

Physicochemical Properties of an Inorganic Polymer (Geopolymer) Sorbent Derived from Local Raw Materials

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Abstract The development of effective and cost-efficient sorbent materials from locally available natural resources is a priority direction in modern materials chemistry and environmental technology. Aluminosilicate sorbents derived from natural clay minerals are of particular interest owing to their wide availability, low cost, and pronounced ion-exchange properties. In this study, an inorganic polymer aluminosilicate sorbent was synthesized by thermochemical activation of clay soil collected from the Amudarya riverbank (Uzbekistan). The raw material, predominantly composed of quartz, kaolinite, and illite, was calcined at 600–650 °C for 4 hours, followed by sequential treatment with 1 M H₂SO₄ and 3 M NaOH solution under autoclave conditions (48 h). Physicochemical characterization was performed using Fourier-transform infrared (FTIR) spectroscopy (Shimadzu IRAffinity-1S; KBr pellet method; 400–4000 cm⁻¹) and X-ray diffraction (XRD) analysis (Shimadzu XRD-6100; Cu K α radiation; 2 θ = 5–80 °). FTIR spectra revealed characteristic absorption bands of the aluminosilicate network in the range 1000–1080 cm⁻¹ (asymmetric Si–O–Si and Si–O–Al stretching vibrations). XRD analysis confirmed the presence of crystalline quartz (2 θ = 20.8°, 26.6°, 36.5°, and 50.1°) and layered aluminosilicate mineral phases (kaolinite and illite). The synthesised material exhibits a partially crystalline structure and is promising for application in the purification of aqueous systems from heavy metals and organic pollutants.

Keywords FTIR spectroscopy, Inorganic polymer, Local clay, Sorbent, XRD analysis

1. Introduction

Adsorbents are widely used to remove pollutants from domestic and industrial wastewater. They are also employed in the oil and gas industry as catalysts and in water treatment as adsorbents and ion-exchange materials for softening hard water [1]. Aluminosilicate-based materials have attracted considerable attention because of their chemical stability, porous structure, and adsorption properties. Natural clay minerals provide an abundant, inexpensive feedstock for these sorbents [2,3]. Their structure supports ion exchange and adsorption of pollutants, including heavy metals, dyes, and organic compounds [4,7,8].

Rapid industrialization and urbanization have made heavy-metal contamination of water an important environmental concern worldwide. Lead (Pb), copper (Cu), cadmium (Cd), chromium (Cr), zinc (Zn), mercury (Hg), and nickel (Ni) are of concern because they do not biodegrade, may accumulate

in organisms, and can be toxic even at low concentrations [9,10,11,12]. Geopolymers, three-dimensional aluminosilicate inorganic polymers, have recently attracted interest because they can be produced at relatively low cost from waste materials, show chemical stability, and have potential for heavy-metal adsorption [13,14,15].

The term “geopolymer” was introduced by Joseph Davidovits in the mid-1970s. Davidovits (1991) described geopolymers as inorganic polymers with amorphous or semicrystalline three-dimensional silicoaluminate frameworks and distinguished three principal structural types:

Poly(sialate) (PS): –Si–O–Al–O– repeating unit; has a Si/Al ratio of 1.

Poly(sialate-siloxo) (PSS): –Si–O–Al–O–Si–O– repeating unit; has a Si/Al ratio of 2.

Poly(sialate-disiloxo) (PSDS): –Si–O–Al–O–Si–O–Si–O– repeating unit; has a Si/Al ratio of 3.

“Sialate” is an abbreviation of silicon-oxo-aluminate. The framework consists of corner-sharing SiO₄ and AlO₄ tetrahedra. Cations such as Na⁺, K⁺, Li⁺, and Ca²⁺ occupy framework cavities to balance the negative charge associated with tetrahedrally coordinated Al³⁺ [16]. The empirical

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polysialate formula is $M_n[-(SiO_2)_z-AlO_2]_n \cdot wH_2O$, where M denotes a cation such as potassium, sodium, or calcium; n is the degree of polycondensation; and z is 1, 2, or 3.

Structural features are central to understanding the physicochemical properties of clay-derived sorbents. Fourier-transform infrared (FTIR) spectroscopy and X-ray diffraction (XRD) are widely used to investigate the structural characteristics and crystalline phases of aluminosilicates [5,6]. XRD identifies mineral components and phase changes during synthesis or modification. This study aimed to synthesize an aluminosilicate sorbent from local clay and examine its phase composition and structural features by FTIR and XRD [17,18].

2. Materials and Methods

The phase composition and crystal structure of the synthesized aluminosilicate sorbent were investigated using a Shimadzu XRD-6100 powder diffractometer (Japan) with a Cu source ($K\alpha_1 = 1.5406 \text{ \AA}$; tube current, 30 mA; voltage, 40 kV). Measurements were performed at room temperature in step-scan mode over $2\theta = 5^\circ\text{--}80^\circ$ at a step size of 0.007° . The diffractometer was calibrated against an $\alpha\text{-Al}_2\text{O}_3$ reference (corundum, NIST SRM 1976), and crystalline phases were identified using the ICDD PDF-2 database [1].

FTIR spectroscopy was used to identify functional groups in the synthesized sorbent. Spectra were recorded using a Shimadzu IRAffinity-1S spectrometer over $400\text{--}4000 \text{ cm}^{-1}$ at 4 cm^{-1} resolution with 32 scans. Samples were prepared as

KBr pellets at a sample-to-KBr mass ratio of 1:100 [19]. Clay was collected from the bank of the Amu Darya River ($41^\circ32' \text{ N}$, $60^\circ38' \text{ E}$). The source material was classified as a heavy sandy loam, with a dominant particle-size fraction of $0.05\text{--}0.25 \text{ mm}$, and consisted mainly of quartz, kaolinite, and illite. The physicochemical properties of the resulting inorganic polymer sorbent were examined by FTIR and XRD [19].

3. Result and Discussion

Local natural clay collected from the banks of the Amu Darya River was used as the starting material for the inorganic polymer aluminosilicate sorbent. The elemental composition of the starting material is presented in Table 1.

The material was placed in a corundum crucible and thermally activated in a laboratory muffle furnace (Nabertherm L9/11/P330) for 4 h to remove organic impurities and increase the structural reactivity of the aluminosilicate framework. After calcination, it was cooled to room temperature in air for 2 h and treated with 1 M sulfuric acid. The material was then rinsed repeatedly with distilled water until neutral. The resulting product was mixed with 3 M NaOH on a magnetic stirrer for 4 h at $60\text{--}80^\circ \text{C}$. A 50 g sample was washed three times with distilled water ($3 \times 200 \text{ mL}$) until the pH reached 6.8–7.2, and subsequently dried at 105°C for 12 h (Figure 1).

The elemental composition of the synthesized inorganic polymer sorbent was measured using a Shimadzu ICPE-9000 instrument.

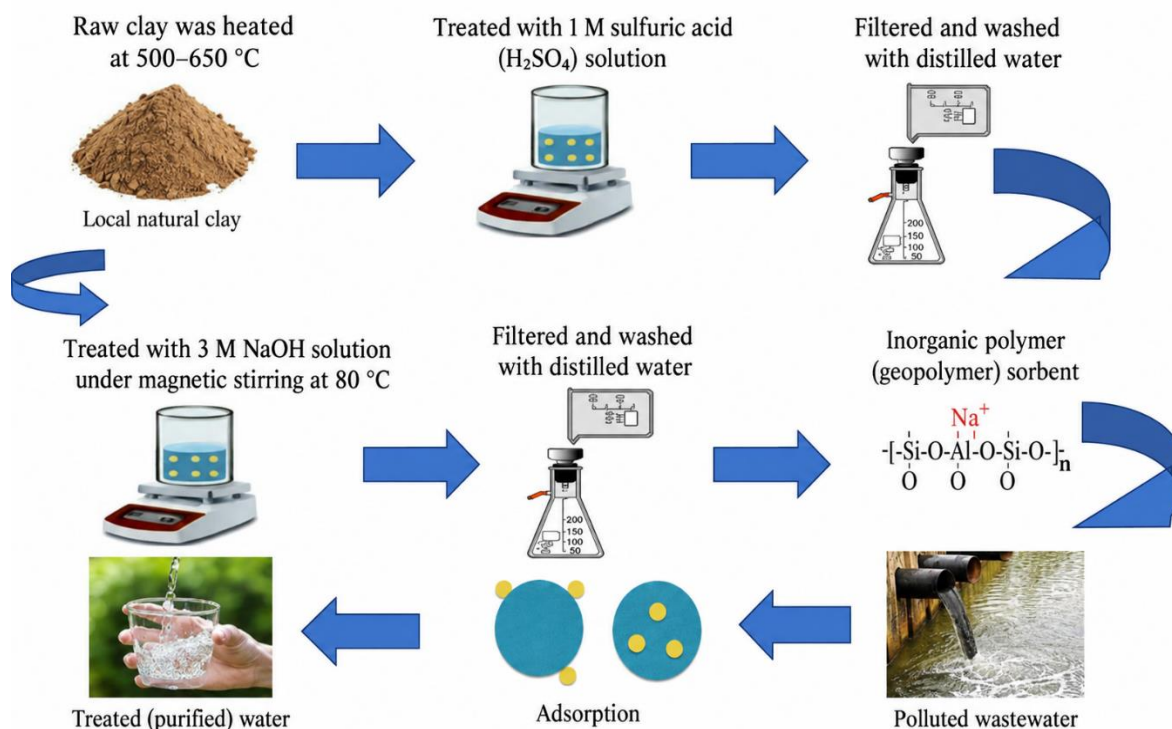


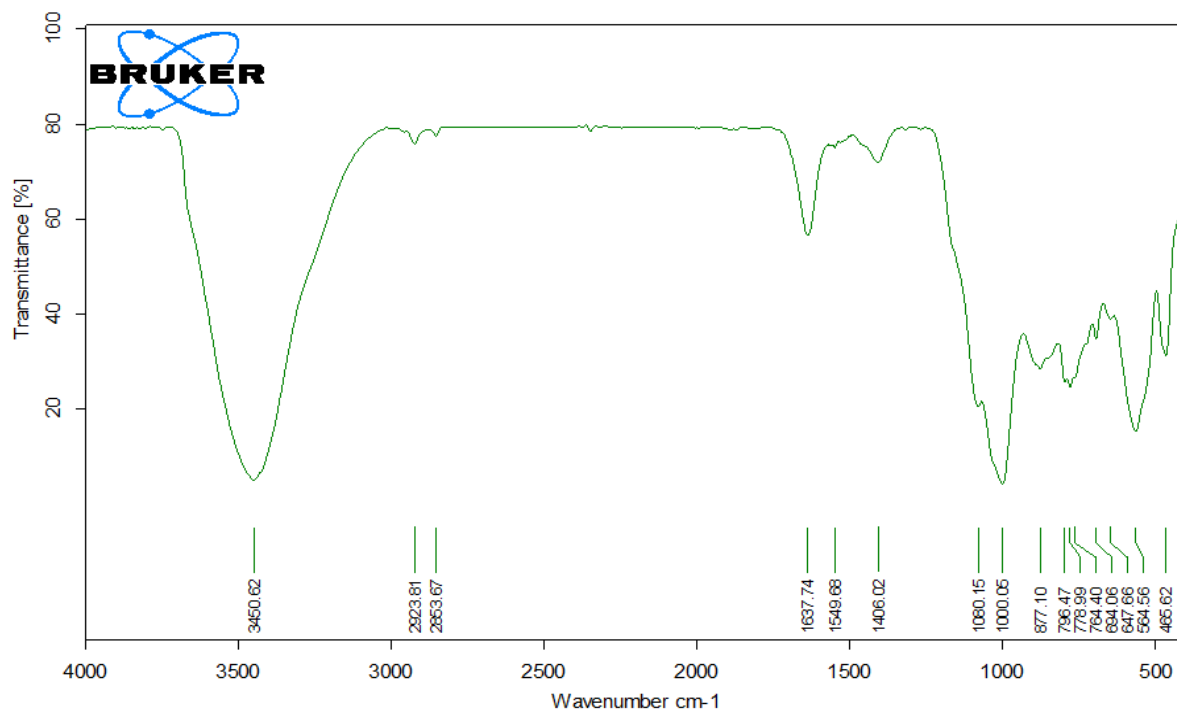
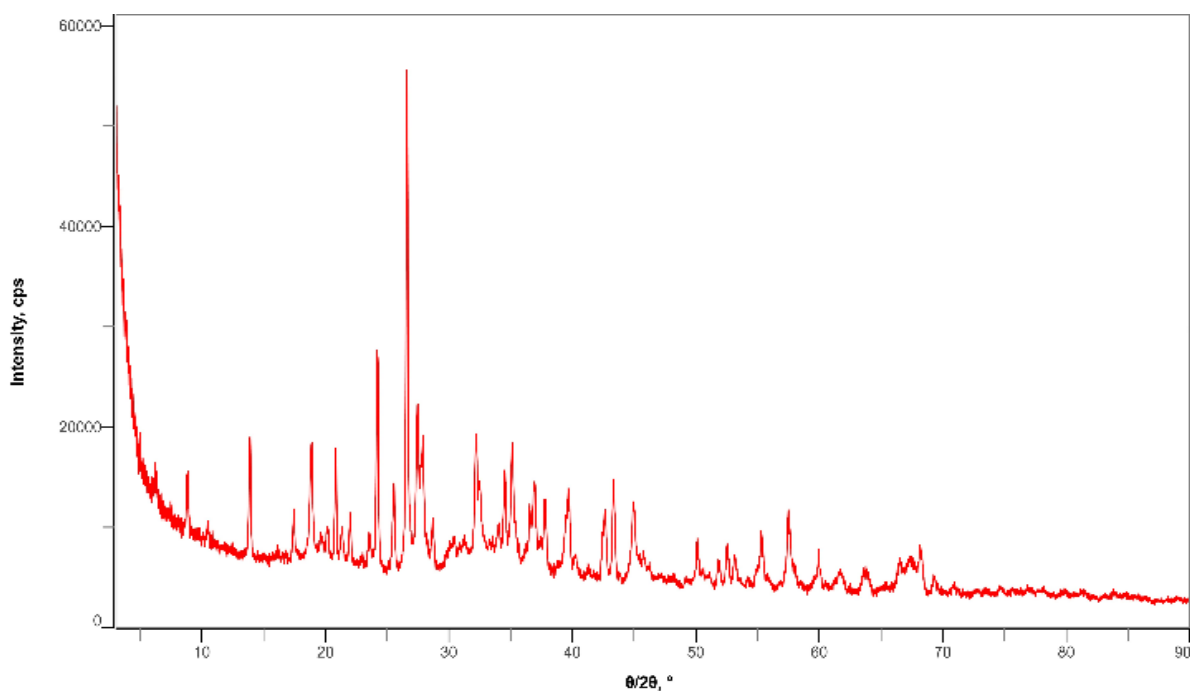
Figure 1. Schematic representation of the synthesis of the inorganic polymer sorbent

Table 1. Elemental composition of the starting clay sample

Starting sample (clay)	Al ₂ O ₃	Ca	Fe	K	Mg	Mn	Na	P	SiO ₂	Other elements
Content, %	13.2	9.68	5.14	0.758	3.23	0.0951	4.16	0.0870	58.7	4.95

Table 2. Elemental composition of the synthesized sorbent

Product	Al ₂ O ₃	Ca	Fe	K	Mg	Mn	Na	P	Si ₂ O ₃
Content, %	34.2	1.08	1.04	0.575	0.572	0.0148	2.4	0.0209	60.5

**Figure 2.** FTIR spectrum of the clay-derived inorganic polymer sorbent**Figure 3.** XRD pattern of the clay-derived inorganic polymer sorbent

The elemental analysis of the synthesized product is presented in Table 2. The clay was collected from the bank of the Amu Darya River (41°32' N, 60°38' E). The source material had a dominant 0.05–0.25 mm fraction and was composed mainly of quartz, kaolinite, and illite.

To remove microorganisms and organic residues, the starting material was heated at 600–650 °C for 4 h. Following cooling in air for 2 h, it was treated with 1 M sulfuric acid. The source also describes subsequent mixing with 3 M NaOH and autoclave treatment for 48 h; the FTIR spectrum of the product is shown in Figure 2.

The FTIR spectrum of the clay-derived inorganic polymer sorbent shows a broad, intense absorption band centered at approximately 3450.62 cm^{-1} , attributable to O–H stretching of hydrogen-bonded surface hydroxyl groups and adsorbed water. Bands at 2853–2920 cm^{-1} correspond to symmetric and asymmetric C–H stretching of aliphatic $-\text{CH}_2-$ groups, possibly arising from residual organic matter. The band near 1637 cm^{-1} is assigned to H–O–H bending, whereas absorption at 1400–1550 cm^{-1} suggests carbonate groups.

The dominant FTIR feature occurs in the 900–1200 cm^{-1} range. Its broad, intense band at 1000–1080 cm^{-1} is assigned to asymmetric stretching of Si–O–Si and Si–O–Al linkages, consistent with an aluminosilicate network.

Bands at 790–800 and 460–470 cm^{-1} are associated with Si–O and Al–O bending vibrations. Bands in the lower-frequency region (600–450 cm^{-1}) may also include metal–oxygen (M–O) vibrations. Together, these features are consistent with an aluminosilicate framework containing metal–oxygen bonds. The phase composition and crystal structure were assessed by XRD (Figure 3) using a Shimadzu XRD-6100 powder diffractometer (Cu $K\alpha_1 = 1.5406 \text{ \AA}$; 30 mA; 40 kV), at room temperature over $2\theta = 5^\circ$ – 80° with a 0.007° step size. Several well-defined diffraction peaks indicate crystalline mineral phases.

The most intense diffraction peaks were assigned to quartz (SiO_2), a common component of natural clay. Characteristic quartz reflections occurred near $2\theta = 20.8^\circ$, 26.6° , 36.5° , and 50.1° .

Additional reflections may be associated with layered aluminosilicate minerals such as kaolinite, illite, or montmorillonite. Peaks within $2\theta = 12^\circ$ – 15° and 24° – 27° are consistent with layered structures containing silicate tetrahedral and alumina octahedral sheets; phase assignments require comparison with reference patterns.

The intensity and distribution of the reflections suggest that the synthesized material is partially crystalline. Thermal activation may have increased the reactivity of the aluminosilicate framework.

The surface morphology of the inorganic polymer sorbent was examined by scanning electron microscopy (SEM; Figure 4).

The micrographs show heterogeneous agglomerates of small particles and an irregular, rough surface with visible interparticle voids. In Figure 4a, small spherical or irregular particles coalesce into larger aggregates. The spaces between aggregates may provide accessible sites for adsorption; adsorption capacity, however, was not directly quantified in this study.

The images do not show large, well-defined single crystals. The granular morphology may reflect a polycrystalline or microcrystalline material. Some regions in Figures 4b and 4c resemble morphologies reported for early stages of zeolite formation, although morphology alone cannot establish a zeolite phase.

4. Conclusions

An inorganic polymer aluminosilicate sorbent was synthesized from local natural clay. Thermal treatment induced partial structural transformation of the clay minerals and produced a mixed crystalline–amorphous aluminosilicate framework. FTIR and XRD findings support formation of a modified aluminosilicate material, while SEM shows an irregular aggregated morphology with interparticle voids. The adsorption performance and mechanisms require direct experimental assessment.

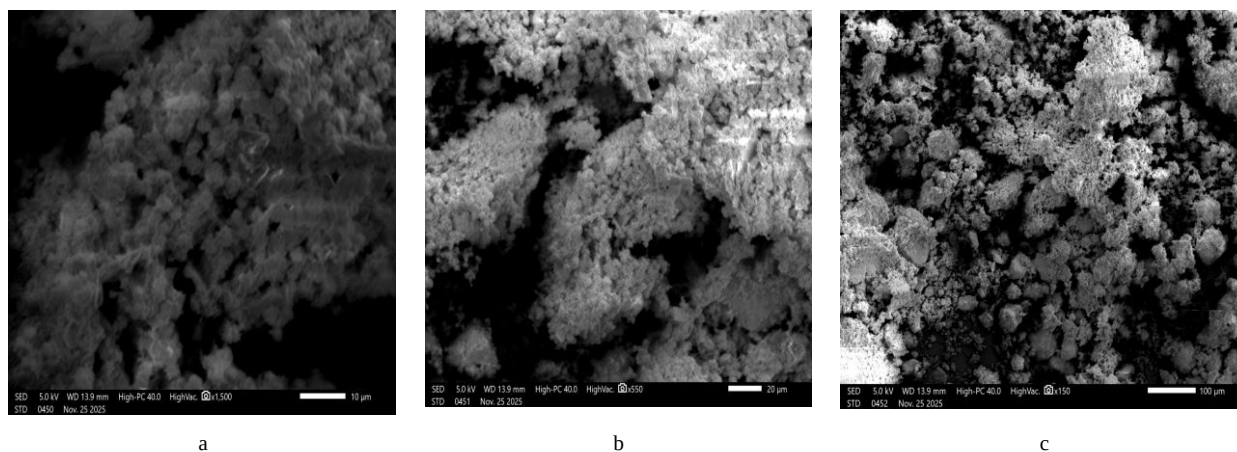


Figure 4. SEM micrographs of the synthesized inorganic polymer sorbent at different scale bars: (a) 10 μm , (b) 20 μm , and (c) 100 μm .

These results indicate that local clay can be used as a feedstock for aluminosilicate sorbents with structural features of interest for water-treatment applications.

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