Strontium(II) Ion Uptake on Poly(*N*-vinyl imidazole)-Based Hydrogels

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ABSTRACT: In this article, we report on the extraction of Sr(II) ions from aqueous solution with a series of poly(*N*-vinyl imidazole)-based hydrogels. The hydrogels were synthesized by the crosslinking of *N*-vinyl imidazole with four different crosslinkers with γ rays as initiators. The well-characterized hydrogels were used as Sr(II) sorbents. Sr(II) uptake was determined with a colorimetric method with Rose Bengal anionic dye. Scanning electron microscopy–energy-dispersive spectroscopy analysis of the Sr(II)-loaded polymers was recorded to ascertain the uptake of Sr(II) ions. The experimental adsorption values were analyzed with the Freundlich and Temkin equations, and the kinetics of adsorption were investigated with a pseudo-second-order sorption kinetic model. The results show that the equilibrium data fit well in the Freundlich isotherm and followed a pseudo-second-order kinetic model. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 124: 3721–3727, 2012

Key words: crosslinking; hydrogels; structure–property relations

INTRODUCTION

Hydrogels having specific functional groups are of great interest for the removal of toxic or radioactive elements from various effluents.[1–4] The removal of toxic and polluting metal ions from industrial effluents, nuclear plants, water supplies, and effluents from mines has received much attention in recent decades.[5–8] After Ca, Sr is the second most abundant metal ion in the Earth's crust. It is present in about 40 different minerals, with celestite ($SrSO_4$) and strontianite ($SrCO_3$) being most common among them.[9] Natural Sr is not radioactive and exists in four stable forms (or isotopes), ^{84}Sr, ^{86}Sr, ^{87}Sr, and ^{88}Sr. In addition, Sr also exists as radioactive isotopes, and ^{90}Sr is its most hazardous radioactive isotope. ^{90}Sr emits β particles and decays to yttrium (^{90}Y), which is also radioactive. ^{90}Sr is an important constituent of wastes produced from nuclear weapons production and is stored in underground tanks. ^{90}Sr is also formed in uranium fission reactions. The presence of these radioactive ions in water bodies poses a serious threat to human health.[10–12] The half-life of ^{90}Sr falls within the human timescale (29 years); hence, it is hazardous to human health. It is potentially mobile in ground water and bioavailable for organisms as a substituent of Ca^{2+} and K^+. Sr has long been known to cause rickets when fed to growing animals at a sufficiently high dose. The intake of Sr present in food products causes rickets in either calcium-replete[13] or calcium-deficient animals.[14] Milk and milk products are found to be significant contributors to the uptake of fallout radionuclides: more than one half of total ^{90}Sr may reach humans through the dairy pathway.[15] The risk of ^{90}Sr intake is mainly based on its carcinogenic and mutagenic mechanisms, problems that occur in cell division, and possible increased infant mortality.[16,17] ^{90}Sr decays to radioactive yttrium, which accumulates in hypophysis and ovaries and subsequently disrupts infant hormonal development and infant growth. The harmful effects of ^{90}Sr are caused by the high energy effects of radiation because ^{90}Sr is taken up into bone itself, and the soft tissues nearby may be damaged by radiation released over time.[18–20]

In view of these facts and also because of the incessantly increasing demand for nuclear material for energy production, effective processes and products/sorbents for the removal of hazardous radioactive ions from discharged wastewater are urgently required. Sorbents for the removal of heavy-metal ions from wastewater should exhibit high efficiency and selectivity in sorption and should be very stable to radiation, chemicals, and thermal energy as well as mechanical stress. Synthetic inorganic cation-exchange materials, such as synthetic micas,[10,11] niobate molecular sieves,[12] and titanate,[21] have been used for the removal of radioactive ions. A few selective separation processes of Sr(II) by liquid–liquid solvent extraction have been reported.[22–24] Synthetic exchange materials are superior to natural

materials in terms of selectivity for radioactive cations.[25] A polymer-based sorbent, Sr resin, synthesized by the impregnation of 40% w/w of a liquid extractant solution of 1M tBuCH18C6 in 1-octanol into the pores of an inert polymeric support (XAD-7) was reported.[26] Recyclable organometallic extractants were also reported for the removal of radioactive ^{90}Sr(II) ions from aqueous media.[27]

In continuation of our earlier work on the use of hydrogels for metal-ion uptake, in this article, we report Sr(II) uptake on poly(N-vinyl imidazole) [poly(N-VIm)]-based hydrogels. Recently, hydrogels based on poly(N-VIm) have been used for the adsorption of some toxic metals,[28,29] including Sr(II).[30] These hydrogels are of special interest, as imidazole is present in proteins and enzymes and poly(N-VIm) is a biocompatible polymer and its action as a sorbent is essentially very similar to the biosorption processes. These are very effective sorbents for Sr(II) ions, as in this study we observed a very rapid (within 60 min) and high uptake ($\sim$ 98%) of Sr(II) ions from aqueous solutions at 35°C.

EXPERIMENTAL

Materials

N-Vinyl imidazole (N-VIm), tripropylene glycol diacrylate (TPGDA; Aldrich, Steinheim, Germany), ethylene glycol dimethacrylate (EGDMA; Merck, Schuchardt, Germany), N,N-methylene bisacrylamide (N,N-MBAAm; Loba Chemie, Mumbai, India), divinyl benzene (DVB; Fluka, Buchs, Germany), strontium bromide (S. D. Fine Chemicals, Mumbai, India), and Rose Bengal (HiMedia Laboratories, Mumbai, India) were used as received.

Synthesis of the hydrogels

To a known amount of N-VIm, 1 wt % of one of four different crosslinkers, EGDMA, TPGDA, N,N-MBAAm, and DVB, was added, and polymerization was carried out by an irradiation method by γ rays at a dose of 12.96 kGy in a γ chamber 900 (Bhabha Atomic Research Centre, Mumbai, India). The networks obtained were washed with methanol to remove any sol fraction and dried first at 70°C in an open-air oven and then in a vacuum desiccator.

Sorption of Sr(II) ions

We studied Sr(II) uptake as a function of time and temperature by stirring 0.1 g of each dry polymer with 25 mL of Sr(II) (1000 ppm) solutions in a chemical reactor (Auto Chem) for 30–360 min at 25°C and at five different temperatures (25–45°C) for 60 min. Sr(II) ion concentration variation was studied from 500 to 1500 ppm for 60 min at 35°C with a 25-mL so-

lution. The same procedure was followed to study the effect of different pHs (4.0, 5.8, 7.0, 9.2, and 13.2) at 35°C, 60 min and 1000 ppm of Sr(II). The network that exhibited the highest uptake was chosen for the evaluation of maximum retention capacity (MRC). MRC was studied with 1000 ppm Sr(II) up to four feeds. The concentrations of the ions left in the solution from different experimental sets were analyzed on an ultraviolet–visible spectrophotometer (Carry 100, Varian Inc, Mulgrave, Victoria, Australia) at a maximum wavelength of 533 nm by the mixture of 200 μL of a 2.0×10^{-4} M solution of Rose Bengal anionic dye into 5.0 mL of the aliquot solution. For the detection of the concentration of the ions, we generated different calibration curves by assaying a series of standards with known concentrations of Sr(II) ions in distilled water at different pHs. The change in the absorbance was plotted versus the concentration to determine the unknown concentration of the ions.

Various adsorption characteristics were evaluated with the following expressions:

Percent uptake (P_u)

$$= \frac{\text{Amount of metal ions sorbed}}{\text{Total ions in the feed solution}} \times 100$$

Equilibrium adsorption capacity (q_e; mg/g)

$$= \frac{(C_o - C_t)V}{m}$$

where q_e is the amount of Sr(II) ions adsorbed onto the unit dry mass of the hydrogel (mg/g), C_o and C_t are the concentrations of ions in the feed and after time t, respectively, in the solution (mg/L), V is the total volume of the aqueous phase (L), and m is the weight of the dry polymer (g).

Characterization of the as-prepared and Sr(II)-loaded hydrogels

The as-prepared hydrogels were characterized by scanning electron microscopy (SEM; JEOL Stereoscan-150 microscope), Fourier transform infrared (FTIR) spectroscopy on a Nicolet 5700, and element analysis (C, H, and N) on a Carlo Erba Instrument 1150 analyzer and an Elementar Vario El. Energy-dispersive spectroscopy (EDS)–SEM X-ray analysis of the Sr(II)-loaded networks was carried out on an Oxford Instruments INCA energy apparatus attached to the scanning electron microscope.

RESULTS AND DISCUSSION

In this article, the terms *hydrogel* and *network* are used interchangeably, and these convey the same meaning. A schematic presentation of the hydrogel structure or
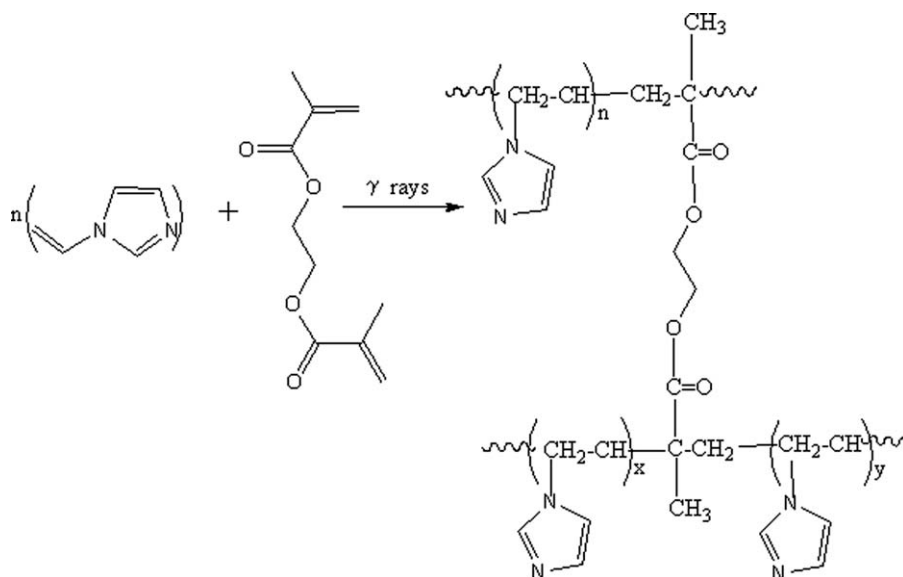
Scheme 1 General structure of the poly(*N*-VIm-*cl*-N,N-MBAAm) network.

network from poly(*N*-VIm) and the bifunctional crosslinker (e.g., EGDMA) is given in Scheme 1.

Evidence of network formation was obtained from the FTIR spectra, SEM, and elemental analysis, and these are briefly discussed here. The FTIR spectra (not presented) contained peaks due to the incorporation of crosslinker in addition to the usual peaks of poly(*N*-VIm). For example, in the spectra of poly(*N*-vinyl imidazole-*cl*-ethylene glycol dimethacrylate) [poly(*N*-VIm-*cl*-EGDMA)] and poly(*N*-vinyl imidazole-*cl*-tripropylene glycol diacrylate) [poly(*N*-VIm-*cl*-TPGDA)], peaks appeared at 1723.4 or 1726.0 cm^{-1} (ester C=O stretching of the crosslinker). In the spectrum of poly(*N*-vinyl imidazole-*cl*-N,N-methylene bisacrylamide) [poly(*N*-VIm-*cl*-N,N-MBAAm)], a peak at 1499.1 cm^{-1} was due to the secondary amide group (from the crosslinker), and in the spectrum of poly(*N*-vinyl imidazole-*cl*-divinyl benzene) [poly(*N*-VIm-*cl*-DVB)], peaks at 1571.9, 1540.9, and 1422.5 cm^{-1} (skeletal vibrations from the benzene ring in the crosslinker) were present. The results of elemental analysis of the crosslinked poly(*N*-VIm) also provided evidence for the crosslinking. The percentages of C and N were found to be different in the crosslinked networks than in poly(*N*-VIm) itself. The surface morphology of the crosslinked networks, as revealed from SEM, revealed a well-defined porous structure across the polymer surface. Such surface morphology of a hydrogel is ideal for use as a sorbent.

Strontium (II) uptake studies

Effect of the contact time, temperature, ion concentration, and pH

A Rose Bengal dye-based spectrophotometeric procedure was used to detect the amount of Sr(II) ions left in the feed solution. The application of the anionic dyes or counter anions with a large molar absorbance led to a sensitive method for the determination of metal ions present in traces, as has been reported for Sr(II) with 18-Crown-6 and Rose Bengal dye with partial least squares.[31] The effects of the time, temperature, and ionic strength on the ion uptake were studied at pH 7.0. The uncrosslinked poly(*N*-VIm) was used as reference polymer in the adsorption experiments. Sr(II) uptake was rapid, and equilibrium was attained within 60 min; this suggested a rapid uptake of the ions (Fig. 1). The effect of the temperature on the ion uptake was studied at five different temperatures at 60 min and with 1000 ppm Sr(II). P_u increased with temperature from 25 to 35 or 40°C, and thereafter, it decreased for all of the networks (Fig. 2). Even at 25°C, $P_u > 96\%$ was observed for all of the polymers studied. The trends
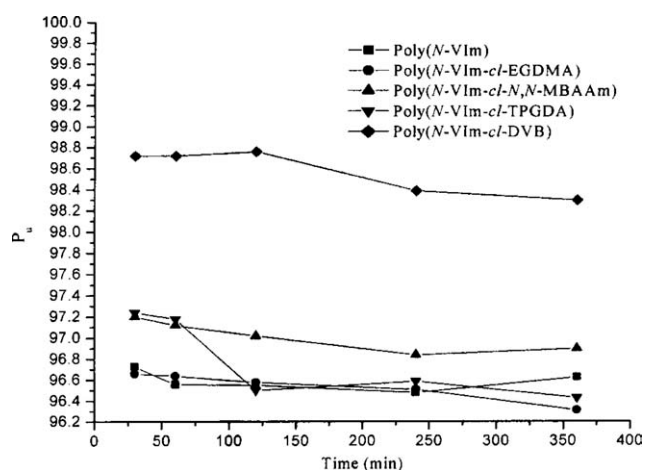

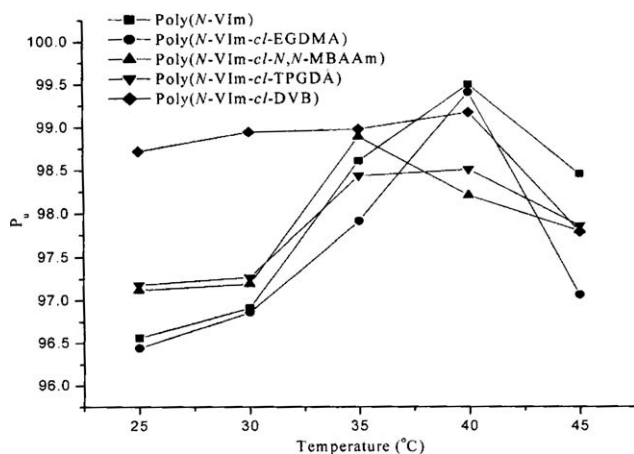**Figure 1** Sr(II) uptake as a function of time at 25°C ([Sr(II)] = 1000 ppm).

Figure 2 Sr(II) uptake as a function of temperature ([Sr(II)] = 1000 ppm, time = 60 min).

in the results on variation of temperature were related to the swelling behavior of the hydrogels. Initially, the percentage swelling (P_s) of the hydrogels increased with increasing temperature up to 35 or 40°C, and thereafter, it decreased irreversibly at the higher temperature. Hence, the lower partitioning of the ions to the inner core of the hydrogel resulted in a low uptake at the higher temperature. On variation of Sr(II) concentration, P_u increased with an increase of concentration, as the maximum values were observed at 1000 ppm for poly(N-VIm-cl-N,N-BAAm) and poly(N-VIm-cl-EGDMA). On the further increase of the ion concentration, it remained constant, and among all of the hydrogels studied, poly(N-VIm-cl-DVB) exhibited the maximum uptake (Fig. 3). The possible electrostatic interactions between a hydrogel and Sr(II) ions resulted through linkage at the tertiary nitrogen atoms of the imidazole rings of different polymer chains, as presented in Scheme 2. Some contribution to the overall ion uptake also came from the adsorption of ions on the
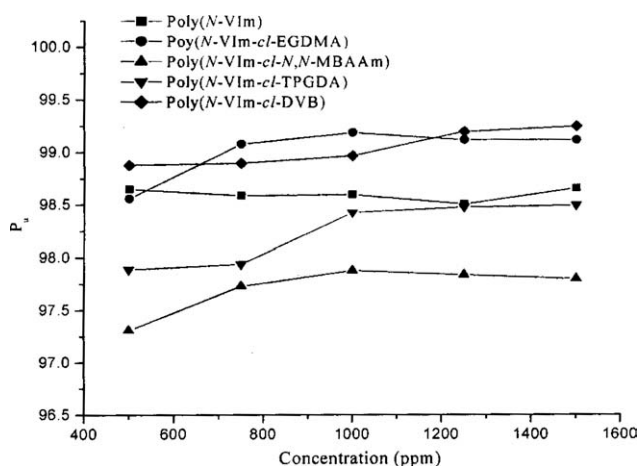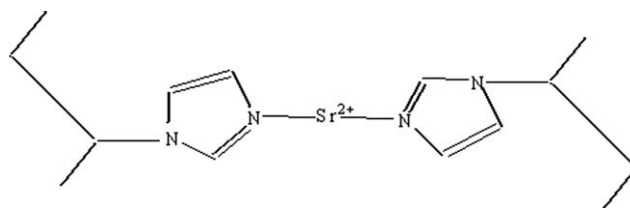

Figure 3 Sr(II) uptake as a function of concentration at 35°C (time = 60 min).

Scheme 2 Representation of possible poly(N-VIm)–Sr^{2+} interaction.

functional groups of the crosslinker-like amide groups of N,N-MBAAm. Hence, the mechanism of the ion uptake was heterogeneous in nature. The absorption peaks present in the FTIR spectra of the Sr(II)-loaded hydrogels were shifted by different magnitudes and appeared at different wave numbers in comparison to those in the FTIR spectrum of poly(N-VIm). The EDS–SEM of the Sr(II)-loaded hydrogels is presented to provide evidence of Sr(II) ion uptake on the candidate hydrogel (Fig. 4).

The effect of pH variation on P_u is presented in Figure 5. P_u by all the hydrogels was lower at acidic pH than at pH 7.0. The structural aspects of the hydrogels were important factors in determining the
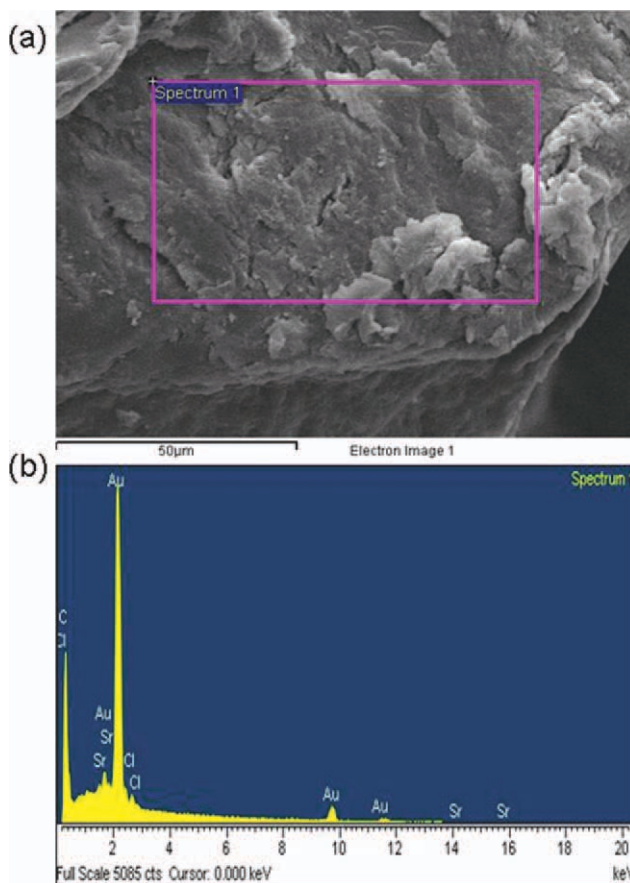

Figure 4 SEM image of (a) Sr(II)-loaded poly(N-VIm-cl-DVB) (b) EDS of Sr(II)-loaded poly(N-VIm-cl-DVB). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
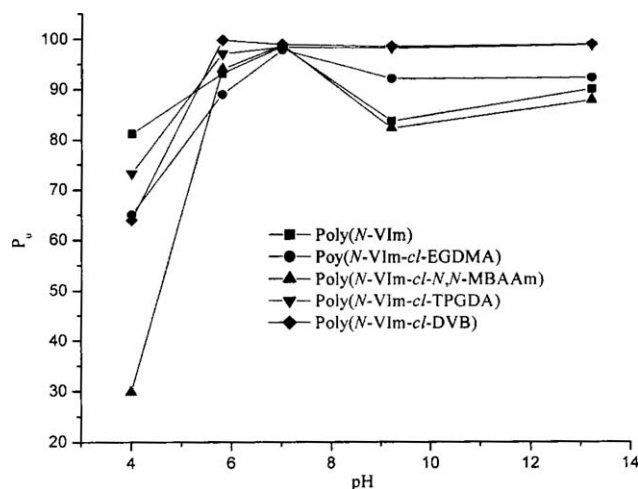
Figure 5 Sr(II) uptake as a function of pH at 35°C. ([Sr²⁺] = 1000 ppm, time = 60 min).

extent of ion uptake. At pH 4.0, it was just 29.84% (minimum uptake among all the networks) for poly(*N*-VIm-*cl*-*N*,*N*-MBAAm) but appreciable (65.04%) for poly(*N*-VIm-*cl*-EGDMA). The decrease in the ion uptake under acidic pH was discernible, as H⁺ ions protonated the active sites (N:) on the networks and made these less available for Sr(II). The interactions of the H⁺ and imidazole ring were also proposed by Annenkov et al.[32]

Evaluation of the MRC

We discussed previously that poly(*N*-VIm-*cl*-DVB) did not only exhibit a higher performance than the other networks under the various parameters studied, but its performance was even appreciable on the

TABLE I
MRC of Sr(II) Ions on Poly(*N*-VIm-*cl*-DVB) with Repeated Treatment at 1000 mg/L

Polymer	P_u	mg of Sr²⁺ sorbed/g
First feed	98.97	247.43
Second feed	98.67	246.68
Third feed	98.15	245.38
Fourth feed	98.14	245.35
MRC		984.84

variation of pH and temperature over a wide range. For these reasons, this network was selected for the evaluation of MRC. MRC was studied at feed concentration of 1000 ppm Sr(II) ions (Table I). The network was subjected to four feeds of solution at the optimized conditions evaluated and as discussed previously. P_u was found to remain almost the same up to the fourth feed with an appreciable value of 98.14%; this amounted to a cumulative MRC of 984.83 mg/g. Such a high uptake and retention resulted from the active sites available for Sr(II) interaction at the tertiary nitrogen of the imidazole rings. The mechanism of the ion uptake was heterogeneous, as many types of electrostatic and other interactions were possible between the ions and the hydrogels.

Adsorption kinetics

Two isotherm equations were tested in this study, namely, the Freundlich and Temkin isotherms. The Freundlich isotherm is a fairly satisfactory empirical isotherm and can be used for nonideal sorption that involves heterogeneous surface energy systems as in this case, and it is expressed by the following equation:
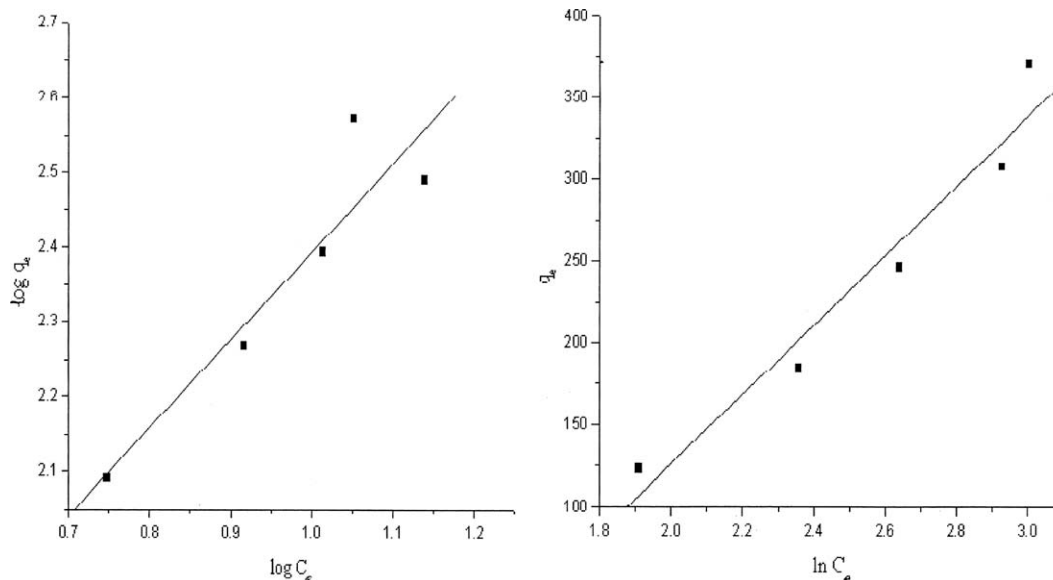

Figure 6 (a) Freundlich plot for adsorption of Sr(II) on poly(*N*-VIm-*cl*-DVB) and (b) Temkin plot for adsorption of Sr(II) on poly(*N*-VIm) (contact time = 60 min, temperature. = 35°C).

TABLE II
TABLE II
Freundlich and Temkin Constants for Sr(II) Sorption

Polymer	Freundlich			Temkin		
	K_F	n	r^2	B	A (L/g)	r^2
Poly(N-VIm)	19.22	1.03	0.995	212.35	0.24	0.973
Poly(N-VIm-cl-EGDMA)	11.76	0.74	0.877	327.02	0.24	0.939
Poly(N-VIm-cl-N,N-MBAAm)	31.77	1.48	0.708	165.03	0.24	0.773
Poly(N-VIm-cl-TPGDA)	8.65	0.87	0.929	252.09	0.15	0.908
Poly(N-VIm-cl-DVB)	16.39	0.84	0.930	250.67	0.28	0.876

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

where C_e is equilibrium concentration and K_F is an indicator of the adsorption capacity and $1/n$ is the adsorption intensity. Generally, as the K_F value increases, the adsorption capacity of an adsorbent for a given adsorbate also increases. The magnitude of the exponent $1/n$ gives an indication of the adsorption conditions. $n > 1$ represents favorable adsorption conditions. One can linearize the previous equation by taking logarithms:

$$\log q_e = \log K_F + 1/n \log C_e \tag{2}$$

Linear plots of $\log q_e$ versus $\log C_e$ show that the adsorption of Sr(II) from the prepared water sample on different networks followed the Freundlich isotherm, as presented for poly(N-VIm-cl-DVB) [Fig. 6(a)]. The values of K_F and $1/n$ were calculated from the intercepts and slopes of the plots and are listed in Table II, along with the correlation constants (r^2's). The results suggest that Sr(II) ions were favorably adsorbed by all of the polymers studied.

The linear form of the Temkin isotherm[33] can be expressed by the following equation:

$$q_e = B \ln A + B \ln C_e \tag{3}$$

where A and B are the Temkin constants. The isotherm constants were determined from linear isotherm graphs for the isotherm equation tested. The values of the isotherm constants with the r^2 values are given in Table II. Linear plots of q_e versus $\ln C_e$ showed that adsorption of Sr(II) from wastewater on different networks followed the Temkin isotherm. The Temkin isotherm equation represents the best fit of the experimental data for poly(N-VIm), for which the value of r^2 was 0.973 [Fig. 6(b)].

The effect of the contact time on Sr(II) ion uptake by different hydrogels, as discussed earlier, suggested rapid ion uptake resulting in a smooth curve and leading to equilibrium within 60 min. The kinetic data were treated with the pseudo-second-order kinetic model[34] with the following differential equation:

$$dq_t/d_t = k_2(q_e - q_t)^2 \tag{4}$$

where q_t is the ion uptake at time t (mg/g) and k_2 is the equilibrium rate constant of pseudo second-order adsorption (g mg^{-1}/min). The integration of this equation for the boundary condition $t = 0$ to t and q_e to q_t gives

$$1/q_e - q_t = 1/q_e + k_2 t \tag{5}$$

which is the integrated rate law for a pseudo-second-order reaction. This equation can be rearranged to obtain a linear form:
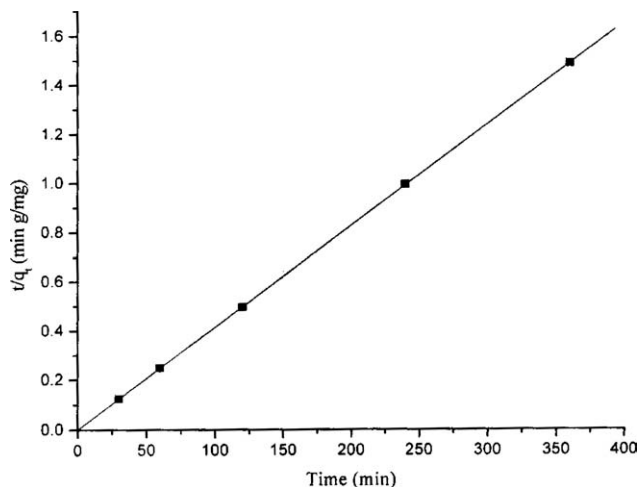
TABLE III
k_2 and Calculated and Experimental q_e Values

Polymer	Experimental		Calculated	
	q_e	k_2	q_e	r^2
Poly(N-VIm)	241.54	14.09×10^{-2}	241.825	1
Poly(N-VIm-cl-EGDMA)	240.96	-6.49×10^{-2}	241.65	1
Poly(N-VIm-cl-N,N-MBAAm)	242.13	3.65×10^{-2}	243.00	1
Poly(N-VIm-cl-TPGDA)	242.13	2.68×10^{-2}	243.10	1
Poly(N-VIm-cl-DVB)	245.70	-1.21×10^{-2}	246.9	1

Figure 7 Pseudo-second-order kinetics for adsorption of Sr(II) on poly(N-VIm-cl-EGDMA) at 25°C.

$$t/q_t = 1/k_2 q_e^2 + 1/q_e \times t \qquad (6)$$

The linear plots of t/q_t versus t are a linear relationship, show good agreement with the experimental data for pseudo-second-order kinetics, and were applicable for all of the networks. A representative example is presented in Figure 7. The r^2 values for this kinetic model were unity. k_2 and q_e were calculated from the intercept and slope of the plots of t/q_t versus t. The calculated q_e values matched very well with the experimental data (Table III).

CONCLUSIONS

We synthesized a series of poly(N-VIm)-based hydrogels by following a clean protocol. The hydrogels exhibited structure–property relationships, as Sr(II) uptake on these hydrogels was found to be dependent on the nature of the crosslinker used. The reported hydrogels were very efficient extractants of Sr(II) ions. The sorption of Sr(II) was rapid, and equilibrium was established within just 60 min. The sorbents were suitable for use under a wide range of external stimuli, such as different values of temperature and pH. Poly(N-VIm-cl-DVB) was found to be the best sorbent among all of the networks studied in diverse pH media; hence, it was used to study the MRC, which was found to be 984.84 mg/g. The evidence of sorption was obtained from SEM–EDS analysis of the Sr(II)-loaded network. The adsorption of ions was both chemical on the tertiary nitrogen of the imidazole ring and physical on the functional groups of the crosslinker used. The match of the experimental equilibrium data with the Temkin and Freundlich isotherms revealed that the mechanism or the nature of sorption was heterogeneous in nature. The adsorption followed pseudo-second-order kinetics; that is, it was dependent on the availability of active sites on the networks and on the feed ion concentration.

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