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Removal of Pb^{2+} Ions Using Rice Husk Ash and Metakaolin Based Geopolymer

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Abstract

Heavy metal contamination in aquatic environments is a serious environmental issue due to its toxicity, persistence, and tendency to accumulate within living organisms. One promising approach to address this problem is adsorption using aluminosilicate-based geopolymers. This study aimed to evaluate the ability of rice husk ash and metakaolin-based geopolymers to remove Pb^{2+} ions and to investigate the effects of Si/Al molar ratio and NaOH concentration on geopolymer characteristics, removal efficiency, and adsorption capacity. Geopolymers were synthesized using rice husk ash as a silica source and metakaolin as an alumina source with varying Si/Al molar ratios and NaOH concentrations. The synthesized geopolymers were characterized using SEM-EDX and FTIR, while their adsorption performance was evaluated based on Pb^{2+} removal efficiency and adsorption capacity from $Pb(NO_3)_2$ solution. The results demonstrated that the geopolymer was successfully synthesized and showed potential as an effective adsorbent for Pb^{2+} removal. SEM-EDX and FTIR analyses confirmed the formation of an aluminosilicate structure and indicated that Pb^{2+} adsorption involved ion-exchange mechanisms and interactions with active functional groups on the geopolymer surface. Variations in the Si/Al molar ratio and NaOH concentration significantly affected the characteristics and adsorption performance of the geopolymer. The optimum adsorption condition was achieved at a Si/Al molar ratio of 4.2 and a NaOH concentration of 10 M, resulting in an adsorption capacity of 7.94 mg/g and a removal efficiency of 99.83%.
Keywords: *geopolymer, rice husk ash, metakaolin, adsorption, Pb^{2+}.*

Abstrak

Pencemaran logam berat di perairan menjadi salah satu permasalahan lingkungan yang serius karena bersifat toksik, sulit terdegradasi, dan dapat terakumulasi dalam rantai makanan. Salah satu metode yang berpotensi untuk mengatasi permasalahan tersebut adalah adsorpsi menggunakan geopolimer berbasis aluminosilikat. Penelitian ini bertujuan untuk mengetahui kemampuan geopolimer berbasis abu sekam padi dan metakaolin dalam removal ion Pb^{2+} serta mengkaji pengaruh variasi rasio mol Si/Al dan konsentrasi larutan NaOH terhadap karakteristik geopolimer, efisiensi removal, dan kapasitas adsorpsinya. Geopolimer disintesis menggunakan abu sekam padi sebagai sumber silika dan metakaolin sebagai sumber alumina dengan variasi rasio mol Si/Al dan konsentrasi NaOH. Karakterisasi dilakukan menggunakan SEM-EDX dan FTIR, sedangkan kinerja adsorpsi dievaluasi berdasarkan efisiensi removal dan kapasitas adsorpsi ion Pb^{2+} dari larutan $Pb(NO_3)_2$. Hasil penelitian menunjukkan bahwa geopolimer berhasil disintesis dan berpotensi sebagai adsorben yang efektif untuk removal ion Pb^{2+}. Analisis SEM-EDX dan FTIR mengonfirmasi terbentuknya struktur aluminosilikat serta menunjukkan bahwa adsorpsi ion Pb^{2+} pada geopolimer melibatkan mekanisme pertukaran ion dan interaksi dengan gugus aktif pada permukaan geopolimer. Variasi rasio mol Si/Al dan konsentrasi NaOH berpengaruh terhadap karakteristik dan kinerja adsorpsi geopolimer. Kondisi optimum diperoleh pada rasio mol Si/Al 4,2 dan konsentrasi NaOH 10 M dengan kapasitas adsorpsi sebesar 7,94 mg/g serta efisiensi removal mencapai 99,83%.
Kata Kunci: *geopolimer, abu sekam padi, metakaolin, adsorpsi, Pb^{2+}*

1. Introduction

Heavy metal contamination of water sources has become a serious environmental concern because of its toxicity, persistence, and bioaccumulation potential. Among various heavy metals, lead (Pb^{2+}) is one of the most hazardous pollutants due to its adverse effects on human health and aquatic ecosystems. Exposure to Pb^{2+} can cause neurological disorders, kidney damage, and developmental impairments. Therefore, the removal of Pb^{2+} from wastewater is essential to protect environmental and public health. Adsorption is widely recognized as an effective method for heavy metal removal because of its simplicity, high efficiency, and relatively low operating cost. Among the various adsorbent materials, geopolymers have attracted considerable attention due to their porous aluminosilicate structure, chemical stability, and abundance of active adsorption sites. Geopolymers are synthesized through the alkali activation of silica and alumina rich precursors, producing a threedimensional network structure suitable for pollutant removal.

Rice husk ash and metakaolin are promising precursor materials for geopolymer synthesis. Rice husk ash is an abundant agricultural by product generated from rice processing activities and is rich in silica, whereas metakaolin serves as an important source of alumina. XRF analysis showed that the rice husk ash used in this study contained 89.7 wt.% SiO_2, while the metakaolin contained 56.1 wt.% SiO_2 and 32.8 wt.% Al_2O_3. The combination of these materials provides the silica and alumina required for the formation of aluminosilicate networks during geopolymerization. In addition, the utilization of rice husk ash supports the valorization of agricultural waste into value added materials.

Several studies have demonstrated the potential of geopolymers derived from rice husk ash and metakaolin for environmental applications. A geopolymer synthesized from rice husk ash and metakaolin achieved an adsorption capacity of 276.9 mg g^{-1} for dye removal [1]. In another study, a rice husk ash based geopolymer exhibited a chromium removal efficiency of 54.42% [2]. These findings indicate that geopolymers produced from rice husk ash and metakaolin possess significant potential as adsorbent materials for pollutant removal. Despite these promising results, limited information is available regarding the combined effects of Si/Al molar ratio and NaOH concentration on the characteristics and adsorption performance of rice husk ash and metakaolin based geopolymers toward Pb^{2+} ions. These parameters play a crucial role in controlling the geopolymerization process, influencing the formation of aluminosilicate networks, surface morphology, and the availability of active adsorption sites. Understanding their effects is therefore important for optimizing geopolymer performance in Pb^{2+} removal applications.

Therefore, this study investigates the removal of Pb^{2+} ions using a rice husk ash and metakaolin based geopolymer. The effects of Si/Al molar ratio and NaOH concentration on geopolymer characteristics, Pb^{2+} removal efficiency, and adsorption capacity were evaluated through material characterization and adsorption experiments. The results are expected to contribute to the development of sustainable geopolymer adsorbents and provide further insight into the utilization of agricultural waste derived materials for wastewater treatment.

2. Material and Methods

Rice husk ash (RHA) was obtained from Hachita Agro, Gresik, Indonesia. Metakaolin, sodium hydroxide (NaOH), lead nitrate [$Pb(NO_3)_2$], and distilled water were purchased from PT Niraku Jaya Abadi, Surabaya, Indonesia. The main equipment used in this study included a magnetic stirrer, analytical balance, oven, pH meter, and laboratory glassware. Material characterization was performed using X-ray Fluorescence (XRF), Fourier Transform Infrared Spectroscopy (FTIR), and Scanning Electron Microscopy Energy Dispersive X-ray Spectroscopy (SEM EDX). The concentration of Pb^{2+} ions was determined using Atomic Absorption Spectroscopy (AAS).

This study investigated the effects of Si/Al molar ratio and NaOH concentration on the characteristics and adsorption performance of RHA and metakaolin based geopolymers. The Si/Al molar ratio was varied at 3.0, 3.3, 3.6, 3.9, and 4.2, while the NaOH concentration was varied at 8, 9, 10, 11, and 12 M. The adsorption performance was evaluated based on Pb^{2+} removal efficiency and adsorption capacity.

The experimental procedure began with the preparation of raw materials. The particle size of RHA was reduced using a mortar and pestle and subsequently sieved through a 200 mesh screen [3]. The chemical compositions of RHA and metakaolin were determined using XRF analysis to identify the SiO_2 and Al_2O_3 contents required for geopolymer synthesis.

Sodium hydroxide solutions were prepared and allowed to cool and stabilize for 24 h at room temperature in sealed glass containers. Geopolymer synthesis was carried out by mixing 30 mL of NaOH solution with metakaolin and rice husk ash according to the predetermined formulation. The mixture was stirred at 150 rpm for approximately 30 min until a homogeneous paste was obtained. The resulting paste was cast into silicone molds and left at room temperature for 24 h. The hardened specimens were then cured at 60 °C for 24 h and subsequently aged at room temperature for 7 days.

After curing, the geopolymer samples were washed repeatedly with distilled water until the washing solution reached a neutral pH (≈7). The samples were then dried at 100 °C for 4 h, ground using a mortar and pestle, and sieved through a 200 mesh screen to obtain geopolymer adsorbent powder [4]. The synthesized adsorbents were characterized using FTIR to identify functional groups and SEM-EDX to evaluate surface morphology and elemental composition.

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Adsorption experiments were carried out by mixing 3.5 g of geopolymer adsorbent with 50 mL of Pb^{2+} solution prepared from $Pb(NO_3)_2$ at an initial concentration of 200 mg L^{-1}. The mixture was stirred at 150 rpm for 120 min [5]. After adsorption, the mixture was filtered to separate the filtrate from the adsorbent. The residual Pb^{2+} concentration in the filtrate was analyzed using AAS. The adsorption performance was evaluated based on Pb^{2+} removal efficiency and adsorption capacity. Furthermore, the spent adsorbents were characterized using FTIR and SEM-EDX analyses to investigate the interaction between Pb^{2+} ions and the geopolymer structure after the adsorption process.

3. Results and Discussion

3.1 Raw Material Characterization

XRF analysis was performed to determine the chemical composition of rice husk ash (RHA) and metakaolin as geopolymer precursors. The results revealed that RHA contained 89.7% SiO_2, whereas metakaolin contained 56.1% SiO_2 and 32.8% Al_2O_3. The high silica content of RHA demonstrates its suitability as a silica-rich precursor, while metakaolin serves as the primary source of alumina required for geopolymer formation. The combination of RHA and metakaolin provides sufficient reactive silica and alumina species to support the geopolymerization process. The silica supplied by RHA contributes to increasing the Si/Al molar ratio, whereas alumina from metakaolin promotes the formation of the aluminosilicate framework. According to [6], the Si/Al ratio plays a critical role in determining the structural characteristics and performance of geopolymer materials.

3.2 SEM–EDX Characterization of Geopolymer Before and After Pb^{2+} Adsorption

The surface morphology of the synthesized geopolymer before and after Pb^{2+} adsorption is presented in **Figure 1**.

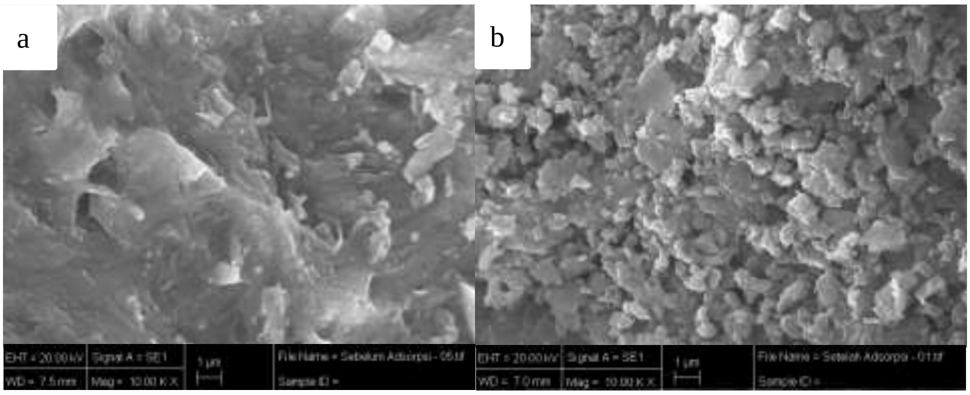

Figure 1. SEM micrographs of geopolymer: (a) before Pb^{2+} adsorption and (b) after Pb^{2+} adsorption

Table 1. Elemental Composition of Geopolymer Before and After Pb^{2+} Adsorption Determined by EDX

Element	Geopolymer before Pb^{2+} adsorption (%wt)	Geopolymer after Pb^{2+} adsorption (%wt)
O	61,20	49,44
Pb	0	19,24
Na	13,83	4,08
C	11,82	12,23
Si	9,31	10,10
Al	3,04	4,20
K	0,80	0,71

SEM micrographs of the geopolymer before and after Pb^{2+} adsorption are presented in **Figure 1**. The morphology was observed at a magnification corresponding to a scale bar of 1 µm. Prior to adsorption, the geopolymer exhibited an irregular surface morphology with visible interparticle voids and cavities, indicating that the geopolymer matrix was not completely compact. These voids may facilitate the diffusion of Pb^{2+} ions toward the active sites located on the geopolymer surface [18].

After adsorption, the geopolymer surface became noticeably rougher and more heterogeneous, accompanied by particle agglomeration and the appearance of deposits on the surface. In addition, several voids that were visible before adsorption appeared partially covered after interaction with Pb^{2+} ions. These morphological changes indicate the attachment of Pb^{2+} ions onto the geopolymer surface and suggest the

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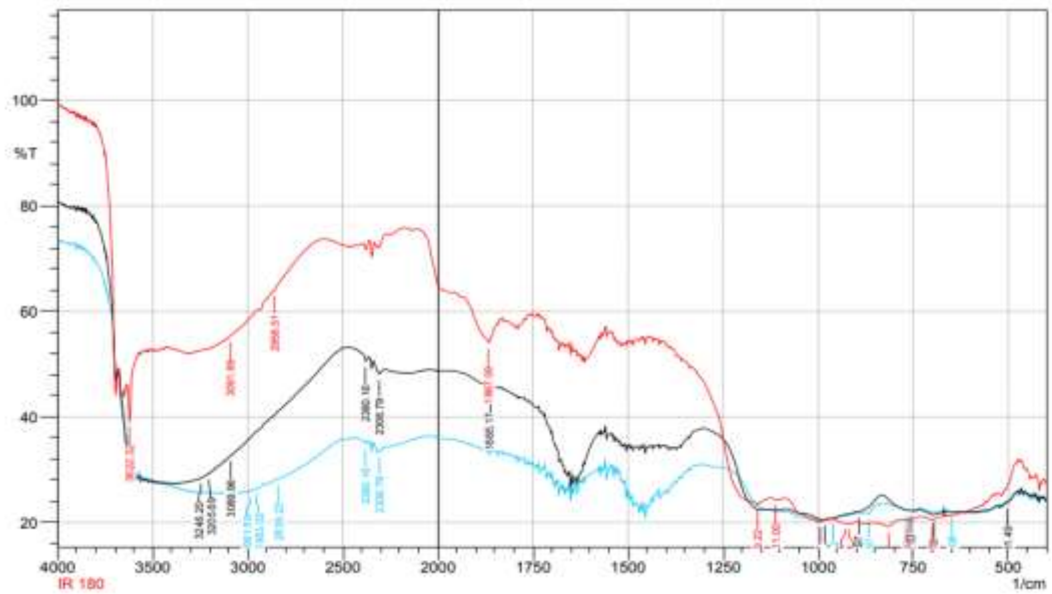
occurrence of adsorption processes within the geopolymer matrix. Similar surface modifications have been observed during Pb^{2+} adsorption by aluminosilicate-based geopolymers [7]. The SEM observations were further supported by EDX analysis, which confirmed the presence of Pb on the geopolymer surface after adsorption.

The EDX results further confirmed the adsorption of Pb^{2+} ions shown in Table 1. Pb was not detected in the fresh geopolymer but appeared at 19.24 wt.% after adsorption. Simultaneously, the Na content decreased from 13.83 wt.% to 4.08 wt.%. The appearance of Pb accompanied by a significant reduction in Na content indicates that Pb^{2+} adsorption occurred through an ion-exchange mechanism, in which Pb^{2+} ions replaced Na^{+} ions within the geopolymer framework [8].

Ion exchange has been recognized as one of the dominant mechanisms governing cation adsorption by geopolymers. The geopolymer structure consists of a three dimensional aluminosilicate framework that carries a net negative charge due to the substitution of Si^{4+} by Al^{3+} in the tetrahedral network. his charge imbalance is compensated by alkali cations, such as Na^{+}, that are bound to the negatively charged sites within the aluminosilicate framework of the geopolymer. Because these charge balancing cations are relatively mobile, they can be exchanged with metal ions present in the surrounding solution. Heavy metal ions with higher charge density and stronger electrostatic interactions generally exhibit a greater affinity for negatively charged aluminosilicate sites than alkali cations. Consequently, Pb^{2+} ions can replace Na^{+} ions and become more stably associated with the geopolymer framework through electrostatic interactions with negatively charged sites [7].

3.3 FTIR Analysis of Geopolymer

The FTIR spectra of metakaolin, geopolymer, and Pb-loaded geopolymer are shown in **Figure 2**. The characteristic Si–O–T (T = Si or Al) stretching bands initially observed at 995.27 and 1111 cm^{-1} shifted to 958.62 and 993.34 cm^{-1} after geopolymerization. This shift confirms the formation of a new aluminosilicate network resulting from the geopolymerization reaction [9]. In addition, the disappearance of the Al–O vibration band at 815.89 cm^{-1} after geopolymer synthesis suggests the dissolution of metakaolin and its incorporation into the geopolymer matrix, indicating the successful formation of the geopolymer structure [10].



: metakaolin (M)
: geopolymer setelah adsorpsi (G-Pb)
: geopolymer sebelum adsorpsi (G)

Figure 2. FTIR spectra of metakaolin, geopolymer, and Pb-loaded geopolymer

Following Pb^{2+} adsorption, several absorption bands shifted, particularly the Si–O–T band, which moved from 958.62 cm^{-1} to 979.84 cm^{-1}. The observed shifts indicate the involvement of oxygen-containing functional groups in Pb^{2+} adsorption [11].

3.4 Effect of Si/Al Molar Ratio and NaOH Concentration on Filtrate pH

The pH of the filtrate after Pb^{2+} adsorption ranged from 9.81 to 10.86, indicating that the adsorption system remained under alkaline conditions. **Figure 3** shows that the filtrate pH was influenced by both the Si/Al molar ratio and NaOH concentration. At 8 M NaOH, increasing the Si/Al ratio from 3.0 to 4.2 decreased the filtrate pH from 10.25 to 9.81. This behavior suggests that a higher Si/Al ratio promoted the interaction between Pb^{2+} ions and active sites on the geopolymer surface, resulting in the consumption of OH^- ions during adsorption processes such as ion exchange and surface complexation [8].

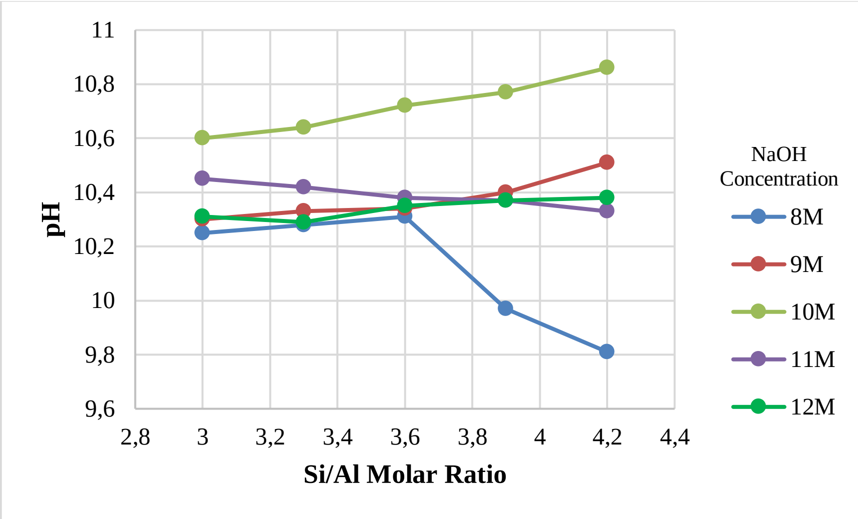


Figure 3. Interactive effect of Si/Al molar ratio and NaOH concentration on adsorption filtrate pH

At higher NaOH concentrations (9–12 M), the filtrate pH generally remained above 10.3 and showed only slight variations with increasing Si/Al ratio. The highest pH values (10.60–10.86) were obtained at 10 M NaOH, indicating sufficient alkalinity to support geopolymer stability and Pb^{2+} adsorption. However, further increases in NaOH concentration did not significantly increase the filtrate pH, suggesting that part of the OH^- ions participated in chemical reactions within the adsorption system rather than remaining as free ions in solution [12,13].

3.5 Adsorption Performance of Geopolymer

3.5.1 Effect of Si/Al Molar Ratio

The effect of the Si/Al molar ratio on Pb^{2+} adsorption capacity and removal efficiency is presented in **Figure 4**. Increasing the Si/Al molar ratio improved both adsorption capacity and removal efficiency, with the highest performance obtained at a ratio of 4.2.

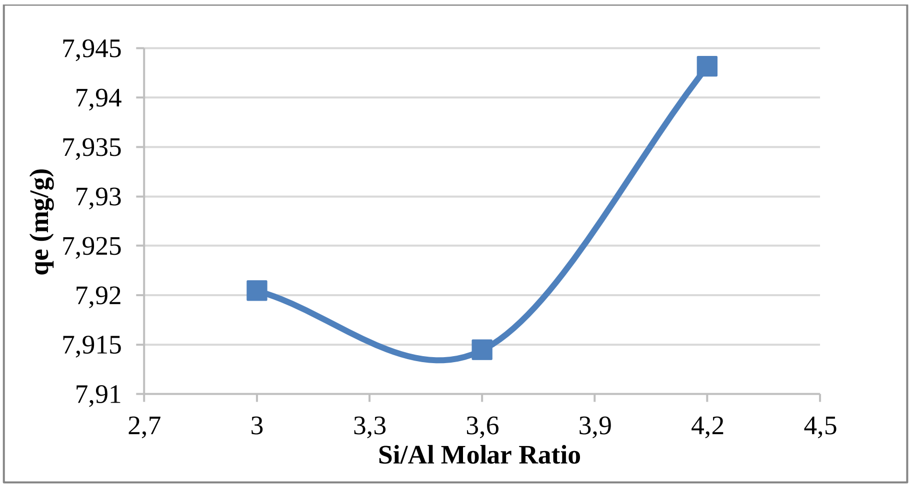


Figure 4. Effect of Si/Al molar ratio on Pb^{2+} adsorption capacity

The enhanced adsorption performance can be attributed to the development of a more extensive aluminosilicate network, which provides a greater number of active sites for Pb^{2+} binding. Moreover, higher

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silica availability promotes the formation of a more stable geopolymer structure and improves pore development, thereby enhancing the adsorption properties of the material [14].

3.5.2 Effect of NaOH Concentration

Figure 5 illustrates the effect of NaOH concentration on the adsorption performance of the synthesized geopolymers. The adsorption capacity increased with increasing NaOH concentration from 8 M to 10 M and subsequently decreased at higher concentrations. The optimum performance was achieved at 10 M NaOH, yielding an adsorption capacity of 7.94 mg g^{-1} and a Pb^{2+} removal efficiency of 99.83%.

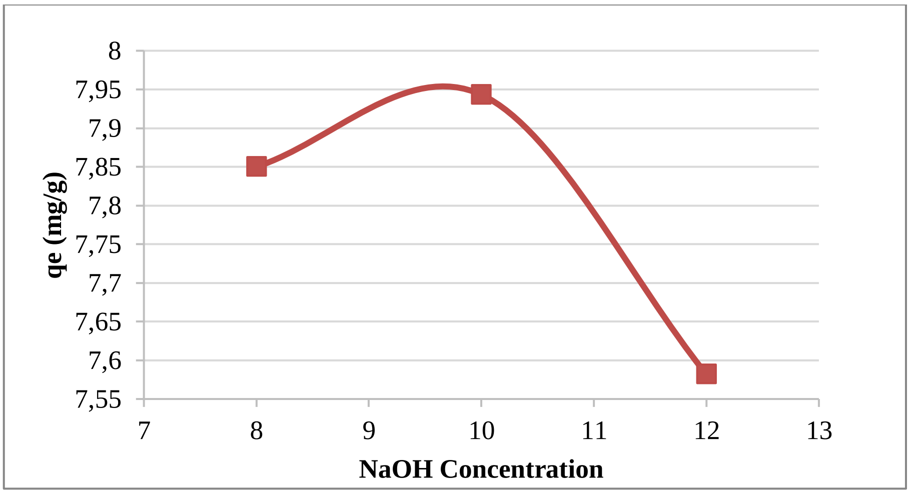

Figure 5. Effect of NaOH concentration on Pb^{2+} adsorption capacity

The improved adsorption performance at 10 M NaOH can be attributed to the enhanced dissolution of silica and alumina species, which promotes the formation of N–A–S–H gel as the primary binding phase in the geopolymer matrix. This process contributes to the development of a more homogeneous structure with improved pore accessibility and adsorption characteristics [15,16].

At higher NaOH concentrations, adsorption performance decreased, likely due to the formation of a denser geopolymer matrix with reduced pore accessibility. Excessive alkali concentrations may accelerate geopolymerization, resulting in a more compact structure that limits the diffusion of Pb^{2+} ions and reduces adsorption efficiency [17].

3.6 Adsorption Mechanism

The adsorption mechanism of Pb^{2+} by the synthesized geopolymer was evaluated based on the SEM–EDX and FTIR results. The significant decrease in Na content after adsorption, accompanied by the appearance of Pb on the geopolymer surface, suggests that ion exchange played a major role in the adsorption process. In this mechanism, Pb^{2+} ions replace Na^{+} ions that act as charge-balancing cations within the negatively charged aluminosilicate framework. Furthermore, the shifts observed in the FTIR absorption bands indicate that Pb^{2+} ions interacted with oxygen-containing functional groups, including Si–O–T, Si–O, and Si–O–Al groups. These interactions contributed to the immobilization of Pb^{2+} ions on the geopolymer surface. Therefore, Pb^{2+} removal by the synthesized geopolymer can be primarily attributed to ion exchange, supported by interactions between Pb^{2+} ions and functional groups within the aluminosilicate framework. The combined contribution of these mechanisms resulted in the high adsorption capacity and removal efficiency observed in this study.

4. Conclusion

A rice husk ash and metakaolin based geopolymer was successfully synthesized and demonstrated excellent potential for Pb^{2+} removal from aqueous solutions. SEM, EDX, and FTIR analyses confirmed the formation of an aluminosilicate geopolymer structure and revealed changes in surface morphology and chemical characteristics after adsorption. The detection of Pb on the geopolymer surface, accompanied by a reduction in Na content, suggested that ion exchange contributed to the adsorption process.

The Si/Al molar ratio and NaOH concentration significantly influenced the adsorption performance of the geopolymer. Increasing the Si/Al molar ratio enhanced the development of the aluminosilicate network and improved the availability of adsorption sites. Meanwhile, NaOH concentration affected the geopolymerization process and the formation of N A S H gel, thereby influencing the resulting adsorption capacity. The optimum adsorption performance was achieved at a Si/Al molar ratio of 4.2 and a NaOH concentration of 10 M, resulting in an adsorption capacity of 7.94 mg g^{-1} and a Pb^{2+} removal efficiency of 99.83%. These findings demonstrate that rice husk ash and metakaolin based geopolymers are promising sustainable adsorbents for the removal of lead from contaminated water.

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