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ORIGINAL PAPER

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# Modifying the thermosensitivity of copolymers of sodium styrene sulfonate and *N*-isopropylacrylamide with dodecyltrimethylammonium chloride

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**Abstract** The interactions between copolymers of sodium styrene sulfonate (SSS) and *N*-isopropylacrylamide (NIPAM), anionic polyelectrolytes, and dodecyltrimethylammonium chloride (DTAC), a cationic surfactant, were studied in aqueous solutions of various ionic strengths. The copolymers were found to be thermoresponsive, showing a lower critical solution temperature (LCST). The influence of the polymer composition, the surfactant concentration, and the ionic strength on the LCST was studied. The surfactant was found to interact strongly with the polymer, forming mixed polymer-surfactant micelles. The critical aggregation concentration (cac) of the polymer-surfactant system was found from fluorescence spectroscopy using pyrene as a fluorescent probe. A strong dependence of the anionic polyelectrolyte-cationic surfactant interactions on the structure of the ionic comonomer was observed.

**Keywords** Lower critical solution temperature · SSS · NIPAM · Stimuli-responsive polymers · Critical aggregation concentration

## Introduction

The interactions between polymers and surfactants, both in solution [1, 2] and at interfaces [3], are of growing interest for both theoretical and practical reasons. The practical applications of polymer-surfactant systems are numerous, including enhanced oil recovery and coatings [4, 5], cosmetics [6, 7], and drug release processes [8].

There are a number of possible polymer-surfactant systems: nonionic polymers and ionic surfactants [9], nonionic polymers and nonionic surfactants [10, 11], hydrophobically-modified polymers and nonionic [12] and ionic [13, 14, 15] surfactants, and polyelectrolytes and oppositely charged surfactants [16]. The latter case is especially interesting due to the particularly strong polymer-surfactant interaction. These polymer-surfactant interactions may result in unusual effects, including enhancing the solubilization capacity of microemulsions enormously by adding an amphiphilic block copolymer (boosting effect) [17, 18, 19]. The polymer-surfactant systems that involve temperature-responsive polymers (smart polymers) are of special interest [20], because these polymers have found many practical applications, such as smart reaction media showing anti- or hyper-Arrhenius behavior [21], or surfactant-induced drug delivery [22].

In our previous paper we reported on studies of the interactions of an anionic thermoresponsive polyelectrolyte, a copolymer based on *N*-isopropylacrylamide (NIPAM) with a small content of sodium 2-acrylamido-2-methyl-1-propanesulfonate (AMPS), with dodecyltrimethylammonium chloride (DTAC) [23]. As a continuation of these efforts, this paper describes studies of a series of copolymers of sodium styrene sulfonate (SSS) and NIPAM with similar contents of ionic comonomer. Both series of copolymers contain a sulfonic acid group, attached to the aliphatic group in AMPS or the aromatic ring in SSS, respectively. The SSS-NIPAM copolymers have already been synthesized by Katsuura et al [24]. However, these authors were interested in interactions of gels of SSS-NIPAM copolymers (with much lower content of SSS) with dodecylpyridinium chloride. Therefore, this paper is the first report on the studies of the interactions of SSS-NIPAM polymers with a surfactant in aqueous solution.

## Experimental

### Materials

The SSS-NIPAM copolymers were synthesized in a radical copolymerization reaction, as described elsewhere [25]. The molecular weight of the polymers was estimated using GPC measurements on a Waters chromatographic system equipped with an Ultrahydrogel Linear column and a refractive index detector using poly(sodium styrenesulfonate) (PSSS) standards obtained from Polysciences. DTAC (99%, Fluka) was used as received. Pyrene (99%, Aldrich) was recrystallized from methanol. Doubly distilled water was used in all measurements.

### LCST measurements

The LCST for each of the polymer solutions was measured as described previously [23]. In brief, the LCST was measured spectrophotometrically using a Hewlett-Packard 8452A spectrophotometer equipped with a Hewlett-Packard 89090A Peltier temperature control accessory. The accessory allowed programmed heating of the solution in a cuvette within the range 15–70 °C. The solution was heated at a rate of about 0.5 °C/min and stirred at the rate of 5 $s^{-1}$. LCST was determined from the changes with temperature of the solution transmittance, $T$, at $\lambda = 400$ nm.

### Fluorescence probe measurements

Steady-state fluorescence emission spectra of pyrene were measured using an SLM-Aminco spectrofluorimeter at an excitation wavelength of 320 nm. The widths of the excitation and emission slits were 8 and 2 nm, respectively. The $I_3/I_1$ ratio of the fluorescence intensities of the first and third vibronic bands was calculated using corrected spectra.

### Surface tension measurements

The surface tension was measured using a K9 Krüss tensiometer.

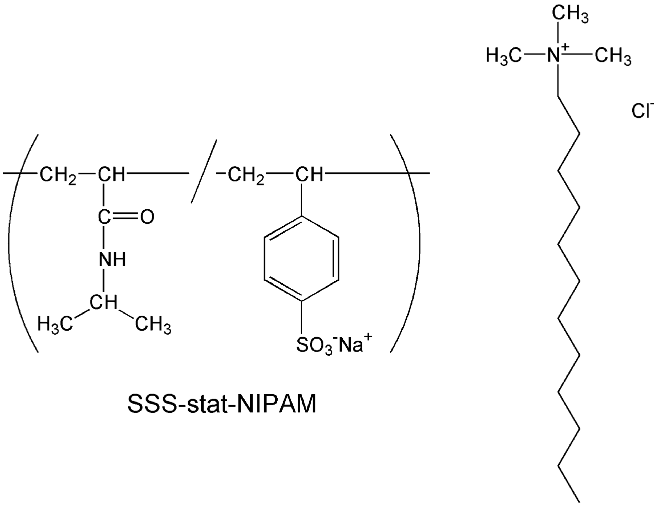
## Results and discussion

The chemical structures of the SSS and NIPAM copolymers and the DTAC surfactant used in these studies are shown in Scheme 1.

The polymers were given acronyms S*x*N where *x* is the SSS content in the polymerization reaction feed, by analogy to AMPS-NIPAM copolymers, A*x*N, where *x* is the AMPS content in the reaction feed [23]. The content of SSS in the polymers, together with their molecular weights is given in Table 1.

In order to determine the LCST of SSS-NIPAM polymers, the turbidity of their aqueous solutions was measured as a function of temperature (see Fig. 1).

The temperatures at which the turbidities of the solutions of S1 N, S2 N and S5 N start to increase are all very similar (31 °C for S1 N and S2 N, and 31.5 °C for S5 N), and similar to that of unsubstituted NIPAM [26] (32 °C). This is in strong contrast with the behavior of A*x*N polymers, for which LCST increased by over 20 °C when the AMPS content was increased by as little as from 1.1 to 5.1 mol% [27]. For both S*x*N and A*x*N polymers the LCST becomes higher than the upper temperature limit accessible to the Peltier device used in the measurements (about 70 °C) when the content of the ionic comonomer is



**Scheme 1** Structures of the copolymers and surfactant studied

**Table 1** Basic characteristics of the polymers

| Polymer | SSS content (mol%) | $M_w$ |
|---|---|---|
| S1 N | 0.94 | $1.1\times10^5$ |
| S2 N | 1.62 | $1.4\times10^5$ |
| S5 N | 3.42 | $7.4\times10^4$ |
| S20 N | 15.40 | $1.1\times10^5$ |

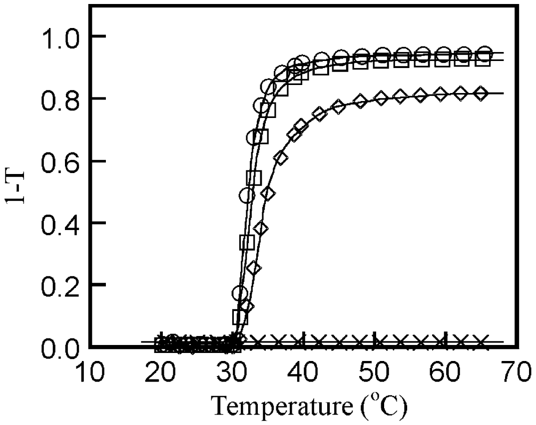


**Fig. 1** Turbidity of the copolymer solutions (expressed as $1-T$ where $T$ is the solution transmittance at 400 nm) versus temperature for S1N (circles), S2N (squares), S5N (diamonds), and S20N (×) at a polymer concentration of 1 g/l

high enough, 15.4 mol% and 9.6 mol%, respectively. The different temperature responses of the S *x*N and A *x*N polymers can be attributed to the different characters of the groups to which the sulfonic groups are attached in the ionic comonomers. Due to the aromatic character of the group attached to the polymeric chain, SSS is more hydrophobic than AMPS. Therefore, the presence of charged sulfonic groups, which increase the LCST of the NIPAM copolymers, is compensated for by the presence of the hydrophobic phenyl groups, which decrease the LCST of the NIPAM copolymers. The influence of SSS on the polymer properties is discussed below in more detail.

Large differences in the responses of S *x*N polymers to temperature appear, however, in the presence of DTAC. The changes in the turbidities of the polymer solutions with temperature in the presence of different concentrations of DTAC are shown in Fig. 2.

It was found that, at low concentrations of DTAC, the rise in the turbidities of the polymer solutions are steep, while at higher DTAC concentrations the increases in turbidity are slower. However, the S20 N solution behaves in a different way to the other polymers. For the S20 N polymer, the increase in turbidity with temperature only appears when the DTAC concentration reaches 0.002 M. In the DTAC concentration range 0.005–0.01 M, the S20 N polymer-surfactant aggregates precipitate at temperatures below 15 °C, which is the lowest temperature limit accessible with the Peltier instrument, so LCST determination was not possible. The precipitate resolubilizes again when [DTAC] exceeds 0.02 M. At DTAC concentrations equal or higher than 0.04 M, the LCST of S20 N becomes higher then the maximum temperature reached by the instrument (about 70 °C).

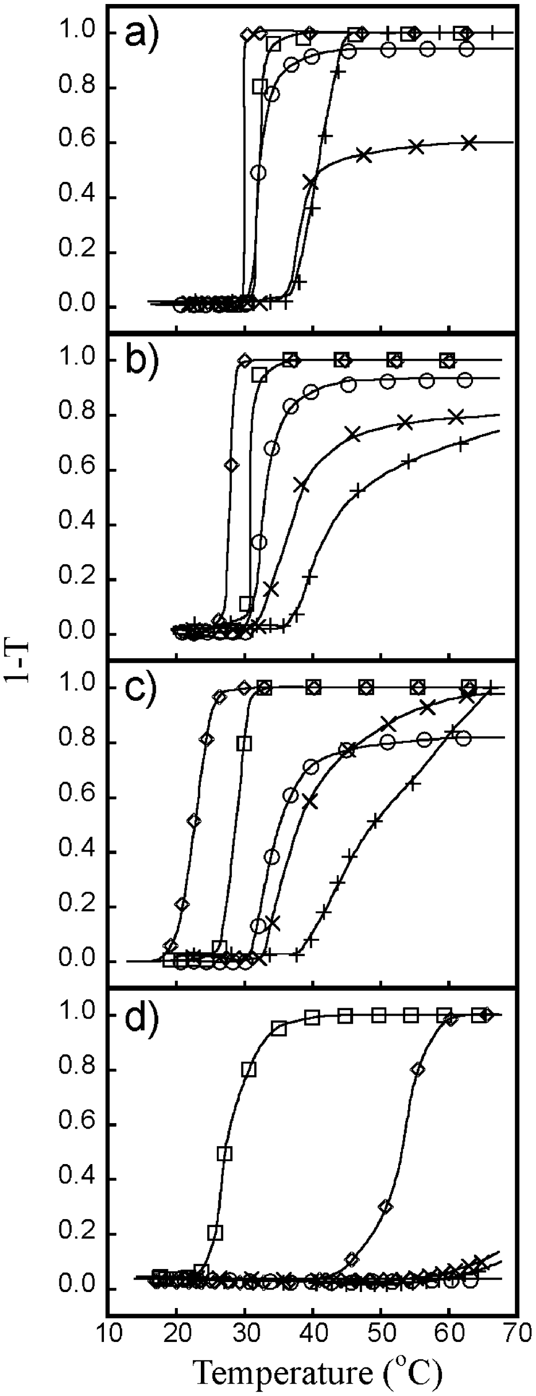


**Fig. 2a–d** Turbidity of the copolymer solutions versus temperature for: **a** S1 N at [DTAC] equal to 0 (circles), 0.001 (squares), 0.01 (diamonds), 0.02 (×), and 0.1 M (+); **b** S2 N at [DTAC] equal to 0 (circles), 0.002 (squares), 0.005 (diamonds), 0.02 (×), and 0.04 M (+); **c**S5 N at [DTAC] equal to 0 (circles), 0.002 (squares), 0.005 (diamonds), 0.02 (×), and 0.1 M (+); **d** S20 N at [DTAC] equal to 0.001 (circles), 0.002 (squares), 0.02 (diamonds), 0.04 (×), and 0.08 M (+). The concentration of the polymer solution was 1 g/L, measurements were carried out in the absence of KCl

Figure 3 shows the dependence of LCST on the concentration of surfactant for S*x*N polymers. The shapes of the plots for S1 N, S2 N, and S5 N polymers are the same: up to the $[\mathrm{DTAC}] < 10^{-3}$ M there is no change in the LCST, at $10^{-3}$ M $< [\mathrm{DTAC}] < 6\times10^{-3}$ M the LCST decreases reaching a minimum, and at $[\mathrm{DTAC}] > 6\times10^{-3}$ M increases again reaching a plateau at $> 4\times10^{-2}$ M. The higher the content of SSS in the polymer, the more pronounced the changes in LCST. Therefore, for S1 N the LCST changes over a range of 10 °C (from 29 to 39 °C), while for S5 N the LCST changes over a range of 22 °C (from 20 to 42 °C). This behavior parallels that shown by A*x*N polymers [23]. The important difference between both polymer-surfactant systems, however, is that in the absence of the salt it is possible to increase the LCST to values above those characteristic of the polymer solution in the absence of the surfactant for S*x*N polymers, while for the polymers containing from 1.1 to 5.1 mol% of AMPS (and probably also for the polymer containing 9.6 mol% of AMPS) the LCST at high DTAC concentrations reached similar values to those in the absence of DTAC [27]. The behavior of S20 N polymer is a special case where the range of LCST values available by varying [DTAC] ranges from $< 15$°C to $> 70$ °C. For S20 N polymer, LCST could only be measured at two DTAC concentrations, $2.0\times10^{-3}$ M and $4.0\times10^{-2}$ M below and above the precipitation region, respectively. For DTAC concentrations of $1.0\times10^{-3}$ M (and lower than that) the LCST is above the upper experimental limit of about 70 °C. For DTAC concentrations in the range of $3.0\times10^{-3}$ to $2.0\times10^{-2}$ M the polymer already precipitates at a temperature below the lower experimental limit of 15 °C. Similarly, at DTAC concentrations higher than $8.0\times10^{-2}$ M, the LCST exceeds the upper experimental limit. Taking into account the above facts, the approximate dependence of the LCST on DTAC concentration was plotted in Fig. 3 (dashed line).

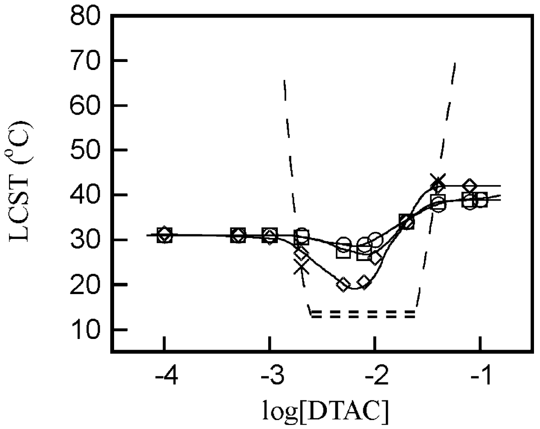


**Fig. 3** The dependence of the LCST of S1 N (circles), S2 N (squares), S5 N (diamonds), S20 N and (×) on [DTAC] in the absence of KCl. The dashed line shows the probable dependence of LCST on [DTAC] for S20 N. The double dashed line shows the precipitation region. The polymer concentration was 1 g/L

As the interactions between S*x*N copolymers and DTAC are expected to contain two components – electrostatic and hydrophobic interactions – it was interesting to determine the effect of changes in electrostatic interactions by observing the influence of simple electrolyte (KCl) on the value of the LCST. It was found that the LCST values of the S*x*N copolymers can be influenced considerably by changes in the ionic strength of the solutions (addition of KCl). The influence of the ionic strength on the turbidity of the polymer solutions is shown in Fig. 4.

Qualitatively, at low concentrations of additive (up to $10^{-3}$ M) and at low temperatures (up to 30 °C) KCl and DTAC produce similar effects, except in the case of S20 N, which does not show a phase transition for concentrations of KCl lower than 1 M. It was observed, however, that for S1 N and S2 N the increase in temperature at high ionic strengths (above 0.5 M KCl) results in a gradual decrease of the solution turbidity. This was also observed for A*x*N polymers, although this effect was less significant [27]. Figure 5 shows the dependence of the LCST value for the S *x*N polymers on the concentration of KCl.

As shown in Fig. 5, increasing the ionic strength decreases the LCST values of the polymers. This may be expected, because the KCl present in the solution leads to the shielding of the repulsive interactions between sulfonic groups on the polymer chain. This results in a shift in the hydrophobic-hydrophilic balance towards hydrophobic interactions. For A*x*N copolymers, a decrease in LCST with increasing ionic strength was also observed [27]. Interestingly, however, the LCST for S*x*N polymers (with different SSS content) changes with ionic strength in the same way (so the plot of the dependence of LCST on concentration of KCl can be shown by one common line for all of the three polymers studied), while for A*x*N polymers the increase in the ionic strength resulted in changes in LCST which were greater for the polymers with higher ionic comonomer content [27]. Another difference is that, by increasing the ionic strength, the LCST can be lowered further (to below 20 °C) for S*x*N polymers than for A*x*N polymers. Again, this can be attributed to the higher hydrophobicity of SSS compared to AMPS.

We have also studied how simultaneously applying both of the factors that influence the LCST (surfactant and increased ionic strength) changes the LCST of a chosen S*x*N copolymer. The results for S5 N are shown in Fig. 6.

In the presence of the salt, the LCST values of the polymers were found to be influenced to different degrees by the addition of surfactant than when the KCl was absent. Increasing the ionic strength decreases the

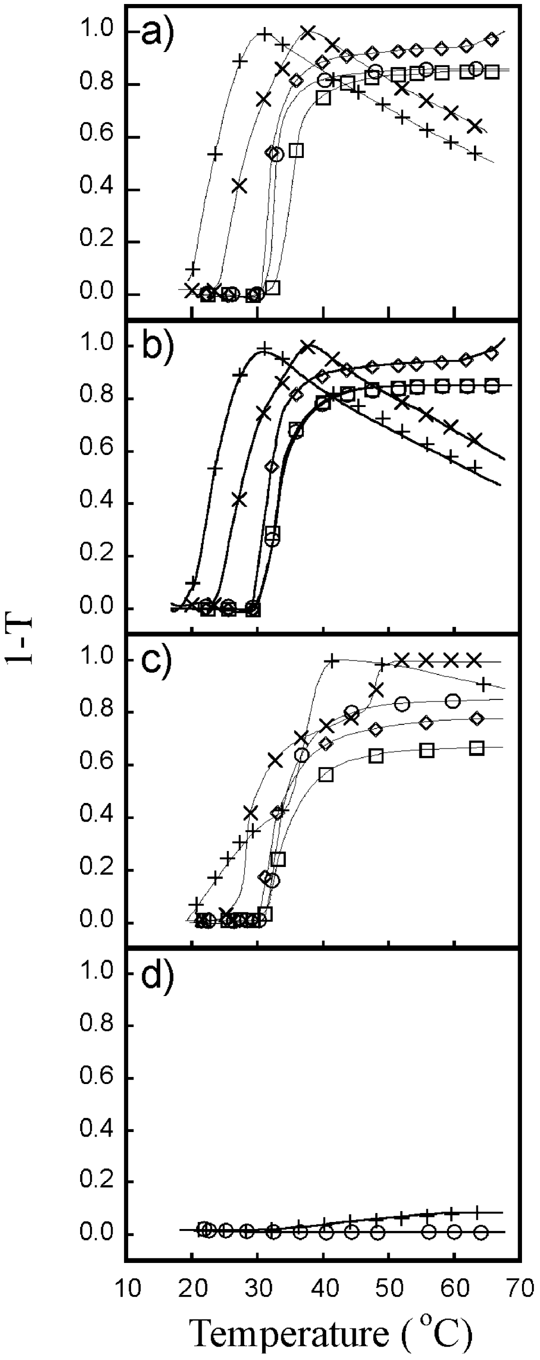


**Fig. 4a–d** Turbidity of the copolymer solutions in the absence of the surfactant for: **a** S1 N; **b** S2 N; **c** S5 N; **d** S20 N at [KCl] equal to 0.001 (circles), 0.01 (squares), 0.1 (diamonds), 0.5 (×), and 1 M (+). The polymer concentration was 1 g/L

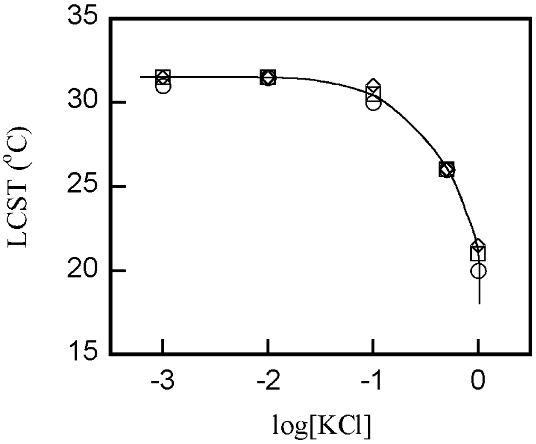


**Fig. 5** The dependence of the LCST of S1 N (circles), S2 N (squares), and S5 N (diamonds) on [KCl] in the absence of DTAC. The polymer concentration was 1 g/L

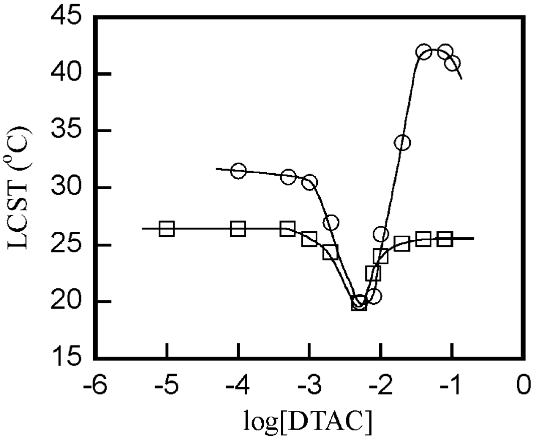


**Fig. 6** The dependence of the LCST of S5 N on [DTAC] in the absence of (circles) and in the presence of (squares) 0.4 M KCl

LCST over the whole range of DTAC concentrations studied, except for the region around the minimum LCST value, which does not change. The greatest decrease in the LCST values caused by the addition of 0.4 M KCl occurs when the surfactant concentration reaches the value close to its critical micellization concentration (cmc) ($9.0\times10^{-3}$ M at 17 °C, as found in a separate measurement).

## Fluorescence probe studies

In order to find the values of the critical aggregation concentration (cac) for these polymer-surfactant systems, we used pyrene, which is a fluorescence probe frequently referred to in studies of polymer-surfactant interactions [28, 29]. Pyrene is a probe used to estimate the hydrophobicity of the environment in which it resides. The ratio of the third and the first vibrational bands, $I_3/I_1$, in the fluorescence emission spectrum of pyrene is a measure of the polarity of the surroundings of the probe molecule. Namely, $I_3/I_1$ is low in polar media and high in hydrophobic environments. Therefore, the formation of surfactant or polymer-surfactant mixed micelles is accompanied by the solubilization of pyrene molecules inside hydrophobic micellar cores, and consequently by a rise in the $I_3/I_1$ ratio. In the case of polymer-surfactant systems, $I_3/I_1$ can be used to determine the cac. Figure 7 shows the plots of $I_3/I_1$ ratios for pyrene solubilized in S$x$N solutions as a function of DTAC concentration.

For the S1 N, S2 N, and S5 N polymers, the plots have similar shape: at some [DTAC] there is a steep rise in $I_3/I_1$ which then reaches a plateau and decreases. The rise in $I_3/I_1$ begins at a [DTAC] of $9.0\times10^{-4}$ M for S1 N and S2 N, and at $3.0\times10^{-5}$ M for S5 N. These values can be considered as the respective cac's for the DTAC-S$x$N polymer systems and are negatively correlated with the SSS content in the polymers. They are also much lower than the cmc of DTAC at 17 °C ($9.0\times10^{-3}$ M). Interestingly, the $I_3/I_1$ ratios of all four polymers studied reach the same highest value of 0.74, which may be considered as characteristic of mixed polymer-surfactant micelles. This value corresponds to the hydrophobicity of ethyl acetate [30]. The onset of the $I_3/I_1$ rise is observed at a concentration of DTAC which is lower than cmc for [DTAC] (equal to $9.0\times10^{-3}$ M at that temperature). At a DTAC concentration equal to the cmc, the $I_3/I_1$ ratios begin to decrease, dropping to a value of $\sim$0.71. This can be explained by assuming that the decrease in the $I_3/I_1$ ratios is caused by the formation of the pure surfactant micelles and the redistribution of pyrene molecules between mixed surfactant-polymer micelles and pure surfactant micelles. This was confirmed by the measurement of the $I_3/I_1$ ratio for pyrene solubilized in DTAC micelles in the absence of the polymer. Therefore, it can be concluded that the hydrophobicity of the surfactant micelles is lower than that of mixed surfactant-polymer micelles. The plot of $I_3/I_1$ ratio versus [DTAC] for S20 N is slightly different to those for the other polymers; the increase in the $I_3/I_1$ ratio begins at an extremely low [DTAC], of about $7.0\times10^{-6}$ M, which is the cac value for this system. Such a low cac originates from particularly strong polymer-surfactant interactions in the DTAC-S20 N system, which results from the high SSS content in S20 N relative to other S$x$N polymers. Another characteristic feature of the plot for S20 N is the lack of the decrease in $I_3/I_1$ ratio in the high [DTAC] regime. This also may be explained by the strong polymer-surfactant interaction. Namely, the portion of the surfactant bound to the polymer forming mixed polymer-surfactant micelles is so large that the concentration of free surfactant is lower than the cmc even if the total concentration of the surfactant in the system is higher than the cmc.

The conclusions drawn from the plots of $I_3/I_1$ ratio versus [DTAC] were confirmed by the measurements of the surface tension of the polymers versus [DTAC] (Fig. 8).

As expected, the formation of mixed polymer-surfactant micelles or pure surfactant micelles decreases the surface tension. The slight increase in the surface tension above the cmc may result from the possible presence of impurities in the surfactant.

A comparison of the behavior of DTAC + S$x$N and DTAC + A $x$N systems leads to the following interesting observations. It was pointed out in the literature that the interactions between polyelectrolytes and surfactants of the opposite charge are strongly influenced by the hydrophobicity of the polyelectrolyte chain [31, 32, 33]. This may explain the very different strengths of the interactions of S$x$N and A$x$N polymers with DTAC, because in the former the hydrophobicity of the ionic comonomer is much greater. As suggested, the strength of polymer-surfactant interactions can be measured by

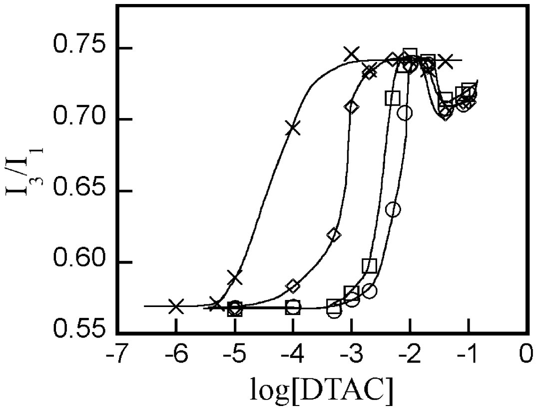


**Fig. 7** The dependence of the $I_3/I_1$ ratio on [DTAC] for S1 N (circles), S2 N (squares), S5 N (diamonds), and S20 N (×) in the absence of the salt at 17 °C

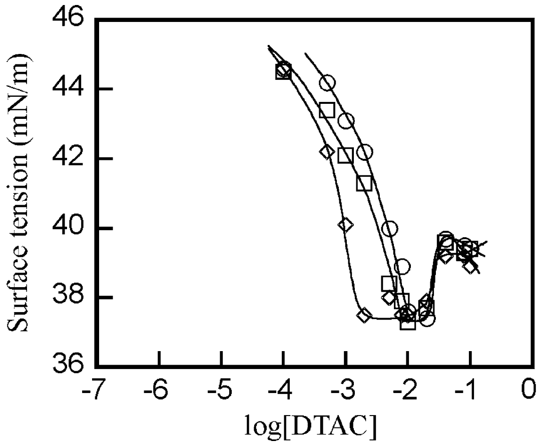


**Fig. 8** The dependence of the surface tension on [DTAC] for S1 N (circles), S2 N (squares), and S5 N (diamonds) at 17 °C. The polymer concentration was 1 g/l

**Table 2** The values of $\mathrm{cmc_{DTAC}}$/cac for A*x*N and S*x*N polymers

| Polymer | Ionic monomer content (mol%)[a] | $\mathrm{cmc_{DTAC}}$/cac[b] |
|---|---|---|
| S1 N | 0.94 | 10 |
| S2 N | 1.62 | 10 |
| S5 N | 3.42 | 300 |
| S20 N | 15.40 | 1285 |
| A2 N | 1.1 | 2 |
| A4 N | 2.6 | 6 |
| A6 N | 5.1 | 6 |
| A10 N | 9.6 | 15 |

[a] Values for A*x*N polymers are taken from [24];

[b] $\mathrm{cmc_{DTAC}}$/cac ratios for S*x*N and A*x*N polymers were calculated for 17 °C

the ratio of $\mathrm{cmc_{DTAC}}$/cac [34]. The values of the ratios for S*x*N and A*x*N polymers, together with the respective content of the ionic monomer, are given in Table 2.

One can notice that the ratio is considerably higher for S*x*N polymers than for A*x*N polymers. As suggested by Kogej and Škerjanc, specific interactions between the surfactant and the polymer backbone can occur for poly(sodium styrene sulfonate) (PSSS) and alkylpyridinium surfactants [35] and alkyltrimethylammonium bromides [34]. These authors postulated the inclusion of the hydrophobic styrene sulfonate group into the alkylpyridinium surfactant micelles, leading to the formation of stable aggregates. Similar behavior can be expected in the case of the S*x*N polymers. This may explain the relatively higher hydrophobicity of the interior of mixed S*x*N-DTAC micelles than the typical DTAC surfactant micelles.

## Conclusions

The LCST values of thermoresponsive copolymers of SSS and NIPAM in pure water were found to be independent of the SSS content and were close to those characteristic of the NIPAM homopolymer. It was observed, however, that the LCST values of the polymers can be changed significantly upon addition of DTAC and by changing the ionic strength of the solution. Through the addition of DTAC it was possible to either increase or decrease the LCST, depending on the DTAC concentration applied, while increasing the ionic strength resulted in a monotonous decrease in the LCST. The polymers were found to interact strongly with DTAC, forming mixed polymer-surfactant micelles as found from fluorescence probe studies. The interactions between S *x*N and DTAC are strong not only because of the opposite charges of the surfactant and the polymer, but also because of the considerable hydrophobicity of SSS.

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