

Optimization of Coal Fly Ash Heating Temperature as an Adsorbent to Improve Acid Mine Water Quality

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Abstract

Fly ash, a fine dust from coal combustion in steam power plants, is an unused waste material that can be repurposed as a low-cost adsorbent. One type of adsorbent that can be synthesized from fly ash is zeolite-like material (ZLM), which has a porous structure capable of absorbing molecules smaller than or equal to its cavity size. In this study, untreated and heat-activated fly ash at 100, 200, and 300°C was used as an adsorbent for acid mine water. Adsorbent mass varied between 6, 8, 10, 12, and 14 g, mixed with 130 mL of mine water and stirred at 150 rpm for 60 minutes. The results showed that heat activation increased the surface area of the fly ash adsorbent, reaching 22.658 m²/g at 300°C. Heat-treated fly ash at 200 and 300°C showed a more significant reduction in Fe and Mn levels, especially with 8 g of adsorbent. Additionally, all adsorbents, whether heated or not, raised the pH of the acid mine water. The total suspended solids (TSS) were reduced to meet quality standards using 14 g of adsorbent heated to 300°C. These results indicate that coal fly ash adsorbent can be effectively applied for treating acid mine water.

Keywords: Fly ash, Adsorbent, Mine acid water pH, TSS, Fe, Mn

Article Info

Received 20 August 2024
Received in revised 27 September 2024
Accepted 29 September 2024
Available online 25 October 2024

Abstrak (Indonesian)

Fly ash, debu halus dari pembakaran batu bara di pembangkit listrik tenaga uap, merupakan material limbah yang tidak terpakai tetapi dapat dimanfaatkan sebagai adsorben berbiaya rendah. Salah satu jenis adsorben yang dapat disintesis dari abu terbang adalah material mirip zeolit (ZLM), yang memiliki struktur berpori sehingga mampu menyerap molekul yang ukurannya lebih kecil atau sesuai dengan ukuran porinya. Dalam penelitian ini, abu terbang yang tidak diolah dan yang diaktivasi melalui pemanasan pada suhu 100, 200, dan 300°C digunakan sebagai adsorben untuk air asam tambang. Massa adsorben divariasikan antara 6, 8, 10, 12, dan 14 g, dicampur dengan 130 mL air tambang dan diaduk pada kecepatan 150 rpm selama 60 menit. Hasil penelitian menunjukkan bahwa aktivasi panas meningkatkan luas permukaan adsorben abu terbang, mencapai 22,658 m²/g pada suhu 300°C. Abu terbang yang diaktivasi panas pada 200 dan 300°C menunjukkan penurunan kadar Fe dan Mn yang lebih signifikan, terutama dengan penggunaan adsorben 8 g. Selain itu, semua adsorben, baik yang dipanaskan maupun tidak, meningkatkan pH air asam tambang. Padatan tersuspensi total (TSS) berkurang hingga memenuhi standar kualitas dengan menggunakan 14 g adsorben yang dipanaskan pada suhu 300°C. Hasil ini menunjukkan bahwa adsorben abu terbang batu bara dapat diterapkan secara efektif untuk pengolahan air asam tambang.

Kata Kunci: fly ash, adsorben, air asam tambang, pH, TSS, Fe, Mn

INTRODUCTION

Coal, which is currently increasingly used as a powerplant fuel, has led to a rise in the amount of waste produced. Indonesia's coal production in 2019 reached 616 million tons, with 74% of this production being

exported, and the remainder used domestically. In 2021, approximately 113 million tons were used for electricity generation, with 63.8 million tons consumed by PLN (State Electricity Company) and 49.2 million tons by Independent Power Producers (IPPs). If the

amount of fly ash and bottom ash (FABA) is assumed to be 10% of the coal content, it is estimated that the total waste produced will be around 11.3 million tons [1].

The main issue with coal mining waste is its low pH (acidity level) and the high content of heavy metals, particularly iron (Fe) and manganese (Mn). To mitigate the environmental impact, an appropriate acid mine drainage management system is essential for coal mining operations.

In recent decades, water pollution resulting from hazardous contaminants, pharmaceutical molecules, and industrial organic compounds has become an important issue because of its direct effects on aquatic and human ecosystems, requiring the development of effective wastewater treatment techniques [2].

Several studies aimed at reducing the impact of acid mine drainage have utilized coal fly ash waste as a coagulant using the concept of reverse logistics, where fly ash is used as a coagulant combined with sulfuric acid (H_2SO_4). The optimal mixture was obtained with fly ash containing 20.56% of Al_2O_3 and H_2SO_4 with ratio of 1:1, while the coagulant was produced with a 1:2 ratio. In research using this coagulant on wastewater at TK 6616 (neutralized water), the optimal dose was found to be 0.5 g/L (500 ppm), resulting in a turbidity reduction of 98.68% and a TSS reduction of 97.41% [3].

Coal contains many rare elements that are retained in coal fly ash [4]. Coal fly ash (CFA) management has become a global environmental concern due to their impact on the environment and the quantities of the waste generated [5]. Coal fly ash (CFA) is the main solid waste from coal-fired power plants. The world's annual growth of this type of waste is about 700–800 million tons [6]. Coal fly ash, a residual by-product of the coal combustion process, promises to be an important source of different rare earth elements associated with minerals in the parent coal [7].

Reducing heavy metals in acid mine drainage through coagulation methods activated by hydrochloric acid using fly ash has also been reported by another research [8]. In their study, the optimal reduction of turbidity and copper (Cu) in artificial acid mine drainage was achieved at an initial pH of 8, with a settling time of 15 minutes, and a coagulant concentration of 100 mg/L, resulting in a 99% reduction in turbidity, with a final turbidity of 1.19 NTU. Additionally, a coagulant concentration of 50 mg/L was used to reduce Cu, achieving a 58% reduction and a final Cu concentration of 1.98 mg/L. After coagulation, the final pH of the sample decreased to 7.25. Coal fly ash, a byproduct of coal-fired industry,

is a promising skeleton material for the fabrication of high-temperature composite [9].

Addressing the significant emissions and severe pollution hazards posed by coal fly ash waste in the coal chemical industry, as well as the challenges in recovering phenolic substances from coal chemical wastewater [10]. An effective strategy for addressing the accumulation of waste from coal combustion in Indonesia is to harness fly ash (FA) as an adsorbent material. The advantages of coal fly ash as an adsorbent stem from its chemical composition, surface area, porosity, particle size, and adsorption capacity [11].

Coal fly ash (CFA) is a kind of byproduct produced by thermal power plants and has been demonstrated to be an environment friendly and low-cost adsorbent for the immobilization of heavy metals and organic dyestuff in water or wastewater due to its characteristics of large surface area and small particle size [12].

In this study, fly ash obtained from the coal combustion residue of PT Bukit Pembangkit Innovative's, a power plant in Lahat, will be used. Its abundance of waste can pose environmental problems, so utilizing it as an adsorbent can help address the environmental issues caused by acid mine drainage.

MATERIALS AND METHODS

Materials

The fly ash to be used as an adsorbent will be obtained from the coal combustion at the Banjar Sari power plant in Lahat Regency, South Sumatera. The fly ash will undergo physical activation through a heat treatment process to enhance its adsorption capacity. This process aims to modify the surface properties and pore structure of the fly ash, making it more effective in adsorbing contaminants from acid mine drainage. Based on **Figure 1** below, is an image of comparison to coal fly ash and coal bottom ash physical form.



Figure 1. Coal Fly Ash & Coal Bottom Ash

The acid mine drainage samples used in the study will be collected at the same time, and an initial analysis will be conducted to determine the Fe and Mn

content before performing adsorption using fly ash. Additionally, pH and TSS measurements will be taken to assess the changes after the adsorption process.

Methods

The first step will involve heating the fly ash at temperatures of 100, 200, and 300°C, with each sample weighing 300 g. Additionally, 300 g of unheated fly ash will also be prepared.

The next step involves treating the mass for the adsorption process of acid mine drainage: Five Erlenmeyer flasks, each containing 130 mL of acid mine drainage, will be added to the fly ash adsorbent with varying masses of 6, 8, 10, 12, and 14 g. This procedure will be performed for each adsorbent, including the unheated adsorbent and the adsorbents heated at 100, 200, and 300°C. The mixtures will be stirred at a speed of 150 rpm for 60 minutes, then allowed to settle for 15 minutes before being filtered using filter paper. The obtained filtrate will then be analyzed for the remaining Fe and Mn content using AAS. Measurements of pH and TSS will also be conducted on each filtrate.

Data Analysis

The characterization of the adsorbent at this stage will be performed using a NOVA instrument with the BET isotherm method to determine the surface area of each adsorbent that has been heated at temperatures of 100, 200, and 300°C.

RESULTS AND DISCUSSION

Characteristics of Fly Ash Based on Surface Area Analyzer

Based on the results from the Surface Area Analyzer, the adsorbent heated at 300°C has a surface area of 22.658 m²/g. This surface area is larger compared to the unheated adsorbent and those heated at 100 and 200°C. Physical activation of the adsorbent through heating enhances the adsorbent's capacity to adsorb substance.

Adsorption is an effective method for removing metal ions from mining wastewater. This method is cost-efficient, especially when using waste materials. Therefore, this study will examine the capability of fly ash as an adsorbent to reduce Fe and Mn contaminants and increase the pH of acid mine drainage by applying physical activation through heat treatment.

According to a study by Rahman, on the purification of used cooking oil using shell-based adsorbents, an increase in surface area results in more open pores in the adsorbent [13]. A similar study by Muttaqi, found that heating natural zeolite at 500°C for 3 hours increased the surface area of the zeolite

from 9.0539 to 9.0746 m². This process causes the loss of water content and reduces impurities trapped in the pores of the natural zeolite [14].

Tabel 1. Results of Adsorbent Surface Area Analysis

No.	Adsorbent	Surface Area (m ² /g)
1	Without	13,692
	Heating	
2	Adsorbent	18,186
	Heating	
3	100 °C	
	Adsorbent	20,977
4	Heating	
	200 °C	
4	Adsorbent	22,658
	Heating	
	300 °C	

According to research by Yefremova, the activation of rice husk biochar at 600°C significantly enhanced its surface area, leading to an improvement in its adsorption capacity for heavy metals from wastewater [15]. The heat treatment resulted in the removal of volatile organic compounds and impurities, which opened up additional micropores and increased the overall porosity of the adsorbent. This finding highlights the importance of thermal activation in enhancing the efficiency of natural adsorbent materials.

Effect of Adsorbent Mass on the Quality of Acid Mine Drainage

The effect of adsorbent mass on the reduction of Fe, Mn, TSS, and pH levels in acid mine drainage using both unheated and heated adsorbents at 100, 200, and 300°C with a contact time of 60 minutes and stirring speed of 150 rpm is presented in **Table 2**.

pH Level After Adsorption

The addition of adsorbents, such as fly ash, to acid mine drainage can significantly alter the pH due to the presence of metal oxides like calcium, magnesium, and aluminum oxides, which exhibit basic properties.

These oxides react with the acidic components of the drainage, neutralizing the solution and raising the pH. Research has shown that as the mass of the adsorbent increases, there is a corresponding increase in pH due to the higher availability of basic oxides to buffer the acidic environment.

Table 2. Results of Acid Mine Drainage Analysis (130 mL) with Adsorbents at a Contact Time of 60 Minutes and Stirring Speed of 150 RPM

No	Adsorbent	Adsorbent mass (g)	pH	TSS (mg/l)	Fe (mg/l)	Mn (mg/l)
1	Without Adsorbent	0	3.2	893	3.42	3.55
1	Adsorbent	6	6.9	740	3.30	3.46
2		8	7.2	573	3.16	3.22
3		10	7.4	403	3.10	2.34
4		12	7.5	303	2.98	2.20
5		14	8.0	177	2.83	2.02
1	Heated Adsorbent 100°C	6	7.4	620	3.24	3.41
2		8	7.7	530	3.12	2.55
3		10	7.9	370	2.98	2.24
4		12	8.0	193	2.70	2.11
5		14	8.3	170	2.46	1.80
1	Heated Adsorbent 200°C	6	7.4	443	3.12	3.34
2		8	7.8	283	2.97	2.52
3		10	8.0	277	2.82	2.19
4		12	8.0	192	2.63	2.07
5		14	8.5	150	2.44	1.67
1	Heated Adsorbent 300°C	6	7.6	407	2.86	3.19
2		8	8.0	193	2.64	2.35
3		10	8.2	173	2.30	2.09
4		12	8.3	163	2.16	1.83
5		14	8.6	90	2.09	1.56
Technical Approval for Compliance BML			6 - 9	100	3.00	3,00

The magnitude of this pH shift depends on factors such as the type of adsorbent used and its activation process. With varying adsorbent masses, based on research results, are obtained as follows:

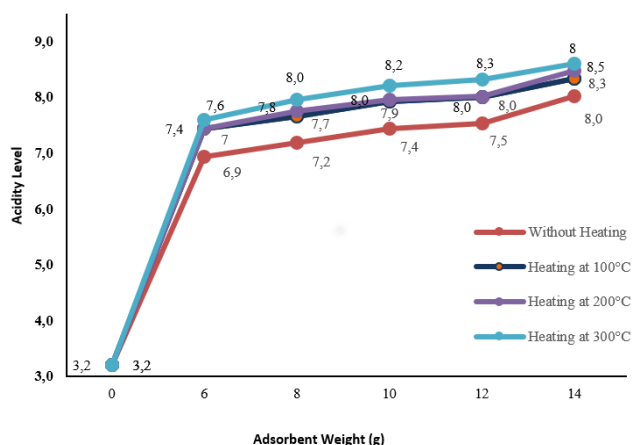


Figure 2. Change in pH of Acid Mine Drainage After Addition of Adsorbent

Based on **Figure 2**, the most significant change in pH in acid mine drainage occurs with an adsorbent mass of 14 g, with pH values increasing from pH = 3.2 to pH = 8.0 (unheated adsorbent), pH = 8.3 (adsorbent heated at 100°C), pH = 8.5 (adsorbent heated at 200°C), and pH = 8.6 (adsorbent heated at 300°C). These results are consistent with previous research on the effectiveness of using coal fly ash as an adsorbent to neutralize acid mine drainage in the settling pond of Banko PT. Bukit Asam (Persero), Tbk [16].

The data conducted by another researcher by [17] from the ABCC curve they created between the titration of HCl and H₂SO₄ shows that the reactivity of fly ash is better against HCl, as indicated by the higher initial pH value. However, the neutralization characteristics of fly ash towards the two types of standard acids do not show a significant difference.

The study found that increasing the mass of fly ash led to notable pH changes in acid mine drainage. This effect is largely attributed to the presence of silicate oxides, which enhance the availability of active sites for the adsorption process. Moreover, the fly ash contains carbonate minerals such as aragonite (CaCO₃), periclase (MgO), and thomsonite (NaCa₂(Al₅Si₅)O₂₀), which act as alkaline compounds.

These minerals help create a basic environment in the solution, effectively neutralizing the acidic components and thus raising the pH of the acid mine drainage. The higher the mass of fly ash used, the greater the pH increase due to the enhanced interaction with these minerals.

Total Suspended Solid (TSS) After Adsorption

Based on **Table 1**, the data show that after mixing with the adsorbent, the total suspended solids (TSS) content in the acid mine drainage generally decreases. As the mass of the adsorbent increases, the TSS content also decreases, the results from the research, are obtained as shown on **Figure 3**.

Based on **Figure 3**, in general, after the addition of the adsorbent, the reduction in TSS content does not meet environmental quality standards, except after adding 14 g of adsorbent heated at 300°C with a contact time of 60 minutes and a stirring speed of 150 rpm. The TSS content decreased by 89.92%, from 893 to 90 mg/L, which meets the technical standard for wastewater quality, i.e., TSS ≤ 100 mg/L. This result is also supported by another research Wardani, who studied the characteristics of fly ash (fly ash) as an adsorbent material in wastewater containing metals, where adsorbents activated through heating were able to reduce total dissolved solids [18].

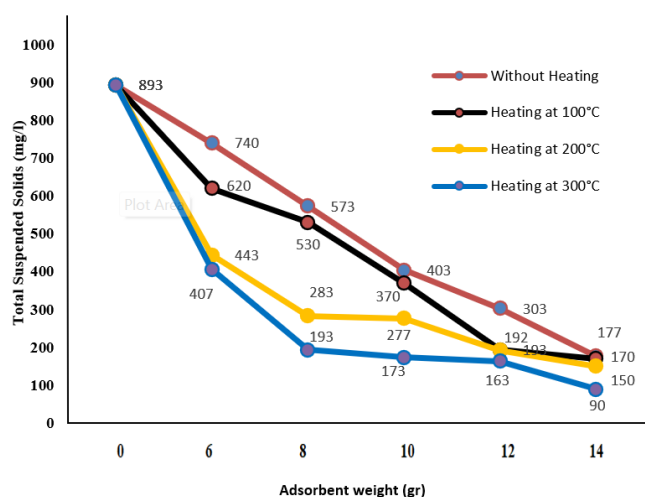


Figure 3. Change in TSS of Acid Mine Drainage After Addition of Adsorbent

The physical activation process can remove impurities, thereby increasing the surface area and opening the pores of the adsorbent [19]. The adsorbent activated by heating at 300°C has a larger surface area and thus better adsorption capacity compared to the adsorbent before heating or those heated at 100 and 200°C.

Another research stated by Ramadhani, shows that her overall result analysis experiments for the optimum filtration time is 180 minutes, during which the filtration results using bottom ash adsorbent indicate the highest percentage reduction of BOD₅, COD, and TSS at 55.4%, 55.3%, and 20.4%, respectively [20]. The filtration results using fly ash adsorbent at 180 minutes show the highest percentage reduction of BOD₅, TSS, and a pH that is neutral.

Iron (Fe) After Adsorption

The iron (Fe) content in acid mine drainage can be reduced after being adsorbed by the adsorbent. The study was conducted using varying adsorbent masses to determine how effectively the Fe content can be reduced to meet the technical standards for wastewater quality. Based on research results, are obtained as shown on **Figure 4**.

Based on **Figure 4**, the iron (Fe) content shows a maximum reduction at a contact time of 60 minutes and a stirring speed of 150 rpm, using an adsorbent with a mass of 14 g heated at 300°C, reducing from 3.42 to 2.09 mg/L, or a decrease of 38.89%, which meets the technical standard for wastewater quality, i.e., Fe ≤ 3.00 mg/L. The pH in acid mine drainage after the addition of the adsorbent can influence the level of iron (Fe) adsorption, which can be explained using the

hydrolysis adsorption mechanism with the following reaction equation.

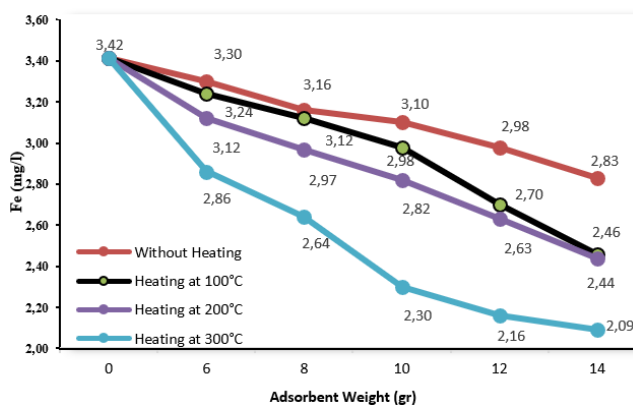


Figure 4. Change in Fe Content of Acid Mine Drainage After Addition of Adsorbent

A similar study was conducted by Roulia using fly ash to remove various heavy metals and two of them are Fe and Mn, she stated that for loadings higher than 5 g/50 mL wastewaters both FAs proved equally effective in (heavy-)metal-removal i.e., Cd, Co, Cu, Fe, Mn, Ni, Pb and Zn were completely removed [21]. Kardia Fly Ash cleaned off Cr as well while Megalopolis Fly Ash retained about 50 % of it. While in our research, the highest percentage of the adsorption are around 85% with the heat treated adsorbent in 300°C.

Here, X represents the surface of the adsorbent, in this case, fly ash. At pH > 4, the adsorption of fly ash onto Fe(II) metal increases. This is because, under these conditions, the concentration of H⁺ ions decrease, and the equilibrium shifts to the right, in accordance with Le Chatelier's principle, which states that if a system at equilibrium experiences a decrease in one component, the equilibrium will shift towards the component that was decreased [22]. An increase in mass and surface area of fly ash leads to a rise in FeOH⁺, enhancing the adsorption capacity of fly ash for Fe(II) metal. However, as the pH exceeds 7, indicating an increase in OH⁻ ions (basic property), the adsorption capacity of fly ash decreases, making it less effective at binding Fe²⁺ ions.

Manganese (Mn) After Adsorption

The adsorbent, which functions to remove contaminants in acid mine drainage, can also adsorb manganese (Mn) content. The process is fundamentally like the reduction of iron (Fe) content. As the mass of the adsorbent increases, the acidity of the acid mine drainage will also increase. The adsorbent's adsorption capacity will decline once the

fly ash can no longer effectively adsorb, leading to an increase in OH^- ions or the solution becoming basic. Based on research results, are obtained as depicted on **Figure 5**:

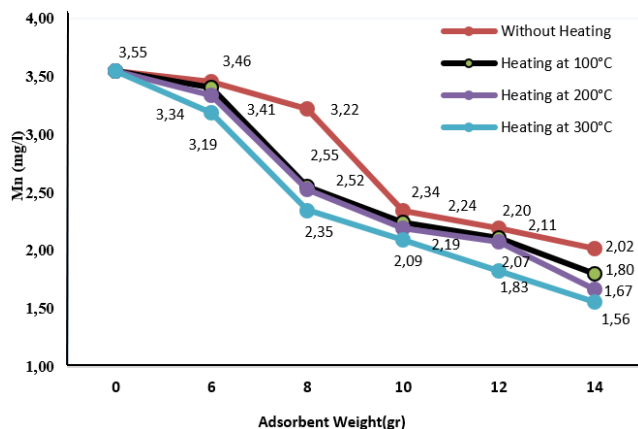


Figure 5. Change in Mn Content of Acid Mine Drainage After Addition of Adsorbent

The manganese (Mn) content shows a maximum reduction at a contact time of 60 minutes and a stirring speed of 150 rpm using an adsorbent heated at 300°C, decreasing from 3.55 to 1.56 mg/L, or a reduction of 56.06%, which meets the technical standard for wastewater quality, $\text{Mn} \leq 3.00$ mg/L. According to previous research by Eka Putri, the physical activation process can remove impurities, thereby increasing the surface area and opening the pores of the adsorbent [19]. This study aligns with the findings that the reduction in Mn content occurs due to the use of a larger adsorbent mass compared to other adsorbents. Additionally, the adsorbent activated at 300°C has a larger surface area, resulting in better adsorption capacity for Mn compared to the unheated adsorbent and those heated at lower temperatures.

Compared to previous research conducted by Pratama, stated that the effectiveness for the heavy metal Fe is achieved with a dosage of 17 grams and a stirring time of 60 minutes, resulting in a reduction of 93.04%. Meanwhile, for the heavy metal Mn, a dosage of 17 grams results in a reduction of 82.64% at a stirring time of 90 minutes [23]. Compared to our research the Mn adsorption percentage is 88% with 14 g of adsorbent mass, and the Fe adsorption percentage is 85% also with 14 g of mass.

This differential was bound to happens because the adsorbent which we use on this research was pre-treated first with heat to increase the adsorbent capability.

Compliance with environmental quality standards for pH reduction is achieved with adsorbent masses of 6, 8, 9, 12, and 14 g, stirring speed of 150 rpm, and

contact time of 60 minutes, using both unheated adsorbent and adsorbents heated at 100, 200, and 300°C. For TSS reduction, compliance with environmental quality standards is only met at a mass of 14 g with the adsorbent heated to 300°C. For Fe and Mn reduction, a decrease starts occurring with an adsorbent mass of 12 g using the unheated adsorbent, with significant reductions observed from a mass of 12 g for the adsorbent heated at 100°C. For fly ash adsorbents heated at 200 and 300°C, reductions in Fe and Mn levels in acid mine drainage begin at an adsorbent mass of 8 g. The results indicate that the activation process of fly ash adsorbent through heating is more effective than using unheated adsorbent, consistent with the surface area of each adsorbent.

CONCLUSION

Activation of fly ash adsorbent by heating at different temperatures shows that a higher surface area is achieved at 300°C (22.658 m^2/g) compared to 100 and 200°C. The heated fly ash effectively reduces Fe and Mn levels in acid mine drainage, particularly at 200 and 300°C, using 8 g of adsorbent, a contact time of 60 minutes, and a stirring speed of 150 rpm. Additionally, the pH of the acid mine drainage increases with both heated and unheated fly ash adsorbents, while the reduction in total suspended solids (TSS) meets quality standards with 14 g of adsorbent heated to 300°C. Previous research has primarily focused on the use of unmodified or chemically modified adsorbents, with less emphasis on the specific role of thermal activation in optimizing adsorption efficiency. The present study fills this gap by demonstrating that heating, particularly at 300°C, significantly enhances the adsorbent's performance, suggesting that thermally activated fly ash can be applied for advanced treatment of acid mine drainage.

ACKNOWLEDGMENT

Thanks are extended to DRTPM Kemdikbudristek for funding this research according to the 2024 fiscal year contract.

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