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Removal of toxic metal ions and their kinetic studies from aqueous solution using zeolite as an adsorbent

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ABSTRACT

In this work, zeolites were prepared from fly ash and characterized by Fourier transform infrared spectroscopy (FT-IR), SEM and energy dispersion spectroscopy. Fly ash is a byproduct of thermal plants. It is essential to use fly ash in other applications viz preparation of cement, water purification and magnetic materials. The obtained zeolites were used for removal of Co(II), Cd(II), Ni(II), Cs(I) and Sr(II) from aqueous solution. The different factors effecting adsorption of metal ions on zeolites viz contact time, initial concentration of metal ions and pH were studied. The batch method has been employed using metal ions in solution from 1,000 to 5,000 μg/L. The obtained adsorption data were used for Freundlich and Langmuir isotherms measurement and both were found applicable for the metal ions adsorption on zeolites. The adsorption data were followed pseudo-second-order reaction kinetics. The results show that zeolites have great potential to remove Co(II), Cd(II), Ni(II), Cs(I) and Sr(II) from aqueous solutions through chemisorption and physisorption.

Keywords: Zeolite; Toxic metal ion; Ion exchange; Adsorption; Isotherm models

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1. Introduction

Recently, toxic metals are major pollutants in water and are becoming severe public health problem. Industrial wastewater contains various kinds of metals [1,2]. These metal ions are present in water from industrial applications viz manufacture of pesticides, batteries, alloys, electroplated metal parts, mining, refining and production of textiles, paints and dyes [3–5]. Toxic metals are not metabolized by the body and accumulated in the soft tissues. Co(II), Ni(II) and Cd(II) are especially common metals that tend to accumulate in organisms, causing numerous diseases and disorders [6,7]. These metals' toxicity can cause hypertension, nephritis, abdominal pain, nausea, vomiting, behavioral changes and development defects [8,9]. On the other hand, accidentally water may get contaminated with long-lived radionuclides such as cesium, strontium, lanthanides and actinides due to operation of nuclear power plants, research facilities, use of radioisotopes in industry and diagnostic medicines. There is possibility that water may get contaminated with radionuclides and their degraded products during nuclear war and accidents in nuclear power reactors [10–13]. Such events have occurred in Japan and Chernobyl due to the earthquake and industrial accident, respectively. Drinking water contaminated with radionuclides will be a severe threat to military as well as civilian population in future. Therefore, there is requirement to develop adsorbents which can remove radionuclides from water.

A number of methods are available for the removal of metal ions from aqueous solutions like ion exchange [14,15], reverse osmosis and nanofiltration [16], precipitation and adsorption [17–19]. Adsorption method is very popular and cost-effective. A number of adsorbents have been reported for removal of these metal ions from water-like inorganic material [20,21] and organic adsorbent alumina [22], bentonite [23], ion-exchanger polyacrylamide cerium titanate polymer [24], titanium tungstate [25] and carbon materials [26]. However, most of these methods have limitations, which

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include high cost, pH range, unavailability and generation of large volumes of secondary waste and poor removal efficiency [27–29]. Considering from the economy and efficiency point of view, adsorption is regarded as the most promising and widely used method among all these [30]. The efficiency of adsorption depends on many factors, including the surface area, pore size distribution, polarity and functional groups of the adsorbent [31].

Zeolites are naturally occurring hydrated aluminosilicate minerals. They belong to the class of minerals known as "tectosilicates" [32]. The structure of zeolites consists of the three-dimensional frameworks of SiO_4 and AlO_4 tetrahedra. The aluminum ion is small enough to occupy the position in the center of the tetrahedron of four oxygen atoms, while the isomorphous replacement of Si^{4+} by Al^{3+} produces a negative charge in the lattice. The net negative charge is balanced by the exchangeable cations (Na, K or Ca). These cations are exchangeable with certain cations in solutions [33]. Zeolites are microporous crystalline solids with well-defined structures, which have unique ion exchange and sorption properties, and are widely used in a large number of water treatment processes [34]. Zeolites have been reported as an adsorbent for removal of heavy metal ions from water [33,35]. Thermal plants produce a large quantity of fly ash. This fly ash contains different oxide viz SiO_2, Al_2O_3 and others. Therefore, fly ash can be converted in zeolite and used to water purification application.

Our laboratory has developed various water purification systems and adsorbents for wastewater treatment [36–40]. Presently, R&D on development of adsorbents for removal of toxic metals ions from contaminated water is initiated. The aim of this work was to examine the efficiency of the prepared zeolite for the removal of toxic metal ions from water. The effect of various parameters viz pH, contact time and initial concentration of metal ions were examined. Langmuir and Freundlich isotherms were applied on obtained adsorption data and calculated different parameters of isotherms. The pseudo-second-order model was considered to describe the adsorption kinetic behavior. Both isotherms and kinetics, to check the applicability of synthesized zeolites for removing of toxic metal ions from water.

2. Experimental setup

2.1. Instrumentation

All atomic absorption spectrometer (AAS; Analytik-Jena-Nova-400) measurements were carried out on flame mode with single beam. AAS equipped with 100 mm burner, a cross-flow nebulizer 5.0 mL/min and 1.2 mm slit were used throughout the experiments. Each experiment was duplicated under identical conditions using this instrument for concentration determination.

IR spectra were recorded on JASCO FT-IR spectrometer model Jasco-610 by using KBr pellets. Scanning electronic microscopy (SEM) images and energy dispersion spectroscopy spectra were performed on Carl-Zeiss EVO-MA-50. Gene matrix make X-ray fluorescence (XRF) spectroscopy were used for quantitative determination of elements in synthesized zeolite. X-ray diffraction pattern was recorded on a PANalytical X'Pert PRO diffractometer with a Cu Kα source. The pH measurements were made on a digital pH meter (Hach, SensION 1, model 51935-00) equipped with a gel-filled pH electrode. The meter was calibrated with the buffers of 4, 7 and 10.

2.2. Chemicals and solutions

Analytical grade reagents were used. All laboratory wares used for analytical determination were cleaned first by trace metal grade nitric acid and hydrochloric acid, followed by repeated rinsing with deionized water. All the solutions were prepared with ultrapure water (resistivity: 18.2 MΩ cm,) from an Elix analytical reagent grade water purification system.

Calibration standard solutions and internal standards were prepared from commercial metal standard solutions. Analytic grade nitric acid (Fisher) was used as acid for the preparation of all the calibration standard solutions and analytical solutions. Standard working solutions 1,000 mg/L of metal ions were prepared from their salts and solutions of varying initial concentrations were prepared from a 1,000 mg/L by serial dilution using distilled deionized water.

2.3. Preparation of zeolites

Mixture of 100 g fly ash and 50 g sodium hydroxide were milled and fused at 600°C for 1 h in furnace. The reaction mixture was cooled to room temperature, grinded and added to water. Crude product was obtained in the form of slurry. It was agitated with mechanical stirrer in a glass beaker for 12 h and kept at 100°C for 6 h. The resultant precipitate was washed with distilled water till neutral pH achieved. Then, it was filtered and dried in oven.

2.4. Adsorption studies with batch method

Batch adsorption experiments were carried out in beakers (100 mL) containing zeolite (50 mg) with 10 mL of metal ions, i.e., Co(II), Ni(II), Cd(II), Cs(I) and Sr(II) of desired concentration (1,000–5,000 μg/L) at 30°C ± 1°C. The beakers were kept for 10–60 min and solutions containing abovesaid metal ions were filtered using Whatman filter paper (No. 42). After each experiment, the residual concentration of metal ions was determined by AAS. The amount of metal ion adsorbed by zeolite (Q_e, mmol/g) was calculated according to the following equation:

$$Q_e = (C_i - C_e) \times V/m \tag{1}$$

where C_i and C_e are initial and equilibrium concentration (μg/L), respectively. V (L) and m (g) are volume of the sample solution and mass of the zeolite. The observed adsorption data were used for Langmuir and Freundlich isotherms measurements.

2.5. Kinetic adsorption experiment

To understand the effect of time on the extent of adsorption and to obtain equilibrium data were used for kinetic study. The equilibrium concentration of Co(II), Ni(II), Cd(II), Cs(I) and Sr(II) were determined at different time interval (5–60 min) with initial concentration of metal ions 2,000 μg/L, zeolite 50 mg and pH 7. The series of samples are quenched at time interval by filtration. The concentrations of the filtrate were analysed by AAS. These results were used to obtain the adsorption kinetic.

2.6. pH effect

The effect of pH on adsorption of metal ions on zeolite was evaluated at constant initial concentration of metal ions 1,000 µg/L (10 mL), adsorbent 50 mg, contact time 30 min and different pH 4, 7, 10 and residual concentration of metal ions determined by AAS.

3. Results and discussion

3.1. Characterization of zeolite

The FT-IR spectrum of zeolite was performed (Fig. 1). The absorption bands localized at 1,006 cm^{-1} due to asymmetric stretching vibrations of bridge bonds Si–O–Si and Si–O–(Al), 726 cm^{-1} symmetric stretching vibrations of bridge bonds Si–O–Si, 555 cm^{-1} (complex band) symmetric vibrations of bridge bonds Si–O–Si and bending vibration, 468 cm^{-1} bending vibrations O–Si–O and O–Al–O. Absorption band present at 3,655 cm^{-1} due to silanol groups and hydroxyl groups. The presence of these absorption bands of groups, confirm the structure of zeolite.

Scanning electronic microscopy (SEM) and energy dispersive analysis of X-rays (EDAX) were preformed of zeolite (Figs. 2 and 3) for analysis of morphology and elemental analysis. Small zeolite crystals are envisioned as cubic particles in order to estimate the particle size and crystals are discrete with narrow size distributions. EDAX results show (Fig. 3) the presence of Si, Al, O and Na elements in zeolite.

The X-ray powder diffraction (XRD) patterns of synthesized zeolite was recorded at 2θ values between 5° and 60°. The XRD pattern of zeolite is represented in Fig. 4. It reveals an amorphous–crystalline nature. Major peaks identified in synthesized zeolite include zeolite-P and mullite.

The quantitative determination of elements contained in the synthesized zeolite was carried out using the XRF technique. Table 1 gives the percentage amount of the major element in the synthesized zeolite.

3.2. Adsorption isotherm

The adsorption isotherms of metal ions onto zeolite from aqueous solution is shown that the adsorption capacity of metal ions onto zeolite increases with increasing the initial concentration of metal ions and continued up to 2,000 µg/L and level off thereafter. The initial concentrations of metal ions (1,000–5,000 µg/L) were used for investigation of adsorption isotherm. The equilibrium concentrations of abovesaid metal ions are obtained after 30 min of contact time.

Langmuir [41] and Freundlich [42] isotherms are employed to describe the adsorption process.

The Langmuir isotherm model is expressed as follows:

$$C_e/Q_e = 1/(Q_m b) + C_e/Q_m \tag{2}$$

where C_e is the equilibrium concentration obtained from the initial concentration upon a certain period of contact time

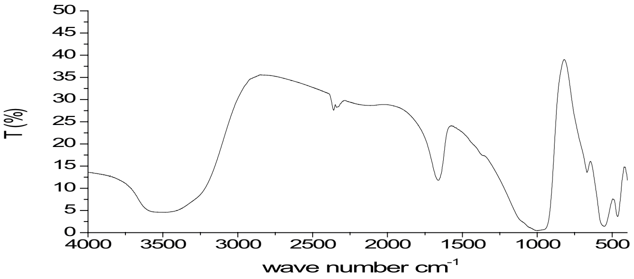

Fig. 1. FT-IR spectra of zeolite.

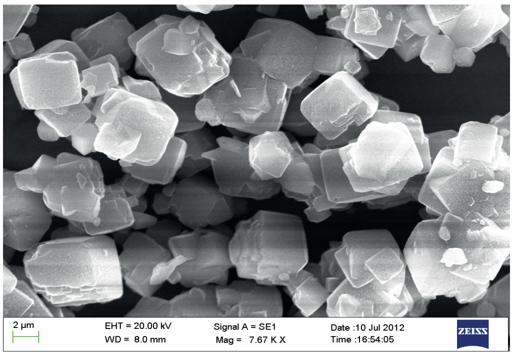

Fig. 2. SEM image of zeolite.

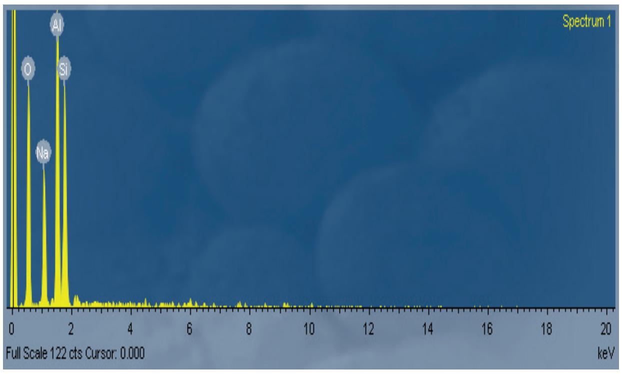

Fig. 3. EDAX of zeolite.

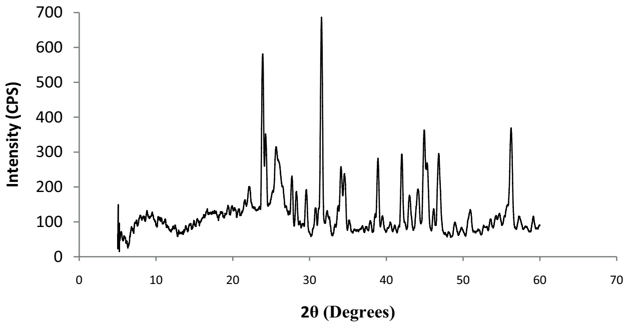

Fig. 4. X-ray diffraction spectrum of synthesized zeolite.

with the zeolite; Q_e is the amount of metal ions adsorbed per gram of zeolite (µg/g) at equilibrium, it is derived from Eq. (1); Q_m is the maximum adsorption capacity (µg/g) and b is the Langmuir parameter related to energy of adsorption.

The linear plot of C_e/Q_e vs. C_e gives the intercept and slope corresponding to $1/(Q_m b)$ and $1/Q_m$, respectively, from which both Q_m and b are derived. A plot of C_e/Q_e vs. C_e obtained straight line if the Langmuir equation is obeyed by the adsorption equilibrium [43]. The adsorption data were used for plot C_e/Q_e vs. C_e and obtained straight line. The values of Q_m and b were calculated from plot and presented in Table 2. Freundlich isotherm is expressed as follows:

$$\log Q_e = 1/n \log C_e + \log K \tag{3}$$

where K and $1/n$ are Freundlich constants, indicating the sorption capacity and sorption intensity, respectively. C_e is the equilibrium concentration of metal ions in aqueous solution and Q_e is the sorption capacity. The plot of $\log Q_e$ against $\log C_e$ gives the intercept and slope corresponding to $\log K$ and $1/n$, respectively, from which both K and n are obtained.

The regression equations parameters Q_m, K, $1/n$ and the correlation coefficient are summarized in Table 2. It is seen that Langmuir as well as Freundlich model are suitable to fit the adsorption data since the correlation coefficients are higher than 0.97.

3.3. Adsorption kinetics

The kinetic property of metal ions adsorbed on zeolite was assessed (Fig. 4). The adsorption rates were determined at pH 7, metal ions concentrations 2,000 µg/L and contact time 5–60 min in aqueous solution.

The adsorption kinetic mechanism was evaluated using one conventional model, namely the pseudo-second-order equations [44]. In the case of pseudo-first-order, very low correlation coefficient values were obtained, so that this model was not suitable for absorption of metal ions on zeolite.

A pseudo-second-order reaction [45] is guided by the following expression:

$$t/Q_t = 1/(k_2 Q_e)^2 + t/Q_e \tag{4}$$

where Q_e (µg/g) is the amount of metal ion adsorbed at equilibrium, Q_t (µg/g) is the amount of metal ion on the surface of the sorbent at time t and k_2 [g/(µg min)] is the rate constant of pseudo-second-order adsorption.

The plot of t/Q_t vs. t is drawn and values of k_2 and Q_e were derived from the intercept and slope of the linear plot (Fig. 5). The obtained values were summarized in Table 3.

High correlation coefficient (R^2) values were obtained for pseudo-second-order reaction of different metal ions. Therefore, it is suitable and applicable for adsorption of metal ions on zeolites. The kinetic data for second-order assessment are listed in Table 3.

3.4. Effect of pH

The activity of the adsorbent's functional groups is strongly affected by the solution of pH. The point of zero charge pH (pH_{pzc}) is the pH of the solution at which the overall observed charge on the surface of adsorbent is zero. This is due to hydrogen ions that are strongly competing with metals. At this pH, protonation of chelating group also

Table 1
Chemical composition of synthesized zeolite

Oxide	%
SiO_2	65.016
TiO_2	0.157
Al_2O_3	34.192
Fe_2O_3	0.093
CaO	0.142
MnO	0.136
K_2O	0.157
Na_2O[a]	ND

[a] Na_2O not detected due to low Z value.

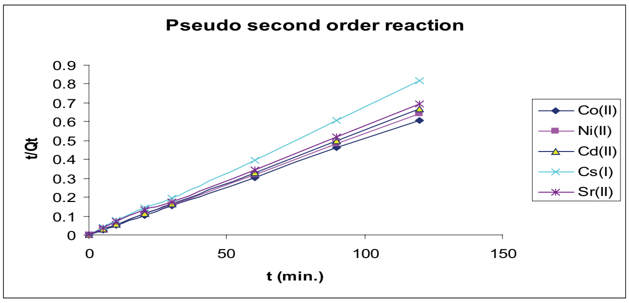

Fig. 5. Second-order kinetic plot for the adsorption of different metal ions.

Table 2
Langmuir and Freundlich parameters of different metal ions adsorption

Langmuir model				Freundlich model		
Metal	Q_m (µg/g)	B (L/µg)	R^2	K (µg/g) ((L/µg^1)$^{1/n}$)	$1/n$	R^2
Co(II)	1,933.5	0.00204	0.9635	5.7226	0.8544	0.9786
Cd(II)	1,763.6	0.00172	0.9762	5.1345	1.023	0.9999
Ni(II)	1,857.4	0.00209	0.9615	5.6754	1.0004	0.9995
Cs(I)	1,541.7	0.00124	0.9732	4.4146	1.0032	0.9991
Sr(II)	1,683.6	0.001659	0.9931	5.0512	1.142	0.9965

Table 3
Kinetic parameters for different metal ions adsorption onto zeolite

Pseudo-second-order				
Metals	C_0 (μg/L)	k_2 [g/(min μg)]	Q_e (μg/g)	R^2
Cs(I)	2,000	13.9706×10^{-2}	1,492.53	0.9996
Sr(II)	2,000	6.1478×10^{-2}	1,754.38	0.9993
Ni(II)	2,000	14.2023×10^{-2}	1,818.18	0.9999
Cd(II)	2,000	19.5230×10^{-2}	1,886.79	0.9999
Co(II)	2,000	36.0642×10^{-2}	1,960.78	0.9999

Table 4
Effect of pH on adsorption of metal ions on zeolite

pH	Removal (%)
4	60–65
7	80–95
10	50–60

takes place. Therefore, complexation with available ligands is reduced and hence the percentage removal decreases. At pH > pH_{pzc} deprotonation of functional group occurs, and salt formation is taken place. Therefore, percentage uptake of metal ions decreases. The removal percentage is shown in range due to different pH_{pzc} value for different metal ions. The pH_{pzc} depends on zeolite and metal ions (Table 4).

4. Conclusions

Zeolite was synthesized from fly ash and characterized using FT-IR, SEM–EDAX and XRD techniques. Synthesized zeolite was used for adsorption of toxic ions viz Co(II), Cd(II), Ni(II), Sr(II) and Cs(I). The results of the experiments indicated that concentration of toxic ions, contact time and pH have very significant effects on adsorption of abovementioned ions on zeolite. The isotherm experimental data fits well to Langmuir and Freundlich models and found high correlation coefficient (R^2 0.96–0.99). The value of adsorption capacity is higher than mentioned in other reference study. Zeolite obtained in this work can be used as an effective and inexpensive adsorbent for toxic ions removal from aqueous solution.

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