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Comparison of Activation Methods for Coal Ash as an Adsorbent in the Removal of Lead (Pb²⁺) from Aqueous Solution

Budi Setya Wardhana ¹, Farrah Fadhillah Hanum ^{1,*}, Zahrul Mufrodi ¹, Annisa Vada Febriani ¹, Siti Salamah ¹



- ¹ Magister of Chemical Engineering, Faculty of Technology Industry, Universitas Ahmad Dahlan, Yogyakarta, Indonesia
- * Corresponding author: farrah.hanum@che.uad.ac.id

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Abstract

Coal ash, also known as fly ash and bottom ash (FABA), is a byproduct of coal combustion that can be utilized as an adsorbent for removing lead (Pb2+) ions from wastewater. While previous studies have explored both unactivated and activated forms of FABA, few have directly compared their adsorption performance under identical operating conditions. This study investigates the characteristics of FABA before and after activation and evaluates its effectiveness in Pb2+ ion removal. Characterization was performed using X-ray fluorescence (XRF) for chemical composition and Brunauer-Emmett-Teller (BET) analysis for surface area and pore structure. XRF results showed that fly ash (FA) and bottom ash (BA) are dominated by Fe₂O₃, SiO₂, and Al₂O₃, which contribute to the adsorption capacity. BET analysis revealed that FA has a higher specific surface area (6.377 m²/g) compared to BA (0.848 m²/g), indicating greater potential for adsorption. The adsorption performance was evaluated using Atomic Absorption Spectroscopy (AAS) after 60 minutes of contact time at an initial Pb2+ concentration of 100 ppm. Both FA and BA exhibited high Pb2+ removal efficiencies, reaching up to 100% under optimal conditions. Physical activation and chemical activation using neutral (H₂O) and alkaline (NaOH and KOH) agents enhanced adsorption efficiency (98-100%), whereas activation with acidic agents (HCl and H₂SO₄) reduced it to 87-97%. These results demonstrate that FABA, particularly FA, has strong potential as an efficient and environmentally friendly adsorbent for heavy metal removal. The findings also suggest that water activation offers a simple and sustainable approach for large-scale wastewater treatment applications.

1. Introduction

The rapid growth of industrialization, agriculture, and domestic sectors has significantly increased wastewater generation and contributed to global warming [1, 2]. Among the hazardous components in wastewater, heavy metals such as lead (Pb), cadmium (Cd), chromium (Cr), mercury (Hg), arsenic (As), and copper (Cu) pose severe risks to human health and the environment [3]. Unlike organic pollutants, heavy metals are non-biodegradable and persist in the environment for decades, originating from sources such as industry, mining, and fossil fuel combustion [4].

Heavy metals persist in the environment and bioaccumulate through water, air, and soil, eventually entering the food chain [5]. Heavy metal contamination, particularly lead (Pb2+) at high concentrations, can cause damage to the central nervous system, impair kidney and liver function, alter blood composition, and disrupt energy production in the body. Research conducted by Bakulski et al. [6] and Yang et al. [7] has shown that the accumulation of heavy metals increases the risk of various diseases, including cancer, chronic kidney disease, neurological disorders, and especially cardiovascular diseases.

Various technologies have been developed to remove heavy metals from wastewater, such as adsorption, coagulation, gamma irradiation, sedimentation, biological treatment, flocculation, and photocatalysis [1, 8]. While these methods can effectively reduce contamination, some are costly and complex. Among these techniques, adsorption is particularly attractive due to its high efficiency, operational simplicity, and costeffectiveness [9, 10]. Adsorption is a process in which ions or molecules (adsorbates) from a liquid or gas adhere to the surface of a solid material (adsorbent) through physical or chemical interactions [11].

Coal ash (FABA), a solid byproduct of coal combustion in power plants, is considered a potential adsorbent for heavy metal removal [12]. FABA is generally categorized into two types: fly ash (FA) and bottom ash (BA) [13]. FA also exhibits a lower bulk density compared to BA [14]. In contrast, BA has a coarse texture and larger particle size, resembling sand, with a smaller surface area [15].

Both FA and BA are primarily composed of silica (SiO_2) and alumina (Al_2O_3) , which enable interaction with heavy metal ions [16]. The adsorption capacity of FABA depends on its surface properties and porosity, which can be enhanced through activation. Physical activation improves surface area and pore development, while chemical activation introduces functional groups that increase binding affinity for metal ions. Although several studies have examined the use of FABA as an adsorbent under both activated and unactivated conditions, few have directly compared different activation methods under identical experimental conditions [17, 18]. Such comparisons are essential to identify the most effective approach and support practical applications in wastewater treatment.

This study investigates the effect of physical and chemical activation on the properties and adsorption performance of coal ash for Pb²⁺ ions removal. Therefore, this study investigates the effect of physical and chemical activation on the properties and adsorption performance of coal ash for Pb²⁺ ions removal. Characterization was conducted using Brunauer-Emmett-Teller (BET) for surface area and porosity analysis, X-ray fluorescence (XRF) for elemental composition, and atomic absorption

spectrophotometry (AAS) for Pb^{2+} ion concentration measurements. The findings are expected to contribute to the development of low-cost, efficient, and sustainable adsorbent materials for mitigating heavy metal contamination in water.

2. Experimental

2.1. Materials

The materials used in this study were FABA obtained from a coal-fired power plant (PLTU X) in Indonesia, lead(II) nitrate (Pb(NO $_3$) $_2$) (Merck, Germany), potassium hydroxide (KOH) (Merck, Germany), sodium hydroxide (NaOH) (Merck, Germany), hydrochloric acid (HCl) (Merck, Germany), sulfuric acid (H $_2$ SO $_4$) (Merck, Germany), and distilled water.

2.2. Preparation of FABA adsorbent

Coal ash (FABA) was initially washed three times with distilled water to remove impurities and then ovendried at 105°C for 6 hours to reduce moisture content. The dried sample was ground and sieved using a 100-mesh sieve to obtain a uniform particle size. After preparation, the samples were characterized using X-ray fluorescence (XRF; PANalytical Epsilon 4) for chemical composition and Brunauer–Emmett–Teller (BET; Quantachrome Nova 2200e) for surface area and pore analysis. The prepared samples were divided into three categories: Unactivated, physically activated, and chemically activated, as illustrated in Figure 1.

2.2.1. Physical Activation

Physical activation was performed by heating the samples in a furnace (Vulcan D550) at 100, 200, 300, 400, and 500°C for 3 hours. This treatment aimed to enhance the surface area and pore structure of the adsorbent, thereby improving its adsorption performance [19].

2.2.2. Chemical Activation

For chemical activation, the samples were soaked in different activating solutions: 3 M NaOH, 3 M KOH, 3 M HCl, 3 M H $_2$ SO $_4$, and distilled water for 24 hours. After soaking, the samples were thoroughly rinsed with distilled water (six times) to remove residual chemicals, then filtered and oven-dried at 105°C for 6 hours.

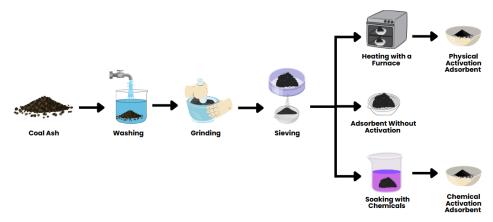


Figure 1. Scheme of the preparation and activation process of FABA adsorbent

2.2.3. Without Activation

Unactivated FABA was used as a control after grinding and sieving. This approach leveraged FABA's inherent surface properties that naturally facilitate adsorption. Based on the preparation stages that have been carried out, the variations in this study for FA and BA-based adsorbents include several special treatments aimed at enhancing the effectiveness of the adsorption process. These treatments are designed to optimize the physical and chemical characteristics of the adsorbent, thereby enhancing its capacity to absorb heavy metal ions more efficiently. The variations of the treatments used are presented in Table 1.

2.3. Preparation of Lead (Pb2+) Metal Solution

A synthetic Pb^{2+} ion solution (100 mg/L) was prepared by dissolving 0.16 g of $Pb(NO_3)_2$ in distilled water and diluting to 1 L in a volumetric flask. The actual concentration was verified using an atomic absorption spectrophotometer (AAS; Shimadzu AA-7000).

2.4. Adsorption Process

Batch adsorption was conducted by mixing 10 g of adsorbent with 200 mL of Pb^{2+} ions solution (100 mg/L) in a 250 mL beaker. The mixture was stirred at 500 rpm for 1 hour at room temperature (30°C) using a magnetic stirrer. After the contact time, the solution was filtered, and the residual Pb^{2+} concentration was measured using AAS. The experimental setup is shown in Figure 2.

2.5. Adsorption and Isotherm Study

The removal efficiency (%RE) of Pb ion was determined after the adsorption process using an AAS. The calculation of %RE was based on Equation (1).

$$\%RE = \frac{(c_0 - c_e)}{c_0} \times 100 \tag{1}$$

Where, C_0 and C_e are the initial and equilibrium concentrations of Pb ion (mg/L), respectively. Equilibrium adsorption studies were conducted by mixing 200 mL of Pb ion solution at various initial concentrations with 10 g of FA or BA. The mixture was stirred for 60 minutes, which was sufficient to reach equilibrium conditions. The resulting data were then analyzed using the Freundlich and Langmuir isotherm models to describe the adsorption mechanism and evaluate the maximum adsorption capacity of the coal ash.

2.5.1. Freundlich Isotherm

The Freundlich model was developed as an empirical approach to represent the adsorption process that occurs on heterogeneous adsorbent surfaces [20]. In this study, the Freundlich model was used to explain the interaction between Pb ions and FA or BA, which is expressed by Equation (2).

$$\ln q_e = \frac{1}{n} \ln C_e + \ln k_F \tag{2}$$

Where, q_e (mg/g) is the amount of Pb adsorbed at equilibrium, C_e (mg/L) is the equilibrium concentration, k_F is the adsorption capacity constant, and 1/n indicates adsorption intensity. The values of k_F and n were obtained from the slope and intercept of the linear plot of $\ln q_e$ versus $\ln C_e$. A value of n > indicates favorable adsorption.

2.5.2. Langmuir Isotherm

The Langmuir isotherm assumes monolayer adsorption on a homogeneous surface without interactions between adsorbed molecules [21]. Its linear form is given in Equation (3).

$$\frac{c_e}{q_e} = \frac{1}{k_L q_e} + \frac{c_e}{q_m} \tag{3}$$

Where, $C_{\rm e}$ (mg/L) is the equilibrium concentration of Pb ions in the solution, $q_{\rm e}$ (mg/g) is the amount of Pb ions adsorbed at equilibrium, $q_{\rm m}$ (mg/g) is the maximum adsorption capacity, and $k_{\rm L}$ (L/mg) is the Langmuir constant related to adsorption energy. The parameters $q_{\rm m}$ and $k_{\rm L}$ are obtained from the slope and intercept of the linear plot of $C_{\rm e}/q_{\rm e}$ versus $C_{\rm e}$.

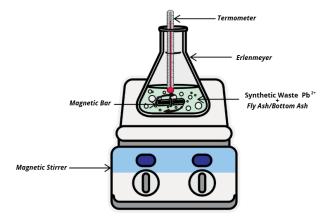


Figure 2. Experimental apparatus for batch adsorption

Table 1. Variation of FA and BA adsorbents

Without activation -	Physical activation (°C)					Chemical activation				
	500	400	300	200	100	NaOH	КОН	HCl	H ₂ SO ₄	H ₂ O
FA										
FA_{o}	FF_1	FF_2	FF_3	FF_4	FF_5	FK_1	FK_2	FK_3	FK_4	FK_5
BA										
BA_0	BF_1	BF_2	BF_3	BF_4	BF_5	BK_1	BK_2	BK_3	BK_4	BK_5

Legend:

FA₀: Fly ash without activation

FF₁-FF₅: Fly ash physically activated at 100-500°C

FK1-FK5: Fly ash chemically activated with H2O, NaOH, KOH, HCl, and H2SO4

 $BF_1\text{--}BF_5\text{:}$ Bottom ash physically activated at 100–500°C

 FK_1-FK_5 : Bottom ash chemically activated with H_2O , NaOH, KOH, HCl, and H_2SO_4

Results and Discussion

3.1. Chemical Composition of FA and BA

XRF analysis was employed to identify the elemental composition of the FA and BA materials used as adsorbents. This chemical composition information was essential to understand the adsorption potential and surface interactions of FA and BA with Pb2+ ions [22]. The XRF analysis results of FA and BA before activation are presented in Table 2. The analysis results indicated that both FA and BA mainly consisted of metal oxides such as Fe₂O₃, SiO₂, CaO, and Al₂O₃, which played a crucial role in the adsorption process of Pb^{2+} ions. The Fe_2O_3 content in FA was 34.8%, which was higher than that in BA (32%). This finding was consistent with the results reported by Ulewicz and Jura [23], who observed that the Fe₂O₃ composition in BA was generally lower than in FA. The presence of Fe₂O₃ contributed to the magnetic properties, high surface area, and positive surface charge of the material, thereby enhancing its interaction with pollutants. In addition, Fe₂O₃ improved the thermal stability of the adsorbent, making both FA and BA more effective as base materials for adsorption processes [24].

In the context of Pb^{2+} ion adsorption, the presence of Fe_2O_3 played an important role. Iron oxides could interact with Pb^{2+} ions through several mechanisms, including ion exchange, surface complexation, and co-precipitation [25]. The hydroxyl groups (-OH) on the Fe_2O_3 surface acted as active sites capable of binding Pb^{2+} ions to form Fe-O-Pb surface complexes, thereby enhancing the adsorption capacity. Moreover, under certain pH conditions, Fe_2O_3 could undergo protonation or deprotonation, influencing the surface charge and strengthening the electrostatic attraction between the adsorbent surface and Pb^{2+} ions [26]. Therefore, the higher Fe_2O_3 content in FA compared to BA was considered one of the main factors contributing to its superior Pb^{2+} ions adsorption capability.

The XRF analysis also revealed that the SiO_2 content in BA (33.20%) was higher than in FA (29.6%). This result was consistent with the findings of Menéndez et al. [27], who reported that BA typically contained higher SiO_2 levels than FA. The presence of SiO_2 was important in forming a porous structure and a large surface area, both of which enhanced the adsorption capacity and efficiency [28]. Thus, both FA and BA exhibited strong potential as adsorbent materials for heavy metal removal, particularly Pb^{2+} ions [29].

In the adsorption process, SiO_2 interacted with Pb^{2+} ions mainly through physical adsorption involving surface complex formation and ion exchange [30]. The hydroxyl groups (-Si-OH) on the SiO_2 surface could undergo deprotonation under certain pH conditions, generating negatively charged sites ($-Si-O^-$) that electrostatically attracted and bound Pb^{2+} ions [31]. Furthermore, chemical interactions between silanol groups and metal ions could result in the formation of strong and stable Si-O-Pb coordination bonds [32]. These mechanisms enabled Pb^{2+} ions to be bound to the SiO_2 surface both physically and chemically.

Table 2. XRF analysis results of FA and BA

Compound	% Chemical composition			
Compound	FA	BA		
Fe ₂ O ₃	34.8	32		
SiO ₂	29.6	33.2		
CaO	23	24.6		
Al_2O_3	9.7	7.7		
TiO_{2}	1.4	1.3		
K_2O	1.2	0.9		
MnO	0.3	0.3		

The XRF results also showed that CaO contents in both ashes were relatively similar, although slightly higher in BA (24.6%) than in FA (23%). This finding aligned with Bertolini *et al.* [33], who noted that BA generally contained higher CaO levels than FA. The presence of CaO played an essential role in imparting alkalinity to the adsorbent material, which supported the coagulation and precipitation of Pb²⁺ ions during adsorption [34].

During the Pb²⁺ ion adsorption process, CaO contributed to improving adsorption efficiency. Upon contact with water, CaO was hydrated to form Ca(OH)₂, which increased the solution's pH and created an alkaline environment [35]. Such conditions facilitated the formation of Pb(OH)₂ or PbCO₃ precipitates on the adsorbent surface, effectively reducing the concentration of dissolved Pb²⁺ ions [36]. Additionally, Ca²⁺ ions released from CaO could compete with Pb²⁺ for active sites on the adsorbent surface, but under certain conditions, they could also promote surface complex formation via ion exchange mechanisms [37].

Another major component identified by XRF was Al_2O_3 , which was found in higher concentrations in FA (9.39%) than in BA (7.42%). This observation was consistent with [27] Menéndez et al. [27] who reported that FA typically contained more Al_2O_3 than BA. The presence of Al_2O_3 contributed to increasing surface area, thermal stability, and adsorption capacity toward pollutants [38].

Furthermore, Al_2O_3 in both FA and BA enabled interactions with Pb^{2+} ions through electrostatic forces and surface complexation (Al–O–Pb), thereby enhancing the overall adsorption capacity [39]. The relatively high Al_2O_3 content also improved the chemical durability and stability of the adsorbent, making it more robust and suitable for reuse after regeneration [38].

Differences in the chemical composition of adsorbent materials could influence their surface physical characteristics, such as specific surface area, average pore size, and total pore volume [40]. To evaluate how these compositional differences affected porosity and surface area, BET analysis was conducted, and the results are presented in Table 3.

Table 3. BET of FA and BA analysis results

Result	Adsorbent			
Result	FA	BA		
Specific surface area (m²/g)	6.377	0.848		
Total pore volume (cm³/g)	13.1×10^{-3}	3.7×10^{-3}		
Average pore diameter (nm)	8.2104	17.6296		

The BET analysis results revealed a significant difference between the adsorbent characteristics of FA and BA. FA exhibited a specific surface area of $6.377~\mathrm{m}^2/\mathrm{g}$ and a total pore volume of $13.1\times10^{-3}~\mathrm{cm}^3/\mathrm{g}$, both of which were substantially higher than those of BA ($0.848~\mathrm{m}^2/\mathrm{g}$ and $3.7\times10^{-3}~\mathrm{cm}^3/\mathrm{g}$, respectively). These values indicated that FA possessed a more porous structure with a greater number of active sites, suggesting a higher adsorption efficiency for pollutants [41, 42]. Meanwhile, BA showed a larger average pore diameter than FA, indicating a mesoporous–to–macroporous structure, whereas FA was predominantly mesoporous [43]. The mesoporous structure of FA was considered more effective for Pb²⁺ ions adsorption, as it enhanced contact between the adsorbent surface and the pollutant [44].

Based on the XRF and BET analyses, the adsorption of Pb^{2+} ions by FA and BA was likely governed by ion-exchange mechanisms supported by the presence of metal oxides such as Fe_2O_3 , Al_2O_3 , SiO_2 , and CaO [45]. Pb^{2+} ions in the solution interacted with negatively charged active surface groups ($-O^-$ and -OH) by replacing other metal ions such as Ca^{2+} , Na^+ , or K^+ [46]. Additionally, the presence of basic CaO increased the solution pH and promoted $Pb(OH)_2$ precipitation, which further enhanced Pb^{2+} ions removal efficiency [37]. Therefore, the adsorption mechanism of Pb^{2+} ions onto FA and BA could be described as a combination of ion exchange and surface complexation processes, both contributing to the high efficiency of Pb^{2+} ions removal.

3.2. Adsorption of Pb2+ Ions by FA and BA Adsorbents

The removal of heavy metal ions was carried out under identical operating conditions for each sample. The adsorption process was conducted for 60 minutes with an initial Pb²⁺ ion concentration of 100 ppm and a stirring speed of 500 rpm. After adsorption, the solution was filtered, and the filtrate was analyzed using AAS to determine the final concentration of Pb²⁺ ions. This value was then used to calculate the adsorption efficiency and evaluate the performance of the adsorbents in removing heavy metals. The analysis aimed to compare the performance of FA and BA adsorbents that underwent physical or chemical activation with those that remained unactivated.

3.2.1. FA-based Adsorbent

The calculated percentage removal of Pb²⁺ ions using FA-based adsorbents is presented in Figure 3. The calculations were performed after the adsorption process to evaluate the effectiveness of each adsorbent in removing Pb²⁺ ions from the synthetic solution.

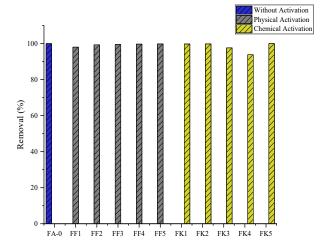


Figure 3. Percentage removal of Pb²⁺ ions using FAbased adsorbents under various physical and chemical activation conditions

Based on Figure 3, the effectiveness of Pb2+ ion removal by FA-based adsorbents was notably high, ranging from 93.85% to 100%. For samples subjected to physical activation via stepwise heating at 100-500°C, the removal efficiency increased significantly at 100°C (FF₅) and 200°C (FF₄), with respective values of 99.83% and 99.73%. However, at higher activation temperatures of 300°C (FF₃), 400°C (FF₂), and 500°C (FF₁), the efficiency decreased slightly to 99.51%, 99.36%, and 98.04%, respectively. This decline was attributed to pore structure alterations caused by excessive heating over prolonged periods, leading to pore shrinkage or collapse and a reduction in the active surface area of the adsorbent [47]. These findings were consistent with the results reported by Mondal et al. [48], who observed that excessive activation temperatures could reduce adsorbent performance due to the loss of active sites, thereby lowering its adsorption capacity.

Chemical activation treatments of FA-based adsorbents showed that Pb2+ removal efficiency varied depending on the activating agent used. Activation with strong bases such as KOH (FK2) and NaOH (FK1), with pH values > 10, resulted in very high adsorption efficiencies of 99.86% and 99.80%, respectively. Alkaline treatments were found to enrich the surface with negatively charged functional groups, particularly -OH and Si-OH groups generated from aluminosilicate depolymerization, thereby enhancing electrostatic interactions with positively charged Pb2+ ions in accordance with Coulomb's law [49, 50, 51]. This mechanism not only increased the number of active surface sites but also improved chemical affinity toward Pb2+ ions [52]. In contrast, activation with strong acids such as HCl (FK₃) and H₂SO₄ (FK₄), with pH values < 3, decreased adsorption efficiency to 97.60% and 93.85%, respectively. This reduction was due to the dissolution of active oxide phases such as SiO₂, Fe₂O₃, and Al₂O₃, along with micropore degradation, which collectively reduced surface area and the number of adsorptive sites [53].

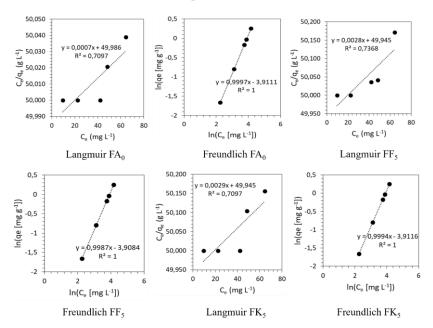


Figure 4. Isotherm curves for the adsorption of Pb²⁺ ions on FA-based adsorbents using the Langmuir and the Freundlich models

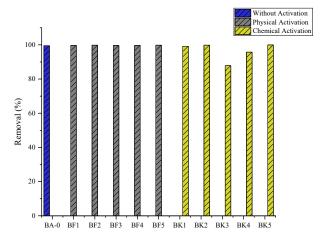


Figure 5. Percentage removal of Pb²⁺ ions using BA-based adsorbents under various physical and chemical activation conditions

Activation using a neutral chemical agent, distilled water (FK5), achieved the highest Pb2+ ion removal efficiency of 100%, surpassing both basic and acidic activations. This result indicated that water, as a simple activation agent, could preserve and even optimize the natural pore structure and surface area of FA without inducing degradation commonly caused by harsh chemical treatments [54]. A similar phenomenon was observed in the unactivated FA sample (FA₀), which also exhibited 100% efficiency, suggesting that natural FA already possessed favorable adsorptive characteristics such as well-developed porosity, adequate surface area, and active functional groups. These results were consistent with the findings of Chowdhury et al. [55], who reported that FA with high silica and alumina contents could remove heavy metal ions by more than 95% even without additional treatments.

Based on Figure 3, the samples with the highest Pb^{2+} ion removal efficiencies were FA_0 , FF_5 , and FK_5 . These three samples were further analyzed using adsorption

isotherm models to understand the interaction mechanism between Pb^{2+} ions and the FA-based adsorbent surface. The applied isotherm models included the Langmuir and the Freundlich models, as shown in Figure 4.

According to Figure 4, the determination coefficients (R^2) for the Langmuir model ranged from 0.70 to 0.73, while the Freundlich model consistently showed R^2 = 1 for all samples. This difference indicated that the Freundlich model better represented the adsorption mechanism of Pb^{2+} ions on FA-based adsorbents. These findings agreed with those of Hussain *et al.* [56], who reported that FA-based adsorbents typically followed the Freundlich model. The suitability of this model suggested that the adsorbent surface was heterogeneous, with non-uniform energy distribution, allowing multilayer adsorption to occur [57].

3.2.2. BA-based Adsorbent

The Pb2+ ion removal efficiency of BA-based adsorbents, as presented in Figure 5, ranged from 87.85% to 99.94%, indicating a high overall performance, although slightly lower than that of FA-based adsorbents. Physical activation of BA-based adsorbents produced results similar to those of FA. Low-temperature activation at 100°C (BF5) and 200°C (BF4) yielded the highest Pb2+ ion removal efficiencies of 99.83% and 99.82%, respectively. However, at higher activation temperatures of 300°C (BF₃), 400°C (BF₂), and 500°C (BF₁), the adsorption efficiency slightly decreased to 99.69%, 99.68%, and 99.64%, respectively. This reduction was attributed to the occurrence of sintering a partial fusion of particles caused by excessive heating that reduced surface area and damaged micropores [58]. This phenomenon was consistent with the observations of Aziz et al. [59], who reported that excessive physical activation temperatures could permanently alter pore structures, thereby reducing the number of active sites and overall adsorption capacity.

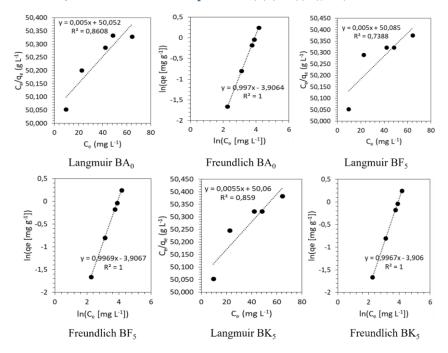


Figure 6. Isotherm curves for the adsorption of Pb^{2+} ions on BA-based adsorbents using the Langmuir and the Freundlich models

For BA-based adsorbents subjected to chemical activation, the Pb^{2+} removal efficiencies varied significantly depending on the activating agent. Activation with distilled water (BK₅) produced the highest efficiency (99.94%), followed by activation with KOH (BK₂) and NaOH (BK₁), which achieved 99.83% and 99.16%, respectively. Strong base activation enhanced adsorbent performance by dissolving inorganic impurities and opening surface pores, leading to increased specific surface area and a greater number of active sites [60, 61]. In addition, alkaline treatments reduced the crystallinity of silica (SiO₂) into a more reactive amorphous phase, thereby improving adsorption capacity [62].

Conversely, activation with strong acids such as HCl (BK₃) and H_2SO_4 (BK₄) reduced adsorption efficiency to 87.85% and 95.75%, respectively, due to the dissolution of active components such as silica and alumina, which damaged pore structures and decreased the number of adsorptive sites [63]. This dissolution effect was more pronounced in BA because of its larger pore diameter (17.63 nm) compared to FA (8.21 nm), allowing acid diffusion into the internal structure more readily [64, 65]. Meanwhile, the unactivated sample (BA $_0$) still exhibited a high efficiency of 99.93%, indicating that natural BA already possessed suitable pore characteristics and functional groups that facilitated effective adsorption.

Based on Figure 5, the samples BA₀, BF₅, and BK₅ showed the highest Pb²⁺ ion removal efficiencies. These samples were further analyzed using Langmuir and the Freundlich isotherm models to evaluate the adsorption characteristics and mechanisms of Pb²⁺ ions on BA-based adsorbent surfaces, as illustrated in Figure 6.

According to Figure 6, the R^2 values for the Langmuir model ranged between 0.73 and 0.86, whereas the Freundlich model consistently exhibited R^2 = 1 for all

samples. Comparatively, the Langmuir R² values for BA were higher than those for FA, indicating a more uniform adsorption interaction. However, overall, the Freundlich model still provided a better fit for both types of adsorbents. These findings were consistent with those of Huda *et al.* [66], who reported that BA-based adsorbents followed the Freundlich model. This conformity suggested that the adsorbent surface was heterogeneous, with a non-uniform distribution of adsorption energies, thereby allowing multilayer adsorption to occur [67].

4. Conclusion

This study successfully evaluated and compared various activation methods applied to coal ash, including fly ash (FA) and bottom ash (BA), for the removal of Pb2+ ions from aqueous solutions. The results showed that FA exhibited superior physicochemical properties compared to BA, characterized by a higher specific surface area and a mesoporous structure that supported higher adsorption capacity. Under optimal conditions (Pb2+ concentration of 100 ppm and 60 minutes of contact time), both FA and BA achieved removal efficiencies of up to 100%, with wateractivated and unactivated FA showing the best performance. Physical and alkaline chemical activations (NaOH and KOH) also improved adsorption efficiency, while acidic activations (HCl and H2SO4) and hightemperature treatments above 300°C tended to reduce performance due to pore structure degradation. The adsorption behavior of both adsorbents followed the Freundlich isotherm model, indicating a heterogeneous surface and multilayer adsorption mechanism. Future work should include detailed isotherm parameter evaluation, pH influence analysis, and replicate experiments to confirm statistical reliability. Overall, the findings highlight the strong potential of coal ash, particularly FA, as a low-cost, efficient, and environmentally friendly adsorbent for wastewater treatment applications.

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