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Interactions of gemini cationic surfactants with anionic azo dyes and their inhibited effects on dyeability of cotton fabric

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Abstract

The interactions of two anionic azo dyes, Methyl Orange (MO) and Congo Red (CR), with a series of gemini cationic surfactants and dodecyl trimethyl ammonium bromide (DTAB) in aqueous solution have been investigated by means of UV–Vis spectroscopy. It was observed that the aggregation of surfactant and dye takes place at surfactant concentrations far below the critical micelle concentration of the individual surfactants. Aggregations with anionic dyes were reflected by hypsochromic shifts with a decrease in the intensity of absorption band. Further addition of surfactant results in an absorption spectrum of the dye characteristic in the presence of cationic micelles. The results also show bathochromic shifts for MO followed by sharp increase in intensity of the absorption bands at λ_{max} after the CMC points of these surfactants. Such a behavior is observed for CR solutions in higher surfactant concentrations. The inhibiting effect of cationic surfactants on dyeability of cotton fabric with CR has also been studied at three different temperatures (30, 50, 90–92 °C). Results show large differences between DTAB and gemini cationic surfactants.

Keywords: Gemini surfactants; Dye interaction; Dyeing inhibition; Methyl Orange; Congo Red

1. Introduction

The various unit operations of textile industry offer numerous opportunities for advantageous use of surface-active agents because they show interesting interfacial and bulk properties. As a consequence, a larger number of such products are used in textile processing than in any other industry [1].

Surfactants are mainly used as wetting, dispersing and levelling agents for improving dyeing process by increasing solubility, stabilizing the dispersed state and promoting uniform distribution of the dye in the textile [2].

Levelling agents act mainly by reducing the dyeing rate, increasing the rate of migration of the dye within the textile, and improving the compatibility of dyes. They can be divided into products with an affinity for dyes, and products with an affinity for fibers. According to the structures of dye and substrate, surfactants used as levelling agents operate by different mechanisms depending on the ionic type of the dye. Products with an affinity for dyes form loosely bound addition compounds with the dyes whose stability is concentration dependent and usually decreases with increasing temperature. Levelling agents with an affinity for fibers are absorbed onto the fibers in competition with the dye. The competitive reaction reduces the absorption rate of the dye and promotes migration [3].

Although, the interaction between dyes and surfactants has been studied in many papers, the studies in this area are still important and interesting for improving the dyeing process from theoretical, technological, ecological and economical points of view. The investigations into the behavior of different dyes in surfactant aqueous solutions can give useful information for understanding the thermodynamics and kinetics of the

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dyeing process and the finishing of textile material. UV–Vis spectroscopy, conductometry and using surfactant selective electrodes are among the most widely used measurement methods for studying this subject [4–16].

The spectral changes of a dye observed in the presence of various amounts of surfactants are consistent with sequential equilibria involving surfactant monomers, micelles, dye aggregates, premicellar dye–surfactant complex and dye incorporated into micelle [17].

The investigation of cationic surfactant–anionic dyes has shown that the importance of long-range electrical forces is basically to bring the dye anion and the surfactant cation close enough to enable the action of short-range noncoulombic attractive van der Waals forces and hydrophobic interactions. The importance of hydrophobic interactions is supported by the fact that the addition of ethanol to water reduces dye–surfactant ion pair formation. So, the long-range electrical forces as well as short-range attractive forces are responsible for the dye–surfactant ion pair formation [9–12,18].

The aggregation of oppositely-charged dyes with surfactants is strongly dependent on noncoulombic interactions. So, the hydrophobicity increase of the surfactant or the dye, increases the binding energy [8]. It has been reported that the type of head group of surfactants has no large influence on the aggregation process [5,6].

The choice of a particular surfactant for a particular purpose depends on its ability to interact with fibers and/or other components in the system.

Changes in the molecular structure and type of surfactant to improve upon their properties have attracted the attention of chemists. This has led to the preparation of new generation of surfactants such as geminis [19].

Gemini or dimeric surfactants are composed of two monomeric surfactant molecules chemically bonded together by a spacer. They have two hydrophilic and two hydrophobic groups in their molecules. The two terminal hydrocarbon tails can be short or long; the two polar head groups can be cationic, anionic or nonionic; the spacer can be short or long, flexible or rigid, polar or non-polar [20].

The advantages of gemini surfactants in comparison with corresponding conventional ones are higher surface activity, much lower values of the concentration C_{20}, lower critical micelle concentration (CMC), lower Krafft temperature and useful viscoelastic properties such as effective thickening.

In terms of concentration, they are about three orders of magnitude more efficient at reducing the surface tension of water and more than two orders of magnitude more efficient in interfacial performances than conventional surfactants. The greater efficiency and effectiveness of geminis over comparable conventional surfactants make them more cost-effective as well as environmentally desirable [21].

In this study a series of gemini cationic surfactants have been synthesized and the interactions of them with two anionic azo dyes, Methyl Orange (MO) and Congo Red (CR) in aqueous solution have been investigated by means of UV–Vis spectroscopy.

2. Experimental

2.1. Material

Decyl bromide, dodecyl bromide, *N*,*N*,*N*′,*N*′-tetramethylethylenediamine, dodecyl trimethyl ammonium bromide (DTAB), Methyl Orange (MO), Congo Red (CR), dry acetone, diethyl ether, *N*,*N*-dimethyldodecylamine, absolute ethanol were obtained from Merck, Acros and Lab-Scan companies.

N,*N*′-didodecyl-*N*,*N*,*N*′,*N*′-tetramethyl-*N*,*N*′-butanediyl-di-ammonium dibromide (14-4-14) was received from Menger's research group and was used without further purification.

The structures of the azoic dyes (MO, CR) and conventional surfactant (DTAB) are shown in Fig. 1.

2.2. Synthesis

The surfactants *N*,*N*′-didecyl-*N*,*N*,*N*′,*N*′-tetramethyl-*N*,*N*′-ethanediyl-di-ammonium dibromide (10-2-10), *N*,*N*′-didodecyl-*N*,*N*,*N*′,*N*′-tetramethyl-*N*,*N*′-ethanediyl-di-ammonium

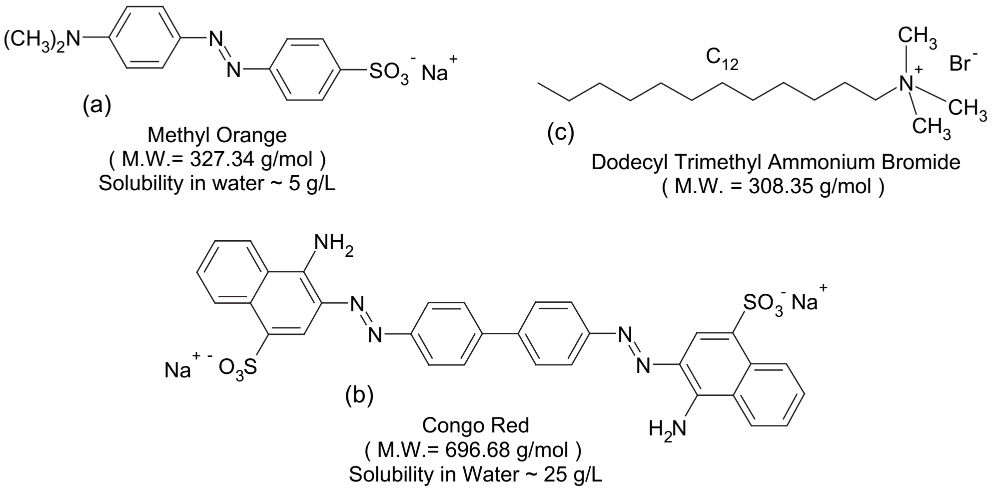


Fig. 1. Chemical structures of (a) Methyl Orange, (b) Congo Red, (c) DTAB.

dibromide (12-2-12), *N,N'*-didodecyl-*N,N,N',N'*-tetramethyl-*N,N'*-butanediyl-di-ammonium dibromide (12-4-12), *N,N'*-didodecyl-*N,N,N',N'*-tetramethyl-*N,N'*-hexanediyl-di-ammonium dibromide (12-6-12) were synthesized by using two different procedures as shown in Schemes 1 and 2. The preferred solvent for both methods is dry acetone [22] or absolute ethanol [23]. However, other solvents such as nitroethane [24] and acetonitrile [25] have also been used. Acetone is an optimal medium for the diquaternization step, as in most cases the dialkylated product precipitated from solution while the monoalkylated compound (the major side product) remained dissolved [22].

2.2.1. Method 1

For synthesis of gemini cationic surfactants type 10-2-10 and 12-2-12, a mixed solution of the corresponding alkyl bromide (5–10% excess) and *N,N,N',N'*-tetramethylethylenediamine in dry acetone was refluxed for 24 h. After cooling, a white solid was filtered which was recrystallized from ethanol and diethyl ether.

Spectral characteristics for 10-2-10: ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.89 (t, 6H), 1.24–1.36 (m, 28H), 1.81 (m, 4H), 3.49 (s, 12H), 3.69 (t, 4H), 4.73 (s, 4H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.03, 22.59, 23.00, 26.23, 29.22, 29.24, 29.43, 31.79, 51.00, 56.73, 65.93.

Spectral characteristics for 12-2-12: ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.8 (t, 6H), 1.17–1.3 (m, 36H), 1.76 (m, 4H), 3.44 (s, 12H), 3.63 (t, 4H), 4.69 (s, 4H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 13.89, 22.45, 22.86, 26.11, 29.13, 29.33, 29.36, 29.43, 29.45, 31.68, 50.78, 56.57, 65.76.

2.2.2. Method 2

For synthesis of gemini cationic surfactants type 12-4-12, 12-6-12, *N,N*-dimethyldodecylamine (5–10% excess) was added to dry acetone in a clean, dry flask. The corresponding alkyl dibromide was added dropwise to the stirring mixture, which was brought to reflux for 24 h. Upon cooling, a white solid precipitated which was filtered and washed with acetone. The solid was recrystallized from ethanol and diethyl ether.

Spectral characteristics for 12-4-12: ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.80 (t, 6H), 1.18–1.28 (m, 36H), 1.69 (m, 4H), 2.03 (m, 4H), 3.25 (s, 12H), 3.37 (t, 4H), 3.81 (s, 4H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 13.90, 19.72, 22.47, 22.72, 26.19, 29.06, 29.12, 29.28, 29.31, 29.41, 31.69, 50.77, 63.33, 64.99.

Spectral characteristics for 12-6-12: ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.82 (t, 6H), 1.19–1.29 (m, 36H), 1.51 (m, 4H), 1.66 (m, 4H), 1.93 (m, 4H), 3.33 (s, 12H), 3.45 (t, 4H), 3.65 (s, 4H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 13.94, 21.61, 22.50, 22.75, 24.48, 26.19, 29.12, 29.15, 29.26, 29.32, 29.42, 31.72, 50.88, 64.02, 64.58.

3. Instruments and measurements

3.1. Conductometry

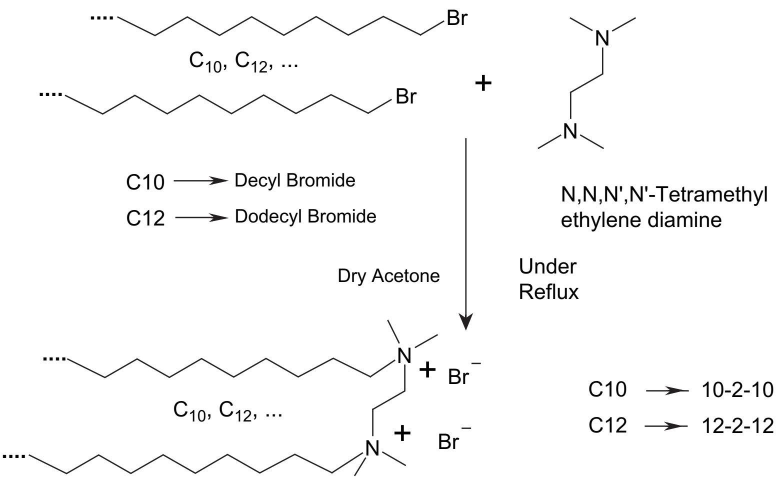
The surface properties of surfactants were determined by the conductivity method. Measurements were performed with 644 METROHM (cell constant = 0.82 cm^{-1}). Experimental temperatures were maintained at 30 °C (in water bath). To perform each series of measurements, an exact volume of 50 ml distilled water was introduced into the bath and the specific conductivity of water measured. The solution was then titrated with surfactant solution and the conductivity measured after each addition.

3.2. UV–Vis spectroscopy

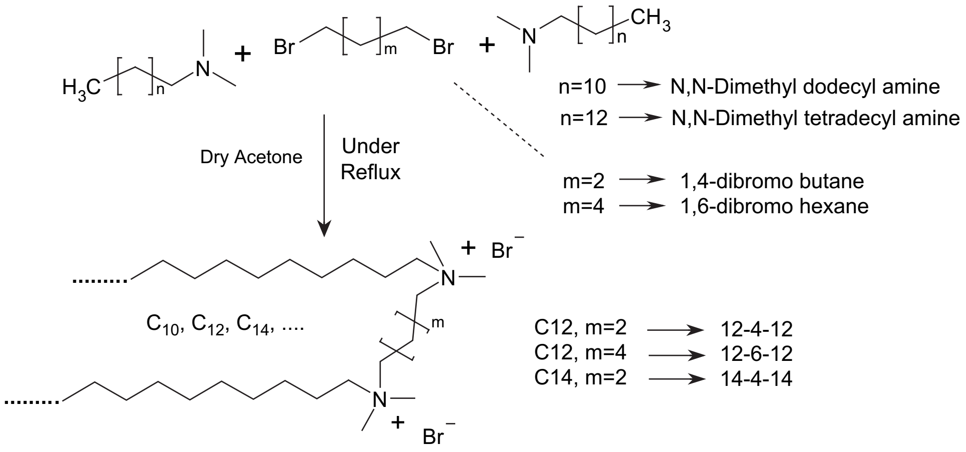
The visible absorption spectra of MO and CR solutions were recorded on a Varian Carry 1E UV–Vis spectrophotometer equipped with a thermostated cell compartment. The dye concentrations were 30 μM for MO and CR. Dye solutions were prepared in double distilled water by addition of different amounts of surfactants below and above the CMC point and kept in appropriate dark bottles. Experiments were performed at 27 °C.

3.3. Dyeing procedure

Cotton samples were washed with nonionic surfactants (2 g/L) at 60 °C for 20 min and dried before dyeing process. A series of dyeing baths were prepared with distilled water



Scheme 1. Synthetic procedure for gemini cationic surfactants of types 10-2-10 and 12-2-12.



Scheme 2. Synthetic procedure for gemini cationic surfactants of types 12-4-12, 12-6-12 and 14-4-14.

including %2 CR (o.w.f.) and liquor to good ratio was 40:1. DTAB and gemini surfactants were investigated in two different submicellar concentrations: (0.1 and 0.2 mM). The effect of each surfactant was studied in three different temperatures (30, 50, 90–92 °C). The dry cotton samples were introduced into the dye baths after reaching to the specified temperature and kept for 1 h. The samples were then rinsed, washed and dried.

The reflectance of cotton samples was measured by a Greta Macbeth Color Eye i 5 spectrophotometer. Each spectrum represents the average of measuring four different points on the samples. The R data (reflectance of opaque dyed samples from 360–740 nm) were used for calculating the K/S values (absorption to scattering coefficients) according to Eq. (1).

$$\frac{K}{S}=\frac{(1-R)^2}{2R} \tag{1}$$

4. Results and discussion

Determination of CMC of 12-2-12 by conductometry at 30 °C has been shown in Fig. 2 as an example. CMCs of all surfactants are listed in Table 1. The CMC of gemini cationic surfactants (12-s-12, s = 2,4,6) are very smaller than DTAB. CMC of 10-2-10 is about six times more than CMC of 12-s-12 series and about 30 times more than CMC of 14-4-14. So, the length of alkyl chain in investigated gemini cationic surfactants affects the CMC greatly but the spacer length has little effect on it.

In addition to the differences in CMC of these surfactants, the morphology of aggregates formed from 12-s-12 gemini surfactants largely depends on the spacer length (s). In the case of 12-s-12 geminis, short spacers (s = 2,3) induce the formation of wormlike micelles; geminis with intermediate spacers (s = 4–12) form spheroidal micelles and vesicles are formed when s = 16,20 [5].

Fig. 3 shows the effect of different concentrations of 12-2-12 on the absorption spectrum of MO. The additions of 12-2-12 (<CMC) cause a hypsochromic shift with a decrease in the intensity of λ_{max}. In submicellar regions (≪1 mM), the dye forms a sparingly soluble precipitate but it becomes soluble as the 12-2-12 concentration reaches the CMC. Different kinds of complexes in the solution can be expected. Although the dye and

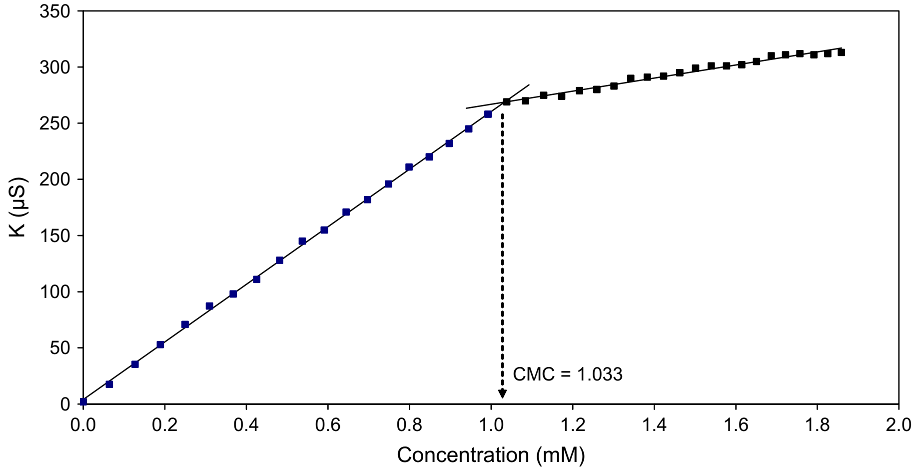


Fig. 2. Determination of CMC of 12-2-12 by conductometry at 30 °C.

Table 1
Critical micelle concentration (CMC) of DTAB and gemini cationic surfactants

Surfactant	CMC (mM) at 30 °C
DTAB	13.55
10-2-10	6.51
12-2-12	1.03
12-4-12	1.18
12-6-12	1.22
14-4-14	0.20

surfactant are individually hydrated in the solution, they can sometimes meet each other in aqueous solution and the long-range electrostatic and short-range hydrophobic forces cause the formation of dye–surfactant complexes. There are strong indications that the dye molecules are arranged in a parallel way (H-type aggregation). It means the complex is a monomer involving electrostatic interaction between the positive charge of cationic surfactant and negatively charged sulphonate group, with the alkyl chain of cationic surfactant in close contact with the rest of the dye molecule and, particularly, the azo group (the chromophoric unit) [5,14]. Some of these complexes can aggregate and precipitate in the solution which is in equilibrium with the precipitates. The loss of absorbance in dye solution in submicellar region is partly due to the precipitation of dye in the solution. Bathochromic shift of about 70 nm occurs at CMC of about 1 mM. Further addition of 12-2-12 ($>$CMC) leads to hyperchromic shift which is characteristic of MO bound to cationic micelles. It is obvious that association of surfactant molecules (micelles) occurs with increase in concentration. This phenomena can increase the solubility of MO, dye–surfactant aggregates and precipitates in the solution. So, bathochromic shift along with an increase in the intensity of absorbance can be seen in UV–Vis spectra (Fig. 3). Effect of concentration of other surfactants on the position of the wavelength of maximum absorption of MO and their absorbance in aqueous solution investigated similarly and summarized in Fig. 4. The positions of the λ_{max} of MO in aqueous solutions show that at concentrations far below the CMC of these surfactants, strong interactions occur. The minimum hypsochromic shift which is about 45–55 nm is in the presence of DTAB.

But, the gemini cationic surfactants show larger hypsochromic shift (75–120 nm). This difference in the hypsochromic shift can be attributed to stronger charge density and hydrophobic forces of geminis in comparison to DTAB and it is obvious that every gemini molecule can bind and complex with two MO molecules. The influence of spacer length of geminis on their aggregation with MO is also visible (Fig. 4) and the aggregation is higher for smaller spacer in 12-*s*-12 series. The hydrophobicities of 12-4-12, 12-6-12 and 14-4-14 are more than 12-2-12 and 10-2-10 and we may expect to have stronger anionic dye–cationic surfactant aggregation because of stronger hydrophobic and noncoulombic interactions. But, the special behavior of geminis ($s = 2$) in aggregation with MO shows that the long-range electrostatic forces and short-range hydrophobic interactions are not the only reason for dye–surfactant aggregation. Thus, other factors such as conformation, mobility and dispersivity of surfactant's molecule may also play important roles for dye–surfactant aggregation. The formation of parallel complexes of dye and surfactant molecules in the solution and different kinds of aggregates may depend on the spacer length. The flexible methylene groups in spacer can affect the conformation and packing parameters of geminis in the dye solution. The nature of spacers has also a large influence on the aggregation of geminis. But, it has been reported that the spacer length hardly affects dye–surfactant aggregation for 12-*s*-12, $s = 4,8,12$ [5]. Regardless of the effect of different pH and measuring method, it can be concluded that in geminis with two methylene groups in their spacer, the aggregation with anionic dyes such as MO is higher than in other species. Thus, further investigation in such series of geminis is necessary.

As an example, the effect of different concentrations of 12-2-12 on the absorption spectrum of CR is shown in Fig. 5. The additions of 12-2-12 ($<$CMC) cause a hypsochromic shift (498 → 462 nm) with a decrease in the intensity of λ_{max}. Further addition of 12-2-12 leads to bathochromic shift and increase in λ_{max} which is not sharp at CMC. Effect of concentration of single-tailed and other gemini cationic

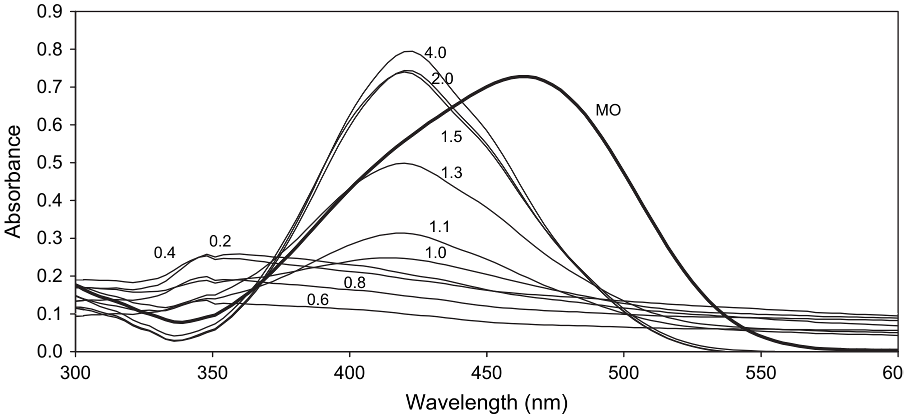


Fig. 3. Effect of 12-2-12 on absorption spectrum of MO (at 27 °C, [MO] = 30 μM). The numbers represent the concentration of 12-2-12 in mM.

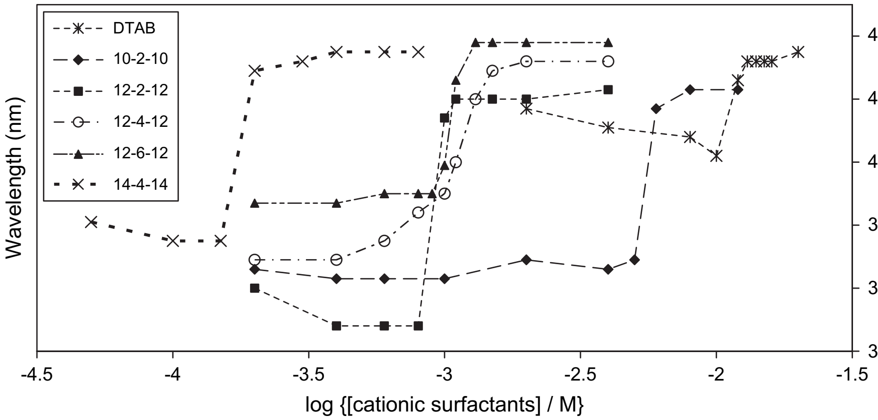


Fig. 4. Effect of DTAB and gemini cationic surfactants on the position of the wavelength of maximum absorption of MO (at 27 °C, [MO] = 30 μM).

surfactants on the position of the wavelength of maximum absorption of CR and their absorbance in aqueous solutions are depicted in Fig. 6. The positions of the λ_{max} of CR in aqueous solutions with the surfactants used show that at concentrations far below the CMC of these surfactants, a strong interaction occurs (decrease in the intensity of λ_{max} and hypsochromic shift). The influence of spacer and alkyl length of geminis on the aggregation with CR is not clearly visible and they have small effects on the aggregation.

Dyeing of cotton fabrics with CR in the presence of different amounts of DTAB and gemini cationic surfactants at different temperatures (30, 50 and 90–92 °C) prove this fact. Fig. 7 shows the *K*/*S* values of dyed cotton samples with CR in the presence of different amounts of cationic surfactants used (0.1 and 0.2 mM) at 30 °C. There is a large difference between the inhibited effects of DTAB and gemini cationic surfactants on dyeability of cotton fabric. Results show that the amounts of dye absorbed on the fabric decrease with increasing hydrophobicity of surfactants. However, the differences are very small between 12-*s*-12 and 14-4-14 at higher temperatures. Cotton (cellulose fiber) has a negative surface charge in water, which repels the dye anion. The affinity and substantivity of an anionic azo dye such as CR to cotton are attributed to hydrogen bonding and van der Waals forces. Due to aggregation of anionic dye–cationic surfactant in aqueous solution, the dyeability of cotton samples reduces greatly. By increasing the temperature, some parts of these complexes break and the dyeability of samples improve that is a very common phenomena. Such dyeing process is a very useful method of studying the dye–surfactant aggregation in aqueous solution. Because the amounts of dye in a same condition in the presence of different cationic surfactants will be investigated and insoluble precipitation and duration of sample preparation will not interfere in measurements. The *K*/*S* values of dyed cotton samples in the presence of gemini cationic surfactants are very similar to each other and there are not much differences between them specially in higher temperatures. The low amounts of geminis (0.2 mM) block CR in dyeing process almost completely. The affinity of this dye to cotton is reduced by cationic surfactants mainly because of dye–surfactant aggregation and precipitation.

The CR molecule has two azo groups, a larger linear structure and two sodium sulphonate groups which lead to higher solubility in water (Fig. 1). So, in comparing with MO, higher

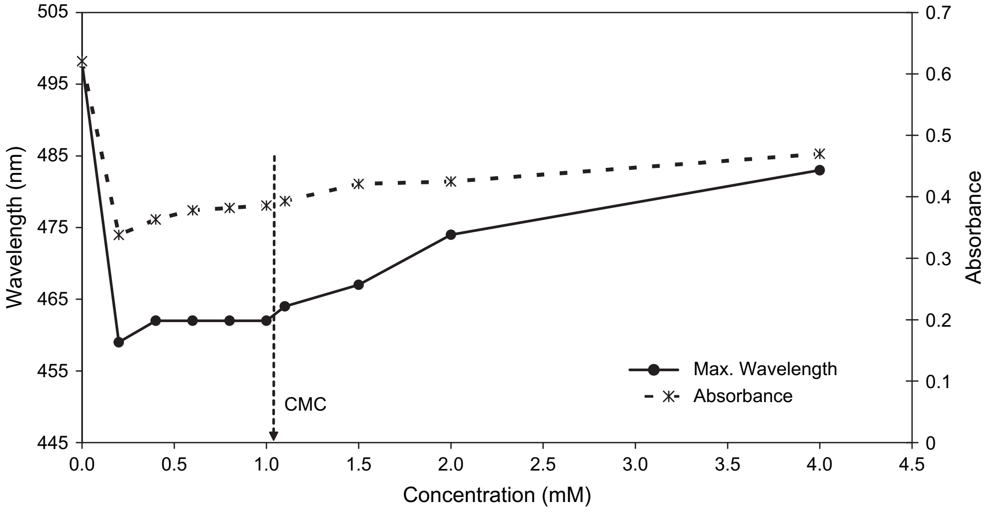


Fig. 5. Effect of concentration of 12-2-12 on the position of the wavelength of maximum absorption of CR and their absorbance (at 27 °C, [CR] = 30 μM).

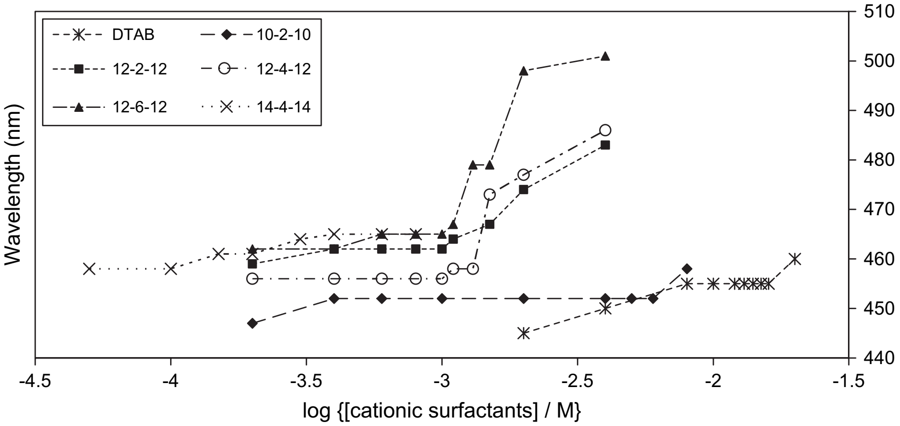


Fig. 6. Effect of DTAB and gemini cationic surfactants on the position of the wavelength of maximum absorption of MO (at 27 °C, [CR] = 30 μM).

electrostatic and smaller hydrophobic interactions with cationic surfactants may occur. The CR samples, in contrast to MO solutions, do not have large and sharp bathochromic shifts in CMC region of the used cationic surfactants. Although, such bathochromic shifts happen at higher concentrations (Figs. 5 and 6).

5. Conclusion

The interactions of a conventional DTAB and a series of gemini cationic surfactants in aqueous solution with two different anionic azo dyes, Methyl Orange (MO) with one sodium sulphonate group and Congo Red (CR) with two sodium sulphonate groups, have been studied by UV–Vis spectroscopy and dyeing process. The results show that aggregation of surfactant and anionic azo dyes takes place at surfactant concentrations far below the critical micelle concentration of the individual surfactants; their λ_{max} have considerable hypsochromic shift along with a decrease in their intensities which strongly depend on combination of bulk hydrophobic and electrostatic interactions. Aggregation of *m*-2-*m* geminis with MO are surprisingly higher than other species. By increasing cationic surfactants' concentration bathochromic shifts occur. This can be due to the increase in the solubility of MO in cationic micelles. So, by reaching to the CMC point of individual surfactants, a bathochromic shift followed by a sharp increase in the intensity of λ_{max} occurs. The sensitivity of small anionic azo dyes such as MO to the polarity of the media makes them extremely suitable for reporting the presence of hydrophobic micro-domains in aqueous solution. Thus, they can be used to determine critical micellar concentration (CMC) of different cationic surfactants. However, such an ideal behavior like MO is not observed for CR solutions and bathochromic shifts occur at higher surfactants' concentrations. This could be due to larger linear structure of CR molecule that has two sodium sulphonate groups which lead to higher solubility in water and smaller hydrophobic interactions with cationic surfactants in comparison with MO. The inhibited effects and large difference between DTAB and gemini cationic surfactants on dyeability of cotton fabrics with CR show that geminis are much more stronger complexing agents (inhibitors) in the same concentration.

It can be concluded that the long-range electrostatic forces and short-range hydrophobic interactions are very important factors of dye–surfactant aggregation in aqueous solutions and other factors such as possible conformation, mobility and dispersivity of surfactant's molecule may also play role in this regard. So, intermolecular interactions are a function of the molecular architecture of both components (dye and

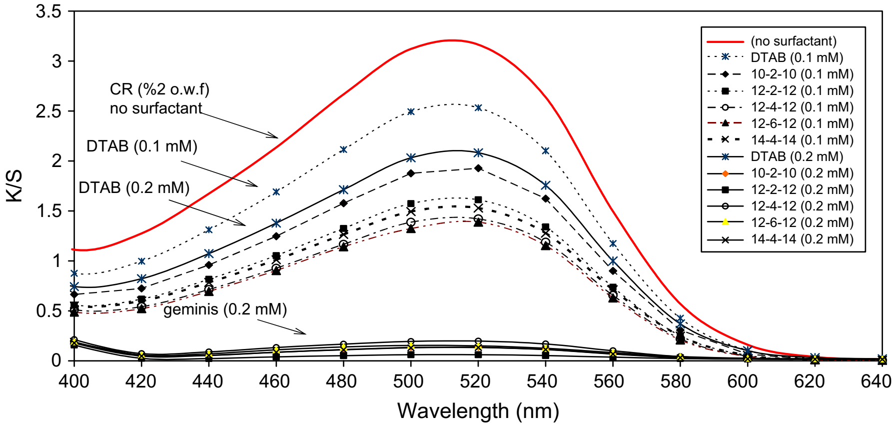


Fig. 7. *K/S* values of cotton samples dyed with CR (%2 o.w.f) in the presence of different amounts of DTAB and gemini cationic surfactants at 30 °C.

surfactant) and complexes of geminis with anionic azo dyes are stronger than conventional cationic surfactants in the same concentration. This may lead to their possible application as dye levelling agents and also dye fixing agents in very low concentrations.

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