

## Research Article

## Fly ash adsorbent for pH improvement and manganese reduction in acid mine drainage

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**ABSTRACT:** Metal solid waste from coal combustion (fly ash) is abundant in Indonesia, as an effective and economical adsorbent in neutralizing acid mine drainage (AMD). Given that the continuous utilization of coal produces environmental challenges in the form of AMD containing acid residues and heavy metals such as manganese (Mn), an appropriate treatment solution is required. The adsorption method was chosen due to its simplicity, cost effectiveness, and ability to remove heavy metal pollutants. The purpose of this research is to characterize fly ash before and after heating by SEM and XRD analysis, and evaluate the effect of fly ash physical activation temperature by heating at 100°C and 200°C for an interval of 60 minutes on the characteristics and adsorption ability of fly ash. In addition, this study also evaluated the effectiveness of the adsorbent mass (fly ash before heating and after heating) in increasing pH and reducing Mn concentration in AMD so that it meets the quality standards of Class 1 river water. The results obtained from this study show a fundamental difference in the properties of fly ash before and after heating. Based on BET analysis, the physical activation process resulted in pore enlargement (0.196 nm) and increased surface area of the adsorbent (0.847 m<sup>2</sup>/g), which significantly affected its binding capacity to solutes (adsorption capacity). The application of fly ash as an adsorbent showed the ability to increase the pH value of acid mine drainage towards neutral conditions. The process of reducing heavy metal ions Mn by using 50 g of fly ash heating at 100°C and 200°C, resulted in a removal percentage of 94.74% and 98.44%. It is hoped that this research can provide innovative and sustainable AMD treatment and increase the use value of fly ash waste.

**Keywords:** Acid mine drainage, Adsorbent, Activation, Fly ash, Mn heavy metal ions

## 1. INTRODUCTION

The Republic of Indonesia, known as the country with the most islands in the world, holds enormous natural resource potential, including substantial coal content that ranks as the sixth largest producer internationally. Although the use of coal as a source of energy is considered cost-efficient, its combustion in the operation of Steam Power Plants (PLTU) leaves waste in the form of fine particles (fly ash) and causes ecological problems in the form of Acid mine drainage (AMD) in mining areas [1,2]. Formation of acid mine drainage (AMD) caused by oxidation of sulphide minerals such as pyrite [3,4], characterized by low pH and high concentrations of heavy metals such as iron (Fe) and manganese (Mn) [5]. This condition has the potential to cause contamination of aquatic ecosystems if not handled properly [6,7].

A number of techniques have been devised to deal with acid mine drainage (AMD) problems, by mitigating the AMD [8], including chemical neutralization processes using lime [9], coagulation-flocculation techniques [10], electro-Fenton [11], membrane techniques [12], and utilization of sulfate-reducing microorganisms [13,14]. In addition, adsorption is emerging as a promising option due to its simplicity, cost-efficiency, and ability to remove heavy metal contaminants from wastewater [15–17]. Exploration of various materials as adsorbents has been car-

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ried out, including plant-based natural materials [18,19], fly ash and Red Mud [20], active carbon [21,22]. However, activated carbon requires relatively expensive activation costs.

Activation of adsorbents can be done through three approaches: physical methods, chemical methods, or a combination of both. One physical activation method involving heating at 100°C has been applied in the production of activated carbon as reported in studies [23,24]. Other studies have also shown the utilization of physical activation method by heating at the same temperature (100°C) on the use of bottom ash as adsorbent [25]. Meanwhile, this research investigates the use of physical activation methods with a heating temperature range of 100°C and 200°C in the context of fly ash production as an adsorbent. Thus, the novelty aspect of this research lies in optimizing the physical activation conditions and evaluating its effectiveness compared to the direct use of fly ash as an adsorbent. While previous studies tend to use chemical activation, this study specifically focuses on a simpler and more environmentally sound physical activation method, namely heating at 100°C and 200°C for 1 hour (60 minutes). This approach is considered more energy-efficient and has the potential for scalability for industrial applications.

In the context of coal waste valorization, fly ash has shown potential as a versatile adsorbent for various types of pollutants [26]. Several studies have applied fly ash for adsorption of phosphorus [26] and heavy metal ions [27,28]. To improve the adsorption effectiveness, modification of fly ash into zeolite-like material (ZLM) through various synthesis methods such as sol-gel, hydrothermal, alkali-fusion, and alkali-leaching has been investigated [29–32].

Although the application of fly ash as an adsorbent in AMD treatment has been investigated, its specific utilization to produce water quality that meets sanitary standards (Class 1 River Water) is still limited and requires further study. Furthermore, the development and characterization of fly ash activated by 100°C and 200°C heating for specific application in neutralizing AMD and reducing Mn levels to meet water quality standards has not been reported. Therefore, this research aims to investigate the potential of fly ash thermally activated at 100°C and 200°C as an adsorbent for AMD treatment. In more detail, the objectives of this research include analysis of AMD characteristics before and after treatment, characterization of fly ash at various stages (before and after activation evaluation of the effect of activation temperature on fly ash adsorption performance, analysis of the impact of fly ash modification on adsorbent properties, and determination of the effectiveness and effect of adsorbent dosage (fly ash) before treatment as well as after heating in improving the quality of coal AMD.

## 2. EXPERIMENTAL AND METHOD

**2.1. Materials.** This research uses Fly ash waste from coal combustion residue at PLTU Tanjung Enim, acid mine drainage from PT Bukit Asam (Persero) Tbk, sodium hydroxide (NaOH, CAS 1310-73-2) and distilled water (H<sub>2</sub>O, CAS 7732-18-5).

**2.2. Method and procedure.** **2.2.1. Activation of the fly ash adsorbent.** A total of 500 g of fly ash was weighed accurately using a digital balance. Next, the fly ash was heated in an oven at 100°C and 200°C for 60 minutes. This heating process was carried out with the aim of removing the water content contained in fly ash

as well as widening its pores. Thus, it is expected that the ability of fly ash to absorb pollutants from acid mine drainage will increase. After heating for one hour, the fly ash was removed from the oven and placed in a desiccator until it reached room temperature before being used in the study. The fly ash before and after activation sent to Scanning Electron Microscopy-Energy Dispersive X-ray (SEM-EDX) characterization to observe the surface morphology and elemental composition. X-ray Diffraction (XRD) technique is also used to identify crystalline phases, determine lattice parameters, and assess the physical properties of materials (fly ash).

**2.2.2. Adsorption of Mn and Fe metal ions in acid mine drainage using fly ash adsorbent.** 250 ml of acid mine drainage sample was put into a 500 ml beaker. The acid mine drainage has Mn concentration of 5.13 mg/L, Fe concentration of 0.06 mg/L, TSS of 26 mg/L, COD of 8 mg/L, BOD of 1.68 mg/L, and TDS of 364 mg/L. Then, fly ash with a mass variation of 10, 20, 30, 40, and 50 g was added to each beaker. This mixture was stirred using a jar test tool at 100 rpm for 30 minutes. After the stirring process was complete, the solution was filtered using Whatman 41 filter paper to separate the solids. Subsequently, pH measurement and concentration analysis of Manganese (Mn) ions in the obtained filtrate were carried out. The same procedure was repeated using fly ash that had been heated at 100°C and 200°C.

**2.2.3. Adsorption percentage.** The amount of solute adsorbed by the adsorbent during the adsorption process is measured through the percentage of adsorption. This calculation is based on the ratio between the initial concentration (C<sub>0</sub>) and the final concentration (C<sub>e</sub>) of the solute in solution. The percentage of adsorption can be determined using the following formula [33]:

$$\% \text{ Adsorption} = \frac{C_0 - C_e}{C_0} \times 100\%$$

Where: % Adsorption indicates the percentage of solute bound by the adsorbent. C<sub>0</sub> represents the initial level of solute before the adsorption process occurs, with units of milligrams per liter (mg/L) or parts per million (ppm). C<sub>e</sub> represents the final level of solute after the adsorption process takes place, with the same units, namely milligrams per liter (mg/L) or parts per million (ppm).

**2.2.4. Fly ash adsorbent analysis by jar test.** A number of fly ash with different masses, namely 10, 20, 30, 40, and 50 g, were weighed for each fly ash condition: without heating, heated at 100°C, and heated at 200°C. Each sample was then mixed into 250 ml of acid mine drainage in a 500 ml beaker container. The beaker containers were then placed in a jar test apparatus with the stirring speed set at 100 revolutions per minute at room temperature for 30 minutes. This step aims to ensure a homogeneous distribution of fly ash and maximize the effectiveness of the adsorption process. After the specified time expired, separation of the mixture was carried out through filtration using filter paper [25]. The acidity (pH) of the acid mine drainage after the treatment process using fly ash adsorbent was measured using a pH meter, while the levels of iron (Fe) and manganese (Mn) metals were analyzed using Atomic Absorption Spectroscopy (AAS) (GBC Scientific Equipment).

**2.2.5. pH measurement.** Before use, the pH measuring device needs to be calibrated first with a pH 4 buffer solution. During this calibration process, set the device to display the number 4.

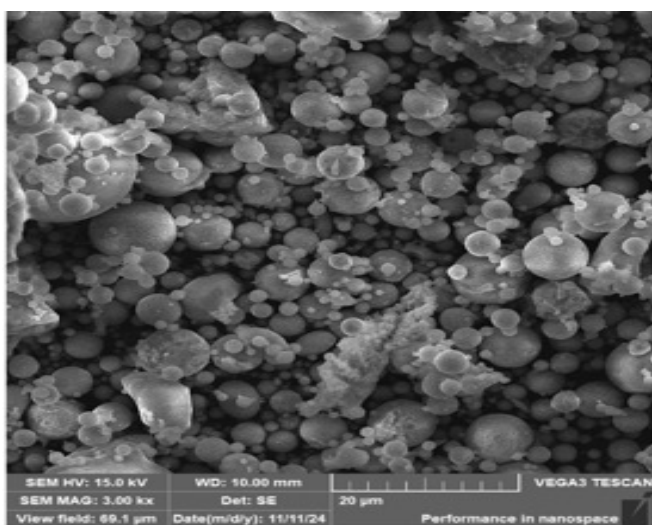
Next, clean the electrodes by rinsing them and then drying them. After that, take measurements using pH 10 buffer solution and adjust the reading of the device until it shows 10 during calibration [34]. After the calibration process was complete, the calibrated electrode was dipped into a vessel containing a sample of acid mine drainage to determine its pH value. Then, pH measurement was carried out on acid mine drainage that had previously been reacted with fly ash adsorbent after going through the jar test stage.

### 3. RESULT AND DISCUSSION

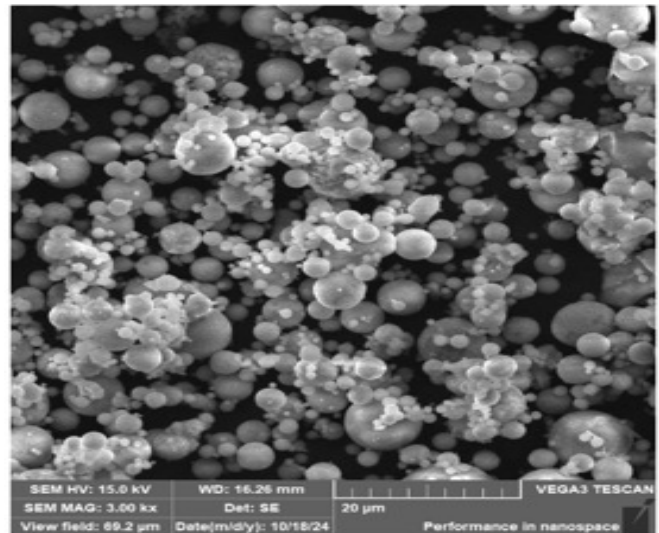
**3.1. SEM analysis of fly ash adsorbent.** The Scanning Electron Microscope with X-ray Energy Dispersion (SEM-EDX) technique is applied to examine the order and characteristics of the outer parts of materials. In addition, this technique is also used to identify the forming elements through X-ray spectroscopic studies carried out with the help of an electron microscope [35]. Scanning Electron Microscopy (SEM) was utilised to observe the external shape or surface arrangement of the cocrystals produced [36]. The characterisation process with Scanning Electron Microscopy (SEM) produces micro-images that display the surface arrangement of the sample. This allows observation of the shape and dimensions of the particles [37].

Observation of fly ash adsorbent morphology using Scanning Electron Microscope (SEM) showed significant changes between conditions before and after heating treatment. Figure 1 (before heating) shows that the pores on the particle surface are not fully open. A variety of particle shapes were observed, including spherical, irregular, and some possibly resembling solid spheres. Overall, the particle surface appears relatively smooth, although there are some particles with uneven surfaces.

The 100°C heating process in Figure 2 shows an increase in the porosity of the particles, characterized by an increase in the number of open pores or empty spaces. Some particles show a hollow structure or have a rougher surface. Figure 3 still demonstrates



**Figure 1.** Surface morphology of fly ash before heating (SEM magnification 3000 times).



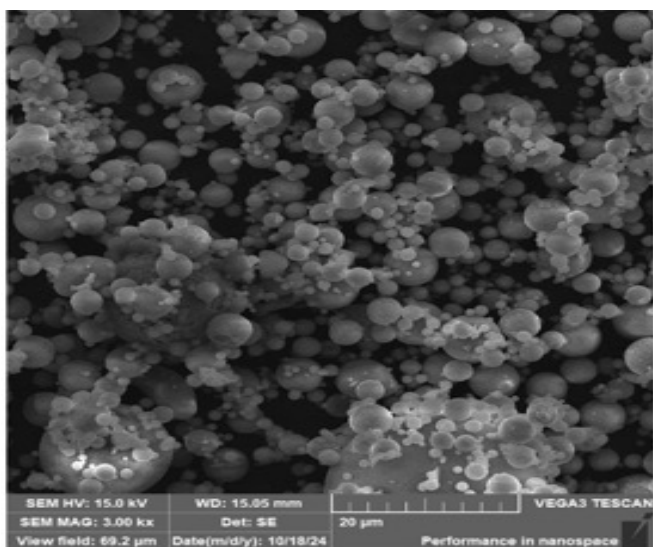
**Figure 2.** Surface morphology of fly ash after heating 100°C (SEM magnification of 3000 times).

most of the spherical shape of the particles as well as Figure 2, after heating the fly ash to 200°C. However, the particles look less bright than depicted in Figure 2, indicate the change in microstructural properties of the fly ash particles because of the higher temperature heating.

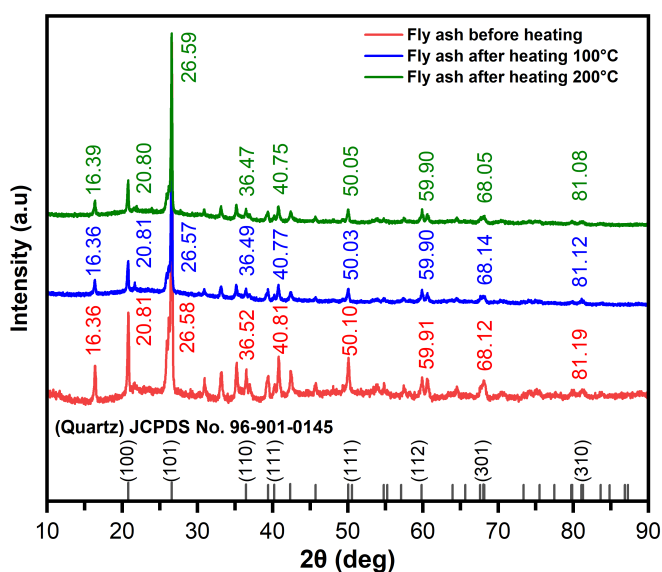
Other researchers described the effect of heating at high temperatures on microstructural properties of fly ash, which is after heating at 200°C, the fly ash still appears to be smooth spheres with soft pores or small cracks beginning to appear, especially on the surface of particles that are not completely covered by gel, causing the fly ash pores to become more open [38]. Under these conditions, the microstructure still retains most of the spherical shape of the particles, but the geopolymer matrix or gel shrinks and small cracks become more visible, making the pores more apparent and weakening the interparticle bonds slightly, as well as reported by Razaq et al in 2022 [39]. Based on the morphological analysis conducted, it can be concluded that the heating process has effectively opened the pores of the fly ash structure and surface. This transformation is expected to have an impact on the characteristics of fly ash, including its reactivity and adsorption capacity. The observed morphological changes indicate the changes that occur because of the heating process.

**3.2. analysis fly ash adsorbent characterization by XRD.** X-ray Diffraction (XRD) analysis technique is applied to qualitatively recognize the presence of crystalline phases in fly ash materials [40]. The identification and characterization of crystal structures in solid materials can be done through an analytical technique called X-ray Diffraction (XRD) [41].

The purpose of characterization using the X-ray Diffraction (XRD) method is to identify the main peaks in the fly ash adsorbent material [28]. A comparison of the peaks appearing in the X-ray Diffraction (XRD) pattern of the fly ash adsorbent before and after the heating process was carried out as part of the analysis. The results of this comparison are visualized in Figure 4.



**Figure 3.** Surface morphology of fly ash after heating 200°C (SEM magnification of 3000 times).



**Figure 4.** Comparison of fly ash diffraction before heating and after heating 100°C and 200°C.

The identification of the phases formed at each peak in the XRD graph of fly ash based on Figure 4 above is by comparing the X-ray diffraction patterns of fly ash before heating, after heating at 100°C, and after heating at 200°C. Phase identification is formed at some significant peaks in the graph: The peak around  $2\theta = 20.8^\circ$ : Before heating, a significant peak is visible. After 100°C heating this peak is still visible. Furthermore, after 200°C heating (green) this peak is also still visible and appears more intense. The identification

results carried out based on the quartz standard pattern at the bottom, this peak most likely corresponds to the crystal plane (100) of Quartz (SiO<sub>2</sub>).

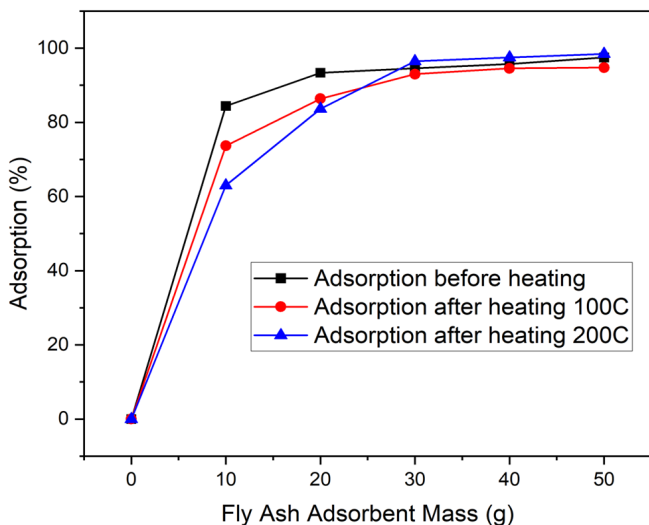
Peak around  $2\theta = 26.6^\circ$ : Before heating a very intense peak is visible. After 100°C heating this peak remains the main peak. Furthermore, after 200°C heating this peak is also very intense. Identification states that this peak closely matches the crystal plane (101) of Quartz (SiO<sub>2</sub>), which is the main characteristic peak of quartz. Peak around  $2\theta = 36.5^\circ$ : Before heating a peak of medium intensity is visible. After 100°C heating this peak is still present. After 200°C heating this peak is also visible. Identification results suggest this peak may correspond to the crystal plane (110) of Quartz (SiO<sub>2</sub>).

Peak around  $2\theta = 40.8^\circ$ : Before heating the peak is of low intensity. After 100°C heating this peak is still visible. After 200°C heating this peak is also present. The identification carried out states that it probably corresponds to the crystal plane (111) of Quartz (SiO<sub>2</sub>). Peak around  $2\theta = 50.1^\circ$ : Before heating the peak is of low intensity. After 100°C heating this peak is still visible. After 200°C heating this peak is also present. The identification results suggest it may correspond to the (111) crystal plane (there are two planes with the same index in the standard pattern) of Quartz (SiO<sub>2</sub>). Other peaks: Other peaks at different  $2\theta$  angles (e.g. around 59.9°, 68.1°, and 81.1°) are also possible crystalline phases present in fly ash. These peaks represent other mineral phases commonly found in fly ash, such as Mullite (Al<sub>6</sub>Si<sub>2</sub>O<sub>13</sub>), Hematite (Fe<sub>2</sub>O<sub>3</sub>), or Calcite (CaCO<sub>3</sub>).

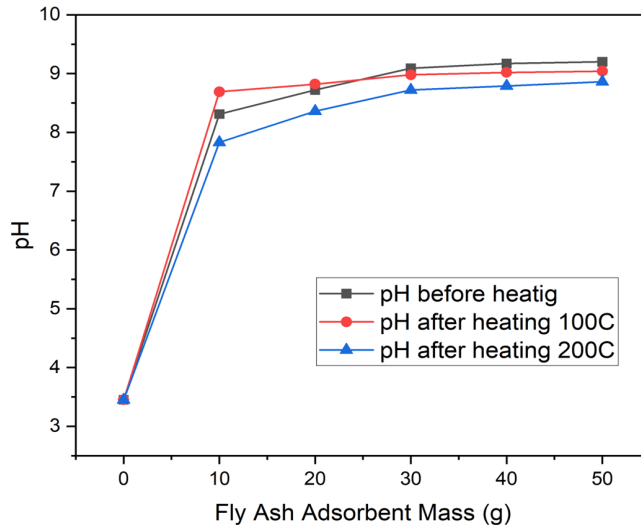
Based on the comparison with quartz standard patterns, it can be concluded that Quartz (SiO<sub>2</sub>) is one of the main crystalline phases present in the fly ash samples, both before and after heating at 100°C and 200°C. Heating to 200°C seems to increase the intensity of the quartz peaks, which could indicate an increase in quartz crystallinity or a change in crystal preference orientation.

**3.3. Percentage adsorption of Mn metal ions with fly ash adsorbent before heating, after 100°C of Heating and 200°C of heating on the sorption of Mn metal ions in acid mine water.** Data on the percentage of Mn metal adsorption with different adsorbent masses from 10 g to 50 g with fly ash adsorbent treatment before heating, after 100°C heating and after 200°C heating can be seen in Figure 5.

At low fly ash adsorbent masses (below 10 g), there were significant differences between the adsorption percentages before heating and after heating. At 10 g adsorbent, the observed adsorption rates of Mn metal before heating, after 100°C heating and after 200°C heating were 84.41%, 773.68% and 62.96%, respectively. When the adsorbent mass was increased to 30 g, the percentage of Mn metal absorption increased to 93.54% before heating, 92.98% after 100°C heating and 96.49% after 200°C heating. Heating tends to increase adsorption. At higher masses (above 30 g), the difference between before heating and after 100°C and 200°C heating became smaller. This suggests that at sufficiently high adsorbent masses, the heating effect becomes less significant. Furthermore, with the increase of adsorbent amount up to 50 g, the removal efficiency of heavy metal ion Mn was recorded as 97.47% before heating, 94.74% after 100°C heating and 98.44% after 200°C heating. The adsorption percentage increased with the increase of fly ash mass. The variation of fly ash adsorbent mass has a significant



**Figure 5.** Effect of fly ash adsorbent mass on adsorption percentage.



**Figure 6.** The effect of fly ash adsorbent mass on pH.

effect on the adsorption of Mn metal in acid mine drainage. The greater the amount of adsorbent used, the higher the adsorption percentage obtained. This shows that the more adsorbent used, the more substances can be adsorbed. Heating fly ash, especially at high masses, can increase the adsorption effectiveness. This may be due to changes in the structure or surface properties of fly ash as a result of heating. These results are relevant in water treatment or environmental applications, where fly ash can be used as an adsorbent to remove pollutants. By understanding the relationship between adsorbent mass and adsorption efficiency, as well as the effect of heating, we can optimise the adsorption process.

**3.4. Effect of fly ash adsorbent mass on pH in acid mine drainage.** Research conducted by Zhou showed that fly ash has the potential to be utilized as an adsorbent [26], Fly ash pellets were applied as an adsorbent in an effort to absorb phosphorus and heavy metals [42], coal fly ash was used as one of the methods in dealing with wastewater. The ability of fly ash to act as an adsorbent can be utilized in the treatment of wastewater generated by the pulp and paper industry [43]. Wastewater treatment applications are the focus in the utilization and management of coal fly ash derived from industrial waste. This action is a form of waste recycling to respond to the challenge of water scarcity [16]. The characteristics of fly ash make it an appropriate adsorption material for a variety of uses [44]. An effective and cost-effective alternative to remove pollutants from water and gas is an adsorbent based on coal fly ash (CFA) [37].

Fly ash has the ability to raise the pH of acid mine drainage, as shown in a study by Keller and colleagues. In that study, the initial pH of 1.9 was successfully increased to 6 after an adsorption process using fly ash [20]. The initial pH level of the acid mine drainage in this study was identified as less than 5, which was around 3.5. Then, the application of fly ash as an adsorbent resulted in a striking increase in pH, as visualized in Figure 6.

From the picture above, the mass of fly ash adsorbent is 0 g, before heating, after 100°C heating and 200°C heating has a relatively low pH value, which is around 3.45. This shows the pH of

the initial acid mine drainage without the addition of fly ash adsorbent. Increase in fly ash adsorbent mass up to 10 g. There was a significant increase in pH in all three conditions. The pH increased sharply from about 3.45 to about 7.8 - 8.7. This shows that the addition of only a small amount of fly ash can drastically increase the pH of the solution.

The mass of fly ash adsorbent has a significant effect on the pH of the solution. An increase in adsorbent mass, especially in the initial range (0-10 g), led to a substantial increase in pH. After reaching a certain mass (about 10 g), further addition of adsorbent did not give a significant increase in pH. This suggests a saturation point or limitation in the adsorbent's ability to affect pH. This is as expected for the pH of coal wastewater Quality Standard of 6-9.

Heating the fly ash adsorbent also affected the pH. Heating at 100°C appeared to slightly increase the pH, while heating at 200°C actually decreased the pH compared to no heating. Within the expected pH of 6-9.

## 4. CONCLUSION

A solid by-product of the coal combustion process, fly ash, is available in large quantities in Indonesia. It has the potential to be used as an efficient and affordable adsorbent to neutralise mine water acidity (AAT). The heating process of fly ash can result in changes in its physical characteristics, such as an increase in pore size and expansion of surface area. These modifications have a positive impact on its adsorption capacity.

Based on the results of the study, fly ash showed its ability to raise the acidity level (pH) of acid mine drainage to near neutral value. The application of 50gram of activated fly ash through heating at 100°C and 200°C proved to be effective in reducing the concentration of heavy metal ions Mn in AAT. The removal rate reached 94.74% and 98.44%, respectively. This study is expected to present an innovative and sustainable alternative to AAT treatment, while increasing the useful value of fly ash waste.

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## CREDIT AUTHOR STATEMENT

**Nurlela:** Conceptualization, Methodology, Writing-Original draft preparation, Formal analysis. **Tuty Emilia Agustina:** Supervision, Methodology, Writing-Reviewing and Editing, Formal analysis. **Susila Arita:** Supervision and Methodology. **David Bahrin:** Supervision and Methodology. **Rianya Gayatri:** Writing-Reviewing and Editing.

## DECLARATIONS

**Conflict of interest** The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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