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Cobalt Removal Mechanism using Metakaolin-based Geopolymer Sorbent

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1. Introduction

Large quantities of radioactive wastewater are produced from nuclear power plants that contain large amounts of radionuclides such as ^{60}Co. ^{60}Co is considered one of the major fission products in nuclear reactors due to its high energy gamma emission and radiological significance [1]. Unlike stable cobalt, ^{60}Co is a highly radioactive isotope that poses serious radiological hazards, potentially leading to hair loss and diseases of the blood system and even death [2].

Sorption is one of the most commonly used methods to treat ^{60}Co-containing effluents because of its high efficiency, simplicity of operation, low cost, environmental friendliness, and reusability of sorbents. Most conventional sorbents have been investigated in powder form, leading to increased sludge generation, difficult recovery, and secondary pollution [2]. Therefore, the development of structurally stable and recyclable sorbents for Co^{2+} removal remains essential. Accordingly, bead or pellet type sorbents have been developed to overcome the limitations associated with powder based materials, particularly in terms of separation and recovery.

Geopolymer, typically used for solidification, can have superior chemical durability characteristics which are attributed to their silicate polymerization. Since geopolymers have a three-dimensional framework structure of oxygen-linked silicon and aluminium, the negative charge associated with the tetrahedral Al sites is typically balanced by alkali cations, including Na^+ or K^+ from the activator. A geopolymer can also be regarded as a disordered pseudo-zeolite. There are active sites on the surface of geopolymer owing to the presence of metal oxide groups and thus, they have increased potential in sorbing heavy metal ions. The types of precursors, water/solid ratio, Si/Al ratio, alkalinity of alkali activation solution, and the curing temperature affect the geopolymerization and could influence the sorption property [3].

In this study, geopolymer was investigated as a sorbent to remove the Co^{2+} ion from aqueous solutions. The optimum geopolymer formulation was determined, and the sorption efficiency of Co^{2+} was evaluated. In addition, various characterization analyses, including XRD, SEM-EDS, and FT-IR, were performed to investigate the Co^{2+} removal mechanism.

2. Materials and methods

2.1 Synthesis of geopolymer sorbent

Geopolymer composition with molar ratios of Si/Al = 1.5, Al/Al = 1, Na/Al = 1 and H_2O/Al = 5.0 was chosen. For obtaining this mixture, NaOH pellets were dissolved in deionized water and sodium silicate solution by stirrer plate. After dissolving of NaOH pellets, metakaolin was added into solution and mixed at 1800 rpm for 4 min and deformed at 2100 rpm for 1 min by thinky mixer. The geopolymer slurry was poured into a mold with an inner diameter of 6 mm and a height of 5 mm. After curing at room temperature for 7 days, the geopolymer were demolded.

2.2 Characterization

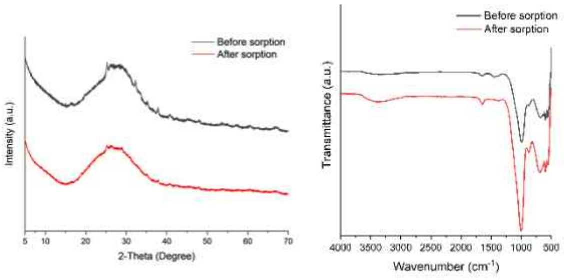
The morphology of geopolymer was observed by scanning electron microscope (SEM) and the energy dispersive spectrometer (EDS) was used to analyze the elements composition of samples. The surface charges of geopolymer were determined using a zeta potential analyzer. The X-ray diffractometer (XRD) was employed to analyze the crystalline phase of geopolymer. Fourier transform-infrared spectrum (FT-IR) were utilized to analyze the chemical bond of the geopolymer.

2.3 Sorption experiments

Non-radioactive Co^{2+} was used as a chemical surrogate for ^{60}Co to avoid radiological hazards and secondary radioactive waste, as stable cobalt isotopes exhibit similar aqueous chemical behavior. Stock solutions of Co^{2+} at concentration of 600 ppm were prepared by dissolving appropriate amounts of $Co(NO_3)_2 \cdot 6H_2O$ in deionized water. The prepared stock solution was directly used as the initial Co solution for the sorption experiments. The sorption experiments were performed at a solid-to-solution ratio of 1 g/L using an initial cobalt concentration of 600 ppm. The mixtures were shaken with 200 rpm at room temperature for 3 and 7 days, respectively. After sorption, the mixtures were separated using a 0.45 μm syringe filter, and the concentration of Cobalt in the supernatants were measured using an inductively coupled plasma-Optical Emission Spectroscopy (ICP-OES).

3. Results and discussion

The XRD pattern of geopolymer is shown in Fig. 1(a). Before sorption, geopolymer is predominantly amorphous as indicated by a broad hump between 20° and 40° 2θ. After sorption, No new crystalline phases were detected, assuming that Co^{2+} was reacted without forming secondary crystalline precipitates.



(a) (b)

Fig. 1. (a) XRD patterns and (b) FT-IR spectra of the geopolymer before and after Co^{2+} sorption

FT-IR spectra of the geopolymer before and after sorption were obtained in the range of 4000-500 cm^{-1} as shown in Fig. 1(b). The broad band around 3400 cm^{-1} and the band near 1630 cm^{-1}, corresponding to surface -OH stretching and H-O-H bending vibrations, respectively, showed no significant changes after sorption. Likewise, the bending vibration region of Al-O-Si (800-500 cm^{-1}) was unchanged, assuming that the primary geopolymer framework was preserved. In contrast, a noticeable variation was observed in the region around 1000 cm^{-1}, assigned to the asymmetric stretching vibration of Si-O-T (T=Si or Al). This change suggests that Co^{2+} ions were interacted with surface sites associated with the aluminosilicate network [4].

Table 1. Sorption capacity and Zeta potential

t (day)	Sorption capacity (mmol/g)
3	0.2111
7	0.8417
	Zeta potential (mV)
0	-43.35
3	-5.86
7	-5.01

As shown in Table 1, the Co^{2+} sorption capacity at 7 days was higher than that at 3 days, indicating increased Co^{2+} uptake with longer contact time. The solution pH (initial pH = 5.20; pH at 3 d = 5.44; pH at 7 d = 4.61) remained nearly constant during sorption. Furthermore, the geopolymer exhibited a uniform green coloration across the outer surface, while the interior remained largely unchanged, assuming that the sorption process was mainly surface-controlled with limited inward diffusion. To support the above results, zeta potential measurements were conducted for each sampling time. Geopolymer inherently possesses negatively charged surface sites, which is consistent with the observed increase in zeta potential from -43.35 mV before sorption to approximately -5 mV after Co^{2+} sorption This change indicates that the initially negatively charged surface was significantly neutralized due to electrostatic attraction between the surface and positively charged Co^{2+} ions. Taken together, these results suggest that the spectral change near 1000 cm^{-1} was associated with surface level interactions rather that bulk structural transformaiton or extensive precipitation.

Fig. 2. Photograph of the geopolymer after Co^{2+} sorption

4. Conclusions

Overall, XRD patterns showed no formation of new crystalline phases after Co^{2+} sorption, indicating that the primary amorphous structure of the geopolymer was maintained. FT-IR analysis revealed a noticeable variation near 1000 cm^{-1}, while most other vibrational regions remained largely unchanged, suggesting interaction between Co^{2+} and surface sites associated with the aluminosilicate framework. The metakaolin-based geopolymer showed sorption capacities of 0.2111 and 0.8417 mmol/g at contact times of 3 and 7 days, respectively. The increase in sorption capacity with contact time indicates enhanced Co^{2+} uptake, which may be related to sorption processes occurring on the geopolymer surface. In addition, the uniform surface coloration with an unreacted interior further supports a surface-controlled sorption mechanism dominated by surface-level interactions. These observations suggest that sorption predominantly occurs at the external surface without significant inward diffusion, implying that regeneration through surface treatment could enable reuse of the geopolymer sorbent. Furthermore, future work will include kinetic and isotherm model analyses to provide a more understanding of the sorption mechanism.

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