Synthesis, Characterization, and Swelling Studies of pH- and Thermosensitive Hydrogels for Specialty Applications

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ABSTRACT: In this article, we describe a novel pH-and temperature-sensitive and moderately salt tolerant hydrogels based on acrylic acid (AAc) and methacrylic acid (MAAc) by employing a novel research plan. AAc and MAAc have been converted to *N*-isopropylacrylamide (*N*-*i*-PAAm) and *N*-isopropyl methacrylamide (*N*-*i*-PMAAm), respectively, via amidation with isopropyl amine, followed by polymerization to respective polymers. These polymers were further converted into networks in the presence of AAc and MAAc and *N*,*N*-methylene bisacrylamide (*N*,*N*-MBAAm). The networks thus formed consist of both amides and carboxylic acid groups. Characterization of

these networks has been carried out by nitrogen analysis, FTIR, NMR, and scanning electron microscopy for the investigation of different structural aspects of the synthesized polymers. Swelling studies of these networks have been carried out as a function of time, temperature, pH, and in the presence of additives, sodium dodecyl sulfate, cetyl trimethyl ammonium bromide, and 5% NaCl solution. © 2008 Wiley Periodicals, Inc. J Appl Polym Sci 109: 47–55, 2008

Key words: hydrogels; salt tolerance; surfactant; swelling; thermosensitivity

INTRODUCTION

High water-absorbing hydrogels sensitive to variation in pH and ionic strength are reported from acrylic acid (AAc) and methacrylic acid (MAAc).¹ The use profile of these hydrogels was improved by way of partial modification with amidation or other polymer analogous reactions like hydrolysis and even by loading of metal ions.² Modification of AAc and MAAc by amidation results in many interesting properties including induction of salt tolerance and thermosensitivity. Iizawa et al.3 have reported the synthesis of thermosensitive poly(*N*-alkylacrylamide) hydrogels by amidation of poly(AAc). The hydrogels based on acrylamide (AAm) and AAc are reported to exhibit good dimensional stability and high yet reversible swelling at 60°C.⁴ Poly(*N-i*-PAAm) macroporous hydrogels have been reported to have high swelling ratio at temperatures below the lowest critical solution temperature (LCST), and these were reported to respond to temperature changes in a sharp manner.⁵ Its macroporous copolymeric hydrogel with poly(2-hydroxy ethyl methacrylate) has

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been reported as support in solid-phase synthesis.⁶ The presence of the poly(sodium acrylate) in the network with poly(N-i-PAAm) was reported not to influence the LCST or the extent of phase transition of the latter.⁷ In the case of poly(*N-i*-PAAm-co-AAc), temperature-induced gradual phase transition is driven by the protonation/ionization of the AAc.⁸ The LCST of poly(N-i-PAAm-co-sodium acrylate-co-N-tert-butylacrylamide) can be adjusted by changing the chemical composition of the polymers and these gels behave as thermosensitive super absorbent.⁹ Hydrogels of N-i-PAAm and MAAc or its esters have also been reported. Dynamic swelling behavior of poly(N-i-PAAm-co-MAAc) hydrogels is also affected by hydrogen bond formation.¹⁰ Swelling and mechanical behavior of the ionized networks of poly(sodium methacrylate)-cl-N,N-MBAAm was reported as a function of temperature in water and NaCl solution.¹¹ Apart from the effect of the copolymeric composition and also of the external environmental factors; surfactant-polymer interactions, especially, at the critical micelle concentration (CMC) of the surfactant affects swelling behavior of the hydro-gels in a significant manner.^{12–16} The presence of surfactants affects swelling behavior of hydrogels by micelle formation.¹⁷ The local mobility of the surfactant molecules those enter association with the hydrogel is considerably higher than in the complex with a compact conformation, and this mobility grows with the increasing number of micelles involved in the associates.¹⁸

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In the present article, we report synthesis and characterization of poly(*N-i*-PAAm) and poly(*N*-iso-propyl methacrylamide) [poly(*N-i*-PMAAm)]-based pH sensitive, salt–resistant, and thermosensitive hydrogels. Different polymers and networks prepared have been characterized by scanning electron microscopy (SEM), nitrogen analysis, FTIR, and NMR. The swelling behavior of the hydrogels has been studied as a function of time, temperature, pH, and also in the presence of various additives including sodium dodecyl sulfate (SDS), cetyl trimethyl ammonium bromide (CTAB), and sodium chloride.

EXPERIMENTAL

Materials

Acrylic acid, methacrylic acid, ammonium persulphate (APS), and isopropyl amine (S.D. Fine, Mumbai), *N*,*N*-methylene bisacrylamide, SDS, sodium chloride, buffer tablets (4.0, 7.0, and 9.2), and cetyl trimethyl ammonium bromide (Merck, Schuchardt, Germany) were used as received.

Modification of AAc and MAAc

AAc (0.28 mol) was treated with an equimolar amount of isopropyl amine. The mixture was allowed to stand for 30 min, and thereafter polymerization was initiated by the addition of APS (1% of combined weight of AAc and amine used and predissolved in 2.0 mL of water). The reaction mixture was heated at 60°C for 30 min in a controlled water bath to ensure complete polymerization. The reaction system was treated with minimum quantity of cold water, and the polymer was separated by filtration, followed by drying the resultant poly(*N-i*-PAAm) in a vacuum oven at 40°C. Following the same procedure, MAAc was modified to poly(*N-i*-PMAAm).

Preparation of networks

The dried sample of poly(*N-i*-PAAm) was grounded to a fine powder. Dry polymer (10.0 g) was taken in

a reaction vessel in 20.0 mL of water at 50°C. It was followed by the addition of 10.0 g of AAc and *N*,*N*methylene bisacrylamide [2.5% by weight of the total weight of the poly(*N*-*i*-PAAm) and AAc] and APS (1% of the total weight). The reaction system was heated to 60°C for 30 min to ensure complete crosslinking. The network was treated with water in a Soxhlet (to extract sol fraction), and dried at 30°C in a vacuum oven. The extraction and drying cycles were repeated to obtain constant weight. Poly(*N*-*i*-PMAAm) was modified following the similar procedure using MAAc.

Characterization of polymers

Different polymers were characterized by using SEM (taken on Jeol JSM-6100 SEM) and FTIR spectra (recorded on Perkin–Elmer and Nicolet FTIR Spectrophotometer in KBr). NMR spectra of the candidate polymers were recorded on Bruker AC-200 MHz using TMS as an internal standard in CDCl₃. Nitrogen analysis was carried out on Carlo Erba EA-1108.

Swelling studies of hydrogels

The known weight (0.1 g) of the different networks was immersed in water. Swelling was measured gravimetrically at different time intervals (30-720 min) at different temperatures (25–45°C) in a temperature-controlled bath. The swollen hydrogels were wiped off with a tissue paper to remove surface water and weighed immediately on Denver TR 203 (minimum readability of 0.001 g). The earlier process was carried out in triplicate sets. Similar process was repeated to study the effect of pH on the swelling kinetics and concentrations of two surfactants (anionic - SDS and other cationic - CTAB) at 35°C at the aforementioned time intervals. Salt tolerance of the polymers was studied by using 5% NaCl solution as the swelling medium at 35°C. Percent swelling (P_s) of the networks was calculated by applying the following relationship:

 $P_s = \frac{\text{Weight of the swollen hydrogel} - \text{weight of the dry hydrogel}}{\text{Weight of the dry hydrogel}} \times 100$

RESULTS AND DISCUSSION

Modification of AAc and MAAc with isopropyl amine to form poly(*N-i*-PAAm) and poly(*N-i*-PMAAm) was followed by network formation with AAc and MAAc as the second component. The formation of amides from AAc and MAAc is exemplified by the reaction of AAc and isopropyl amine as shown below:

$$CH_2 = CH - COOH + H_2N - CH(CH_3)_2$$
$$\longrightarrow CH_2 = CHCONH - CH(CH_3)_2 \quad (1)$$

In the presence of APS, (1) polymerizes to poly(*N-i*-PAAm), and addition of AAc in the second step results in the grafting and consequent crosslinking to three-dimensional network of poly(AAc) and poly(*N-i*-PAAm) in the presence of crosslinker. The two networks, thus obtained, contain both amide and carboxylic acid groups and are presented as poly(*N-i*-PAAm-*co*-AAc)-*cl*-*N*,*N*-MBAAm and poly (*N-i*-PMAAm-*co*-MAAc)-*cl*-*N*,*N*-MBAAm.

Characterization of polymers

FTIR spectra of polymers

FTIR spectrum of poly(N-i-PAAm) has peaks at 3445 cm⁻¹ (NH stretching), 1657 cm⁻¹ (C=O stretching of secondary amide), 1227 cm⁻¹ (C–N stretching), 1167 cm⁻¹, and 1127 cm⁻¹ (assigned to isopropyl gemdimethyl group) [Fig. 1(A)]. In the spectrum of poly(N-i-PAAm-co-AAc)-cl-N,N-MBAAm, important peaks appear at 3485 cm⁻¹ (NH stretching), 1719 cm⁻¹ (C=O acid stretching), 1654 cm^{-1} (C=O stretching of amide), 1235 cm^{-1} (C—N stretching), and 1123 cm^{-1} (assigned to isopropyl gemdimethyl group) [Fig. 1(A)]. FTIR spectrum of poly(N-i-PMAAm) has peaks at 3415 cm^{-1} (NH stretching) and 1642 cm^{-1} (C=O stretching) of amide) [Fig. 1(B)]. The spectrum of poly(N-i-PMAAm-co-MAAc)-cl-N,N-MBAAm has peaks at 3440 cm^{-1} (NH stretching), 1720 cm^{-1} (C=O acid stretching), 1647 cm⁻¹ (C=O stretching of amide), 1265 cm⁻¹ (C-N stretching), and 1138 cm⁻¹ (assigned to isopropyl/gemdimethyl groups) [Fig. 1(B)].

¹H and ¹³C NMR spectra of the poly(N-i-PAAm) and poly(N-i-PMAAm)

¹H NMR spectra of the candidate polymers have been shown in Figure 2(A,B) and ¹³C NMR spectra in Figure 2(C,D). The interpretations of NMR were used to elucidate the structure of these polymers. The amidation is supported by the (i) presence of substituents both from the precursor amines and monomers, (ii) absence of signal due to the expected absorption of the reacting groups, and (iii) presence of new groups generated after amidation reactions.

The ¹H NMR spectrum of poly(*N*-*i*-PAAm) shows a doublet at 0.8–0.95 δ equivalent to 6 H of two –CH₃ groups (assigned as a). A septet at 3.0–3.4 δ equivalent to H of –CH group (assigned as b) is deshielded due to adjacent N. A singlet at 3.8 δ due to –NH proton (assigned as c) is more deshielded. A quintet at 2.0–2.2 δ equivalent to H of –CH (assigned as d) and a triplet at 1.5 δ equivalent to 4 H of two –CH₂ group (assigned as e) have been observed [Fig. 2(A)]. In the ¹H NMR spectrum of poly(*N*-*i*-PMAAm), a doublet at 0.8–1.0 δ equivalent to 6 H of two $-CH_3$ groups (assigned as a) and a septet at 3.2–3.5 δ equivalent to 1 H of two --CH (assigned as b) have been observed. Apart from these, a singlet at 4.0 δ equivalent to H of -- NH (deshielded and assigned as c), a singlet at 1.7 δ equivalent to 2H of two $-CH_2$ groups (assigned as d), and a singlet at 1.9 δ equivalent to 3H of $-CH_3$ group (assigned as x) have also been observed [Fig. 2(B)]. The downfield shift of the signals up to a fraction of a ppm in the case of the poly(*N-i*-PMAAm) is explained by the fact that it can assume different tacticity because of the presence of a $-CH_3$ group, which is not possible by the other polymer. These modifications are further supported by ¹³C NMR spectrum. The ¹³C NMR spectrum of poly(*N-i*-PAAm) shows a quartet at 20 ppm due to two $-CH_3$ groups (assigned as a). Two doublets at 42 ppm and 50 ppm are equivalent to two --CH (assigned as b and d, respectively). A triplet at 30–35 ppm is due to two –CH₂ groups (assigned as e) and a singlet at 182 ppm is equivalent N-alkyl amide (assigned as y) [Fig. 2(C)]. The ¹³C NMR spectrum of poly(*N-i*-PMAAm) shows a quartet at 20 ppm equivalent to two -CH₃ groups (assigned as a), a doublet at 50 ppm is equivalent to --CH (assigned as b), and a triplet at 40-45 ppm is equivalent to -CH₂ (assigned as d). A quartet at 25–35 ppm is due to $-CH_2$ (assigned as x) and a singlet at 186 ppm is equivalent to N-alkyl amide [Fig. 2(D)]. From this data, the structure assigned are:

Nitrogen analysis

The high percent of nitrogen has been found in both AAm/MAAm and their networks. A comparison of the N present in a known weight of each of the polymer analyzed is given in Table I. In the case of poly(*N-i*-PAAm), the % amine incorporated has been found to be 51.32 (5.68% N), meaning thereby almost 100% conversion. But when this copolymer is modified by adding AAc and simultaneously crosslinked with *N*,*N*-MBAAm to form crosslinked poly(*N-i*-PAAm-*co*-AAc), % N in the sample was found to be only 3.39% because of the higher incorporation of AAc compared to the crosslinker which has two nitrogen per molecule. The result reveals that poly (AAc) has been incorporated into the crosslinked



Figure 1 (A,B) FTIR spectra of poly(*N-i*-PAAm) and poly(*N-i*-PAAm-*co*-AAc)-*cl*-N,*N*-MBAAm. (C,D) FTIR spectra of poly(*N-i*-PMAAm) and poly(*N-i*-PMAAm-*co*-MAAc)-*cl*-N,*N*-MBAAm.

networks. Similar trends have been observed for poly(*N-i*-PMAAm) and crosslinked poly(*N-i*-PMAAm-*co*-AAc).

Scanning electron micrography

SEMs of the two networks at different magnifications are shown in Figure 3(A,B). The contrast in the morphologies of the two networks is evident. Poly(*N*-*i*-

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PAAm-*co*-AAc)-*cl*-*N*,*N*-MBAAm is less intensely crosslinked, as compared to the other network. In this case, pores of different sizes are visible. While from the SEMs of poly(*N*-*i*-PMAAm-*co*-MAAc)-*cl*-*N*,*N*-MBAAm, more intense crosslinking and smaller pores are visible. These differences in the morphologies of the two networks are expected to affect the property profile, especially the water uptake of the two networks.





Figure 2 (A,B) ¹H NMR spectra of poly(*N-i*-PAAm) and poly(*N-i*-PMAAm) in CDCl₃. (C,D) ¹³C spectra of poly(*N-i*-PAAm) and poly(*N-i*-PMAAm).

Swelling studies of hydrogels

Hydrophilicity and the presence of ionizing groups affect swelling behavior or swelling extent of the polymeric networks. The nature and concentration of the crosslinker, polymerization conditions, and network composition also affects swelling behavior of the network, as these affect crosslinking density, and consequently, size of the pores of a network. Swelling behavior of a network is also strongly affected by the nature of the swelling medium and other components of the swelling environment. In the present study, the networks contain amides as well as carboxylic groups; hence, these have large hydrophilic area.

Swelling behavior as a function of time and temperature

The effect of swelling time on swelling behavior of different networks at different temperature is pre-

sented in Figure 4(A,B). The effect of the nature of copolymeric composition is evident from the variation and temperature with time. Generally, equilibrium swelling is attained at 480 min and 35°C. *P_s* of poly(*N-i*-PAAm-*co*-AAc)-*cl*-*N*,*N*-MBAAm is much higher than poly(N-i-PMAAm-co-MAAc)*cl-N,N-MBAAm.* The sharp contrast in the P_s values of the two hydrogels, especially, the lower P_s in the latter is attributed to the presence of $-CH_3$ in its precursor (MAAc) that contributes to the hydrophobic regions in the hydrogels and consequently decreases its water interaction capacity. Further, its morphology also reveals more intense crosslinking as compared to the other hydrogel; hence, lower water uptake by this hydrogels is expected. It is interesting to observe that in the case of poly(AAc)-based hydrogel [Fig. 4(A)] there is differentiation in the swelling behavior with time, while it does not vary much in the other

TABLE I Nitrogen Analysis of the Polymers

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Polymer	Weight of sample (mg)	% N found	Weight of N (mg)	Weight of amine (mg)	% Amine incorporated
Poly(N-i-PAAm)	2.704	5.68	0.335	1.411	51.32
Poly(N-i-PAAm-co-AAc) ^a	2.167	3.39	0.341	1.436	21.72
Poly(<i>N-i-</i> PMAAm)	1.880	4.8	0.207	0.858	45.66
Poly(N-i-PMAAm-co-MAAc) ^a	1.95	3.20	0.2151	0.891	20.64

^a N,N-MBAAm.



Figure 3 (A) SEMs of poly(*N-i*-PAAm-*co*-AAc)-*cl*-*N*,*N*-MBAAm at different magnifications. (B) SEMs of poly(*N-i*-PMAAm-*co*-MAAc)-*cl*-*N*,*N*-MBAAm at different magnifications.

hydrogel [Fig. 4(B)]. Before attaining equilibrium, a rapid change in the swelling rate with an increase in temperature has been observed for both the hydrogels [Fig. 4(A,B)]. It is more so in the case of poly(*N*-*i*-PAAm-*co*-AAc)-*cl*-*N*,*N*-MBAAm, which shows a sharp increase in P_s from 8157 to 12,074 when temperature was increased from 30 to 35°C. The positive sharp volume transition in poly(AAc)-based hydrogel manifests that the LCST of poly(*N*-*i*-PAAm) is not expressed in the presence of poly(AAc) in the network.

Swelling behavior as a function of pH

The effect of the nature of copolymeric composition is also evident on swelling when the pH was varied from in the range of 4, 7.0, and 9.0 [Fig. 4(C,D)]. At 35° C, pH sensitivity of these hydrogels is evident, as these hydrogels show sharp volume transitions on change of pH from 4.0 to 7.0. Thereafter, change in pH to 9.0 does not affect the swelling behavior in poly(*N-i*-PAAm-*co*-AAc)-*cl*-*N*,*N*-MBAAm; however, in poly(*N-i*-PMAAm-*co*-MAAc)-*cl*-*N*,*N*-MBAAm, *P*_s decreased significantly. AAc-based hydrogel exhibited much higher *P*_s as compared to MAAc-based hydrogel. These trends in swelling behavior originate from the suppression of the ionized carboxylic groups at the lower pH and result is shriveling of

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the hydrogels. These results are also manifestation of the interpolymer complexation between poly(AAc) and poly(*N-i*-PAAm) side chains due to strong hydrogen bonding that results in the formation of a transient network of poly(*N-i*-PAAm/poly(AAc).¹⁹ Such complex formation makes the hydrophilic groups less available for the formation of hydrogen bonds with water. In the alkaline pH (i.e., 9.2), the hydrogels interact with the basic species, again restricting the interaction of water molecules with those of acids. In other words, the expected interactions, especially, with the nonionized carboxylic groups and water are reduced, as the former shows preference for the basic moieties of the buffer.

Swelling behavior in the presence of surfactants

Swelling studies were carried on different hydrogels in the presence of SDS and CTAB. As aforesaid, SDS and CTAB are known to affect swelling by micelle formation.¹⁷ Swelling studies of the hydrogels were carried out in SDS solutions in the concentration range from 4.05 to 16.20 mM/L at 35°C in different swelling times [Fig. 4(E,F)]. Generally, swelling increased with time and reached the maximum at 480 min. At the same time, P_s increased with the increase in [SDS] from 4.05 to 8.1 mM/L, i.e., CMC of SDS, and thereafter, it decreases. In contrast to the



Figure 4 (A) P_s of poly(*N-i*-PAAm-*co*-AAc)-*cl*-*N*,*N*-MBAAm as a function of temperature at different time intervals (pH = 7.0). (B) P_s of poly(*N-i*-PMAAm-*co*-MAAc)-*cl*-*N*,*N*-MBAAm as a function of temperature at different time intervals (pH = 7.0). (C) P_s of poly(*N-i*-PAAm-*co*-AAc)-*cl*-*N*,*N*-MBAAm as a function of pH at different time intervals at 35°C. (D) P_s of poly(*N-i*-PAAm-*co*-AAc)-*cl*-*N*,*N*-MBAAm as a function of pH at different time intervals at 35°C. (E) P_s of poly(*N-i*-PAAm-*co*-AAc)-*cl*-*N*,*N*-MBAAm as a function of pH at different time intervals at 35°C. (E) P_s of poly(*N-i*-PAAm-*co*-AAc)-*cl*-*N*,*N*-MBAAm as a function of [SDS] at different time intervals at 35°C. (F) P_s of poly(*N-i*-PAAm-*co*-AAc)-*cl*-*N*,*N*-MBAAm as a function of [SDS] at different time intervals at 35°C. (G) P_s of poly(*N-i*-PAAm-*co*-AAc)-*cl*-*N*,*N*-MBAAm as a function of [CTAB] at different time intervals at 35°C. (I) P_s of poly(*N-i*-PAAm-*co*-AAc)-*cl*-*N*,*N*-MBAAm as a function of [CTAB] at different time intervals at 35°C. (I) P_s of poly(*N-i*-PAAm-*co*-AAc)-*cl*-*N*,*N*-MBAAm as a function of [CTAB] at different time intervals at 35°C. (I) P_s of poly(*N-i*-PAAm-*co*-AAc)-*cl*-*N*,*N*-MBAAm and poly(*N-i*-PAAm-*co*-AAc)-*cl*-*N*,*N*-MBAAm in 5% NaCl as a function of time at 35°C.

sharp volume phase transitions in case of poly (MAAc)-based hydrogel [Fig. 4(F)], swelling did not increase sharply in the case of poly(AAc)-based

hydrogel [Fig. 4(E)]. The swelling of hydrogels in the presence of CTAB was studied in the concentration range from 0.46 to 1.84 mM/L [Fig. 4(G,H)]. The

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Figure 4 (Continued from the previous page)

observed results show almost same trends as have been observed in the presence of SDS, and the maximum P_s was observed at 0.92 mM/L. However, the extent of swelling was lesser as compared to that was observed in SDS. This observation supports that poly(AAc) or poly(MAAc) is not much ionized in the hydrogels; hence, the anionic surfactant (SDS) interacts more than the cationic surfactants (CTAB), with the hydrogels at the $-CO_2H$ groups. Micelle formation is further supported by the observation that maximum volume transition has been observed near the reported CMC of both the surfactants at 35°C.^{20,21} Further, the copolymer network formation offers suitable environment for micellization because of the presence of both hydrophobic and hydrophilic groups, which otherwise is not present in the homopolymer networks, as poly(AAc)-cl-N,N-MBAAm and poly(MAAc)-cl-N,N-MBAAm swell far lesser than the two copolymeric hydrogels in the presence of surfactants.²²

Salt tolerance of the hydrogels: Swelling in the presence of sodium chloride

The high swelling of the hydrogels in the presence of salts is important from the technological viewpoint. Hydrogel shriveling results from the loss of hydrophilic/hydrophobic balance of the networks in the presence of electrolyte salts. Thus, the preswollen gels shrink quickly and regain their original shape and weight by deswelling when they are subjected to swelling in the presence of electrolyte solutions. In the present study, the two networks show lesser swelling as compared to that in pure water, yet significant swelling and hence salt tolerance in 5% NaCl (w/v) solution [Fig. 4(I)]. The order of salt tolerance of networks is the same, as is their swelling behavior in pure water. Amidation of AAc improves salt tolerance. In the presence of 5% NaCl solution, poly (N-i-PAAm-co-AAc)-cl-N,N-MBAAm shows higher P_s (hence more salt tolerance), as compared to poly(N-i-PMAAm-co-MAAc)-cl-N,N-MBAAm.

CONCLUSIONS

It follows form the foregone discussion that these hydrogels respond very quickly to even small changes in their external environment and are thus are smart or environment-sensitive hydrogels. It can be concluded that structural differences like hydrophilicity of the incorporated monomers or crosslinkers and external environmental factors affect and govern swelling behavior of these hydrogels. The results reported in this article are promising both from the fundamental and technological point of view. These absorb appreciable amount of water, with a percent swelling as high as 13,000. The maximum water absorption by these hydrogels at 35°C, at pH 7.0, interactions with surfactants by means of micellization and appreciable salt tolerance make these hydrogels candidate supports in bioseparation, enzyme immobilization, and also in the field of metal ion sorption including uranyl ions.

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