

# Assessing The Performance of Acid Mine Drainage Sludge in Removing Anionic Dye from Aqueous Solution

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**Abstract**— Acid mine drainage (AMD) presents substantial environmental issues due to its acidity and high metal content. Traditional treatment methods, such as lime neutralization, produce a substantial amount of sludge, which poses disposal issues. This study assessed the efficacy of utilizing AMD sludge as an adsorbent for congo red dye from aqueous solutions. AMD samples from Khwezela Colliery were neutralized using lime and under the determined optimum conditions (dosage of 0.3 g and a contact time of 30 minutes), AMD's pH was increased from 2.19 to 7.89. The resulting sludge was characterized for surface, elemental, and structural characteristics using Scanning Electron Microscopy (SEM) combined with Energy Dispersive X-Ray Analysis (SEM-EDX), X-ray Fluorescence (XRF), and Fourier Transform Infrared Spectroscopy (FTIR) analysis. Under optimal conditions of 10 mg/L dye concentration, 10 minutes contact time, and 1 g dosage the sludge removed up to 87% of dye from the solution. The results suggest that AMD sludge can be a cost-effective and sustainable adsorbent for dye removal in wastewater treatment, lowering both sludge disposal costs and environmental impacts. The treated AMD water achieved a neutral pH and low metal ion concentrations, suitable for recycling in industrial applications, particularly for construction.

**Keywords**— Acid mine drainage, dye removal, lime neutralization, and sludge.

## I. INTRODUCTION

AMD is a critical environmental problem that arises from the exposure of sulfide minerals to air and water during mining operations, leading to the generation of acidic, metal-laden effluents. When sulfide minerals such as pyrite ( $\text{FeS}_2$ ) undergo oxidation, they produce sulfuric acid, which dissolves surrounding metals, creating a highly toxic and acidic discharge. The release of untreated AMD into aquatic systems severely impacts the environment by contaminating water bodies, degrading ecosystems, and posing risks to human health through the bioaccumulation of toxic metals [1]. Given the scale and intensity of mining activities globally, especially in countries with rich mineral resources, AMD

represents a persistent and costly challenge to both the mining industry and environmental protection agencies [2].

Traditional methods for AMD treatment involve neutralization processes that aim to raise the pH of the acidic waters, thereby precipitating dissolved metals into a sludge. Among the most widely used neutralizing agents is lime ( $\text{Ca}(\text{OH})_2$ ), due to its affordability and effectiveness in rapidly increasing pH and promoting metal hydroxide precipitation [3]. This treatment not only reduces the acidity of AMD but also converts hazardous dissolved metals into a solid form, preventing them from further contaminating water systems. However, this process results in the production of large volumes of metal-laden sludge, which presents its own set of environmental challenges, such as the need for proper disposal or storage [4]. In some cases, poorly managed sludge disposal can lead to secondary pollution, where metals are leached back into the environment, negating the benefits of AMD treatment [5].

In recent years, there has been growing interest in the potential reuse of AMD sludge as a resource rather than waste. This concept aligns with the principles of sustainable waste management and circular economy, where industrial by-products are repurposed to reduce environmental impact and create value [6]. AMD sludge, which is typically rich in iron, aluminum, and other metal oxides, has been identified as a promising material for use in environmental applications, particularly as an adsorbent for removing pollutants from water. Research has shown that metal oxides present in the sludge can provide active sites for the adsorption of various contaminants, including heavy metals, phosphates, and organic pollutants such as dyes [7]. The reuse of AMD sludge in this manner not only reduces the burden of sludge disposal but also offers a low-cost, sustainable solution for treating industrial effluents.

Among the various pollutants found in industrial wastewater, synthetic dyes, especially anionic dyes, have garnered significant attention due to their widespread use and environmental persistence. Anionic dyes, commonly used in industries such as textiles, leather, and paper, are not only highly toxic but also resistant to degradation, making their removal from wastewater a priority [8]. These dyes can pose severe environmental and health risks, including the potential to form carcinogenic by-products when exposed to certain

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conditions. Conventional dye removal techniques, such as coagulation-flocculation, membrane filtration, and advanced oxidation processes, are often costly, energy-intensive, and less efficient at low dye concentrations [9]. As a result, there is increasing interest in the development of alternative, low-cost adsorbents for dye removal, particularly from waste materials like AMD sludge.

This study focuses on neutralizing AMD with lime and assessing the performance of AMD sludge in the removal of anionic dye, particularly congo red from an aqueous solution. By exploring the sludge's adsorption capacity and percentage removal under optimized conditions, this research aims to determine whether this by-product can be effectively repurposed for environmental applications. Furthermore, this study contributes to the broader context of sustainable water treatment practices by addressing the dual challenges of AMD management and industrial dye contamination. Through a combination of laboratory experiments and analytical techniques, this research seeks to provide insights into the feasibility of using AMD sludge as a cost-effective, environmentally friendly adsorbent for the treatment of dye-laden wastewater.

## II. MATERIALS AND METHODS

### A. Sampling

AMD samples were collected from Khwezela colliery in a 5 L high-density polyethylene bottle and refrigerated to avoid precipitation and oxidation of metal ions.

### B. Preparation Of AMD Samples

A cleaned graduated cylinder was used to measure 10 mL of AMD to be diluted. This AMD was diluted with 90 mL of deionized water (10:100 ratio) and then mixed thoroughly. The samples were then stored at 4 °C to maintain integrity until analysis.

### C. Characterization Of AMD

A pH/EC meter (HACH HQ40D, Aqualytic, South Africa) was used to determine the AMD and treated sample's EC ( $\mu\text{S}/\text{cm}$ ) and pH throughout the experiment. The meter was first calibrated with standard buffer solutions before each measurement. The metal content was determined using the ICP-MS instrument with method ME-011.

### D. AMD Neutralization Experiments

#### Neutralization of AMD as a Function of Lime Dosage

A 100 mL of AMD was poured into 6, 500 mL beakers, and 0.3 g, 0.6 g, 1 g, 2 g, 3 g, and 4 g of lime (calcium hydroxide) were added to each beaker. The mixture was then stirred on a 4-paddle stirrer (Model 1924, Electronics, India) at a speed of 150 rpm. The experiments were carried out in triplicate at room temperature of 25 °C. After equilibrium, the mixtures were filtered through a 0.45  $\mu\text{m}$  pore size filter membrane and analyzed. The results were compared and the dosage which worked best in neutralizing the AMD was chosen as the optimum dose.

#### Neutralization of AMD as a Function of Contact Time

100 mL of AMD was poured into 6, 500 mL beakers. Using the optimized dosage (0.3 g), samples were stirred at 15, 30, 45, 60, 90, and 120 minutes at 150 rpm using a 4-paddle stirrer (Model 1924, Electronics, India) at room temperature to determine the optimum time for the reaction. After stirring, the beakers were left undisturbed for 30 minutes for the sludge to settle. When the sludge had settled, the supernatant was transferred to another beaker and characterized.

### E. Sludge Preparation

The resulting sludge, produced at the optimal conditions, was collected and dried using an oven at 105 °C for 2 hours to remove excess moisture. It was then crushed into a fine powder using mortar and pestle and sieved to ensure homogeneity. The powdered sample was then characterized.

### F. Characterization Of Lime And AMD Sludge

The powdered samples (lime and AMD sludge) were characterized using Scanning electron microscopy with energy dispersive x-ray for surface morphology and chemical mapping, X-ray fluorescence for elemental composition, and Fourier-transform infrared spectroscopy for structural composition.

### G. Congo Red Adsorption Experiment

A stock solution of 200 mg/L was prepared by dissolving 0.2 g of CR dye in deionized water and a 10 mg/L diluted solution was prepared from it. Optimized conditions of 10 minutes contact time, 1 g adsorbent dosage, and 10 mg/L initial CR dye concentration as given by Rasilingwani et al., (2024) were applied for the adsorption experiment. 100 mL of 10 mg/L CR dye solution was poured into 3, 500 mL beaker and mixed with 1 g AMD sludge (adsorbent). The solution was then agitated for 10 minutes at a speed of 250 rpm using the 4-paddle stirrer and then left for 30 minutes to settle. After settling, the supernatant was transferred into centrifugal tubes and centrifuged for 15 minutes at 4 000 rpm to separate solid particles from the liquid. The sample was then transferred into cuvettes and the absorbance was measured using Jenway UV/Visible spectrophotometer at 498 nm.

### H. Calculation of %Removal And Adsorption Capacity

The % Removal of CR dye was computed by the following equation:

$$\% \text{ Removal} = \left( \frac{C_0 - C_f}{C_0} \right) \times 100 \quad (1)$$

Where  $C_0$  = initial concentration of CR dye and  $C_f$  = the final concentration of CR dye after adsorption.

Then the adsorption capacity of the AMD sludge for CR dye adsorbed per unit mass of adsorbent ( $q$ ) was computed by the following equation:

$$q = \frac{(C_i - C_e)V}{M} \quad (2)$$

Where  $C_i$  = Initial dye concentration (mg/L),  $C_e$  =

equilibrium concentration (mg/L),  $V$  = volume of adsorbate (dye solution) in (L), and  $M$  = mass of AMD sludge in (g).

### III. RESULTS AND DISCUSSIONS

#### A. Characterization of AMD Results

##### Physicochemical Parameters Of AMD

Table I and II below present the physicochemical parameters of AMD before and after lime neutralization. The treated AMD showed substantial improvement in water quality. The pH increased from 2.68 to 7.89, effectively neutralizing acidity and making the water suitable for industrial use. Electrical conductivity decreased from 748  $\mu\text{S}/\text{cm}$  to 392  $\mu\text{S}/\text{cm}$ , well below the 2,500  $\mu\text{S}/\text{cm}$  limit, indicating reduced ionic content. Turbidity dropped from 8.42 NTU to 1.32 NTU, reflecting the effective removal of suspended particles. Total dissolved solids decreased from 374 mg/L to 196 mg/L, staying well under the 1,000 mg/L guideline. These improvements make the treated AMD compliant with industrial standards.

TABLE I  
pH, EC, TURBIDITY, AND TDS RESULTS OF AMD BEFORE AND AFTER TREATMENT

	pH	EC ( $\mu\text{S}/\text{cm}$ )	Turbidity (NTU)	TDS (mg/L)
Before treatment	2.68	748	8.42	374
After treatment	7.89	392	1.32	196

Table II presents results from ICP-MS analysis determining the metal ion concentrations.

TABLE II  
ICP-MS RESULTS SHOWING THE ELEMENTAL COMPOSITIONS OF AMD BEFORE AND AFTER TREATMENT.

Element composition	Initial (mg/L)	Final (mg/L)	Permissible limits (mg/L)
Sulphate as $\text{SO}_4$	1049.74	219.21	$\leq 250.00$
Calcium as Ca	55.38	43.86	$\leq 200.00$
Magnesium as Mg	51.08	23.08	$\leq 100.00$
Sodium as Na	6.62	3.36	$\leq 200.00$
Zinc as Zn	0.70	ND	$\leq 5.00$
Aluminium as Al	18.42	7.05	$\leq 0.20$
Iron as Fe	102.39	11.78	$\leq 0.30$
Cobalt as Co	0.08	ND	$\leq 0.05$
Nickel as Ni	0.09	ND	$\leq 0.02$
Manganese Mn	7.28	0.003	$\leq 0.10$

Key: ND=Not Detected

The ICP-MS analysis of treated AMD revealed improvements in water quality, but some elements still exceeded permissible limits. Sulphate concentration decreased

to 219.21 mg/L, below the 250 mg/L limit, showing effective neutralization. Potassium, sodium, calcium, and magnesium levels were within acceptable limits, confirming suitability for industrial reuse. However, aluminum (7.05 mg/L), iron (11.78 mg/L), and vanadium (0.181 mg/L) exceeded thresholds, indicating the need for further treatment. Heavy metals like zinc, chromium, cobalt, and nickel were absent, demonstrating the successful removal of trace metals. Despite these improvements, additional treatment is required for some metals to meet stricter standards.

#### B. AMD Neutralization Experiments

##### Neutralization of AMD as a Function of Lime Dosage

Fig. 1 shows a graph of lime dosage versus the pH of AMD. Lime was added at varying dosages and pH levels were monitored over time. The results showed a significant increase in pH with increasing lime dosage.

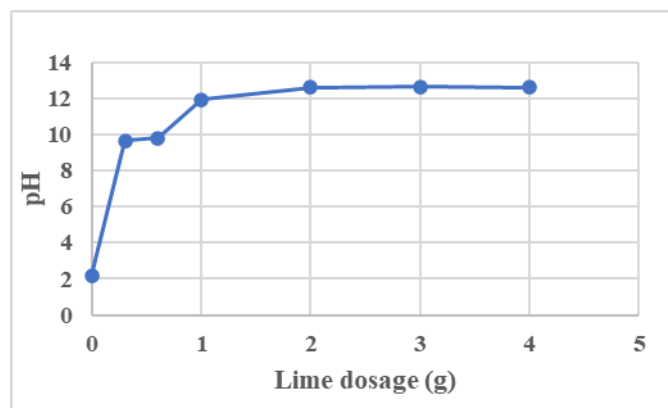


Fig. 1: Neutralization of AMD as a function of lime dosage

The lime neutralization experiment demonstrated that a dosage of 0.6 g of lime per 100 mL of AMD was sufficient to achieve near-neutral pH levels (9.80) without excessive alkalinity. Higher lime dosages (1.0 g and above) resulted in pH levels exceeding 11, which may lead to unnecessary lime consumption and increased operational costs. Additionally, a highly alkaline environment may cause the re-dissolution of some metal precipitates, reducing the overall effectiveness of the treatment. Therefore, 0.6 g was determined to be the optimum lime dosage for this study, providing effective neutralization while avoiding the negative effects of over-treatment.

##### Neutralization of AMD as a Function of Contact Time

Fig.2 shows a graph of AMD neutralization as a function of contact time. The determined optimum dosage was used throughout this experiment to determine the optimum time. The samples were stirred at varying times and pH levels were monitored.

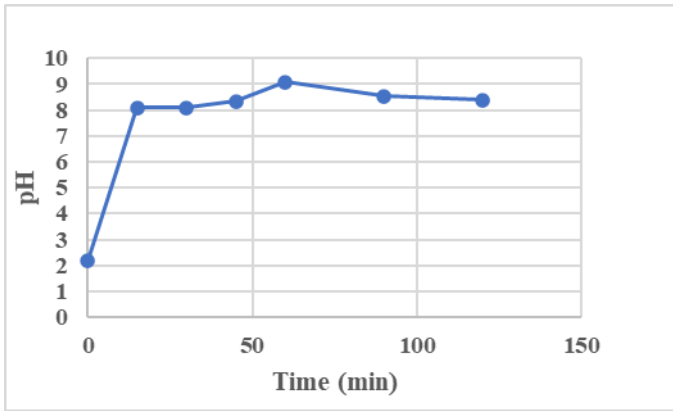


Fig.2: Neutralization of AMD as a function of contact time

The time variation results revealed that most of the pH change occurred within the first 30 minutes of treatment. This suggests that a contact time of 30 minutes is enough to achieve effective neutralization. Extending the reaction time beyond 30 minutes showed minimal additional improvement, supporting the conclusion that this is the optimum contact time for practical applications.

C. Characterization of lime and AMD sludge results

SEM-EDX Analysis Results

Fig. 3 below shows the SEM images of lime and AMD sludge.

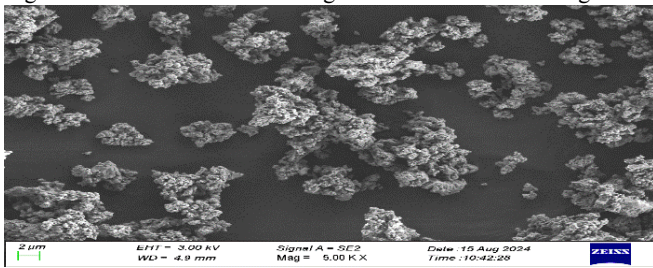


Fig 3(a): surface morphology of lime

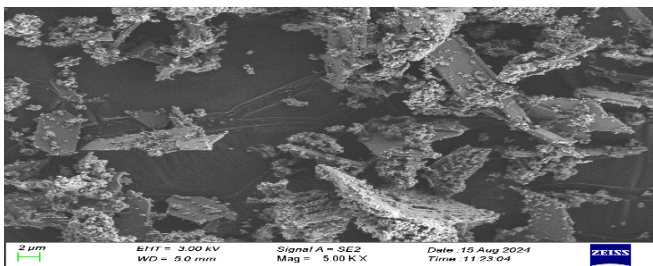


Fig 3(b): surface morphology of AMD sludge

The SEM image of lime reveals a predominantly agglomerated and irregular particle structure with a relatively smooth surface. The SEM image of sludge shows a significant transformation in surface morphology compared to the lime. The sludge particles appear more fragmented, with a rough structure. Additionally, there are visible aggregates and flake-like formations, which indicate the precipitation of metal ions during the AMD neutralization process and the formation of new minerals such as iron oxides, aluminum oxides, and sulfides as corroborated by XRF results.

Fig 4. Below shows the EDX live maps of lime and AMD sludge

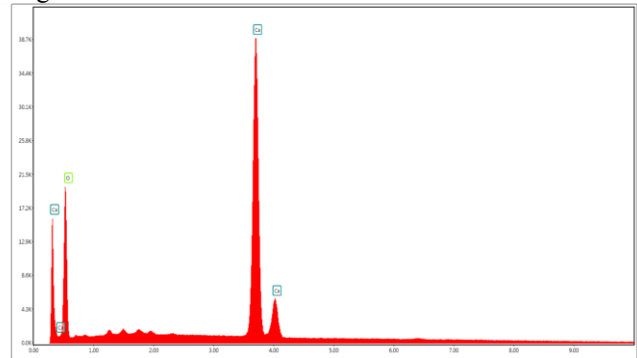


Fig 4(a): Live map of lime

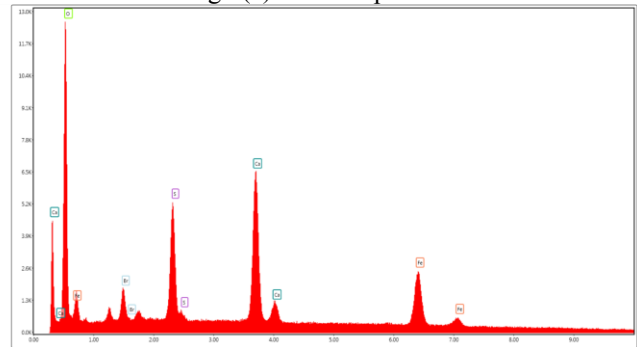


Fig 4(b): Live map of AMD sludge

The elemental analysis of lime before AMD treatment and the resulting sludge after treatment revealed significant compositional changes due to neutralization processes. The lime consisted primarily of calcium (49.83%) and oxygen (50.17%), typical of its structure used for AMD neutralization. The treated sludge showed a more complex composition, with high levels of iron (27.18%), calcium (20.29%), oxygen (43.63%), and sulphur (7.3%), indicating reactions between the lime and acidic components like sulfuric acid. The high iron content highlights effective iron removal through precipitation, and the presence of iron hydroxides enhances the sludge's adsorptive properties, particularly for removing anionic dyes.

D. XRF Analysis Results

Table III shows the elemental composition of lime before and after AMD treatment.

TABLE III  
XRF DERIVED CHEMICAL COMPOSITIONS OF ELEMENTS IN LIME AND AMD SLUDGE.

	Lime (before treatment)	AMD sludge (after treatment)
Element Names	%	%
CaO	71.969	28.343
Fe <sub>2</sub> O <sub>3</sub>	0.407	22.341
MgO	1.431	1.861
Al <sub>2</sub> O <sub>3</sub>	0.000	2.573
S	0.000	13.496



The XRF analysis of lime before and after AMD neutralization showed significant chemical changes, reflecting the treatment process. Magnesium oxide increased from 1.431% to 1.861%, indicating magnesium retention in the sludge. The most significant shift was a reduction in calcium oxide from 71.969% to 28.343%, due to lime consumption during neutralization. Sulphur content rose to 13.496% in the sludge, highlighting effective sulphate removal. Iron oxide increased dramatically from 0.407% to 22.341%, confirming iron precipitation, while aluminium oxide reached 2.573%, reflecting aluminium removal. These changes underscore successful metal and sulphate precipitation during treatment.

**E. FTIR Analysis Results**

FTIR spectrum of lime and AMD sludge are represented in Fig. 3.

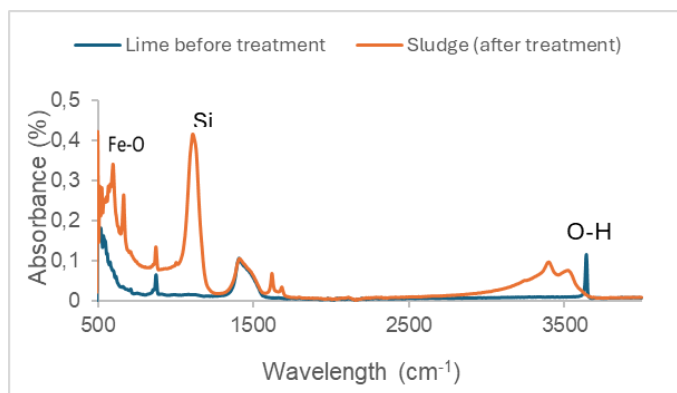


Fig. 3: FTIR scatter graph showing the spectrum of lime particles and AMD sludge reacted with lime.

The FTIR analysis highlights chemical transformations during AMD neutralization, particularly the precipitation of metals and sulphates. Before treatment, the lime spectrum showed strong hydroxyl (O-H) stretching peaks (3200-3600  $\text{cm}^{-1}$ ), indicating free hydroxyl ions in lime. These peaks diminished after treatment, showing that hydroxyl ions were consumed in reactions with sulfuric acid and dissolved metals. Peaks in the sludge spectrum (1400-1500  $\text{cm}^{-1}$ ) corresponded to carbonate groups, suggesting calcium carbonate formation from calcium hydroxide reacting with atmospheric  $\text{CO}_2$ . New peaks (500-800  $\text{cm}^{-1}$ ) indicated iron-oxygen and aluminium-oxygen bonds, confirming the precipitation of metal hydroxides like Fe (III) and Al hydroxides.

**F. Adsorption Experiment Results**

Table IV represents results from adsorption experiment under optimized conditions as given by Rasilingwane *et al.*, (2024).

TABLE IV  
Adsorption of CR dye using AMD sludge

Initial concentration (mg/L)	Final average concentration (mg/L)	%Removal efficiency	Adsorption capacity
10	1.3	87	870

The use of AMD sludge for removing Congo Red (CR) dye demonstrated impressive efficiency, with 87% of the dye removed from a 10 mg/L solution, reducing it to 1.3 mg/L. The sludge’s adsorption capacity of 870 mg/g highlights its potential as a low-cost alternative to conventional adsorbents like activated carbon. The removal process is driven by electrostatic interactions and chemical bonding, with metal hydroxides in the sludge, such as calcium and iron oxides, promoting the adsorption of the anionic dye. This rapid and effective adsorption, observed within 10 minutes, is attributed to the high number of active sites available in the sludge.

Compared to other low-cost adsorbents, the performance of AMD sludge is favorable, particularly in terms of its cost-effectiveness and sustainability. As a waste byproduct from acid mine drainage treatment, it is readily available, especially in mining regions like South Africa. This offers dual benefits—reducing mine drainage waste while providing a sustainable, low-energy solution for wastewater treatment. The environmental advantages of using AMD sludge, which requires minimal processing compared to synthetic adsorbents like activated carbon, make it an attractive, eco-friendly option for industries.

**IV. CONCLUSION**

This study addressed the dual environmental challenges of AMD and dye pollution, focusing on using lime to neutralize AMD and repurposing the resulting sludge for dye removal. Lime treatment effectively raised the pH of AMD and precipitated heavy metals, transforming contaminated water into a safer resource for reuse, though additional treatment is recommended for metals like aluminium, vanadium, and iron. Characterization of the sludge via SEM-EDX, XRF, and FTIR showed favorable properties for adsorption, with experiments achieving a remarkable 87% removal of anionic dyes under optimal conditions.

The study identified a lime dosage of 0.3 g and a contact time of 30 minutes as optimal for neutralizing AMD, achieving a neutral pH suitable for industrial recycling. The sludge’s high dye removal efficiency suggests its potential as a sustainable adsorbent for wastewater treatment. Both the treated water and the dye-laden effluent met South African industrial water quality guidelines. This integrated approach addresses AMD and dye pollution while promoting water resource conservation.

In conclusion, this research highlights the importance of repurposing waste materials like AMD sludge for sustainable industrial practices. It demonstrates that AMD sludge can serve as a low-cost, effective solution for dye removal, reducing the environmental impacts of mining and manufacturing. Future research should focus on scaling this approach, exploring its effectiveness for other pollutants, and assessing its economic feasibility to further support responsible environmental management and water security in water-scarce regions.

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#### REFERENCES

- [1] Gupta, G., Singh, S. K. and Chandra, R. Environmental Impact of Acid Mine Drainage: Challenges and Solutions. *Journal of Water Resource and Protection*, 12(7), pp. 612-625, 2020.
- [2] Johnson, D. B. and Hallberg, K. B. Acid Mine Drainage Remediation Options: A Review. *Science of the Total Environment*, 603, pp. 144-154, 2019.
- [3] Kalin, M., Fyson, A. and Wheeler, W. N. The Chemistry of Acid Mine Drainage: Its Treatment and the Development of Sustainable Solutions. *Mine Water and the Environment*, 39(2), pp. 257-267, 2021.
- [4] Rao, G. V., Bhargava, M. and Chakrabarti, S. Valorization of Acid Mine Drainage Sludge for Environmental Applications. *Journal of Hazardous Materials*, 424, pp. 127-134, 2022.
- [5] Akcil, A. and Koldas, S. Acid Mine Drainage (AMD): Causes, Treatment, and Case Studies. *Journal of Cleaner Production*, 64, pp. 701-710, 2019.
- [6] Choi, J., Lee, J. and Kim, S. Neutralization of Acid Mine Drainage Using Lime: An Efficient Approach for Sustainable Mining Practices. *Water Research*, 184, p. 115947, 2020.
- [7] Rodríguez, L., Pérez, M. and López, E. Adsorption of Heavy Metals Using Acid Mine Drainage Sludge: A Novel Approach for Waste Valorization. *Journal of Hazardous Materials*, 421, pp. 1-12, 2022.
- [8] Qiu, X., Zhao, W. and Shi, L. Removal of Anionic Dyes from Wastewater Using Adsorbents Derived from Waste. *Journal of Environmental Chemical Engineering*, 9(5), p. 105892, 2021.
- [9] Gong, J., Liu, Y. and Yue, X. Adsorption of Anionic Dyes by Metal Oxide-Based Adsorbents Derived from Mining Wastes. *Journal of Environmental Management*, 259, p. 109679, 2020.