Microscopy (SEM) to evaluate their surface morphology before being applied to the CPO bleaching process and compared with fresh Bleaching Earth (BE). The results showed that the initial CPO color of 22.0/22.0 (red/yellow) was optimally reduced to 7.6/7.6 using RSBE I, outperforming fresh BE, which reduced the color to 11.0/11.0. RSBE I also exhibited the highest performance among all samples. SEM characterization showed that the reactivation process produced a cleaner surface morphology, supporting the improved adsorption performance of RSBE. These findings demonstrate the potential of reactivated SBE as an efficient and sustainable alternative adsorbent for the palm oil refining industry.

Keyword: Crude Palm Oil, Reactivation, Spent Bleaching Earth

ABSTRAK
Penelitian ini bertujuan untuk mengevaluasi kinerja *Spent Bleaching Earth* (SBE) yang direaktivasi terhadap penurunan warna *Crude Palm Oil* (CPO). SBE direaktivasi melalui aktivasi kimia menggunakan HCl dan H_2SO_4 secara tunggal, campuran, dan bertahap, diikuti kalsinasi pada 600°C, menghasilkan sampel *Reactivated Spent Bleaching Earth* (RSBE A–K). Material hasil reaktivasi dikarakterisasi menggunakan *Scanning Electron Microscopy* (SEM) sebelum diaplikasikan pada proses bleaching CPO dengan menggunakan *Bleaching Earth* (BE) segar sebagai adsorben pembanding. Evaluasi dilakukan berdasarkan penurunan warna sebagai indikator utama efisiensi pemucatan. Hasil menunjukkan warna awal CPO 22,0/22,0 (*red/yellow*) dapat diturunkan optimal menjadi 7,6/7,6 menggunakan RSBE I, lebih baik dibanding BE segar yang mencapai 11,0/11,0. RSBE I juga menunjukkan kinerja tertinggi di antara seluruh sampel. Karakterisasi SEM menunjukkan bahwa proses reaktivasi menghasilkan morfologi permukaan yang lebih bersih sehingga mendukung peningkatan kinerja adsorpsi RSBE. Hasil penelitian ini menunjukkan bahwa SBE yang direaktivasi berpotensi menjadi adsorben alternatif yang efisien dan berkelanjutan dalam industri pemurnian minyak sawit.

Kata Kunci: *Crude Palm Oil*, Reaktivasi, *Spent Bleaching Earth*

1. Introduction

Bleaching Earth (BE) is a widely used adsorbent in palm oil refining processes, particularly during the Crude Palm Oil (CPO) bleaching stage, due to its ability to remove unwanted compounds such as pigments (carotenoids and chlorophyll), trace metals, phospholipids, and oxidation products [1]. As the production of crude palm oil (CPO) in Indonesia increased to 50.07 million tons in 2023 [2], the demand for Bleaching Earth (BE) has also risen, leading to a corresponding increase in waste generation in the form of Spent Bleaching Earth (SBE). This waste is classified as hazardous and toxic waste (B3) due to its residual oil content of 20–40% (w/w), which poses potential environmental impacts [3-4].

The management of SBE has become a significant challenge because, in addition to its environmental impacts, it also requires high handling and disposal costs. One promising approach is the reactivation of SBE to restore its adsorption capacity, allowing it to be reused in the bleaching process [5]. Reactivation methods can be carried out through chemical treatment, thermal treatment, or a combination of chemical activation and calcination. Chemical activation commonly uses strong acids such as HCl and H_2SO_4, while calcination is performed at high temperatures to improve the surface characteristics of the adsorbent [6-7].

Previous studies have shown that reactivated SBE has the potential to exhibit performance comparable to fresh Bleaching Earth (BE). However, its effectiveness strongly depends on the activation method and operating conditions used [8]. Therefore, further investigation is required to evaluate the effect of variations in chemical activation on adsorption performance in the CPO bleaching process.

Based on these considerations, this study focuses on evaluating the performance of reactivated SBE prepared using a combination of chemical activation and calcination at 600°C. The adsorbent performance was assessed based on its ability to reduce the color of CPO compared to fresh Bleaching Earth (BE). This study is expected to contribute to the development of more efficient and sustainable alternative adsorbents in the palm oil refining industry.

2. Material and Method

2.1. Equipments

The equipment used in this study included a Soxhlet extractor, analytical balance, beaker glass, hot plate with magnetic stirrer, furnace, oven, filter paper, Lovibond tintometer, glass cell, and desiccator. These were used for sample preparation, reactivation, bleaching, and color analysis.

2.2. Materials

The materials used in this study included Spent Bleaching Earth (SBE) obtained from a palm oil refinery industry and fresh Bleaching Earth (BE) as a reference adsorbent. Crude Palm Oil (CPO) was used as the feedstock for the bleaching process. Hydrochloric Acid (HCl) and Sulfuric Acid (H_2SO_4) were used as chemical activating agents for the reactivation process.

2.3. Pretreatment

Spent Bleaching Earth (SBE) used in this study was obtained from bleaching unit of an oleochemical industry located in the Sei Mangkei Special Economic Zone, North Sumatra, Indonesia. The SBE had been used once as an industrial adsorbent before being collected for reactivation. Prior to reactivation, the SBE was extracted using the Soxhlet method with *n*-hexane to remove the residual oil retained in the adsorbent, then dried before further treatment.

2.4. Experimental Design of SBE Reactivation

Reactivation of Spent Bleaching Earth (SBE) was carried out using chemical and thermal treatments based on different experimental conditions. The reactivation scheme and sample variations are presented in Table 1.

Table 1. Experimental design of SBE reactivation

Code	Activation Type	Acid Composition	%
SBE A	Chemical	HCl	20
SBE B	Chemical	H_2SO_4	20
SBE C	Chemical	HCl : H_2SO_4 (1:1)	10 : 10
SBE D	Chemical	HCl → H_2SO_4	20 : 20
SBE E	Chemical	H_2SO_4 → HCl	20 : 20
SBE F	Physical	-	-
SBE G	Chemical + Physical	HCl	20
SBE H	Chemical + Physical	H_2SO_4	20
SBE I	Chemical + Physical	HCl : H_2SO_4 (1:1)	10 : 10
SBE J	Chemical + Physical	HCl → H_2SO_4	20 : 20
SBE K	Chemical + Physical	H_2SO_4 → HCl	20 : 20

2.5. Chemical Activation

Chemical activation of SBE was carried out using HCl and H_2SO_4, applied in single, mixed, and sequential modes. The SBE was contacted with acid solution at a solid-to-liquid ratio of 1:5 (w/v) and soaked for 24 hours at room temperature. For sequential activation, the process was conducted in two stages, each lasting 12 hours, followed by washing with distilled water until neutral pH (±6–7) and drying at 105 °C to constant weight.

2.6. Thermal Activation (Calcination)

Thermal activation was performed through calcination of chemically treated SBE samples. The samples were heated in a furnace at 600 °C for 90 minutes. After calcination, the furnace was allowed to cool to approximately 100 °C before the samples were removed. The samples were then cooled in a desiccator to room temperature to prevent moisture adsorption prior to further use.

The activation conditions used in this study, including a 20% acid concentration, 24 h activation period, and calcination at 600 °C for 90 min, were adopted from the method reported by [3]. These conditions were selected because they have been demonstrated to effectively regenerate spent bleaching earth through the removal of residual oil and impurities while improving its adsorption performance.

2.7. SEM Characterization

The surface morphology of fresh Bleaching Earth (BE), Spent Bleaching Earth (SBE), and RSBE I, which exhibited the highest bleaching performance, was characterized using Scanning Electron Microscopy (SEM). The analysis was conducted to observe the morphological changes after the reactivation process.

2.8. Bleaching Test

A 100 mL sample of Crude Palm Oil (CPO) was heated to 105 °C. Subsequently, Reactivated Spent Bleaching Earth (RSBE) or fresh Bleaching Earth (BE) was added at 2 wt % of the CPO mass. The bleaching process was conducted under atmospheric pressure with continuous stirring at 105 °C for 30 min to ensure sufficient contact between the oil and the adsorbent. After the bleaching process, the adsorbent was separated from the oil by filtration using filter paper. Fresh Bleaching Earth (BE) was used as the reference adsorbent for comparison with the performance of RSBE

2.9. Color Analysis [9]

Crude Palm Oil (CPO) was ensured to be free from any sediment and completely in liquid form. The sample was then poured into a clean and dry glass cell until full, ensuring no air bubbles were present. The Lovibond tintometer was switched on and the internal lighting was stabilized prior to analysis. The glass cell containing the sample was placed inside the Lovibond tintometer. The color of the sample was matched using a combination of red (R), yellow (Y). The color ratios were adjusted until a visual match was achieved. The final results were recorded and expressed in Lovibond Units (LU).

The bleaching test was conducted once for each treatment condition. The Lovibond color of each bleached oil sample was measured in duplicate using a Lovibond Tintometer, and the reported color values represent the average of the duplicate measurements. Bleaching power was calculated based on the average Lovibond color values.

3. Result and Discussion

3.1. Oil Recovery

Prior to the reactivation process, Spent Bleaching Earth (SBE) underwent Soxhlet extraction using n-hexane to remove the residual oil retained in the adsorbent. As presented in Table 2, the initial residual oil content of SBE was 23.01%, indicating that a considerable amount of oil remained trapped on the surface and within the pores of the adsorbent after the industrial bleaching process. Such residual oil may block the active surface and reduce the effectiveness of the subsequent reactivation process [10].

Table 2. Residual oil content of SBE before and after oil recovery

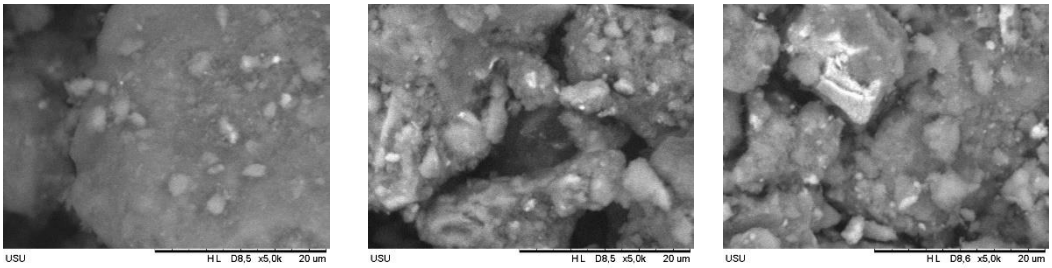
Sample	Residual Oil (%)
SBE before oil recovery	23.01
SBE after oil recovery	0.21

After the oil recovery process, the residual oil content decreased significantly to 0.21%, corresponding to an oil removal efficiency of approximately 99.09%. This result indicates that Soxhlet extraction effectively

removed most of the retained oil, thereby exposing the adsorbent surface for subsequent chemical activation and calcination. The removal of residual oil is expected to facilitate better interaction between the adsorbent and the activating acid, leading to more effective regeneration of the adsorption properties of SBE[11], [12].

3.2. SEM Characterization

Scanning Electron Microscopy (SEM) analysis was performed to observe the surface morphology of fresh Bleaching Earth (BE), Spent Bleaching Earth (SBE), and RSBE I, which exhibited the highest bleaching performance among all reactivated samples. The morphological observations were used to evaluate the effect of the reactivation process on the surface characteristics of the adsorbent and to relate these changes to its bleaching performance.



(a) (b) (c)

Figure 1. SEM micrographs of (a) fresh BE, (b) SBE, and (c) RSBE I at 5000× magnification.

Figure 1 presents the SEM micrographs of fresh BE, SBE, and RSBE I at a magnification of 5000×. Fresh BE (a) exhibited a relatively clean and homogeneous surface morphology, indicating that the adsorbent had not yet been exposed to residual oil, pigments, and other impurities during the bleaching process. In contrast, SBE (b) displayed a rougher and more compact surface morphology with noticeable particle agglomeration. This morphological change is likely associated with the accumulation of residual oil and impurities on the adsorbent surface after its use in the bleaching process, which may reduce the accessibility of adsorption sites.

Following chemical activation and calcination, RSBE I (c) exhibited a cleaner and less compact surface morphology than SBE. The reduction of surface deposits indicates that the reactivation process effectively removed a portion of the residual oil and impurities retained on the adsorbent surface, thereby partially restoring its surface characteristics. Although the surface morphology of RSBE I had not completely returned to that of fresh BE, the reactivation process noticeably improved the cleanliness of the adsorbent surface. The cleaner surface morphology observed in RSBE I is in agreement with previous findings reported by [3] and [13], who reported that acid activation and subsequent thermal treatment facilitated the removal of surface contaminants, thereby restoring the adsorbent surface and improving its adsorption performance.

3.3. Color Reduction Performance

Color reduction of Crude Palm Oil (CPO) was evaluated using Lovibond color values (red and yellow) as the main indicator of bleaching performance. The performance of Reactivated Spent Bleaching Earth (RSBE) was compared with fresh Bleaching Earth (BE) to assess the effectiveness of the reactivation process in improving adsorption capacity. The reduction in color intensity after bleaching indicates the ability of the adsorbent to remove pigments and other color forming compounds present in CPO. The resulting oil after the bleaching process is referred to as Bleached Palm Oil (BPO). The visual appearance of CPO and BPO is shown in Figure 2.

From the figure 2, a clear color change can be observed between the initial CPO and BPO using both BE and RSBE. The initial CPO exhibits a darker color, while after the bleaching process, a decrease in color intensity is observed, indicating a decolorization process.The color results of CPO and Bleached Palm Oil (BPO) using BE and RSBE are presented in Table 3. The data show a reduction in color values after the bleaching process compared to the initial CPO. These differences indicate the effectiveness of pigment adsorption by each adsorbent.

Figure 2. Visual appearance of Crude Palm Oil (CPO) before bleaching and Bleached Palm Oil (BPO) obtained using fresh Bleaching Earth (BE) and Reactivated Spent Bleaching Earth (RSBE A–K). Samples are arranged from left to right as CPO, BE, and RSBE A–K.

The observed color reduction is closely related to the ability of the adsorbent surface to adsorb carotenoids and other color-forming compounds present in CPO. Therefore, differences in bleaching performance among the RSBE samples reflect differences in the effectiveness of the reactivation treatments in restoring the adsorption capability of spent bleaching earth.

Table 3. Lovibond Color of CPO and BPO

Sample	R	Y
CPO	22.0	22.0
BE	11.0	11.0
RSBE A	15.0	15.0
RSBE B	13.0	13.0
RSBE C	14.5	14.5
RSBE D	16.0	16.0
RSBE E	12.0	12.0
RSBE F	14.4	14.4
RSBE G	10.0	10.0
RSBE H	10.6	10.6
RSBE I	7.6	7.6
RSBE J	8.8	8.8
RSBE K	8.2	8.2

The identical Lovibond Red (R) and Yellow (Y) values presented in Table 3 represent the actual readings obtained for each sample using the Lovibond Tintometer. Therefore, the reported values reflect the direct measurement results under the experimental conditions.

The initial CPO showed a Lovibond color value of 22.0/22.0 (R/Y), indicating a high content of color pigments in the oil. After bleaching using fresh BE, the color value decreased to 11.0/11.0, indicating that BE has good performance in reducing the color intensity of CPO.

For the use of RSBE, the color reduction results varied, with values ranging from 16.0 to 7.6. All RSBE samples showed the ability to reduce the color of CPO compared to the initial condition. Among all RSBE samples, RSBE I exhibited the best performance with the lowest color value of 7.6/7.6, even lower than that obtained using BE. Meanwhile, several other RSBE samples showed lower performance compared to BE. These results are in agreement with the study by [1], which also reported that RSBE exhibited better bleaching performance than BE, where the color value of BE was 18/79 (R/Y), while RSBE achieved a lower value of 16/76.

3.4. Effect of Reactivation Method

The bleaching performance of RSBE was strongly influenced by the reactivation method applied. Different activation treatments resulted in varying color reduction performances, indicating that both chemical activation and calcination play important roles in restoring the adsorption capability of spent bleaching earth [14].

Acid activation plays an important role in regenerating spent bleaching earth by removing residual oil, pigments, and inorganic impurities that remain attached to the adsorbent surface after the bleaching process. Hydrochloric Acid (HCl) is effective in dissolving inorganic impurities and metal-containing compounds, whereas Sulfuric Acid (H_2SO_4) facilitates the decomposition of organic residues and enhances the acidic characteristics of the adsorbent surface. Consequently, the effectiveness of the reactivation process depends

not only on the type of acid used but also on the interaction between chemical activation and subsequent thermal treatment [3], [11], [15].

RSBE samples that only underwent chemical activation (RSBE A–E) generally showed lower bleaching performance compared to samples that received combined chemical and thermal activation. Among the chemically activated samples, RSBE E showed the best performance with a color value of 12.0/12.0, while RSBE D exhibited the lowest performance with a value of 16.0/16.0. These results indicate that the type and sequence of acid treatment affected the effectiveness of the reactivation process [16].

The relatively lower bleaching performance observed in several chemically activated samples suggests that chemical activation alone was insufficient to completely regenerate the adsorption capability of SBE. Although acid treatment removed a portion of the retained impurities, residual organic compounds may still remain on the adsorbent surface, limiting its effectiveness during the subsequent bleaching process [17-18].

The physically activated sample (RSBE F) also showed relatively limited bleaching performance with a color value of 14.4/14.4. This suggests that thermal treatment alone was not sufficient to significantly restore the adsorption capacity of SBE [14]. Similarly, thermal activation alone mainly promotes the decomposition of residual organic compounds through calcination but is less effective in removing inorganic contaminants strongly bound to the adsorbent surface. Therefore, calcination without prior chemical treatment was unable to fully restore the adsorption performance of SBE [5], [8].

In contrast, RSBE samples treated through combined chemical activation and calcination (RSBE G–K) exhibited significantly better bleaching performance, with color values ranging from 10.6 to 7.6. The improvement in bleaching performance indicates that the combination of chemical treatment and thermal activation was more effective in regenerating the adsorbent properties of SBE [19].

Among all samples, RSBE I showed the best bleaching performance with the lowest Lovibond color value of 7.6/7.6. RSBE I was prepared using mixed acid activation (HCl: H_2SO_4, 1:1) followed by calcination at 600 °C. The combined acids likely enhanced the removal of both inorganic impurities and residual organic compounds from the adsorbent surface, while calcination further eliminated remaining organic matter and promoted the regeneration of the adsorbent surface. This mechanism is consistent with the SEM observations, which showed a cleaner surface morphology for RSBE I than for SBE, indicating successful removal of surface deposits during the reactivation process. Similar findings have been reported by [3] , who demonstrated that acid activation combined with thermal treatment effectively regenerated spent bleaching earth and improved its adsorption performance.

Although RSBE I exhibited a lower Lovibond color value than fresh Bleaching Earth (BE), the color values reported in this study represent Bleached Palm Oil (BPO) obtained after the bleaching stage only. In industrial palm oil refining, the final product quality is typically evaluated after the complete refining process, including deodorization, according to customer or refinery specifications. Therefore, the present results primarily demonstrate the comparative effectiveness of the different reactivation methods rather than compliance with final industrial color specifications. Nevertheless, the superior performance of RSBE I indicates its potential for application as an alternative adsorbent in industrial bleaching processes.

3.5. Bleaching Power

The bleaching power of BE and RSBE was calculated based on the reduction in Lovibond color values after the bleaching process. The bleaching power was determined using the following Equation 1:

$$Bleaching\ Power\ (\%) = \frac{C_i - C_f}{C_i}\ x\ 100\% \qquad \text{(Equation 1)}$$

Where : C_i = Initial color of CPO
C_f = Final color after bleaching

Table 4. Bleaching Power

Sample	%
BE	50.00
RSBE A	31.82
RSBE B	40.91
RSBE C	34.09
RSBE D	27.27
RSBE E	45.45
RSBE F	34.55
RSBE G	54.55
RSBE H	51.82
RSBE I	65.45
RSBE J	60.00
RSBE K	62.73

The bleaching power values obtained are presented in Table 4. The results showed significant differences depending on the reactivation method applied to the spent bleaching earth. Fresh Bleaching Earth (BE) exhibited a bleaching power of 50.00%, indicating its effectiveness in reducing the color intensity of CPO. In comparison, several RSBE samples showed lower bleaching power values, particularly RSBE D with the lowest value of 27.27%, followed by RSBE A and RSBE C with values of 31.82% and 34.09%, respectively.

On the other hand, RSBE samples subjected to combined chemical and thermal activation generally demonstrated higher bleaching power compared to samples treated only by chemical or physical activation. RSBE G, RSBE H, RSBE I, RSBE J, and RSBE K all showed bleaching power values above 50%, indicating better adsorption performance than fresh BE.

Among all samples, RSBE I showed the highest bleaching power of 65.45%, followed by RSBE K and RSBE J with values of 62.73% and 60.00%, respectively. These results indicate that the combination of acid activation and calcination treatment produced the most effective reactivation method for restoring and improving the adsorption performance of SBE. These results are consistent with the study conducted by [3], which reported a bleaching power value of 72.92% for RSBE reactivated through combined chemical activation and calcination, even higher than that of fresh BE.

Overall, the bleaching power results are consistent with the Lovibond color reduction data, where samples with lower final color values also exhibited higher bleaching power. This confirms that the reactivation treatment significantly affected the bleaching efficiency of RSBE in the adsorption of color pigments from crude palm oil.

In the present study, the bleaching performance was evaluated based on Lovibond color reduction and bleaching power, as the primary objective was to compare the effectiveness of different SBE reactivation methods in reducing the color of crude palm oil. Although additional quality parameters, such as carotenoid content, Free Fatty Acid (FFA), peroxide value, and phosphorus content, would provide a more comprehensive evaluation of the bleaching process, these parameters were beyond the scope of the present study and are recommended for future investigation. Similarly, material recovery and mass loss during the acid activation, washing, and calcination processes were not evaluated. Future studies should include mass balance analysis to assess the economic feasibility and practical applicability of the proposed reactivation process.

From an environmental and economic perspective, the reactivation of spent bleaching earth (SBE) offers the potential to reduce solid waste generation and the consumption of fresh bleaching earth. However, the process also involves acid consumption, wastewater generation during washing, and energy use during calcination. Therefore, appropriate acid management, wastewater treatment, and energy-efficient calcination should be considered to improve the overall sustainability of the proposed reactivation process.

4. Conclusion

This study demonstrated that the reactivation method significantly affected the performance of SBE in the bleaching process of CPO. All RSBE samples were able to reduce the color of CPO, where the combination of chemical activation and calcination produced better results compared to single activation methods. RSBE I, reactivated using a mixed acid treatment of HCl:H_2SO_4 (1:1) followed by calcination at 600 °C, showed the

best performance with the lowest Lovibond color value of 7.6/7.6 (R/Y) and the highest bleaching power of 65.45%, even better than fresh BE. SEM characterization further showed that the reactivation process produced a cleaner surface morphology in RSBE I than in SBE, which was consistent with its improved bleaching performance. These findings indicate that SBE reactivation through combined chemical and thermal treatments has strong potential as an effective and sustainable alternative adsorbent in the palm oil refining process.

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6. Conflict of Interest

Authors declare no conflicts of interest.

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