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Enhancing the adsorption of strontium (II) using TOPO impregnated Dowex 50 W-X8 resin

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In this research work, the adsorption of strontium ions from aqueous solutions was studied using Dowex 50 W-X8 resin loaded with TOPO extractant. This study introduces a novel TOPO-impregnated adsorbent with potential applications for efficient and cost-effective nuclear wastewater treatment. The influence of effective operational parameters such as initial pH of the solution, adsorbent dosage, initial strontium concentration in the solution, and contact time was evaluated. In order to investigate the kinetics of the adsorption process, pseudo-first-order and pseudo-second-order models were used. The results indicated a very good agreement between the experimental data and the pseudo-first-order kinetic model. Langmuir and Freundlich models were used to model the equilibrium of the adsorption. Langmuir isotherm model showed good agreement with the experimental data. The maximum adsorption capacity of Dowex 50 W-X8 resin loaded with TOPO for strontium sorption was obtained 163.43 mg/g of adsorbent calculated by the Langmuir model. The result of the present study showed that Dowex 50 W-X8 resin loaded with TOPO extractant can be an effective adsorbent for the sorption of strontium from aqueous solutions and industrial wastewater.

Keywords Adsorption, Strontium, Dowex 50 W-X8 resin, TOPO extractant

The management of the entry of radioactive materials into the environment is one of the critical issues in various industries, especially in the nuclear industry[1,2]. Strontium-90 is one of the most important radionuclides that can pollute the environment. This radionuclide with a half-life of 28.9 years is a beta emitter that is produced during nuclear fission reactions[3]. Strontium, particularly its radioactive isotopes, is utilized in the nuclear industry as a source of energy and in the production of chemical materials, ceramics, glass production, and health sciences[4–6]. However, strontium can have detrimental effects on the environment and living organisms, as its accumulation in the food chain and contamination of water and soil from human activities can lead to chronic poisoning and ecological disturbances[3,7].

Numerous techniques exist for eliminating strontium and other metal ions, including processes such as chemical precipitation[8], oxidation and reduction[9], membrane technology[10], solvent extraction[11], ion exchange and adsorption[12–24].

Ion exchange resins have been widely used for the removal of heavy metals, including strontium, from industrial wastewater due to their high efficiency, easy handling, and regeneration capability[25]. Dowex 50 W-X8, a strong acid cation exchange resin with sulfonic acid functional groups, has shown promising results for the adsorption of strontium[26–28]. Given that Dowex 50 W-X8 resin impregnated with TOPO (trioctylphosphine oxide), as a solvent-impregnated resin (SIR) has not yet been investigated for strontium adsorption, and considering TOPO's demonstrated ability to enhance adsorption capacity compared to other solvents, this study evaluates its potential for strontium adsorption from aqueous solutions. TOPO is a neutral organophosphorus compound that can form stable complexes with metal ions, thereby increasing the adsorption capacity and selectivity of the resin[29,30]. Lee et al.[31] used TOPO to enhance the adsorption capacity of PVA (polyvinyl alcohol) for strontium removal from aqueous solution and obtained satisfactory results. Studies have shown that the use of extractants as modifiers can improve the adsorption properties of resins[21]. SIRs have several advantages over conventional extraction systems including the selectivity of extractants, ease of phase separation, easy recovery of the loaded materials in the desorption step, and the wide variety of extractants[32].

The purpose of this research work is to investigate the adsorption potential of Dowex 50 W-X8 resin impregnated with TOPO extractant as a new SIR adsorbent for strontium removal from aqueous solutions and also to evaluate the effect of impregnation on the adsorption capacity of the resin. In this regard, the effect of the main effective parameters on the adsorption process such as initial pH of the solution, adsorbent dosage, initial

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strontium concentration in the solution, and contact time have been investigated. Also, the kinetics and the equilibrium of the adsorption process have been studied.

Research theories

First- and second-order reaction rate kinetic models commonly used to describe the rate of the adsorption processes. The pseudo - first and -second-order kinetic models are as follows respectively[33]:

$$q_t = q_e\left(1 - e^{-k_1 t}\right) \tag{1}$$

$$q_t = \frac{k_2 q_e^2 t}{1 + k_2 q_e t} \tag{2}$$

where q_e and q_t (mg/g) are the amount of strontium adsorbed at equilibrium and at time t (min), respectively, k_1 (1/min) and k_2 (g/mg.min) show the equilibrium rate constant of pseudo first and second order adsorption, respectively.

The Langmuir and Freundlich isotherms can be used to determine the relationship between adsorbed strontium ions on the SIR adsorbent (q_e) and unadsorbed strontium ions in solution (C_e). The Langmuir and Freundlich isotherms are given by the following equations, respectively[33]:

$$q_e = \frac{q_m b C_e}{1 + b C_e} \tag{3}$$

$$q_e = K_F {C_e}^{1/n} \tag{4}$$

where q_e (mg/g) is the amount adsorbed at the equilibrium, C_e (mg/L) is the equilibrium concentration, q_m (mg/g) is the Langmuir constant representing the maximum monolayer adsorption capacity and b (L/mg) is the Langmuir constant related to the energy of adsorption and the affinity of the binding sites, K_F (L/g) is the Freundlich constant which is related to the adsorption capacity, and n is the dimensionless Freundlich constant.

Materials and methods

Materials

Strontium nitrate ($Sr(NO_3)_2$ salt for the solution of metal ions, Sodium hydroxide (NaOH), Nitric acid (HNO_3) used to adjust the pH of the solution were all prepared by Merck. Organic solvent toluene ($C_6H_5CH_3$) with 99.99% purity was purchased from Merck and used as a diluent. TOPO as extractant was supplied by the Sigma-Aldrich. The Dowex 50 W-X8 resin used in this work was purchased from Sigma-Aldridge. Dimensions of the resin are 90 to 250 microns, its density are equal to 0.8 g/cm^3, exchange capacity of 640–720 g/kg water. All chemicals used in the present study were of analytical grade.

Instrumentation

An Italian-made Gallenkamp mechanical shaker was used to stir the solutions. To measure the solution concentration of Sr (II) ions, the inductively coupled plasma-atomic emission spectrometry (ICP-AES) device model PerkinElmer, Optima 7300 DV. USA, was used. To determine the pH of the solution, a pH meter of the Swiss model 780 was used. Characterization of the impregnated resins (SIRs) using the fourier transform infrared (FTIR), PerkinElmer Spectrum, 10.30.06, USA, between 500 and 4000 cm^{-1} wavenumber was obtained. For the morphology scanning electron microscopy (SEM), VEGA/SEM, Tescan, A.S, Czech Republic was used. The BET analysis was done using Quantachrome NOVA 2200 USA, instrument.

Impregnation

To impregnate Dowex 50 W-X8 resin with TOPO extractant, first, 50 g of resin were washed three times with deionized water to remove impurities and prepare the internal pores of the resin for the impregnation process. Then the washed resin was filtered and completely dried at room temperature for 24 h. Then, 10 g of dry resin was contacted with 30 and 60 g of TOPO in a 0.5 M toluene solution at room temperature using a constant speed stirrer for two hours[1]. Then the resin beads were separated from the organic phase and washed several times with 2 M nitric acid. Finally, the impregnated resin beads were washed with deionized water and completely dried for 24 h at room temperature.

Adsorption experiments

To carry out the strontium adsorption process using Dowex 50 W-X8 resin with and without impregnation with TOPO, a certain amount of adsorbent was added to a set of 250 mL Erlenmeyer flasks containing 100 mL of strontium nitrate solution with predetermined concentrations. The flasks were shaken for a certain period in the incubator shaker at a speed of 150 rpm and a temperature of 25 °C. After the adsorption process was completed, Whatman filter paper was used to filter the strontium solution. Sampling was done at specified time intervals and the remaining strontium concentration was measured by ICP-AES device. To minimize error, all experiments were repeated twice, and the average value was reported.

The amount of adsorbed metal ions per unit mass of resin (mg/g) was calculated using the following Eq. (6).

$$\mathrm{q} = (C_i - C_e) \times \frac{V}{M} \tag{5}$$

where C_i and C_e (mg/L) is the initial and the equilibrium concentrations, respectively. V (L) is the volume of test solution and M (g) is the amount of the adsorbent in the solution. The percentage of metal ion adsorption was calculated by Eq. (7).

$$\% \text{ Adsorption} = \frac{C_i - C_e}{C_i} \times 100 \quad (6)$$

Results and discussion
Characterization
FTIR spectra of TOPO and Dowex 50 W-X8 resin before and after impregnation with TOPO extractant, and after adsorption process are shown in Fig. 1. The range of these spectra are between 500 and 4000 cm^{-1}. In these spectra, the peak observed at 2932.9 cm^{-1} is related to C-H stretching. The stretch 1647.2 cm^{-1} is related to C = C in the alkene group and the peak 1413 cm^{-1} is related to the stretch of C = C ring. The peaks 1185 cm^{-1}

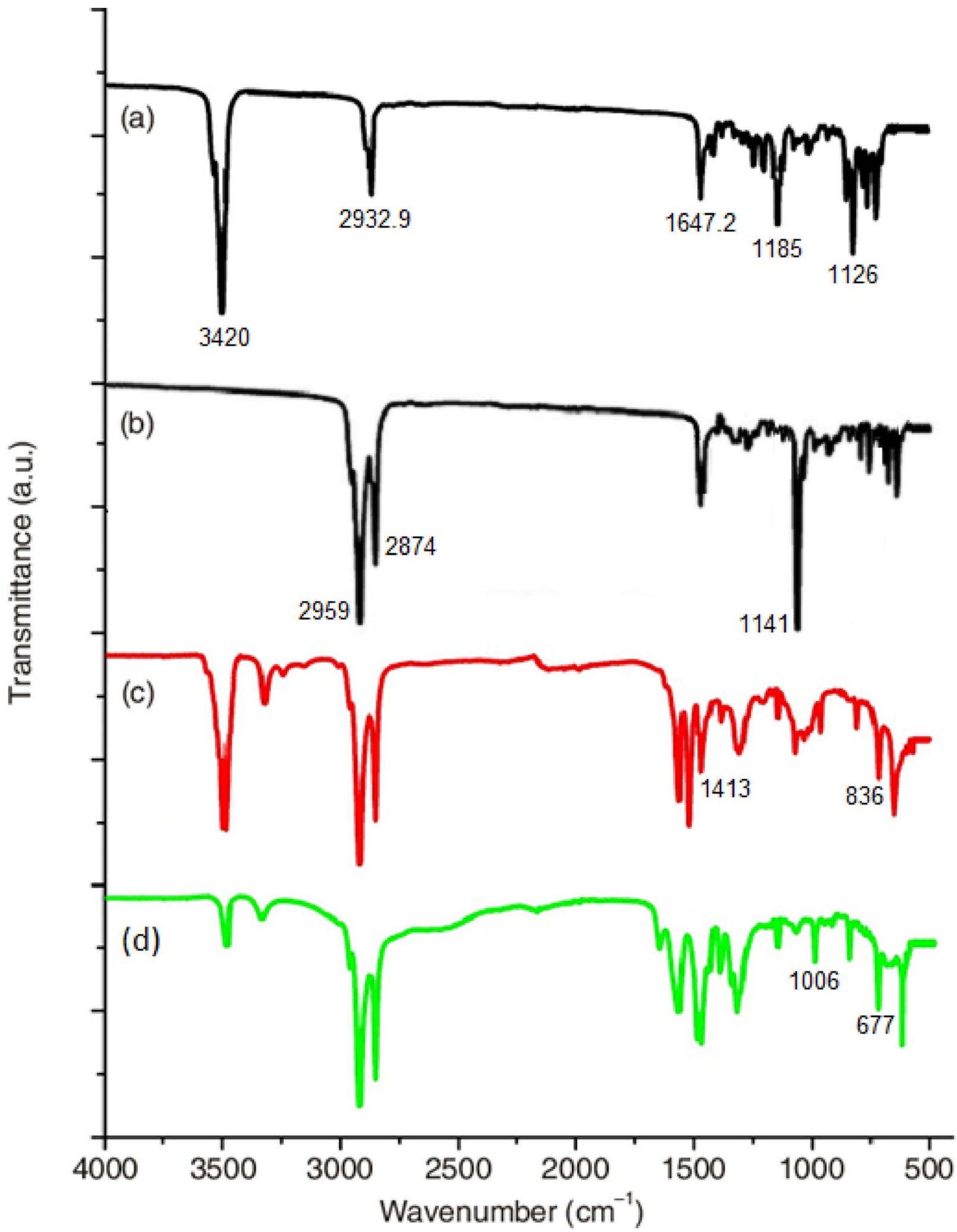


Fig. 1. FTIR spectra of Dowex 50 W X-8 resin (**a**), TOPO (**b**) Dowex-TOPO before (**c**) and after strontium adsorption (**d**).

and 1126 cm^{-1} are for S = O and S-OH, respectively. Two peaks of 1006 cm^{-1} and 1035 cm^{-1} are related to S-O-C stretching. The C = C bond in the alkene appeared in the 836 cm^{-1} peak. The stretching caused by C-S can be seen in the peak of 677 cm^{-1}. The spectra of 2959 cm^{-1} and 2874 cm^{-1} are related to asymmetric and symmetric stretching of CH_2, and a band at 3420 $cm^{−1}$ ascribed to hydroxyl –OH group stretching. Also, the stretching vibration of P = O at the peak of 1141 cm^{-1}. Comparing the spectra of resin and TOPO with the spectrum of the sample after impregnation indicates the successful impregnation of the resin. (Fig. 1a,b,c)[2]. Comparing the FTIR spectra before and after the adsorption of strontium ions (Fig. 1c,d), it is observed that the peaks in the region of 2900–3500 cm^{-1} have been changed. Change and/or shift of the majority of bands indicates that the adsorption of strontium on the impregnated resin has occurred successfully.

SEM analysis was used to study the physical structure of the resin surface and its changes due to impregnation and then strontium adsorption. According to Fig. 2, Dowex 50 W-X8 has a smooth and flat surface (Fig. 2a), while its surface after impregnation has a rougher surface due to impregnation with trioctylphosphine oxide (Fig. 2b). Figure 2b shows that the surface of the resin has been modified with TOPO and the solvent has filled almost all the pores. It seems that the strontium ions are sitting in the form of bright spots on the impregnated adsorbent surface (Fig. 2c).

BET analytical technique was employed to evaluate the specific surface area and pore volume. The surface area and pore size distributions of Dowex 50 W-X8 and Dowex-TOPO were analyzed by N_2 adsorption–

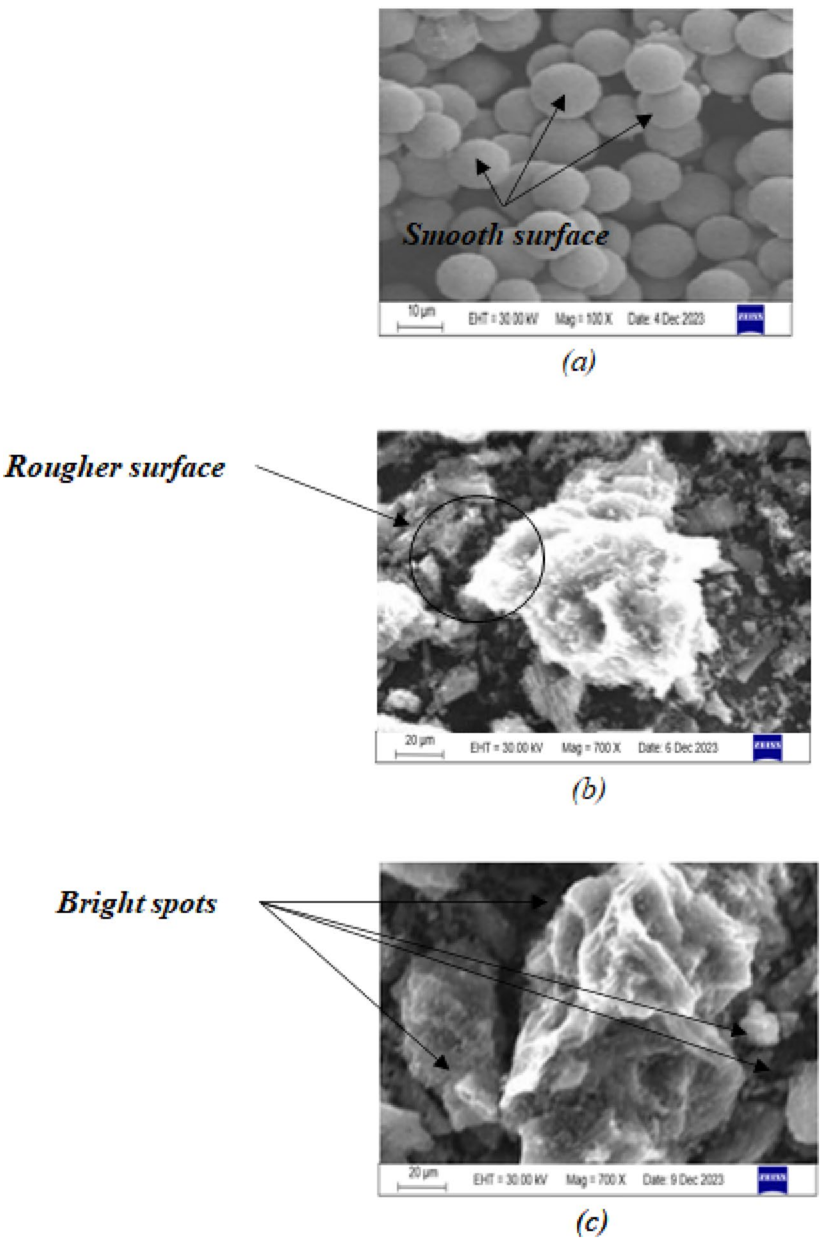


Fig. 2. SEM images of Dowex 50 W X-8 resin before (**a**) and after (**b**) impregnation, and after strontium adsorption (**c**).

Resin	BET surface area (m^2/g)	BJH adsorption average pore volume(cm^3/g)	Adsorption pore diameter (Å)
Dowex 50 W-X8	10.04	0.011	22.31
Dowex-TOPO	1.29	0.0047	35.95

Table 1. BET analysis for resin berfore and after impregnation (0.496 g/g SIR).

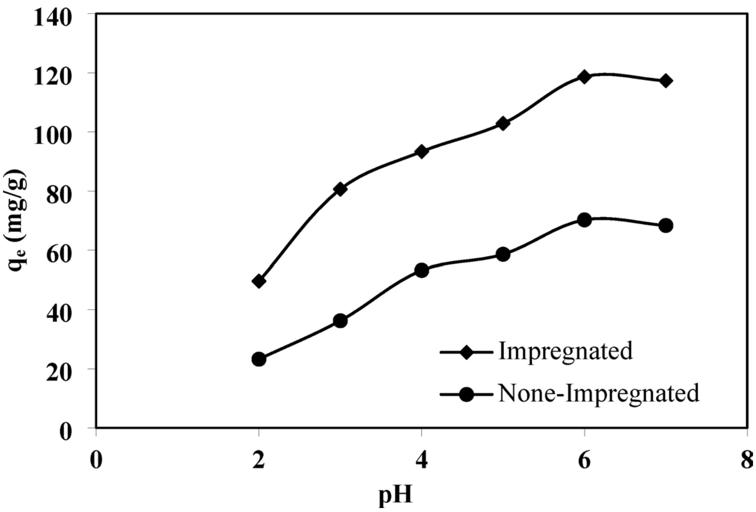


Fig. 3. Effect of pH on strontium adsorption by Dowex 50 W-X8 with and without impregnation (C_o: 90 mg/L, Adsorbent dosage: 0.5 g/L, contact time: 24 h).

desorption isotherms are shown in Table 1. According to the results, after the impregnation process, BET surface area and BJH pore volume of the Dowex 50 W-X8 resin decreased due to the filling of its pores by the TOPO extractant. Therefore, increasing the pore diameter of the resin after impregnation from 22.31 to 35.95 Å can be an explanation for filling the internal cavities of the particles.

Effect of pH

The effect of pH on the adsorption of strontium from aqueous solutions was investigated using Dowex 50 W-X8 resin and resin impregnated with TOPO in the range of 2 to 7. Figure 3 shows the amount of strontium adsorption by these two adsorbents. According to the figure, the maximum amount of adsorption of both adsorbents occurs at pH 6. Also, the comparison of strontium adsorption curves by these two adsorbents shows that impregnation of the adsorbent has a significant effect on increasing the adsorption rate. At lower pH values, the competition between H^+ and Sr^{2+} cations for the exchange sites on the adsorbent causes a noticeable reduction in the amount of strontium adsorption. With the increase of pH, the adsorption rate increases, and with the increase of pH higher than about 6, the adsorption rate starts to decrease. At alkaline pHs, adsorption was not investigated due to the possibility of strontium precipitation[34]. Therefore, in the next tests, pH 6 is considered as the optimal pH.

Effect of contact time

The adsorption process of strontium ions by Dowex 50 W-X8 were evaluated before and after impregnation in different time intervals, and the adsorption results are shown in Fig. 4. According to this figure, during the first 120 min of the adsorption process, the concentration of strontium adsorbed increases with time, and after this period, no significant change in the concentration of strontium is observed. Therefore, according to the results, the time to reach the adsorption equilibrium can be considered 120 min. Also, as expected, the amount of strontium adsorption by Dowex impregnated with TOPO is higher than Dowex without impregnation in all time intervals.

Effect of adsorbent dosage

The effect of the adsorbent dosage on the amount of adsorption and the removal percentage of strontium ions was investigated in the range of 0.1 to 1.2 g/L, and the results are shown in Fig. 5. According to the results, increasing the amount of adsorbent causes an increase in the percentage of adsorption because more sites for adsorption are available for metal ions. On the other hand, with increasing the adsorbent dosage, the amount of adsorption decreases because the amount of adsorption is defined based on the amount of unit weight of the adsorbent. Based on this, next series of adsorption experiments have been performed using an adsorbent dosage of 0.5 g/L.

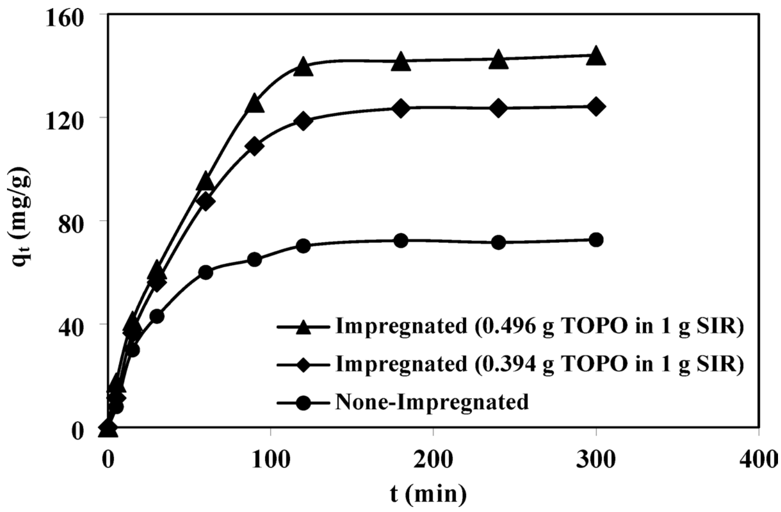


Fig. 4. Effect of contact time on strontium adsorption by Dowex 50 W-X8 with and without impregnation (pH: 6, Co: 90 mg/L, Adsorbent dosage: 0.5 g/L).

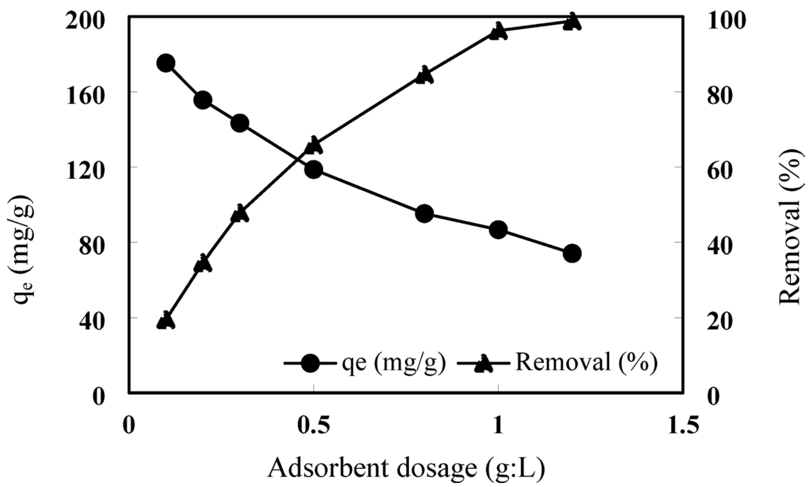


Fig. 5. Effect of adsorbent dosage on equilibrium sorption of strontium by TOPO impregnated Dowex 50 W-X8 (pH: 6, Co: 90 mg/L).

Effect of initial solution concentration

Figure 6 shows the results of investigating the effect of the initial concentration of strontium on its adsorption amount for adsorbents with and without impregnation. According to the figure, as the initial concentration of strontium increases, the amount of adsorption increases. The increase in the amount of adsorption with the increase in initial concentration is due to the availability of more adsorbent sites for the adsorption of strontium ions, which gradually approaches a constant value. At higher initial solution concentrations, due to the saturation of the adsorbent active sites, the amount of adsorption remains constant.

Effect of impregnation ratio

To investigate the effect of resin impregnation ratio on the amount of strontium adsorption, experiments were performed in three series, i.e., without impregnation, impregnation with ratios of 0.394 and 0.496 g/g SIR. In Fig. 6, it can be seen that the adsorption amount has been significantly increased by the impregnation. According to the figure, with the increase in the impregnation ratio (extractant/resin) in the impregnation process, the amount of adsorption has increased. This phenomenon indicates that in the impregnated resin, both the polymeric support (Dowex 50 W-X8) and the solvent (TOPO) are involved in the adsorption of strontium and increase the amount of adsorption compared to the resin without impregnation. Also, according to Fig. 6, with the increase of impregnation ratio from 0.394 to 0.496 g/g SIR, the amount of adsorption has also increased from 119.17 to 142.36 mg/g.

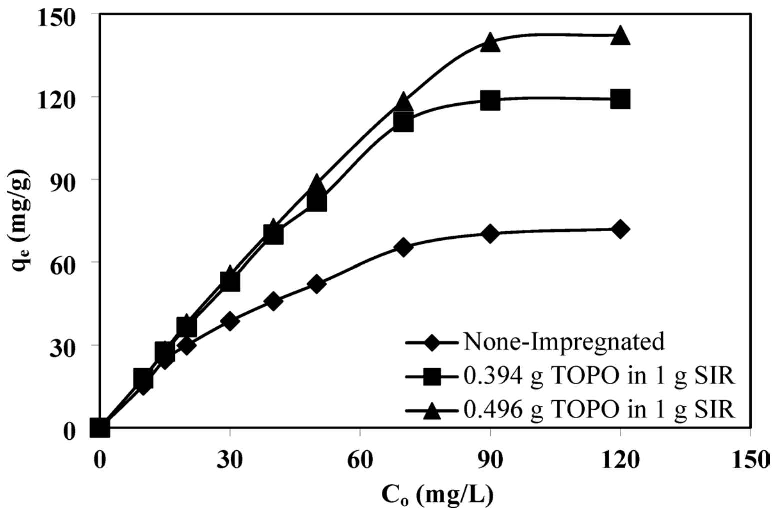
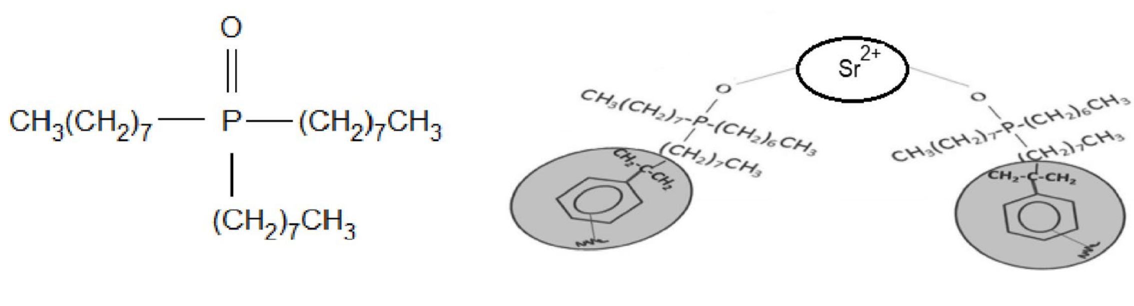


Fig. 6. Effect of impregnation ration on equilibrium sorption of strontium at different initial metal concentrations (pH: 6, Adsorbent dosage: 0.5 g/L).



(a) (b)

Fig. 7. The TOPO chemical structure (**a**) and simple representation for the strontium ions complexing with TOPO.

Sorption mechanism

In order to explain the mechanism of binding of strontium (II) to the active sites of adsorbent surfaces, it is essential to know the chemistry of strontium solution. The distribution of Sr species depends on both the total concentration of strontium ions in solution and the pH of the equilibrium solution. Knowledge of speciation is important for determining the interactions of a species with SIR or ion exchange materials. Strontium may exist in three main forms in aqueous solution. The speciation of strontium can be interpreted based on the following two equations that represent its sequential hydrolysis in solution[35]:

$$Sr^{2+}_{(aq)} + OH^{-}_{(aq)} \leftrightarrows SrOH^{+}_{(aq)} \quad (7)$$

$$SrOH^{+}_{(aq)} + OH^{-}_{(aq)} \leftrightarrows Sr(OH)^{0}_{2(aq)} \quad (8)$$

The obtained results showed that by increase of OH^- (with increasing pH) the divalent Sr ions (Sr^{2+}) shift to monovalent ions ($Sr(OH)^+$) and with further increasing pH shift to $Sr(OH)_2$.

The chemical formula of TOPO is $C_{24}H_{51}OP$ and its chemical structure is shown in Fig. 7a. TOPO contains an oxygen ion in the oxygen-phosphorus bond, which can be seen in the FTIR graph with a peak at 1141 cm[-1] corresponding to the P=O stretching vibration (Fig. 1). Divalent strontium ions (Sr^{2+}) can potentially form complexes with oxygens present in two TOPO molecules (Fig. 7b). However, monovalent $Sr(OH)^+$ ions require only one TOPO molecule to form the complex. Therefore, complexing of monovalent ions with TOPO is easier than that of divalent ions. Given that at higher pHs, according to solution chemistry, the presence of monovalent ions is greater than that of divalent ions and the higher accessibility of adsorption sites for monovalent ions compared to divalent ions[36], the adsorption capacity increases. These evidences suggest that the main adsorption mechanism in the adsorption of Sr(II) ions by Dowex-TOPO SIR is ion exchange by the resin and complex

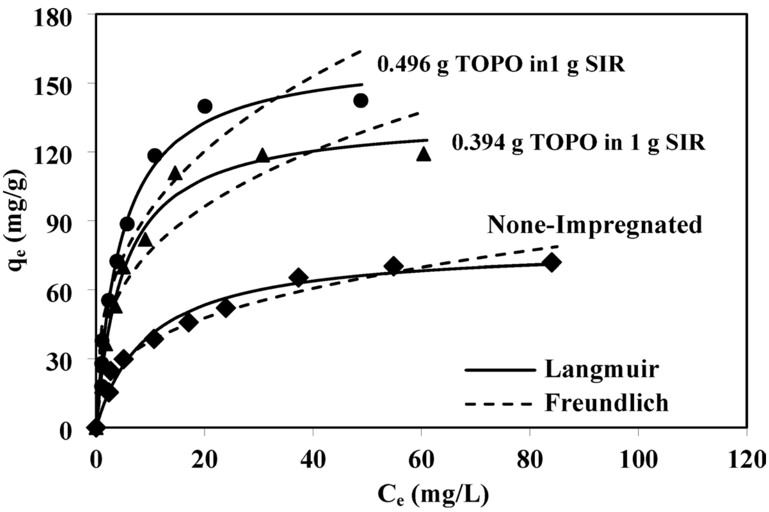


Fig. 8. Adsorption isotherms of Sr^{2+} ion adsorbed onto none-impregnated Dowex 50 W-X8, 0.394 g TOPO in 1 g SIR, and 0.496 g TOPO in 1 g SIR adsorbents (pH: 6, Adsorbent dosage: 0.5 g/L).

Model	Parameter	Adsorbent		
		None-impregnated	0.394 g TOPO in SIR	0.496 g TOPO in SIR
Langmuir	q_m (mg/g)	79.36	135.25	163.43
	b (L/mg)	0.1028	0.2010	0.2157
	R^2	0.9891	0.9911	0.9909
	RMSE	0.1275	0.2560	0.3860
Freundlich	K_F (L/g)	16.95	36.49	42.52
	n	2.8938	3.0913	2.8819
	R^2	0.9728	0.9034	0.9020
	RMSE	0.1855	0.9280	1.2340

Table 2. Adsorption isotherm parameters.

formation by the extractant. Other researchers have also reached similar results for the metal ions, chromium and lanthanum[37,38].

Equilibrium modeling

Experimental equilibrium data were modeled using Langmuir and Freundlich isotherms (Fig. 8). The constants of isotherms obtained from non-linear modeling are presented in Table 2. According to the results (R^2 and RMSE values), the Langmuir isotherm model has better compatibility with the experimental data, which means that the adsorption mechanism can be considered as a monolayer and the adsorbent surface is homogeneous in terms of adsorption energy. The physical significance of the better fit of strontium adsorption on to the studied SIR with the Langmuir model is only a monomolecular layer of noninteracting solutes is formed dynamically on this adsorbent surface, with all adsorption sites being of equal adsorption energy[39]. The Langmuir model predicts the maximum adsorption capacities of 79.36, 135.25, and 163.43 mg/g for the none-impregnated, 0.394 g TOPO in 1 g SIR, and 0.496 g TOPO in 1 g SIR adsorbents, respectively.

The obtained maximum adsorption capacity of this impregnated resin is compared with the results of strontium adsorption by the other similar adsorbents and shown in Table 3. Based on the data presented in this table, the maximum adsorption capacities of the adsorbents examined in this study are significant.

Kinetic modeling

The kinetic study of the adsorption process of strontium ions by impregnated resin shows that this adsorption process is relatively fast at the beginning, with approximately 70% of the adsorption occurs in the first 50 min. Experimental kinetic data were modeled using non-linear pseudo-first-order and pseudo-second-order models (Fig. 9). The obtained constants of kinetic models are presented in Table 4. According to the results (R^2 and RMSE values), the experimental data show better agreement with the pseudo-first-order kinetic model for both impregnated and nonimpregnated Dowex 50 W-X8 resin. The impregnation of resin with a solvent, i.e. TOPO in this research, can alter the surface properties of the resin. These changes can lead to an increase in adsorption capacity, as the solvent may cause the resin's surface properties to open up, thereby increasing the contact surface

Adsorbent	q_m (mg/g)	pH	T (°C)	Reference
TiO2/DCH18C6/octanol	48.97	[HNO3] = 6 M	25	Ma et al.[40]
Hybrid gel	77.11	2–12	20	Li et al.[41]
Zr-Sb oxide/PAN	43.67	4.7	60	Cakir et al.[42]
Almond green hull	116.53	6	22	Hamza et al.[43]
Sulfonylcalix arenetetrasulfonate loaded Resin	76.43	7	25	Liu et al.[44]
Dowex 50 W-X8 resin	79.36	6	25	Present work
Dowex 50 W-X8 resin impregnated by TOPO	163.43	6	25	Present work

Table 3. Comparison of the strontium adsorption capacities of different adsorbents.

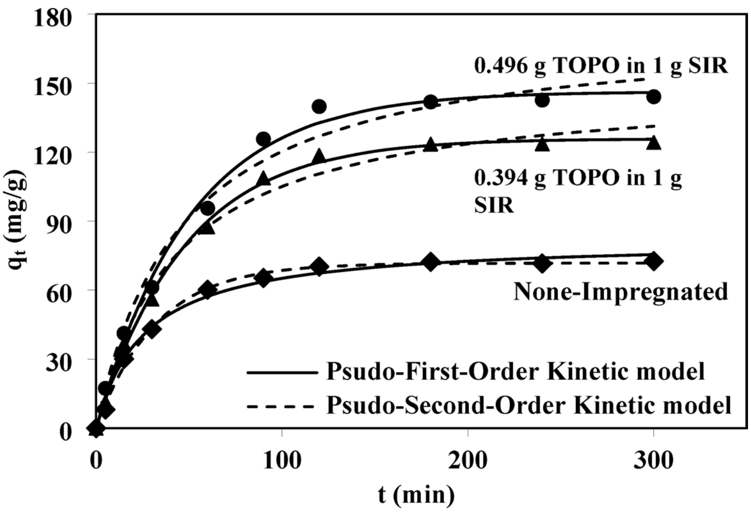


Fig. 9. Kinetics of strontium adsorption onto impregnated and nonimpregnated Dowex 50 W-X8 (pH: 6, Co: 90 mg/L, Adsorbent dosage: 0.5 g/L).

Model	Parameter	Adsorbent		
		None-impregnated	0.394 g TOPO in SIR	0.496 g TOPO in SIR
Pseudo-first-order	k_1 (1/min)	0.0312	0.0209	0.0198
	q_{eq} (mg/g)	71.76	125.88	146.37
	R^2	0.9968	0.9982	0.9945
	RMSE	0.0297	0.0500	0.0886
Pseudo-second-order	k_2 (g/mg.min)	4.71×10^{-4}	1.56×10^{-4}	1.25×10^{-4}
	q_{eq} (mg/g)	82.01	149.98	175.24
	R^2	0.9917	0.9888	0.9849
	RMSE	0.3357	0.5770	0.2307
Experimental	$q_{eq, ex}$	72.65	124.25	144.12

Table 4. Kinetic modeling parameters.

with the adsorbates. However, these changes may also result in a decrease in adsorption rate, as the diffusion path of the adsorbates into the resin becomes more complex. This is evident by comparing the results in Table 4.

Conclusion

Adsorption of strontium ions was performed using Dowex 50 W-X8 resin with and without impregnation with TOPO extractant and the effect of impregnation on the adsorption was investigated. The results showed that the impregnation of resin using TOPO significantly increases the adsorption capacity of the adsorbent. Also, the effect of effective operational parameters such as pH, contact time, adsorbent dosage, and solution initial concentration on adsorption was evaluated and their optimal values were determined. Kinetic investigation of strontium adsorption using this adsorbent shows that the kinetics follows the pseudo-first-order model. The adsorption equilibrium data showed a very good agreement with the Langmuir isotherm model. According to the findings of this study, it can be concluded that Dowex 50 W-X8 resin loaded with TOPO extractant can be an effective adsorbent to adsorb and remove strontium ions from real wastewater. The potential of this adsorbent for removing other radionuclides can also be studied.

Data availability

Data sets generated and/or analyzed during the current study available from the corresponding author on reasonable request.

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Author contributions

Both authors contributed equally to this work.

Declarations

Competing interests

The authors declare no competing interests.

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