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Research Article

Study of boiler ash as bio-adsorbent to decolorize palm oil mill effluent final discharge (POMEFD)

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Abstract
Palm oil mill effluent final discharge (POMEFD) has a persistent brown color, potentially affecting ecosystems and water quality if not properly treated before being discharged to the watercourse. Meanwhile, boiler ash from palm oil mills poses challenges in waste management due to unsustainable disposal practices. To address both issues, this study investigated the potential use of boiler ash as bio-adsorbent to decolorize POMEFD. The effects of adsorbent dosage (4-20% w/v) and contact time (5-45 min) were evaluated in the adsorption experiment. The color intensity of raw and treated POMEFD was measured using the UV-Vis spectrophotometer and tintometer. Furthermore, the morphological and elemental characteristics of the boiler ash were analyzed using field emission scanning electron microscopy (FESEM) coupled with energy dispersive X-ray spectroscopy (EDX). The highest color removal percentage of POMEFD was achieved at 78.4% using 20% w/v dosage, while 77.8% color removal was attained at 5 minutes of contact time for 12% w/v dosage. The Field Emission Scanning Electron Microscopy with Energy Dispersive X-ray Spectroscopy (FESEM-EDX) images revealed the porous structure of boiler ash, which became smoother and less porous after treatment. EDX analysis identified the presence of metal and oxygen elements in the boiler ash, suggesting the formation of metal oxides that facilitated the adsorption of POMEFD color. This finding was further supported by tintometer analysis, which showed a significant reduction in POMEFD color intensity. Brunauer–Emmett–Teller (BET) analysis was conducted to examine the surface characteristics of the boiler ash bioadsorbent. The initial surface area was measured at 5.09 m^2/g, which increased significantly to 39.51 m^2/g after POMEFD decolorization, indicating the adsorption of contaminants onto the bioadsorbent. These findings can enlighten palm oil industry producers in dealing with POMEFD and boiler ash waste and promote the reusability of industrial waste.

Keywords: adsorption, palm oil mill effluent final discharge, decolorization, boiler ash, waste management

Introduction

The palm oil industry in Malaysia is a significant global player, contributing over 29% of world palm oil production and 37% of world exports [1]. However, this industry commonly generates 0.31-0.35 tons of palm oil mill effluent (POME) for every ton of fresh fruit bunches [2] processed in the milling factory. Waste management is often seen as a serious and challenging environmental issue due to the increase in waste generation annually. In general, palm oil mills in Malaysia use ponding systems for POME treatment and successfully comply with the Environmental Quality (Prescribed Premises) (Crude Palm-Oil) Regulations 1977. However, the effluent in the final pond before discharge into the watercourse, known as palm oil mill effluent final discharge (POMEFD), still has an intense brownish color. It appears that the commonly used ponding system has an insignificant impact on the color removal. This could stem from excessive concentration of tannins, melanoidin and lignin compounds [3,4]. The physical appearance of POMEFD contributes to public concern as it is associated with environmental pollution [5]. The initial form of the color compound may not be cytotoxic or carcinogenic, but the decomposition of these compounds after discharge into the environment can cause the formation of aromatic amines that can induce cancer or tumors [6] in humans. According to Madaki and Seng [7], even after treatment, this effluent still contains significant amounts of organic matter, which is a food source for microorganisms. The growing microbial population will compete with other aquatic life for dissolved oxygen consumption. Thus, the decolorization of POMEFD before discharge into the watercourse using the adsorption approach is prominent.

Apart from POMEFD, this industry is also plagued by the generation of large amounts of boiler ash. It is a by- product of burning oil palm fibres, kernel shells and empty fruit bunches in the boiler at high temperatures for energy. As an underutilized waste, it is typically disposed of in landfills or used as fertilizer; however, the heavy metal content leads to inconsistent chemical stability [8]. Uncontrolled disposal of boiler ash can cause soil and groundwater contamination and contribute to more complex waste management problems. According to Wang et al. [9], boiler ash has potential as an adsorbent due to its porous nature and high content of oxide elements, such as silica and alumina, which can improve the adsorption efficiency of pollutants.

The outstanding performance of boiler ash as the adsorbent has been documented in the literature. It was used by Mayyoga et al. [10] to reduce fatty acids by 2.99% and water content by 0.26% in crude palm oil. Manikam et al. [11] reported that boiler ash is an effective, low-cost and eco-friendly adsorbent to remove chemical oxygen demand (COD), ammoniacal nitrogen, nitrate and phosphorous from sewage water at 63.39 mg/L, 6.01 mg/L, 0.63 mg/L, and 0.43 mg/L, respectively. On another note, Hamzah et al. [12] decolourized POMEFD using microwaved-boiler ash and acquired a color concentration of 19.20 ADMI, complying with Malaysia’s effluent discharge standard class A. Ghani et al. [13] treated POME with boiler ash and found that the material could reduce total suspended solids (41.38-42.06%), biochemical oxygen demand (39.83-42.87 mg/L), and COD (122.33-150.67 mg/L). Nevertheless, boiler ash has not received due attention from the palm oil millers to commercialize it as a prospective adsorbent in decolorizing POMEFD color due to lack of related studies.

Therefore, this study investigated the potential use of boiler ash in the POMEFD decolorization process. The performance of this bio-adsorbent was evaluated by varying adsorbent dosages and contact times and determining color removal efficiency using UV-Vis spectrophotometer and tintometer. The morphological and elemental characteristics of boiler ash were examined using scanning electron microscopy (SEM) and energy dispersive X- ray (EDX) before and after the adsorption process. This study is expected to provide a more sustainable solution in dealing with POMEFD and boiler ash waste and promote the reusability of industrial waste.

Materials and Methods

Collection of boiler ash and POMEFD samples

Boiler ash and palm oil mill effluent final discharge (POMEFD), as depicted in **Figure 1**, were collected from Sime Darby Guthrie Pagoh Palm Oil Mill, Johor. As can be seen from **Figure 1(b)**, the color of POMEFD remained even after undergoing biological treatments. The boiler ash was stored in a tightly closed container to prevent absorption of surrounding moisture and did not undergo pre-treatment.

(a) (b)

Figure 1. (a) Boiler ash; (b) Palm oil mill effluent final discharge (POMEFD)

Adsorption experiment

The adsorption experiment evaluated the performance of boiler ash in decolorizing POMEFD. Two parameters were selected: adsorbent dosage (4, 8, 12, 16, and 20% w/v) and contact time (5, 15, 25, 35, and 45 min). This experiment was divided into two stages, where contact time remained constant at 30 min in the first stage, and adsorbent dosage was varied. In the second stage, the dosage was fixed at 12% w/v, and the contact time was varied. The specific amount of adsorbent was added to a constant volume of POMEFD at 50 ml in the 100 ml beaker at room temperature without stirring and pH alteration. The pH of the raw POMEFD was 8.5. All samples were filtered using Whatman No.41 filter paper to avoid interference from adsorbent fines before analysis.

Analysis of samples

To further understand the behavior of boiler ash as the bio-adsorbent, field emission scanning electron microscopy (FESEM) (ZEISS, MERLIN) coupled with energy dispersive x-ray spectroscopy (EDX) was employed to observe its morphology and composition before and after treatment, respectively. The surface area of boiler ash was determined using Braeuer–Emmett–Teller (BET) surface area analyzer, micromeritics Tristar II-Mesopore. The color intensity of raw and treated POMEFD samples was analyzed using UV-Vis spectrophotometer (Agilent Cary 60) at 290 nm wavelength and manual tintometer (Lovibond) with 5 ¼-inch glass cell. From the UV-Vis measurement, the color absorbance of raw POMEFD was 2.8756. The color removal efficiency (%) of each sample at different conditions was calculated by the following equations:

$$\text{Color removal \%} = \frac{Abs_{before\ treatment} - Abs_{after\ treatment}}{Abs_{before\ treatment}} \times 100$$

where, $Abs_{before\ treatment}$ is the color absorbance of raw POMEFD and $Abs_{after\ treatment}$ is the color absorbance of treated POMEFD.

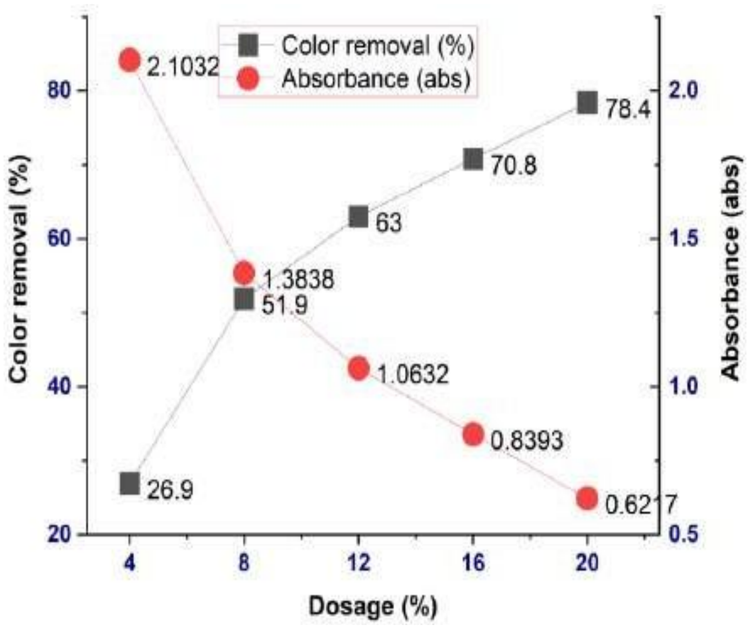

Figure 2. Effect of dosage on POMEFD brown color removal

Results and Discussion

Effect of adsorbent dosage on brown color removal

At constant contact time of 30 min, the effectiveness of boiler ash dosage on brown color removal indicated a significant positive correlation (**Figure 2**). Specifically, the color removal efficiency increased from 26.9% to 78.4% at boiler ash dosages between 4% and 20% w/v. This trend implied that higher adsorbent dosages may provide more surface areas and active sites for adsorption. The initial color removal efficiency was low at 4% w/v dosage, which was likely due to the limited interaction between the adsorbent and colorant molecules in the effluent. As the dosage increases, more binding sites are available, facilitating more effective adsorption of color compounds [14]. The observed color removal efficiency was aligned with the findings reported by Zainuddin et al. [15], in which the color pigment from the coating industry effluent (methylene blue) was significantly reduced by increasing the ratio of bio-adsorbent from sugarcane bagasse activated carbon.

Furthermore, the increment in color removal efficiency at higher dosages (16-20%) was smaller, with a difference of 7.6%, compared to the lower dosage (4-12%), which ranged from 11 to 20%. It suggests that the removal efficiency was less pronounced at higher dosages, particularly beyond 16%, demonstrating that the adsorption was approaching saturation. Moreover, at higher dosages, a significant portion of unutilized adsorbent particles can increase the risk of particle agglomeration, hindering the effective interactions between adsorbent and color molecules [16]. Similar finding has been reported in studies using biochar for organic pollutant removal, where lignin-rich effluent exhibited strong adsorption through hydrogen bonding, π–π stacking with carbonaceous adsorbent, van der Waals forces and physical interaction such as pore filling [17]. Additional adsorbent offers limited advancement in color removal, highlighting crucial optimum dosage to balance operational cost and treatment effectiveness, particularly in industrial applications. Ultimately, understanding these dynamics will enhance the practical application of boiler ash as a sustainable adsorbent in treating POMEFD.

In addition, **Figure 3** depicts the effect of boiler ash dosages on POMEFD color removal based on visual. It was explicit that effective color removal began from 12% w/v to 20% w/v boiler ash dosages. Therefore, 12% boiler ash dosage was selected for further study in the second stage to balance minimal operating costs and optimal treatment efficiency.

Effect of contact time on brown color removal

The effect of contact time on the POMEFD brown color removal efficiency exhibited a compelling trend at constant 12% w/v boiler ash dosage, as depicted in **Figures 4** and **Figure 5**. Based on **Figure 4**, the highest color removal (77.8%) was achieved within 5 min of contact time. However, the removal efficiency decreased to 72.2% when the contact time rose to 15 min and then remained at around 75% for the subsequent time intervals of 25 min (75.1%) and 35 min (75.3%). Notably, at 45 min, the color removal efficiency declined to 68.3% (**Figure 4**). Rapid initial color removal at 5 min suggested that a substantial quantity of the colorant was readily accessible for adsorption, with higher kinetics at the beginning of the process. This is supported by Marczewski et al. [18], who stated that adsorption occurs in a multi-stage kinetic process. The decrease in removal efficiency at longer contact times may be attributed to the saturation effect, where the adsorbent's active sites become occupied and the available binding sites for additional color molecules are diminished.

In addition, the desorption phenomena and competing reactions may be responsible for the decrease in color removal efficiency with prolonged contact time. Although strong adsorption occurred at 5 min of contact time, some color molecules may start desorbing into the solution. Weak interaction between the adsorbent and colorant could be the cause, particularly if the adsorption site loses its favorable characteristics [19]. Moreover, other compounds in POMEFD are also likely to interfere with the adsorption process. Over time, competitive interactions may diminish the overall efficiency of color removal by altering the equilibrium between the adsorbed color molecules and those in the solution. If the condition changes, for example, a decrease in dye concentration can lead to the desorption of previously adsorbed dye molecules back into the solution [14]. This trend underscored the importance of optimizing the contact time of the treatment process. Although longer contact times may allow for more thorough interactions, they can also reduce efficiency due to the factors mentioned earlier. Therefore, a contact time of 5 min was favorable for maximum POMEFD color removal using boiler ash as bio- adsorbent.

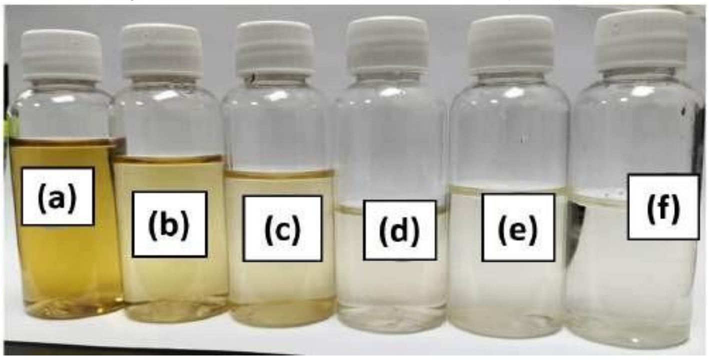

Figure 3. Visual representation of POMEFD decolorization as affected by different boiler ash dosages: (a) raw POMEFD, (b) 4% w/v, (c) 8% w/v, (d) 12% w/v, (e) 16% w/v, and (f) 20% w/v

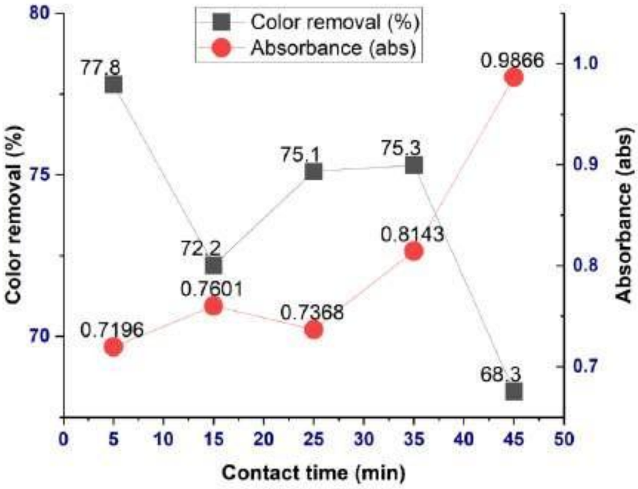

Figure 4. Effect of contact time on POMEFD brown color removal

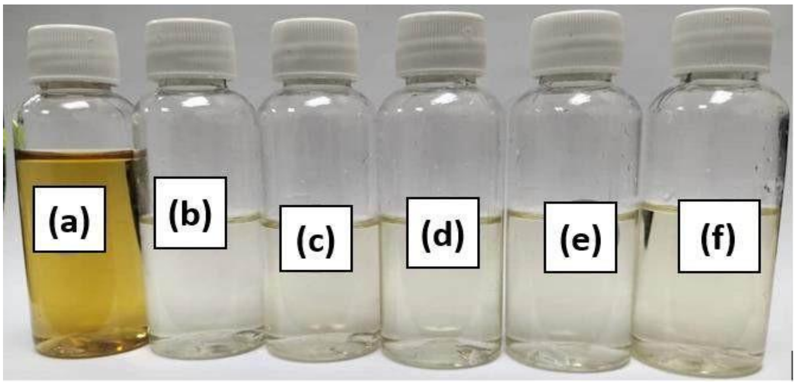

Figure 5. Visual representation of POMEFD decolorization as affected by contact time: (a) raw POMEFD, (b) 5 min, (c) 15 min, (d) 25 min, (e) 35 min, and (f) 45 min

Morphology and elemental characteristics

Figure 6 displays the surface morphology of the boiler ash before and after POMEFD decolorization treatment. In **Figure 6(a)**, the FESEM micrograph showed that the surface of the boiler ash was highly porous, sponge-like structure with numerous well-defined pores and cavities considering its adequate active sites for the adsorption of POMEFD. The uniform porosity and interconnected voids suggest that the material has undergone significant thermal treatment, leading to the formation of a highly developed pore network [20]. After the treatment, the boiler ash undergoes morphological transformations where the surface expanded, as shown by the arrow in **Figure 6(b)**. Increased contaminant accumulation caused changes in the surface morphology of the bio-adsorbent, leading to a smoother surface, swelling-like structure and reduce porosity. This proved that the boiler ash's pores interacted with POMEFD color molecules during adsorption. The morphological changes of boiler ash before and after adsorption of POMEFD are like those reported by Zainal et al. [21], demonstrating that the pores of bio- adsorbent were packed with contaminants and POMEFD constituents. Reduction in porosity after treatment indicates the capacity of ash for additional adsorption is diminished, suggesting the importance of regenerating or replacing the adsorbent for continued efficacy.

Furthermore, in **Figure 7**, the elemental composition from EDX analysis revealed that carbon (C) and oxygen (O) were the dominant elements before and after adsorption. However, the intensity of these two elements decreased after the adsorption process due to oxidation and chemical reactions involving carbon bonding in plant fiber that captured POMEFD impurities. The EDX analysis of boiler ash after adsorption exhibited that the peaks corresponding to elements such as magnesium (Mg), phosphorous (P), silicon (Si), calcium (Ca), potassium (K), and aluminum (Al), increased, possibly due to the adsorbed POMEFD impurity molecules [22]. Sequestration of inorganic element from the effluent might be due to ion exchange between cations from boiler ash are replaced by metal ions from the effluent. This mechanism is supported by studies showing a decrease in Cd (II) ion from aqueous solution by using white yam waste as bio-adsorbent via ion-exchange and adsorption complexation [23].

Moreover, according to EDX spectra, the presence of metals (Mg, P, Si, Ca, K and Al) alongside them with oxygen suggests that these elements are likely to exist in their oxides. Boiler ash is generally high in metal oxides due to the combustion process which promotes the formation of stable metal oxide rather than pure metals [24]. Metal oxide in boiler ash plays an important role in facilitating the adsorption process of lignin and tannin. The adsorption of negatively charged lignin and tannin molecules is enhanced by the surface charges created by metal oxides [25].

Tintometer analysis

The results of the tintometer analysis demonstrated a significant reduction in the color intensity of POMEFD after adsorption with boiler ash, as shown in **Table 1**. The POMEFD brown color was matched with red and yellow glasses, with red values from 2.9 to 3.9 and yellow values from 8.9 to 9.9. High red and yellow values are typically attributed to the effluent's residual organic and inorganic compounds, commonly associated with oil extraction by-products, mainly tannin and lignin [5]. In contrast, the treated effluent color improved significantly after adsorption with boiler ash. The red values were reduced to 0.4, while the yellow values decreased to 1.9. These findings implied that the boiler ash bio-adsorbent effectively removed the color-causing substances, leading to a clear effluent solution. This substantial color transformation was visually apparent and implied the removal of various impurities, which is a critical aspect of meeting effluent discharge standards.

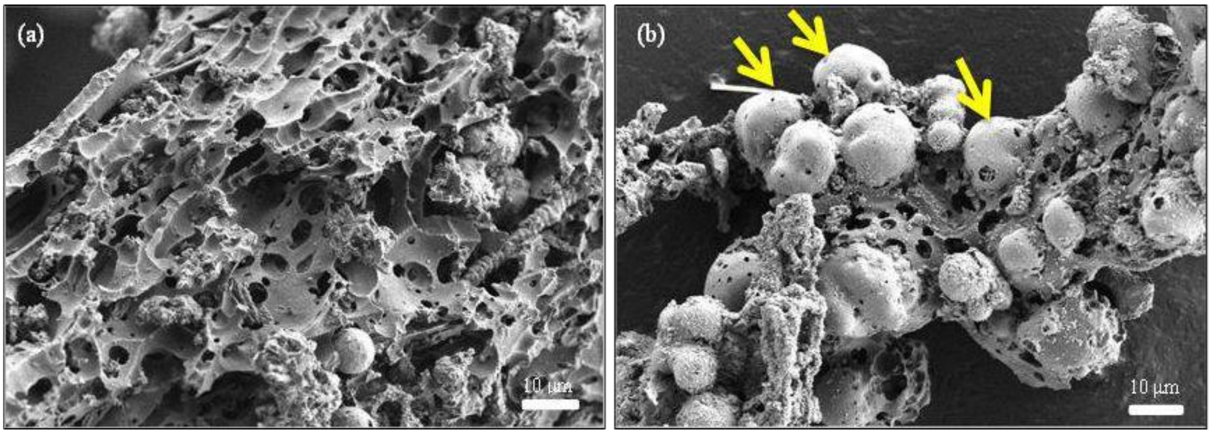

Figure 6. FESEM images of boiler ash bio-adsorbent (a) before and (b) after adsorption of POMEFD

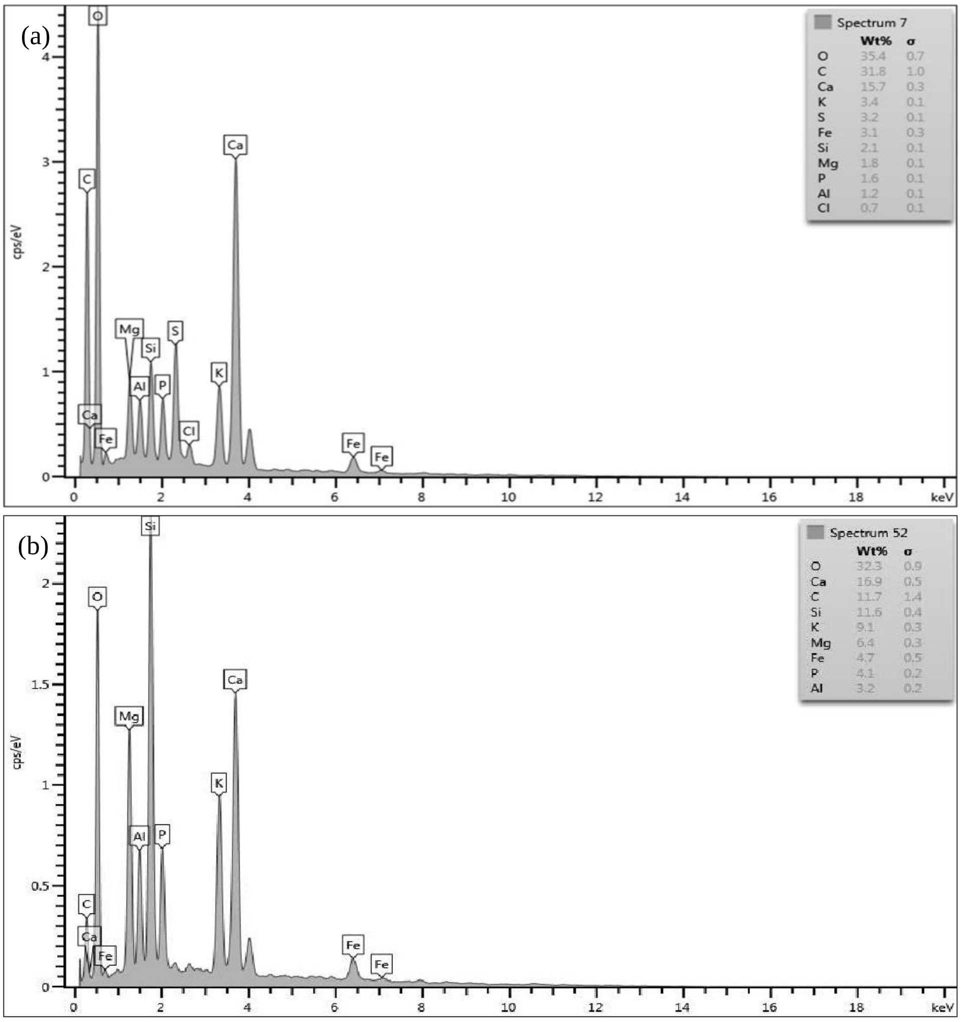

Figure 7. EDX spectra of boiler ash bio-adsorbent (a) before and (b) after adsorption of POMEFD

Table 1. Tintometer analysis of raw and treated POMEFD

Before treatment		**After treatment**	
Red	Yellow	Red	Yellow
2.9 – 3.9	8.9 – 9.9	0 – 0.4	1.9
			

Brunauer–Emmett–Teller (BET) analysis

The feature of porous structures for the obtained ash bioadsorbent was characterized by N_2 adsorption-desorption method. **Table 2** lists the particulate properties of the samples, such as specific surface area (SSA), pore size and volume distributions. A type IV curve with a comparable hysteresis loop was recorded in **Figure 8**. This finding suggests that ash bioadsorbent contains mesopores. Before decolorization wastewater treatment of POMEFD, the BET surface area of the ash bioadsorbent was found to be 5.09 m2/g, which is less than the 39.51 m2/g of ash bioadsorbent after decolorization wastewater treatment of POMEFD. This significant increase in BET surface area indicates that the decolorization wastewater treatment process enhances the porous structure of the ash bioadsorbent, resulting in improved adsorption capacity. The larger surface area allows for more active sites for adsorption, making the ash bioadsorbent more effective in removing contaminants from wastewater.

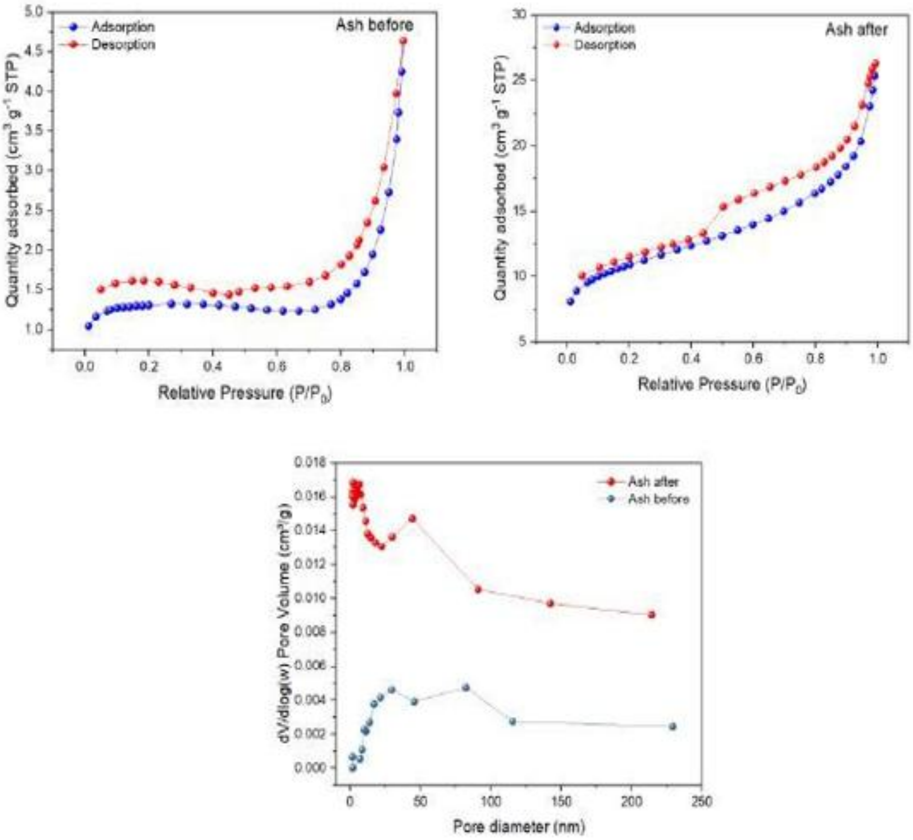

Figure 8. N_2 adsorption-desorption isotherms (a, and b) and pore size distribution (c) for ash before and after decolorization POMEFD wastewater treatment.

Table 2. Textural properties of ash bioadsorbent before and after decolorization wastewater treatment of POMEFD

Bioadsorbents	S_{BET} ($m^2 g^{-1}$)	Average pore diameter	Pore volume ($cm^3 g^{-1}$)
Ash before	5.09	3.34	0.0055
Ash after	39.51	3.23	0.0304

Conclusion

This study demonstrated the effectiveness of boiler ash as a bio-adsorbent for decolorizing palm oil mill effluent final discharge (POMEFD). The highest color removal efficiency of 78.4% was achieved using a 20% w/v adsorbent dosage, while the optimal contact time for decolorization was determined to be 5 minutes at a 12% w/v dosage, achieving 77.8% color removal. FESEM-EDX analysis revealed that the porous structure of boiler ash played a significant role in adsorption. BET analysis further supported these findings, with a significant increase in surface area from 5.09 m^2/g to 39.51 m^2/g after adsorption, indicating efficient pollutant capture. Additionally, tintometer analysis confirmed a substantial reduction in POMEFD color intensity after treatment. Overall, boiler ash demonstrated potential as a sustainable and effective bio-adsorbent for treating POMEFD. Future studies should investigate the underlying adsorption mechanisms over time and assess the potential to regenerate the adsorbent to maintain its efficiency in prolonged applications.

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