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ORIGINAL CONTRIBUTION

Tuncer Çaykara
Melike Demiray
Olgun Güven

Effect of type and concentration of surfactants on swelling behavior of poly[*N*-[3-(dimethylamino) propyl]methacrylamide-co- *N,N*-methylene-bis(acrylamide)] hydrogels

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T. Çaykara (✉) · M. Demiray
Department of Chemistry,
Faculty of Science, Gazi University,
06500 Besevler, Ankara, Turkey

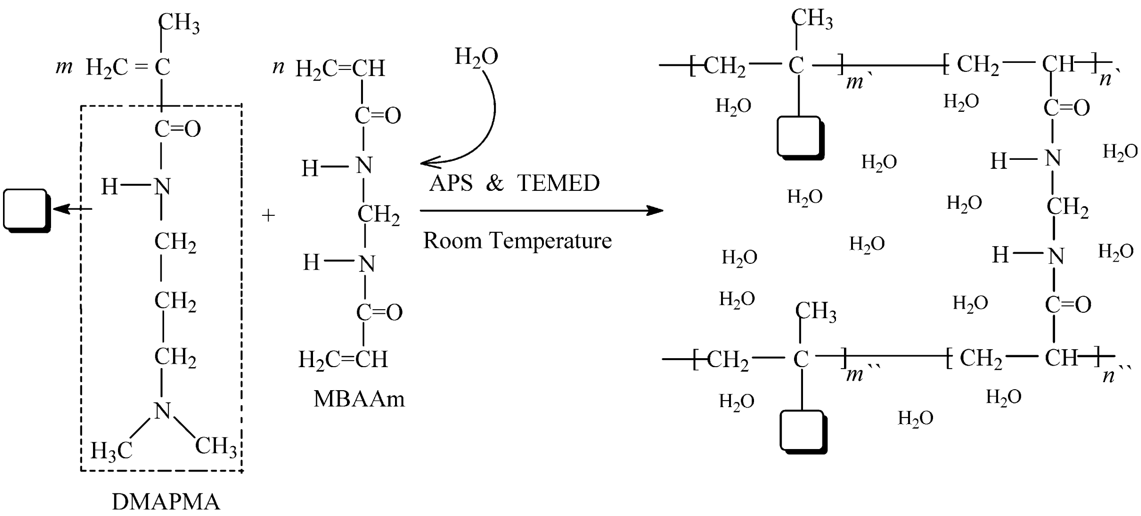
O. Güven
Department of Chemistry,
Hacettepe University,
06532 Beytepe, Ankara, Turkey

Abstract A series of thermosensitive hydrogels were prepared from *N*-[3-(dimethylamino)propyl]methacrylamide (DMAPMA) monomer by using 11.6–17.8% (m/m) *N*,*N*-methylenebis(acrylamide) (MBAAm) as the crosslinker and comonomer in water. A kinetic study of the absorption determined the transport mechanism. The diffusion coefficients of these hydrogels were calculated for the Fickian mechanism. It was shown that the swelling behavior of the P(DMAPMA-co-MBAAm) hydrogels can be controlled by changing the amount of MBAAm. The swelling equilibrium of the P(DMAPMA-co-MBAAm) hydrogels was also investigated as a function of temperature in aqueous solutions of the anionic surfactant sodium dodecyl sulfate (SDS) and the cationic surfactant dodecyltrimethylammonium bromide (DTAB). In pure water, irrespective of the amount of MBAAm, the P(DMAPMA-co-MBAAm) hydrogels showed a discontinuous phase transition between 30 and 40 °C. However, the transition changed from discontinuous to continuous with the addition of surfactants, this is ascribed to the conversion of non-ionic P(DMAPMA-co-MBAAm) hydrogel into polyelectrolyte hydrogels due to binding of surfactants through the hydrophobic interaction. Additionally, the amount of free SDS and DTAB ions was measured at different temperatures by a conductometric method, it was found that the electric conductivity of the P(DMAPMA-co-MBAAm) – surfactant systems depended strongly on both the type and concentration of surfactant solutions.

Keywords Hydrogel · Swelling · *N*-[3-(dimethylamino)propyl]methacrylamide · Surfactants

Introduction

The interaction of nonionic gels with charged surfactants was extensively studied in recent years [1–4]. This process was shown to be governed mainly by electrostatic and hydrophobic forces. The electrostatic interactions prevail when a gel and a surfactant are oppositely charged, while in other cases (a gel and a surfactant of similar charges, uncharged gel and charged surfactant) hydrophobic interactions dominate. The interaction between surfactants and hydrogels is also of great importance in understanding the fundaments of hydrogel volume phase transition because of the amphoteric property of surfactants. Hydrogel can be ionized upon binding of ionic surfactant molecules to the polymeric network through hydrophobic interactions. The swelling ratios and the volume phase transition temperatures of hydrogels were found to be remarkably enhanced, which was interpreted on the basis of electrostatic repulsion due to binding of ionic surfactants to polymer chains. The change in the transition temperature was found to be influenced strongly



Scheme 1

by the addition of small amounts of ionic surfactants [5–7].

In this investigation, the following two types of surfactants which have dodecyl groups as the hydrophobic chain have been used: the anionic surfactant $CH_3(CH_2)_{11}SO_4Na$ (sodium dodecyl sulfate, SDS) and the cationic surfactant $CH_3(CH_2)_{11}N(CH_3)_3Br$ (dodecyltrimethylammonium bromide, DTAB).

In this study, poly(*N*-[3-(dimethylamino)propyl]methacrylamide-co- *N, N*-methylenebis(acrylamide)) [P(DMAPMA-co-MBAAm)] hydrogels with different levels of crosslinking were synthesized from DMAPMA monomer and MBAAm comonomer or crosslinker in water. The effects of ionic surfactants on the equilibrium swelling ratio of P(DMAPMA-co-MBAAm) hydrogels were studied in water and in aqueous system as a function of surfactant type and their respective concentrations. The association of ionic surfactants with the P(DMAPMA-co-MBAAm) chains was further investigated by conductometric measurements for aqueous solutions including both the surfactant and hydrogel.

Experimental procedure

Materials

The monomer *N*-[3-(dimethylamino)propyl]methacrylamide (DMAPMA), the crosslinker or comonomer *N,N*-methylenebisacrylamide (MBAAm), the initiator ammonium persulfate (APS), the accelerator *N,N,N′,N′*-tetramethylethylenediamine (TEMED), surfactants SDS and DTAB. were purchased from Aldrich Chemical Co. and used as received. All aqueous solutions were prepared using deionized water.

Hydrogel synthesis

P(DMAPMA-co-MBAAm) hydrogels were prepared by the free-radical crosslinking polymerization of the DMAPMA in aqueous solution at 22 °C for 24 h in the presence of predetermined concentrations of the MBAAm crosslinking agent or comonomer (see Scheme 1). APS (0.056 M) and TEMED (0.32 M) were used as the redox initiators in the gelation process. The DMAPMA (1.0 mL), APS (1.0 mL) and MBAAm (0.12 g) were dissolved in distilled water (4 mL) and the solution was purged with nitrogen gas for 10 min. After the addition of TEMED (0.5 mL), the solution was placed in poly(vinylchloride) straws of 4 mm diameters and about 10 cm long. The poly(vinylchloride) straws were sealed and immersed in a thermostated water bath at 22 °C, and the polymerization was conducted for 24 h. Upon completion of the reaction, the hydrogels were cut into specimens of approximately 10 mm in length and immersed in large excess of water to wash out any unreacted monomers and the initiator. The hydrogel samples were then dried at 50 °C under vacuum to constant weight. The ratio between mass of dried copolymer and calculated copolymer mass for 100% conversion for these samples was found in the range of 1.10–1.18. An analysis of these values shows both the presence of bound water and conversion of monomer to polymer to be close to 100%. The cross-linked *N*-substituted acrylamide hydrogels always contain about 10–20 wt% of bound water, even after several months of drying under vacuum [8].

Determination of swelling kinetics

The swelling kinetics of the P(DMAPMA-co-MBAAm) hydrogels containing different MBAAm content were measured gravimetrically. The dried samples were placed in distilled water at 22 °C and removed from water at regular time intervals. After the water on the surfaces of the hydrogels was wiped off with moistened filter paper, the weights of the hydrogels were recorded. The swelling ratio was defined as follows:

$$\mathrm{SR} = \frac{m_t - m_\mathrm{d}}{m_\mathrm{d}} \tag{1}$$

where m_d and m_t are the masses of the dry and swollen hydrogels at time t, respectively.

Determination of equilibrium swelling ratio

For the temperature-response studies, hydrogels were equilibrated in distilled water at temperatures ranging from 10 to 60 °C. The hydrogels were allowed to swell in distilled water for at least 24 h at each predetermined temperature, controlled up to ±0.1 °C in a constant-temperature water bath (Thermo Haake K10). After immersion in distilled water at a predetermined temperature, the hydrogels were removed from the water and blotted with wet filter paper for the removal of excess water on the hydrogel surface; they were then weighed. After this weight measurement, the hydrogels were re-equilibrated in distilled water at another predetermined temperature, and their swollen weight was determined. The average values of three measurements were taken for each hydrogel, and the equilibrium swelling ratio was calculated as follows:

$$\mathrm{ESR} = \frac{m_\mathrm{s} - m_\mathrm{d}}{m_\mathrm{d}} \tag{2}$$

where m_s is the mass of the swollen hydrogel, respectively.

Similarly, for the swelling measurements in aqueous surfactant solutions, the P(DMAPMA-co-MBAAm) hydrogels were immersed in vials filled with aqueous surfactant solutions (SDS or DTAB). Aqueous surfactant solutions were prepared by dilution of the 30 mM surfactant stock solutions with various volumes of water. The volume of the surfactant solutions in the vials was much larger than the hydrogel volume so that the concentration of the solution was practically unchanged. The hydrogels were equilibrated in aqueous surfactant solutions at temperatures ranging from 10 to 60 °C. The hydrogels were allowed to swell in aqueous surfactant solutions for at least 24 h at each predetermined temperature. After immersion in aqueous surfactant solutions at a predetermined temperature, the hydrogels were removed from the aqueous surfactant solutions and blotted with wet filter paper for the removal of excess water on the hydrogel surface; they were then weighed. After this weight measurement, the hydrogels were re-equilibrated in aqueous surfactant solutions at another predetermined temperature, and their swollen weight.

Conductivity measurements

The conductivity of the P(DMAPMA-co-MBAAm) hydrogel and surfactant (DTAB or SDS) systems was measured in the concentration range of 3.8–30 mM surfactant solutions at different temperatures by using a conductometer (Model WPA CM 35). The temperature was controlled with water circulation using a thermostatic circulator (Thermo Haake K10).

Results and discussion

Effect of cross-linker content on swelling kinetic

When the hydrogels was immersed in water, they readily swelled up to size depending on the degree of crosslinking. The water adsorption of P(DMAPMA-co-MBAAm) hydrogels containing different MBAAm content was determined gravimetrically as a function of time. Figure 1 shows the swelling ratio–time curves for the P(DMAPMA-co-MBAAm) hydrogels depending on the crosslinker content. The data show that the swelling rate decreased with the increase in the crosslinker content, as from 11.6 to 17.8%. The hydrogel with 11.6% MBAAm has about 6.3 swelling ratio within 96 min, or 17.2 within 420 min, whereas the hydrogel with 17.8% MBAAm has about 3.2 and 6.5, respectively, within the same time frames.

The swelling process is a complicated phenomenon and involves three successive steps [9–11]: (1) the diffusion of water molecules into the polymeric network, (2)

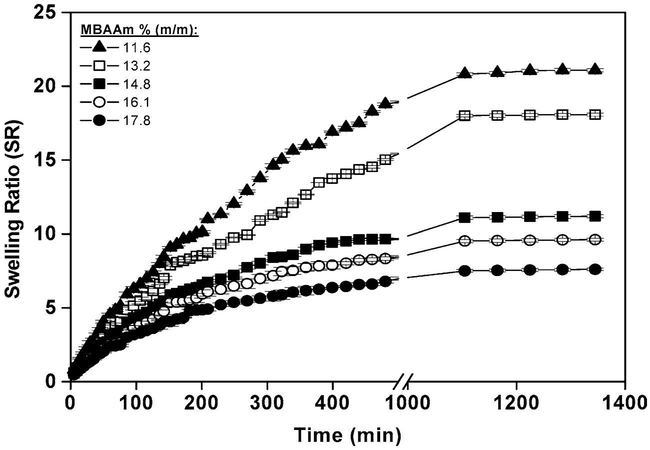


Fig. 1 Swelling kinetics of the P(DMAPMA-co-MBAAm) hydrogels containing different MBAAm content. The crosslinker contents of the hydrogels are indicated as the *insert*

the relaxation of the hydrated polymer chains, and (3) the expansion of the polymeric network into the surrounding aqueous solution. Before the swelling, there existed strong intermolecular and/or polymer–polymer interactions, such as hydrogen bonds and hydrophobic interactions, in the dried hydrogel samples, which remained in a glassy state [9]. This suggests that a glassy inner core might exist in a dried hydrogel having a higher crosslinking level because the high crosslinking may lead to strong such interactions. As a result, with increasing amount of MBAAm, the swelling ratio of the hydrogels reduced due to dense three-dimensional structure formed at the high crosslinker concentration.

To determine the nature of water diffusion into the hydrogel, the following equation was used [12]:

$$\frac{M_t}{M_\infty} = kt^n \qquad (3)$$

where M_t and M_∞ represent the amount of water absorbed by the hydrogel at time t and at equilibrium, k is a constant characteristic of the system, n is an exponent which takes into account the mode of water transport. A value of $n = 0.5$ indicates a Fickian diffusion mechanism, while a value of $0.5 \le n \le 1$ indicates that diffusion is anomalous or non-Fickian. The exponents n and k values were calculated from the slope and intercept of the plots of log M_t/M_∞ versus log t for the P(DMAPMA-co-MBAAm) hydrogels (Fig. 2). Eq. 3 is valid for the first 60% of normalized solvent absorbed [12]. From the intercept and the slope of the curves, the values of the kinetic constant, k, and diffusion exponent, n, are obtained. These results are shown in Table 1. A slight variation of diffusion exponent with MBAAm content is observed, and its value higher than 0.50, indicating diffusion of water to the interior of all the hydrogels, follows an anomalous mechanism and reveals the existence of certain coupling between molecular diffusion and tension relaxation developed during swelling of the hydrogels. The highly anomalous behavior of these hydrogels is due to the regularity of the chain and strong interchain interactions via the formation of hydrogen bonding (Fig. 3), leading to a compact structure which would accentuate the anomalous aspects of diffusion even for a molecule as small as water.

Diffusion coefficients are important penetration parameters of some chemicals to polymeric systems. Using n and k values, the diffusion coefficient (D) of water in the hydrogel could be calculated using the following equation [12]:

$$D = \left(\frac{k}{4}\right)^{1/n} \pi r^2 \qquad (4)$$

where D is the diffusion coefficient and r is the radius of the hydrogel. The D values of the hydrogels are also presented in Table 1. The diffusion coefficients D slightly

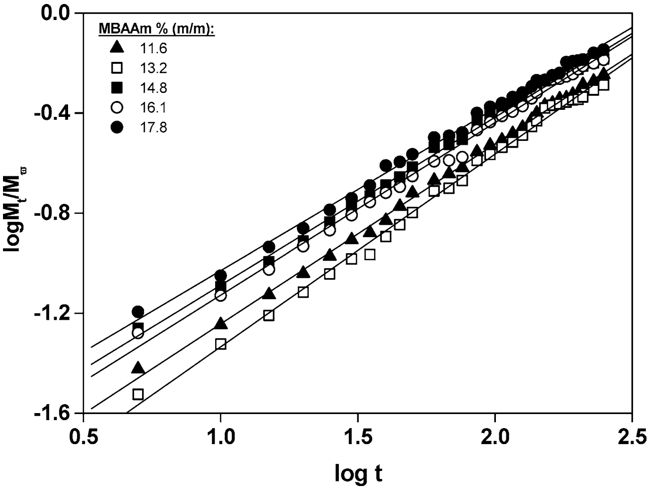


Fig. 2 The plots of log M_t/M_∞ versus log t for the P(DMAPMA-co-MBAAm) hydrogels containing different MBAAm content. The crosslinker contents of the hydrogels are indicated as the *insert*

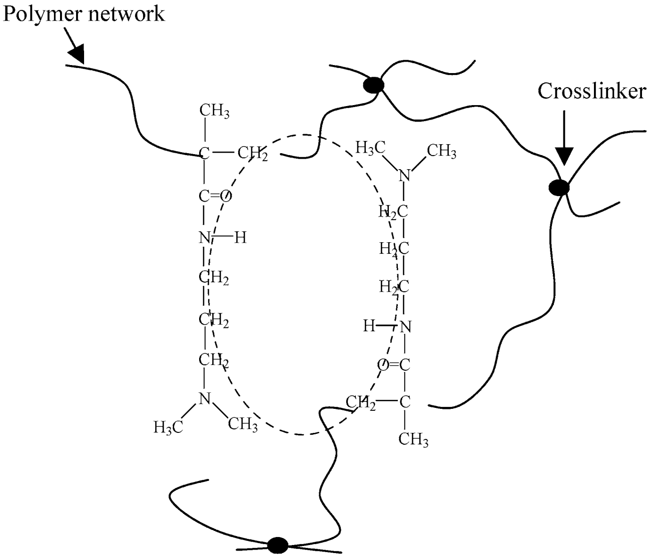


Fig. 3 Proposed hydrogen-bonding interactions in the P(DMAPMA-co-MBAAm) hydrogel

Table 1 The parameters of diffusion of water into the P(DMAPMA-co-MBAAm) hydrogels

MBAAm % (m/m)	k×100	n	D×10^7/cm^2 .s^{-1}
11.6	1.09	0.72	2.55
13.2	0.79	0.77	2.85
14.8	1.74	0.67	2.82
16.1	1.52	0.69	2.88
17.8	2.09	0.65	2.87

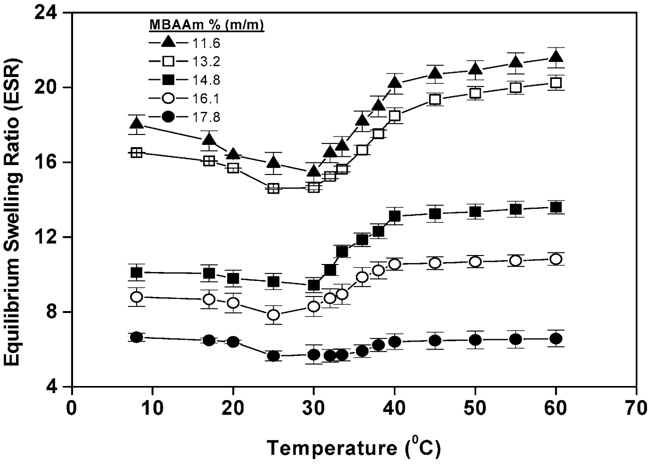


Fig. 4 Equilibrium swelling ratios of the P(DMAPMA-co-MBAAm) hydrogels in water shown as a function of temperature. The crosslinker contents of the hydrogels are indicated as the *insert*

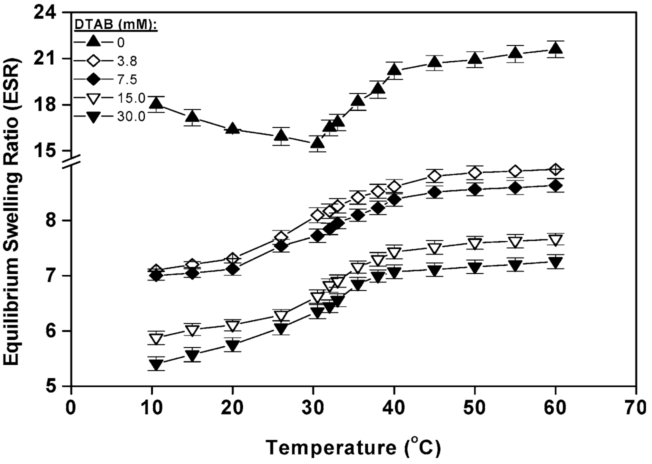


Fig. 5 Equilibrium swelling ratio of the P(DMAPMA-co-MBAAm) hydrogel with 11.1% MBAAm in DTAB solutions shown as a function of temperature. The concentrations of DTAB solutions are indicated as the *insert*

increase with an increase in MBAAm content in the hydrogel. Because, at a high crosslinking level, the pore numbers of the hydrogels with 16.1 and 17.3% MBAAm were also increased and large numbers of channels were available for the surrounding water to diffuse into, and this slightly increased the diffusion coefficients for the hydrogels with 16.1 and 17.3% MBAAm.

Effect of cross-linker content on swelling equilibrium

Figure 4 illustrates the temperature dependence of the equilibrium swelling ratio of P(DMAPMA-co-MBAAm) hydrogels with different amount of cross-linker in water when the temperature increased from 10 to 60 °C. The data show that all the P(DMAPMA-co-MBAAm) hydrogels, regardless of the amount of the MBAAm, have similar swelling behaviors as a function of temperature, and the phase-transition temperatures of these hydrogels are in the range of 30–40 °C. It is also shown that all P(DMAPMA-co-MBAAm) hydrogels exhibited a positive temperature-coefficient, which swells at higher temperature and shrinks at lower temperature. Under equilibrium swelling conditions, all P(DMAPMA-co-MBAAm) hydrogels show increasing swelling at higher temperatures, but they arewell below their phase-transition temperatures because of the aggregation of the network chains. When the external temperature was increased from 10 °C toward phase-transition temperature, the volume or water content inside P(DMAPMA-co-MBAAm) hydrogels first decreased slowly up to 30 °C and then increased drastically between 30 and 40 °C (at phase transition temperature range) and finally remained almost constant. Even though the phase-transition temperature range of the P(DMAPMA-co-MBAAm) hydrogels were virtually not affected by the amount of MBAAm within the ranges studied in this work, the data in Fig. 4 clearly show also that, the equilibrium swelling ratios of these hydrogels decreased 67% at 10 °C and 73% at 60 °C with increasing amount of MBAAm from 11.1 to 17.3%. It is believed that an increase in the level of crosslinking agent would reduce the free volume within the hydrogel network structure in which water would reside during swelling and would also led to the reduction of pore size of the corresponding hydrogels, which, in turn, reduced the water holding capacity because of the decreased pore volume.

Effects of surfactants on swelling equilibrium

The effect of each ionic surfactant on the swelling process of P(DMAPMA-co-MBAAm) hydrogel containing 11.1% MBAAm, which has the highest equilibrium swelling ratio in water among the other hydrogels prepared and studied in this work was investigated in detail. Figures 5 and 6 show the temperature dependence of the equilibrium swelling ratio of P(DMAPMA-co-MBAAm) hydrogel with 11.1% MBAAm in the presence of two different ionic surfactants with various concentrations. The overall effect observed is a reduction in the equilibrium swelling ratio of the hydrogel in surfactant solutions for the whole temperature range investigated when this was compared with in pure water. The DTAB and SDS molecules consists of a long aliphatic hydrocarbon chain and P(DMAPMA-co-MBAAm) has hydrophobic units such as $(CH_3)_2N(CH_2)_3$ and CH_2CCH_3 as pendant group and main chain, respectively. When DTAB or SDS molecules diffuse into the gel network of the P(DMAPMA-co-MBAAm), strong association should take place through the hydrophobic interaction between the hydrophobic groups of the P(DMAPMA-co-MBAAm) and long-chain alkyl groups of the DTAB or SDS, in this case, the equilibrium swelling ratio of hydrogel decreases.

In the absence of surfactants, this hydrogel underwent a discontinuous phase transition (chain collapse) at 30–40 °C temperature range with a swelling ratio change at this temperature range defined by ratio $SR_{swollen}/SR_{collapsed} \sim 1.3$. In solutions containing DTAB (Fig. 5), sharp discontinuity in the phase transition the P(DMAPMA-co-MBAAm) hydrogel was converted into a more smooth change while maintaining a break in the curves corresponding to onset of transition. However, in the SDS solutions (Fig. 6), the P(DMAPMA-co-MBAAm) hydrogel has almost the same swelling ratio irrespective of the concentration of SDS solutions and exhibited a negative temperature-coefficient with increasing of temperature from 10 to 60 °C. This behavior may be attributed to the differences in the counterions, the ionizable groups and the binding amounts of the surfactants. In the absence of P(DMAPMA-co-MBAAm) hydrogel, pH of DTAB and SDS solutions was measured as 6.1 and 7.8, respectively. Therefore pH dependent protonation of amine groups is very unlikely. On the other hand, the equilibrium swelling ratio of the hydrogels with binding surfactant molecules depend on the types and nature of water binding sites such as $-O-SO^{-}_{3}$ and $-N^{+}(CH_3)_3$ of the surfactant molecules. Their high electric fields not only polarize, immobilize, and electrostrict nearest neighbor molecules, but they also induce additional order beyond the first layer of water molecules. Ions like $-O-SO^{-}_{3}$, however, can immobilize the water molecules of only the first layer [13]. The higher attractive field that can be felt to several layers in the case of $-N^{+}(CH_3)_3$ probably causes many more layers of water to be associated to the first layer, which is in the immediate vicinity of the polymer in comparison to $-O-SO^{-}_{3}$ group, and hence more water uptake is seen in the hydrogel with the binding of DTAB containing $-N^{+}(CH_3)_3$ groups.

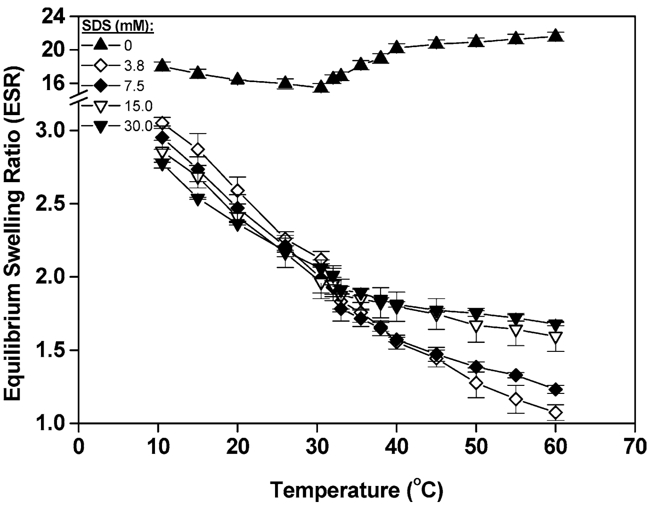


Fig. 6 Equilibrium swelling ratio of the P(DMAPMA-co-MBAAm) hydrogel with 11.1% MBAAm in SDS solutions shown as a function of temperature. The concentrations of SDS solutions are indicated as the *insert*

The binding of surfactant molecules to hydrogels has been studied by many groups [1, 14, 15]. Generally, increased binding of charged surfactants would increase the charge density of hydrogels and thus lead to enhanced swelling ratio. The effect of SDS in inducing some measure of polyelectrolyte behavior with chain expansion has been demonstrated by viscometric measurements on aqueous solutions of the uncharged polymer, poly(*N*-vinyl-2-pyrrolidone) [14]. Binding of SDS has also been observed for thermosensitive crosslinked hydrogels of modified cellulose ethers [15]. However, Shinde et al. [1] observed non-uniform swelling of poly(*N*-isopropylacrylamide) hydrogels in SDS solutions, but did not analyze surfactant binding or its relation to micellisation or aggregation. They have attributed this to the presence of heterogeneous phases as a result of uneven binding of surfactant molecules. But in this present study, our hydrogels are always transparent and swelling is uniform throughout the bulk, so there is homogeneous interaction of SDS with our hydrogels. Therefore, we are unable to attribute the decrease of the swelling ratio with increasing surfactant concentration to heterogeneous distribution of surfactant molecules in the hydrogel. However, the observed swelling ratio changes can be attributed to the micellar-like hydrophobic domains, which act as physical crosslinkers to restrain the swelling of the hydrogel networks, arise from hydrophobic side groups dangling on the hydrogel networks (Fig. 9). This is in agreement with the low swelling behavior of the P(DMAPMA-co-MBAAm) hydrogels observed in more concentrated surfactant solutions. The intermolecular aggregation force of polymer segments may play a dominant role in the formation of the micellar-like hydrophobic domains because of the disappearance of protonation effect on the basic dimethylamino group in slightly basic SDS solutions. In this case, in contrast to the DTAB solutions with slightly acidic pH, the micellar-like hydrophobic domains form only in the presence of lightly basic SDS solution.

On the other hand, in this study, the high surfactant concentrations (SDS and DTAB) were used. In this case, the equilibrium swelling ratio of the P(DMAPMA-co-MBAAm) hydrogel in the surfactant solutions was appreciably decreased comparing to the values measured in deionized water. This is due to decrement in the expansion of the hydrogel network because of repulsive forces of counter ions acting on the polymeric chain shielded by the binding ionic changes. Therefore, the

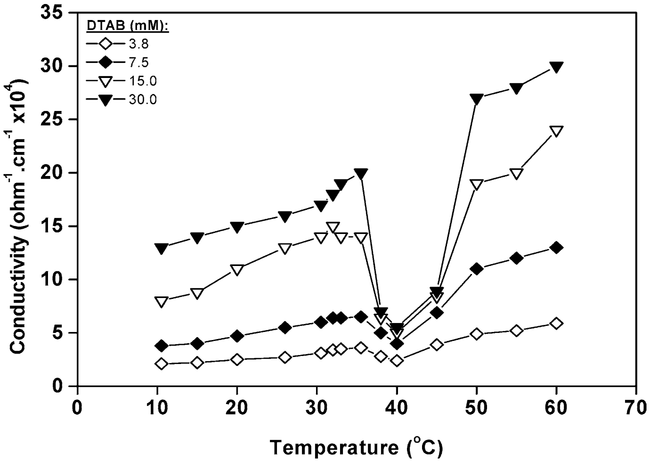


Fig. 7 Temperature dependence of conductivity of DTAB solutions in the presence of the P(DMAPMA-co-MBAAm) hydrogel with 11.1% MBAAm. The concentrations of DTAB solutions are indicated as the *insert*

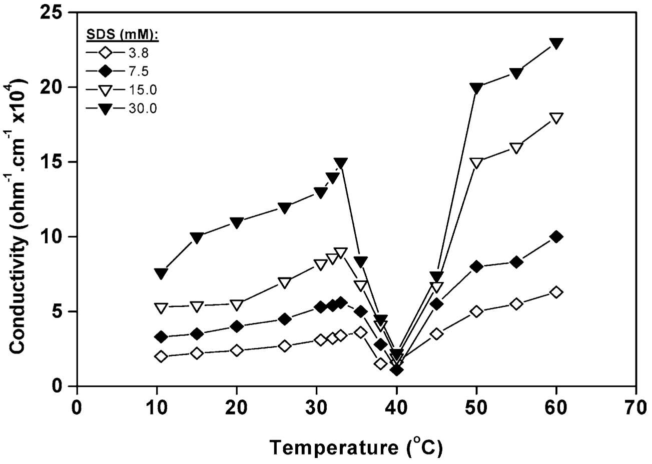


Fig. 8 Temperature dependence of conductivity of SDS solutions in presence of the P(DMAPMA-co-MBAAm) hydrogel with 11.1% MBAAm. The concentrations of SDS solutions are indicated as the *insert*

difference in the osmotic pressure between the hydrogel network and the external solution decreased with an increase in the ionic strength of the surfactant concentration, as a result of which the hydrogel exhibited the trends to shrink.

Conductivity measurements were used to determine the free DTAB and SDS amounts in their aqueous solutions in the presence of the P(DMAPMA-co-MBAAm) hydrogel. This hydrogel is expected to absorb a portion of the DTAB and SDS molecules and since the polymers within the hydrogel structure are non-ionic, the conductivity should be proportional to the amount of free ionic surfactant molecules. Figures 7 and 8 show the temperature dependence of conductivity in the presence of this hydrogel in the respective surfactant solutions. The conductivity was linearly dependent with respect to temperature variation for the temperature range investigated, except in the temperature range of 30–40 °C, where a significant sudden decrease in conductivity was observed, this did not change with the type of surfactant. This temperature range can be thus referred to as the binding temperature [16, 17]. The magnitude of the decrease in the free DTAB or SDS concentrations, indicated by the decrease in conductivity, was found to increase with the surfactant concentration. The observed effects of both the cationic surfactant DTAB and anionic surfactant SDS on the P(DMAPMA-co-MBAAm) hydrogel can thus be readily understood by assuming that the hydrogel have a partially ionic character upon binding of DTAB or SDS molecules the polymer net-

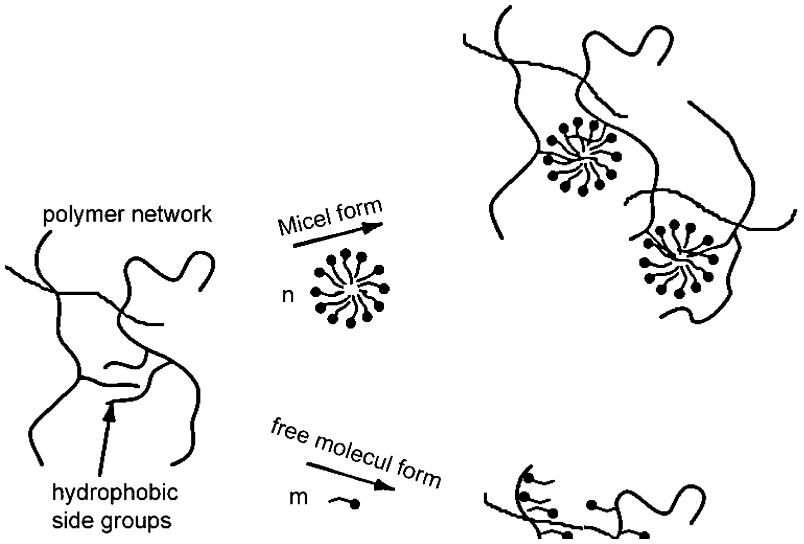


Fig. 9 Schematic representation of the adsorption via free molecular and micelle (*sphere*) forms of the surfactants onto the P(DMAPMA-co-MBAAm) hydrogel

work. It is reasonable to suppose that the DTAB or SDS binds to the hydrogel within the gel phase though hydrophobic interactions. Because, it is also evident that the P(DMAPMA-co-MBAAm) hydrogel does not contain ionizable groups in their chemical structure; hence, there should not be any electrostatic repulsion forces between the polymer chains. The strong hydrophobic interactions between DTAB or SDS molecules and the P(DMAPMA-co-MBAAm) hydrogel enables the surfactants to bind to polymer networks not only from free DTAB or SDS molecules but also from micelles of these, though the destruction or breakdown of the later [13, 14] (Fig. 9). The critical micelle concentrations (CMC) of DTAB and SDS are 15.6 and 8.3 mM at room temperature, respectively [18]. It is known that surfactant micelle formation in the presence of polymer is observed at the surfactant concentration beyond critical aggregation concentration (CAC) that below CMC. At surfactant concentrations beyond this characteristic concentration, the excess 'free' surfactant is in a dual equilibrium with micelle formation and binding to hydrogel. Both cases would result in a saturation value for amount of surfactant bound to the hydrogel.

However, after 40 °C, the conductivity of the hydrogel– surfactant systems increased again linearly with increasing temperature. This conductivity behavior must be the result of the desorption of the DTAB and SDS molecules at around the surface of hydrogel. Because, the hydrophobic interactions between the hydrogel and the surfactant molecules having a low energy would be overcome with increasing temperature and in this case, the conductivity of the hydrogel– surfactant systems increases.

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