Valorization of industrial waste red mud into geopolymer adsorbent for fluoride removal from aqueous solution

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ABSTRACT

Red mud, a waste by-product of the Tan Rai alumina plant, was utilized as a precursor for geopolymer synthesis and evaluated for fluoride removal from water. Geopolymers were prepared by alkali activation with NaOH (1%, 5%, and 10%) and subsequently calcined at 200–800 °C. Structural and surface characterizations were performed using XRD and FT-IR. Among the tested conditions, the sample activated with 1% NaOH and calcined at 200 °C exhibited the best performance, achieving a maximum fluoride adsorption capacity of 48.31 mg/g with an equilibrium time of 4 hours, and was effective at pH ≤ 8.4. Adsorption data were best fitted to the Freundlich isotherm model compared with Langmuir and Dubinin–Radushkevich models, indicating multilayer adsorption on a heterogeneous surface. These findings highlight the potential of red mud-based geopolymer as a cost-effective adsorbent for efficient fluoride removal from contaminated water.

Keywords: Geopolymer; Red mud; Fluoride; Adsorption.

1. INTRODUCTION

Fluorine in water predominantly occurs in the form of fluoride ions. At appropriate concentrations, fluoride plays a beneficial role by protecting tooth enamel and reducing the risk of dental caries; however, excessive intake can cause adverse effects on both dental and skeletal health [1]. Fluoride contamination originates from both natural and anthropogenic sources. Natural contributions include the weathering of fluoride-bearing minerals and volcanic emissions, while anthropogenic sources are primarily linked to industrial activities such as steel making, aluminum production, ceramics manufacturing, and phosphate fertilizer application. In Vietnam, fluoride concentrations exceeding the permissible limit have been reported in several provinces, including Phu Yen, Quang Nam, and Thai Binh. Notably, in Khanh Hoa, fluoride concentrations in groundwater wells range from 3 to 14 mg/L, far surpassing the safe threshold [2]. Given these health and environmental concerns, the development of effective and low-cost methods for fluoride removal from water has become an urgent priority.

Among the available methods for removing excess fluoride from water, adsorption stands out due to its operational simplicity and cost-effectiveness, making it particularly suitable for small-scale or decentralized water treatment systems. A wide range of fluoride adsorbents has been investigated, ranging from synthetic materials such as α-Fe_2O_3 and γ-Al_2O_3 powders [3], Al-MOFs [4], Zr-MOF-801 [5], layered double hydroxides (LDHs) [6], Ce–Al composite oxides supported on activated carbon [7], to waste-derived materials such as gypsum [8], alum sludge [9]. Red mud, a waste by-product of the Bayer process in alumina production, has been studied to use as an adsorbent for removing fluouride in water and showed relatively high adsorption ability [10, 11]. However, the direct use of red mud as an adsorbent is still limited by poor mechanical properties, as it tends to disintegrate, partially dissolve, and release a reddish color associated with iron oxides.

Geopolymers are inorganic polymers of a three-dimensional network structure composed of $[SiO_4]$ and $[AlO_4]$ tetrahedra sharing an oxygen atom as a linking bridge, typically formed through the polymerization reaction between alkaline solutions (NaOH, KOH, or sodium silicate) and aluminosilicate precursors [12]. The presence of alkalinity promotes the dissolution of Si^{4+} and Al^{3+} in alkaline media, which is the crucial step for geopolymer formation [13]. The main composition of red mud including aluminum oxide, iron oxide, and silicon oxide, together with its fine particle size and inherent alkalinity derived from residual NaOH in the Bayer process, makes it highly suitable for geopolymerization. The ability to transform red mud into geopolymer material could bring a way to overcome the shortcomings mentioned above of the use of red mud as an adsorbent and improve its structural stability through alkali activation [14, 15].

Based on these advantages, this study aims to synthesize geopolymer materials from red mud and evaluate their fluoride adsorption performance. The approach seeks to achieve dual benefits: valorizing industrial waste into low-cost adsorbents and effectively remediating fluoride contamination in aqueous environments.

2. EXPERIMENTS

2.1. Materials

Red mud was collected from the Tan Rai alumina plant of Lam Dong Aluminum Company Limited, Vietnam. The composition of red mud was SiO_2 (6.01%), Fe_2O_3 (48.29%), Al_2O_3 (16.16%), Na_2O (4.27%) and others [16]. Sodium hydroxide was analytical grade (≥ 98% purity, China). NaF (≥ 99% purity), HCl (35%), and NaCl (≥ 99% purity) were purchased from Aladdin Biochemical Technology Co., China.

2.2. Synthesis of red mud-based geopolymers

Red mud obtained from the Tan Rai alumina plant was dried at 105 °C for 24 h and then ground into a fine powder. Different amounts of alkaline activators could give rise the differences in the geopolymerization of adsorbent material. To study the effect of activator amount, the red mud was mixed with NaOH at different mass ratios of 1%, 5%, and 10%, followed by the addition of water at a solid-to-liquid ratio of 1:1. The resulting mixtures were allowed to stand for 24 h, then partially dried, and granulated using a sieve with a mesh size of 1.6 mm. The granulated materials were subsequently dried at 80 °C for 24 h. The geopolymer samples prepared with NaOH activation ratios of 1%, 5%, and 10% were denoted as RN1, RN5, and RN10, respectively. Thermal treatment can affect the structure and integrity of the geopolymers prepared, resulting in different adsorption abilities. To investigate the effect of calcination temperature, the sample with the optimal alkali activation ratio was further subjected to thermal treatment at 200 °C, 400 °C, 600 °C and 800 °C.

2.3. Evaluation of fluoride adsorption capacity

Batch adsorption experiments were conducted in polyethylene (PE) bottles with a solution to adsorbent ratio of 100 mL/g at room temperature. The adsorption performance of the materials was evaluated in terms of adsorption capacity and removal efficiency.

$$q = \frac{(C_0 - C_e)V}{m} \quad (1)$$

$$H = \frac{(C_0 - C_e)}{C_0} x100\% \quad (2)$$

where q is adsorption capacity (mg/g), H is removal efficiency (%), C_0 is the initial fluoride concentration before adsorption (mg/L), C_e is the equilibrium fluoride concentration after adsorption (mg/L), V is the solution volume (L) and m is the adsorption mass (g).

The factors influencing the fluoride adsorption capacity of red mud-based geopolymers include

NaOH activation ratio, calcination temperature, equilibrium adsorption time, pH effect, and fluoride concentration. The ranges of these parameters are summarized in Table 1.

Table 1. *Parameters for evaluating the fluoride adsorption on red mud-based geopolymers.*

Parameters	Values
Alkali activation ratio	1%, 5%, 10%
Calcined temperture	200 ºC, 400 ºC, 600 ºC and 800 ºC
Adsorption time	0.5 h ÷ 6 h
pH value	3 ÷ 10
Fluoride concentration	10 ÷ 900 ppm

The adsorption process of fluoride onto the optimized material was described using three adsorption isotherm models: Langmuir, Freundlich, and Dubinin–Radushkevich (D–R) [17].

The Langmuir model is commonly applied to solid–liquid systems to describe monolayer adsorption on a homogeneous surface of the adsorbent with finite identical sites. The Langmuir isotherm equation can be expressed in a linear form as:

$$\frac{C_e}{q_e} = \frac{1}{K_L q_{\max}} + \frac{1}{q_{\max}} C_e \tag{3}$$

where q_e is the equilibrium adsorption capacity (mg/g); q_{max} is the maximum theoretical adsorption capacity (mg/g); C_e is the equilibrium concentration of fluoride (mg/L); K_L is the Langmuir adsorption constant. The Freundlich model assumes adsorption occurs on heterogeneous surfaces, where the adsorption sites have different energies. The linear form of the Freundlich isotherm is represented by:

$$\ln q_e = \ln K_F + (1/n) \ln C_e \tag{4}$$

where q_e, C_e are the adsorption capacity and equilibrium concentration, respectively; K_F is the Freundlich constant related to adsorption capacity and intensity; 1/n is the heterogeneity factor indicating adsorption intensity and the distribution of active sites. The Dubinin–Radushkevich (D–R) model assumes pore-filling as the governing adsorption process and suggests a multilayer adsorption mechanism associated with van der Waals forces, typical of physical adsorption. The D–R isotherm is expressed as:

$$\ln q_e = \ln q_{\max} - \beta \varepsilon^2 \tag{5}$$

$$\varepsilon = RT \ln\left(1 + \frac{1}{C_e}\right) \tag{6}$$

$$\mathrm{E} = \frac{1}{\sqrt{2\beta}} \tag{7}$$

where q_e is the adsorption capacity, q_{max} is the theoretical maximum capacity, ε is Polanyi potential, β is Dubinin-Radushkevich constant, and E is the mean free energy of adsorption (J/mol).

2.4. Determination of the point of zero charge (pH_{pzc}) of the material

The point of zero charge (pH_{pzc}) is defined as the pH value at which the surface of the material is electrically neutral. The determination method is based on the assumption that protons (H^+) and hydroxyl groups (OH^-) are the charge-determining ions, and that the surface charge of the material depends on the solution pH. To determine the pH_{pzc}, 50 mL of 0.01 M NaCl solution was placed in Erlenmeyer flasks. The initial pH of the solution (pH_i) was adjusted in the range of 2–12 using 0.1 M HCl or 0.1 M NaOH. Subsequently, 0.5 g of the adsorbent was added to each flask, shaken for 24 h, allowed to settle, and then filtered through filter paper. The final pH values (pH_f) were measured, and the difference was calculated as $\Delta pH = pH_f - pH_i$. A plot of ΔpH versus pH_i was constructed, and the intersection of the curve with the x-axis corresponds to the pH_{pzc} of the material.

2.5. Analytical method

Fluoride concentration was determined using the 4500 F^- D: SPADNS method [18] in accordance with the Standard Methods for the Examination of Water and Wastewater of the American Public Health Association (APHA). The principle of the method is based on the reaction between fluoride and the zirconyl–SPADNS color complex. Fluoride ions react with zirconyl in the complex to form a colorless anionic complex (ZrF_6^{2-}). As the fluoride concentration increases, the color intensity of the solution decreases proportionally.

The fluoride concentration was quantified by measuring the decrease in absorbance of the color complex at a maximum wavelength of 570 nm, using a calibration curve to establish the linear relationship between absorbance and fluoride concentration. Each measurement was performed in triplicate, and the average value was reported.

The crystalline phases of the materials were characterized using an X-ray diffractometer (Bruker D8 Advance, Germany) equipped with a Cu anode X-ray tube (Cu-Kα, λ = 1.5406 Å). Fourier-transform infrared (FT-IR) spectra were recorded on an FT/IR-4600 spectrometer (Jasco, Japan).

3. RESULTS AND DISCUSSION

3.1. Structural characterization by X-ray diffraction (XRD)

Figure 1a presents the XRD patterns of the synthesized materials prepared with different NaOH activation ratios. The results show the characteristic peak of aluminosilicate geopolymer (N-A-S-H gel) at 2θ = 29.4° [19], which was observed in all alkali-activated samples from 1% to 10%, confirming the formation of the geopolymer network. Several crystalline phases originally present in red mud, such as gibbsite (2θ = 18.2°; 20.2°), goethite (2θ = 18.2°), and hematite (2θ = 33.1°; 53.8°), were also detected [20]. In general, the diffraction peaks exhibited relatively low intensity, indicating a low degree of crystallinity and a large proportion of amorphous or microcrystalline phases in the materials.

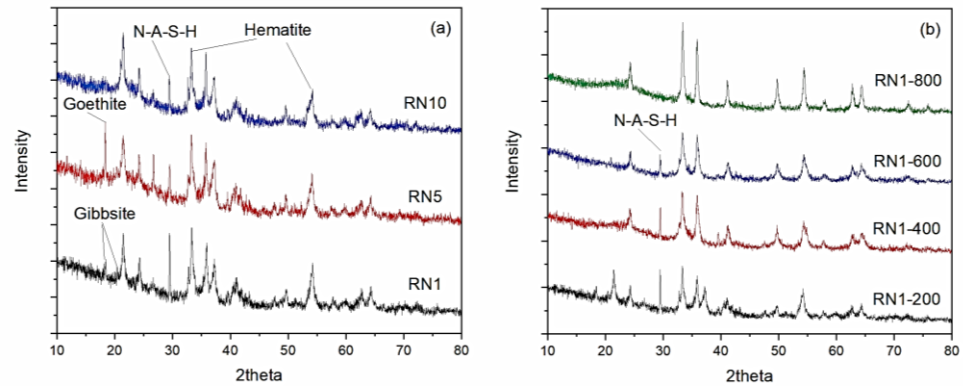

Figure 1. *XRD patterns of geopolymers: (a) synthesized with different NaOH activation ratios of 1%, 5%, 10%; and (b) calcined at different temperatures.*

The XRD patterns of samples calcined at different temperatures are shown in Figure 1b. At 200 °C, the crystalline structure remained similar to that of the uncalcined RN1 sample, with the characteristic N-A-S-H peak at 2θ = 29.4° still present along with the original crystalline phases of red mud. As the calcination temperature increased, the crystalline peaks became sharper and more intense, while the characteristic geopolymer peak gradually decreased and disappeared in the sample calcined at 800 °C. These results suggest that higher temperatures promote crystallization, and excessive crystallization is generally unfavorable for adsorption processes.

3.2. Functional group characterization by FT-IR spectroscopy

As shown in Figures 2a and 2b, all FT-IR spectra of the synthesized materials exhibited typical

absorption bands of geopolymers. The asymmetric stretching vibrations of Si–O–T (T = Si or Al) appeared at around 1000 cm^{-1}, while the bands at 462 cm^{-1} and 563 cm^{-1} correspond to the bending vibrations of Si–O/Al–O and Si–O–Al bonds, respectively [20]. Peaks at 816 cm^{-1}, attributed to the asymmetric stretching of Al or Si tetrahedra in the geopolymer framework [20], were observed in all samples with different NaOH ratios. These peaks gradually shifted toward longer wavelengths as the calcination temperature increased from 200 to 600 °C, and eventually disappeared in the RN1-800 sample. This observation is consistent with the XRD results.

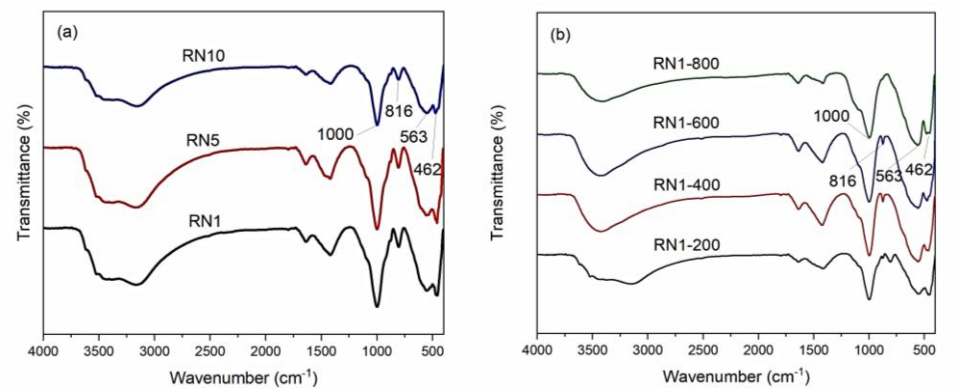

Figure 2. *FT-IR spectra of geopolymers prepared at different: (a) NaOH ratios and (b) calcination temperatures.*

The FT-IR spectra revealed the characteristic absorption bands of geopolymers, confirming that geopolymerization was successfully achieved at all investigated NaOH ratios. Furthermore, calcination below 200 °C did not significantly affect the geopolymer structure.

3.3. Effect of NaOH ratio and calcination temperature on fluoride adsorption

Three geopolymer samples, RN1, RN5 and RN10 synthesized from red mud with NaOH activation ratios of 1%, 5% and 10%, respectively, were evaluated for fluoride adsorption. The results presented in Figure 3 showed that the RN1 sample, activated with 1% NaOH, exhibited the highest fluoride adsorption capacity. With increasing alkali content, the adsorption capacity decreased. This reduction may be attributed to the excessive alkalinity, which accelerates the dissolution of Al_2O_3 and SiO_2, leading to rapid geopolymer network formation. As a result, the synthesized materials could become denser and contain fewer voids, thereby reducing their adsorption capacity.

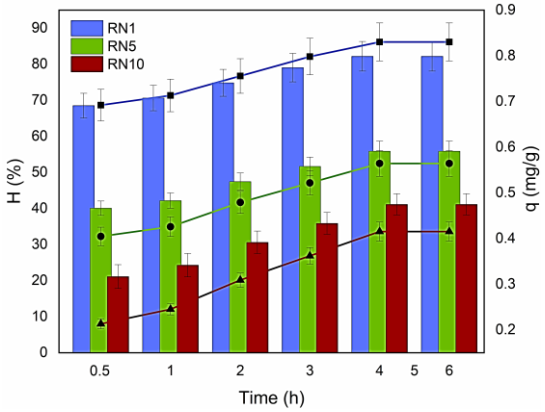

Figure 3. *Effect of NaOH ratio on fluoride adsorption.*

Based on the results of NaOH activation ratio, the RN1 material synthesized from red mud with 1% NaOH activation was further calcined at temperatures ranging from 200 °C to 800 °C and

evaluated for fluoride adsorption. As shown in Figure 4, the RN1 sample calcined at 200 °C exhibited the highest adsorption capacity. Thermal treatment at this temperature likely generated a more porous and open surface structure, thereby enhancing adsorption performance. However, further increasing the calcination temperature above 200 °C reduced the amorphous phase and promoted crystallization, as evidenced by the XRD patterns and FT-IR spectra, leading to a decline in adsorption capacity.

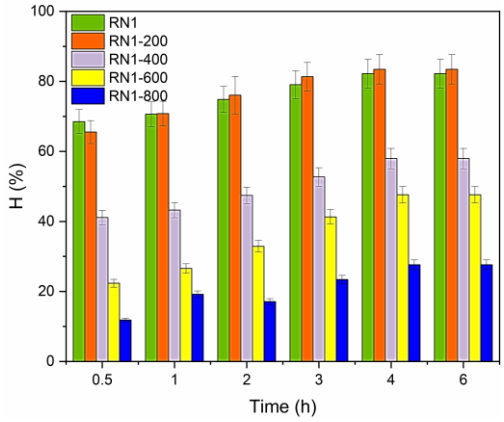

Figure 4. *Effect of calcination temperature on fluoride adsorption.*

From the results of NaOH activation ratio and calcination temperature, the optimal synthesis and modification conditions for the geopolymer material were identified as red mud activated with 1% NaOH and subsequently calcined at 200 °C. The results also indicated that the adsorption capacity did not change after 4 hours, suggesting that the equilibrium adsorption time was 4 hours.

3.4. Determination of pH_{pzc} of the optimal material and effect of pH on fluoride adsorption

As shown in Figure 5, the point of zero charge (pH_{pzc}) of the optimal material RN1-200 was determined to be 8.4. This implies that when the solution pH is below 8.4, the surface of the material carries a positive charge, whereas at pH values above 8.4, the surface is negatively charged. Since fluoride exists in water predominantly as the anion F^-, the adsorption process is more favorable when the material surface is positively charged. This finding is consistent with the results of the pH effect on fluoride adsorption, as shown in Figure 6. The adsorption capacity was high in the pH range of 4–8, but decreased at pH 9 and 10. Thus, the adsorption process proceeds favorably under the pH conditions commonly found in natural water environments, which is of great practical significance.

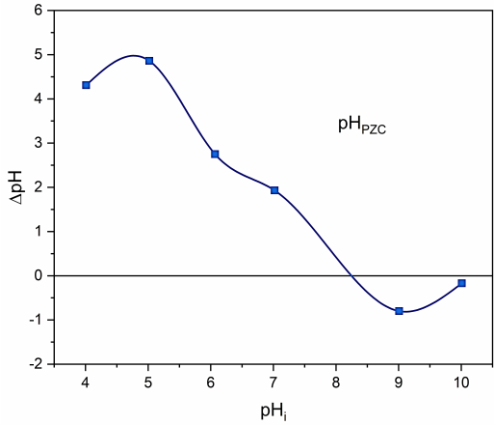

Figure 5. *Plot used to determine pH_{pzc} of the optimal material.*

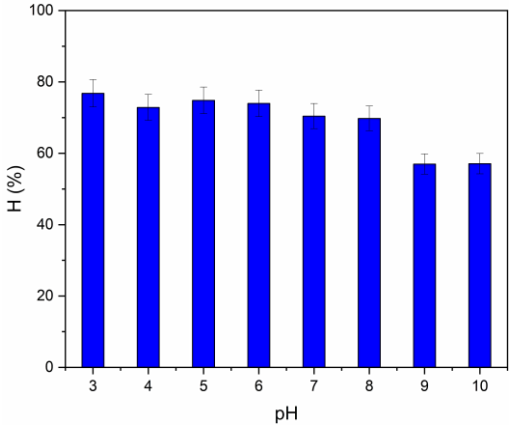

Figure 6. *Effect of pH on fluoride adsorption.*

3.5. Adsorption isotherms of fluoride on the optimal material

Based on the experimental data, the linearized plots of the Langmuir, Freundlich, and Dubinin–Radushkevich isotherm models were constructed and are presented in Figure 7, with the corresponding adsorption parameters summarized in Table 2.

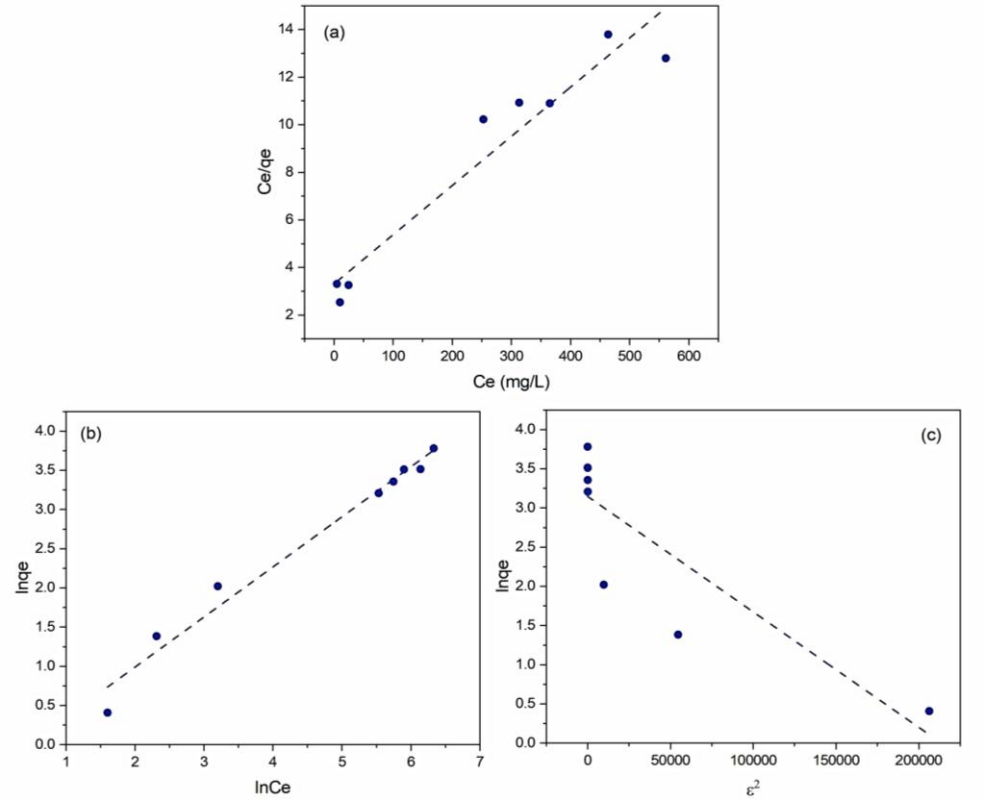

Figure 7. *The Langmuir (a), Freundlich (b), and Dubinin-Radushkevich (c) linear plots describing the adsorption of fluoride onto the optimized material.*

From the regression coefficients (R^2), it was observed that among the investigated models, the Freundlich isotherm provided the best fit for describing the adsorption of fluoride onto the optimized geopolymer material. This indicates that the adsorption process follows a multilayer adsorption mechanism occurring on a heterogeneous surface. Moreover, the adsorption intensity value ($1/n < 1$) confirmed that the adsorption process was favorable.

Table 2. *Adsorption constants and regression coefficients of the Langmuir, Freundlich, and Dubinin–Radushkevich equations for fluoride adsorption on the optimized geopolymer material.*

Model	**Parameters**	**Value**
Langmuir	q_{max} (mg/g)	48.31
	K_L (L/mg)	0.007
	R^2	0.929
Freundlich	K_F (mg/g)(L/g)^n	0.749
	1/n	0.639
	R^2	0.978
Dubinin-Radushkevich	q_m (mg/g)	23.26
	β	$1x10^{-5}$
	E (J/mol)	224.0
	R^2	0.746

The maximum adsorption capacities estimated from the Langmuir isotherm was 48.31 mg/g. When compared with previously reported adsorbents derived from red mud or other fluoride adsorbents (Table 3), it can be seen that synthesizing geopolymers from red mud of this study significantly enhances fluoride adsorption capacity compared to raw red mud, red mud activated by other agents, and various conventional adsorbents.

Table 3. *Maximum adsorption capacities obtained from the Langmuir isotherm model for several adsorbents.*

Adsorbents	q_{max} (mg/g)	Ref.
Mg-impregnated red mud	11.14	[11]
Ce-impregnated red mud	14.45	[11]
RN1-200	**48.31**	**This study**
Zr-modified MOF	19.4	[5]
LDH-Biochar composite	7.24	[6]
Ce-Al/Active carbon	31.65	[7]

4. CONCLUSIONS

Geopolymer adsorbents were successfully synthesized from industrial waste red mud, with the optimal synthesis condition determined at 1% NaOH activation and calcination at 200 °C. Structural analyses confirmed that calcination below 200 °C did not alter the geopolymer framework, while adsorption experiments revealed superior fluoride uptake capacity under these conditions. Fluoride adsorption was best fitted by the Freundlich model and followed a multilayer process on heterogeneous surfaces, with the optimized material (RN1-200) achieving a maximum adsorption capacity of 48.31 mg/g. The thermally modified geopolymer exhibited significantly enhanced adsorption efficiency, a rapid equilibrium time of 4 hours, and effective performance across a wide pH range (≤ 8.4). The findings highlight the potential of red mud-based geopolymers as low-cost, sustainable, and high-capacity adsorbents for practical applications in fluoride remediation from aqueous environments.

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TÓM TẮT

Tận dụng bùn đỏ thải công nghiệp làm chất hấp phụ geopolymer để loại bỏ florua ô nhiễm khỏi dung dịch nước

Bùn đỏ, một phụ phẩm thải của nhà máy alumina Tân Rai, đã được sử dụng làm tiền chất để chế tạo geopolymer và được đánh giá khả năng loại bỏ florua khỏi dung dịch nước. Geopolymer được điều chế bằng cách hoạt hóa kiềm với NaOH (1%, 5% và 10%) và sau đó nung ở nhiệt độ 200 - 800 °C. Cấu trúc của vật liệu geopolymer được đặc trưng bằng XRD và FT-IR. Trong điều kiện thử nghiệm, mẫu được hoạt hóa với 1% NaOH và nung ở 200 °C cho thấy hiệu quả tốt nhất, đạt khả năng hấp phụ fluoride tối đa là 48,31 mg/g với thời gian cân bằng là 4 giờ, và có hiệu quả ở pH ≤ 8,4. Kết quả hấp phụ phù hợp nhất với mô hình đẳng nhiệt Freundlich so với các mô hình Langmuir và Dubinin–Radushkevich, cho thấy sự hấp phụ đa lớp trên bề mặt không đồng nhất. Kết quả từ nghiên cứu này cho thấy geopolymer chế tạo từ bùn đỏ có tiềm năng sử dụng như là một chất hấp phụ tiết kiệm chi phí để loại bỏ fluoride hiệu quả khỏi nước bị ô nhiễm.

Từ khoá: Geopolymer; Bùn đỏ; Florua; Hấp phụ.