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CLOUD POINT OF MIXED SOLUTIONS OF IONIC-NONIONIC SURFACTANTS: INFLUENCE OF ELECTROLYTES

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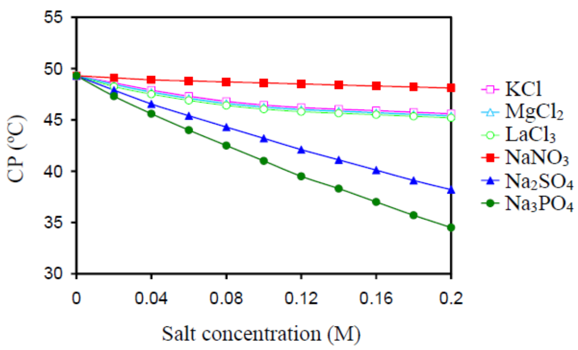
Influence of several inorganic salt's additives, ionic and nonionic surfactants on the cloud point (CP) of linear nonionic surfactant, $C_{12}EO_6$ were investigated. The cloud point of this surfactant depends on both its concentration and the additive concentration. Sodium dodecyl sulfate (SDS) and cetyl trimethyl ammonium bromide (CTAB) presence in the system, causes in considerable increase in the cloud point of 1 wt % $C_{12}EO_6$ micellar solution. The effect of different electrolytes [including KCl, $MgCl_2$ and $LaCl_3$ for anionic (SDS)-nonionic ($C_{12}EO_6$) surfactant solution, and $NaNO_3$, Na_2SO_4 and Na_3PO_4 for cationic (CTAB)-nonionic ($C_{12}EO_6$) surfactant mixtures] on the cloud point of anionic-nonionic and cationic-nonionic surfactant solutions has also been extensively studied. For a concentration of less than 0.1 M of the electrolytes, the CP is a slightly decreases. However, addition inorganic salts to the $C_{12}EO_6$ solution in the presence of SDS or CTAB, is lowering the cloud point significantly. After adding the electrolytes to SDS-$C_{12}EO_6$ mixed micellar solutions, the activity of polyvalent cations is more than the univalent cation, the order of decreasing effect is $LaCl_3 > MgCl_2 > KCl$. However, polyvalent anions are more effective than the univalent anion, in case of addition of the electrolytes to CTAB-$C_{12}EO_6$ mixed micellar solutions. The order of decreasing effect is $Na_3PO_4 > Na_2SO_4 > NaNO_3$.

ABSTRACTED PARAGRAPH



Effect of electrolytic additives on the cloud point temperature of 1 wt % $C_{12}EO_6$ solutions.

INTRODUCTION

Clouding behavior refers to the phenomenon of separation homogeneous solutions of an amphiphilic substances into a surfactant-rich and a surfactant-poor phase at a definite temperature. The cloud point temperature (CPT) is the temperature, at which phase separation occurs. This reflects the importance of the threshold temperature of clouding of nonionic surfactants.[1] At a definite temperature, clouding is attributed to the efficient dehydration of hydrophilic portion of micelles. The clouding phenomenon is due to the interaction of nonionic surfactant micelles via an attractive potential, whose well depth increases with temperature.[2] When these micelles approach the cloud point, they attract each other and form clusters.[3] The value of CP depends on the structure and concentration of the surfactant and the presence of additives.[4–9]

Due to their amphiphilic nature, surfactants have versatile applications in solubilization of substrates,

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which are insoluble, both in aqueous and non aqueous media.[10,11] They are widely used in the extraction and preconcentration of polycyclic aromatic hydrocarbons,[12–16] mobility of drugs in aqueous and lipid media,[17,18] probing of biological systems,[19–23] etc. Mixed ionic-nonionic surfactants are also of considerable applied and fundamental interest. In the area of enhanced oil recovery for example, such surfactant systems might show advantageous solubility behavior, exhibiting cloud points (CPs) higher than those of the pure nonionic surfactant, along with krafft points lower than those of the pure ionic surfactants. Where this is the case, mixed surfactants could be employed over a wider range of temperature, salinity, and hardness conditions than the individual surfactants. Starting from this consideration, many experiments have shown that the CPs of nonionic surfactants are dramatically increased on adding small amounts of ionic surfactant, either anionic or cationic.[24–26] On the other hand, the CPs of mixed nonionic-ionic surfactant systems decrease when small amounts of inorganic salts are added.[27–31] These results can be explained by considering that the mixed micelles contain ionic monomers. This factor is the origin of the electrostatic repulsion between micelles.[25–27,30,31] When inorganic salts are added to the solution, the micellar charge is screened and the CP is lowered.[32,33]

Non-ionic linear ethoxyl alcohol ($C_{12}E_6$) is an attractive surfactant for environmental and industrial applications, including cloud point extraction and biodegradation of pollutants. To the author's knowledge, the clouding phenomenon of $C_{12}E_6$ in the presence of electrolyte additives and mixed ionic and non-ionic surfactants has not been previously discussed. The novelty and originality of this research concerns the study of the effect of electrolytes on the cloud point of $C_{12}EO_6$ without any additives. Subsequently, mixtures of $C_{12}EO_6$ + ionic surfactants were studied, where significantly high cloud points were obtained. We attempted to reduce the cloud point of the mixed mixtures of $C_{12}EO_6$ + ionic surfactants by adding electrolytes, which resulted in a drastic reduction in the cloud point. The goal was to make extraction processes economical and improve the cloud point extraction of organic compounds.

This paper presents the experimental results showing the effect of $C_{12}EO_8$ nonionic surfactant, SDS and CTAB ionic surfactants and various inorganic salts on the cloud point of $C_{12}EO_6$ nonionic surfactant. In addition, the effect of different inorganic salts (including KCl, $MgCl_2$ and $LaCl_3$ for SDS- $C_{12}EO_6$ surfactant solutions. $NaNO_3$, Na_2SO_4 and Na_3PO_4 for CTAB- $C_{12}EO_6$ surfactant mixtures) on the CP of SDS- $C_{12}EO_6$ and CTAB- $C_{12}EO_6$ surfactant solutions was also carried out.

EXPERIMENTAL

Materials

Nonionic surfactants, hexaethylene glycol mono-n-dodecyl ether ($C_{12}E_6$) [$CH_3(CH_2)_{11}(OCH_2CH_2)_6OH$] and octaethylene glycol mono-n-dodecyl ether ($C_{12}E_8$) [$CH_3(CH_2)_{11}(OCH_2CH_2)_8OH$] were purchased from Fluka with a purity of 98%. Ionic surfactants, SDS [$C_{12}H_{25}SO_4Na$] and CTAB [$C_{19}H_{42}NBr$] were purchased from Merck. Reagent grade of inorganic salts were obtained from Fluka, Alfa Aesar, Aldrich, Panreac and Chem-Lab. These inorganic salts include KCl, $MgCl_2$, $LaCl_3$, $NaNO_3$, Na_2SO_4 and Na_3PO_4. All the chemicals were used as received without further purification. Doubly distilled water was used to prepare sample solutions.

Methods

CPs of surfactant solutions were determined visually point by point by preparing aqueous solutions containing various surfactants. Aliquots (1–2) cm^3 were enclosed in small capped vials immersed in a thermostated bath. The temperature was progressively raised until the solution became turbid. The uncertainty range could be decreased up to ± 0.1 ºC. The temperature at which the turbidity disappeared on cooling was also noted. CPs values stated throughout this article are averages of the appearance and disappearance temperatures of the clouds. These temperatures did not differ by more than 0.4 ºC.

RESULTS AND DISCUSSION

Cloud point as a function of surfactant concentration

Dependence of CP of $C_{12}E_6$ on its concentration in water is shown in Fig. 1. It shows a cusp on the curve of CP as a function of $C_{12}E_6$ concentration.

With increasing $C_{12}E_6$ concentration, the CP decreases in dilute regime until reaching a minimum value, and then increases. The critical concentration (C_C) of $C_{12}E_6$ solution is 2.4% w/w and the corresponding critical temperature (T_C) is 48.5°C. The obtained T_C in this paper is rather low compared with that obtained by Schubert et al (51.3°C).[11] It may be ascribed to the fact that the transition temperatures are extremely sensitive to impurity ($C_{12}E_6$ used in this work without farther purification). Similar trend on change in CPT with increasing surfactant concentration in dilute regimes has been reported elsewhere on some other surfactants.[33] Below the curve of CP, there exists only one liquid phase (micellar phase), whereas two coexisting liquid phases, surfactant rich micellar phase and water phase, are found in the region above the curve.

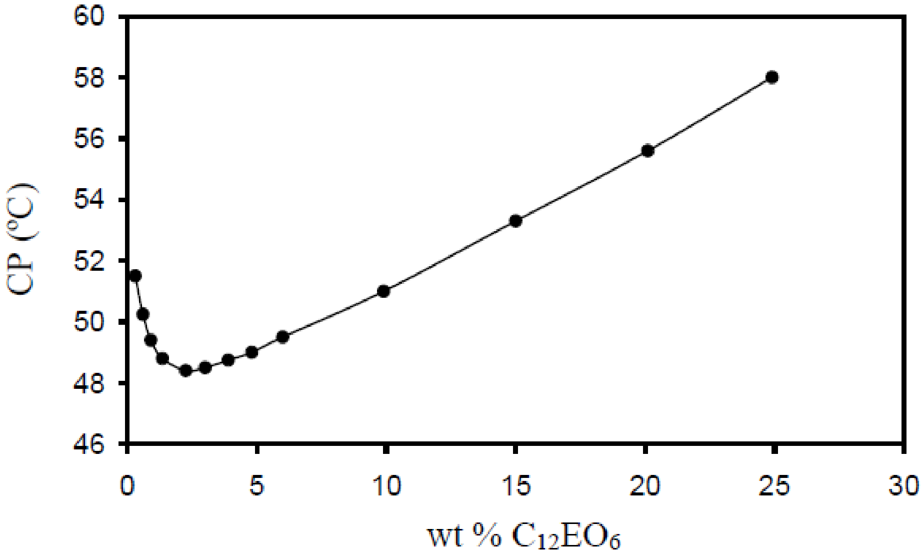

Fig. 1 – Cloud point (CP) of $C_{12}EO_6$ as a function of wt % of $C_{12}EO_6$ in solution.

The cloud point of $C_{12}E_6$ + $C_{12}E_8$ mixtures

In many applications, mixtures of surfactants rather than a pure surfactant have to be used to maintain desired physical properties of the surfactant solution and to achieve desired performance such as high solubilization capacity. The CP of binary surfactant mixtures of $C_{12}E_6$ with $C_{12}E_8$ were measured (Fig. 2).

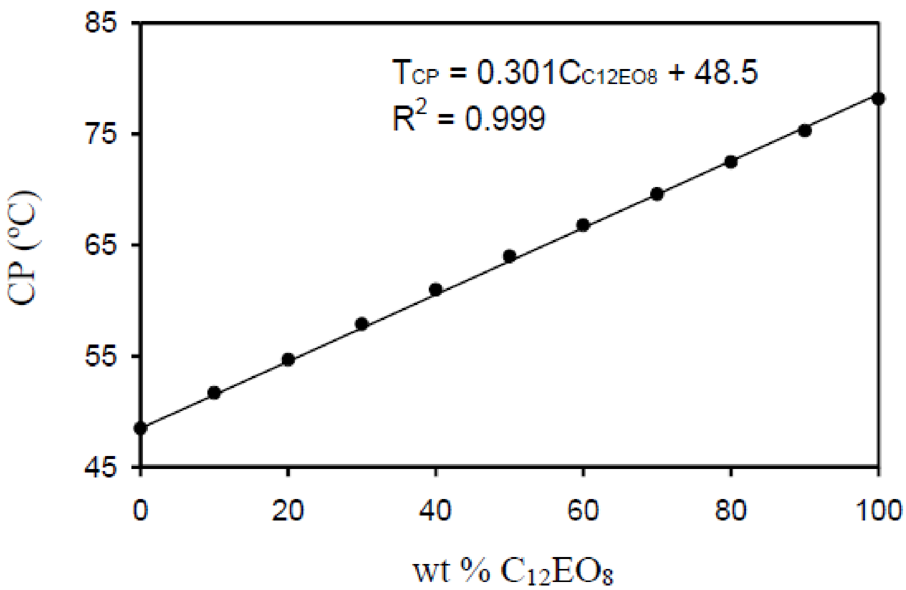

Fig. 2 – The cloud point curve of mixtures of ($C_{12}EO_6$ + $C_{12}EO_8$) in water solution at a total concentration of 2 wt % nonionics.

The total surfactant concentration in the respective binary mixture was fixed at 2 wt %. It is observed that the CPs of the binary mixed surfactants are laid between those of individual component surfactants. Hence, the CP of mixed surfactants can well be ascribed by a linearly additive model.

For $C_{12}E_6$ and $C_{12}E_8$ mixed surfactants, the following correlation was obtained:

$$T_{CP} = 0.301\ C_{C12EO8} + 48.5, \qquad R^2 = 0.999$$

where T_{CP} and C_{C12EO8} denote the CP temperature of mixed surfactants and concentration of $C_{12}E_8$ in wt %, respectively. This linear model provides us with a convenience in formulating mixed surfactant systems to have desired CP for specific applications.

The effect of small amounts of ionic surfactants on the cloud point of $C_{12}E_6$ solutions

Figure 3 displays the CPs of 1 wt % $C_{12}E_6$ solution in presence of two ionic surfactants, sodium dodecyl sulphate (SDS) and cetyl trimethyl ammonium bromide (CTAB).

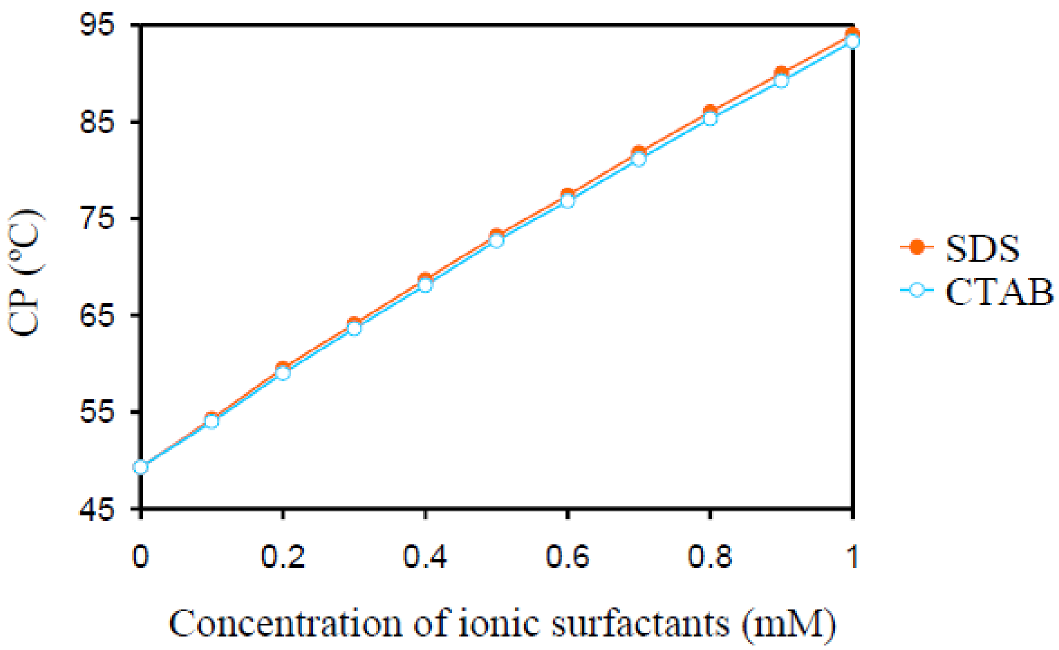

Fig. 3 – The effect of ionic surfactants (SDS and CTAB) on the cloud point of 1 wt % $C_{12}EO_6$ solutions.

The concentrations of the added SDS or CTAB were far below the concentration of $C_{12}E_6$ and, in most cases; the concentrations of SDS or CTAB used were lower than their critical micelle concentrations (CMC_S). It is observed that, in general, presence of small amounts of these two ionic surfactants increased the CP of $C_{12}E_6$. For example, the addition of 1 mM SDS or CTAB raise the CP of 1 wt % $C_{12}E_6$ solution from 49.3ºC to 93.5ºC. Charged ionic surfactant molecules can be either adsorbed on nonionic surfactant micelles or can form mixed micelles with nonionic surfactant molecules. As a result of adding these ionic surfactants, the electrostatic repulsion between nonionic micelles increases and, consequently making it more difficult for the micelles to aggregate together and leading to an increase in the CP.[25,26] This effect becomes more prominent when concentrations of ionic surfactants approaching their CMC_S.

Valaulikar and Manohar[25] considered the rise in the clouding temperature of Triton X-100 (TX- 100) upon addition of very small concentrations of anionic surfactants in terms of increase in the surface charge of the micelle. At such small concentrations (far below the CMC of ionic surfactant used) the ionic surfactant molecules were present either as monomers or as mixed micelles with TX- 100. According to the authors, this additional surface charge increases the repulsion between the micelles, thus supporting the view regarding the cloud point as the aggregation of micelles rather than the growth of micelles.

The effect of inorganic salts on the cloud point of $C_{12}E_6$ solutions

The effect of inorganic salts on the CP of $C_{12}E_6$ at 1 wt % concentration is shown in (Fig. 4).

The figure indicates that the addition of KCl, $MgCl_2$, $LaCl_3$, $NaNO_3$, Na_2SO_4 and Na_3PO_4 can decrease the CP of the $C_{12}E_6$. The change is almost linearly proportional to the concentrations of the inorganic salts in the solutions. The observed effects of the inorganic salts on the CPs of nonionics have been explained in terms of so-called "salting-in" and "salting-out" effects.[17,34] The difference in the effects of the salts could be attributable to the different effects of their ions on modifying the solvent property of water.[34] Ions that enhance the property of solvent of water can rise the solubility of surfactants, which as a result increases in CPs. This kind of ions is termed as water structure-breaking ions. Presence of structure-breaking ions can hinder self-association of water molecules and hence, will lead to an increasing extent of hydrogen bond formation between water molecules and ether groups in nonionic surfactants. In contrast, structure-making ions can lower the solvent property of water and result in decrease of CPs. According to various measures of the effects of ions on the structure of water, cations such as Li^+, Na^+, K^+, NH_4^+, Ca^{2+}, Mg^{2+} and anions, such as F^-, SO_4^{2-}, CO_3^{2-} and PO_4^{3-} are structure-making ions, while Cl^-, Br^-, I^- and NO_3^- are structure-breaking ions.[35]

Interestingly, the overall effect of a salt on the CP of a surfactant solution depends both on its cation and anion. However, effects of cations are relatively smaller than anions, especially with the large polyatomic anions. Similarly, a salt having an overall effect in decreasing the CP of a nonionic surfactant is denoted as a "salting-out". Presence of this kind of salts reduces the solubility of surfactants. Conversely, salts with "salting-in" effect increase the aqueous solubility of surfactants and in turn increase their CPs. Hence in the present work, the observed decrease of CP in the presence of both cations and anions KCl, $MgCl_2$, $LaCl_3$, $NaNO_3$, Na_2SO_4 and Na_3PO_4, can be attributed to their salting-out effects. However, it is clearly observed that the addition of $NaNO_3$ has a very low influence on the CP of $C_{12}E_6$. The order in the CP depression is as follows:

$$Na_3PO_4 > Na_2SO_4 > LaCl_3 > MgCl_2 > KCl > NaNO_3.$$

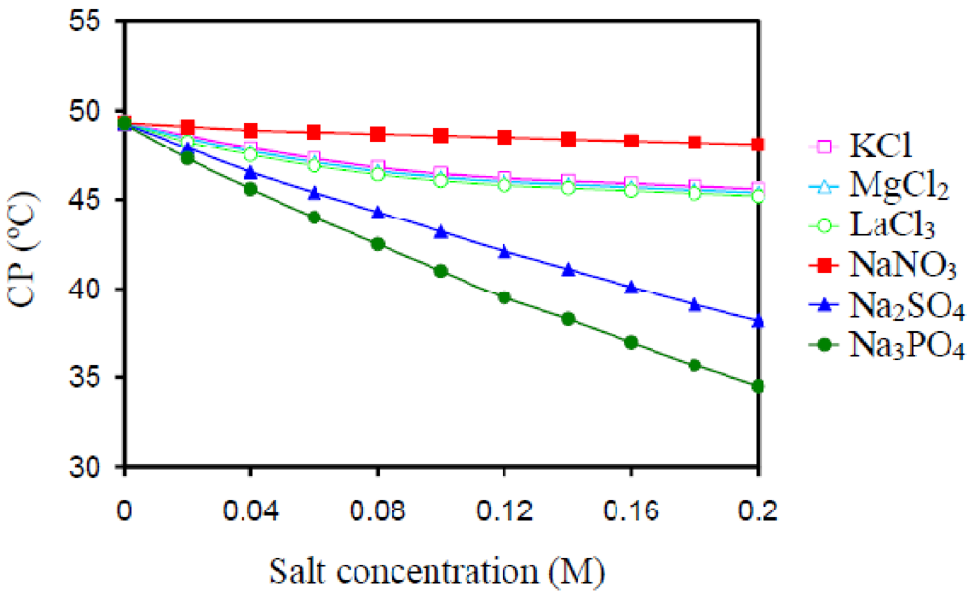

Fig. 4 – Effect of electrolytic additives on the cloud point temperature of 1 wt % C12EO6 solutions.

The effect of small amounts of inorganic salts on the CPs of ionic- $C_{12}E_6$ surfactant solutions

Figures 5 and 6 show the dependence of the CPs for 1 wt % $C_{12}E_6$ solution in the presence of 1 mM SDS and 1 mM CTAB, respectively, as a function of the molar concentrations of different added inorganic salts.

From the results of the previous section, it is known that these salts have only small effects on the CP of $C_{12}E_6$ (Fig. 4). However, when salts are added to $C_{12}E_6$+SDS or $C_{12}E_6$+CTAB mixed systems, a pronounced minimum in the CP was observed, CP of these mixed systems was greatly influenced with the concentration of added inorganic salt leading to the depression of CP below 50ºC (Figs. 5 and 6).

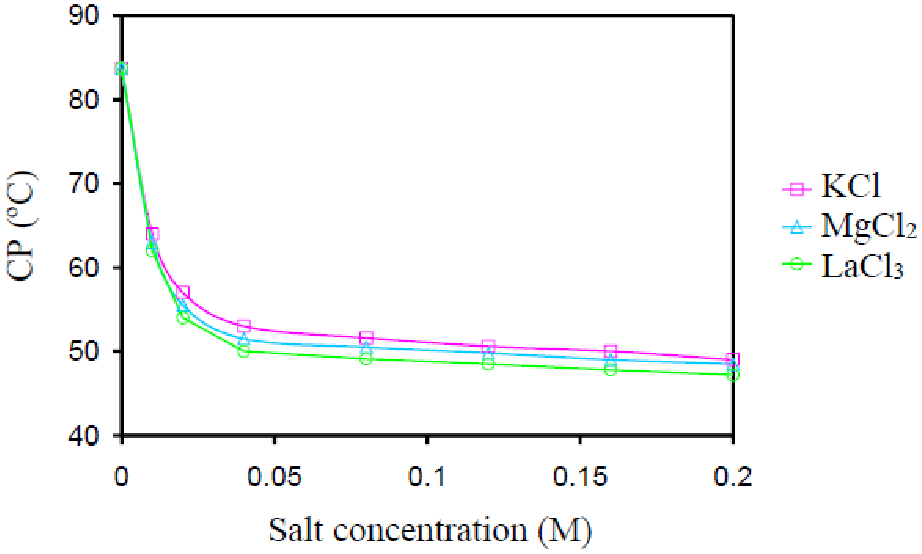

Fig. 5 – The effect of the addition of different electrolytes on the cloud point of 1 wt % C12EO6 solutions in the presence of 1 mM SDS.

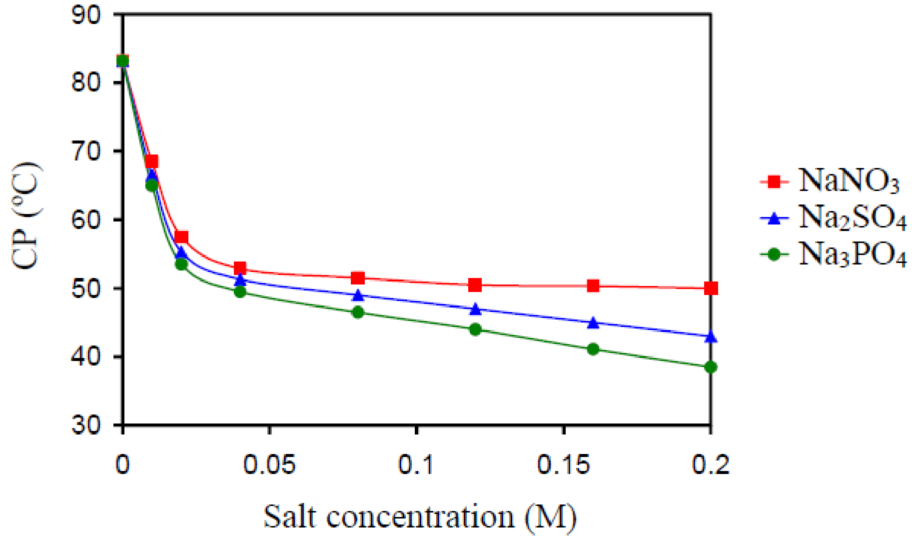

Fig. 6 – The effect of the addition of different electrolytes on the cloud point of 1 wt % C12EO6 solutions in the presence of 1 mM CTAB.

Marszall.[27] has investigated the effect of electrolytes on the cloud point of ionic-nonionic surfactant solutions. When ionic surfactant is added to an TX-100 solution, the cloud point increases as expected. However, when small amounts of electrolytes (NaC1 or NaSCN) are added to the TX-100 solution in the presence of very low concentrations of sodium dodecyl sulfate (one SDS molecule per micelle of TX-100), the cloud point decreases in a ratio depending on the concentration of electrolyte and the nature of co-ion. The authors suggested that this effect is due to the change of counterion binding on the mixed ionic-nonionic micelles. It is also interesting to compare the action of different inorganic salts on the CP of the same mixed micelles. In view of the formation of ionic-nonionic mixed micelles, it is reasonable to expect that for negatively charged anionic-nonionic mixed micelles, the valency of the added cation is a decisive factor in the CP modification. On the contrary, for positively charged cationic-nonionic mixed micelles, the valency of the anion plays the dominant role. Gu *et al.*[26] have shown that this statement is true for the system TX-100 in the presence of SDS or CTAB. It also shown from Fig. 5 that for SDS- $C_{12}E_6$ mixed micellar solution, the polyvalent cations are more active than the univalent cation, in the order $LaCl_3$>$MgCl_2$>KCl. Similarly, Fig. 6 shows that for CTAB- $C_{12}E_6$ mixed micellar solution, the polyvalent anions are more active than the univalent anion, in the order Na_3PO_4>Na_2SO_4>$NaNO_3$. As pointed out by Gu *et al.*[26] Furthermore, if the valency of the added counterion is the factor that plays the dominant role in the CP depression and not the valency of the co-ion, one may anticipate that the CP of a particular ionic-nonionic surfactant solution is only a function of the counterion concentration, no matter what types of inorganic salts are used. This is the case for the system CTAB- $C_{12}E_6$ (1 wt % $C_{12}E_6$ solution in the presence of 1 mM CTAB) shown in Fig. 7.

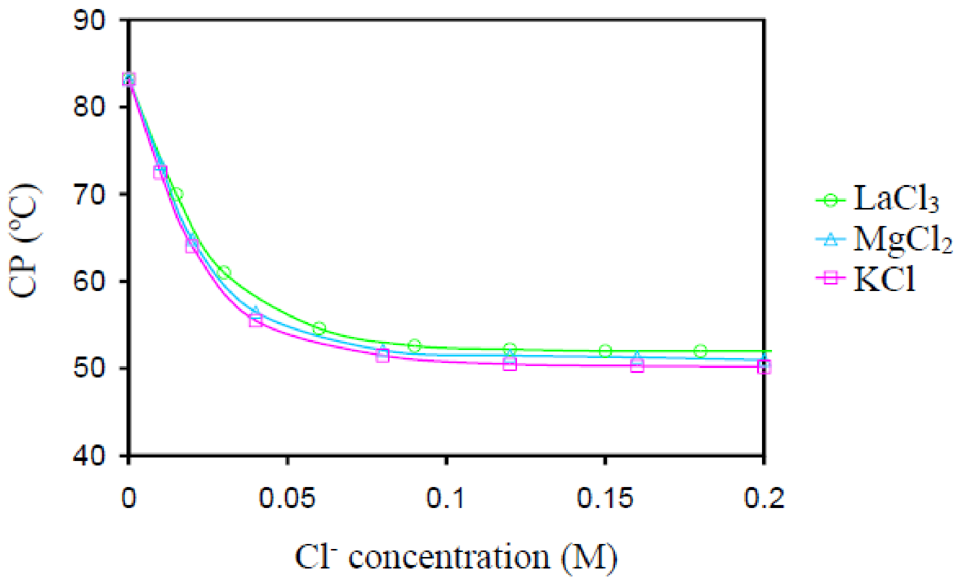

Fig. 7 – The effect of the concentration of chloride ions (with different cations) on the cloud point of 1 wt % solutions of C12EO6 in the presence of 1 mM CTAB.

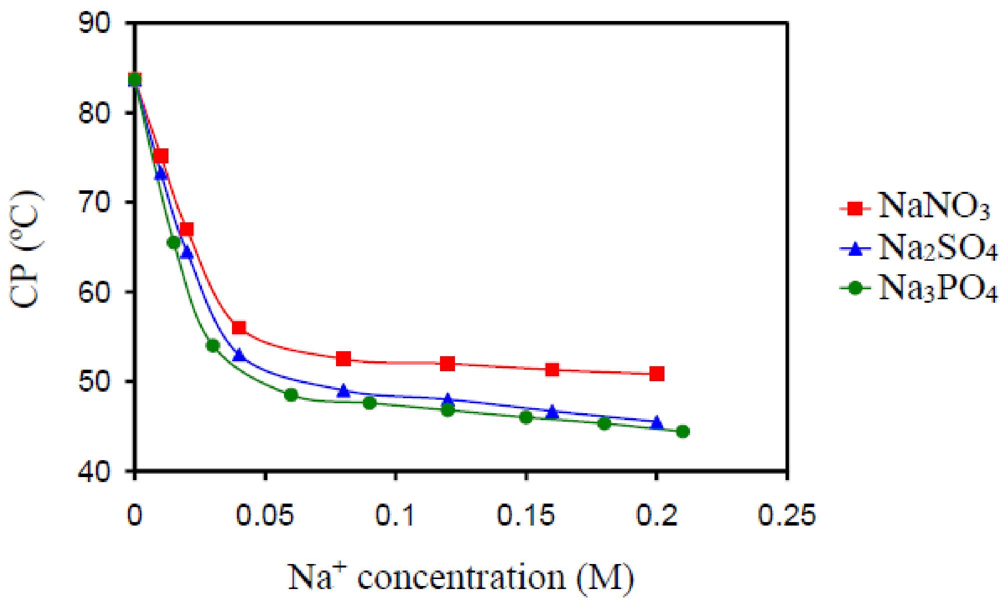

Fig. 8 – The effect of the concentration of sodium ions (with different anions) on the cloud point of 1 wt % solutions of C12EO6 in the presence of 1 mM SDS.

The CP curves are obtained for the mixed CTAB- $C_{12}E_6$ solutions in the presence of chlorides with different valencies of cations (KCl, $MgCl_2$ and $LaCl_3$) using the concentration of chloride ions as the abscissa. However, for the system SDS-$C_{12}E_6$ (1 wt % $C_{12}E_6$ and 1 mM SDS) in the presence of sodium salts with anions of different valencies ($NaNO_3$, Na_2SO_4 and Na_3PO_4), the CP curves are somewhat different from each other when using the concentration of sodium ions as abscissa (Fig. 8). This implies that some significant specific interactions of the anions are present. Marszall[30] have also noted the importance of co-ion species in the counterion binding on the CP of mixed micelles (SDS-TX-100) in the presence of NaSCN and NaCl.

CONCLUSION

The influence of various factors on the CPs of nonionic linear ethoxylated alcohol surfactant, $C_{12}E_6$, is investigated. We choose this surfactant due to its potential environmental and industrial applications, such as CP extraction and biodegradation of pollutants. The CP of $C_{12}E_6$ showed a minimum in variation with concentration. Presence of inorganic salts (KCl, $MgCl_2$, $LaCl_3$, $NaNO_3$, Na_2SO_4 and Na_3PO_4) can decrease the cloud point. Mixed nonionic-nonionic surfactant system shows clouding phenomenon at temperature, which are intermediate to that of corresponding pure surfactants. The obtained results showed that the presence of ionic surfactants, SDS and CTAB, increased the cloud point of $C_{12}E_6$. Such effects became more prominent when concentrations of ionic surfactants approached their critical micelle concentrations. Results in this report also show that for SDS- $C_{12}E_6$ mixed micellar solutions, the polyvalent cations of the added inorganic salts are more active than the univalent cation, the order of decreasing effect is $LaCl_3$>$MgCl_2$>KCl; while for CTAB- $C_{12}E_6$ mixed micellar solutions, the polyvalent anions of the added inorganic salts are more effective than the univalent anion, the order of decreasing effect is Na_3PO_4>Na_2SO_4>$NaNO_3$. Mixed micelle cloud point extraction is a highly promising methodology for establishing an analytical and extraction procedure for organic or mineral materials. The extent of extraction is influenced by the presence of additives and the concentrations of surfactants used. The small volume of the surfactant-rich phase obtained using the proposed cloud point methodology allows for the design of an extraction strategy characterized by robustness, low cost, good extraction efficiency, and low toxicity compared to those using organic solvents. Mixed micelle systems are highly flexible and allow cloud point extraction at room temperature. This study demonstrates an innovation in the use of surfactants in analytical applications. The results of this work clearly demonstrate the potential and versatility of this method, which can be applied to the extraction of toxic organic materials and enables identification of these substances in samples of environmental and toxicological interest.

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