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Uranyl ions uptake on poly(AAc/AAm)-*cl*-*N*,*N*-MBAAm hydrogel

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Abstract In the present article, we report two well-characterized hydrogels for the adsorption of uranyl ions from 5% NaCl solution. The hydrogel was synthesized by free radical initiation from acrylamide and acrylic acid using *N*,*N*-methylenebis-acrylamide as crosslinker. In order to improve ion uptake performance, the hydrogel was hydrolyzed to partially convert some of the amide groups into carboxylate groups. The uranyl ion adsorption was studied as a function of hydrogel structure, uranyl ions concentration, pH, temperature, and mass of hydrogel. The partially hydrolyzed hydrogel exhibited the maximum uranyl ion uptake of 236.6 mgg^{-1} in 480 min at 45 °C and at pH 13. Good reproducibility of results was observed and the evidence of sorption at the optimum pH and ion concentration was obtained by comparison of FTIR spectra of the precursor and uranyl ions loaded hydrogels. The performance of both the hydrogels was found to be higher than that of a commercial resin, Amberlite IRC-718. The experimental data shows good match with Langmuir and Freundlich isotherms and pseudo-first order kinetics.

Keywords Hydrogel · Partial hydrolysis · Swelling · Uranyl ions

Introduction

Enrichment, separation, and removal of actinides, like uranyl ions from the sea water and waste water, are imperative to enrich energy resources as well as an environmental corrective. The polymeric resins are materials of choice in adsorption, extraction, and ion exchange processes. Uranyl ions uptake has been reported using membranes [1–3], polymer imprinting [4, 5], and polymeric supports which have active groups like azobenzene [6], complexing groups like amidooxime [7–9],

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ion-exchange groups like phosphoric acid [10], and carboxylic acids [11, 12]. The polymeric supports those have more than one functional groups have also been reported for the uranyl-ion uptake [13]. Among these materials, hydrogels are the most frequently reported supports to enrich and separate uranyl ions from the aqueous solutions [12, 14–21]. The uranyl-ion uptake on these hydrogels either from the single cation solution or competitive from multi-cation solutions is guided by the structure–property relationship of the hydrogels as well as by the environmental factors, such as pH, temperature, or the ionic strength.

The hydrogels offer many advantages as materials of choice. The structure of the hydrogels can be suitably manipulated by polymer analogous reactions to generate additional functional groups [22]. The technological importance of hydrogels in enrichment, separation, and purification technologies emanates from their capacity to absorb large amount of water. Absorption of large amount from the metal ion solutions results in their partitioning to the active sites on the polymer phase [22–25].

In view of the above and in continuation to our earlier study, in this article, we report the use of a hydrogel based on acrylamide (AAm) and acrylic acid (AAc) crosslinked with *N*,*N*-methylenebisacrylamide [poly(AAm/AAc)-*cl*-*N*,*N*-MBAAm] as a sorbent for uranyl ions. The hydrogel was functionalized by alkaline hydrolysis to increase its efficacy in the targeted end-use, and both the hydrogels were characterized by physical and chemical methods including scanning electron micrography (SEM), nitrogen analysis, and Fourier transform infrared spectroscopy (FTIR), and were used for the sorption of uranyl ions from 5% NaCl solution. The extent of uranyl-ion uptake was dependent on the hydrogel structure as well as on the environmental factors like pH and temperature, as the maximum uranyl-ion uptake of 224.98 and 236.6 mgg^{-1} was observed using the unhydrolyzed and partially hydrolyzed hydrogel in 480 min at 45 °C and at pH 13, which reveal 89.99 and 93.21% adsorption, respectively.

Experimental

Materials

Acrylamide (AAm), *N*,*N*-Methylenebisacrylamide, ammonium persulfate, uranyl nitrate, sodium chloride, sodium carbonate, and buffer tablets all from S.D. Fine Chem. Ltd. (Mumbai, India), acrylic acid (AAc) from Merck (Schuchardt, Germany), were used as received.

Synthesis of the hydrogels

An equimolar amount of acrylic acid and acrylamide, *N*,*N*-methylenebisacrylamide (2% of the total weight of the monomers), and ammonium persulfate (1% of the combined weight of two monomers) were placed in 5 mL of water. The reaction system was placed in the Autochem Chemical Reactor at 25 °C for 30 min. The self-insolubilized network was purified, dried, and functionalized further by alkaline hydrolysis as per the procedure described elsewhere [21]. The hydrogels were cut to thin disks, washed again, and dried in a hot air vacuum oven at 45 °C to obtain a constant weight. Poly(AAc/AAm)-*cl*-*N*,*N*-MBAAm and its partially hydrolyzed form were designated as HG_1 and HG_2, respectively.

Characterization of hydrogels

Hydrogels were characterized by SEM (Joel Stereoscan-150 microscope), FTIR (Nicollet 5700 FTIR spectrometer), and nitrogen analysis (Carlo Erba Instrument 1150). The swelling behavior of hydrogels was studied as a function of time (10–720), temperature (25–45 °C), and pH (4.0, 7.0, and 9.0), and in 1 and 5% NaCl solution as per the procedure reported earlier [21]. The percent swelling (P_s) of the hydrogels was calculated as:

$$P_s = (M_t - M_o)/M_o) \times 100,$$

where M_t is the mass of swollen gel at time t, and M_o is the initial mass of the dry gel. Salt sensitivity of the hydrogels was calculated as per the following relationship:

$$f(\text{Salt sensitivity}) = 1 - (\text{Water uptake in a salt solution}/\text{Water uptake indistilled water}).$$

Uranyl ions adsorption studies

Solution of uranyl nitrate in a concentration range of 0.5–2.5 mM/L was prepared in the presence of 5% NaCl and buffered by adding Na_2CO_3. To this solution (50 mL), 0.1 g of dry hydrogel was added. The adsorption process was allowed to equilibrate for 8 h at 25 °C. Hydrogels were separated by filtration and the filtrate was analyzed at λ_{max} 215 nm for the rejected ions on Cary 300 UV–Vis spectrophotometer. The adsorption per unit mass of the dry hydrogel was expressed as:

$$q_e = (C_o - C_e)V/W,$$

where q_e is the amount of uranyl ions adsorbed onto unit dry mass of the hydrogels (mgg^{-1}), V is volume of solution taken, and W is weight of dry gel used (g), C_o and C_e (mg L^{-1}) are concentrations of uranyl ions in the initial solution and that left after adsorption for a certain period of time, respectively.

Result and discussion

Monomers and crosslinker being hydrophilic in nature form a three-dimensional hydrogel HG_1 with a high crosslinking efficiency (yield = 98.3%). The ratio of AAm and AAc in HG_1 is determined by their respective monomer reactivity ratio as reported earlier [25]. Hence, HG_1 is richer in AAc and the overall uranyl ion adsorption process will have more contribution from the ion-exchange process. After partial hydrolysis, the resultant hydrogel HG_2 has terpolymer composition as apart from the crosslinker it has three components viz poly(AAc), poly(AAm), and poly(sodium carboxylate).

Characterization of hydrogels

Evidence of the network formation by observing surface morphology is provided by the SEM of the hydrogels. In the HG_1, the intense crosslinking results in small pore formation due to the high crosslinking density. On partial hydrolysis, the structure of the network opens up as large pores are visible in the SEM of HG_2 (Fig. 1). The evidence of network formation and partial hydrolysis was deduced from the analysis of the characteristic peaks of the functional groups present in hydrogels. Peaks at 3,415.5 and 1,658.7 cm^{-1} due to –NH stretching and –C=O stretching of $–CONH_2$ groups and a weak peak at 1,700 cm^{-1} due to –C=O stretching of –COOH group are present in the FTIR spectrum of HG_1. In the spectrum of HG_2, the intensity of the peaks due to the amide absorption decreases while the peak due to the –C=O stretching of the carboxylate groups increases. The results of nitrogen analysis of the hydrogels are presented in Table 1. Percent nitrogen analysis reveals that the monomers react as per their reactivity ratios. The mole fractions of the two monomers have been calculated from the obtained nitrogen values and it comes out there from that HG_1 is richer in poly(AAc) than poly(AAm). Partial hydrolysis of $–CONH_2$ groups to $–COO^-$ lowers %N in HG_2 (2.74%) than HG_1 (7.71%).

Swelling behavior

Investigation of the swelling behavior of the hydrogels as a function of time, temperature, pH, and in brine is important to define their end uses. The swelling of HG_1 increased with time until 240 min, and thereafter equilibrium was reached. When studied as a function of temperature, the maximum P_s was obtained at 35 °C as thereafter it was observed to decrease (Fig. 2a). With rise in temperature, the water uptake by the polymer network increases due to the expansion of the network chains. However, at the higher temperatures, the hydrogel cannot retain much water due to the higher thermal motion of water molecules and more opening up of the hydrogel structure. Swelling was lower in HG_1 than HG_2. These results manifest high crosslinking density of HG_1, while HG_2 is more porous and can accommodate more water in the bulk. Further, more ionic groups are present in the latter those contribute to more interaction with water resulting in the higher water uptake (Fig. 2b). These hydrogels are sensitive to the pH of the swelling medium. On change of pH from 4.0 to 9.2 at 35 °C, P_s was observed to increase in a linear fashion. In the acidic pH, the ionization of the $–CO_2H$ groups of the hydrogels is suppressed and shriveling of the hydrogels was observed, while in the alkaline pH, there is more interaction between the hydrogels and the swelling medium. Hydrogels do not swell appreciably in the presence of electrolyte salts due to the exo-osmosis as even the swollen hydrogels shrank dramatically in the presence of salt (Fig. 2c). The maximum P_s in 1 and 5% NaCl solution was 343.3 and 106.6 for HG_1 and 2,316.6 and 1,156.6 for the HG_2, respectively. The salt sensitivity values of the hydrogels are presented in Fig. 2d. Although these hydrogels are sensitive to the

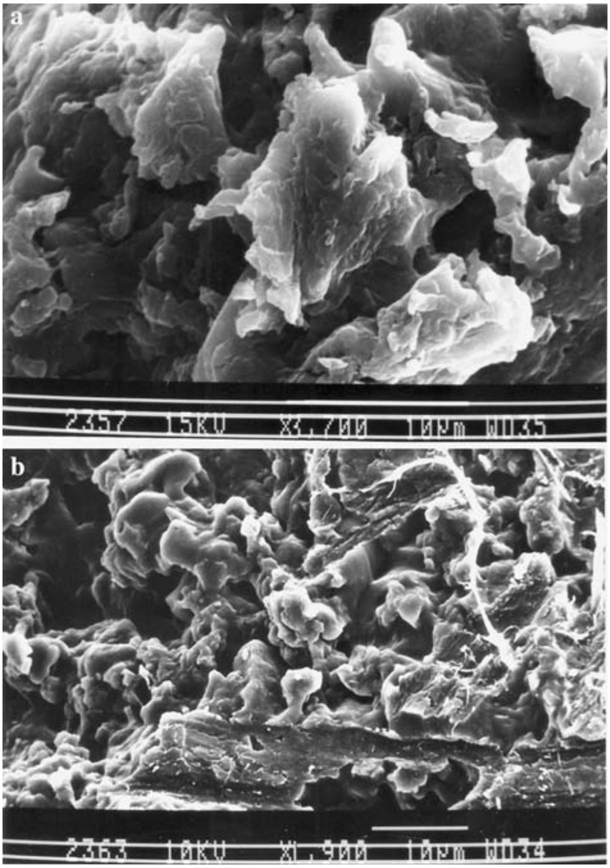

Fig. 1 Scanning electron micrograph of (**a**) HG_1 and (**b**) HG_2

Table 1 Nitrogen analysis of hydrogels

Hydrogels	Weight of samples (mg)	N found (%)	Amount of N (mg)	AAm (mg)	AAm:AAc	Hydrolysis (%)	Mole fraction of AAm
HG_1	1.912	7.71	0.147	0.745	1:1.54	–	0.397
HG_2	1.942	2.74	0.0532	0.269	1:6.14	63.89	0.16

presence of salt, yet the water uptake by HG_2 in brine is high, thus exhibiting appreciable salt tolerance.

Adsorption of uranyl ions: effect of time, temperature and pH

Uranyl ions uptake is a combination of ion exchange at $–CO_2H$ and $–CO_2Na^+$ groups of HG_1 and HG_2, respectively. Some adsorption also occurs on the amide groups of poly (AAm) and crosslinker. The ion uptake was studied using 502 ppm

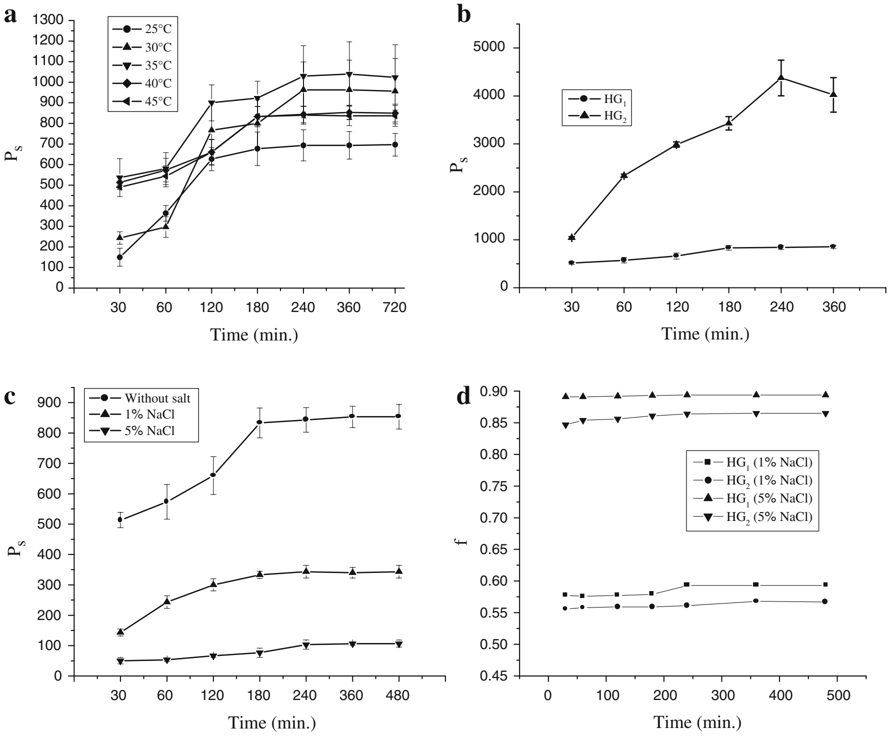

Fig. 2 **a** P_s of HG_1 as a function of time and temperature. **b** Comparative plot of P_s of both HG_1 and HG_2 as a function of time. **c** P_s of HG_1 as a function of time at 35 °C in different concentration of salt. **d** Salt sensitivity (f) of HG_1 and HG_2 as a function of time at 40 °C in 1 and 5% NaCl

of the (1 mM/L) of uranyl nitrate in the presence of 5% NaCl solution. q_e values were observed to increase with time affording the highest q_e of 224.98 mgg^{-1} corresponding to a P_u of 89.99% for HG_1 at pH 13 (Fig. 3a). This is due to polyelectrolyte hydrogels comprise acidic and basic groups, respectively, those can be ionized. The hydrogel has –COOH groups and its partially hydrolyzed product contain $–COO^-$ groups. In the basic medium, these get ionized, and the result is higher ion uptake with change of pH from acidic to basic media. In an acidic medium, the acid group-containing hydrogel form a transient network that does not allow deprotonation of the hydrogel. On increase of temperature from 25 to 45 °C, ion uptake was observed to increase regularly affording the highest q_e at 45 °C. Increase in temperature opens up hydrogel structure making it more accessible to the ions. Consequently, the diffusion of the metal ions inside the pores of the hydrogels is facilitated more. Variation of pH from 4.0 to 13.0 at a fixed concentration of uranyl nitrate (502 ppm) at 45 °C, ion uptake increased on moving from acidic to basic pH (Fig. 3b). Since the pK_a of poly (AAc) is 4.3, the water uptake, hence the partitioning of the ions varies with pH. At the lower pH, ionization of the $–CO_2H$ of poly (AAc) is suppressed as the hydrogel was observed to shrivel. This results in low swelling and limits the access of ions to the bulk of the

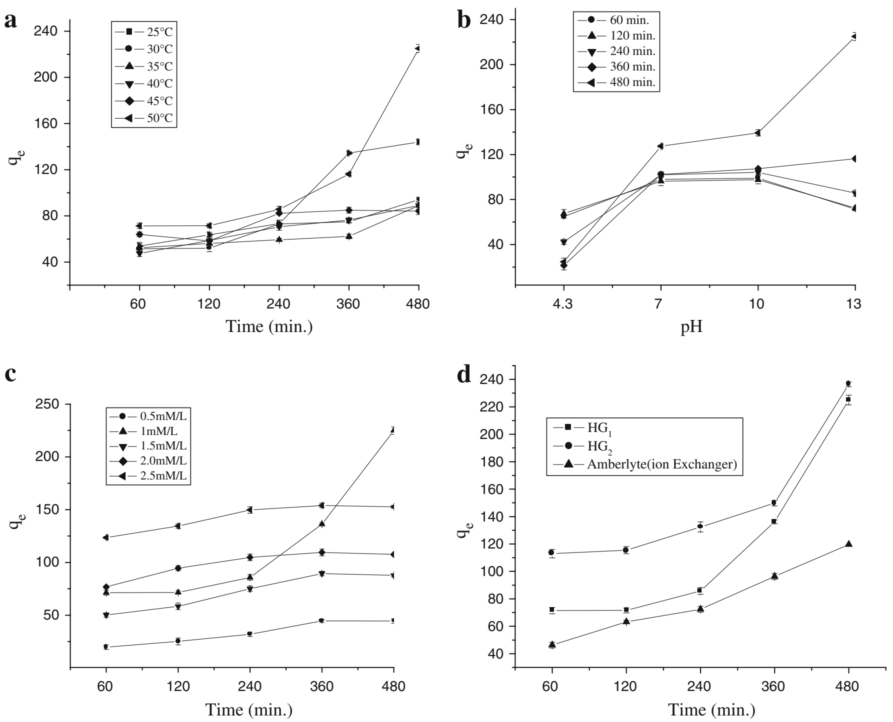

Fig. 3 Uranyl uptake by **a** HG_1 as a function of time and temperature (uranyl nitrate = 502 ppm), **b** HG_1 as a function of pH at 45 °C, **c** HG_1 as a function of time and concentration at 45 °C, and **d** Uranyl uptake by HG_1, HG_2, and Amberlite IRC-718 as a function of time at 45 °C, at pH 13 (uranyl uptake = 502 ppm)

hydrogels. Adsorption of the uranyl ions was observed to obey linear behavior when the uranyl concentration was varied between 251 and 1,255 ppm at 45 °C and 60–480 min. An increase in the concentration of the uranyl nitrate from 0.5 to 2.5 mM/L, results in an increase in the adsorption as observed with a maximum adsorption at 502 ppm after 480 min (Fig. 3c). After that it remains almost constant suggesting the attainment of equilibrium. The maximum q_e of 236.825 mgg^{-1} for HG_2 was also observed at 502 ppm. This shows the saturation of active binding sites on the sorbent above that concentration.

Comparison with Amberlite IRC-718

In order to ensure efficacy of the results obtained, the uranyl adsorption study was compared with that of HG_2 and a commercial resin Amberlite IRC-718 at 45 °C, pH 13.0, and 502 ppm of uranyl ions. It exhibited a q_e value 119.65 which corresponds to 65% and is comparable to that of HG_1, and is far lower than that of the partially hydrolyzed hydrogel (Fig. 3d). The maximum adsorption capacities of both the hydrogels reported in this article are higher than those reported in literature for hydrogel having $-CO_2H$ groups [26].

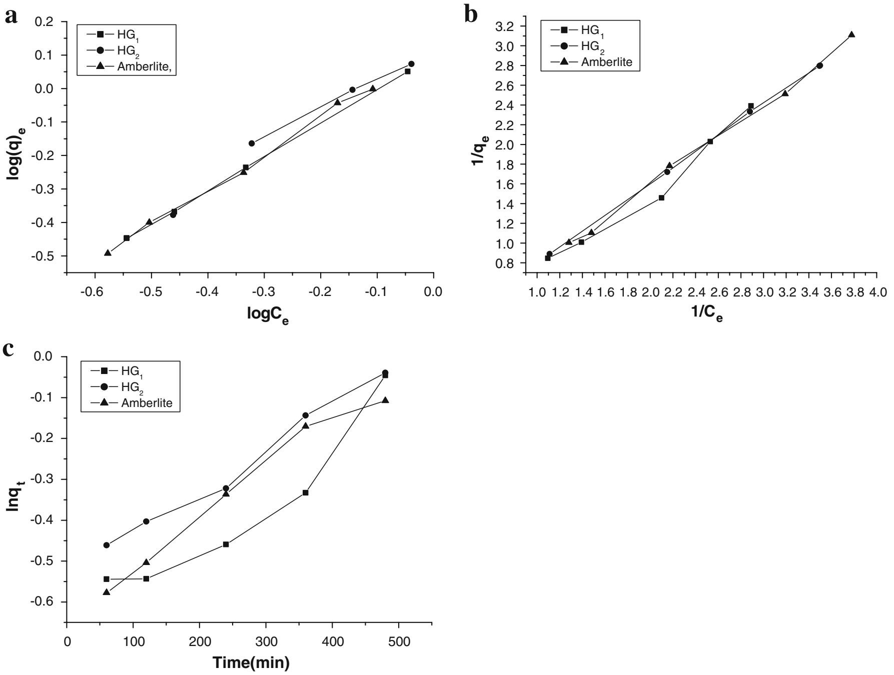

Fig. 4 **a** Freundlich adsorption isotherm of uranyl ion uptake on HG_1, HG_2, and Amberlite IRC-718 at 45 °C. **b** Langmuir adsorption isotherm of uranyl ion uptake on HG_1, HG_2, and Amberlite IRC-718 at 45 °C. **c** Pseudo-first order kinetics plot of uranyl ion uptake on HG_1, HG_2, and Amberlite IRC-718 at 45 °C

Adsorption isotherms and kinetics

The linear fit plots of the Freundlich (Fig. 4a) and Langmuir isotherms (Fig. 4b) were compared with the experimental values, and a good match was observed for the both adsorption isotherms. The applicability of the Langmuir isotherm is understandable in the hydrogels due to the complexation processes, while that of the Freundlich isotherm is indicative of the heterogeneities in the adsorption process, as apart from the $–CO_2H$ or $–CO_2Na^+$ groups, adsorption also takes place at the $–CONH_2$ groups of the poly(AAm) and in the bulk of hydrogels at the crosslinks of *N*,*N*-MBAAm (Table 2, Fig. 4a, b). Two kinetic models viz. pseudo-first order and pseudo-second order were also applied to the experimental data, and the former exhibited higher applicability over the whole range of time studied suggesting predominantly the complexation or chemisorption (Table 3, Fig. 4c).

Evidence of the uranyl uptake

The uranyl uptake on hydrogels is supported by the changes in the FTIR of the uranyl-loaded hydrogels [27]. The comparison of the spectra of the precursor HG_1 (Fig. 5a) and its uranyl-loaded form (Fig. 5b) reveals that in the latter a shift in –C=O stretching of acid and amide groups is observed with the peak corresponding to the –C=O stretching

Table 2 Correlation coefficients for isotherm models of HG_1, HG_2, and Amberlite

Polymer	Freundlich adsorption isotherm (linear regression)			Langmuir adsorption isotherm (linear regression)			Freundlich adsorption isotherm	Langmuir adsorption isotherm
	R	R^2	SD	*R*	R^2	SD	$\log q_e = 1/n \times \log C_e + \log k$ $(Y = mX + C)$	$1/q_e = 1/ab \times 1/C_e + 1/a$ $(Y = mX + C)$
HG_1	0.9994	0.9998	0.00273	0.99989	0.9998	0.01419	Y = 1.00233X + 0.9672	Y = 0.80099X + 0.00341
HG_2	0.98759	0.9753	0.03836	0.98742	0.9749	0.12013	Y = 1.05089X + 0.13591	Y = 0.86223X − 0.17968
Amberlite	0.99759	0.9959	0.01579	0.99824	0.9965	0.06202	Y = 1.05436X + 0.12	Y = 0.83698X − 0.09015

Table 3 Correlation coefficients for different kinetic models of HG_1, HG_2, and Amberlite

Polymer	Pseudo-first order (linear regression)			Pseudo-second order (linear regression)			Pseudo-first order	Pseudo-second order
	R	R^2	SD	*R*	R^2	SD	$\ln q_t = -k't + \ln q_0$ $(Y = mX + C)$	$t/q_t = 1/q_e \times t + 1/kq_e^2$ $(Y = mX + C)$
HG_1	0.94489	0.8928	0.07879	0.65867	0.4338	194.447	Y = 0.00115X − 0.67392	Y = 0.85798X + 309.49
HG_2	0.99289	0.9856	0.02453	0.88121	0.7765	85.179	Y = 0.00103X − 0.53255	Y = 0.80033X + 199.85
Amberlite	0.98904	0.9782	0.03472	0.92839	0.8629	65.54	Y = 0.00117X − 0.63469	Y = 0.8253X + 247.72

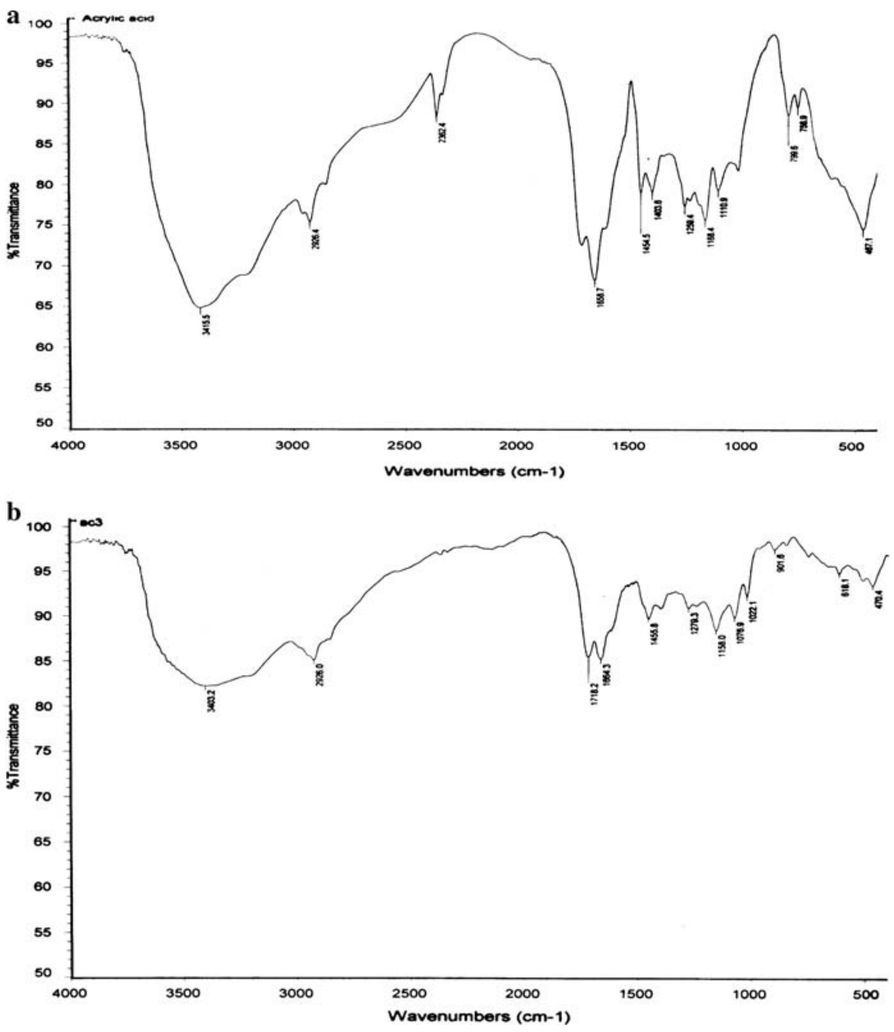

Fig. 5 **a** FTIR spectrum of HG_1. **b** FTIR spectrum of uranyl-loaded HG_1

appears at 1,664.3 cm^{-1} as compared with 1,658.7 cm^{-1} in its precursor HG_1. This indicates strong interaction of uranyl ions and the amide and carboxylate groups of the sorbents. The strong evidence of the uranyl ions uptake and binding with the hydrogel comes from the additional peaks near 901.6 cm^{-1} which are characteristics of O=U=O stretching. Similar conclusions can be drawn for the uranyl-loaded HG_2.

Conclusions

Poly(AAm/)-*cl*-*N*,*N*-MBAAm behaves as an environmentally sensitive hydrogel as it undergoes wide changes when subjected to even small changes in its external environment. However, its partially hydrolyzed form is better water absorbent and shows good salt tolerance even in 5% NaCl solution. It exhibited higher uranyl uptake than its precursor. The uranyl ions uptake capacities of both these hydrogels were found to be higher than a commercial resin Amberlite IRC-718. The partial hydrolysis of the hydrogel had positive impact on its property-profile that includes uranyl ion uptake at the environmentally and technologically desirable conditions from brine. The hydrogels are potential candidates for the uranyl ions uptake from different sources including seawater.

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