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A comparative behavior of photophysical properties of Pluronic F127 and Triton X-100 with conventional zwitterionic and anionic surfactants

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Abstract

The mixed micellar properties of a triblock copolymer, Pluronic F127, $(EO)_{97}(PO)_{69}(EO)_{97}$ and a nonionic surfactant, Triton X-100 (TX-100), in aqueous solution with conventional zwitterionic (alkyldimethylammoniopropane sulfonates) and anionic (sodium dodecylsulfate, SDS, and dodecylbenzene sulfonate, SDBS) surfactants were investigated with the help of fluorescence measurements at 25 °C. From the variation of I_1/I_3 intensity ratio of pyrene fluorescence, the critical micelle concentration (cmc) and other related micellar parameters were determined. Apart from this, photophysical properties of pyrene due to its quenching as well as excimer formation in the interior of the mixed micelles were also studied and explained on the basis of stability of the mixed micelles. It was observed that the mixed micelle formation between zwitterionic/anionic +F127 occurred due to favorable interactions, the magnitude of which increased with the increase in hydrophobicity of zwitterionic component. On the other hand, close to ideal or unfavorable mixing was observed in the mixtures of zwitterionic + TX-100, which was attributed to the incompatibility due to the bulkiness of the hydrophilic groups of zwitterionic and TX-100 components.

Keywords: Zwitterionic surfactants; Anionic surfactants; Triblock copolymer; Mixed micelles; Fluorescence measurements

1. Introduction

Mixtures of surfactants, often in combination with polymers, are used widely in industrial and domestic formulations. A surfactant may help in surface tension lowering, wetting, active ingredient deposition and control of stability or rheology, but its properties generally change in the complex mixture. Understanding of the interactions between the components of a surfactant mixture is, therefore, important to get their best performance. Thus, a significant research has been focused on the field of polymer–surfactant interactions in recent decades and several reviews exist [1–3]. The case of polymer–surfactant interaction pairs in which the polymer is a water-soluble poly(ethylene oxide)–poly(proplylene oxide)–poly(ethylene oxide) (PEO–PPO–PEO) copolymer which is commonly called triblock polymers (TBP) and commercially known as Pluronic or Poloxamers, is of special interest. The structure of TBP micelle is well described by a core-corona model in which a spherical core composed of PPO is surrounded by a corona composed of strongly hydrated PEO [4–9]. The variation of their molecular characteristics (PPO/PEO ratio, molecular weight) allows the best use of these macromolecules in number of applications, such as emulsifiers, gelling agents in food processing, in pharmaceutical, cosmetic and agricultural formulations [10–16]. The TBPs also act as nonionic surfactants since amphiphilic character of block copolymer leads to a self-assembled behavior resembling to that of classical nonionic surfactants [5,17–22]. Since most of the industrial applications involve the usage of nonionic surfactants, therefore, their fundamental behavior is equally important to understand. Moreover, the physicochemical properties of the aqueous solution consist of two or more surfactants often present a very different character in comparison to those formed by single surfactant.

Using ^{13}C NMR and fluorescence quenching measurements, Almgren et al. [23] have investigated the interaction of L64 $(EO_{13}PO_{30}EO_{13})$ and F68 $(EO_{78}PO_{30}EO_{78})$ with sodium dodecyl sulfate (SDS) in the dilute regime. The experiments demonstrated that TBPs formed mixed micelle with SDS at concentration well below the cmc of SDS and that addition of SDS reduces the size of TBP micelles markedly. The shape of the mixed

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micelles was assumed to be spherical, with coiled PO blocks solubilized in the interior of a SDS micelle. Li et al. [24] studied the interactions between cationic surfactant, tetradecyltrimethylammonium bromide (TTAB) and F127, and showed that small amount of TTAB decreases the cmc of F127. Marinov et al. [25] studied the state of L64 in aqueous solution at concentration below cmc and temperature below critical micelle temperature (cmt) by fluorescence probing, ultrasonic absorption and conductivity. They observed that on addition of SDS, L64 aggregates bind with pyrene and SDS.

Similarly, zwitterionic surfactants are also of particular interest because they are commonly utilized in many commercial formulations, particularly in personal care products (including soaps, shampoos and lotions), where they are typically found to be less irritating to skin than many ionic surfactants [26,27]. They have hydrophilic polar heads, which carry both positive and negative charges. This leads to head group hydrophilicity an intermediate between that of ionic and nonionic surfactants [28]. They have thus many applications when combined with ionic and nonionic surfactants in cosmetic and pharmaceutical industries. Despite this, not many studies exist for zwitterionic + nonionic surfactant mixtures to understand their fundamental mixing behavior [29–31]. Thus, the novelty of such mixtures and their wide implications, have prompted us to study the mixed micelle behavior of a series of zwitterionic surfactants with a triblock polymer (F127) and a conventional nonionic surfactant, TX-100. This is to discriminate the mixing behavior of F127 from that TX-100 with a same series of zwitterionic surfactants so that the mixtures with better mixing behavior can be defined. The results have been compared with similar mixtures of anionic surfactants (SDS and SDBS) with F127 and TX-100 in order to evaluate the head group effect. The microenvironment of mixed micelles of various mixtures has been studied with the help of pyrene fluorescence by utilizing both quenching and excimer formation.

2. Experimental

2.1. Materials

The triblock copolymers, Pluronic F127, $(EO)_{97}(PO)_{69}(EO)_{97}$ (MW = 12,600), was obtained from Aldrich and polyoxyethylene (10) iso-octyl phenyl ether, which is also known as Triton X-100 (TX-100), was obtained from Nacalai Tesque Inc., Kyoto, Japan. Both surfactants were used without purification. 3-(*N*,*N*-dimethyldodecylammonio)propane sulfonate (DPS), 3-(*N*,*N*-dimethyltetradecylammonio)propane sulfonate (TPS) and 3-(*N*,*N*-dimethylhexadecylammonio)propane sulfonate (HPS), all more than 99% pure from Flüka. Sodium dodecyl sulfate (SDS) and sodium dodecylbenzene sulfonate (SDBS) were obtained from Sigma. Pyrene was obtained from Aldrich and quencher, hexadecylpyridinium chloride (HPyCl), was procured from Lancaster Synthesis, UK. All zwitterionic and anionic surfactants, pyrene and HPyCl were used after repeated purifications (at least three times) from ethanol. Water was purified by deionisation followed by double distillation. The solutions were prepared by mass with an accuracy of 0.01 mg. All measurements have been performed at 25 °C after giving overnight time for the stabilization.

2.2. Sample preparation

The samples of binary mixtures of zwitterionic/anionic surfactant + F127/TX-100 were prepared over the whole mixing range by dissolving appropriate amounts of respective components in pure water or aqueous pyrene. The desired mole fraction range was covered by mixing precalculated amounts of stock solutions of binary components. The mole fraction of each component is expressed only on surfactant basis. The mixed micelle formation was carried out in the form of titration by adding successive amounts of stock solutions in aqueous pyrene reference solution.

2.3. Fluorescence measurements

The pyrene fluorescence of the present mixtures has been measured by using Hitachi 2500 fluorescence spectrophotometer at 25 °C. It is well known that the ratio of first to third vibronic peaks (I_1/I_3) of pyrene emission spectrum indicates the polarity of the medium in which it is dissolved. The I_1/I_3 values for all the binary mixtures covering whole mole fraction range were measured in order to evaluate the micropolarity. Fluorescence emission spectra of pyrene in these solutions were recorded employing an excitation wavelength of 335 nm, and the detection wavelengths were $I_1 = 373$ nm and $I_3 = 384$ nm. For the micelle formation measurements, the concentration of pyrene was fixed at 10^{-6} mol dm^{-3} and no pyrene excimer formation was observed in these experiments. Therefore, the excimer formation, on the other hand, for these surfactant solutions was determined by keeping [pyrene] = 10^{-4} mol dm^{-3} and pyrene emission intensity, $I_E = 470$ nm. The errors in cmc values were estimated to be less than 5%.

3. Results and discussion

3.1. Critical micelle concentration and micellar parameters

Fig. 1 shows the variation in the intensity ratio of I_1/I_3 of the pyrene spectrum with respect to surfactant concentration. This represents the micelle formation process of pure TX-100 and its equimolar mixture with SDS. In all cases, the cmc was evaluated from break in I_1/I_3 curve as demonstrated in Fig. 1 [32]. The cmc values thus obtained for the pure components have been compared with literature values in Table 1, and found to be in good agreement with each other. The cmc values over the whole mole fraction range for all binary mixtures have been given in Supplementary Table along with their uncertainties. The ideality in the mixed micelle formation of binary mixtures can be evaluated by using Clint equation (1) [33] based upon the pseudophase thermodynamical model. It is represented in the form

$$\frac{1}{\text{cmc}^*} = \frac{\alpha_1}{\text{cmc}_1} + \frac{1-\alpha_1}{\text{cmc}_2} \qquad (1)$$

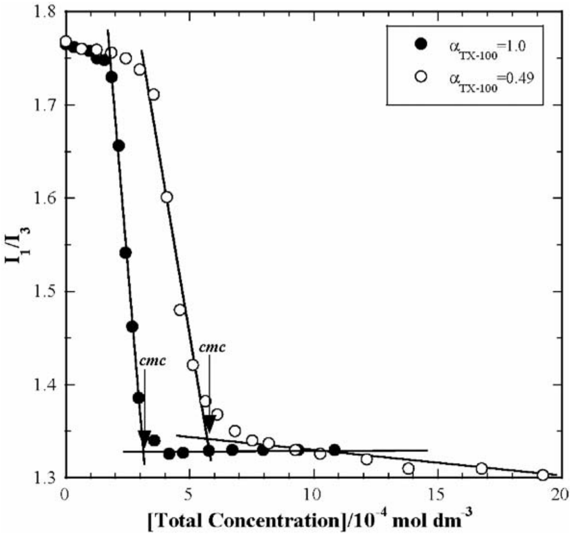

Fig. 1. Variation of the pyrene intensity I_1/I_3 ratio with total concentration for pure TX-100 and $\alpha_{\text{TX-100}} = 0.49$ in SDS + TX-100 binary mixture at 25 °C.

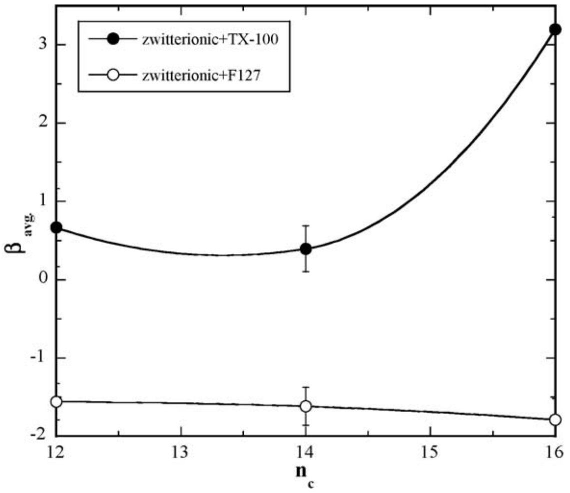

Fig. 2. Plot of β_{avg} vs. n_{c} for DPS/TPS/HPS + TX-100/F127 surfactants binary mixtures.

where cmc* is the theoretical ideal state mixed cmc, α_1 the mole fraction of surfactant 1 (DPS/TPS/HPS/SDS/SDBS) in total mixed solute, and cmc_1 and cmc_2 are the critical micellar concentrations of pure components 1 and 2, respectively. The cmc* values thus calculated have also been mentioned in Supplementary Table. The cmc values for DPS/TPS/HPS + TX-100 mixtures deviate positively from the cmc* values, while these values for all other mixtures (i.e. DPS/TPS/HPS + F127 and SDS/SDBS + TX-100/F127) deviate negatively from the cmc*. Also a qualitative comparison suggests that the magnitude of the positive and negative deviations from the ideality increases with an increase in the length of hydrophobic chain of zwitterionic component. A positive or negative deviation in cmc values from the corresponding ideality can be explained in terms of unfavorable and favorable mixing, respectively, among the unlike monomers of the binary mixtures. This means that the mixtures of all surfactants with F127 and that of anionic surfactants with TX-100 undergo micellization due to increasing attractive interactions with increasing hydrophobicity. A quantitative estimate has been made on the basis of regular solution approximation [34] based upon pseudophase thermodynamical model, from which one can calculate the regular solution interaction parameter, β, by using the following equations.

$$\frac{X_1^2 \ln(\text{cmc}\alpha_1/\text{cmc}_1 X_1)}{(1-X_1)^2 \ln(\text{cmc}(1-\alpha_1)/\text{cmc}_2(1-X_1))} = 1 \tag{2}$$

$$\beta = \frac{\ln(\text{cmc}\alpha_1/\text{cmc}_1 X_1)}{(1-X_1)^2} \tag{3}$$

where X_1 is the micelle mole fraction of the first component.

The average β values (β_{avg}) thus obtained have been plotted against the number of carbon atoms (n_{c}) of the zwitterionic component in Fig. 2. One can see that the β_{avg} values are positive for zwitterionic + TX-100 and negative for zwitterionic + F127 binary mixtures. The magnitude of β_{avg} seems to increase with the increase in n_{c} of zwitterionic component in both series of mixtures. The positive and negative β_{avg} values indicate that the mixed micelle formation takes place due to antagonistic (repulsive) and synergistic (attractive) interactions between the unlike components, respectively. On the other hand, the β_{avg} are negative for SDS/SDBS + TX-100 ($\beta_{\text{avg}} = -1.34$, -1.67) and SDS/SDBS + F127 ($\beta_{\text{avg}} = -6.31$, -5.72) mixtures with significant higher values obtained in the latter case.

These results suggest that the mixed micellization between the components of zwitterionic + F127 and SDS/SDBS + TX-100/F127 mixtures takes place due to favorable mixing, while it is close to ideal or due to the unfavorable mixing in the case of zwitterionic + TX-100 mixtures. It means that zwitterionic surfactants interact differently with TX-100 from that of F127. Recently, da Silvia et al. [35] have reported from the calorimetry measurements that SDS interacts strongly with F127 than cetyltrimethylammonium chloride. No clear reason has been observed for this discrimination but the removal of water from the Stern layer of SDS micelles and reduction in the polar head group repulsions upon incorporation of F127 is reported to be the main factor [36]. Similar arguments can be extended for the favorable mixed micellization between zwitterionic and F127 mixtures. Apart from this, F127 is a polymeric surfactant and is expected to interact with zwitterionic

Table 1
Values of critical micelle concentrations (cmc/10^{-4} M) of different surfactants at 25 °C

Surfactants	cmc	Literature values
DPS	36.0	37 [53]
TPS	4.2	–
HPS	0.70	–
SDS	90.0	83 [54], 85 [55], 88 [56], 80 [57]
SDBS	30.0	–
F127	4.5	4.7 [51]
TX-100	3.0	2.5 [58], 3.3 [59]

micelles by wrapping around them and leading to a traditional kind of polymer–surfactant complex. This kind of association is expected to reduce the polar head group repulsions and would enhance the hydrophobic interactions in return.

However, similar polymer–surfactant complex is not expected in the case of zwitterionic + TX-100 mixtures. Due to the presence of electrical neutrality in the head group of present zwitterionic surfactants, they are not expected to interact electrostatically with TX-100. Previous studies have reported almost ideal mixing in the case of similar mixtures, i.e. C_{12}maltoside + DPS [29] and C_{12}BMG + $C_{12}E_8$ [37]. However, increase in the positive β_{avg} value with increasing hydrophobicity of zwitterionic + TX-100 mixtures demonstrates the presence of steric hindrances. It seems that bulkiness of zwitterionic head group and that of poly(oxyethylene) phenyl group of TX-100 might be responsible for steric hindrances which become significant with increasing hydrophobicity of the zwitterionic component.

3.2. Quenching process

The above results can further be explained on the basis of quenching of pyrene by a suitable quencher (Q), such as HPyCl under steady state conditions. It is being considered that the fluorescence lifetime of pyrene is longer than the residence time of the quencher in a micelle. A suitable [pyrene]/[mixed micelle] and [Q]/[mixed micelle] ratios ensure the Poisson distribution. The fluorescence intensity of the first vibronic band of pyrene decreases with the increase in [Q] without appearance of any new band (not shown). A Stern–Volmer relationship can be used to explain the collision quenching under the steady state conditions [38–42].

$$\frac{I_0}{I} = 1 + K_{SV}[Q] \qquad (4)$$

where I_0 and I are the fluorescence intensities without and with quencher, respectively, and K_{SV} is the collisional quenching constant, called the Stern–Volmer constant. A linear variation of I_0/I versus [Q] (Fig. 3) will give the value of K_{SV}. These values have been plotted in Fig. 4 for zwitterionic + TX-100/F127 mixtures. Fig. 4(i) shows that these values vary almost linearly and lie much close to the ideal behavior (dotted lines) for DPS + TX-100/F127 mixtures. In the case of both TPS + TX-100/F127 as well as HPS + TX-100/F127, K_{SV} shows a significant nonlinear variation (Fig. 4(ii) and (iii), respectively) with large negative deviations from the ideality. A lower value of K_{SV} than intended ideal state suggests that the probability of finding excited state pyrene (Py*) with Q (HPyCl) is much reduced. The quenching efficiency is related to the probability of finding both Py* and HPy$^+$ species in a confined environment [42]. The solubilization of pyrene in the micellar phase mostly restricts to the palisade layer [43–45]. Hence, if an effective nonpolar hydrophobic environment is available in the mixed micelle, it would facilitate the solubilization of both Py* and HPy$^+$, and bring them in close vicinity for an effective quenching. The positive β_{avg} values of zwitterionic + TX-100 mixtures (Fig. 2) suggest that the mixed micellar environment of these mixtures is less hydrophobic and might not be suitable for an effective quenching. This could be the reason why little deviations are observed for these mixtures from an ideal behavior (Fig. 4). On the contrary, the zwitterionic + F127 mixtures with negative β_{avg} values should have shown higher K_{SV} than the ideal state due to its favorable mixed micellization. But a significant lower K_{SV} value further points to a quite weak quenching efficiency. It suggests that despite the presence of strong hydrophobic environment of these mixed micelles, HPy$^+$ ion seems unable to approach Py*. The reason can be related to the polymeric nature of F127, which mostly exists in large micellar domains of polydisperse nature [9,46,47]. As the amount of a surfactant increases, these domains are converted into mixed micelles with relatively much smaller size. At higher surfactant concentration, the size of mixed micelle even falls below the actual micellar size of a pure surfactant [9]. These observations are quite clear in the present mixtures where whole mixing range exhibits three mixing regimes, i.e. surfactant-rich, TBP-rich and intermediate [9]. One can see that such deviations are prominent in surfactant-rich and close to intermediate regimes (Fig. 4). It demonstrates thus the expected small mixed micelles in these regions (and especially in the intermediate regime) are occupied with relatively much smaller number of Py* and HPy$^+$, that significantly reduces the overall quenching efficiency of these mixtures.

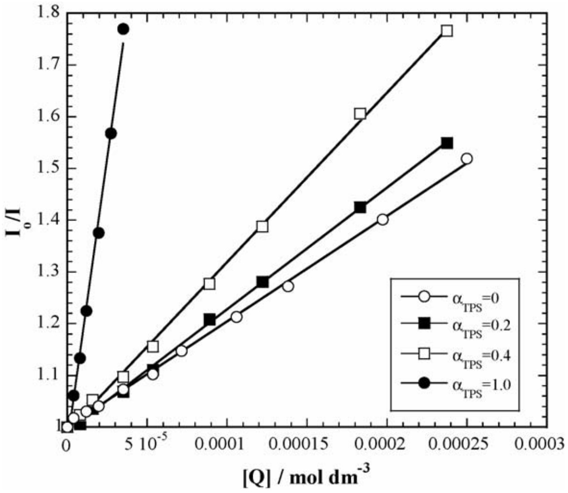

Fig. 3. Plot of I_0/I vs. [Q] for pure TPS, F127 and $\alpha_{TPS} = 0.2, 0.4$ in TPS + F127 binary mixture at 25 °C keeping total surfactant concentration for both pure and mixed components at least 10 times the respective cmc values.

3.3. Excimer formation

The steady state emission spectrum of pyrene shows five vibronic bands in 350–420 nm region (Fig. 5(i)), while a broad structureless band with a maximum at 470 nm represents the excimer formation. This occurs mainly due to the interactions involve between two pyrene species. As the amount of solubilized pyrene increases in a hydrophobic environment, such as of micelle, the intensity of the monomer bands decreases while that of excimer (I_E) increases. Fig. 5(ii) shows the variation of I_E/I_1 with respect to an increase in the [total surfactant] for various mixtures of DPS + TX-100. The I_E/I_1 represents a parameter,

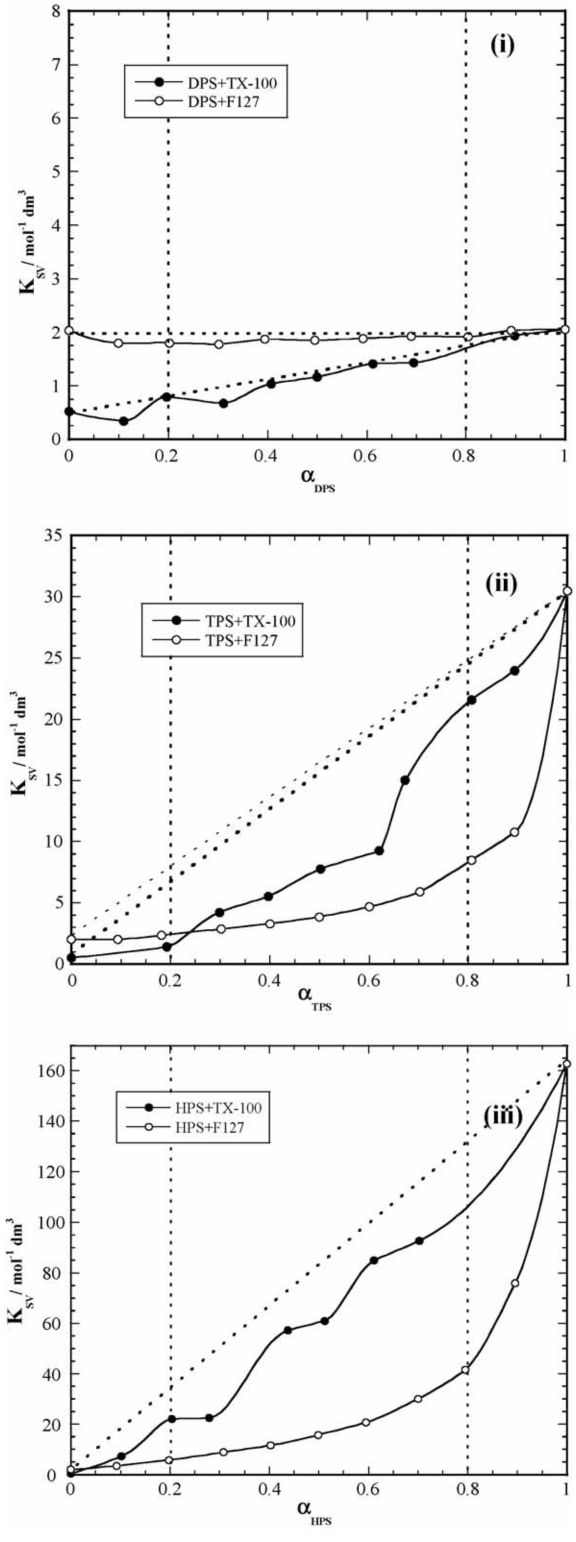

Fig. 4. Plot of K_{SV} vs. $\alpha_{DPS/TPS/HPS}$ (i) DPS + TX-100/F127; (ii) TPS + TX-100/F127; (iii) HPS + TX-100/F127 binary mixtures.

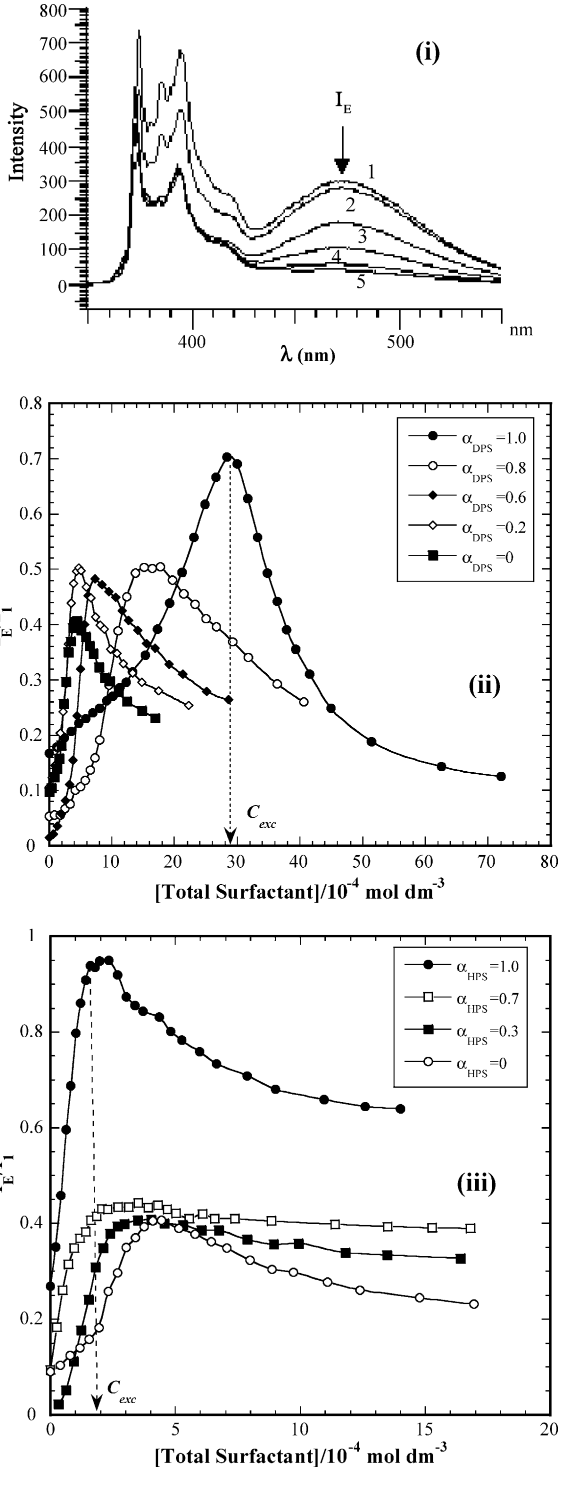

Fig. 5. (i) Plot of excimer formation of DPS in DPS + TX-100 mixtures: 1, [DPS] = 180 × 10^{-4} M; 2, [DPS] = 135 × 10^{-4} M; 3, [DPS] = 103 × 10^{-4} M; 4, [DPS] = 81 × 10^{-4} M; 5, [DPS] = 58.5 × 10^{-4} M. (ii) Plot of intensity ratio of I_E/I_1 vs. [total surfactant]/10^{-4} mol dm^{-3} for DPS + TX-100 binary mixtures. (iii) Plot of intensity ratio of I_E/I_1 vs. [total surfactant]/10^{-4} mol dm^{-3} for HPS + TX-100 binary mixtures.

which measures the amount of excimer formation at a particular [surfactant] [36,48,49]. As the concentration of surfactant increases, it produces only monomers in the bulk solution and hence pyrene is uniformly distributed through out the bulk phase. This gives weak excimer intensity. As soon as the micelles start forming, more and more pyrene gets solubilized in the interior of micelles that entrap large number of pyrene molecules. Thus, a greater probability of finding pyrene in a confined environment leads to a large increase in the excimer formation. At this

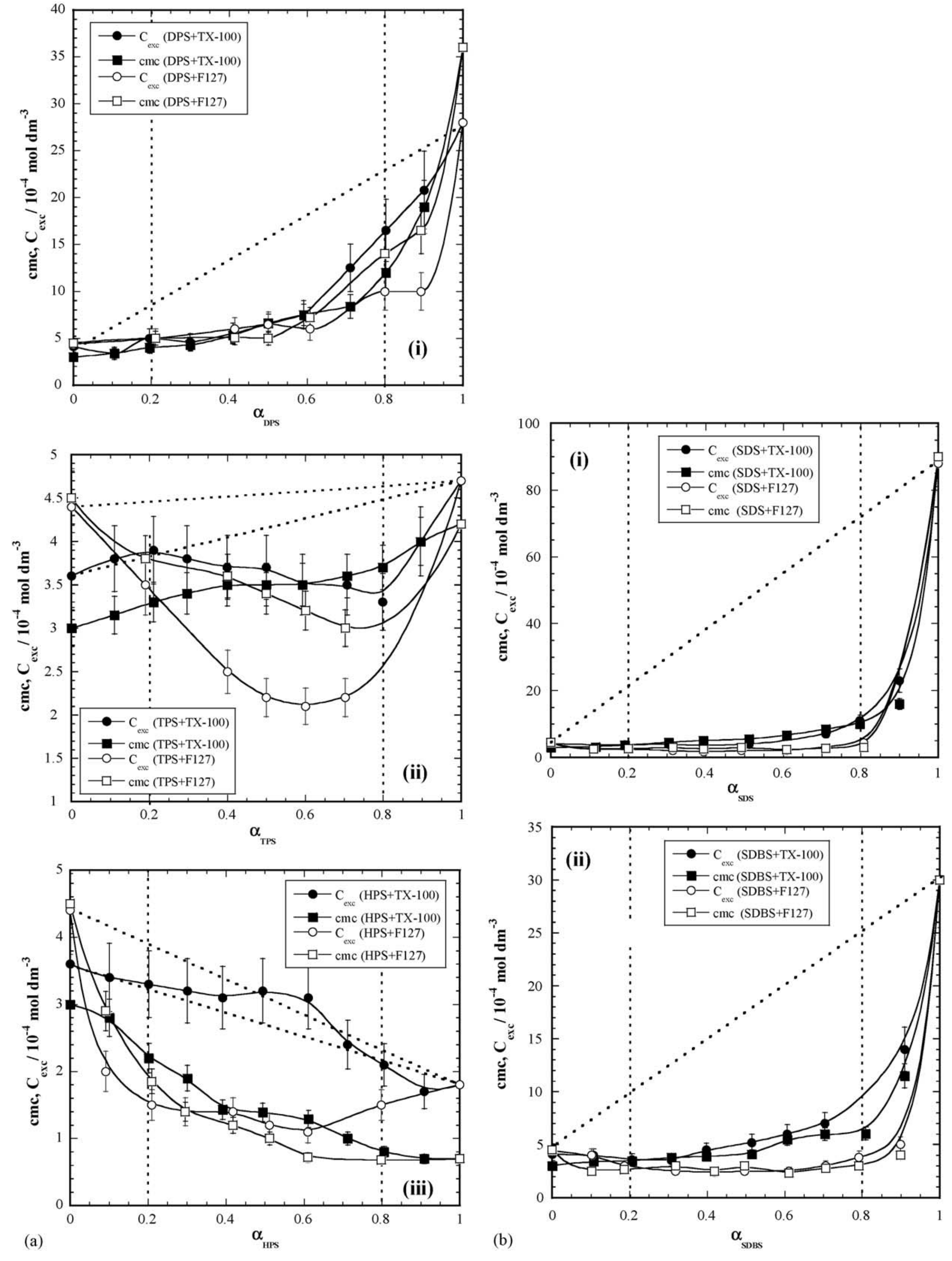

Fig. 6. (a) Plot of cmc, C_{exc} vs. $\alpha_{\text{DPS/TPS/HPS}}$ (i) DPS + TX-100/F127, (ii) TPS + TX-100/F127 and (iii) HPS + TX-100/F127 binary mixtures. (b) Plot of cmc, C_{exc} vs. $\alpha_{\text{DPS/TPS/HPS}}$ (i) SDS + TX-100/F127 and (ii) SDBS + TX-100/F127 binary mixtures.

point, I_E/I_1 passes through a maximum (where maximum solubilization of pyrene is achieved due to the micelle formation) and then decreases with the further increase in [surfactant], since less number of pyrene monomers are now available in large number of micelles.

It is interesting to note (Fig. 5(ii)) that the concentration at which the maximum in I_E/I_1 curve occurs in the case of pure component, shifts towards the lower values in its mixtures. This behavior can be observed more clearly by plotting the surfactant concentration at maximum excimer formation (C_{exc}) versus the mole fraction (Fig. 6). This figure also shows a good comparison among the respective C_{exc} and cmc values for most of the mixtures, however large uncertainties in determining the C_{exc} values in some cases (e.g. see Fig. 6(iii)) demonstrate relatively poor agreement. One can see that DPS/TPS + TX-100/F127 mixtures (Fig. 6a(i) and (ii)) show significant negative deviations from ideality and which are more prominent in the mixtures of F127. On the other hand, C_{exc} values for HPS + TX-100 close to equimolar mixing show some positive deviations from ideality, while the values for HPS + F127 remain significantly lower than the ideal state (Fig. 6a(iii)). In the case of SDS/SDBS + TX-100/F127 mixtures (Fig. 6b(i) and (ii)), C_{exc} values always show large negative deviations and which are again greater for the mixtures of F127. A stronger hydrophobic environment as in the mixed micelles of these mixtures will facilitate the pyrene solubilization and hence will promote the excimer formation. Therefore, excimer formation at lower [total surfactant] can be related to the favorable mixed micellization. This observation is fully compatible with the favorable mixing derived previously (Fig. 2) for these mixtures. The latter effects even become more significant as the hydrophobicity of the zwitterionic component increases from DPS to HPS. Similar explanation can be extended for the mixing behavior of SDS/SDBS + TX-100/F127 mixtures (Fig. 6b). The results of zwitterionic + TX-100 (Fig. 6a) further need some explanation as far as their dependence on the hydrophobicity of the zwitterionic component is concerned. Although, most of the points of C_{exc} curve lie close to the ideal mixing, some high values than the ideal state for HPS + TX-100 mixture indicate the formation of mixed micelles even at higher concentrations than the ideal mixing particularly close to the equimolar mixing range. Such a delay in the mixed micelle formation fully supports the antagonistic behavior derived from the variation of positive β_{avg} value for this mixture (Fig. 2).

All above results conclude that the present zwitterionic as well as anionic surfactants have significant favorable interactions with F127 in comparison to TX-100 despite the fact that both TX-100 and F127 belong to a common category of nonionic surfactants. However, there is still a tremendous difference between the natures of TX-100 and F127. The latter is a polymeric surfactant and at a concentration higher than its cmc, it also behaves like a neutral water-soluble polymer. The surfactant micelles are expected to adsorb on the backbone of such TBP and giving rise to typical surfactant–polymer aggregates [36,50–52]. However, at high [TBP], these TBPs possess large hydrophobic domains, which act as an ideal harbor for surfactants. This polymeric property is very much absent in the case of a typical nonionic surfactant like TX-100. Apart from this, all mixed micellar properties of present F127 mixtures with SDS/SDBS also show three prominent regions (see Fig. 6b), the one is F127 rich-region where a micellar property does not show any significant variation, the other surfactant rich-region which shows a drastic change in a property, and an intermediate between these two [9]. Thus, a favorable solubilization of present surfactants in the polymeric micelles of F127 could be considered as the driving force for the favorable mixing.

4. Conclusions

The following conclusions can be made from this study:

1. The evaluation of various micellar parameters have suggested that the present zwitterionic + F127 and anionic + F127/TX-100 mixtures undergo mixed micellization due to favorable interactions between the unlike components, while close to ideal or unfavorable mixing has been observed for zwitterionic + TX-100 mixtures.
2. These results have been fully supported by the photophysical properties of pyrene by determining the quenching efficiency and excimer formation. The former is significantly reduced in the mixtures of F127 and has been attributed to the solubilization of pyrene in large hydrophobic domains, which protect its quenching by HPy^+ ions. This is further supported by the maximum excimer formation at a particular surfactant concentration, which significantly decreases in the mixtures of F127 due to the favorable mixed micellization.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jphotochem.2006.03.008.

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