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A Comparative Study on the Effectiveness of Iron (Fe) Adsorption from Acid Mine Drainage Using Single and Composite Adsorbents

Mycelia Paradise*[1], Yudha Agung Pratama[2], Faisol Mukarrom[3], Triyono[4], Akhmad Zamroni[5]
[1,3,4]Department of Mining Engineering, Faculty of Engineering and Planning, Institut Teknologi Nasional Yogyakarta, 55281, Indonesia
[2]Department of Geophysical Engineering, Faculty of Mineral and Energy Technology, Universitas Pembangunan Nasional "Veteran" Yogyakarta, 55283, Indonesia
[5]Department of Geological Engineering, Institut Teknologi Nasional Yogyakarta, 55281, Indonesia
Author emails: [1]mycelia@itny.ac.id, [2]yudha.agung@upnyk.ac.id, [3]faisol@itny.ac.id, [4]triyono@itny.ac.id, [5]akhmadzamroni@itny.ac.id

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ABSTRACT

Acid mine drainage (AMD) is mining waste that has high acidity and dissolved metal content, especially iron (Fe), which has the potential to pollute the aquatic environment. This research aimed to evaluate the performance of coconut shell activated charcoal and local composite-based adsorbents (active charcoal–clay–zeolite) in reducing Fe concentrations in AMD. The research was conducted using two types of samples: real AMD collected from the mine site and synthetic AMD prepared in the laboratory using a $FeSO_4 \cdot 7H_2O$ solution. The composite adsorbent was tested using real AMD (baseline pH 2.6; Fe 42.75 ppm), while activated charcoal was tested on synthetic AMD (baseline pH 4.7; Fe 40.37 ppm). The main variables observed were dissolved Fe concentration and pH changes at various contact time variations. Analysis of Fe concentrations was carried out using AAS, while pH was measured directly with a pH meter. Adsorbent characterization was performed with BET to identify the specific surface area of the adsorbents. Results showed that activated charcoal was able to increase pH to 8.6 and decrease Fe to 0.04 ppm (99.9% efficiency). The primary mechanism of Fe reduction is dominated by $Fe(OH)_3$ precipitation, which occurs due to an increased pH. In contrast, the composite produced a final pH of 4.3 but still decreased Fe to 0.52 ppm (98.8% efficiency). BET analysis showed that although activated charcoal had a greater surface area, the composite maintained better pore stability after adsorption. The integration of the two has the potential to enhance the adsorption effectiveness and stability in the field for the remediation of acid mine drainage.

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A. INTRODUCTION

Acid mine drainage (AMD) is one of the most serious environmental problems in mining areas because it contains high levels of acidity and significant concentrations of dissolved metals, especially iron (Fe), manganese (Mn), and aluminum (Al) (Gobel, 2025; Riekert et al., 2025; Wintah, 2025). Water formed through the oxidation of sulfide minerals can pollute surface water bodies, damage aquatic ecosystems, and endanger the health of communities surrounding mines (Skousen, 2014). Therefore, AMD processing is an essential focus in efforts to mitigate the environmental impacts of mining activities.

Various methods have been developed to reduce heavy metal levels in AMD, including chemical precipitation, phytoremediation, and membrane technology (Sheoran & Sheoran, 2006; Masindi et al., 2020). Such methods have proven effective but relatively expensive and complex, prompting the development of more straightforward and more economical alternatives (Qasem et al., 2021; Maura, Atreya, & Arshi, 2023). Adsorption then becomes an option because it is easy, not too expensive, and can significantly reduce heavy metal levels (Kurniawan et al., 2006; Thakur, 2022; Biswal & Balasubramanian, 2023)

Previous research has demonstrated that activated charcoal (activated carbon) can effectively reduce the heavy metal content in wastewater (Yang, 2020; Rofikoh et al., 2023; Wang et al., 2023; Primary & Paradise, 2025). Several studies show that zeolites and claystone as aluminosilicate materials have high ion exchange capacity and surface sites so they are effective at adsorbing multivalent metal ions such as Pb(II), Cu(II), Cd(II), and Fe(II) (Paradise et al., 2021; Paradise et al., 2022; Shah et al., 2023; Hakim et al., 2024; Middea, 2024; Paradise & Pratama, 2024; Xie, 2024)

Despite various efforts, comparative studies directly comparing the performance of single and composite adsorbents under real AMD and artificial AMD conditions are very limited. In fact, the chemical characteristics of the two types of AMD are very different: native AMD generally has a very low pH and a complex ion composition, while artificial AMD has simpler conditions with a relatively higher pH. These differences can affect the dominant mechanism of Fe removal, both through precipitation due to increased pH and chemical interactions with mineral surfaces. Historically, the authors' research has included studies on adsorbent characterization, adsorption isotherms, and the development of laboratory-scale automation for adsorption systems. This research was conducted as a continuation aimed at confirming the role of mineral-based composites in enhancing the effectiveness of iron (Fe) adsorption compared to single adsorbents, while contributing to novelty through testing at low pH conditions typical of acid mine drainage. In many previous studies, adsorption testing was generally carried out under more controlled conditions rather than under the highly acidic conditions commonly found in AMD. In this study, the adsorbents were evaluated at low pH to assess their performance and stability under conditions closer to those of actual AMD.

B. METHODS

This research utilized two types of acid mine drainage (AMD) samples: synthetic AMD and real AMD. Synthetic AMD samples were prepared in the laboratory by dissolving the compound $FeSO_4 \cdot 7H_2O$ in aquadecs. AMD samples were taken directly from coal mine sumps.

Adsorption Procedure

1. Preparation of test solutions
    a. Real AMD was obtained directly from coal mine sumps
    b. Synthetic AMD wa made in the laboratory
    Main ingredients: $FeSO_4 \cdot 7H_2O$ salt (molar mass 278 g/mol).
    Target Fe concentration: 42 ppm.
    Amount of Fe required = 42 ppm = 7.52×10^{-4} mol Fe.
    Equivalent $FeSO_4 \cdot 7H_2O$ mass = 7.52×10^{-4} mol × 278 g/mol = 0.209 g.
    Procedures:
    1) $FeSO_4 \cdot 7H_2O$ was weighed at 0.209 g using an analytical scale.
    2) The solid was dissolved in 1 Liters of water in a volumetric flask.
    3) The solution was homogenized until completely dissolved.

4) The solution was left open ±24 hours to allow some of Fe(II) to oxidize to Fe(III), thus resembling the real condition of AMD.
5) Concentration Test
   The Fe concentration of the solution was tested using an Atomic Absorption Spectrophotometer (AAS).

2. Experimental conditions: The adsorption process was carried out in a batch system with contact time variations (0, 2, 3, 4, 5, 6, 9, 12 minutes) at room temperature. A total of 100 mL of the AAT sample was placed in the stirring pool, then an adsorbent at a dose of 1 g/L was added. The adsorption process was carried out using a prototype assembled tool (Auto-Adsorption Assembly) that was operated with an automation system using a microcontroller at 1,000 rpm during variations in contact time.

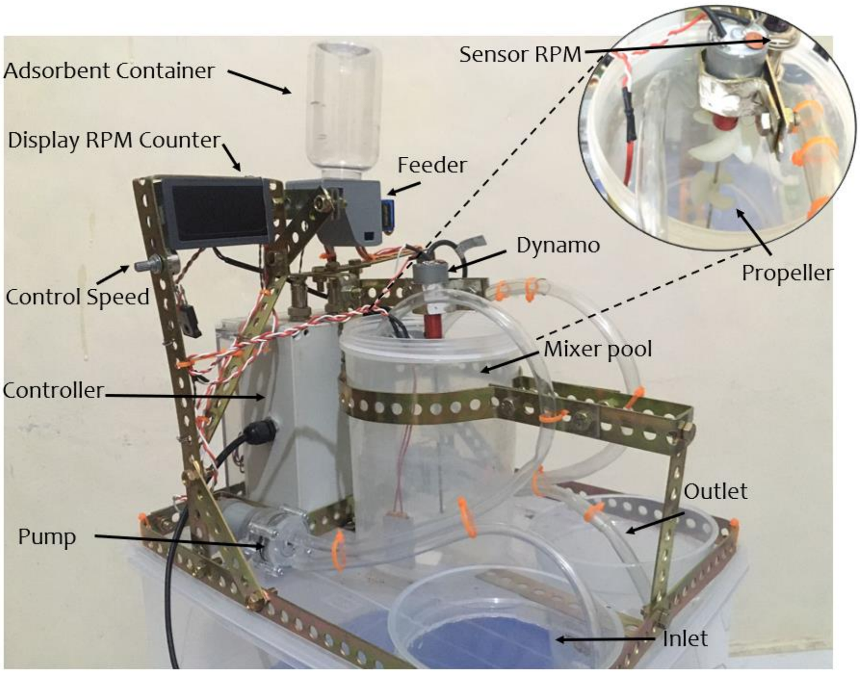

Figure 1. Auto-Adsorption Assembly

Adsorbent materials used: activated charcoal, zeolite, and clay.

Figure 2. Activated adsorbent material: A)claystone, B)zeolite, C)coconut shell

Adsorbents used: activated charcoal (single adsorbent) and activated charcoal composite–claystone–zeolite (50:25:25 weight %). Adsorbent Characterization: BET analysis (Brunauer-Emmett-Teller).

3. Sampling: Aliquots of the solution are taken at certain time intervals, then filtered using a Whatman filter no. 41.
4. Fe Analysis: Residual Fe concentrations were measured using Atomic Absorption Spectrophotometry (AAS).

This study evaluates the comparative adsorption performance under controlled experimental conditions. The present study does not include replication tests and sensitivity analysis, but future work may incorporate them to further assess the consistency and reliability of the adsorption system.

C. RESULT AND DISCUSSION

Initial Characteristics of AMD

The AMD used in the study was of two types: real AMD and synthetic AMD. The real AMD showed an initial pH condition of 2.6 with a Fe concentration of 42.75 ppm, while the synthetic AMD had an initial pH of 4.7. The test results showed a Fe concentration in synthetic AMD of 40.37 ppm, slightly lower than the theoretical target (42 ppm). This is thought to be due to the partial oxidation of Fe^{2+}, adsorption on the container wall, or the purity of the reagent. These differences in initial characteristics indicate the variation between field conditions and laboratory-controlled conditions.

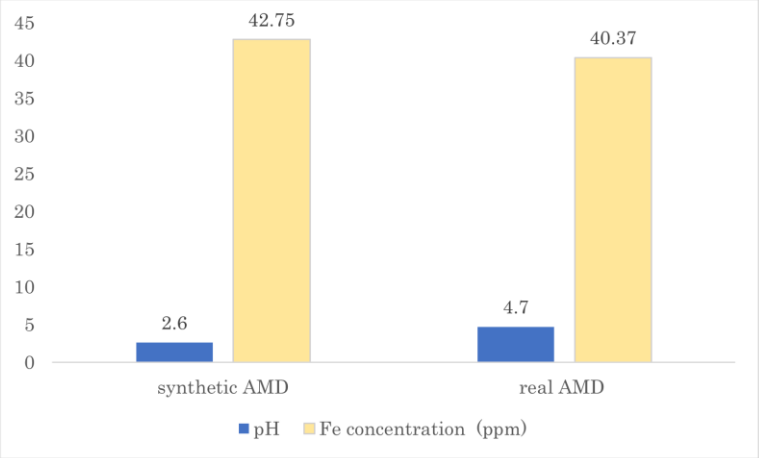

Figure 3. AMD Characteristics

Changes in pH During the Adsorption Process

Based on Figure 5, there are two different patterns of pH change: (1) in experiments with composites (real AMD, initial pH 2.6) the pH rose rapidly in the range of 4.15–4.35 in 2–6 minutes and then remained relatively stable at around 4.18–4.32 in minutes 9–12; (2) in experiments with activated charcoal (synthetic AMD, initial pH 4.7) pH jumped sharply to a peak of 10.3 at the 4th minute then decreased and stabilized at around 8.4–8.6 towards the 12th minute.

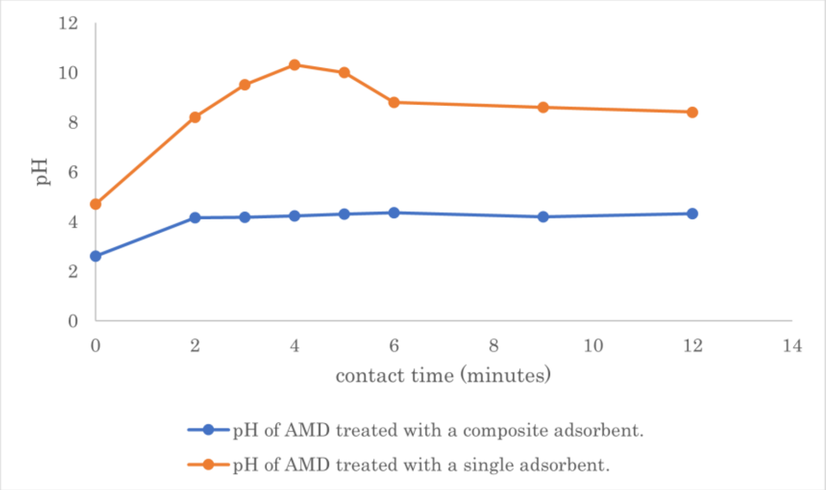

Figure 4. AMD pH vs Contact Time

The phenomenon of increasing pH in activated carbon-based adsorption systems is generally associated with the release of surface base groups, such as –OH, or the deprotonation of functional groups when the adsorbent interacts with the solution, resulting in a decrease in the concentration of dissolved H^+ ions (Elewa et al., 2023). In addition, a decrease in the concentration of H^+ ions in solution can occur through ion exchange reactions and complexation interactions with the surface functional groups of the adsorbent, thereby increasing the overall pH of the solution (Al-Badaani et al., 2023). A very significant increase in pH, as observed in this study (from an initial pH of 4.7 to >10 in a relatively short time), indicates conditions that strongly favor the formation of iron hydroxide ($Fe(OH)_3$) deposits. The solubility of iron compounds in oxidized form (Fe^{3+}) decreases drastically under neutral to basic conditions, so iron precipitation occurs quickly and contributes to a decrease in the concentration of dissolved Fe ions (Furcas et al., 2024). Therefore, the decrease in Fe concentration from 40.37 ppm to 0.04 ppm in the use of activated charcoal is not solely controlled by the surface adsorption process, but is also triggered by the accelerated precipitation mechanism due to the increase in pH.

The use of charcoal-based composites, clay, and zeolite results in a moderate pH increase, from the initial condition of 2.6 to approximately 4.2–4.3. This is due to the nature of the cation exchange capacity (CEC) and the buffer property (buffering capacity), which can withstand drastic changes in pH. Both minerals absorb H^+ ions through a cation exchange mechanism, so that even though the pH increase is not significant, the process is effective in absorbing metal ions through an ion exchange and surface complexation mechanism (surface complexation) (Fadilah et al., 2023; Bakalar et al., 2020). Because the pH increase is relatively small, the formation of iron hydroxide ($Fe(OH)_3$) deposits is not dominant; As a result, Fe removal in composite use occurs more through adsorption and ion exchange at mineral surface sites than massive precipitation (Bhattacharyya & Sen Gupta, 2006; Bakalár et al., 2020).

Characterization of Adsorbents by Surface Area Test

The variation in pH change between the two types of adsorbents is also in line with the results of specific surface area characterization (BET). Activated charcoal exhibits a high initial surface area (116 m^2/g), providing many active sites that play a role in chemical interactions and micro pH regulation around the particle surface. However, after the adsorption process, the surface area decreased to 56 m^2/g, indicating that most of the pores had been filled by adsorption and surface reaction products (Al-Badaani et al., 2023). In contrast, charcoal-based composites–clay–zeolite experienced a more moderate decrease in surface area (from 86.8 m^2/g to 63.4 m^2/g). This condition indicates that the absorption process occurs more evenly on the mineral surface through ion exchange and surface complexation mechanisms, rather than just deposition in micro pores, resulting in a more homogeneous distribution of adsorbates (Elewa et al., 2023).

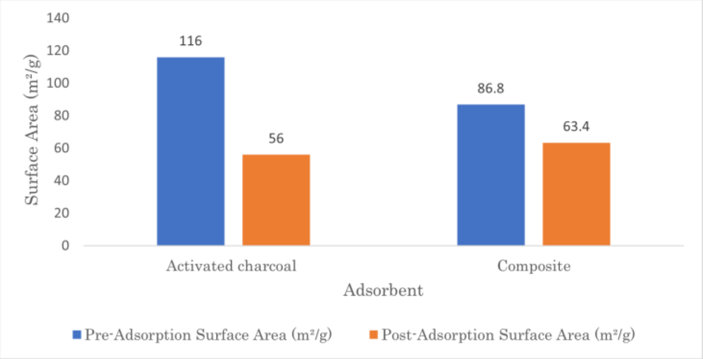

Figure 5. Surface area of the adsorbent

Changes in Fe concentration During the Adsorption Process

The data in Table 10 show changes in iron (Fe) concentration with respect to contact time during the adsorption process using two types of adsorbents: composite and activated charcoal. Initial concentrations of Fe in the composite system and activated charcoal were 42.75 ppm and 40.37 ppm, respectively. After 2 minutes, these concentrations decreased sharply to 1.37 ppm and 0.23 ppm, respectively, and then further reduced to reach 0.5157 ppm (composite) and 0.0406 ppm (activated charcoal) after 12 minutes of contact time. This phenomenon indicates that the initial stage of adsorption occurs rapidly, suggesting that most of the active sites on the adsorbent surface remain empty and can interact directly with Fe ions in solution (Kalenskii et al., 2023).

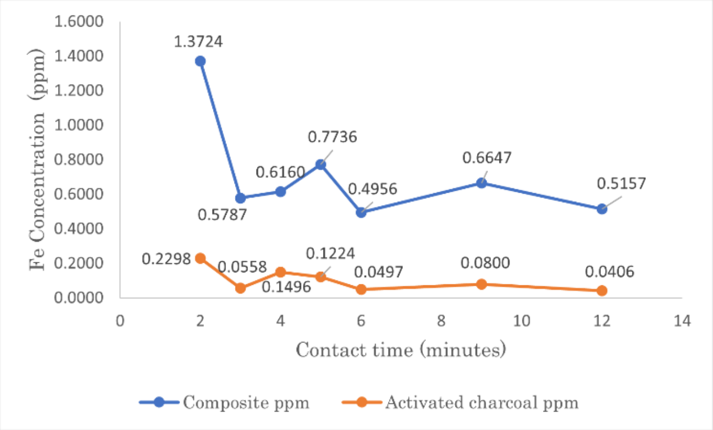

Figure 6. Concentration of Fe during adsorption

A rapid drop in the early minutes indicates that the adsorption rate is controlled by external mass transfer, when Fe ions move rapidly towards the particle surface by film diffusion. After a few minutes, the absorption rate slows and tends to approach the equilibrium state, indicating a decrease in the number of free active sites. Such kinetic patterns typically follow a pseudo-second-order model, where the rate of adsorption is directly proportional to the number of empty sites and the concentration of dissolved metal ions (Blankenship et al., 2022). A comparison of performance between the two adsorbents reveals that activated charcoal exhibits higher adsorption capabilities than the composites. This is evident from the lower residual Fe concentration at each observation time. Activated charcoal has a broad microporous structure with a pore size distribution that supports the rapid diffusion of metal ions towards internal sites, as well as oxygen functional groups (carboxyl and hydroxyl) that can interact strongly through complexation with Fe ions (Kalenskii et al., 2023). On the other hand, composites generally exhibit more heterogeneous porosity and a lower specific surface area, resulting in slower mass transfer of Fe ions to the active surface (Hidayat et al., 2024).

Small fluctuations in the Fe concentration of the composite system between the 3rd and 9th minutes can be attributed to the presence of a more dominant intraparticle diffusion process following the rapid adsorption step. Fe ions remaining in the solution must penetrate the pore layer or inner surface of the composite material to interact with the active site, causing equilibrium to be reached more slowly (Kalenskii et al., 2023). Additionally, the presence of non-carbon phases, such as metal oxides, in the composite can cause a competitive effect between the adsorption mechanism and $Fe(OH)_3$ precipitation at the surface. In terms of mechanism, Fe adsorption by activated charcoal is predominantly driven by chemical interactions (chemisorption) through ion exchange and the formation of surface complexes. At the same time, composites rely more on physico-adsorption and weaker electrostatic interactions (Blankenship et al., 2022). This explains why activated charcoal can reach very low final concentrations (0.0406 ppm) in a short time, whereas composites require longer contact times to reach values close to equilibrium. These results align with the research by Ratnawati et al. (2024), which states that the combination of activated carbon with chitosan-modified zeolites can simultaneously adsorb Fe and phenol ions through ion exchange and complexation mechanisms, confirming the importance of pore structure and active surface groups in determining the effectiveness of adsorption. Thus, the difference in effectiveness between composites and activated charcoal is mainly due to variations in pore morphology, specific surface area, as well as surface chemical properties that determine speed and total adsorption capacity.

The results also showed that contact time affected Fe removal during adsorption. However, adsorption kinetics such as pseudo-first-order and pseudo-second-order models were not discussed in detail in this study. Further studies are needed to better understand adsorption behavior under different AMD conditions and in larger-scale applications. In actual field conditions, AMD characteristics can vary depending on pH, metal content, and water flow. These conditions may affect adsorbent performance differently compared to laboratory testing.

D. CONCLUSION AND SUGGESTIONS

This study showed that both adsorbents were effective in reducing Fe concentrations in acid mine drainage (AMD), although they exhibited different adsorption characteristics. Activated charcoal achieved the highest Fe removal efficiency by increasing pH from 4.7 to 8.6 and reducing Fe concentration from 40.37 ppm to 0.04 ppm (99.9%), mainly through $Fe(OH)_3$ precipitation. Meanwhile, the charcoal–clay–zeolite composite increased the pH from 2.6 to 4.3 and reduced Fe concentration from 42.75 ppm to 0.52 ppm (98.8%), indicating more stable performance under acidic conditions through ion-exchange and surface-complexation mechanisms. BET analysis further showed that the composite adsorbent maintained better pore stability after adsorption. These findings suggest that activated charcoal is more suitable for initial AMD treatment, while the composite adsorbent has potential for stable treatment under acidic conditions. The integration of both adsorbents may improve the effectiveness of AMD remediation and operational stability in field applications.

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