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Adsorption Characterization of Strontium on PAN/Zeolite Composite Adsorbent

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Abstract

This work reports the adsorption of strontium from aqueous solutions onto PAN/zeolite composite. The strontium adsorption on the composite adsorbent was studied as a function of initial strontium concentration, pH of the solution, contact time and temperature. Adsorption isotherms like Langmuir, Freundlich, Dubinin-Radushkevich (D-R) and Temkin were used to analyze the equilibrium data at the different concentrations. Adsorption process well fitted to Temkin isotherm model. Thermodynamic parameters such as the changes in enthalpy, entropy and Gibbs' free energy were determined, showing adsorption to be an exothermic and spontaneous process.

Keywords: Composite Adsorbents, Strontium, Sorption Behavior, Adsorption Isotherms, Thermodynamic Parameters

1. Introduction

Radioactive strontium occurs in the environment as ^{89}Sr and ^{90}Sr with half-lives of 51 days and 29 years respectively and it is not produced only as a waste fission product from nuclear power plants, also produced in the reprocessing of nuclear fuels [1]. Sr-90 as a main species is a soft β-emitter of 0.5460 MeV energy. The most of ^{90}Sr existing in the environment can form to deposition alone with rain or other precipitation. Radiostrontium undergoes decay while emitting β-radiation and forms to ^{90}Y with a half-life of 64 h, which is very strong β-emitter. Strontium-90 is relatively mobile and can move down with percolating water to underlying layers of soil and into groundwater. Strontium preferentially adheres to soil particles, and the amount in sandy soil is typically about 15 times higher than in interstitial water, concentration ratios are typically higher in clay [2].

Chemically, strontium resembles calcium, it is taken up via the gastrointestinal tract and collects in the body becoming part of the bone marrow tissue and damaging blood-producing cells. It is easily incorporated into bone and continues to irradiate localized tissues with the eventual development of bone sarcoma and leukemia. So, ^{90}Sr is considered as one of the most hazardous element in fission products. Furthermore, the high content of Sr as a heat-generator is unfavorable for vitrifying high level liquid waste (HLLW). Therefore, from the viewpoint of safety, eliminating Sr from the radionuclides is quite necessary prior to the final disposal of HLLW [1,3].

Separation of strontium is particularly important in the determination of the radioactive isotopes of strontium ^{89,90}Sr in natural samples. Isotope ^{89,90}Sr are pure β-emitters and cannot be determinate without separating strontium from all natural and artificial radioactive isotopes present as well as from the elements contained in large quantities of natural samples such as potassium, sodium, calcium and iron [4].

The composite ion exchangers have been used in several studies to treatment of low and medium level liquid radioactive wastes [5-12], to investigate of sorption behavior of I to V group elements (Fr, Ra, Pb, Bi, Eu, Zr, Hf, Th, Nb, Pa, U) [13], to purification of ^{223}Fr from its decay products [14], to removal of some basic dyes [15], and Na from irradiated samples [16] and to mineralization of biological materials in neutron activation analysis [17].

PAN (polyacrylonitrile) is one of the most favorable binding materials for any inorganic materials, due to its physicochemical properties such as excellent pelletizing property, strong adhesive force with inorganic materials, good solubility for organic solvents and chemical stability [18]. Polyacrylonitrile is an inexpensive raw material and effectively immobilize to ion exchange materials into useful forms, such as spherical beads. PAN beads are highly porous and can accommodate very high loadings of ion exchange material (5 - 95%wt) iFnto the PAN matrix. These highly porous PAN beads exhibit a number of advantages over other granular sorbents, namely; significantly improve kinetics and sorbent capacity due to increase availability of the sorbent material, easy modification of physico-chemical properties (hydrophilicity, porosity, mechanical strength), and simplified production [19].

To remove Sr ions selectively from low and medium level liquid radioactive wastes, organic/inorganic composite ion exchangers have been widely used [20-28].

In this study, PAN/zeolite composite adsorbent was prepared and studied adsorption of strontium from aqueous solution as a function of the initial strontium concentration, pH, shaking time and temperature. The sorption data were interpreted using Langmuir, Freundlich, Dubinin-Radushkevich (D-R) and Temkin equation. Various thermodynamic parameters, including the mean free energy of sorption, were also determined.

2. Experimental

2.1. Materials and Methods

The zeolite (clinoptilolite) which is originated from Manisa-Gordes/Turkey was obtained from Pamukkale University, Turkey. The clinoptilolite was ground and wet sieved to a particle size of –200 mesh. Polyacrylonitrile (PAN) fiber was obtained from the Industry of Acrylic Chemistry (AKSA), Istanbul, Turkey.

The composite adsorbent was prepared from inorganic adsorbent, a natural zeolite clinoptilolite, as an active component and polyacrylonitrile (PAN) as a binding polymer. The composite beads were prepared in a flask with reflux as a reactor. Very fine colloidal particles of the clinoptilolite (–200 mesh) were stirred with the solution of PAN which was solved in 11.86 g of n-dimethylformamide (DMF) at 70˚C for 1 hour to form homogeneous solution. The mass ratio of the PAN to clinoptilolite was adjusted at 1:1 [29]. The mixture was fed into the nozzle to obtain the spherical composite beads. Ultra pure distilled water (Millipore) was used as a gelatin agent. The composite beads were washed repeatedly by ultra pure distilled water to remove the solvent and then dried at 60˚C. The obtained spherical composite beads were sieved and fractionated according to the particle size. The dried composite adsorbent was stored in wide mouth plastic bottle for further use. It was assumed that the composite beads have homogeneous distribution of the inorganic particles in their matrix structure.

Arsenazo III and $SrCO_3$ were obtained from Merck Co. All chemicals reagents used in the present study were of analytical reagent grade. The stock solution of strontium was prepared by dissolving an accurately weighed amount of strontium (II) carbonate ($SrCO_3$) in distilled water to achieve a concentration of 500 µg/mL, and subsequently diluted to the required concentrations. The buffer solutions (pH: 4, 7 and 9) to calibrate the pH-meter model 8521 from Hanna Instruments were purchased from Merck. In all experiments, ultra pure distilled water was used for analytical purposes.

2.2. Adsorption Experiments

Adsorption studies were carried out by batch technique to obtain the equilibrium data. The experiments were carried out by agitating 10 mL of a solution containing different concentrations of Sr(II) in a thermostated water bath with 0.03 g of PAN/zeolite for various contact time. Batch adsorption experiments were carried in a (GFL-1083 model) with a constant shaking rate. Filter-separating of solid phase from liquid was followed by centrifuging at 300 rpm for 10 min. The strontium was determined spectrophotometrically using Arsenazo III method as complexing agent at 640 nm against reagent blank, employing Shimadzu UV-VIS 260 Spectrophotometer [30]. In order to calculate the strontium concentration, the sorption of solution was compared with a working curve that was a plot of absorbance versus standard concentration of strontium. The amount of adsorbed strontium was estimated from the difference between the initial and final concentrations of strontium (II) by means of UV-VIS spectrophotometer (Model Shimadzu UV-VIS 260). All experiments were carried out at 298 K and in duplicate. ±5% was the limit of experimental error of each duplicates, any experiment resulted in higher than this limit was repeated.

The percentage sorption of strontium from aqueous solution was computed as follows:

$$\% \text{ Adsorption} = \left[\left(C_i - C_e\right)/C_e\right] \times 100 \quad (1)$$

where C_i: concentration of the initial solution (µg/mL), C_e: concentration of the solution in equilibratio (µg/mL).

2.3. Adsorption Isotherm

Adsorption isotherms were studied by mixing a known amount of PAN/zeolite (0.03 g) with various initial Sr(II) solution concentrations ranging from 25 to 175 µg/mL at 298 ± 2 K and at pH 5. The adsorption isotherms were obtained by analyzing solutions in contact with composite adsorbent before and after equilibrium and plotted in terms of the equivalent fraction of strontium in the composite phase against the equivalent fraction in the solution phase. The experimental data obtained in the present work was tested with the Langmuir, Freundlich, Dubinin-Radushkevich (D-R) and Temkin isotherm equations. Linear regression is frequently used to determine the best-fitting isotherm, and the applicability of isotherm equations is compared by judging the correlation coefficients.

2.4. Thermodynamic Parameters

Evaluation of thermodynamic parameters was made to assess the spontaneity of the sorption process. The influence of temperature variation was examined on the sorption of Sr(II) of fixed concentration 150 µg/mL onto PAN/zeolite composite using 20 min of equilibration time and temperature from 293 K to 333 K. The thermo- dynamic parameters viz: Gibbs free energy change (ΔG°), heat of sorption (ΔH°) and entropy change (ΔS°) for sorption of Sr(II) on PAN/zeolite composite were calculated for the system.

3. Results and Discussion

3.1. Adsorption Experiments

The effects of various parameters such as initial strotium concentration, pH, contact time and temperature were investigated.

3.1.1. Effect of Initial Concentration

Sorption of Sr ions over the surface of the composite adsorbent was studied at the different concentrations (25 - 175 µg/mL) of strontium ions at 25˚C. **Figure 1** shows the adsorption yields for Sr ions on the composite adsorbent as a function of the initial strontium concentration. As shown in **Figure 1**, an initial rise of sorption attained an apparent saturation in 50 µg/mL of strontium concentration and percentage of adsorption increased with increasing of strontium concentration. The percentage adsorption remained almost constant for strontium solution concentrations of 150 - 175 µg/mL.

3.1.2. Effect of pH

The pH of solution has been identified as one of the most important parameters affecting metal ion sorption. The effect of pH on the adsorption capacity of PAN/zeolite was investigated using solution of 150 µg/L Sr (II) for a pH range of 2.0 - 8.0 at 25˚C for 120 min. The pH of the solution was adjusted with 1M NaOH and 1M HNO_3. After the sorption equilibrium, the supernatant solutions were filtered and the concentrations of strontium were determined spectrophotometer. The adsorption percentage of strontium on the composite adsorbent was calculated.

Figure 2 shows the effect of pH, ranging from pH 2 to pH 8 on the removal of Sr by composite adsorbent. As seen from **Figure 2**, the highest adsorption levels for composite between pH 5 ± 0.1 and 6 ± 0.1 which indicate that a high affinity for strontium ions is predominant in this region. Therefore, pH 5 ± 0.1 was selected for further experiments with 84.6% adsorption yield.

According to strontium(II), hydrolysis constantsthe distribution of strontium species in demineralized water at the pH range from 1 to 11 can be present in the form of Sr^{2+} and a very negligible $Sr(OH)^+$ which increases in concentration above 10 and becomes the predominant specie above pH 13. Therefore, at the pH range of 2 - 8, strontium (II) has been found to be in the form of Sr^{2+} [31]. When pH is low (pH 2), the adsorbent almost has no more affinity to Sr(II) ions. Thus, the strong acidity results in replacing the adsorbed Sr(II) ions by the H^+, which will decrease the adsorption capacity of Sr(II) ions. When pH values increase beyond 6.1 ± 0.1, precipitation starts due to the formation of complexes in aqueous solution and then the adsorption decreases [21,32].

3.1.3. Effect of Contact Time

In order to compare the adsorption ability of composite adsorbent, the effect of contact time on the adsorption of

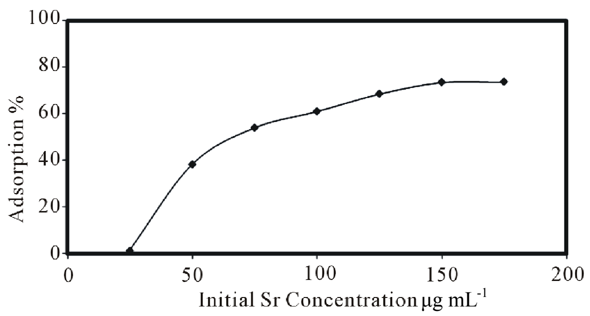

Figure 1. Effect of initial strontium concentration on the adsorption.

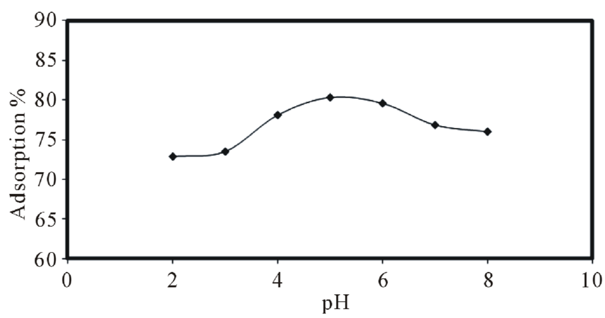

Figure 2. Effect of pH on the strontium adsorption.

Sr(II) was investigated. **Figure 3** shows the variation of percentage adsorption with shaking time. As demonstraed in **Figure 3**, the adsorption yield of Sr(II) onto composite adsorbent slightly increased with an increasing the contact time. These results show that the sorption process is rapid and equilibrium is reached almost instantaneously after mixing; no significant change occurs up to 240 min. Therefore, 20 min reaction time was selected and used for all further studies. The instantaneous uptake of strontium by PAN/zeolite composite may be due to adsorption and/or exchange of the metal ions with some ions on the surface of the adsorbent.

3.1.4. Effect of Temperature

Temperature has a pronounced effect on sorption. So as to investigate the effect of temperature, the sorption of Sr (II) ions onto composite adsorbent was studied in the temperature range of 293 - 333 K under optimized conditions. **Figure 4** illustrates the effect of temperature for adsorption of Sr(II) ions on the composite adsorbent. It is shown that the adsorption of Sr(II) on PAN/zeolite composite decreased as the solution temperature increased. This can be explained by the exothermic spontaneity of the adsorption process and by the weakening of bonds between Sr(II) and active sites of adsorbents at high temperatures.

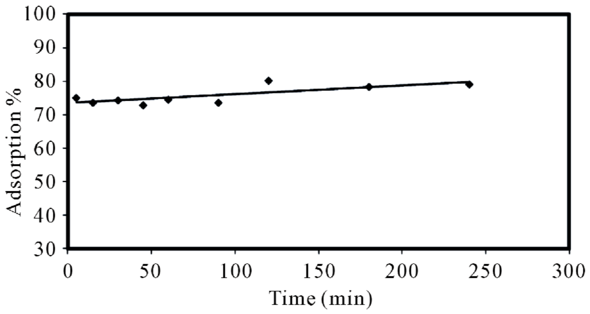

Figure 3. Variation of percentage sorption of strontium as a function of time.

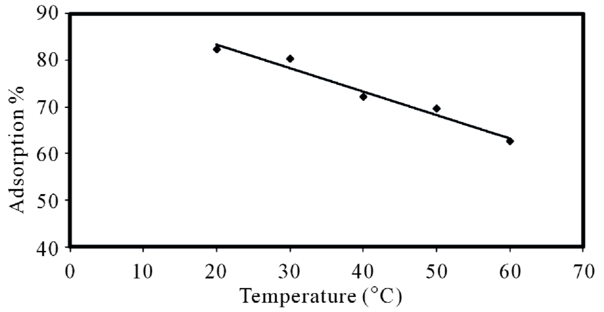

Figure 4. Effect of temperature on the strontium adsorption.

3.2. Adsorption Isotherms

Equilibrium data, commonly known as adsorption isotherms, are basic requirements for the design of adsorption systems. The distribution of metal ions between the liquid phase and the adsorbent is a measure of the position of equilibrium in the adsorption process and can generally be expressed by one or more of a series of isotherm models. In order to choose the best isotherm model to represent Sr(II)-PAN/zeolite adsorption system, a set of equilibrium data has been tested on the Langmuir, Freundlich, D-R and Temkin isotherm models, respectively. The model equations are shown below (Equations 2-5):

$$C_e/q_e = C_e/Q^\circ + 1/bQ^\circ \tag{2}$$

$$\log q_e = \log K_f + 1/n \log C_e \tag{3}$$

$$\ln C_{ads} = \ln X_m - \beta\varepsilon^2 \tag{4}$$

$$q_e = B_1 \ln K_T + B_1 \ln C_e \tag{5}$$

where q_e is the adsorption capacity in equilibrium (mg/g), C_e is the sorbate equilibrium concentration (mg/L), b (L/mg) is the Langmuir constant associated to energy of adsorption and Q° denotes the theoretical monolayer adsorption capacity (mg/g). K_f is the Freundlich constant (mg/g) while 1/n represents dimensionless heterogeneity factor. C_{ads} is the amount of sorbate sorbed by the composite (mol/g), X_m is the maximum sorption capacity of sorbent (mol/g) under investigation, β is a constant (kJ^2/mol^2) related to energy and *ε* is polany potential which is mathematically equal to $RT\ln(1+1/C_e)$, R is the universal gas constant in kJ/molK, T is absolute temperature in Kelvin and C_e is the equilibrium concentration of Sr(II) in solution (mol/L) and β (mol^2/kJ^2) indicates the activation energy, E (kJ/mol) of adsorption per molecule of sorbate when it is transferred to the surface of the solid from infinity in the solution, where $E = 1/\sqrt{-2\beta}$

Prior to equilibrium data plotting, all model equations were linearized accordingly [33].

The Temkin isotherm equation assumes that the heat of adsorption of all the molecules in layer decreases linearly with coverage due to adsorbent-adsorbate interactions, and that the adsorption is characterized by a uniform distribution of the bonding energies, up to some maximum binding energy [34]. The Temkin isotherm equation is given in Equation (5) above. B_1 is the Temkin constant related to heat of adsorption (kJ/mol). K_T is the equilibrium binding constant (L/mol) corresponding to the maximum binding energy. A plot of q_e vs. $\ln C_e$ at studied temperature is given also in **Figure 5**.

The values of the constants for Langmuir, Freundlich, D-R and Temkin isotherm models at 303 K for the removal of Sr(II) onto PAN/zeolite are presented in **Table 1**. The R^2 value range obtained for the Temkin model is high when compared to those of the Langmuir, Freundlich and D-R models. Therefore the Temkin model is most suitable and that applicability follows the order; Temkin > D-R > Freundlich > Langmuir adsorption models. This is a sign of a strong interaction between strontium ions and the adsorbents surface. The Temkin model shows a strong conformation to experimental data judging on satisfactorily values of the obtained linear regression coefficient, R^2 (**Table 1**) [33,35,36].

3.3. Adsorption Thermodynamics

The thermodynamic parameters viz: Gibbs free energy change (ΔG°), heat of sorption (ΔH°) and entropy change (ΔS°) for sorption of Sr(II) on PAN/zeolite composite system. These parameters were calculated from following equations:

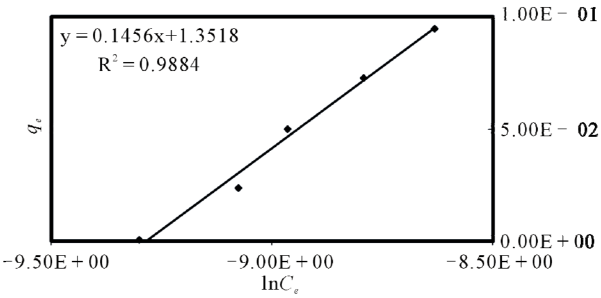

Figure 5. Temkin plot for the adsorption of Sr(II) by PAN/zeolite: pH: 5; adsorbent dosage, 150 μg/L; contact time, 20 min.

Table 1. Adsorption isotherm constants for the adsorption of Sr(II) onto PAN/zeolite (at 293 ± 2 K, pH 5).

Model	Linearized equuation	Parameters values for the adsorption of Sr(II) on PAN/zeolite
Freundlich	$\log q_e = \log K_f + 1/n \log C_e$	$K_f = 7.31\times10^{-13}$ mg/g ; $1/n = 7.6642$; $R^2 = 0.7729$
Temkin	$q_e = B_1 \ln K_T + B_1 \ln C_e$	$K_T = 0.03991$ L/mg ; $B_1 = 3.5384$; $R^2 = 0.9884$
D-R	$\ln C_{ads} = \ln X_m - \beta\varepsilon^2$	$\beta = 6.04\times10^{-7}$ (mol/J) ; $X_m = 6.4695$ mmol/g ; $E = 0.91$ kJ/mol ; $R^2 = 0.8407$
Langmuri	$C_e/q_e = C_e/Q^\circ + 1/bQ^\circ$	$Q^\circ = 0.011$ mg/g ; $b = 0.00237$; $R^2 = 0.4838$;

$$\Delta G^\circ = -RT \ln K_d \quad (6)$$

where, R is the universal gas constant (8.314 J/molK), T is temperature (K) and $K_d\left(q_e C_e^{-1}\right)$ is the distribution coefficient [37,38]. The enthalpy (ΔH°) and entropy (ΔS°) parameters were estimated from the following Equation:

$$\ln K_d = \Delta S^\circ/R - \Delta H^\circ/RT \quad (7)$$

According to Equation (7), the values of ΔH° and ΔS° can be calculated from the slope and intercept of the plot of $\ln K_d$ vs $1/T$ yields, respectively (**Figure 6**). Results are shown in **Table 2**.

The negative ΔH° indicates the exothermic nature of the adsorption process and the positive value of ΔS° suggests the increase in randomness at the solid/solution interface during the adsorption process. This occurs as a result of redistribution of energy between the adsorbate and the adsorbent [35]. The negative value ΔG° indicates the degree of spontaneity of the process and higher negative value reflects a more energetically favorable sorption. The negative value of Gibbs free energy for Sr(II) adsorption on PAN/zeolite indicated the feasibility of the process and spontaneous nature of the adsorption. The degree of spontaneity of the process also increased with increasing temperature [39].

4. Conclusions

The present study focuses on adsorption of Sr(II) from aqueous solutions using the PAN/zeolite composite as a low cost sorbent. The adsorption characteristic has been examined with the variations in the parameters of concentration of Sr(II), pH, contact time and temperature.

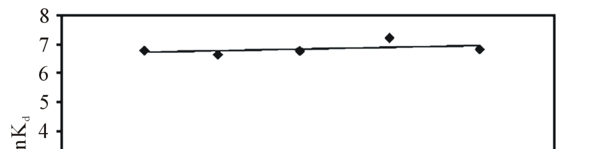

Figure 6. A plot against $\ln K_d$ to $1/T$ for removal of Sr(II) from PAN/zeolite composite.

Table 2. Thermodynamic parameters for the adsorption of Sr(II) on PAN/zeolite composite.

ΔH°, kJ/mol	ΔS°, kJ·mol/K	ΔG°, kJ/mol				
		293 K	303 K	313 K	323 K	333 K
–5.20	0.04	–16.92	–17.32	–17.72	–18.12	–18.52

The pH experiments showed that the governing factors affecting the adsorption characteristics of adsorbent. Sorption is relatively high at pH 5. This phenomenon can be explain by competition of the H^+ ions with strontium ions at low pH values and precipitation of hydroxyl species onto the adsorbents (pH 6 - 8) at higher pH.

The experimental results were analyzed by using the Langmuir, Freundlich, D-R and Temkin equations. The Temkin model appears to be the best fitting model for Sr(II) sorption on the adsorbent due to its high regression coefficient, R^2 (0.9884). Also thermodynamic results support this suggestion. Thermodynamic parameters revealed that the adsorption process is exothermic and spontaneous with a increased randomness in nature.

The results provide information for the implementation of permeable reactive barrier technology to control the transport of radioactive Sr(II) and its species in natural surface and groundwater.

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