A Lime-Kaolin Clay Composite for the Active Treatment of Acid Mine Drainage from Sibanye Stillwater, Gauteng Province, South Africa

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***Abstract*—Acid mine drainage, a byproduct of large-scale mining in South Africa, continues to impact aquatic ecosystems and water resources. Therefore, this study aimed to synthesize and characterize a lime-kaolin clay composite and evaluate its efficiency in treating AMD relative to lime and kaolin clay used individually. Inductively coupled plasma mass spectrometry analysis of AMD confirmed a significant reduction in heavy metals, addressing one of the major water challenges at Sibanye-Stillwater. Characterization of the composite using Scanning electron microscopy, Fourier-transform infrared spectroscopy, and X-ray Fluorescence revealed a successful combination of lime and kaolin clay traits, along with interactions and introduction of metals on the surface of the sludge. Lime-only treatment increased the pH above 10; kaolin-only treatment raised the pH to near-neutral levels. At the same time, the composite achieved stable neutralisation (pH ≈ 8.3) AMD under both circumneutral and acidic conditions. The composite further achieved zinc removal efficiencies of 99.4% in raw AMD and 88.2% in pH-adjusted AMD. These results highlight the composite as a low-dose and effective material with strong potential for sustainable AMD remediation.**

***Keywords*— Acid mine drainage, lime-kaolin composite, pH neutralization, and metal remediation.**

I. Introduction

Acid mine drainage (AMD) is one of the pressing causes of water pollution, primarily driven by the oxidation of sulphide minerals upon exposure to atmospheric, biological, and hydrological elements such as oxygen, chemoautotrophic bacteria, and water. The uncontrolled release of AMD from abandoned coal, gold, and platinum mines has critically damaged and degraded aquatic and terrestrial ecosystems in South Africa. Approximately 60,000,000 liters of metal-rich mine wastewater are discharged into nearby aquatic ecosystems daily [1]. These effluents are typically characterized by very acidic pH, elevated concentrations of toxic metals, and high electrical conductivity, which leads to poor water quality and the inability to support aquatic life.

Sibanye-Stillwater is among the largest producers of gold and platinum group metals in South Africa, operating extensive mining activities in the Witwatersrand Basin and Bushveld Complex. The legacy of these operations has resulted in the generation of vast quantities of mine waste, including tailings and mine-impacted water, which continue to pose long-term environmental challenges [2]. Gold and platinum mining waste streams are particularly problematic due to their high sulphide mineral content. For example, gold mine tailings in South Africa often contain substantial pyrite, and sulphide minerals have been shown to continue generating AMD even after mining operations have ceased [3]. Interestingly, while AMD is commonly associated with very low pH, it may also occur at circumneutral (pH 6-8) or even strongly basic (pH 8-12), depending on site-specific conditions [4]. Despite this, circumneutral AMD can still contain elevated metal loads and sulphates, posing significant risks to water quality, aquatic ecosystems, and human health in mining-affected areas.

Globally, AMD is considered a significant environmental issue due to its persistence, treatment challenges, and severe impacts on water bodies and ecosystems. To mitigate its impacts, AMD must be properly treated to reduce contaminant levels to those acceptable under the Department of Water and Sanitation regulations. Various technologies have been developed for AMD treatment, including precipitation, adsorption, biosorption, ion exchange, membrane techniques, and desalination, with some also enabling the recovery of valuable metals. However, these methods often face limitations at an industrial scale. Neutralization remains the most widely applied treatment, where AMD is reacted with alkaline agents such as lime, sodium hydroxide, or sodium carbonate to raise pH and precipitate metals [5]. Natural adsorbents, including clay minerals, chitin, and coconut shells, are increasingly utilized for AMD treatment due to their low cost, widespread availability, and effectiveness in reducing heavy metal concentrations [6]. Kaolin clay possesses active O-H sites capable of binding positively charged metal ions, fine particle size, and high surface area, making it effective in adsorption and cation exchange [7].

This study, therefore, has prepared a composite material from lime and kaolin clay. The lime-kaolin clay composite works synergistically by combining the properties of lime and kaolin clay to enhance the treatment of AMD. Their combined action provides better pH control, heavy metal removal, sulfate reduction, and sludge stability than using each material alone.

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While kaolin clay offers buffering capacity, lowering extreme pH fluctuations, and adsorbing the precipitated metals, lime quickly increases pH, neutralizing AMD acidity and precipitating metals. Using lime-kaolin clay composite reduces the total amount of lime required for neutralization of AMD. This composite provides an eco-friendly solution to AMD issues.

II. MATERIALS AND METHODS

A. Sampling

The AMD sample used in this study was collected from the Sibanye Stillwater mine in a 25 L plastic drum and preserved using standard measures. For analysis, the sample was transferred into glass bottles and stored in the refrigerator at 4 °C to maintain chemical integrity

B. Preparation of lime-kaolin clay composites

A series of lime-kaolin clay composites with varying lime-to-kaolin ratios was prepared to investigate their effectiveness in treating acid mine drainage (AMD). The composites were prepared using a mortar and pestle to ensure thorough mixing. For each composite, a total of 30 g or 20 g of material was prepared according to the following ratios: 2:1 ratio: 20 g of lime to 10 g of kaolin clay, 1:2 ratio: 10 g of lime to 20 g of kaolin clay, and 1:1 ratio: 10 g of lime to 10 g of kaolin clay. After mixing, the composites were passed through a 106 µm test sieve to ensure homogeneity.

C. Characterization of AMD samples

The pH, electrical conductivity (EC), and total dissolved solids (TDS) of the AMD samples were measured at three stages: before pH adjustment, after pH adjustment, and after treatment with the composites. Measurements were conducted using a Hach multiparameter water quality meter (Model: HQ40d, Hach, USA) for EC, and a benchtop pH meter (Accsen pH 8, Lasec, South Africa) for pH. TDS values were calculated by multiplying the measured EC by a constant factor of 0.85.

D. pH Adjustment of AMD samples

Circumneutral AMD was acidified to a pH of 2.59 using 1M HCI to provide consistent initial conditions for better assessment of the lime-kaolin clay composite's neutralization and metal removal efficiency.

E. Evaluation of Composite Efficacy in AMD Treatment

To assess the effectiveness of each composite, 0.5 g of each lime-kaolin clay ratio was added separately to 100 mL of AMD in individual beakers. The mixtures were stirred at 200 rpm using a jar test apparatus for 1 hour. The pH and EC were measured at 20-minute intervals (0, 20, 40, and 60 minutes). The composite demonstrating the fastest and most effective pH neutralization was selected for further optimization.

F. Treatment Procedure for Raw AMD and pH-Adjusted Sample with Individual Lime and Kaolin Clay Applications.

A mass of 0.2 grams of each material, lime and kaolin clay (equivalent to the optimal dose of the composite), was used to treat both the raw AMD sample and pH-adjusted AMD samples. Each treatment was performed in triplicate, and the mixtures were stirred at 200 rpm for 10 minutes to ensure uniform mixing and sufficient contact between the materials and the solution. After stirring, EC, TDS, and pH of the samples were measured to assess the effectiveness of the treatment using individual materials.

G. Zinc Recovery Using Optimized Composite

For zinc recovery experiments, 100 mL of both raw and pH-adjusted AMD samples were placed in separate beakers. The optimized composite (2:1 lime-to-kaolin ratio) was added in two doses (0.1 g and 0.2 g) to the respective beakers. Each mixture was stirred continuously at 200 rpm for 10 minutes. The target pH for effective zinc precipitation was between 8.0 and 8.5 [12]. Following stirring, the solutions were allowed to settle for 20 minutes and then filtered using 0.45 µm filter paper. The filtrates were collected for zinc analysis using ICP-MS to confirm zinc recovery.

H. Characterization of Materials and Treatment Residues

Material characterization was conducted to assess the properties of lime, kaolin clay, their composites, and the resulting sludge. Particle size distribution was determined through laser diffraction and sieve analysis using a Mastersizer 3000, while surface morphology and elemental composition were examined via SEM–EDS. Functional groups were identified using FTIR spectroscopy in the 4000–400 cm^{-1} range, and chemical composition, including major oxides and trace metals, was quantified using XRF analysis. These methods collectively provided insight into the structural, chemical, and morphological characteristics influencing AMD treatment performance.

I. Calculation of the % metal recovery efficiency of lime-kaolin clay composite.

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

Given that: C_o = initial concentration of the Zn^{2+} before neutralization, and C_e = equilibrium Zn^{2+} concentration.

III. RESULTS AND DISCUSSIONS

A. Characterization of AMD Results

Physicochemical Properties of AMD

Tables I below present the physicochemical properties of AMD before and after treatment with the lime-kaolin clay composite. The composite achieved slightly alkaline conditions (8.33) in both acidic and circumneutral AMD while simultaneously decreasing the ionic load of the AMD.

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TABLE I: PHYSICOCHEMICAL PARAMETERS OF RAW AND PH-ADJUSTED AMD SAMPLES

Parameters	Raw Sample	pH-Adjusted Sample
EC (µS/cm)	260.30	418.33
pH	5.94	2.59
TDS (mg/L)	221.25	355.58

TABLE II: PHYSICOCHEMICAL PARAMETERS OF TREATED RAW AND PH-ADJUSTED AMD SAMPLES

Parameters	Raw sample	pH-adjusted Sample	South African Water Quality Guidelines Volume 4
EC (µS/cm)	252.67	335.33	≤ 400
pH	8.33	8.32	6.5-8.4
TDS (mg/L)	214.77	285.03	≤ 260

TABLE III: ICP-MS ANALYSIS OF ELEMENTAL CONCENTRATIONS BEFORE AND AFTER TREATMENT WITH THE LIME-KAOLIN CLAY COMPOSITE.

Elemental composition	Initial metal content of raw AMD	Final metal content of raw AMD	Initial metal content of PH-adjusted AMD	Final metal content of pH-adjusted AMD	SA Water Quality Guide line Volume 4
Zinc	0.018	0.0001	0.034	0.004	≤1.0
Copper	0.023	0.002	0.001	-0.001	≤2.0
Fluoride	0.08	0.21	0.17	ND	≤2.0
Magnesium	11.20	61.09	7.59	5.87	0.02- - 10.0

Key: ND = Not Detected

The ICP-MS results show that the lime-kaolin clay composite achieved significant zinc removal from AMD. In raw AMD, the initial Zn concentration of 0.018 mg/L was reduced to 0.0001 mg/L, corresponding to a 99.4% removal efficiency. Similarly, in pH-adjusted AMD, Zn was reduced from 0.034 mg/L to 0.004 mg/L, equivalent to an 88.2% removal efficiency. These results demonstrate effective removal to levels well below the South African water quality guidelines, making the treated water suitable for agricultural use. In the acidic sample, significant removal of other metals like F, Mg, and Cu was observed, whereas in the raw sample, these metals increased, suggesting possible desorption or dissolution from the treatment medium. Although the results demonstrate promising metal removal efficiency, inconsistencies in F and Mg behavior highlight the need for further optimization of the composite ratio, contact time, and pH control to improve stability and reproducibility.

A. *Neutralization Experiment Results*

Figure 1 presents the effect of different composite dosages on the AMD sample over 10 minutes, illustrating their efficiency in neutralizing pH.

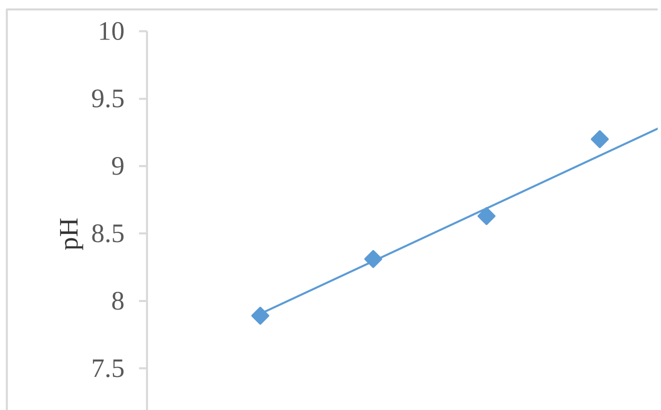

Fig. 1: Optimization of Composite Dosage Based on pH Adjustment over 10 minutes. (Experimental conditions: 200 rpm agitation speed, 10 min contact time, 25 °C, 2 mL HCl per 100 mL AMD).

As shown in Fig. 1, both 0.2 g and 0.3 g dosages of the lime-kaolin clay composite rapidly increased the pH of AMD from 2.59 to above 8.0 within 5 minutes. After 10 minutes, the pH stabilized between 8.2-8.6, suggesting that equilibrium was reached. Notably, there was minimal difference between the two dosages, indicating that a smaller dosage (0.2 g) was sufficient to achieve the desired neutralization effect.

Fig. 2 presents the pH-adjusted measurements compared with the raw AMD sample, highlighting the effect of treatment on acidity.

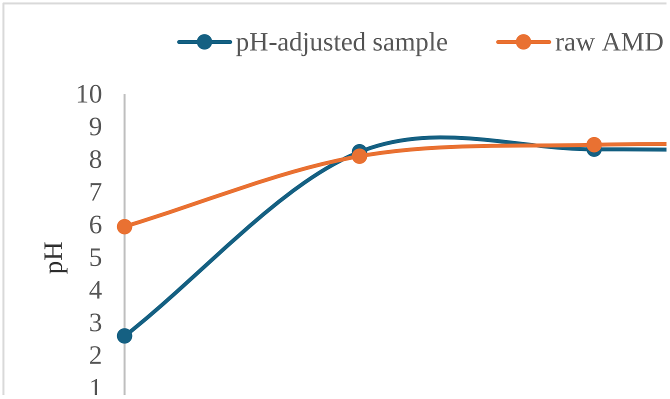

Fig. 2. Time optimization profile of raw and pH-adjusted AMD samples.

The pH curves of both the raw and pH-adjusted AMD samples following treatment with 0.2g of lime-kaolin composite are shown in Fig. 2. A rapid increase in pH was observed from 2.59 to approximately 8.2 in the pH-adjusted sample and from 5.94 to 8.1 in the raw AMD sample. After this sharp initial increase, the pH values gradually stabilized, reaching equilibrium around the 10-minute mark. Beyond 10 minutes, no significant changes in pH were recorded, with values remaining within the range of 8.2-8.5 for both samples. Therefore, 10 minutes was chosen as the optimal contact time, since it reduces unnecessary reaction time and energy input.

B. *Composite Efficacy in AMD Treatment*

TABLE IV: COMPARISON OF THE EFFICIENCY OF COMPOSITE RATIOS IN NEUTRALIZING AMD

Composite	Time (minutes)	pH	EC
Composite 1 (1:1) (lime: kaolin)	20	8.05	229.29
	40	8.74	298.33
	60	8.55	234.07
Composite 2 (1:2) (lime: kaolin)	20	8.40	239.85
	40	8.79	301.56
	60	8.39	304.33
Composite 3 (2:1) (lime: kaolin)	20	9.25	242.71
	40	9.32	290.33
	60	9.35	288.67

Table IV shows that all composites effectively raised the AMD pH to alkaline conditions within 20 minutes. However, the 2:1 lime-to-kaolin ratio (Composite 3) achieved the highest pH values (up to 9.35), indicating superior neutralization capacity. The higher lime content in Composite 3 increased hydroxide ion availability, facilitating metal hydroxide precipitation. Kaolin likely contributed additional adsorption sites, improving stability and buffering effects. Thus, the 2:1 ratio was identified as the optimal composite formulation for AMD treatment.

C. *Characterization of lime and AMD sludge results*

Figure 3 below presents particle size distribution analysis conducted to assess the textural characteristics of the materials.

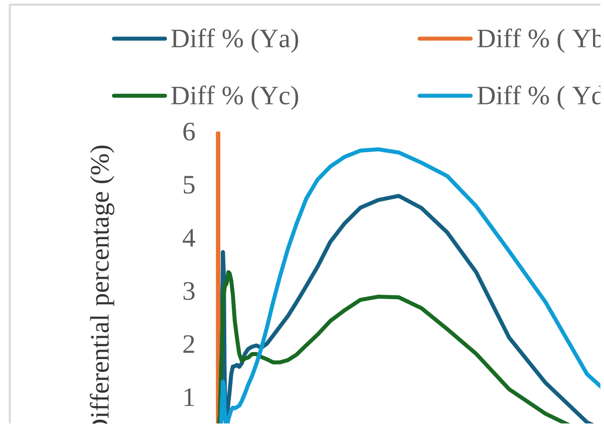

Fig. 3: Differential particle size distribution of lime (Ya), kaolin (Yb), lime-kaolin composite (Yc), and reacted AMD sludge (Yd).

Kaolin (Yb) exhibited the highest differential percentage (Diff%) at finer particle sizes, which was indicative of a large specific surface area. This fine texture was advantageous for AMD treatment because it enhances adsorption capacity and facilitates chemical interactions with dissolved metals. In contrast, lime (Ya) and sludge (Yd) displayed peak Diff% at larger particle diameters, suggesting lower surface areas and slower reactivity. The lime-kaolin composite (Yc) demonstrated an intermediate Diff%, reflecting a combination of the fine reactive kaolin and the coarser lime particles. This blended PSD likely provides a balanced performance: the finer kaolin fraction promotes surface-mediated metal removal, while the lime fraction contributes to rapid neutralization of AMD acidity.

Fig. 4 below shows the SEM images of lime, kaolin clay, composite, and AMD-reacted sludge at 10.00 magnification.

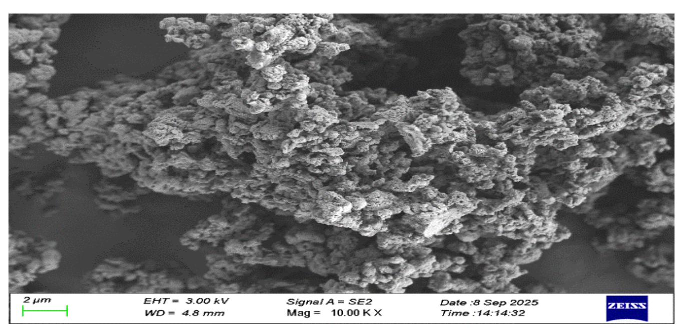

Fig. 4.1: surface morphology of lime

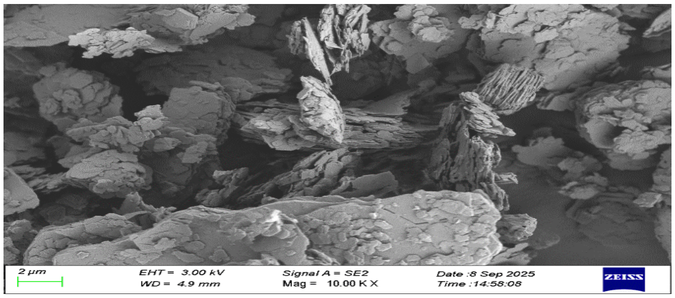

Fig. 4.2: surface morphology of kaolin clay

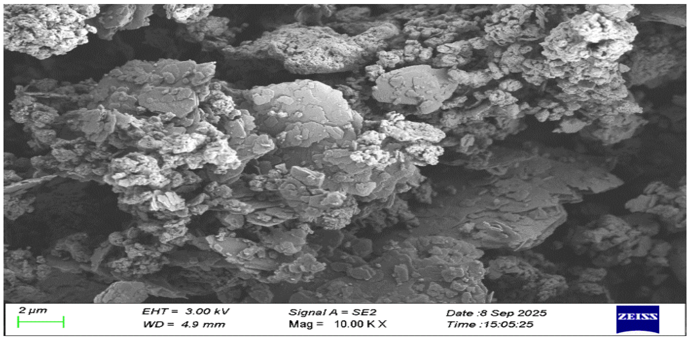

Fig. 4.3: surface morphology of lime-kaolin clay composite

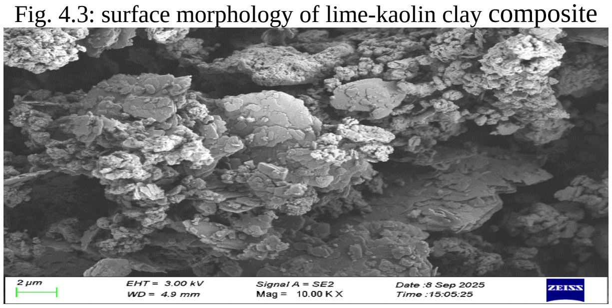

Fig.4.4: surface morphology of AMD-reacted Sludge

The kaolin clay (Fig. 4.2) has irregularly shaped particles with rough surfaces and various sizes. This diverse morphology is important because the rough, uneven surfaces increase the reactive area, which can boost the adsorption of metal ions and interaction with AMD components. In contrast, lime (Fig. 4.1) shows larger, more angular particles with smoother surfaces, reflecting its main role in pH neutralization rather than adsorption. The lime-kaolin composite (Fig. 4.3) exhibits features of both materials, with finer kaolin particles attached to or mixed with coarser lime particles, suggesting a structure that may enhance both neutralization and metal removal. AMD sludge (Fig. 4.4) contains aggregated, irregular particles, indicating the heterogeneous mix of precipitated metals and residual solids from the AMD. SEM characterization confirms that the surface morphology and particle arrangement directly influence reactivity, adsorption capacity, and settling behavior, which are critical factors in optimizing the performance of the composite material in AMD treatment.

D. Energy Dispersive X-ray Spectroscopy (EDS) results

TABLE V: EDS ELEMENTAL COMPOSITION OF KAOLIN, LIME, AMD SLUDGE, AND LIME-KAOLIN CLAY COMPOSITE.

Sample	Element	Weight %
Kaolin clay	O	53.33
	AI	16.76
	Si	21.87
Lime	O	49.73
	Ca	46.76
Lime-kaolin Composite	O	56.50
	Al	6.65
	Si	7.43
	Ca	26.04
AMD-reacted Sludge	Mg	0.20
	Mn	1.71
	Zn	0.60
	Fe	18.86

As shown in Table V, Kaolin was dominated by oxygen, silicon, and aluminum, confirming its clay structure, which contributes to its adsorption capacity. Lime contained a high calcium content, consistent with its role in neutralizing acidity and precipitating metals as hydroxides. The 2:1 lime-kaolin composite possesses the high calcium content of lime with the silicon and aluminum of kaolin, demonstrating a material with both neutralization and adsorption potential.

E. FTIR Analysis results

Fig. 5. below presents the absorbance spectra of the raw reagents (lime and kaolin), lime-kaolin clay composite, and the AMD-reacted sludge.

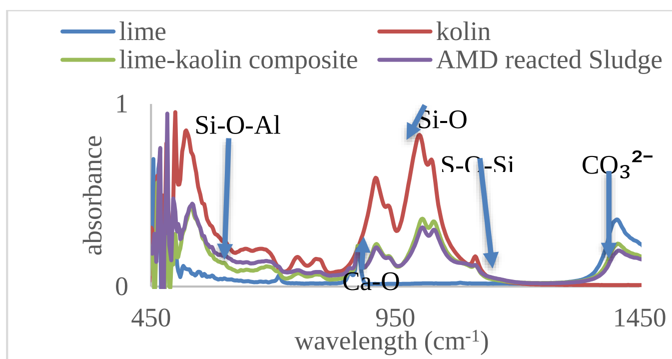

Fig. 5: FTIR characterization of lime, kaolin, lime-kaolin composite, and sludge.

Figure 5presents the absorbance spectra of lime, kaolin, the lime-kaolin composite, and the AMD-reacted sludge across the 450-145o cm^{-1} range. The spectrum of kaolin displayed strong, sharp peaks around 450-500 cm^{-1} and 950 cm^{-1}, which are characteristic of hydroxyl stretching and Si-O vibrations in aluminosilicate minerals. In contrast, lime (CaO) exhibits weak and broad absorbance features in the 450-1450 cm^{-1} region due to its ionic Ca-O bonds, which lack the complex vibrational modes found in silicate and hydroxyl-bearing minerals. The 2:1 lime-kaolin composite showed attenuated kaolin peaks and enhanced lime-related features, confirming the influence of the higher lime fraction and the dominance of CaO. After treatment, the AMD-reacted sludge spectrum displayed diminished kaolin peaks and the appearance of broader absorptions, particularly near 950-1450 cm^{-1} and in the 3450-3550 cm^{-1} region, suggesting the formation of new phases such as gypsum and iron oxyhydroxides, which overlapped the original lime and kaolin signals.

F. XRF Analysis results

TABLE VI: ELEMENTAL COMPOSITION IN LIME, KAOLIN CLAY, LIME-KAOLIN CLAY COMPOSITE, AND AMD-REACTED SLUDGE.

Elemental Composition	Lime %	Kaolin clay %	Lime-kaolin clay Composite %	AMD-reacted Sludge %
MgO	0.846	0.257	0.538	0.730
Al_2O_3	0.000	33.512	9.001	8.149
SiO_2	0.000	44.500	20.593	15.547
Zn	0.000	0.000	0.000	0.0041
MnO	0.004	0.003	0.006	0.858

The lime-kaolin composite exhibited a mixed composition with CaO (40.657%) as the dominant oxide, followed by SiO_2 (20.593%) and Al_2O_3 (9.001%), which enhances its neutralization capacity, while the kaolin fraction contributes surface functional groups that support metal adsorption (Table IV). The AMD-reacted sludge showed significant compositional changes compared to the raw materials, elevated Fe_2O_3 (13.745%) and MnO (0.858%) in the sludge, confirming the removal of iron and manganese from AMD through precipitation. Furthermore, the detection of trace metals such as Cu, Zn, Ni, and As in the sludge, which were absent in the initial materials, demonstrates effective immobilization of these contaminants.

G. Performance of Individual lime and kaolin clay compared to the composite.

TABLE VII: THE PHYSICOCHEMICAL CHARACTERISTICS OF THE TREATED RAW AND PH-ADJUSTED AMD SAMPLES AFTER LIME AND KAOLIN CLAY APPLICATION.

Parameters	Lime(Raw AMD)	Kaolin clay (Raw AMD)	Lime (pH-adjusted AMD)	Kaolin Clay pH-adjusted AMD)
EC (µS/cm)	253	255	273	361
pH	10.46	8.82	7.76	2.92
TDS (mg/L)	215.05	216, 75	215.05	216,75

Lime treatment rapidly increased the pH of the raw acid mine drainage (AMD) sample to 10.46, demonstrating strong alkalinity due to its high CaO content, while kaolin clay raised the pH to 8.82, indicating moderate neutralization. For the acidic (pH 2.59) sample, lime achieved effective neutralization (pH 7.76), whereas kaolin showed limited buffering capacity (pH 2.92). The lime-kaolin composite produced balanced pH values of 8.33 (raw) and 8.32 (pH-adjusted), moderating lime's high alkalinity and enhancing kaolin's weak neutralization, yielding a stable pH suitable for discharge.

At a dosage of 0.2 g, the composite achieved comparable treatment efficiency to individual materials while reducing lime usage and sludge formation. This synergistic interaction between lime and kaolin improved water quality and reduced waste generation, highlighting the composite's potential as a sustainable material for AMD treatment.

IV. CONCLUSION

The study evaluated the potential of a lime-kaolin clay composite in treating acid mine drainage, focusing on the neutralization of acidity and the removal of heavy metals compared to lime- and kaolin-only treatments. This approach responds to the persistent environmental challenge of AMD and the need for low-cost, readily available adsorbents suitable for South African conditions. Among the tested ratios, the 2:1 lime-kaolin composite exhibited the highest performance, rapidly neutralizing AMD and stabilizing the pH from strongly acidic conditions to approximately pH 8, which falls within the South African Water Quality Guidelines Volume 4 for agricultural use.

The composite was highly effective under optimal conditions(10 minutes contact and 0.2 g dosage), achieving results comparable to lime- and kaolin-only treatments. This shows that a smaller amount of each material can achieve similar treatment efficiency, thereby minimizing the sludge generation and improving the recovery of useful resources.

Characterization using FTIR, XRF, SEM, and PSD analysis confirmed that the composite successfully combined the alkaline reactivity of lime with the adsorptive surface properties of kaolin. Although zinc (Zn^{2+}) was the primary metal studied, other metals such as Fe and Mg were also detected in the sludge, indicating that the composite can immobilize a broader range of metals. ICP-MS results showed Zn removal efficiencies of 99.4% in raw AMD and 88.2% in pH-adjusted AMD, demonstrating that the composite remained effective even under highly acidic conditions. The composite's performance at low dosages, coupled with its fabrication from locally available, low-cost materials, underscores its practicality and economic suitability for large-scale AMD treatment in South Africa.

Future work must focus on pilot-scale testing to evaluate large-scale application, optimization of lime-to-kaolin ratios and dosages for different AMD compositions, long-term stability assessment, and cost-benefit evaluation of the composite in AMD treatment.

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