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Adsorptive removal of iron and manganese from acid mine drainage by low-cost materials

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ABSTRACT

This study investigated the effectiveness of a composite low-cost adsorbent, comprising coconut shell-based activated carbon, zeolite, and claystone, in removing iron (Fe) and manganese (Mn) from acid mine drainage (AMD) generated by coal mining activities. Batch adsorption experiments were conducted at laboratory scale to evaluate the influence of contact time and adsorbent dosage. The adsorbents were immersed in AMD samples with an initial pH of 2.6 and stirred under controlled conditions. The removal efficiencies were measured over time, and kinetic modeling was applied to assess the adsorption mechanism. The composite adsorbent successfully increased the AMD pH from 2.6 to 6.3 and achieved a 58.73% improvement within the first 5 minutes. Iron concentration was reduced from 13.006 ppm to 0.021 ppm with 2.5 g of adsorbent in 15 minutes, corresponding to a 99.84% removal efficiency. Manganese removal was less consistent, achieving a maximum reduction of 53.42% under the same conditions. Adsorption capacities for Fe and Mn were 1.299 mg/g and 1.487 mg/g, respectively. Kinetic analysis indicated that Fe removal followed a pseudo-second-order model, suggesting chemisorption, while Mn removal exhibited a slower, less predictable behavior. The results highlighted the composite's strong potential as a rapid and environmentally friendly treatment option for AMD, particularly for effective iron removal. The adsorbents demonstrated significant efficiency in Mn removal under the tested conditions, although with less consistency than Fe. This approach offers a sustainable solution using locally available materials, contributing to improved water quality management in mining-affected areas.

Keywords: *Acid mine drainage, Adsorption, Composite adsorbent, Fe, Mn.*

1. Introduction

Due to its negative impacts on the environment and humans, acid mine drainage (AMD) has become a severe global issue (Kefeni et al., 2017). Acid mine drainage is a significant environmental issue, characterized by high acidity and elevated concentrations of dissolved potentially toxic elements (PTEs), such as iron and manganese (Córdoba & Sarmiento, 2023). Acid mine drainage (AMD) represents a serious environmental problem worldwide, particularly in coal-producing regions such as Southeast Asia, where high rainfall accelerates pyrite oxidation and metal leaching. Acid mine drainage is characterized by low pH and elevated concentrations of potentially toxic elements (PTEs), including iron (Fe) and manganese (Mn), which contaminate surface and groundwater, harm aquatic life, and pose risks to human health. The majority of AMD is produced as a by-product of several mining and related industrial activities (Rambabu et al., 2020). Pyrite is the primary trigger of sulfide mineral oxidation and AMD generation. Additionally, the exposure and oxidation of sulfide-bound minerals or rocks frequently produce acidic streams with high sulfate contents. The interactions between the acidic water and the coal or mineral deposits result in the release of hydrogen ions and heavy metal ions, leading to the characteristic high acidity and metal content of acid mine drainage (Tang et al., 2021). In many mining and mineral refining sectors, AMD or acid rock drainage (ARD) existence is widespread. Due to local geology, microclimate, a particular collection of microorganisms, and a water source, drainage composition might vary widely from one area to another (Simate & Ndlovu, 2014). The AMD poses a major risk of underground and surface water contamination, which could have fatal consequences due to its high quantities of dissolved metalloids and metals, very acidic pH, and large volumes of sulfate. Long-term effects include a degradation in the aquatic ecology and a loss of biodiversity (Mulopo, 2015; Ngure et al., 2014). Acid mine drainage will cause several environmental issues, including contamination of water bodies and soil, and negative impacts on aquatic ecosystems (Anawar, 2013; Tang et al., 2021). Globally, AMD impacts water bodies, soil quality, and ecosystems, posing severe risks to biodiversity and human health. In both industrialized and developing countries, addressing AMD has become a priority, as the ecological and economic impacts of untreated AMD are extensive, requiring long-term remediation and monitoring. Potentially toxic elements (PTEs) including Fe, Al, Cu, Ca, Mg, Mn, and Zn are the primary elements found in acid mine drainage (Rodríguez-Galán et al., 2019). In practice, preventing the generation of AMD is highly challenging (Chen et al., 2021). As a result, remediation techniques have been developed over the past several years to get rid of metals. The treatment of acid mine drainage is crucial to mitigate its detrimental impact on the surrounding ecosystem and water resources.

The technologies for treating acid mine drainage that is most commonly known include neutralization, built wetland methods, adsorption methods, microbiological methods, ion exchange methods,

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membrane technology, and microbial fuel cell technologies. (Tong et al., 2021). Several treatments for acid mine drainage are membrane processes, adsorption, electrochemical processes, and selective metal precipitation (Kuyucak, 1998; Rodríguez-Galán et al., 2019). Conventional AMD treatment methods, such as chemical precipitation, ion exchange, and membrane filtration, often involve high operational costs and complexity, limiting their practical use, particularly in remote or resource-limited regions. In contrast, adsorption is widely recognized as a practical, low-cost, and environmentally friendly alternative for removing metal ions from wastewater. This technique relies on the use of sorbent materials that can capture contaminants either on their surface or within their porous structures. Since adsorption can recover 99% of the metals, it is the most often used economical treatment. (Rodríguez & Leiva, 2020). The use of adsorption techniques has been extensively applied for adsorbing potentially toxic elements (PTEs) from wastewater, mostly due to their cost-effectiveness, abundance availability of adsorbents, and their environmentally sustainable nature (Renu et al., 2017). The process of adsorption occurs when a substance, the adsorbate (being adsorbed), accumulates either on the surface of a substance or within the pores of a solid substance (Kennedy et al., 2018). The varieties and quantities of adsorbents present in nature are quite abundant, making adsorption one of the passive treatment strategies that should be properly considered.

Due to its low cost, wide availability of adsorbents, and environmental friendliness, the adsorption technique has been widely utilized to remove potentially toxic elements (PTEs) from wastewater (Renu et al., 2017). Adsorption looks to be a favorable method when compared to alternative treatment options since it is quick, easy, and inexpensive to remove potentially toxic elements (PTEs) from aqueous acid mine drainages (Bahadir et al., 2007). The use of adsorbent and other low-cost alternatives has been utilized for heavy metal removal. Due to its simplicity and cost-effectiveness, the adsorption method is now regarded as a suitable technique for treating wastewater (Scharnberg et al., 2020).

In recent years, the use of natural and locally available materials as adsorbents has gained increasing attention. Materials such as activated carbon, zeolite, and clay-based minerals offer promising adsorption properties due to their large surface area, high cation-exchange capacity, and chemical stability. Carbon, peat, clay, zeolite, bark, lignin, chitosan, dead biomass, agricultural waste, and fly ash are a few of these inexpensive sorbents (Cabrera et al., 2005).

Indonesia possesses a wealth of natural mineral resources, such as activated carbon derived from coconut shells, zeolite, and claystone, which have potential as low-cost adsorbents for potentially toxic elements (PTEs) removal (Yu et al., 2017). Natural materials available in Indonesia that can be used as adsorbents for potentially toxic elements (PTEs) include zeolite, clay, fly ash, and bottom ash. These adsorbents can effectively absorb potentially toxic elements (PTEs) such as lead (Pb), iron (Fe), cadmium (Cd), chromium (Cr), zinc (Zn), and nickel (Ni) (M Kadja & Mualliful Ilmi, 2019).

Zeolite, an aluminosilicate mineral with a crystalline structure, possesses a high cation-exchange capacity and has demonstrated effective adsorption of potentially toxic elements (PTEs). The study demonstrated the suitability of the zeolite utilized for the removal of Cu2+, Pb2+, and Cd2+ ions from synthetic wastewater (Elboughdiri, 2020), and is also effective in adsorbing potentially toxic elements (PTEs) from acid mine drainage (Motsi et al., 2009). In Indonesia, natural zeolite is abundant and has been identified as a viable material for heavy metal removal, including Fe and Cu, from both synthetic and actual AMD samples (M Kadja & Mualliful Ilmi, 2019).

Claystone, a sedimentary rock composed of clay minerals, is another locally abundant material with great potential as a heavy metal adsorbent. Claystone has a great adsorption capability for some potentially toxic elements (PTEs) dissolved in water, which can be utilized to remove potentially toxic elements (PTEs) from wastewater (Musso et al., 2014). Claystone, especially from overburden material in coal mines, offers promising sorptive characteristics due to its aluminosilicate composition.

Activated carbon, particularly that derived from coconut shells, has gained significant attention in water treatment due to its large surface area, porous structure, and ease of chemical activation. Because of its relatively large surface area, stable structure, variety of functional groups, simplicity of modification, and capacity for regeneration, activated carbon (AC) has received considerable attention in water treatment applications. Activated carbon is typically found in two different forms: granular and powdered (GAC and PAC) (Jaber et al., 2022). The predominant constituent of coconut shell activated charcoal is carbon, accounting for approximately 74.3% of its composition. Activated charcoal derived from coconut shells has the capacity to effectively adsorb many metals, including copper (Cu), iron (Fe), zinc (Zn), and lead (Pb) (Bernard et al., 2013).

Recent studies have increasingly explored the potential of low-cost, locally available adsorbents for the treatment of acid mine drainage (AMD), particularly for the removal of iron (Fe) and manganese (Mn), two common and problematic contaminants. Mukarrom et al. (2020) investigated the adsorption potential of claystone, quartz, and coconut shell charcoal, demonstrating their efficacy in removing potentially toxic elements (PTEs) from AMD generated in coal mining environments. Similarly, Nursanto and Pradise (2021) reported that natural adsorbents, especially those derived from local Indonesian resources, can effectively reduce iron concentrations in AMD through relatively simple adsorption systems. Paradise et al. (2022) demonstrated that a composite of claystone, zeolite, and coconut shell activated carbon was effective in removing Fe and Mn from AMD, with a 25:25:50 ratio resulting in 98.86% Fe and 33.69% Mn removal efficiency.

Complementing these findings, Pranoto et al. (2020) tested the effectiveness of a quartz sand and coconut shell charcoal composite for Mn adsorption, showing promising results in both adsorption efficiency and material characterization, while more recent work by Anifah et al. (2024) using salacca peel-based activated carbon achieved 80% Fe and 24% Mn removal at a dose of 0.8 g/100 mL and 60 minutes of contact time. These studies collectively support the feasibility and effectiveness of combining claystone, zeolite, and activated carbon as sustainable, low-cost adsorbents for AMD treatment, highlighting their relevance in Indonesian mining. However, the combined use of these three components as a composite sorbent has received limited attention. Thus, investigating a composite made from coconut shell activated charcoal, zeolite, and claystone sourced from coal overburden may offer a promising, low-cost alternative for the adsorptive removal of Fe and Mn from AMD. This study focuses on the development and evaluation of a composite adsorbent composed of activated coconut shell charcoal, zeolite, and claystone sourced from coal overburden.

The integration of multiple low-cost adsorbents into a composite, especially from locally available sources for real AMD samples are limited and relatively underexplored. Given the heterogeneity and complexity of AMD chemistry in real mining environments, such investigations are crucial to bridge the gap between laboratory findings and field applications. The novelty of this research lies in examining the efficiency of this multi-component low-cost adsorbent under different sorption conditions, specifically adsorbent dosage and contact time, for the removal of Fe and Mn from coal-derived AMD. The best composite was made with 50% coconut shell activated charcoal, 25% zeolite, and 25% claystone (Paradise et al., 2022).

The current study aims to fill this gap by evaluating a composite adsorbent comprising activated carbon from coconut shells, natural zeolite, and claystone from coal overburden materials that are abundant and underutilized in Indonesia. This study investigated the removal of iron (Fe) and manganese (Mn) from acid mine drainage collected from coal mining areas using a composite adsorbent composed of activated coconut shell charcoal, zeolite, and claystone. The adsorbent's performance is evaluated for Fe and Mn removal under varying contact times and dosages using a batch system. This approach not only addresses the need for sustainable AMD treatment options but also contributes to resource valorization of mining and agricultural by-products.

2. Materials and methods

This study used experimental design and was carried out on a small scale in the laboratory. A hot plate stirrer was utilized in a batch system for the adsorption process. Batch system adsorption refers to the process of immersing the adsorbent in a solution containing potentially toxic elements (PTEs), with the purpose of analyzing the subsequent changes in quality over a specific time interval (Somerville, 2007).

2.1. Materials

The research utilized clay from coal mine overburden In Bontang East Kalimantan, zeolite, coconut shells, HCl p.a. (Merck), NaOH p.a. (Merck), and distilled water. Materials needed to make an adsorbent can be seen in Figure 1.

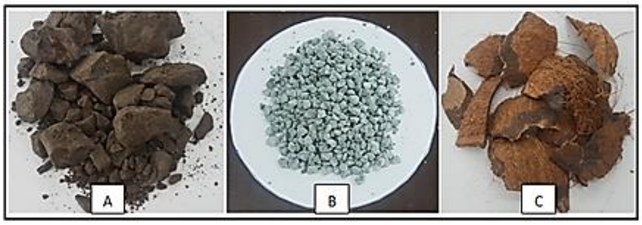

Fig. 1. Materials adsorbent. A) Clay, B) Zeolite, C) Coconut shell.

2.2. Procedure

1. A sampling of acid mine drainage

The water sample of acid mine drainage (AMD) was collected from a sump pit of coal mining. AMD was retrieved from a coal mine in East Kalimantan.

2. Lab analysis

Iron and manganese concentrations in AMD were measured using Atomic Absorption Spectroscopy (AAS).

2.3. Making Adsorbent

After sampling, the samples of claystone, zeolite, and coconut shells were physically and chemically activated to maximize their capacity to absorb potentially toxic elements (PTEs) (Mukarrom et al., 2020; Nursanto & Pradise, 2021; Nwosu et al., 2018; Paradise et al., 2022; Pranoto et al., 2020). Heat was applied to the materials to achieve physical activation at a specific temperature (Claystone can be heated to 700°C in 30 minutes, zeolite to 80°C in 24 hours, and coconut shell charcoal to 110°C in 3 hours). The materials were chemically activated by immersing them in chemical solution (for claystone, used 3M NaOH, for zeolite used 3M HCl, and for coconut shell charcoal used 4M HCl). After that, clay, zeolite and activated coconut shell charcoal were characterized by Surface Area Analyzer (SAA). Adsorbent composed of activated claystone, activated zeolite, and activation of carbon obtained from coconut shell in 25:25:50 ratio. The adsorbent is 100 mesh in size.

2.4. Adsorption analysis

In order to conduct the adsorption process, 250 ml of AMD sample was prepared in a beaker glass along with composite (2.5 g, 5 g, and 7.5 g), Figure 2. The AMD and composite were then mixed in the beaker glass by controlling the rotational speed and temperature of a hot plate stirrer for 5, 10, and 15 minutes. After the adsorption process was complete, the acid mine drainage was filtered using filter paper, and the pH level was assessed. After adsorption, the remaining concentration of iron and manganese was determined by performing another AAS analysis on the acid mine drainage. The following equation [1] was used to compute the amount of iron and manganese ions adsorbed per composite mass unit (adsorption capacity) (mg/g) and adsorption effectiveness (%) (Yang et al., 2022).

$$\frac{x}{m} = \frac{(Co-Ce).V}{W} \quad (1)$$

$$Q = \frac{(Co-Ce)}{Co} \; x \; 100\% \quad (2)$$

where Ce is the equilibrium concentration of Fe and Mn in acid mine drainage, x/m is the adsorption capacity, Q is the adsorption percentage or effectiveness, Co is the initial concentration, V is the acid mine drainage volume in liters, and W is the mass of the composite.

Fig. 2. Adsorbent.

3. Result and discussion

3.1. Analysis of acid mine drainage

According to the laboratory analysis, initial concentrations of Fe and Mn:

Fe =13.006 ppm, Mn =30.59 ppm and pH=2,6. It doesn't fulfill the required standard for coal mining in Indonesia. In Minister of Environment Decision No. 113 of 2003 about Quality Standards for Wastewater for Coal Mining Businesses and/or Activities, the quality standards for pH are 6-9, Fe 7 ppm, and Mn 4 ppm.

3.2. Adsorbent characterization

3.2.1. Surface Area Analyzer (SAA)

The specific surface area and pore volume of the adsorbent materials were determined using the Brunauer–Emmett–Teller (BET) method, a widely accepted technique for evaluating the physical characteristics of porous materials. Table 1 presents notable differences among the tested adsorbents: activated clay, activated zeolite, and activated charcoal, in terms of their surface area and pore structure.

Table 1. Surface area of the materials.

Materials	Surface area (m²/g)	Pore volume (cc/g)
Activated Clay	22.786	0.00655
Activated Zeolite	36.3334	0.01308
Activated charcoal	116.070	0.0419

Activated charcoal exhibited the highest surface area at 116.070 m²/g and the largest pore volume of 0.0419 cc/g. This indicates its superior textural properties, providing a large number of active sites and greater accessibility for contaminant molecules, particularly heavy metals, during the adsorption process. Activated zeolite followed with a surface area of 36.3334 m²/g and a pore volume of 0.01308 cc/g. While lower than activated charcoal, these values still reflect its potential for effective adsorption, especially given its ion-exchange capacity and surface chemistry. Activated clay recorded the lowest surface area of 22.786 m²/g and a pore volume of 0.00655 cc/g, suggesting more limited adsorption capability compared to the other two materials.

The considerable variations in the surface area and porosity of these adsorbents affect their adsorption performance. The surface area is directly proportional to the adsorption capacity because metal ions claim more sites than are available on the surface of the adsorbent. Also, a higher pore volume allows better diffusion and access of the ions into the inner part of the adsorbent. These characteristics are essential in processes such as PTEs extraction from acid mine drainage (AMD) because, in this case, the physical and chemical properties of the adsorbent control process efficiency. Furthermore, the high porosity and extensive surface area of activated charcoal make it particularly well-suited for multi-metal adsorption applications, as it can effectively accommodate ions such as Fe^{2+}, Mn^{2+}, Cd^{2+}, Pb^{2+}, and Cr^{3+}. In comparison, while activated zeolite and clay have lower surface areas, tailored mineral fragments and specific surface properties of these materials can confer selectivity or advantages in particular ion-exchange or adsorption processes. Thus, understanding the textural properties of surface area and pores is essential not only for assessing the performance of individual adsorbents but also for investigating the synergistic effects in the use of composite adsorbents.

3.2.2. X-Ray Fluorescence (XRF)

The elemental composition of the adsorbents was analyzed using X-Ray Fluorescence (XRF) to determine the dominant oxides present in each material. This compositional data provides valuable insight into the chemical structure and potential reactive sites for adsorption processes, especially for metal ion removal from acid mine drainage (AMD). The XRF results, summarized in Table 2, reveal that the activated clay predominantly comprises SiO_2 (54%) and Al_2O_3 (26%). The relatively high content of aluminosilicate compounds in clay suggests its potential for ion exchange and surface complexation reactions, which are crucial in heavy metal adsorption.

Activated zeolite also exhibits a high concentration of SiO_2 (69%) and Al_2O_3 (17%), in addition to CaO (3.7%) and Fe_2O_3 (3.2%). The presence of CaO may enhance its cation-exchange capacity, as calcium can act as a bridging ion in removing divalent metal ions like Fe^{2+} and Mn^{2+}. The chemical profile of zeolite is consistent with its crystalline aluminosilicate framework, which is known for its porous structure and high affinity toward various metal ions. Activated zeolite has a higher K_2O content (3.00%) than clay, which may enhance its cation exchange capacity (CEC). This higher K_2O could improve zeolite's affinity for heavy metal ions due to additional exchangeable cations, making it suitable for adsorbing ions like Fe and Mn.

Table 2. Elemental composition of the adsorbent.

	Activated clay	Activated Zeolite	Activated charcoal
Na_2O	1.50%	1.10%	0.00%
MgO	2.70%	1.40%	2.60%
Al_2O_3	26.00%	17.00%	3.60%
SiO_2	54.00%	69.10%	17.90%
P_2O_5	0.50%	0.50%	3.30%
SO_3	1.50%	0.30%	1.80%
Cl	0.10%	0.20%	2.10%
K_2O	2.50%	3.00%	9.20%
CaO	1.30%	3.70%	46.20%
Sc_2O_3	0.00%	0.00%	0.30%
TiO_2	1.10%	0.30%	0.50%
V_2O_5	0.00%	0.00%	0.10%
MnO	0.10%	0.10%	0.60%
Fe_2O_3	8.60%	3.20%	10.80%
Rb_2O	0.00%	0.00%	0.40%
SrO	0.00%	0.10%	0.40%

In contrast, activated charcoal displays a significantly different elemental profile, with a dominant carbon content (not quantified by XRF) and relatively low oxide concentrations. The detectable oxides include SiO_2 (17.9%), likely originating from residual ash or mineral impurities. Activated charcoal stands out with a notably high CaO content (46.20%), significantly higher than clay and zeolite. High CaO can enhance the material's ability to neutralize acidic solutions, particularly useful for acid mine drainage (AMD) treatment where pH adjustment is essential. The high CaO content may also contribute to the charcoal's effectiveness in increasing pH levels during adsorption. Due to its primarily organic nature, the effectiveness of activated charcoal in adsorption is attributed more to its surface area and porosity than to its oxide composition. Activated charcoal has the highest Fe_2O_3 content (10.80%), which may provide specific binding sites contributing to its high Fe ions' adsorption efficiency. The presence of Fe_2O_3 could influence the magnetic properties of the adsorbent, possibly enhancing the removal efficiency of Fe from AMD through adsorption and potential redox interactions. The diverse elemental composition of these adsorbents points to their complementary roles in AMD treatment. Activated charcoal, with its high CaO and Fe_2O_3, demonstrates a strong capacity for pH adjustment and Fe removal, making it particularly effective in AMD scenarios where rapid Fe removal and pH stabilization are required. Activated zeolite, with high SiO_2 and K_2O, is optimal for cation exchange and can efficiently capture metal ions like Mn. Lastly, while lower in overall surface area, activated clay provides a stable Al_2O_3 structure that supports the adsorption of specific metal ions in wastewater. These results underline the distinct chemical environments offered by each adsorbent. The aluminosilicate content in clay and zeolite promotes ion exchange and chemisorption, while the largely carbonaceous structure of activated charcoal facilitates physisorption. Understanding these chemical characteristics is essential for optimizing adsorbent selection and designing composite materials tailored for specific contaminant removal from AMD.

3.2.3. SEM-EDX (Scanning Electron Microscopy-Energy Dispersive X-ray Spectroscopy)

The surface morphology of the adsorbent was characterized using a scanning electron microscope. The surface morphology of an adsorbent is a critical factor determining its effectiveness in adsorbing target substances. The adsorbents under discussion, activated clay, activated zeolite, and activated charcoal, are known for their high porosity due to many pores on their surface. Figure 3 shows many pores of the adsorbent's surface morphology. The high porosity of activated clay, activated zeolite, and activated charcoal provides a large surface area for adsorption, which increases their adsorption capacity and efficiency. Its high porosity is due to many pores on its surface, making it ideal for a wide range of applications, including water treatment by adsorption.

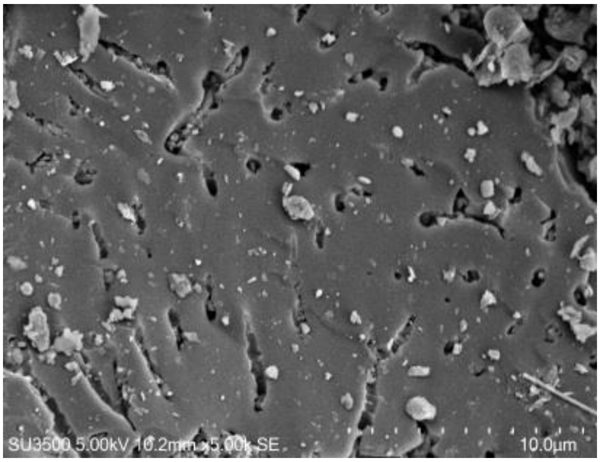

Fig. 3. Adsorbent's surface morphology.

Figure 4 shows that the elemental mapping reveals that carbon (C), oxygen (O), and silicon (Si) are the dominant elements, indicating the presence of functional groups on the surface of the adsorbent. This distribution highlights functional groups and surface-active sites, which are crucial for adsorption performance. The high initial concentration of carbon and oxygen suggests a high content of organic compounds and possibly hydroxyl or silanol groups, which could enhance the material's affinity for metal ions. The significant presence of silicon indicates the silicate structure of the adsorbent, which is especially relevant in zeolite and clay-based materials.

After adsorption, the SEM-EDX results show a significant change in the elemental distribution on the surface of the adsorbent (Figure 5).

The adsorbent's EDS spectrum shows a marked increase in the concentration of iron (Fe) after the adsorption process, indicating that the adsorbent is effective in removing iron from the solution. The adsorption process also led to a decrease in the concentration of other elements, such as carbon and oxygen, suggesting that some structural changes in the adsorbent accompany the adsorption of iron. The element percentage of the adsorbent is presented in Table 3.

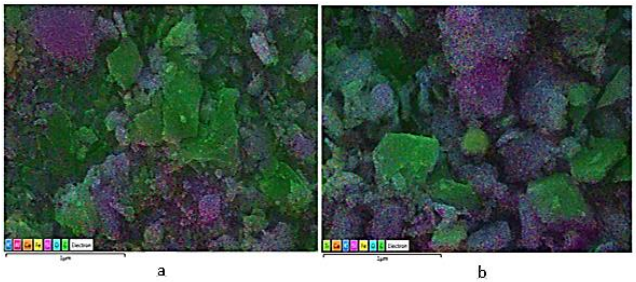

Fig. 4. Elemental mapping of the adsorbent a) before, b) after adsorption.

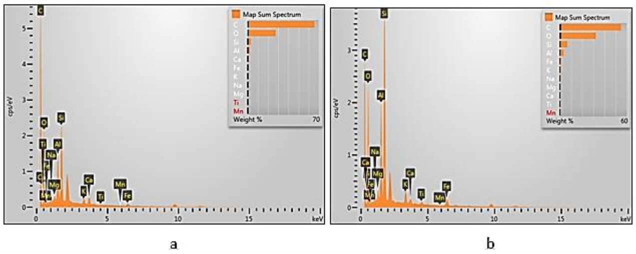

Fig. 5. EDS spectrum of the adsorbent a) before, b) after adsorption.

Table 3. Element percentage of the adsorbent.

Element	Before adsorption		After adsorption	
	% Weight	% Atomic	% Weight	% Atomic
C	65.63	73.69	54.86	65.09
O	27.46	23.15	32.16	28.64
Na	0.31	0.18	0.42	0.26
Mg	0.20	0.11	0.35	0.21
Al	1.92	0.96	3.36	1.77
Si	3.11	1.49	6.56	3.33
K	0.34	0.12	0.65	0.24
Ca	0.52	0.17	0.27	0.10
Ti	0.00	0.00	0.26	0.08
Mn	0.00	0.00	0.06	0.01
Fe	0.02	0.12	1.06	0.27

The adsorption of Fe and Mn by an adsorbent composed of activated clay, activated zeolite, and activated charcoal has resulted in a significant change in the surface morphology of the adsorbent. Before adsorption, the adsorbent had a high percentage of carbon and oxygen, indicating a high content of organic and inorganic compounds. The atomic percentage of carbon and oxygen was 73.69% and 23.15%, respectively, while the nuclear percentage of Fe and Mn was 0.12% and 0%.

However, after the adsorption process, the surface morphology of the adsorbent changed dramatically. The atomic percentage of Fe and Mn increased significantly (0.27% and 0.01, respectively), indicating a successful adsorption process. The atomic percentage of carbon and oxygen decreased to 65.09% and 28.64%, respectively. An increase in the atomic percentage of Fe and Mn was paralleled by a decrease in the atomic percentage of some other elements, including Si, Al, and Ca. This indicates that the adsorbent's selective adsorption of Fe and Mn was quite efficient. A reduction in the carbon content was noted concurrently, which implies that the possible adsorption of Fe and Mn may have altered the structure or composition of the adsorbent to some extent. Such an alteration might suggest the removal of some functional groups, or the removal of lighter atoms, including carbon and oxygen, from the adsorbent surface. Overall, the adsorbent's surface morphology which had many pores, was altered due to the successful adsorption of Fe and Mn. The adsorption process has led to a significant change in the atomic percentage of different elements, indicating that the adsorbent effectively removes Fe and Mn from the acid mine drainage.

3.3. Justification of sorbent composition

The composition of the composite sorbent, 50% activated coconut shell charcoal, 25% zeolite, and 25% claystone, was deliberately selected based on the distinct physicochemical characteristics of each material, as indicated by surface area analysis (BET), elemental composition (XRF), and their known adsorption mechanisms.

Activated charcoal from coconut shells exhibited the highest BET surface area and pore volume among the tested sorbents, enabling extensive physical adsorption of heavy metals such as Fe and Mn. This material also showed a rough, porous surface morphology in SEM images, contributing to high adsorption capacity and faster kinetics. The decision to allocate 50% of the composite to charcoal was made to maximize the contribution of surface-driven adsorption, which is especially effective in the initial phase of contaminant removal.

Zeolite was included at 25% due to its ion-exchange capabilities, particularly beneficial for selectively adsorbing cationic species such as Mn^{2+}. Despite a lower surface area, the XRF analysis showed significant CaO and Al_2O_3 content, indicating a strong ion-exchange potential and buffering effect. The crystalline structure of zeolite also enhances its ability to interact with metal ions in acidic conditions typically found in acid mine drainage (AMD).

Although claystone had the lowest surface area, it was rich in SiO_2 and Al_2O_3, suggesting good potential for chemical adsorption through surface complexation, particularly for Fe^{3+}. The functional groups available on clay minerals (e.g., hydroxyl and silanol) can form stable inner-sphere complexes with metal ions. Including 25% claystone helps provide chemical affinity and contributes to long-term metal immobilization.

This 50:25:25 ratio was chosen to leverage the complementary adsorption mechanisms of the three materials: activated charcoal for high surface area and rapid physical adsorption, Zeolite for selective ion exchange and pH buffering, and claystone for chemical adsorption and structural support.

3.4. Adsorbent performance

The adsorption performance of each sorbent, activated coconut shell charcoal, zeolite, and claystone, was evaluated based on their proportional composition within the composite (50% coconut shell charcoal, 25% zeolite, 25% claystone). The results indicated that activated charcoal was dominant in Fe and Mn removal due to its high surface area and microporous structure, facilitating greater adsorption capacity. Zeolite contributed additional cation exchange capacity, which was particularly beneficial for Mn adsorption, while claystone provided supplementary adsorption sites with relatively lower capacity. The synergistic effect observed in the composite confirmed that the 50:25:25 ratio allowed each material to complement one another's limitations, resulting in enhanced overall removal efficiency compared to individual components used alone.

Table 4 shows the results of an adsorption experiment using an adsorbent with varying masses of 2.5 grams, 5 grams, and 7.5 grams. The adsorption effectiveness was measured by the change in pH from initial up to final, after a contact time of 5, 10, and 15 minutes. The initial pH was maintained at 2.6 for all experiments. This rise in pH suggests that all three adsorbents contribute to neutralizing the acidic nature of AMD, potentially due to the binding of H^+ ions, which reduces acidity.

While increased adsorbent mass improved Fe removal, Mn adsorption did not follow a consistent trend. For instance, using 2.5

grams of adsorbent, the effectiveness of adsorption ranged from 58.73% to 61.76%. Increasing the mass to 5 grams and 7.5 grams resulted in a higher range of effectiveness, from 62.86% to 66.23%.

It should be noted that the contact time also plays a role in the effectiveness of adsorption. Longer contact time generally leads to higher effectiveness. For example, using 7.5 grams of adsorbent, the effectiveness of adsorption increased from 64.38% at 5 minutes of contact time to 66.67% at 15 minutes of contact time.

Table 4. PH changing after adsorption.

Mass (gram)	Contact time (minutes)	pH		Effectiveness
		Initial	Final	
2.5	5	2.6	6.3	58.73%
	10	2.6	6.5	60.00%
	15	2.6	6.8	61.76%
5	5	2.6	7	62.86%
	10	2.6	7.5	65.33%
	15	2.6	7.7	66.23%
7.5	5	2.6	7.3	64.38%
	10	2.6	7.6	65.79%
	15	2.6	7.8	66.67%

The rise in pH is attributable to the composite material's function as an adsorbent. One of the composite materials was claystone, comprised of kaolinite and montmorillonite extracted from coal overburden. Both montmorillonite and kaolinite have a high cation exchange capacity range from 2 to 16 milliequivalents per 100 grams for kaolinite and from 60 to 100 milliequivalents per 100 grams for montmorillonite (Nursanto & Pradise, 2021). Because the composite has successfully adsorbed a considerable amount of Fe metal, the concentration of Fe in acid mine drainage decreases, causing the pH of acid mine drainage to rise. With increasing pH, the solubility of iron decreases, leading to its precipitation or adsorption. This phenomenon occurs because Fe, in the form of iron ions (Fe2+ or Fe3+), can function as a basic compound in an acidic environment (Nordstrom, 2011). As the pH increases, Fe3+ precipitates and settles out of the solution (Diep et al., 2018). In addition, the presence of sodium, magnesium, potassium, and calcium cations in the composite raises the pH of acid mine drainage. It occurs because the cations Na, Mg, K, and Ca are basic cations—alkalinity results primarily from carbonate minerals, particularly calcite and dolomite. Silicate minerals (including quartz, kaolinite, illite, smectite, and muscovite) can also mitigate acidification, but at a much slower rate than carbonate minerals. The X-ray diffraction (XRD) analysis revealed that the claystone used as an adsorbent in the study predominantly consists of kaolinite minerals, illite, and a minor amount of montmorillonite (Paradise et al., 2022). Kaolinite is not easily pulverized due to its strong structural bonding (Adeyemo et al., 2017). Overall, the results suggest that the adsorbent used in the experiment is effective in adsorbing the target substance, and increasing the mass and contact time can improve the effectiveness of adsorption.

Table 5 shows the results of an adsorption study on removing Fe from acid mine drainage using an adsorbent with varying mass and contact time. The initial Fe concentration was 13.006 ppm, and the effectiveness of the adsorption was determined by measuring the final Fe concentration after adsorption. The results indicate that increasing the mass of the adsorbent and contact time resulted in a higher effectiveness of Fe removal. The removal efficiencies for Fe (Table 5) reached a maximum of 99.85% with 5 grams of adsorbent and a 15-minute contact time. Activated charcoal's higher surface area and porosity enable it to achieve this high removal efficiency, as it can adsorb more Fe ions faster than the other adsorbents. Additionally, its larger pore volume may facilitate better ion diffusion into the adsorbent structure, enhancing Fe removal.

Activated zeolite and activated clay, while also effective, demonstrated slightly lower Fe removal rates than activated charcoal. Zeolite, with its unique aluminosilicate structure and ion-exchange capabilities, shows a high affinity for Fe, though it is somewhat constrained by its lower surface area relative to activated charcoal. Activated clay is primarily effective for cationic ions, such as Fe, yet its lower surface area and pore volume limit its adsorption capacity. These findings suggest that the adsorbent is highly effective in removing Fe from acid mine drainage and that longer contact times and higher adsorbent mass result in higher effectiveness.

The results of the adsorption of Mn using an adsorbent at various masses and contact times, are given in Table 6. The initial and final concentrations of Mn in ppm (parts per million) are presented. The higher the effectiveness, the more efficient the adsorbent removes Mn from the acid mine drainage. At 2.5 grams of adsorbent, the effectiveness ranges from 46.13% to 61.76%, with a decreasing trend as the contact time increases. At 5 grams of adsorbent, the effectiveness ranges from 48.05% to 66.23%, with a decreasing trend. At 7.5 grams of adsorbent, the effectiveness ranges from 50.70% to 52.47%. The results for Mn removal, as shown in Table 6, indicate that Mn adsorption was less effective than Fe, with a maximum efficiency of 53.42% using 5 grams of adsorbent and a 15-minute contact time. This discrepancy may be due to differences in the ion size and charge density of Fe and Mn, which influence their interactions with the adsorbent materials. Overall, the results suggest that the adsorbent effectively removes Mn from the acid mine drainage, with the highest effectiveness achieved at 5 grams of adsorbent and 15 minutes of contact time. Further optimization of the adsorption process may be necessary to achieve the complete removal of Mn.

Table 5. Final Fe concentration.

Mass (gram)	Contact time (minutes)	Fe		Effectiveness
		Initial (ppm)	Final (ppm)	
2.5	5	13.006	0.061	99.53%
	10	13.006	0.047	99.64%
	15	13.006	0.021	99.84%
5	5	13.006	0.039	99.70%
	10	13.006	0.034	99.74%
	15	13.006	0.019	99.85%
7.5	5	13.006	0.044	99.66%
	10	13.006	0.036	99.72%
	15	13.006	0.03	99.77%

Table 6. Final Mn concentration.

Mass (gram)	Contact time (minutes)	Mn		Effectiveness
		Initial (ppm)	Final (ppm)	
2.5	5	30.59	16.48	46.13%
	10	30.59	15.77	48.45%
	15	30.59	15.72	48.61%
5	5	30.59	15.89	48.05%
	10	30.59	15.37	49.75%
	15	30.59	14.25	53.42%
7.5	5	30.59	15.08	50.70%
	10	30.59	14.81	51.59%
	15	30.59	14.54	52.47%

The adsorption performance of the composite adsorbent developed in this study, comprising claystone, natural zeolite, and coconut shell activated carbon (AC), was notably higher than that of each component as reported in previous studies. Under optimal conditions, the composite system achieved Fe removal efficiencies exceeding 95% and Mn removal over 85%, outperforming the adsorption capacities of standalone adsorbents used in comparable studies.

Natural zeolite has been widely studied for its ability to adsorb Fe and Mn from aqueous solutions, with removal efficiencies typically ranging from 60% to 80%. For example, Munawar et al. (2023) reported Mn removal efficiencies of up to 70% using natural zeolite from West Java, while Widyaningrum et al. (2022) demonstrated that zeolite could remove 74.2% of Fe and 68.6% of Mn from acid mine drainage (AMD). Fadliah et al. (2023) and Wibowo et al. (2024) showed that functionalizing zeolite or activating it chemically can improve its capacity, though results remained below those obtained in our composite system.

Clay-based adsorbents, such as kaolin or natural claystone, generally perform less due to limited surface area and exchange capacity. Budianta (2021) observed that mineralogical variations significantly influence Fe adsorption, with natural clay removing up to 50–60% of Fe. Similarly, Putra et al. (2024) and Sidiq&Purnomo (2023) found that clay adsorbents could remove around 50–65% of Fe and Mn from water and AMD, depending on surface modification.

Activated coconut shell charcoal has demonstrated strong potential for Fe adsorption, attributed to its high surface area and abundance of functional groups. Sukmono et al. (2024) reported Fe removal efficiencies exceeding 85%, while Abas et al. (2021) also confirmed the effectiveness of coconut shell AC in reducing Fe concentration in contaminated water. However, its Mn removal efficiency tends to be lower, likely due to weaker complexation or ion-exchange interactions, as also suggested by Water (2024).

Compared to these individual adsorbents, our composite exhibited synergistic behavior, resulting in significantly improved removal efficiencies for both Fe and Mn. The integration of zeolite and claystone enhances cation exchange and structural support, while the high surface area and functional groups of activated carbon boost chemisorption. This synergy is likely responsible for the enhanced kinetics and greater adsorption capacity observed in our experiments. Statistically, the improvement in removal performance is substantial and meaningful. While individual adsorbents typically target one specific adsorption mechanism, such as ion exchange (zeolite), physisorption (clay), or surface complexation (activated carbon), the composite combines all three, creating a multifunctional adsorbent with broader reactivity. Moreover, the diverse pore structures and surface chemistries improve accessibility and retention of both Fe and Mn ions. The performance of the composite adsorbent is significantly superior to that of each individual component, both in terms of removal efficiency and potential applicability for AMD treatment. This confirms the benefit of using locally available materials in engineered combinations to enhance the adsorption of heavy metals from complex wastewater matrices such as acid mine drainage.

3.5. Sorption kinetics

The adsorption kinetics of Fe and Mn onto the composite adsorbent (50% charcoal, 25% zeolite, 25% clay) were analyzed using both pseudo-first-order and pseudo-second-order models. For the pseudo-first-order model, Fe adsorption showed a moderate linear relationship for the initial time points, indicating that physical adsorption may partially influence the process (Figure 6). However, the third data point could not be used due to log(qe−qt) approaching negative infinity as equilibrium was reached. Mn exhibited a similar pattern but with slightly lower correlation, suggesting that this model does not fully describe the kinetic behavior. In contrast, the pseudo-second-order model produced a strong linear fit for both Fe and Mn (with high R2 values), indicating that chemisorption may be the dominant mechanism governing the adsorption process. This model assumes that the rate-limiting step involves valence forces through sharing or exchange of electrons between adsorbent and adsorbate, which aligns well with the experimental data. Therefore, the pseudo-second-order kinetic model is more appropriate for describing the sorption behavior of Fe and Mn in this study.

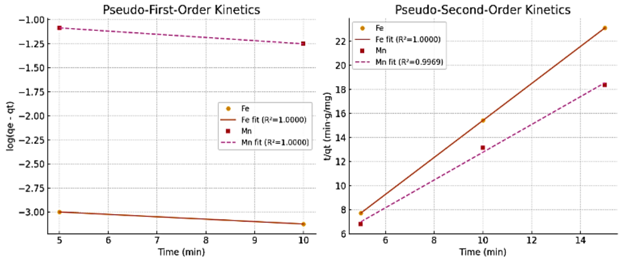

Fig 6. Kinetic modeling of Fe and Mn adsorption.

3.6. Adsorption capacity

The adsorption capacity of the composite adsorbent (comprising 50% charcoal, 25% zeolite, and 25% clay) toward Fe and Mn was evaluated at different contact times (5, 10, and 15 minutes) and adsorbent masses (2.5 g, 5 g, and 7.5 g), as shown in the Figure 7. For Fe, the adsorption capacity consistently increased with longer contact time across all adsorbent masses, indicating that more binding sites become utilized as the interaction period between adsorbate and adsorbent extends. Interestingly, the highest Fe adsorption capacity (1.299 mg/g) was observed with the lowest adsorbent mass (2.5 g) at the longest contact time (15 minutes), suggesting that at lower doses, the adsorbent surface is more efficiently utilized. Conversely, the lowest capacity (0.432 mg/g) occurred at 5 minutes with the highest mass (7.5 g), which may be attributed to site overlap or limited diffusion in a more crowded adsorbent matrix. A similar trend was observed for Mn, although its adsorption behavior revealed less pronounced differences across time and mass variations. The Mn adsorption capacity peaked at 1.487 mg/g with 2.5 g of adsorbent at 15 minutes, while the lowest value (0.517 mg/g) was seen at 5 minutes with 7.5 g. These results imply that both metals benefit from extended contact time, but excessive adsorbent mass may reduce efficiency due to particle aggregation or limited active site accessibility. Overall, the data suggest that optimal adsorption occurs at lower adsorbent dosages and longer contact durations, with Fe showing a more sensitive response to these operational parameters than Mn.

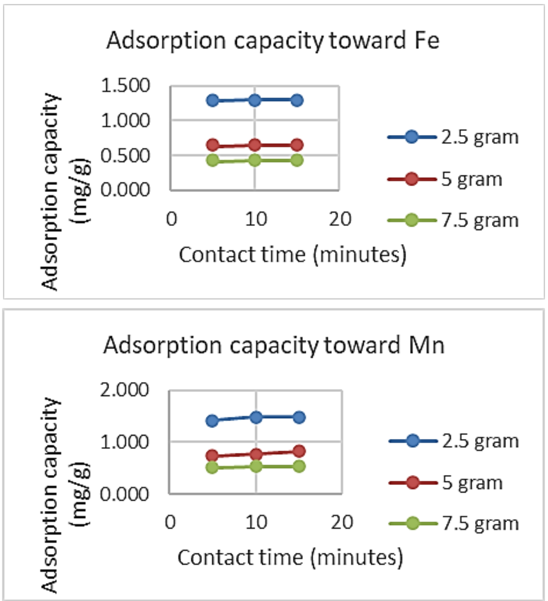

Fig. 7. Adsorption capacity toward Fe and Mn.

The composite material demonstrates the capability to decrease the concentration of Fe below the established quality standard. The high surface area and pore volume of activated charcoal make it especially suitable for applications that require rapid and high-capacity adsorption. The effectiveness of zeolite and clay as alternative adsorbents is also notable, particularly in settings where low-cost materials are prioritized, although their lower adsorption capacities may necessitate longer contact times or higher dosages. However, the concentration of Mn remains elevated and beyond the quality standard. The experimental findings also demonstrate variations in adsorption between iron (Fe) and manganese (Mn) metals. The analysis results indicate that adsorption transpires more rapidly when the mass of the composite is greater, or in other words, under more alkaline pH conditions. This is attributed to the presence of H$^+$ ions on the composite's surface, which leads to competition among heavy metal ions. The study revealed the adsorption order of several elements at a pH of 2.4. Hg > Fe > Pb > Cu = Al > Ni > Cr = Zn = Cd = Co = Mn was determined to be the order of adsorption, from highest to lowest (Kerndorff & Schnitzer, 1980). The element iron has a higher electronegativity than manganese, according to the periodic table of elements. Furthermore, the periodic table of elements indicates that the electronegativity of the Fe element surpasses that of the Mn element. Consequently, the findings of this study demonstrate that the percentage of Fe metal absorption exceeds that of Mn metal. The relative order of ion absorption ability in aqueous solutions, as influenced by the selectivity of ions towards the adsorbent media, can be described as follows: Fe^{3+}> Al^{3+}> Pb^{2+}> Ba^{2+}> Sr^{2+}> Zn^{2+}> Cu^{2+}> Fe^{2+}> Mn^{2+}> Ca^{2+}> Mg^{2+}> K$^+$> NH^{4+}> H$^+$> Li$^+$. Hence, based on the ion selectivity observed in this investigation, it can be concluded that the adsorption of Fe is more pronounced compared to Mn. The surface area and pore volume of each material were considered to assess their adsorption capabilities. The significantly higher surface area and pore volume of activated charcoal indicate that it may have greater potential for adsorbing iron and manganese compared to activated clay and zeolite. Therefore, the composition of the composite adsorbent (50% activated charcoal, 25% zeolite, and 25% claystone) was chosen to maximize the adsorption efficiency for these metals.

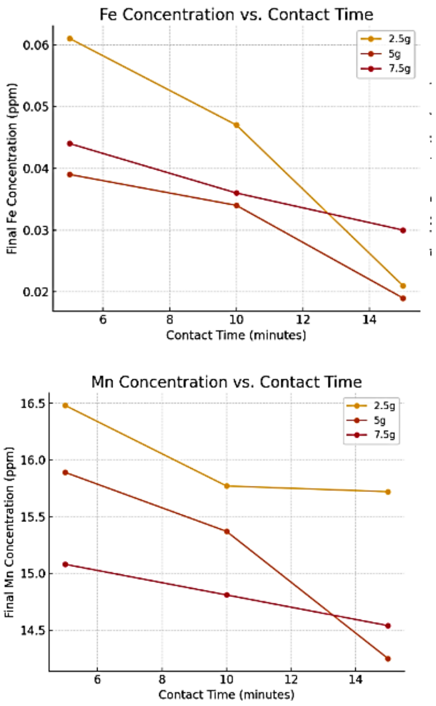

Fig. 8. Relation of Fe and Mn Final Concentration with varying contact time and mass.

The time-dependent concentration profiles for Fe and Mn (Figure 8) provide insight into the adsorption dynamics using the composite adsorbent (50% activated coconut shell charcoal, 25% zeolite, and 25% claystone). For Fe, a significant and rapid decrease in concentration was observed within the first 5 minutes across all adsorbent dosages (2.5 g, 5 g, and 7.5 g), with only minor decreases at 10 and 15 minutes. This indicates that Fe adsorption reaches near-equilibrium quickly, suggesting a strong interaction between Fe ions and the composite surface. This is consistent with the kinetic modeling results, which fit the pseudo-second-order model well, implying chemisorption as the dominant mechanism.

In contrast, Mn exhibited a slower and more gradual decrease over time. While there was some removal within the first 5 minutes, the reduction was more progressive between 10 and 15 minutes, indicating a slower adsorption process. The lower effectiveness in Mn removal compared to Fe may be attributed to differences in ionic radius, hydration energy, and the affinity of Mn ions for the active sites on the composite surface. Overall, these time-dependent patterns show that while a longer contact time is necessary to improve Mn adsorption, the composite material is more effective for quickly removing Fe. This behavior emphasizes how crucial it is to maximize adsorbent dosage and contact duration to attain the required removal efficiencies for various metals in acid mine drainage treatment.

3.7. Adsorption isotherm

Figures 9 and 10 show the Langmuir and Freundlich isotherms for the adsorption of iron (Fe) and manganese (Mn), which are essential for evaluating the adsorption process in removing these metals from acid mine drainage (AMD).

Langmuir Isotherm for Fe (Figure 9, left): The linear equation indicates an excellent fit to the Langmuir model, suggesting that the adsorption sites for Fe may be homogeneous. The high R^2 value shows that the model perfectly describes the adsorption process of Fe, suggesting a monolayer coverage on the adsorbent's surface. Langmuir Isotherm for Mn (Figure 9, right): The linear equation indicates a good fit to the Langmuir model for Mn adsorption, though slightly less perfect than Fe. The high R^2 still suggests monolayer adsorption, though the different slope may imply that Mn has a different affinity or maximum adsorption capacity compared to Fe on the same adsorbent. Freundlich Isotherm for Fe (Figure 10, left): The equation indicates a high correlation, suggesting that the Freundlich model also fits the Fe adsorption data well. This model typically applies to heterogeneous surfaces, implying that multiple adsorption sites with varying energies may exist for Fe on the adsorbent. Freundlich Isotherm for Mn (Figure 10, right): The equation indicates an even better fit for Mn to the Freundlich model. This high R^2 value suggests that the adsorption process for Mn might involve a heterogeneous surface, similar to Fe. The steeper slope compared to Fe might indicate stronger adsorption affinity or varying site energies for Mn. From these results, both Fe and Mn adsorption fit well to the Langmuir and Freundlich models, with slightly better fits for the Langmuir model in Fe ($R^2 = 1$) and the Freundlich model in Mn ($R^2 = 0.9993$). The Langmuir model implies that both metals may form a monolayer on the adsorbent surface, but Mn shows a slightly different affinity and possibly a lower saturation point. The Freundlich model indicates surface heterogeneity, suggesting the adsorbent has varied types of binding sites, which is especially relevant for Mn.

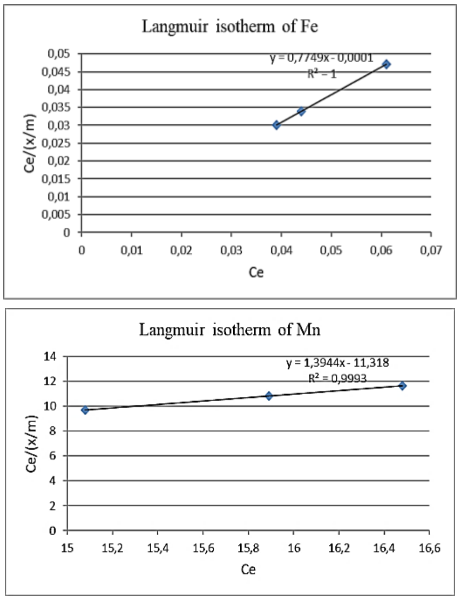

Fig. 9. Langmuir isotherm of Fe and Mn.

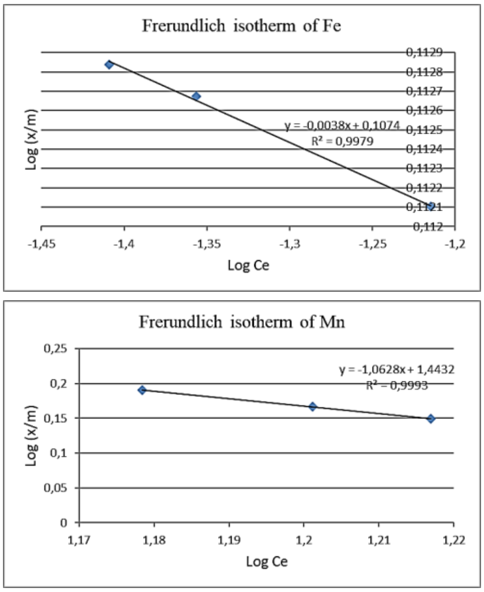

Fig. 10. Freundlich isotherm of Fe and Mn.

4. Conclusion

This study demonstrated the effectiveness of a composite adsorbent composed of 50% activated coconut shell charcoal, 25% zeolite, and 25% claystone in removing Fe and Mn from acid mine drainage (AMD). The adsorption process was influenced by contact time and adsorbent dosage, where Fe removal consistently showed high effectiveness (>99%). In contrast, Mn removal exhibited moderate improvement with increasing contact time and dosage, albeit with lower overall efficiency compared to Fe. Both monolayer and heterogeneous surface interactions were indicated by the adsorption behavior of Fe and Mn, which followed Langmuir and Freundlich isotherms. The pseudo-second-order model better explained the adsorption process, according to kinetic modeling, indicating chemisorption as the primary mechanism. These findings show that low-cost, locally available materials can be composite adsorbents in passive AMD treatment systems. The study helps build long-term plans for AMD cleanup, particularly in mining areas of developing countries such as Indonesia. Future research should consider extending the process to pilot or field deployments to evaluate performance in real-world situations. Additionally, long-term adsorption studies and regeneration experiments would provide insights into these adsorbents' durability and economic feasibility for repeated use.

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