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Separation Science and Technology

Publication details, including instructions for authors and subscription information:
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Online publication date: 20 December 2010

To cite this Article Chauhan, Kalpana and Chauhan, Ghanshyam S.(2011) 'Separation of Uranyl Ions on Starch-Based Functional Hydrogels: Mechanism and Kinetics', Separation Science and Technology, 46: 1, 172 — 178

To link to this Article: DOI: 10.1080/01496395.2010.484005

URL: http://dx.doi.org/10.1080/01496395.2010.484005

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Separation Science and Technology, 46: 172–178, 2011
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ISSN: 0149-6395 print/1520-5754 online
DOI: 10.1080/01496395.2010.484005

Separation of Uranyl Ions on Starch-Based Functional Hydrogels: Mechanism and Kinetics

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In this article, we report the mechanism and kinetics of adsorption of uranyl ions on starch-based functional hydrogels. The hydrogels were prepared from starch in native or hydrolyzed/oxidized form by crosslinking with *N,N*-methylenebisacrylamide. The hydrogels synthesized from the oxidized starch have carboxylic groups at C-6 position. The effect of the structure and external environmental factors, i.e., contact time, temperature, ion strength, and simulated seawater (0.55 M NaCl and 3 mM $NaHCO_3$), was investigated on the uranyl adsorption behavior of hydrogels. The adsorption of uranyl ions was rapid as the highest adsorption was observed after 6 h and at 40°C. The sorbents also exhibited appreciable ion uptake even from the simulated seawater. The equilibrium data was analyzed using Langmuir and Freundlich adsorption isotherms and pseudo-first order and pseudo-second order kinetic models. Evidence of adsorption was obtained by characterization of the uranyl ions-loaded hydrogels by FTIR spectroscopy and also by elution with 0.1 N HCl.

Keywords adsorption isotherms; functional starch; hydrogels; simulated seawater; uranyl ions

INTRODUCTION

Uranyl ions or radionuclides are toxic to all the life forms even if these are present in the undetectable quantities in the natural water bodies or waste water of nuclear plants. The recovery of uranyl ions from various water bodies is also essential to supplement its stock as resource material for nuclear energy production. These twin aims constitute an area of current interest in the separation, purification, and recovery technologies. The high salinity and alkaline pH adversely affect uranyl ions uptake. The problem of uranium recovery from seawater or any other saline water systems is difficult in view of the high salinity and presence of the competitive cations. Further, the removal of uranyl ions is highly dependent on the pH of the solution, which affects speciation of the adsorbate species. Four major uranyl complex ions UO_2^{2+}, $(UO_2)_2(OH)_2^{2+}$, UO_2OH^+, $(UO_2)_3(OH)^{5+}$ and a dissolved solid schoepite ($4UO_3 \cdot H_2O$) exits in the solution in the pH range of acidic to near neutral (1). UO_2^{2+} ions are the most dominant ionic form at the lower pH of the system, but the lower pH suppresses the enhancement of uranyl ion adsorption. On the other hand, the non-ionic dissolved solid schoepite appears in the solution at the high pH and consequently the uranyl ions sorption is retarded due to decrease in the ion concentration.

In view of the above, the separation of the uranyl ions is augmented by using functional polymeric supports/hydrogels which may be of synthetic or bio-origin. The use of the latter is imperative in view of the easy availability, cost-effectiveness, high efficiency, and non-waste generating nature of biomass. The use of biosorbents is thus attractive for the adsorption or immobilization of uranyl ions from the aqueous solution. Biosorbents can be easily prepared by the functionalization of biopolymers which provide a wide range of reactive and functional materials for diverse applications with a wide end-use spectrum (2). The biosorbents are suitable candidates for the enrichment, separation, and water management technologies (3–8). The biosorption is a complex process that involves ion exchange, chelation, adsorption by physical forces, and ion entrapment in inter and intra-fibrillar capillaries and spaces of the polysaccharide network as a result of the concentration gradient and diffusion through cell walls and membranes (9). The biosorption of uranyl ions (UO_2^{2+}) has been reported on bacteria, algae, fungus, green plants, or biopolymers both in the modified and native forms (10). The biosorption on the polysaccharide rich biomass is aided by the participation of different polar groups. The efficacy of ion sorption on a natural biosorbent can be enhanced many a time by its modification to a crosslinked hydrogel. It is desirable that such a bioresource-based hydrogel has carboxylic/acidic functional groups, as the pendant acidic units enhance ion uptake processes and efficiency of the sorbents in the uranyl ions uptake from the aqueous solutions (11,12). The incorporation of active hydrophilic moieties such as –OH, –COOH, –CH=NOH, $-NH_2$ and $-SO_3H$ improves ion binding and chelating

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Received 22 November 2009; accepted 6 April 2010.

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capacities of these polymers by ion partitioning and improved ion exchange (13). The hydrophilic character of the hydrogel has been reported to be a significant determinant in the uranyl uptake (14). The enhancement of the adsorption capacity of hydrogels with the concentration of metal ions results in the shrinking of the hydrogels and the resultant proximity of polymer chains increases in the intermolecular contacts (15). The amount of adsorbed UO_2^{2+} also depends on the concentration of the chelating groups in the hydrogel as well as free volume of gel available for diffusion (16).

The hydrogels based on the biomass are sorbents of choice for the reasons delineated above. Hence, in this article we report the uranyl ions uptake on new sorbents synthesized from the functional starch. Starch is the third largest available biopolymer after cellulose and chitosan. It was partially hydrolyzed and consequently changed to hydrogels by crosslinking reactions. The crosslinked matrices were oxidized at C-6 to generate a carboxyl group. The effect of the hydrogel composition, ion strength, temperature, and simulated seawater (0.55 *M* NaCl and 3 m*M* $NaHCO_3$) on the uranyl ions sorption was investigated using the colorimetric method for the determination of the rejected ions. The effect of functionalization of starch is evident as the structure-property relationship was reflected in the sorption behavior of hydrogels, as the one synthesized from the partially hydrolyzed and oxidized starch exhibited the maximum sorption efficiency. The work is novel and we have not encountered any study where such hydrogels are reported for uranyl ions separation.

EXPERIMENTAL

Materials

Uranyl acetate [$UO_2(CH_3COO)_2 \cdot 2H_2O$], potassium ferrocynate (1 gL^{-1}), sodium chloride (NaCl), and sodium bicarbonate ($NaHCO_3$) and all analytical grade (BDH, India) were used as received without any purification. The concentrations of UO_2^{2+} ions in the sorption experiments were determined with UV-VIS spectrophotometer (Cary 300 UV- spectrophotometer).

Synthesis of Hydrogels

The hydrogels were prepared by following an earlier reported procedure (17). Starch (S) was modified by acidic hydrolysis to obtain low molecular weight hydrolyzed starch (S_H). S and S_H were crosslinked separately with *N,N*-methylenebisacrylamide (*N,N*-MBAAm), and the hydrogels thus obtained are referred as S-*cl*-*N,N*-MBAAm, and S_H-*cl*-*N,N*-MBAAm. The crosslinked hydrogels synthesized were functionalized by oxidation reactions with nitrogen oxides to generate carboxyl groups. The oxidized series is referred to as S_O-*cl*-*N,N*-MBAAm and S_{HO}-*cl*-*N,N*-MBAAm, where O in the subscript presents oxidized state of starch.

Adsorption Procedure, Isotherm Evaluation and Kinetic Models

The uranyl acetate was taken as acetate anion has a positive effect on the uptake of uranyl ions adsorption (18). The adsorption experiments were carried out by immersion of a 0.1 g sample in 50 mL of 230 ppm UO_2^{2+} ions concentration (resultant pH 4.53) at 25°C. The hydrogels were allowed to equilibrate for 24 h without any mechanical agitation. The residual aliquots were withdrawn at definite time interval (30 min, 1 h, 2 h, 4 h, 6 h, and 24 h) and the concentration of the rejected ions from different experimental sets was studied from a calibration curve that was generated by assaying a series of standards with known concentration of UO_2^{2+} ions in the distilled water and simulated seawater, separately. 0.5 mL solution of potassium ferrocynate (1 g/L) was added to 5 mL of UO_2^{2+} solutions and the mixture was kept for 30 min. Potassium ferrocynate was used as a complexing agent to elevate the absorbance in visible region (11). The spectrophotometric measurements were carried out using UV-VIS spectrophotometer. The effect of temperature (25° to 45°C) on the adsorption capacity of hydrogels was investigated at the optimized time and candidate hydrogels those afforded the high percent uptake. The UO_2^{2+} ions concentration was varied between 230 ppm to 3000 ppm at the optimum time and temperature. The effect of electrolyte on adsorption was also studied in the simulated seawater (0.55 M NaCl and 3 *m*M $NaHCO_3$) at 1000 ppm concentration (resulting solution pH 7.7). The results of the adsorption were evaluated by using the following expressions.

$$\text{Percent uptake } (P_u) = \frac{\text{Amount of metal ions sorbed}}{\text{Total ions in the feed solution}} \times 100$$

$$\text{Adsorption Capacity } (Q)(mg/g) = \frac{(C_o - C_t)V}{W}$$

Where, Q is the amount of UO_2^{2+} ions adsorbed onto unit dry mass of the hydrogel or xerogel (mg/g), C_o and C_t are the concentrations of ions in the feed solutions and in the aqueous phase after treatment for a certain period of time t, respectively (mgL^{-1}), V is the volume of the aqueous phase (L), and m is the weight of dry polymer (g).

Characterization of Metal Ions-Loaded Hydrogels

To get evidence of the uranyl ions uptake, the FTIR spectra of the ions-loaded hydrogels were recorded in the range 4000–500 cm^{-1} in KBr on Nicolet 5700 FTIR Spectrophotometer. Evidence of sorption was also obtained by elution experiments on the ion-loaded hydrogels with 0.1 *N* HCl.

RESULTS AND DISCUSSION

Different starch-based hydrogels were synthesized from the native or partially hydrolyzed starch. These were further functionalized by oxidation at the primary hydroxyl groups at C-6 position of the anhydroglucose units of starch. Though the oxidation process is selective, yet some secondary groups are also oxidized to carbonyl groups. The degree of modification was found to be 23.52–27.84% by weight, and the particle size of the sorbent in dry state was 2.0–10 µm. The resultant hydrogel has ionizable carboxylic groups and it is expected to be an efficient sorbent in the uranyl ions uptake. Since the main contribution to the ion uptake in this type of hydrogels is by ion exchange, the consequent release of H^+ from the carboxylic groups of the hydrogel decreases the pH of the solution to the desirable lower levels (17).

Adsorption of UO_2^{2+} on Hydrogels: Effect of Contact Time, Temperature and Ionic Strength

Initially the rate of ion uptake was slow, and it attained equilibrium in 6 h. The effect of functionalization by oxidation on the ion uptake was evident as the non-oxidized S-*cl-N,N*-MBAAm and S_H-*cl-N,N*-MBAAm exhibited the maximum P_u of 5.92% and 7.56% only than P_u of 61.74% and 55.36% exhibited by S_O-*cl-N,N*-MBAAm and S_{HO}-*cl-N,N*-MBAAm, respectively (Fig. 1). The effect of temperature on UO_2^{2+} uptake was evaluated in the temperature range of 25° to 45°C and 230 ppm concentration at 6 h. The adsorption capacity was observed to increase from 61.17 to 80.00 mg/g for S_O-*cl-N,N*-MBAAm and 50.33 to 76.50 mg/g for S_{HO}-*cl-N,N*-MBAAm, when the temperature was increased from 25° to 45°C (Fig. 2). These results are a manifestation of the opening up of the hydrogel structure with temperature that enhances the accessibility of the ions to the active sites on the hydrogel. The effect of ionic strength on the UO_2^{2+} adsorption capacities of hydrogels was investigated within 6 h and at 40°C. P_u increased with an increase in uranyl ions concentration as equilibrium was not achieved even at the high concentration of 3000 ppm, which suggests the abundance of the active binding sites on the sorbent. The adsorption capacity values for S_O-*cl-N,N*-MBAAm were observed to increase from 76 to 578 mg/$g_{xerogel}$ with change of concentration from 230 to 3000 ppm (Fig. 3). Such values for uranyl ions uptake have also been reported in literature, as in the case of *Sargassum*, the adsorbed uranyl

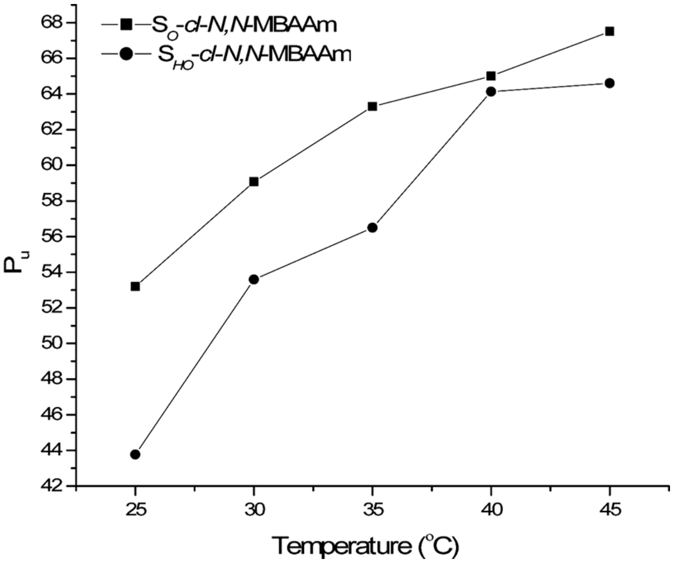

FIG. 2. Sorption of UO_2^{2+} ions with temperature (time = 6 h, sorbent = 0.1 g, concentration = 230).

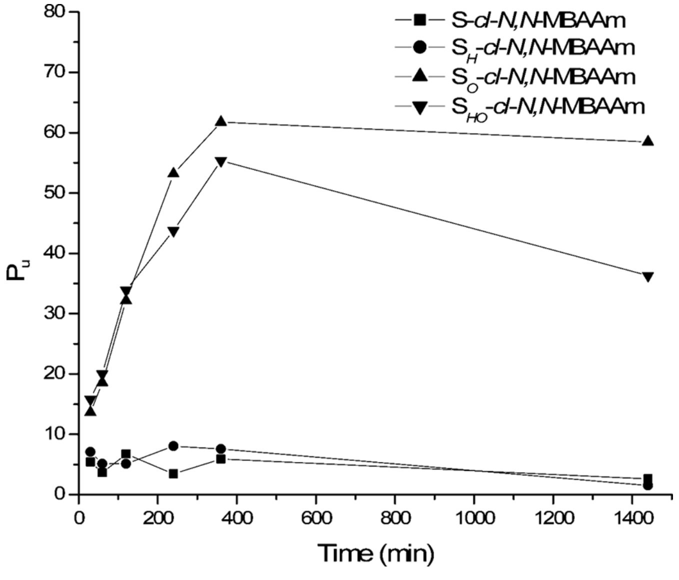

FIG. 1. Effect of hydrogel structure on P_u (temperature = 25°C, concentration = 230 ppm, sorbent = 0.1 g).

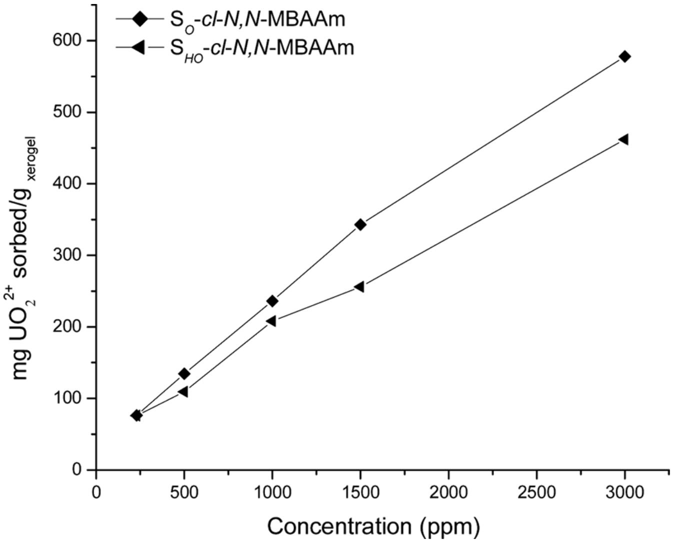

FIG. 3. Sorption of UO_2^{2+} ions with concentration (time = 6 h, temperature = 40°C, sorbent = 0.1 g).

ions constituted more than half of the dry weight of biomass (> 500 mg U/g, the amount exceeding the stoichiometric ion exchange predictions) (19).

Effect of Presence of Electrolyte/s (Saline Water)

From the above discussion it is evident that S_O-*cl-N,N*-MBAAm exhibited the highest efficacy in the uranyl ion uptake from the distilled water. When the sorption of uranyl ions was studied on different hydrogels from the 0.5 *M* NaCl and 3 mM $NaHCO_3$ solution at 6 h and 40°C with 230 ppm of uranyl ions, the effect of the composition of the medium was not appreciable on the adsorption capacity. However, the hydrogels exhibited far rapid adsorption from the distilled water within a much shorter time of 30 min than the saline solution. The maximum adsorption for the S_O-*cl-N,N*-MBAAm was 234 mg/g in sea water compared to 236 mg/g from the distilled water at 240 min, while for the S_{HO}-*cl-N,N*-MBAAm, the maximum adsorption from the saline solution was within 120 min with the magnitude of 222 mg/g in contrast to almost a similar value of 216 mg/g in pure water (Fig. 4). The decrease in the adsorption capacity with time is related to the less swelling or deswelling of the hydrogels in the strong saline solution, hence, less partitioning of the ions to the hydrogel phase from the solution phase.

Evaluation of Adsorption Isotherms and Kinetic Models

The adsorption isotherm plays an important role in the determination of the course taken by the adsorbate. In order to evaluate, the equations of Langmuir and Freundlich have been considered. The Langmuir equation assumed a weak physical sorption of the metal cations on a surface with homogeneous adsorption. In contrast, the Freundlich model is a non-ideal sorption that involves heterogeneous adsorption over independent sites (20). The suitability of the isotherm equations was assessed by the correlation coefficient values (R^2) presented in Table 1. The Freundlich adsorption isotherm exhibits better correlation than the Langmuir adsorption isotherm as the correlation coefficients values (R^2) obtained for the S_O-*cl-N,N*-MBAAm and S_{HO}-*cl-N,N*-MBAAm approaches 1.0 in the case of Freundlich adsorption isotherm with a respective R^2 values of 0.9989 and 0.9878, respectively, while for the Langmuir isotherm these are far lower at 0.8897 and 0.6788 (Table 1). The experimental adsorption capacity results for S_O-*cl-N,N*-MBAAm and S_{HO}-*cl-N,N*-MBAAm exhibited good match with the Freundlich isotherm. Thus the heterogeneity of the sorption process is obvious as ion uptake occurs by ion exchange at the carboxylic groups generated by oxidation process at the C-6 of the anhydroglucose units of starch and also by simple adsorption and diffusion into

TABLE 1
Correlation coefficients for Langmuir and Freundlich isotherm models

Polymers	Constants (R^2) Langmuir	Constants (R^2) Freundlich
S_O-*cl-N,N*-MBAAm	0.8897	0.9989
S_{HO}-*cl-N,N*-MBAAm	0.6788	0.9878

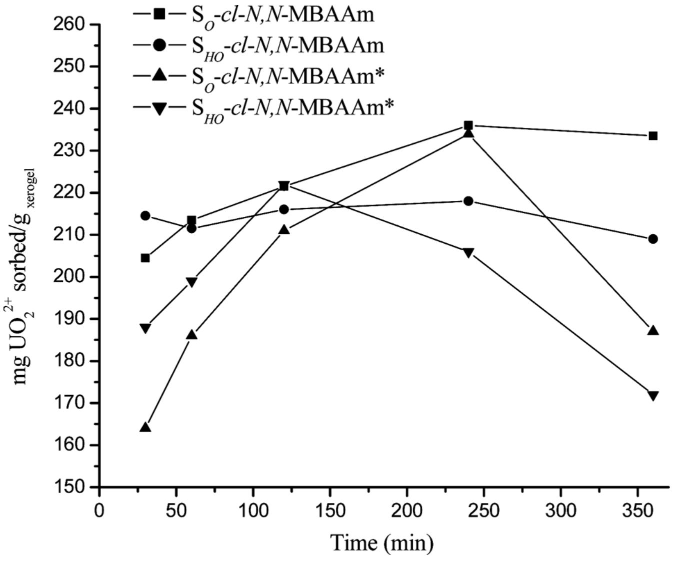

FIG. 4. Comparison of UO_2^{2+} sorption in pure water and simulated sea water (* = simulated sea water, concentration = 1000 ppm, temperature = 40°C, sorbent = 0.1 g).

TABLE 2
Adsorption capacities calculated from Langmuir and Freundlich isotherms for S_O-*cl-N,N*-MBAm and S_{HO}-*cl-N,N*-MBAm their comparison with the experimental values

C_e (mg/L)	Experimental (Q)	Langmuir (Q)	Freundlich (Q)
S_O-*cl-N,N*-MBAm			
230	76.5	67.735	74.862
500	134.5	139.486	138.867
1000	236	254.155	241.157
1500	343	350.09	333.0427
3000	578	562.366	578.096
S_{HO}-*cl-N,N*-MBAm			
230	76	56.26	70.372
500	109.5	114.73	122.462
1000	208	205.846	200.86
1500	256	279.955	268.287
3000	462	437.445	439.542

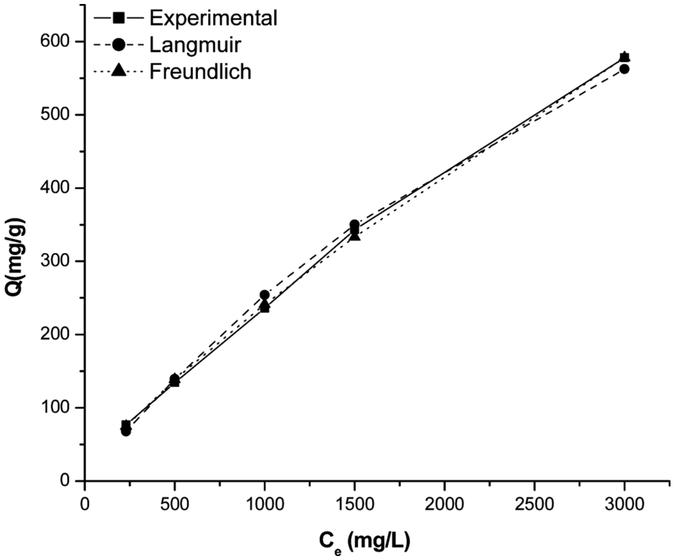

FIG. 5. Equilibrium isotherms (Langmuir ($C_e/Q_e = (1/K_L)+(a_L/K_L)C_e$) and Freundlich ($\log Q_e = \log K_F + (1/n)\log C_e$)) comparison for S_O-*cl-N,N*-MBAAm with experimental results.

the bulk of the hydrogels, especially at the amide groups of the crosslinker (Table 2 and Figs. 5 and 6, respectively).

The kinetics of the adsorption is related to the specificity of the interaction of heavy metal ions with the polymeric matrix. In order to examine the mechanism of biosorption processes, such as mass transfer and chemical reaction, the two kinetic models were analyzed (21).

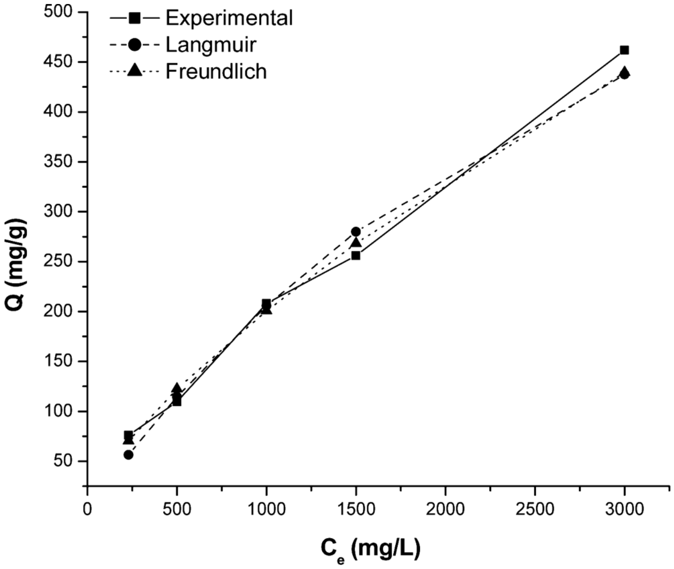

FIG. 6. Equilibrium isotherms (Langmuir ($C_e/Q_e = (1/K_L) + (a_L/K_L)C_e$) and Freundlich ($\log Q_e = \log K_F + (1/n)\log C_e$) comparison for S_{HO}-*cl-N,N*-MBAAm with experimental results.

TABLE 3
Correlation coefficients for pseudo-first order and pseudo-second order kinetic Models

	Constants (R^2)	
Polymers	Pseudo First-order	Pseudo Second-order
S_O-*cl-N,N*-MBAAm	0.9972	0.9996
S_{HO}-*cl-N,N*-MBAAm	0.9667	0.9996

The pseudo-second order equation provided the best correlation for the sorption process of uranyl ions for S_O-*cl-N,N*-MBAAm and S_{HO}-*cl-N,N*-MBAAm, as is evident from the R^2 values from the first-order and second order kinetic models (Table 3). The applicability of the pseudo-second order kinetic model in this case signifies dominance of chemisorption/complexation mechanism. It was found to be applicable over the whole range of the contact time studied both for S_O-*cl-N,N*-MBAAm and S_{HO}-*cl-N,N*-MBAAm (Table 4, Figs. 7 and 8, respectively).

Evidence of Ion Sorption by Characterization

The IR spectra of the UO_2^{2+}-loaded hydrogels and their precursor are presented in Fig. 9. The characteristic absorption peaks for –OH stretching near 3200 cm^{-1} to 3400 cm^{-1}, carbonyl group stretching peak at 1750 cm^{-1},

TABLE 4
Adsorption capacities calculated for S_O-*cl-N,N*-MBAm and S_{HO}-*cl-N,N*-MBAm from pseudo-second order kinetic equation and their comparison with the experimental values

T(min)	Experimental (Q)	Pseudo Second-order (Q)
S_O-*cl-N,N*-MBAm		
30	164	150.45
60	186	187.852
120	211	214.512
240	234	230.902
360	240	236.936
S_{HO}-*cl-N,N*-MBAm		
30	188	175.336
60	199	199.933
120	206	215.015
240	222	223.443
360	225	226.401

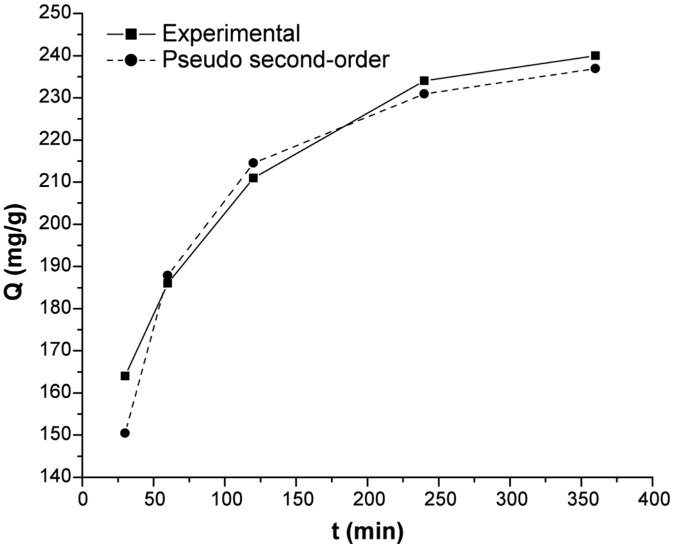

FIG. 7. Kinetics of uranyl ions sorption for S_O-*cl-N,N*-MBAAm.

carboxylate anion peaks at 1650 cm^{-1} and 1400 cm^{-1} were observed in all the hydrogels (Fig. 9). The comparison of these spectra with the UO_2^{2+} ions-loaded hydrogels reveals that there is a shift in the position and intensity of the characteristics bands initially observed in the precursor hydrogels. The strong interactions between UO_2^{2+} ions and the functional groups of the hydrogel are also confirmed from the presence of an additional peak around 925 cm^{-1}, which is characteristic of O=U=O stretching vibration (22). Recovery of uranyl ions from the hydrogels was quantitative when eluted with 0.1 *N* HCl.

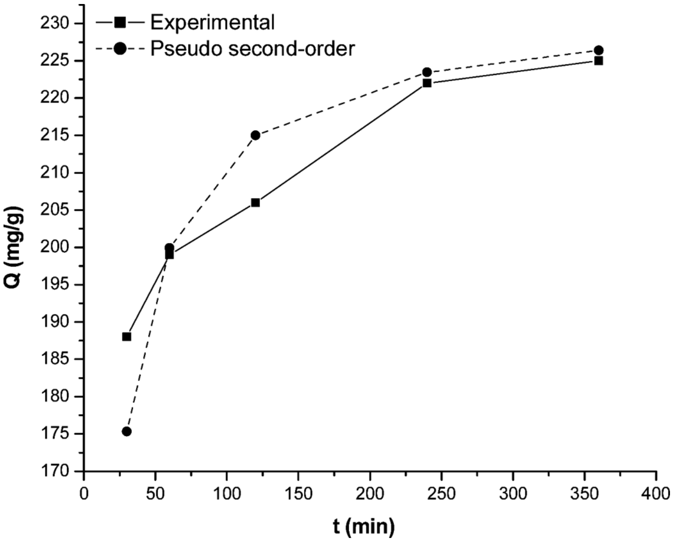

FIG. 8. Kinetics of uranyl ions sorption for S_{HO}-*cl-N,N*-MBAAm.

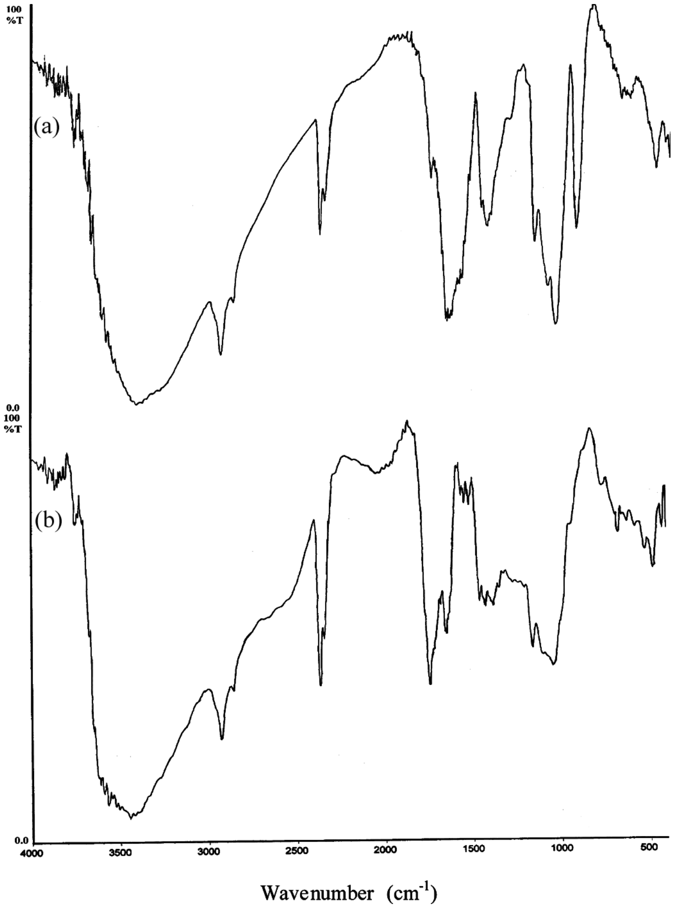

FIG. 9. FTIR spectra of (a) S_O-*cl-N,N*-MBAAm; (b) S_O-*cl-N,N*-MBAAm-loaded with UO_2^{2+}.

CONCLUSIONS

In the continuing search for a suitable biosorbent to supplement environmentally recalcitrant synthetic polymers, the functionalization of starch is a useful option to synthesize efficient and environmentally friendly sorbents. Functionalization of starch to hydrogels by the controlled hydrolysis and oxidation affected its structure-property relationship and increased its proficiency as sorbent for UO_2^{2+} ions. The sorption was affected by the hydrogel structure as well as external stimuli such as temperature and ion concentration. The hydrogels exhibited high adsorption capacity up to 578 $mg/g_{zerogel}$. The hydrogels follow the Freundlich isotherm and pseudo-second order kinetic models, which signify the heterogeneity of UO_2^{2+} ions adsorption, suggesting chemisorption as the predominant mode of sorption.

ACKNOWLEDEGEMENTS

This study was supported by the University Grants Commission, New Delhi, India (under Grant No. F. No. 30/47/2004(SR) November 1, 2004). The facilities provided by the Department of Chemistry of Himachal Pradesh University under UGC-SAP and DST-FIST are also gratefully acknowledged.

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