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Synthesis of Zeolite from Coal Bottom Ash Using Hydrothermal Method as An Adsorbent for Heavy Metals

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ABSTRACT

This study aims to synthesize zeolite from coal combustion bottom ash using the hydrothermal method and evaluate its effectiveness in adsorbing heavy metals—specifically cadmium (Cd), lead (Pb), iron (Fe), and copper (Cu). Bottom ash was treated via alkali fusion at 750°C, followed by acid activation with 1 M HCl to extract silica and alumina. Zeolite synthesis was hydrothermal at 100°C with 6, 12, and 24 hours of crystallization. Sodium aluminate ($NaAlO_2$) was added to adjust the Si/Al ratio and enhance structural stability. The synthesized zeolites were characterized using X-ray Fluorescence (XRF) and Scanning Electron Microscopy (SEM). The sample crystallized for 12 hours and exhibited the highest SiO_2/Al_2O_3 ratio, measuring 3.73. Adsorption tests revealed optimal performance at pH 5–9 and 120 minutes contact time, achieving removal efficiencies of 97.84% for Pb^{2+}, 99.47% for Cd^{2+}, 99.73% for Fe^{2+}, and 99.91% for Cu^{2+}. These findings demonstrate that bottom ash-derived zeolite is a promising, low-cost adsorbent for heavy metal removal from wastewater. It supports use in sustainable and long-term industrial effluent treatment systems.

Keywords: *Adsorption; Bottom ash; Heavy metals; Hydrothermal; Zeolite*

ABSTRAK

Penelitian ini bertujuan untuk menyintesis zeolit dari abu bawah hasil pembakaran batu bara menggunakan metode hidrotermal serta mengevaluasi efektivitasnya sebagai adsorben logam berat, terutama kadmium (Cd), timbal (Pb), besi (Fe), dan tembaga (Cu). Abu bawah diproses melalui fusi alkali pada suhu 750°C dan aktivasi asam dengan HCl 1 M untuk meningkatkan ekstraksi silika dan alumina. Sintesis dilakukan secara hidrotermal pada suhu 100°C dengan variasi waktu kristalisasi 6, 12, dan 24 jam. Natrium aluminat ($NaAlO_2$) ditambahkan untuk mengatur rasio Si/Al serta memperkuat struktur kristal. Karakterisasi dilakukan menggunakan XRF dan SEM. Zeolit dengan waktu kristalisasi 12 jam memiliki rasio SiO_2/Al_2O_3 terbaik sebesar 3,73. Uji adsorpsi menunjukkan efisiensi optimal pada pH 5–9 dan waktu kontak 60 menit, dengan penyerapan sebesar 97,84% untuk Pb^{2+}, 99,47% untuk Cd^{2+}, 99,73% untuk Fe^{2+}, dan 99,91% untuk Cu^{2+}. Hasil ini membuktikan bahwa zeolit dari abu bawah merupakan alternatif murah dan efektif untuk menghilangkan logam berat dalam air limbah. Kemampuan zeolit sintesis semakin meningkatkan potensinya dalam pengolahan limbah industri secara berkelanjutan.

Keywords: *Abu dasar batubara; Adsorpsi; Hidrotermal; Logam berat; Zeolit*

INTRODUCTION

Indonesia possesses vast coal resources, and coal remains a vital source of energy, particularly for electricity generation. This reliance on coal has led to the production of large amounts of combustion residues, especially fly ash and bottom ash (FABA). Among these by-products, bottom ash accounts for approximately 10–20% and contains notable levels of silica (SiO_2) and alumina (Al_2O_3), which are valuable raw materials for zeolite production [1].

The increasing accumulation of bottom ash without proper treatment raises environmental concerns due to its alkaline nature and the possibility of leaching heavy metals into surrounding ecosystems. Improper disposal can contribute to soil degradation and the loss of arable land [2]. Despite its potential, only about 25% of coal ash is currently reused in Indonesia, with the rest discarded [3]. Zeolites are microporous crystalline materials composed of aluminosilicates. Due to their high surface area and ion-exchange capabilities, they are widely recognized as efficient adsorbents for removing heavy metals from water. Zeolites can be synthesized from bottom ash through methods such as hydrothermal processing, which can be enhanced by chemical treatments to increase porosity and functionality. Moreover, their reusability through thermal regeneration makes zeolites a sustainable choice for wastewater remediation [4].

This study investigates zeolite synthesis via a hydrothermal method preceded by alkali fusion, using coal bottom ash sourced from the coal combustion industry. A distinguishing feature of this research is its emphasis on the use of coal bottom ash, a material whose utilization as a zeolite precursor is less frequently reported in the literature compared to fly ash. Furthermore, the combined alkali fusion and hydrothermal treatment method was selected due to its assessed effectiveness and potential for greater cost-efficiency in converting this waste into a high-value adsorbent material. Further novelty of this study lies in the systematic evaluation of the influence of varying hydrothermal crystallization times (6, 12, and 24 hours) on the physicochemical characteristics (structure, morphology, and composition) of the resulting zeolite and its subsequent adsorption performance for a combination of four key heavy metals: Cd^{2+}, Pb^{2+}, Fe^{2+}, and Cu^{2+}. The synthesized zeolite was characterized using X-ray Fluorescence (XRF) and Scanning Electron Microscopy with Energy Dispersive X-ray Spectroscopy (SEM-EDX), and its adsorption capacity was evaluated under various pH and contact time conditions to determine optimal parameters and its environmental applicability.

METHOD

Preparation of Zeolite Adsorbent

First of all, the coal bottom ash was washed thoroughly with distilled water to remove impurities. It was then dried in an oven at 105°C for 12 hours until completely dry. The dried sample was sieved using a 200 mesh sieve to obtain uniform particle size. A total of 50 grams of the sieved bottom ash was mixed with solid sodium hydroxide (NaOH). The mixture was placed in a crucible and fused in a furnace at 750°C for 3 hours. After the fusion process, the product was allowed to cool at room temperature. The cooled fusion product was then dissolved in distilled water in a specific ratio and stirred using a magnetic stirrer for 1 hour until homogeneous. The solution was filtered using Whatman No. 42 filter paper to separate the insoluble residue. The filtrate was then added to a 1.5 M sodium aluminate ($NaAlO_2$) solution to enhance the aluminum content. The pH of the mixture was adjusted using 1 M hydrochloric acid (HCl) to the desired value. The solution was transferred into a stainless-steel autoclave reactor and subjected to hydrothermal treatment at 100°C for varying crystallization times of 6, 12, and 24 hours. After the hydrothermal process, the product was filtered, washed with distilled water until a neutral pH was achieved, and then dried in an oven at 105°C for 12 hours. The dried zeolite was ground using a porcelain mortar and pestle until it became a fine powder and stored in polyethylene bottles for further use.

Heavy Metal Degradation Using Zeolite Adsorbent

The synthesized zeolite adsorbent was tested for its ability to degrade heavy metal concentrations (Cd^{2+}, Pb^{2+}, Fe^{2+}, and Cu^{2+}) using a standard heavy metal solution with 20 mg/L of concentration. A total of 0.1 grams of the zeolite adsorbent was weighed and contacted with 50 milliliters of the 20 ppm heavy metal solution in a glass beaker equipped with a magnetic stirrer. The mixture was stirred using a magnetic stirrer, and samples were taken at specific contact times of 15, 30, 60, and 120 minutes to observe the adsorption performance over time. After the designated contact time, the mixture was filtered to separate the adsorbent from the filtrate. The concentration of heavy metals remaining in the filtrate was then analyzed using Atomic Absorption Spectrophotometry (AAS) to determine the efficiency of the zeolite adsorbent in reducing heavy metal concentrations.

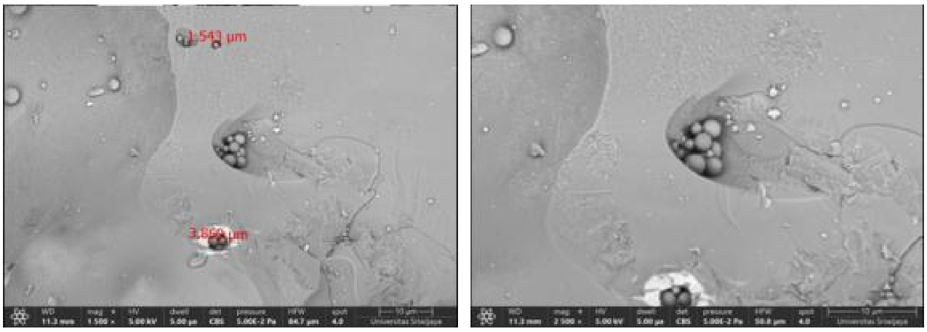
Sample Analysis

The analysis of the zeolite adsorbent made from bottom ash coal is conducted using several equipment and methods, including X-ray Fluorescence (XRF) to analyze the adsorbent structure, determine the degree of crystallinity, and observe the diffraction pattern. Scanning Electron Microscopy (SEM) is used to analyze the microstructure of the adsorbent and determine the surface morphology. Heavy Metal Degradation Analysis is performed using Atomic Absorption Spectrophotometry (AAS) to determine the concentration of heavy metals in the sample. The sample is converted into vapor and atomized into free atoms using a flame. The free atoms, in their ground state, absorb light at a wavelength corresponding to the element's properties. The intensity of the absorbed light is measured using a detector. The amount of energy absorbed is proportional to the concentration of the metal in the sample, according to Lambert-Beer's law.

RESULTS AND DISCUSSION

Zeolite Characteristic Results

The Scanning Electron Microscopy-Energy Dispersive X-ray (SEM-EDX) analysis method is a highly valuable technique for characterizing zeolites, primarily used to analyze the morphology and surface structure of the adsorbent particles and the shape of the pores on the zeolite surface (source). SEM enables high-resolution visualization of the surface morphology of zeolites, providing information on surface roughness and porosity, which are crucial factors in determining the zeolite's adsorption properties. Meanwhile, EDX is used to provide both qualitative and quantitative surface chemical composition analysis. SEM-EDX analysis was conducted to gather information about the physical structure and shape of the zeolite, which are closely related to its adsorption properties. The chemical composition also determines the zeolite's ability to interact with specific molecules and its potential in developing more efficient synthesis processes for zeolites, including those derived from industrial waste [5].



(a) (b)

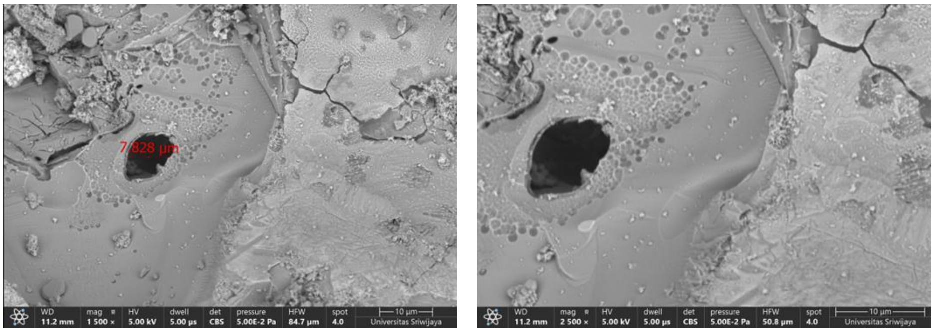
Figure 1. SEM analysis results of synthetic zeolite before activation at magnifications of (a) 1500× and (b) 2500×.

Table 1. SEM-EDS results of zeolite before activation.

Element	% Atomic	% Weight
C	4.5	2.1
O	62.9	39.1
Al	6.4	6.7
Si	21.0	22.9
Pm	5.2	29.2

The SEM analysis of zeolite before the hydrothermal process is shown in Figure 1, with magnifications of 1500x and 2500x. Based on the analysis, the morphology of the zeolite is still in the form of irregular stone-like plates. Additionally, the zeolite particles exhibit a wide variation in size and lack uniformity, with relatively smooth surface textures [6]. The observed morphology indicates that the zeolite particles have not yet developed significant surface roughness, possibly due to the presence of impurities or associated minerals adhering to the zeolite structure. Furthermore, the pores are not fully opened, suggesting the need for an activation process involving thermal treatment and chemical addition to remove impurities, open the pore structure, and clean the surface, thereby increasing the adsorbent capacity.

According to Table 1, the composition analysis of zeolite before activation shows the presence of key framework elements, including 21% silica, 6.4% aluminum, and 62.9% oxygen. The high content of silica, aluminum, and oxygen confirms the aluminosilicate framework typical of zeolite materials. Additionally, the sample contains 4.5% carbon, which may originate from organic impurities or surface contamination.



(a) (b)

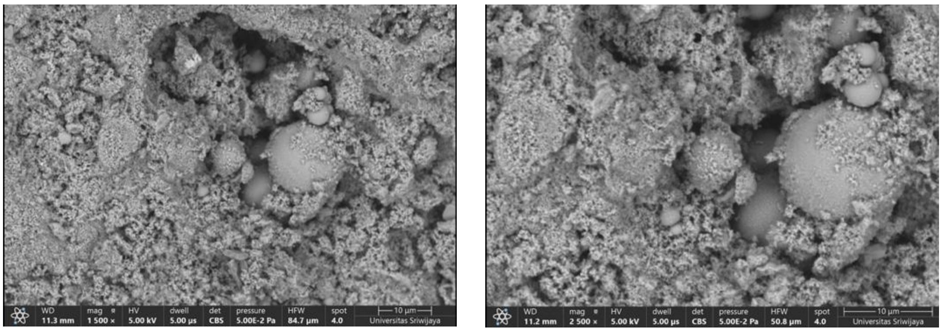
Figure 2. SEM analysis results of synthetic zeolite activated for 6 hours at magnifications of (a) 1500× and (b) 2500×.

Table 2. SEM-EDS results of synthetic zeolite activated for 6 hours.

Element	% Atomic	% Weight
C	3.8	2.1
O	60.0	48.4
Al	8.2	11.1
Si	17.7	25.0
Na	9.8	11.4
As	0.5	1.8

In Figure 2, the characterization results of the zeolite sample after hydrothermal activation for 6 hours show changes in both morphology and elemental composition. SEM observations reveal modifications on the zeolite surface, with the formation of more distinct pores or cavities approximately 7.8 µm in size. The surface also appears different, with some areas displaying a finer granular structure, while incomplete cracks are observed in certain regions.

Elemental composition analysis using EDS indicates an increase in the aluminum (Al) content compared to the previous sample, rising from 6.7 wt% to 11.1 wt%. Furthermore, sodium (Na) is detected in relatively high amounts, with a concentration of 9.8 mol% or 11.4 wt%. This is attributed to the addition of sodium aluminosilicate compounds, which successfully incorporated into the zeolite framework as charge-balancing cations [7].



(a) (b)

Figure 3. SEM analysis results of synthetic zeolite activated for 12 hours at magnifications of (a) 1500× and (b) 2500×.

Table 3. SEM-EDS results of synthetic zeolite activated for 12 hours.

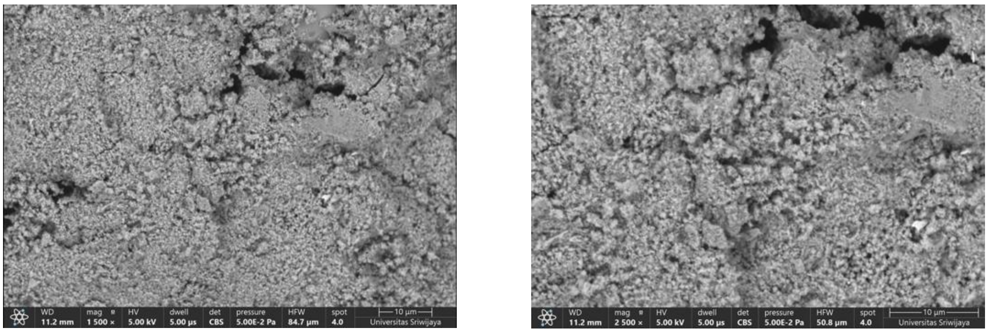
Element	% Atomic	% Weight
C	3.8	2.3
O	57.3	45.2
Al	11.3	15.1
Si	18.8	26.1
Na	8.3	9.4
As	0.5	1.9

In Figure 3, the characterization of the zeolite sample after hydrothermal activation for 12 hours shows further changes in the structural transformation and chemical composition compared to the previous 6-hour activation. Morphologically, at 1500× magnification, the sample surface is now dominated by aggregates of fine particles, giving it a rougher and more uniform texture. The macroporosity observed at 6 hours is no longer dominant; instead, the porosity appears to be interparticle within the fine aggregates [8].

According to Table 3, the aluminum (Al) content increased significantly to 15.1 wt% from the previous 11.1 wt%, and the silicon (Si) content rose from 25.1 wt% to 26.1 wt%. Meanwhile, the sodium (Na) content decreased from 11.4 wt% to 9.4 wt%, indicating a shift in charge balancing or the formation of a more complex phase.

In Figure 4, the SEM analysis after 24 hours shows a morphology similar to that observed in Figure 3 (12-hour SEM analysis). Both samples are dominated by fine particles that create a comparable texture; however, the 24-hour sample exhibits more uniform particle sizes and the disappearance of coarse particles [9].

EDS analysis revealed significant changes, with the aluminum (Al) content decreasing to 5 wt% from 15.1 wt% in the 12-hour sample, and the silicon (Si) content dropping to 20.1 wt% from 26.1 wt%. The decrease in Al and Si content indicates the occurrence of a dealumination process, where Al atoms are removed from the aluminosilicate framework. This process commonly occurs in zeolites under hydrothermal conditions, particularly in zeolites with low or intermediate Si/Al ratios, which are less stable and prone to Al bond breakage when exposed to high-temperature water [10].



(a) (b)

Figure 4**.** SEM analysis results of synthetic zeolite activated for 24 hours at magnifications of (a) 1500× and (b) 2500×.

Table 4. SEM-EDS results of synthetic zeolite activated for 24 hours.

Element	% Atomic	% Weight
C	4.8	2.2
O	60.4	37.5
Al	4.8	5.0
Si	18.5	20.1
Na	3.3	3.0
As	-	-

XRF Analysis Results

X-ray Fluorescence (XRF) analysis was used to determine the elemental composition of the zeolite material, identifying key elements such as Si, Al, Na, K, and Ca, as well as to determine the SiO_2/Al_2O_3 ratio, which influences the physical and chemical properties of the zeolite.

Table 5. XRF analysis results of zeolite sample.

Compound	Concentration (%)
Al_2O_3	14.61
SiO_2	54.51
Fe_2O_3	6.74
K_2O	3.48
Na_2O	3.37
CaO	2.08
MgO	0.98
TiO_2	0.48

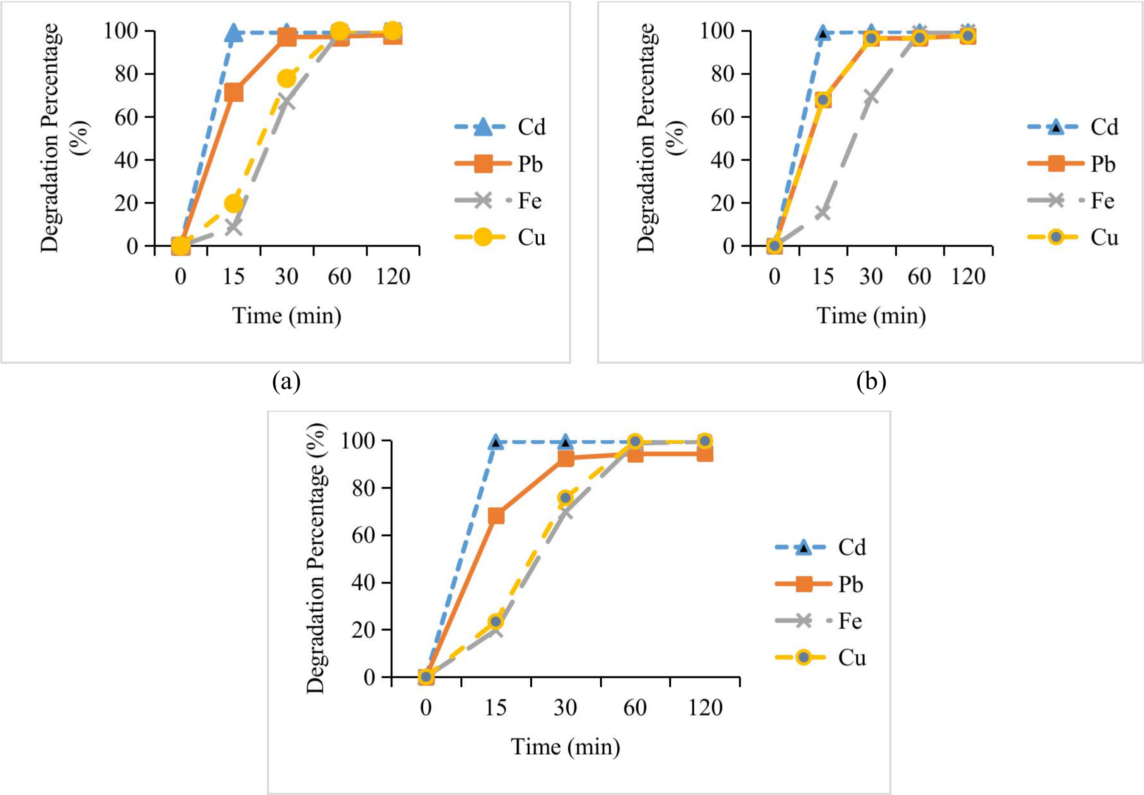
Based on the XRF analysis results of the synthesized zeolite, the highest composition was SiO_2 at 54.51% and Al_2O_3 at 14.61%. From this data, the SiO_2/Al_2O_3 ratio was calculated to be 3.73. The SiO_2/Al_2O_3 ratio plays an important role in determining the framework of zeolite, which is composed of aluminosilicate compounds. In the zeolite structure, each aluminum (Al) atom that replaces a silicon (Si) atom in the tetrahedral framework generates a negative charge. This negative charge must be balanced by positively charged cations, such as sodium ions, present within the zeolite structure. A higher aluminum content leads to a lower SiO_2/Al_2O_3 ratio and requires more cations to maintain charge stability, which significantly impacts the ion-exchange capacity of the zeolite. Based on the obtained ratio of 3.73, the synthesized zeolite can be categorized as a zeolite with an intermediate Si/Al ratio. This ratio influences the zeolite's affinity for certain molecules in adsorption applications and its selectivity in catalytic applications [11].

The XRF analysis also detected the presence of alkali metal oxides, such as Na_2O at 3.37%, which originates from the use of alkali activators during the hydrothermal reaction process. These activators play a role in dissolving silica and alumina from the coal bottom ash, aiming to form the porous structure of the zeolite. Additionally, the presence of alkali and alkaline earth metals in the zeolite serves as ion exchangers with heavy metals in solution, which is one of the primary adsorption mechanisms in zeolites. The high content of Na_2O and K_2O indicates that the analyzed zeolite has a good ion-exchange capacity.

The Fe_2O_3 content, recorded at 6.74%, influences the adsorption ability of zeolite by providing additional sites for heavy metal interactions, particularly through surface complexation or specific adsorption, and can modify the surface properties of the zeolite to enhance affinity towards certain heavy metals. However, a high iron content can negatively affect the crystallinity and thermal stability of the zeolite, potentially hindering crystal growth if accumulated as impurities [11].

Effect of Degradation Time and pH on the Reduction of Heavy Metals Cd, Pb, Fe, and Cu

The reduction of heavy metals is illustrated in Figure 5, showing the effect of contact time on the percentage of heavy metal removal for Cd, Pb, Fe, and Cu at pH 5. Contact time plays a crucial role in the adsorption process for all four metals, especially for Pb and Cd. For Cd, after 15 minutes, a removal efficiency of 99.03% was achieved, followed by Pb with 71.35%. Pb removal continued to increase, reaching 96.95% at 30 minutes. In contrast, Fe and Cu showed slower adsorption rates, with only 8.64% and 19.63% removal, respectively, at 15 minutes.



(a)

(b)

(c)

Figure 5. Effect of adsorption on heavy metal degradation at (a) pH 5, (b) pH 7, and (c) pH 9.

The effect of pH on heavy metal removal during the adsorption process plays an important role, as pH influences the adsorbent's surface charge, metal solubility, and ionic competition in the solution [9]. Cadmium (Cd) adsorption in this study achieved a removal percentage of up to 99.47% at pH 7 with a contact time of 120 minutes, or at pH 9 with a contact time of 30 minutes.

These results are highly comparable to the research by [12], who utilized zeolite from coal bottom ash modified with dithizone, achieving a Cd adsorption efficiency of 98% at pH 6. The optimal reduction of Pb was achieved at pH 5 with 97.84%, followed by 97.49% at pH 7 and 94.52% at pH 9. This performance is superior to that reported by [13], who used silica gel from coal bottom ash and achieved an efficiency of 70.84% for Pb removal, and by [10], [14], who used zeolite from sugarcane bagasse and reported an efficiency of 70.19% for Pb removal. This study indicates that Pb adsorption is more favorable under acidic conditions at pH 5. The observed effectiveness across the pH range of 5 to 7 also demonstrated a high adsorption capacity for Cd under acidic, neutral, and slightly basic conditions. The ability of the zeolite to perform effectively across a wide pH range offers significant practical advantages for wastewater treatment, where pH fluctuations are common. Adjusting pH before treatment could otherwise increase operational costs.

For Fe, the highest removal efficiency was achieved at pH 5 with 99.73%, followed closely by 99.61% at pH 7 and 99.46% at pH 9. The finding that pH 5 is a highly effective condition for Fe removal using zeolite is also supported by the research of [15], who reported that pH 5 is the optimum pH for Fe removal using zeolite, with an efficiency reaching 98.86%. Ismail et al. [15] also concluded that the optimal Fe removal capacity is achieved under acidic conditions. Although Fe ion adsorption occurred more slowly compared to Cd and Pb, Fe exhibited the highest overall heavy metal removal among the three. As shown in Figure 10, Fe adsorption at 15 and 30 minutes was more effective at higher pH values. The optimal reduction of Cu was achieved at pH 5 with 99.91%, followed by 99.74% at pH 9 and 97.49% at neutral pH 7. At 15 minutes of contact time, Cu removal was higher under neutral pH 7 conditions, reaching 67.77%, compared to only 19.63% at acidic pH 5 and 23.36% at basic pH 9. After 30 minutes of contact time, the removal of Cu remained most optimal at neutral pH 7 with 96.32%, while at acidic pH 5 it was 77.72%, and at basic pH 9 it was only 75.67%. These results indicate that the most favorable condition for Cu heavy metal adsorption is at neutral pH 7.

CONCLUSION

Zeolite synthesized from coal bottom ash with the optimal SiO_2/Al_2O_3 ratio of 3.73 through a hydrothermal process for 12 hours demonstrated effective characteristics as an adsorbent. The optimum conditions for heavy metal degradation occurred within a pH range of 5–9, where longer contact times between the adsorbent and heavy metals led to greater reductions in metal concentrations until reaching equilibrium. Cadmium (Cd) exhibited the highest degradation percentage of 99.47% at pH 7 and 99.9% at pH 9 within 30 minutes. Lead (Pb) achieved a degradation percentage of 97.34% at pH 5 within 120 minutes, iron (Fe) achieved 97.84% at pH 5 within 120 minutes, and copper (Cu) achieved 99.91% at pH 5 within 120 minutes of contact time.

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