Utilization of Petroleum Sludge Waste as an Al-Fe Oxide/C Adsorbent and Its Characterization

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Abstract. This research addresses the conversion of hazardous petroleum oil sludge (POS) into an Al-Fe Oxide/C adsorbent via sulfuric acid (H_2SO_4) activation and hydrothermal treatment. Leveraging the inherent C (51.7%), Al (1.6%), and Fe (10.8%) content in the waste, the study aims to synthesize an effective adsorbent for methylene blue (MB) and eriochrome black T (EBT) removal. Characterization using SEM-EDX and FTIR confirmed the formation of active acid sites. The results showed total acidity of 5.6338 mmol/g. Adsorption performance was optimized based on pH and contact time, following the Langmuir-Freundlich isotherm model. This study demonstrates a sustainable approach to B3 waste management by producing a high-value adsorbent for industrial wastewater treatment.

Keywords: Oil sludge, Al-Fe oxide/C, adsorbent, methylene blue, eriochrome black T, hydrothermal synthesis.

1 Introduction

The petroleum industry remains the backbone of global energy supply; however, its operations simultaneously generate massive quantities of hazardous waste streams annually [1]. Among these waste fractions, Petroleum Oil Sludge (POS) is recognized as one of the most toxic and challenging byproducts due to its complex and persistent composition of hydrocarbons, water, and solids, and its classification as Hazardous and Toxic Material (B3) due to high levels of polycyclic aromatic hydrocarbons (PAHs) and heavy metals like iron (Fe) and aluminum (Al) [2,3]. The sheer volume of this waste, such as the estimated 51,000 cubic meters produced annually in Indonesia [4], necessitates urgent action. Improper disposal methods, like landfilling or land farming, severely

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threaten the environment by damaging soil properties and allowing PAHs and hydrocarbons to leach into groundwater, thus compromising aquatic ecosystems and public health [5,6]. Consequently, there is an imperative for innovative and sustainable waste management focusing on valorizing POS by transforming it into functional, value-added materials [7].

In response to the imperative for sustainable POS management, valorization into high-performance adsorbents presents a highly promising solution, offering significant advantages over disposal [8,9]. The adsorption process is widely recognized as a relatively efficient and economically viable technique for waste water purification due to its low operating costs [10]. This study specifically focuses on removing synthetic dyes, such as Methylene Blue (MB) and Eriochrome Black T (EBT), which are major, persistent contaminants from the textile industry known for their toxic, mutagenic, and carcinogenic properties [11]. Although research has explored the valorization of POS into activated carbon [12], this approach exhibits significant limitations: the resultant material often displays a relatively low adsorption capacity, and critically, its functionality typically proves effective for adsorbing only a single type of pollutant charge [13]. Considering that real industrial wastewater contains complex mixtures of both cationic and anionic pollutants simultaneously, a critical scientific gap remains: the necessity of developing a truly multifunctional adsorbent capable of optimal, simultaneous removal of both charge types.

This study, therefore, addresses this critical gap by proposing the synthesis of an Al-Fe Oxide/C adsorbent from POS via a hydrothermal route. The primary novelty of this research lies in leveraging the synergistic effect between the carbon matrix (derived from POS) and the dual active sites provided by the inherent iron (Fe) and added aluminum (Al) oxides, which are specifically designed to achieve optimal, simultaneous adsorption of both cationic and anionic pollutants. Based on this, the research aims are two-fold: to synthesize and comprehensively characterize the novel Al-Fe Oxide/C adsorbent from POS utilizing a hydrothermal method, and to systematically evaluate its adsorption performance in removing Methylene Blue and Eriochrome Black T, thereby determining the optimal conditions (such as pH, contact time, and concentration). The findings of this work are expected to provide in-depth insights into the adsorption mechanisms on waste-derived, multi-site materials, thus contributing significantly to sustainable environmental remediation and materials science.

The petroleum industry generates substantial quantities of Petroleum Oil Sludge (POS) waste, reaching 51,000 m^3 annually in Indonesia. Although previous studies have explored the conversion of POS into activated carbon, these methods are often limited by low adsorption capacity and are typically effective for only a single type of pollutant charge. This study offers a novel approach using a hydrothermal method compared to conventional thermal activation, ensuring a more uniform distribution of active sites between the carbon matrix and Al-Fe metal oxides. The low-temperature hydrothermal process (180°C) is more environmentally friendly and efficient in pore formation compared to high-temperature pyrolysis. Consequently, this work addresses a significant research gap concerning multifunctional adsorbents capable of simultaneously removing cationic and anionic pollutants through the synergistic effects of dual active sites.

2 Methodology

2.1 Materials

The primary raw material, Petroleum Oil Sludge (POS), was obtained from PT Pertamina RU III Plaju, South Sumatra, Indonesia. Chemical reagents including Hydrochloric acid (HCl), Sulfuric acid (H_2SO_4 98%), Sodium hydroxide (NaOH), and demineralized water (H_2O) were used for activation and pH adjustment. Adsorbates used in this study were Methylene Blue (MB) and Eriochrome Black T (EBT), both analytical grades purchased from Merck.

2.2 Synthesis of Al-Fe oxide/C adsorbent

The synthesis followed a two-stage process: pre-treatment and hydrothermal activation. Initially, raw POS was calcined at 500°C to remove volatile organic compounds. The stabilized material was ground and sieved using a 200-mesh screen.

For the chemical activation, 10 g of the POS powder was mixed with 50 mL of demineralized water (solid-to-liquid ratio of 1:5) and 3 mL of H_2SO_4 98%. The mixture was stirred magnetically at 200 rpm for 1 hour at room temperature to ensure homogeneous acid distribution. The resulting slurry was transferred into a 100 mL stainless-steel hydrothermal autoclave and heated at 180°C for 12 hours. This temperature was chosen to facilitate the formation of the carbon-metal oxide framework. The solid product was filtered, washed to neutral pH, dried at 110°C, and finally calcined at 700°C for 3 hours to stabilize the Al-Fe Oxide/C phase [12]

2.3 Material characterization

Characterization was performed to evaluate the structural and chemical transformation of the POS into an adsorbent. Scanning Electron Microscopy-Energy Dispersive X-ray (SEM-EDX) was employed to observe surface morphology and elemental distribution (Al, Fe, and C). X-ray Diffraction (XRD) was used to identify phase changes from amorphous to crystalline metal oxides. Fourier Transform Infrared (FTIR) spectroscopy identified functional groups and peak shifts before and after adsorption.

2.4 Surface acidity and pHpzc analysis

The surface acidity was quantified using the base vapor adsorption method (Ammonia and Pyridine). 1.0 g of sample was exposed to the respective vapors in a closed desiccator for 24 hours, followed by oven drying at 110°C to remove physically trapped molecules. The increase in mass represented the concentration of acid sites (mmol/g).

The pH Point Zero Charge (pHpzc) was determined by adding 0.05 g of adsorbent into 25 mL of 0.1 M NaCl solution (ratio 1:500). The initial pH (pHi) was adjusted from 3 to 10 using 0.1 M HCl or 0.1 M NaOH. After shaking at 150 rpm for 24 hours, the final pH (pHf) was measured. pHpzc is defined as the point where ΔpH (pHf - pHi) equals zero [14].

2.5 Batch adsorption studies

Adsorption experiments were performed in a batch system using 25 mL of dye solution (60 mg/L) with 0.05 g of adsorbent. The effect of pH was investigated in the range of 3–10. Kinetic studies were performed at the optimum pH with contact times ranging from 10 to 210 minutes. Adsorption isotherms were evaluated by varying the initial concentration of MB and EBT from 10 to 100 mg/L.

Post-adsorption, the solution was filtered and analyzed using a UV-Vis Spectrophotometer at the maximum wavelength for each dye. The adsorption capacity (qe) was calculated, and the data were fitted to Pseudo-first-order and Pseudo-second-order kinetic models, as well as Langmuir and Freundlich isotherm models to determine the maximum adsorption capacity and the nature of the interaction (monolayer or multilayer) [16, 17].

3 Result and discussion

3.1 Material characterization

The characterization of the calcined oil sludge powder was essential to confirm its suitability for composite synthesis and adsorption. SEM-EDX analysis, presented at 500x magnification in **Figure 1**, revealed a rough surface morphology with an irregular and uneven particle distribution. The particle structure appeared coarse, lacking visible pores at this magnification, though pores are reportedly identifiable at a higher magnification (160, 000x) [18].

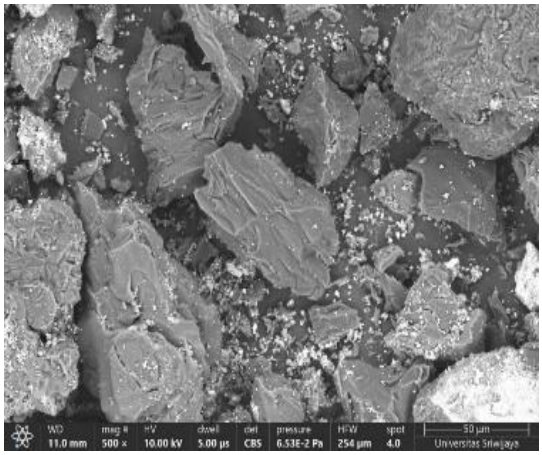

Fig. 1. SEM-EDX test results of petroleum sludge powder at 500x magnification

The elemental composition, summarized in **Table 1**, showed that the powder was predominantly composed of Carbon (C) at 63.4% At and 51.7% Wt. Furthermore, high Oxygen (O) content (26.8% Wt) indicated the presence of metal oxide compounds formed during the calcination process.

Crucially, the detection of Iron (Fe) (2.9% At, 10.8% Wt) and Aluminum (Al) (0.9% At, 1.6% Wt) confirmed that the waste possesses inherent metal oxides capable of acting as active sites in the final Al-Fe/C adsorbent [19].

Table 1. Elemental components of petroleum sludge powder

Component	Percentage weight (%Wt)
C	51.7
N	5.6
O	26.8
Na	0.2
Al	1.6
Si	2.0
S	1.3
Fe	10.8

3.2 Surface acidity and FTIR spectroscopy analysis

The acid properties and specific surface area were analyzed using the base vapor adsorption method, providing critical insights into the material's pore structure and active sites. Ammonia (0.26 nm) was used to determine the total acidity due to its small molecular size, which allows deep penetration into the pores, whereas pyridine (0.57 nm) was used to measure surface acidity [20,21]. The analysis revealed a total acidity of 5.6338 mmol/g. This value was significantly higher than the measured surface acidity (0.646 mmol/g), which indicated that the majority of acid sites (4.9878 mmol/g) are located within the internal pore structure.

This finding was further corroborated by the specific surface area measurements, where the ammonia-based surface area (8.8180 m^2/g) was substantially greater than the pyridine-based area (2.2166 m^2/g, confirming the oil sludge's potential as a mesoporous material. Furthermore, FTIR spectroscopy (**Figure 2**) confirmed the functional nature of these sites. The analysis confirmed the existence of Brønsted acid sites (~1643 cm^{-1} from pyridine interaction) and Lewis acid sites (~ 1604 cm^{-1} from ammonia interaction). The presence of these combined acid sites is vital, as they enhance the material's ability to facilitate diverse electrostatic and chemical interactions with various dye molecules.

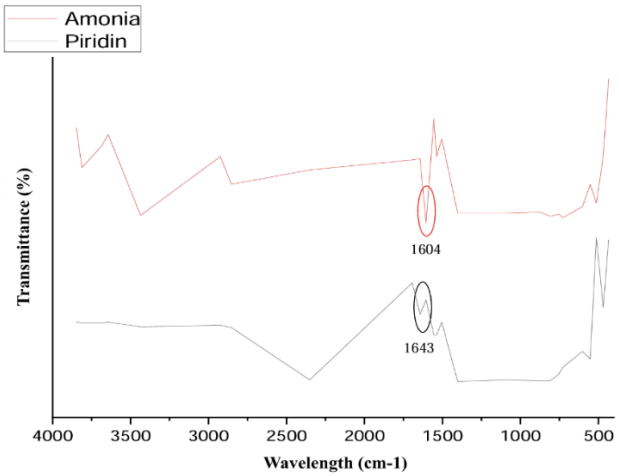

Fig. 2. FTIR spectra of petroleum sludge powder using ammonia and pyridine

3.3 PH pzc analysis

The synthesis of the Al-Fe Oxide/C adsorbent was executed via chemical activation (H_2SO_4) and subsequent hydrothermal treatment. Initial calcination of the oil sludge at 500°C for 6 hours was necessary to remove volatile organic components. The chemical activation, utilizing 98% H_2SO_4, aimed to enlarge the active sites and increase the material's acidity by forming sulfonate groups (-SO_3H) through interaction with carbon [12]. The hydrothermal process, conducted at 180°C for 12 hours, was critical for promoting a more uniform crystalline structure and evenly distributing the Al and Fe oxides within the carbon matrix. Following this, a final calcination at 700°C for 3 hours was performed to eliminate acid residues and enhance the interaction between carbon and metal oxides, yielding a more stable structure. This entire activation process resulted in a mass reduction from 10 grams to 7.540 grams, primarily due to the loss of volatile compounds and organic residues [22].

The operational efficiency of the Al-Fe Oxide/C adsorbent was initially assessed by determining the optimal solution pH, which directly controls the electrostatic interactions. The adsorption capacity exhibited a strong and inverse dependence on the charge of the dye and the surface of the adsorbent, confirming the dominance of the electrostatic mechanism. For the Cationic dye Methylene Blue (MB), maximum capacity (27.4457 mg/g) was achieved at pH 7 (**Figure 4**).

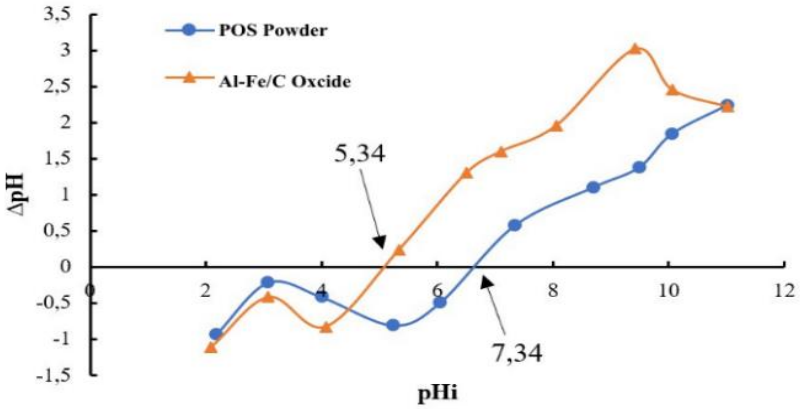

Fig. 3. pH pzc POS powder and Al-Fe oxide/C

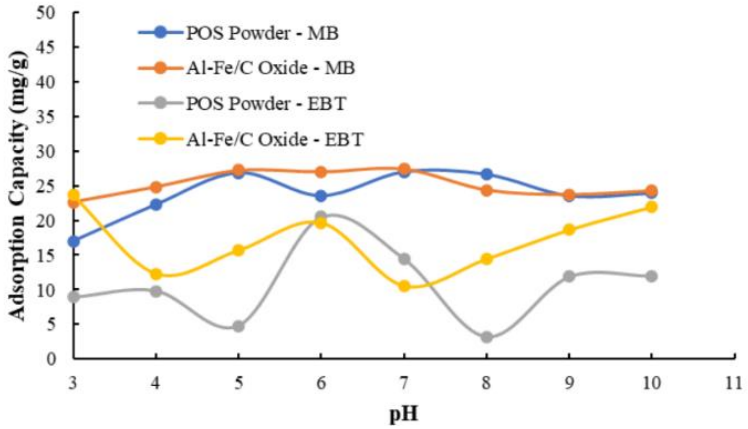

Fig. 4. Effect of initial pH on the adsorption capacity (qe) of MB and EBT

This finding is fully consistent with the material's surface being negatively charged when the operating pH (7.0) is greater than the pHpzc (5.34) (**Figure 3**), resulting in the strongest attraction for the positive MB ions. Conversely, for the Anionic dye Eriochrome Black T (EBT), the highest capacity (23.662 mg/g) was recorded at pH 3 (**Figure 4**). At this acidic pH, the adsorbent surface maintains a positive charge (pH < pHpzc) (**Figure 3**), facilitating optimal removal of the negative EBT ions. These results clearly demonstrate that the synthesized Al-Fe Oxide/C adsorbent is capable of controlling the selective adsorption of both cationic and anionic pollutants simply by adjusting the solution pH.

3.4 Adsorption performance

The optimization of contact time was crucial for determining the operational efficiency and underlying kinetic mechanisms of the adsorbents. The kinetic study established that the Al-Fe Oxide/C adsorbent exhibited superior kinetics compared to the petroleum oil sludge. For the cationic dye Methylene Blue (MB), the Al-Fe Oxide/C reached equilibrium faster (120 minutes) compared to the POS powder (210 minutes). Similarly, for the anionic dye Eriochrome Black T (EBT), the Al-Fe Oxide/C reached its maximum capacity (25.915 mg/g) at 180 minutes, significantly reducing the required contact time compared to the POS powder (210 minutes). This consistent trend demonstrates that the chemical and hydrothermal treatments successfully increased the available active sites and accelerated the mass transfer rate, confirming the enhanced efficiency of the synthesized material. **Figure 5** visually illustrates the difference in equilibrium time and capacity between the two materials.

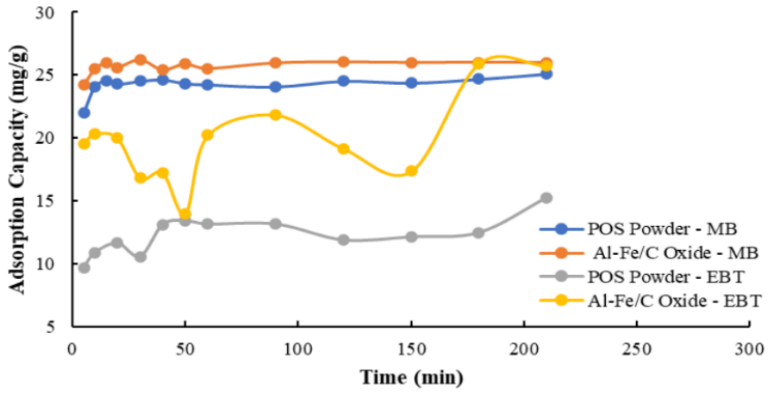

Fig. 5. The effect of contact time on the adsorption capacity (qe) of MB and EBT

Kinetic modeling for both MB and EBT on the Al-Fe Oxide/C was best described by the Pseudo-Second-Order model (R_2 = 1 for MB; R_2 = 0.9179 for EBT). The strong adherence to the PSO model suggests that the adsorption rate-limiting step is controlled by chemisorption, involving valence forces and electron exchange at the active sites. The specific kinetic parameters are detailed in **Table 2** and **Table 3**. The Al-Fe Oxide/C showed a superior rate constant (k_2) (0.0712 g/mgmin) and higher equilibrium capacity (qe) (26.04 mg/g) for MB compared to the POS powder. Although the POS powder exhibited a higher k_2 for EBT, the Al-Fe Oxide/C achieved a substantially higher overall capacity (24.44 mg/g VS. 13.69 mg/g on POS powder). This increase in total binding capacity confirms that the synthesis successfully augmented the number of available active sites [23].

Table 2. Kinetic model of methylene blue dye adsorption with POS powder and Al-Fe oxide/C

Kinetic Model	POS Powder	Al-Fe Oxide/C
Pseudo-Orde 1		
Equation	y = -0.0118x + 0,4876	y = 0.0039x + 0.4506
R^2	0.42696	0.7052
k_1	0.0272 min^{-1}	0.0089 min^{-1}
Pseudo-Orde 2		
Equation	y = 0.0402x + 0.0409	y = 0.0384x + 0.0207
R^2	0.9996	1
k_2	0.0394 g/mg.min	0.0712 g/mg.min
qe	24.87 mg/g	26.04 mg/g

The investigation into the effect of initial concentration was conducted to determine the maximum adsorption capacity (q_{max}) and to elucidate the equilibrium mechanism. The capacity for the cationic dye Methylene Blue (MB) increased with concentration, reaching a maximum capacity of 39.380 mg/g on the Al-Fe Oxide/C at 90 mg/L, slightly higher than the 36.815 mg/g achieved by the POS powder. Conversely, for the anionic dye Eriochrome Black T (EBT), the Al-Fe Oxide/C reached its maximum capacity (6.936 mg/g) at a lower optimal concentration (70 mg/L) compared to the raw sludge (6.091 mg/g at 100 mg/L). This phenomenon suggests that the smaller pore structure resulting from the activation process may limit optimal performance at very high concentrations. The relationship between concentration and capacity is visually presented in **Figure 6**.

Table 3. Kinetic model of eriochrome black T dye adsorption with POS powder and Al-Fe oxide/C

Kinetic Model	POS Powder	Al-Fe Oxide/C
Pseudo-Orde 1		
Equation	y = -0.0132x + 1.1161	y = 0.0008x + 1.0223
R^2	0.3746	0.1827
k_1	0.0303 min^{-1}	0.0018 min^{-1}
Pseudo-Orde 2		
Equation	y = 0.073x + 0,3735	y = 0.0409x + 0.5857
R^2	0.9757	0.9179
k_2	0.073 g/mg.min	0.0028 g/mg.min
qe	13.69 mg/g	24.44 mg/g

Isotherm modeling was crucial to validate the material's novelty as a dual-site adsorbent. The adsorption data for MB on the Al-Fe Oxide/C was best described by the Freundlich isotherm model (R2 = 0.9491), which suggests a multilayer adsorption process occurring on a heterogeneous surface. This mechanism consistent with MB utilizing the bulk, irregular carbon matrix of the synthesized material. In sharp contrast, EBT adsorption was best fitted by the Langmuir isotherm model (R2 = 0.9221).

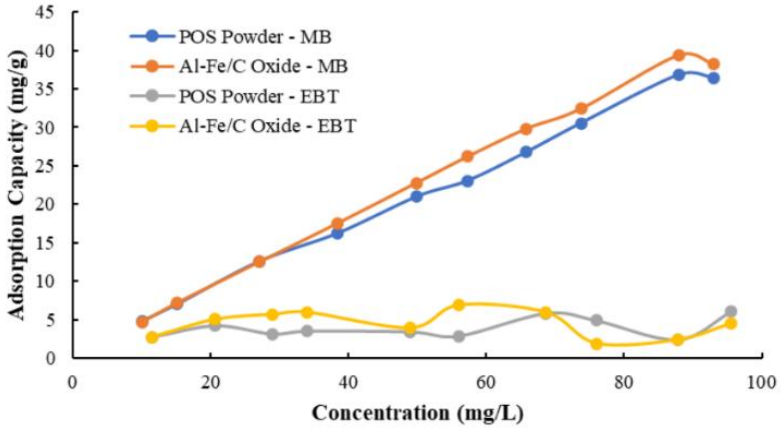

Fig. 6. Effect of initial concentration on the adsorption capacity (qe) of MB and EBT

The Langmuir model implies monolayer adsorption on a homogeneous surface, strongly suggesting that EBT primarily interacts with the uniformly distributed Al/Fe oxide active sites. This divergence in optimal isotherm models provides the core evidence that the Al-Fe Oxide/C successfully facilitates specialized dual active sites for both cationic and anionic pollutants, overcoming the single-charge limitation of conventional POS-derived carbon adsorbents. The isotherm constants are summarized in **Table 4** and **Table 5**.

Table 4. Adsorption isotherm of methylene blue dye with POS powder and Al-Fe oxide/C

	Langmuir Isotherm			Freundlich Isotherm		
	R^2	K_L	q_m	R^2	K_F	n
POS Powder	0.845	0.152	44.642	0.9676	7.426	1.897
Al-Fe Oxide/C	0.9325	6.4293	56.497	0.9491	7.610	1.465

Table 5. Adsorption isotherm of eriochrome black T dye with POS powder and Al-Fe oxide/C

	Langmuir Isotherm			Freundlich Isotherm		
	R^2	K_L	q_m	R^2	K_F	n
POS Powder	0.8362	0.054	6.666	0.5932	1.720	3.938
Al-Fe Oxide/C	0.9221	0.709	4.837	0.2166	12.246	6.422

4 Conclusion

This study concludes that POS is a highly effective precursor for a new Al-Fe Oxide/C adsorbent, contributing significantly to hazardous waste valorization. The practical implication includes an eco-friendly solution for textile dye removal in industrial settings. Future research should focus on the regeneration capability of this adsorbent and BET surface area measurements.

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