

Enhanced Removal of Heavy Metals from Acid Mine Drainage Using Alkali-Leached Fly Ash as a Modified Adsorbent

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Abstract

Acid mine drainage (AMD) is a major environmental concern characterized by low pH and elevated concentrations of heavy metals such as iron and manganese, requiring effective and sustainable treatment methods. This study investigates the use of industrial fly ash waste as a low-cost adsorbent, enhanced through alkali-leaching modification to produce a zeolite-like material with improved adsorption performance. The novelty of this work lies in the application of alkali-leached fly ash to simultaneously enhance adsorption capacity and neutralization ability for AMD treatment. Adsorbent characterization was conducted using scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDX), X-ray diffraction (XRD) and Brunauer–Emmett–Teller (BET) analyses. BET results showed that modification significantly increased the specific surface area (14 m²/g) and pore volume (0.025 cm³/g). SEM-EDX results revealed a predominantly spherical morphology and changes in elemental composition following modification. XRD analysis showed partial dissolution of minor phases during alkali leaching, resulting in increased dominance of the quartz (SiO₂) phase with a main peak at 2θ = 26.59°. Batch adsorption experiments were performed under varying adsorbent dosages (10–100 g) to evaluate metal removal and pH improvement. The results demonstrated that modified fly ash increased AMD pH from 3.45 to 7.21 and achieved high removal efficiency of Mn, reducing its concentration to 0.05 mg/L (removal efficiency > 95%). Compared to untreated fly ash, the modified adsorbent exhibited significantly enhanced performance. The treated AMD met Indonesian Class I water quality standards. These findings highlight the potential of alkali-leached fly ash as an effective and sustainable adsorbent for AMD remediation.

Keywords

acid mine drainage, adsorption, alkali leaching, fly ash, zeolite-like material

1 Introduction

The formation of acid mine drainage (AMD) is caused by the oxidation of sulphide minerals, such as pyrite (FeS₂), which are exposed to the soil surface during mining [1]. Acid mine drainage is an outcome of the oxidative reaction of sulfide minerals [2]. The oxidation process occurs due to the presence of pyrite, water and oxygen [3]. These metal sulphides then form acids after oxidation. Acid mine drainage is acidic water (high acidity, as can be seen by its low pH value below 4 (pH < 4) and high metal solubility, such as Mn [4]. Improper treatment of acid mine drainage can be harmful to several constituents in the environment due to environmental pollution caused by the oxidation of sulphides carried by water flow. The release of untreated

AMD into natural waters poses a serious threat to aquatic ecosystems, damaging biodiversity and polluting surface water and groundwater sources used for human consumption and the environment [5]. Faced with this threat, the development of effective, efficient, and sustainable AMD treatment methods has become a top priority in environmental engineering. The solution to this problem is to treat acid mine drainage so that it does not pollute the surrounding environment [6].

One alternative method for treating acidic mine drainage is adsorption methods. Adsorption is one of the preferred process units that can be used to remove pollutants from various type of wastewater. The capacity of

the adsorbent to absorb a solution affects the wastewater process [7]. Currently, adsorption is the most effective, low-cost process and is often used to reduce heavy metal pollutants [8–10]. In addition, adsorption is a simple, easy-to-use, and cheaper method [11, 12]. The types of adsorbent materials that have been widely developed are fly ash [5, 13–15]; fly ash and red mud [16]; bottom ash and oyster shells [17]; ion-exchanged zeolite and fly ash [18]; zeolite-type adsorbents from fly ash [19].

Currently, the utilization of waste from steam power plants (PLTU) as a source of adsorbent raw materials [20] is a growing research focus, one of which is fly ash. This material is dominated by amorphous aluminosilicate content and other minerals that have the potential for adsorbent properties. However, raw fly ash still has limitations, particularly low specific surface area and suboptimal pore distribution, which limits its pollutant absorption capacity. To overcome these constraints, physical and chemical modifications are required, one of the most promising methods being alkali leaching treatment. This study aims to analyze changes in fly ash characteristics before processing, after heating, and after modification through various characterization techniques, including scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDX), X-ray diffraction (XRD) and Brunauer–Emmett–Teller (BET) analysis to evaluate the changes in specific surface area, pore volume, morphology and elemental composition. Development efforts were carried out through an alkali leaching-based zeolite-like modification (ZLM) approach to improve the function of fly ash as an adsorbent. The alkali leaching method was recognized as an efficient, cost-effective, and straightforward technique for synthesizing zeolitic materials from coal combustion residues. This method effectively extracts reactive silica and alumina prior to the hydrothermal process, facilitating the formation of zeolite structures with desirable physicochemical properties.

Although the potential of modified fly ash has been widely studied, the studies that specifically highlight morphological and compositional changes due to alkali leaching and its application in the adsorption of certain heavy metal ions, such as manganese in AMD, are still limited. The novelty of this study lies in the development of a ZLM material produced through an alkali leaching treatment of fly ash, which has not been widely studied to achieve AMD quality in accordance with Class 1 of water quality standards in Indonesia, making it suitable for sanitation purposes. The results of this research

are expected to contribute to the development of sustainable and cost-effective remediation technology by utilizing industrial waste as a valuable resource. This research offers an innovative approach to the treatment of AMD by utilizing other industrial solid waste (fly ash) in an effort to neutralize the negative environmental impact of both types of waste. Particular focus is placed on raising the pH and reducing the concentration of heavy metals, particularly manganese, which is often difficult to remove to meet clean water standards.

2 Methodology

2.1 Materials

The materials used in this study consisted of fly ash obtained from coal combustion at the Tanjung Enim PLTU, acid mine drainage (pH 3.45) collected from the operational area of PT Bukit Asam (Persero) Tbk, both located in South Sumatera, Indonesia. Supporting chemicals, 1 M aqueous sodium hydroxide solution (CAS No. 1310-73-2) from CV Ilham Jaya Palembang, Indonesia and distilled water (CAS No. 7732-18-5) were used for sample modification and preparation.

2.2 Methods

2.2.1 Characterization of acid mine drainage

In this study, the key parameters analyzed in the treatment of acid mine drainage included pH, total suspended solids (TSS), chemical oxygen demand (COD), biochemical oxygen demand (BOD), and total dissolved solids (TDS), Fe and Mn concentrations using American Public Health Association (APHA) Standard Methods. The quantification of metal ions (Mn and Fe) was conducted by using atomic absorption spectroscopy (AAS equipment, manufactured by GBC Scientific Equipment, Australia).

2.2.2 Characterization of fly ash

The physicochemical characteristics of fly ash were systematically analyzed to evaluate the influence of modification on its structural and surface properties. The morphological features and elemental composition were examined using SEM–EDX (Phenom ProX Desktop Scanning Electron Microscope). Surface morphology and elemental composition were characterized using a Phenom ProX Desktop SEM manufactured by Thermo Fisher Scientific, Netherlands. The crystalline phases were identified by X-ray Diffraction (SmartLab Rigaku, Cu-K α radiation source manufactured by Rigaku Corporation, Japan), while the specific surface area, pore volume, and pore

size distribution were determined using the BET method. BET instrument was NOVA 4200e BET Surface Area Analyzer, manufactured by Quantachrome Instruments, United States (USA). Nitrogen gas was used as the probe gas at liquid nitrogen temperature ($-196\text{ }^{\circ}\text{C}$).

All spectroscopic and imaging data were processed and interpreted using OriginPro software. The experimental datasets obtained from SEM–EDX, XRD, and BET analyses were imported into the software for further processing. OriginPro (OriginLab Corporation, USA) was utilized to analyse the surface morphology, construct 3D microstructural visualizations, and evaluate the porosity characteristics of the activated materials. For XRD data, the software facilitated the identification of diffraction peaks, determination of crystalline phases, and estimation of crystallographic parameters [21]. The integrated features such as Peak Analyzer and Image Analysis were employed to enhance data interpretation and visualization, providing a comprehensive understanding of the physicochemical transformation of fly ash after activation.

2.2.3 Activation of fly ash adsorbent

A total of 500 g of fly ash was accurately weighed using a digital balance and subsequently heated in an oven at $100\text{ }^{\circ}\text{C}$ for 60 min. The heating step aimed to remove residual moisture and promote pore expansion within the fly ash particles. This thermal treatment was expected to enhance the adsorptive capability of the material toward contaminants present in acid mine drainage. Upon completion of the heating process, the fly ash samples were removed from the oven and stored in a desiccator until they reached room temperature before being used in the subsequent experimental stages. The procedures were repeated by using temperature of $200\text{ }^{\circ}\text{C}$.

2.2.4 Alkali leaching method of fly ash

The process of producing synthetic zeolite from fly ash raw material using the alkali leaching method involves several steps. First, fly ash is ground to a size of 200 mesh (0.074 mm) to increase its reactivity. Next is the leaching stage, where fly ash is reacted with an alkali solution at a ratio of fly ash to NaOH solution of 1:10, with the aim of extracting silica and alumina from the fly ash. Then, this mixture is heated at $100\text{ }^{\circ}\text{C}$ for 2 h to help dissolve the silica and alumina. After that, the washing stage is carried out on the leaching mixture using demineralized water to remove residual alkali. The washed mixture is then reheated at a temperature between $70\text{ }^{\circ}\text{C}$ and $100\text{ }^{\circ}\text{C}$ using an oven or furnace to form zeolite. Finally, the resulting

zeolite is rinsed and dried to produce a synthetic zeolite that is ready for application [22].

2.2.5 Removal of metal ions from acid mine drainage using fly ash adsorbent

A series of fly ash samples with varying masses of 10, 20, 30, 40, and 50 g were prepared under four different conditions: untreated (without heating), thermally treated at $100\text{ }^{\circ}\text{C}$, thermally treated at $200\text{ }^{\circ}\text{C}$, and chemically modified through the alkali leaching process. Each prepared adsorbent was introduced into 250 mL of acid mine drainage contained in a 500 mL glass beaker. The mixtures were then agitated using a jar test apparatus at a constant stirring speed of 100 rpm for 30 min under ambient temperature to ensure homogeneous dispersion and effective interaction between the adsorbent and dissolved metal ions. After agitation, the suspensions were filtered using qualitative Whatman filter paper [23] separate the solid phase from the liquid. The adsorption process was conducted for a total contact time of 30 min under constant stirring. Following the agitation period, the mixture was allowed to settle for 15 min to facilitate initial phase separation before the filtration process using filter paper. To ensure the reliability and reproducibility of the data, all adsorption experiments, including pH measurements and metal ion concentration analyses, were performed in triplicate. The pH of the treated solution was measured using a calibrated pH meter, while the residual concentrations of Fe and Mn ions were analyzed through AAS to evaluate the adsorption efficiency of each treatment condition.

2.2.6 Measurement of adsorption efficiency

The adsorption efficiency was expressed as the percentage of adsorption, representing the proportion of solute transferred from the liquid phase onto the surface of the adsorbent during the experimental process. The calculation was performed by comparing the initial solute concentration (C_0) with the equilibrium concentration after treatment (C_e), as defined by Eq. (1) [24]:

$$\% \text{ Adsorption} = \frac{C_0 - C_e}{C_0} \times 100\% \quad (1)$$

The adsorption percentage represents the fraction of solute that transfers from the liquid phase to the surface of the adsorbent during the adsorption process. The concentrations are expressed in milligrams per liter (mg/L). The adsorption measurements were performed at room temperature ($30\text{ }^{\circ}\text{C}$).

3 Result and discussion

3.1 Characterization of acid mine drainage

Acid mine drainage from coal mining is not harmful to the environment if it complies with acid mine drainage quality standards in Indonesia, as listed in Table 1.

However, the results of the analysis of the characteristics of acid mine drainage show the values presented in Table 2.

Based on the characteristics of acid mine drainage above, the parameters such as Fe, TSS, COD, BOD, and TDS are below the quality standards, indicating conditions that are better and not harmful to the environment. Meanwhile, the main problems with acid mine drainage are the very low pH and the manganese content that exceeds quality standards.

The extreme acidity level of pH 3.45 is characteristic of acid mine drainage analysis results, as is the high manganese content of 5.13 mg/L. Therefore, we have conducted research on the treatment of acid mine drainage using fly ash adsorbents modified by the alkali leaching method to neutralize acidity and to remove manganese heavy metals. The results of the study show that the treated acid mine drainage meets the acid mine drainage quality standards and reaches the national water quality standard of Class I. Thus, this treated water is safe for the environment and can be used for sanitation purposes.

Table 1 Quality standard of acid mine drainage (Regulation of The Minister of Environment and Forestry of The Republic of Indonesia Number 5 of 2022) [25]

Parameter	Unit	Standards of acid mine drainage
pH	-	6-9
Mn	mg/L	4
Fe	mg/L	7
TSS	mg/L	400
COD	mg/L	-
BOD	mg/L	-
TDS	mg/L	-

Table 2 Analysis of acid mine drainage sample

Parameter	Unit	Analysis
pH	-	3.45
Mn	mg/L	5.13
Fe	mg/L	0.06
TSS	mg/L	26
COD	mg/L	8
BOD	mg/L	1.68
TDS	mg/L	364

3.2 Characterization of fly ash before and after activation and fly ash after modification (alkali leaching)

3.2.1 Scanning electron microscope analysis

The SEM technique is applied to examine the order and characteristics of the outer parts of materials. In addition, this technique is also used to identify the forming elements through X-ray spectroscopic studies carried out with the help of an electron microscope [25]. SEM was utilized to observe the external shape or surface arrangement of the cocrystals produced [26]. The characterization process with SEM produces micro-images that display the surface arrangement of the sample. This allows observation of the shape and dimensions of the particles [27].

Morphological analysis of the fly ash adsorbent using SEM revealed notable differences between samples before treatment, after heating at 100 °C, 200 °C, and following the alkali leaching modification. Fig. 1 presents SEM micrographs of the fly ash surface under a magnification of 2000 times, illustrating the morphological evolution of the material at each treatment stage-namely, before heating, after heating at 100 °C and 200 °C, as well as after the alkali leaching process.

Fig. 1(a) shows an SEM image of fly ash before heating; the surface of the fly ash particles appears relatively smooth with tightly packed agglomerates. The morphology is dominated by spherical granules and several large particles that remain attached to each other, indicating that the pores have not yet developed significantly in the untreated condition [16].

Fig. 1(b) shows an SEM image of fly ash after heating 100 °C, noticeable morphological changes occur. The particles begin to separate, and micro-pores start to emerge on the surface. This transformation is likely attributed to the evaporation of physically and chemically bound water, as well as the decomposition of volatile components, which promotes initial pore formation [28].

Fig. 1(c) shows an SEM image of fly ash after heating 200 °C, the surface morphology becomes rougher and more porous [29]. The number and distribution of pores increase, suggesting that higher heating temperatures enhance the specific surface area by promoting the opening of closed pores and reducing particle agglomeration [30].

Fig. 1(d) shows an SEM image of fly ash after modification (alkali leaching), exhibits a markedly different morphology. The surface shows numerous macro- and micro-pores with a more irregular and porous texture. This structural transformation indicates that the alkaline leaching treatment

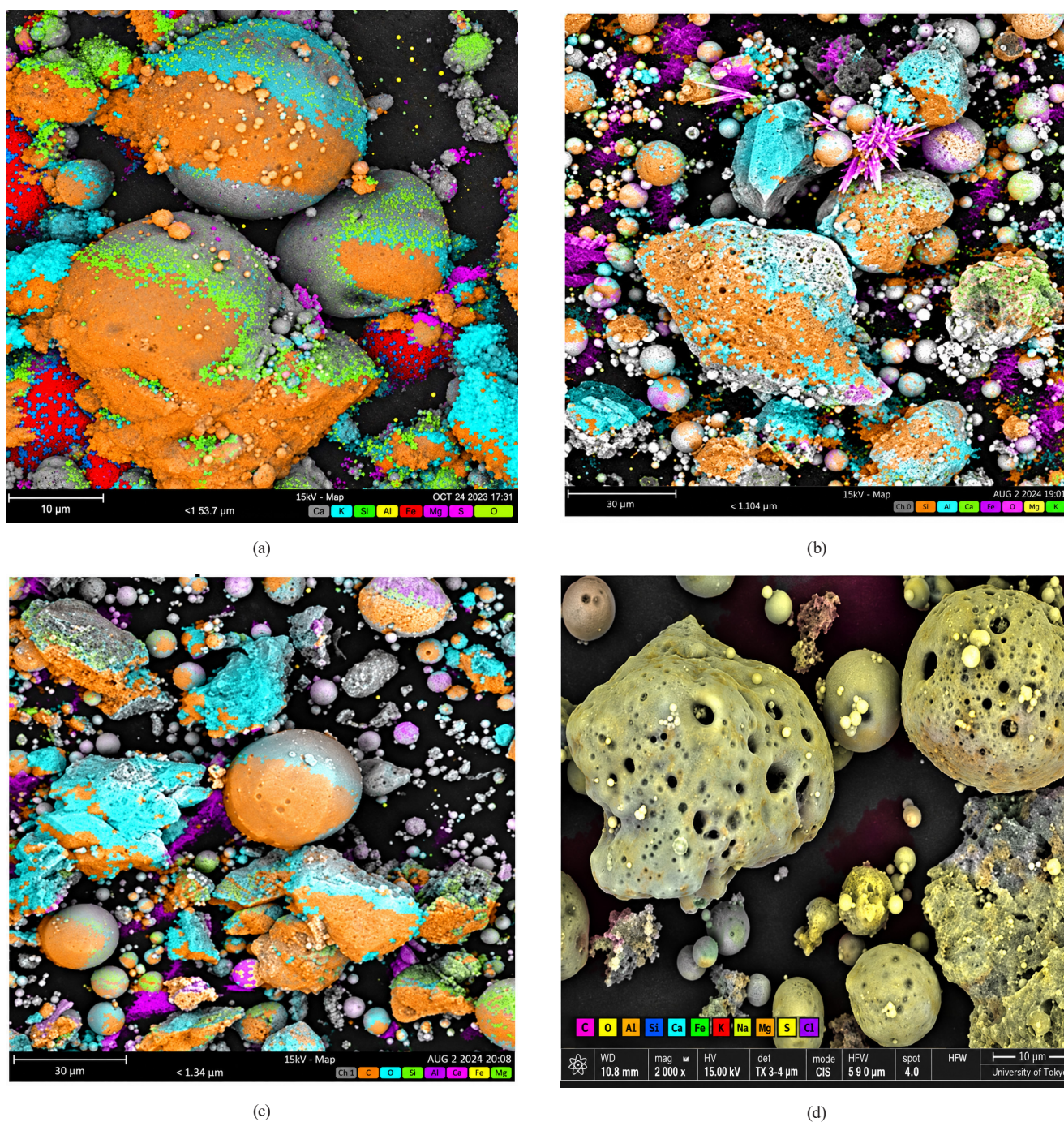


Fig. 1 SEM image of the fly ash: (a) before heating, (b) after heating 100 °C, (c) after heating 200 °C, (d) after modification (alkali leaching)

effectively dissolved portions of the amorphous aluminosilicate phases, particularly silica and alumina, resulting in a highly porous surface. Consequently, the modified fly ash is expected to possess a larger specific surface area and enhanced adsorption capacity for metal ions and contaminants present in acid mine drainage.

The SEM analysis reveals a significant morphological transformation of the raw fly ash, transitioning from smooth, spherical to a highly roughened and porous structure following the alkali leaching modification. This

structural evolution indicates the successful dissolution of silica and alumina phases, leading to the formation of an open aluminosilicate framework

3.2.2 3D image of fly ash before treatment, after heating at 100 °C, at 200 °C and after modification (alkali leaching)

The 3D visualization of fly ash at various treatment stages—including the untreated condition, thermal activation at 100 °C and 200 °C and subsequent chemical modification

through the alkali leaching process- reveals distinct variations in surface elevation and topographical morphology. In the 3D model, the X, Y, and Z axes represent the principal spatial dimensions describing the surface distribution and morphology of the material. The X and Y axes denote the lateral coordinates that define the observed surface area, while the Z axis reflects the surface elevation, corresponding to variations in surface roughness and pore depth.

The visualization distinguishes two major structural components: the solid volume, representing compact regions of the material, and the pore volume, corresponding to the void spaces within the fly ash structure. The contrast between these regions illustrates the enhanced porosity resulting from thermal and chemical activation processes. Such structural transformation contributes to

an increase in the specific surface area and adsorption capacity of the material, thereby improving its potential performance as an adsorbent for acid mine drainage treatment or other catalytic applications. The 3D visualization of fly ash morphology is presented in Fig. 2.

Fig. 2(a), which illustrates the morphology of fly ash prior to thermal treatment, reveals a heterogeneous surface characterized by noticeable variations in elevation. The measured maximum surface height ($H_{\max}$) reached 65.600 μm , while the minimum height ($H_{\min}$) was 2.200 μm . Such irregularities suggest the presence of particle agglomeration and indicate that amorphous phases remain predominant within the material structure. The porosity value of 0.54 (54.15%) implies that a significant portion of the material's volume consists of pores; however, some of these

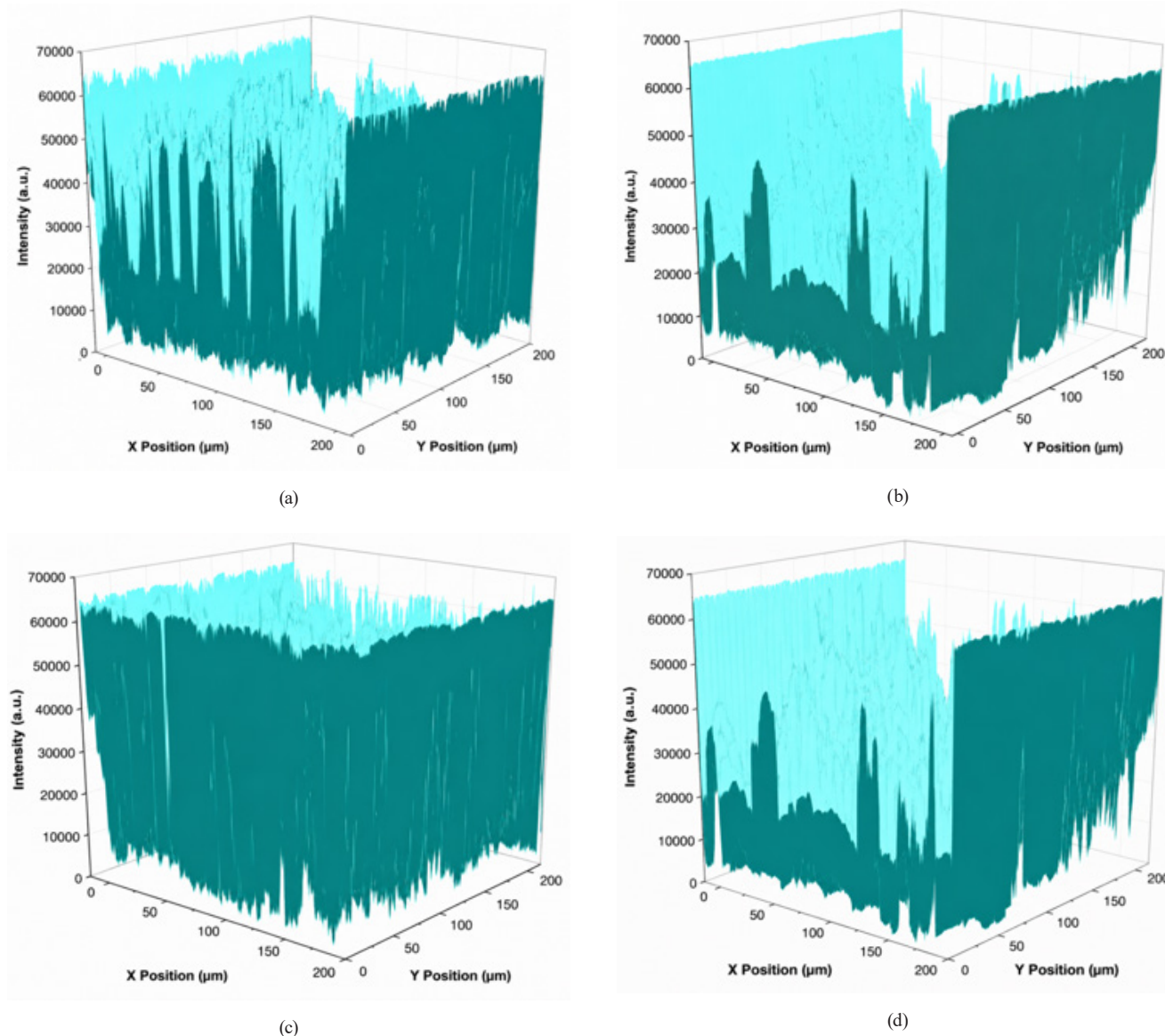


Fig. 2 3D image of fly ash: (a) before heating, (b) after heating 100 °C, (c) after heating 200 °C and modification (alkali leaching)

pores appear to be partially obstructed by amorphous silica and alumina layers, which may limit the effective surface accessibility for adsorption interactions [31].

Fig. 2(b), representing the surface topography of fly ash after heating at 100 °C, reveals partial surface leveling accompanied by a noticeable reduction in elevation irregularities. The thermal treatment at this temperature promotes the evaporation of free and weakly bound water within the fly ash matrix, leading to increased particle densification and a reduction in certain microporous structures. Nevertheless, minor pore openings begin to emerge due to the shrinkage of fine particles, resulting in a morphology that appears slightly more stabilized compared to the untreated sample [32].

Fig. 2(c), corresponding to fly ash after heating at 200 °C, displays a smoother surface morphology with a more pronounced development of open pores compared to the previous condition. Thermal treatment at this temperature leads to the removal of most crystallized water and promotes partial recrystallization of aluminosilicate phases, which strengthens interparticle bonding. A slight increase in porosity relative to the 100 °C treatment indicates the generation of new pores resulting from internal structural reorganization within the material [33].

Fig. 2(d), representing fly ash after the alkali leaching modification, exhibits the most pronounced transformation among all treatment stages. The surface morphology becomes considerably rougher and more porous, with a more uniform distribution of pore structures. The alkali leaching process effectively dissolves portions of the amorphous phase, particularly reactive silica and alumina, resulting in an expanded pore volume and increased specific surface area. Quantitatively, the porosity rises to 0.58 (58.22%), with a total volume of $12.35 \times 10^9 \mu\text{m}^3$, a solid volume of $5.16 \times 10^9 \mu\text{m}^3$ and a pore volume of $7.12 \times 10^9 \mu\text{m}^3$.

This enhancement in porosity and surface openness confirms that the combination of thermal activation and chemical modification successfully enhances the physico-chemical properties of fly ash, rendering it a highly porous and active adsorbent. Overall, the topographical evolution from Figs. 2(a) to 2(d) illustrates that progressive heating followed by alkali leaching effectively transforms fly ash from a moderately porous material into a high-porosity adsorbent with an enlarged active surface, directly contributing to improved metal ion uptake and acid mine drainage neutralization performance.

3.2.3 X-ray diffraction analysis

Analysis using XRD techniques was applied to qualitatively identify various crystalline phases present in fly ash [34]. This approach allows for in-depth characterization of the solid crystal structure, thereby providing a comprehensive understanding of the type, composition, and degree of regularity of the crystalline phases formed [35].

The main objective of XRD characterization is to determine the main diffraction peaks that reflect the crystalline components of fly ash adsorbents [18]. The analysis process was carried out by comparing the XRD diffraction patterns of fly ash samples under three different conditions, namely before treatment, after heating, and after modification through the alkali leaching process. The results of the diffraction pattern comparison are presented in Fig. 3, which shows changes in the structure and intensity of the diffraction peaks as an indication of crystalline transformation during the treatment stages.

Fig. 3 shows the X-ray diffraction results of fly ash at various treatment stages, namely before heating, after heating at 100 °C and 200 °C, and after modification through alkali leaching. The diffraction patterns show that the fly ash samples before and after thermal treatment up to 200 °C are still dominated by the crystalline phase of quartz (Quartz, JCPDS No. 96-901-0145) with main diffraction peaks appearing at positions $2\theta = 16.36^\circ$; 20.81° ; 36.52° ; 40.81° ; 50.10° ; 59.91° ; 68.12° ; and 81.19° . Heating to a temperature of 200 °C only caused a slight decrease in peak intensity without the formation of new phases, so that quartz remained the dominant phase in the material

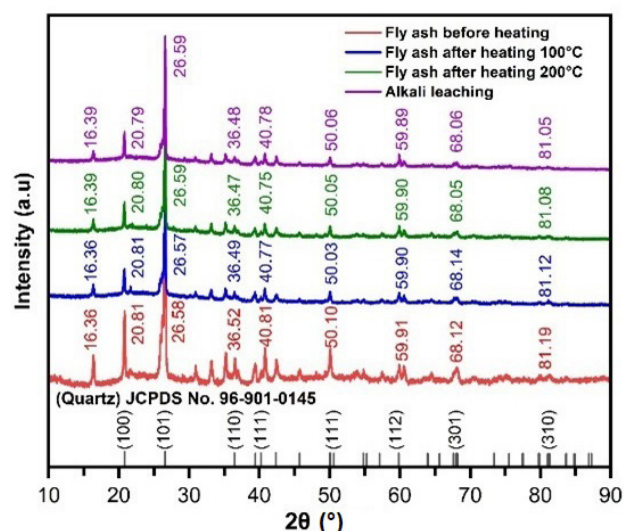


Fig. 3 Xray diffraction results of fly ash before heating, after heating at 100 °C, at 200 °C and after alkali leaching modification

structure. In contrast, the results of analysis on samples that had been modified through alkali leaching showed a significant decrease in quartz peak intensity, indicating a reduction in crystallinity and an increase in the amorphous fraction in the fly ash structure. These changes confirm that the alkali leaching process is effective in altering the mineral structure of fly ash, thereby increasing its reactivity potential and adsorption capacity towards metal ions and contaminants in acid mine drainage.

3.2.4 Brunauer-Emmett-Teller analysis

The BET method is an analytical technique applied to determine the specific surface area of solid materials such as fly ash [36]. The characterization results using the BET method showed significant changes in the specific surface area, pore size, and total pore volume parameters after fly ash underwent thermal treatment and chemical modification through alkali leaching (Table 3).

Fly ash before treatment had a surface area of 9 m²/g with a pore size of 1.69 nm and a pore volume of 0.017 cm³/g. After heating at 100 °C, the surface area increased to 10 m²/g with a pore size of 1.89 nm and a pore volume of 0.019 cm³/g, indicating the opening of some pores due to the release of volatile molecules and bound water. However, at 200 °C, the surface area decreased to 8 m²/g with a pore size of 1.91 nm and a pore volume of 0.016 cm³/g, possibly due to mild sintering or partial pore blockage. The most significant change was observed after the alkali leaching process, where the surface area increased dramatically to 14 m²/g with a pore size of 1.70 nm and a pore volume of 0.025 cm³/g. These results indicate that the alkali leaching process is capable of dissolving some amorphous components, such as silica and alumina, resulting in a more porous structure with a greater number of active sites. Overall, this modification increases the potential of fly ash as an adsorbent in acid mine drainage treatment.

3.2.5 Porosity analysis of the fly ash

Table 4 shows for the unheated fly ash, the total volume was recorded at $5.06 \times 10^9 \mu\text{m}^3$, with a solid volume of

Table 4 Porosity percentage and fly ash volume

Material	Parameter	Value	Unit
Fly ash before heating	Solid volume	2.32×10^9	μm^3
	Solid volume	2.74×10^9	μm^3
	Pore volume	5.06×10^9	μm^3
	Porosity	0.54	-
	Porosity Percentage	54.15	%
Fly ash after heating at 100 °C	Solid volume	3.76×10^9	μm^3
	Solid volume	3.22×10^9	μm^3
	Pore volume	6.98×10^9	μm^3
	Porosity	0.46	-
	Porosity Percentage	46.05	%
Fly ash after heating at 200 °C	Solid volume	4.15×10^9	μm^3
	Solid volume	4.86×10^9	μm^3
	Pore volume	9.01×10^9	μm^3
	Porosity	0.54	-
	Porosity Percentage	54.00	%
Fly ash after modification (alkali-leaching)	Solid volume	5.16×10^9	μm^3
	Solid volume	7.19×10^9	μm^3
	Pore volume	12.35×10^9	μm^3
	Porosity	0.58	-
	Porosity Percentage	58.22	%

$2.32 \times 10^9 \mu\text{m}^3$ and a pore volume of $2.74 \times 10^9 \mu\text{m}^3$, resulting in a porosity of 0.54 (54%). This relatively high initial porosity indicates the presence of irregular voids between agglomerated particles, typical of unprocessed fly ash derived from coal combustion. However, many of these pores are likely closed or partially obstructed by amorphous aluminosilicate phases, limiting the accessibility of the inner pore structure.

After heating at 100 °C, a noticeable reduction in porosity to 0.46 (46.05%) was observed. This decrease is primarily attributed to the evaporation of physically adsorbed water and the contraction of fine particles, which compact the structure and reduce available pore space. The solid volume increased slightly due to the densification effect, while the total volume expanded minimally, indicating a reduction in void fraction.

Upon heating at 200 °C, the porosity increased again to 0.54 (54%), accompanied by a higher pore volume

Table 3 BET characterization of fly ash adsorbents

Measurement subject	Fly ash before heating	Fly ash after heating at 100 °C	Fly ash after heating at 200 °C	Fly ash after modification (alkali leaching)
Specific surface area (m ² /g)	9	10	8	14
Pore size (nm)	1.69	1.89	1.91	1.70
Total pore volume (cm ³ /g)	0.017	0.019	0.016	0.025

($4.86 \times 10^9 \mu\text{m}^3$) and total volume ($9.01 \times 10^9 \mu\text{m}^3$). This behavior suggests the release of chemically bound water (crystalline water) and the initiation of microstructural rearrangement within the fly ash matrix. The resulting structural relaxation creates additional micro voids and enhances pore connectivity, indicating the onset of partial phase transformation within the amorphous matrix.

The most significant structural improvement was obtained after alkali leaching modification, where the total volume reached $12.35 \times 10^9 \mu\text{m}^3$ and the pore volume increased dramatically to $7.12 \times 10^9 \mu\text{m}^3$, yielding a porosity of 0.58 (58%). This result confirms that the alkaline treatment effectively dissolved portions of the amorphous aluminosilicate phases, particularly SiO_2 and Al_2O_3 , leading to the development of a more open and interconnected pore network. The increased porosity and expanded pore volume are critical improvements that directly enhance the specific surface area, thereby improving the adsorption capacity and reactivity of fly ash for environmental applications such as acid mine drainage treatment and heavy metal removal.

Overall, the progressive evolution of the porosity profile—from 54% (unheated) to 58% (alkali leached)—illustrates that the combination of thermal activation and alkaline modification plays a synergistic role in optimizing the surface and internal pore structure of fly ash. These morphological and textural changes confirm that controlled pre-treatment significantly improves its potential as a cost-effective and sustainable adsorbent material.

3.3 The effect of fly ash adsorbent mass on the pH of acid mine drainage

The pH of AMD before the addition of adsorbent (fly ash before heating, after heating at 100°C , and after heating at 200°C) was relatively low, at around 3.45. This indicates

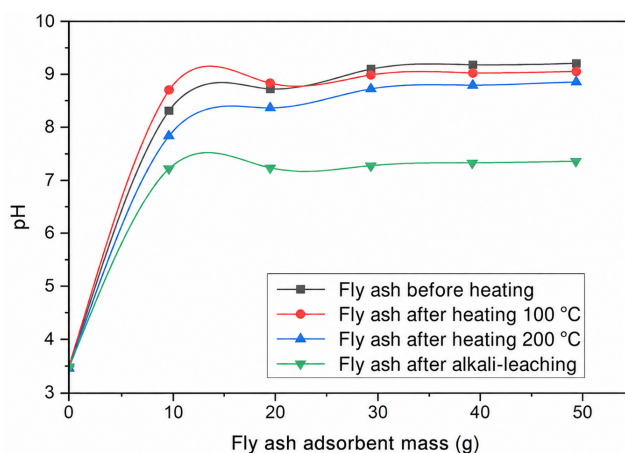


Fig. 4 Effect of fly ash adsorbent mass on pH

the initial pH of the acid mine drainage without the addition of fly ash adsorbent. Fig. 4 shows the effect of variations in fly ash adsorbent mass on the pH value of acid mine drainage under four treatment conditions, namely without heating, after heating at 100°C and after heating at 200°C , and after modification with alkali leaching. The results show that modified fly ash adsorbents (alkali leaching) are most effective in neutralizing acid mine drainage, with a stable pH in the range of 7.21 to 7.71.

An increase in adsorbent mass results in a significant increase in pH, especially in modified fly ash (alkali leaching), from an initial pH of around 3.45 to more than 7. This indicates that chemical modification of fly ash significantly increases its neutralization capacity against hydrogen ions in acid mine drainage, resulting in a final pH within the Class I of water quality standard range as stipulated in Government Regulation No. 22 of 2021, namely between pH 6 and 9. The modified fly ash showed a moderate increase in pH with stabilization in the neutral pH range (7.3), even at high adsorbent mass. This indicates that the modification process (alkali leaching) focuses on increasing heavy metal adsorption capacity and surface stability but does not predominantly increase free base characteristics as in natural fly ash. The modification (alkali leaching) generally increases the number of polar functional groups ($-\text{OH}$, $-\text{COO}^-$), but with limited H^+ ion neutralization capacity compared to strong base compounds [37].

Overall, this graph shows that thermal and chemical treatment conditions of fly ash affect pH neutralization capabilities differently. Untreated fly ash and lightly heated fly ash (100°C) have the highest pH due to their intact active base mineral content. Meanwhile, modified fly ash tends to be more stable and optimal for maintaining neutral pH, especially when the main focus is heavy metal adsorption rather than pH increase only.

3.4 Effect of fly ash adsorbent mass on metal degradation

The metal selected for analysis was manganese because the concentration of iron in the sample already met the applicable quality standards. Data on the percentage degradation of metal ions with differences in adsorbent mass from 10 g to 50 g with fly ash adsorbent treatment before heating, after heating at 100°C and after heating at 200°C can be seen in Fig. 5.

Fig. 5 shows the effect of fly ash adsorbent mass variation on the percentage of Mn degradation under three different treatment conditions: before heating, after heating

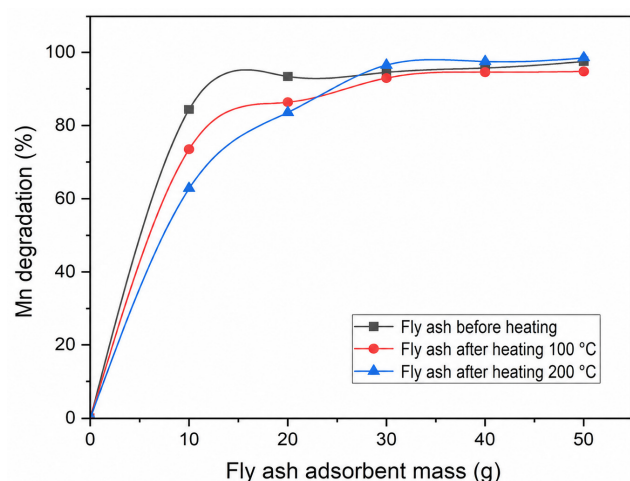


Fig. 5 Effect of fly ash adsorbent mass on Mn removal before heating, after heating at 100 °C and at 200 °C

at 100 °C and after at 200 °C. In general, this graph shows that an increase in fly ash adsorbent mass tends to increase the percentage of Mn removal for all treatment conditions until it reaches a saturation point.

In the initial adsorbent mass range (0–10 g), a sharp increase in the percentage of Mn removal is observed for all three curves. This increase can be attributed to an increase in the number of active sites available for adsorption as the adsorbent mass is added. As the adsorbent mass continues to increase, the rate of increase in the percentage of Mn removal begins to slow down, indicating that most of the adsorbate has been adsorbed and the system has been approached the equilibrium [3].

Fly ash adsorbents that have not undergone heat treatment (before heating, black curve) show good adsorption percentages. Heat treatment at 100 °C (red curve) and 200 °C (blue curve) shows an interesting pattern. At low adsorbent masses, fly ash heated at 100 °C shows slightly lower performance than unheated fly ash, while heating at 200 °C shows even lower performance. However, as the adsorbent mass increases, these three curves (pre-heating, 100 °C, and 200 °C) tend to converge and reach relatively similar maximum adsorption percentages at higher adsorbent masses (>30 g).

Heating can affect the structure and surface properties of fly ash, which in turn affects the adsorption capacity [33]. At higher temperature (200 °C), densification or structural changes may occur that reduce the availability of active sites in the lower adsorbent mass, although this effect can be overcome by adding a larger adsorbent mass. Overall, these findings underscore the potential of fly ash as an effective adsorbent and highlight the importance of optimizing the adsorbent mass to achieve maximum adsorption efficiency.

3.5 Effect of modified fly ash adsorbent mass on Mn removal

The relationship between the mass of fly ash adsorbent modified through an alkali leaching process and the efficiency of heavy metal adsorption from acidic mine drainage solutions. An increase in adsorbent mass from 0 to 100 g causes a drastic jump in the percentage of Mn removal, from close to 0% to around 99.03%. This reflects an increase in the number of active surface sites available to bind metal ions in solution, as shown in Fig. 6.

Fig. 6 clearly shows that fly ash modification is effective as an adsorbent for Mn removal. Mn removal is highly dependent on the mass of adsorbent used, with a significant increase at low to medium masses, and then reaching a saturation phase at higher masses. The optimal adsorbent mass to achieve a high Mn removal rate (around 90% and above) appears to be around 60–70 g, as further mass addition beyond this point provides minimal degradation improvement.

Adsorption efficiency continues to increase with increasing fly ash adsorbent mass, reaching 99.3% Mn degradation at 100 g, thus meet the water quality standards of Class I. This phenomenon is consistent with the principle of adsorption, where the number of active sites becomes limited after reaching maximum capacity, even though adsorbent mass continues to be added [20].

The removal of Mn and Fe in the present study is a combined (synergistic) mechanism involving several physical and chemical processes. This study clarifies that the mechanism of heavy metal removal from acid mine drainage via ZLM is a complex combined process. Precipitation occurs as a logical consequence of the increase in solution pH triggered by the basic nature of the adsorbent.

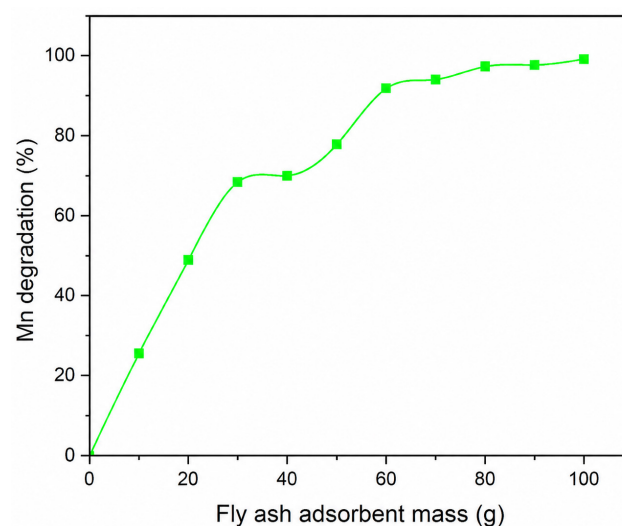


Fig. 6 The effect of modified fly ash adsorbent mass on Mn removal

The removal mechanism was further interpreted by considering the contribution of pH-induced metal precipitation alongside adsorption. The application of alkali-modified fly ash (zeolite like material) significantly increased the pH of AMD from 3.45 to 7.21, which plays a key role in reducing metal solubility. Under acidic conditions, metals such as Mn and Fe predominantly exist as dissolved ions; however, as the pH rises, these ions tend to form insoluble hydroxide precipitates (e.g., $\text{Mn}(\text{OH})_2$). Therefore, metal removal in this system is governed by a synergistic mechanism, where adsorption onto the enhanced surface area and pore structure of zeolite like material occurs simultaneously with chemical precipitation facilitated by the alkaline environment, leading to improved overall removal efficiency.

The efficient removal of Mn is attributed to two primary mechanisms. First, the ZLM facilitates an ion-exchange process where Mn^{2+} ions replace exchangeable cations in the aluminosilicate framework. Second, the inherent alkalinity of the modified fly ash raises the AMD pH to 7.21, promoting the precipitation of manganese as hydroxides. Concurrently, the zeolite-like structure of ZLM facilitates ion exchange and chemisorption at homogeneous surface-active sites. It is this synergy between precipitation and adsorption that enables the removal of Mn by up to 99% (reaching 0.05 mg/L), thereby meeting Class 1 water quality standards.

The ZLM demonstrated exceptional performance, achieving a Mn removal efficiency of over 99%. This indicates that the alkali leaching process successfully optimized the adsorbent's surface for metal ion entrapment

The modification process (alkali leaching) significantly alters the morphology and surface chemistry of fly ash, increasing the availability of ion exchange sites and chelation capacity against Mn metal removal. Thus, these

results confirm that modified fly ash is an efficient adsorbent for acid mine drainage treatment.

3.6 Adsorption capacity and adsorption isotherm

The presentation of adsorption capacity (q_e , mg/g) is essential for comparative analysis of the literature. Adsorption capacity (mg/g) for the four adsorbent conditions were calculated at a fixed adsorbent mass of 50 g using an initial concentration (C_0) of 5.13 mg/L and a solution volume (V) of 0.25 L.

Table 5 shows that the q_e value indicates how many mg of manganese is successfully adsorbed by every 1 g of fly ash adsorbent. The result also presents the advantages of ZLM, although the difference in mg/g values appears small, ZLM has the highest q_e value (0.0254 mg/g). On an industrial scale or in the treatment of large volumes of acid mine drainage, this small difference will have a significant impact on overall efficiency.

An increase in adsorption capacity is directly proportional to an increase in the percentage of efficiency. This proves that alkali leaching modification genuinely enhances adsorption performance per unit mass.

In addition to the percentage of removal, the adsorption capacity was also calculated to evaluate the performance of the adsorbent. Based on the table above, it can be seen that the ZLM modification provides the highest adsorption capacity of 0.02535 mg/g compared to the other conditions. This indicates that the active sites formed through the alkali leaching process are more effective at binding Mn ions from the solution than fly ash that has only undergone heat treatment (100 °C and 200 °C).

The following table (Table 6) presents $q_{\max}$ (theoretical maximum adsorption capacity). This value is derived from the constants of the Langmuir equation and serves as

Table 5 Adsorption capacity of Mn under various adsorbent conditions (C_0 : 5.13 mg/L, Adsorbent mass: 50 g, Solution volume: 250 mL)

Adsorbent conditions	Final concentration (mg/L)	Adsorption capacity (mg/g)	Efficiency (%)
Fly ash - before heating	0.13	0.025	97.46
Heated at 100 °C	0.27	0.024	94.73
Heated at 200 °C	0.08	0.025	98.44
Modified zeolite like material	0.06	0.025	98.83

Table 6 Comparison of maximum adsorption capacity for various adsorbent conditions (based on the Langmuir isotherm model)

Adsorbent conditions	Langmuir equation	$q_{\max}$ (mg/g)	KL constant (L/mg)	R^2
Fly ash before heating	$C_e/q_e = 0.0080 C_e + 0.11$	0.125	0.072	0.982
Heated 100 °C	$C_e/q_e = 0.0085 C_e + 0.13$	0.118	0.065	0.975
Heated 200 °C	$C_e/q_e = 0.0079 C_e + 0.10$	0.126	0.079	0.988
Modified ZLM	$C_e/q_e = 0.0078 C_e + 0.09$	0.128	0.086	0.996

a standard for comparing the performance of the four fly ash adsorbent conditions. The Langmuir equation has followed this formula:

$$\frac{C_e}{q_e} = \frac{1}{(q_{\max} \times KL)} + \frac{C_e}{q_{\max}} \quad (2)$$

C_e (mg/L): Equilibrium concentration of adsorbate remaining in solution after adsorption.

q_e (mg/g): Amount of adsorbate adsorbed per gram of adsorbent at equilibrium.

$q_{\max}$ (mg/g): Maximum adsorption capacity of the adsorbent.

KL (L/mg): Langmuir constant.

The table above shows that the highest $q_{\max}$ value (0.128 mg/g). This indicates that ZLM has the highest theoretical adsorption capacity. This demonstrates that the active sites produced by alkali leaching are more numerous and bind Mn more strongly than those produced by the conventional heating method. ZLM also has the highest KL value (0.086 L/mg). This means the bond between the ZLM surface and

Mn ions is very strong, so that even when the Mn concentration in the water is low, ZLM is still able to 'attract' the Mn until its concentration reaches 0.05 mg/L.

The result also shows the Model Fit ($R^2 = 0.996$). The R^2 value for ZLM is very high. This demonstrates that the adsorption mechanism on ZLM is highly regular and homogeneous based on the good fit of the experimental data to the isotherm model, which suggests uniform adsorption sites and consistent adsorption energy across the surface.

The comparison of adsorption capacities (mg/g) in the table above, which shows the maximum capacity values ($q_{\max}$) based on the Langmuir isotherm model. The results indicate that the ZLM modification exhibits the highest $q_{\max}$ value of 0.128 mg/g compared to the other treatments. The increase in the $q_{\max}$ value and the affinity constant (KL) for ZLM confirms that the formation of a zeolite-like structure on the fly ash surface significantly enhances the availability of functional active sites for the removal of manganese.

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4 Conclusions

The alkali leaching modification of fly ash has proven to be an effective approach to enhance its physicochemical and adsorption properties, as evidenced by the significant increase in specific surface area (14 m²/g), facilitating a removal efficiency exceeding 99% and total pore volume (0.025 cm³/g). Characterization results from SEM, XRD, and BET analyses confirmed substantial morphological, structural, and compositional transformations, where the dissolution of amorphous silica and alumina phases produced a more porous and reactive surface. When applied in the treatment of acid mine drainage, the modified fly ash effectively increased the pH from 3.45 to 7.21 and reduced Mn concentration from 5.13 mg/L to 0.05 mg/L, which meet the Class I of water quality standard in Indonesia. These findings demonstrate that the ZLM developed from industrial fly ash through alkali leaching successfully transforms this waste material into a high-value, eco-friendly, and sustainable adsorbent. These findings suggest that ZLM not only meets strict environmental discharge standards but also offers a sustainable pathway for industrial waste valorization in mine water remediation. The resulting ZLM exhibits strong potential for heavy-metal removal and broader environmental remediation applications, highlighting the effectiveness of this modification strategy in upgrading fly ash into a functional and environmentally beneficial material.

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