



## Separation of congo red by surfactant mediated cloud point extraction

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### Abstract

An attempt has been made to remove color from wastewater containing congo red (a toxic cationic dye) by surfactant mediated cloud point extraction (CPE) in batch mode using Triton X-100 (TX-100) as non-ionic surfactant. Most of the dye molecules get solubilized in the coacervate phase leaving a dye free dilute phase. The effects of the concentration of feed mixture (both dye and surfactant), temperature and salt concentration on the concentration of both dye and surfactant in the coacervate phase and dilute phase have been studied. An optimum set of surfactant concentration, temperature and salt concentration are obtained for removal of dye. Experimental investigations have also been carried out for the recovery of surfactant from dilute phase applying solvent extraction (SE) in batch condition using heptane as extracting medium.

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**Keywords:** Congo red; Cloud point extraction; Non-ionic surfactant; Coacervate phase; Solvent extraction

### 1. Introduction

Color removal from effluent is one of the most difficult requirements faced by the textile finishing, dye manufacturing, pulp and paper industries. Among the various types of dye, congo red (CR) dye is used in wool and silk to give red color with a yellow fluorescence. The effluents containing dyes are highly colored resulting in major environmental problems. So these colored wastes need to be treated before disposal. Many investigators

have studied different techniques for removal of colored dye from wastewater, e.g., micellar-enhanced ultrafiltration [1], several oxidation processes [2–4], ozonations [5], nanofiltration [6] and adsorption onto (i) agricultural solid waste [7] (ii) different bentonites [8] (iii) various types of activated carbon [9] and (iv) surfactant impregnated montmorillonite [10], etc. Ultrafiltration (UF) and nanofiltration (NF) can be used for complete removal of all classes of dye, but care is needed to avoid membrane fouling which decreases the flux. Due to low biodegradability of dyes, a conventional biological wastewater treatment process is not very efficient in treating dye wastewater [11]. Physical or chemical treatment

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processes are generally used to treat it [12]. Adsorption is a common treatment process. Previous investigators have studied the adsorption of dyestuffs using a variety of natural adsorbents, but yet these adsorbents are impractical for the effluent treatment of modern textile industries. Activated carbon can effectively be used to remove color although it is ineffective for dispersed and vat dye [13]. Many investigators have found that depending on the type of adsorbent and adsorbate, the adsorption capacity is greatly influenced by changing the pH of solution. Experimentally it has been found that some dyes change their color with change in pH. For example, crystal violet: purple at pH  $\sim 6$ , green at pH  $\sim 4$  and yellow at pH  $\sim 2$ ; chrysoidine: red in acidic pH and changes its color from red to yellow at strong alkaline pH. This change in color is due to the shifting of absorption band to the longer and shorter wavelengths because of bathochromic and hypsochromic effects, respectively. Therefore, for pH sensitive dyes, it is better to think about a suitable process where pH change is not a process parameter.

In the last decade, increasing interest on the use of aqueous micellar solution has been found in the field of separation science [14,15]. At certain temperature, aqueous solution of a non-ionic surfactant becomes turbid. With further increase of temperature, solution separates in two phases: first, surfactant rich phase, which has small volume compared to the solution and is called coacervate phase and the other is bulk aqueous solution containing surfactant concentration slightly above the critical micelle concentration (CMC) [16]. This temperature is known as cloud point temperature (CPT) of the surfactant. The cloud point is strictly defined at a particular surfactant concentration (e.g., 1 wt%), but because the phase boundary between the two phases is fairly independent of concentration, the cloud point is generally quite close to the lower consolute solution temperature [17]. The solute present in aqueous solution of non-ionic surfactant is distributed between the two phases at the cloud point temperature [18]. This phenomenon is known as cloud point extraction (CPE).

To make the CPE process more economical, it is necessary to recover surfactant from both coacervate phase and aqueous phase. Unlike ionic

surfactant, precipitation method is not applicable for non-ionic surfactant. For volatile solute, it is easy to recover surfactant from coacervate phase by vacuum, steam, or gas stripping [19]. But problem arises for non-volatile solute like dye, which is used in the present case. Although regeneration of surfactant from coacervate phase is not studied here, an experimental investigation has been performed to observe the potential of solvent extraction to recover surfactant from dilute phase. The same process may be applicable to separate surfactant from coacervate phase. Once, the surfactant is transferred to the extracting medium (non aqueous medium: hexane, heptane etc.), it is recovered by evaporating the solvent.

In the present work, CPE has been adopted to remove cationic dye (congo red) from wastewater using TX-100 as non-ionic surfactant. The effects of temperature, concentrations of surfactant, dye and salt on extraction of both dye and TX-100 have been studied. Heptane is used to extract surfactant from dilute phase. The efficiency of solvent extraction for surfactant recovery from dilute phase has also been studied with different surfactant concentration and the volume of extracting medium.

## 2. Cloud point extraction

### 2.1. Micellar system

Surfactant molecules are amphiphilic in nature having a hydrophilic (head) and hydrophobic (tail) part. The hydrophobic part is generally a long chain hydrocarbon or aromatic ring. On the other hand, head part of the surfactant is hydrophilic due to the presence of some electrophilic or nucleophilic groups. In aqueous solution, and at low concentrations, surfactant molecules are found in monomer form. The concentration above which surfactant molecules are dynamically associated ( $\sim 50$ – $150$  of monomer) to form molecular aggregates (micelles) is called critical micellar concentration (CMC). Depending on the nature of surfactant and solvent, the aggregates may form micelles, reverse micelles, micro emulsion, vesicles, monolayers and bilayers [20].

## 2.2. Mechanism of phase separation

An aqueous solution of non-ionic surfactant changes from single isotropic phase to two isotropic phases when the temperature of the solution exceeds the CPT. This change of phase is reversible and on cooling, again it reverts to a single isotropic phase. Actual mechanism of phase separation above CPT is not known. But, several authors proposed some mechanism using the phase separation phenomenon. Nilsson et al. [21] suggested that the dielectric constant of water decreases with increase in temperature, which reduces the interaction between the hydrophilic portion of surfactant and water. Thus above the CPT, dehydration process occurs in the external layer of micelles of non-ionic surfactant [21]. Others have proposed that the phase separation above CPT is due to the micellar attraction. This is because at lower temperature (below CPT), inter micellar repulsive force is predominant which becomes attractive when temperature exceeds the CPT [22]. Some authors have also proposed that the phase separation is due to the increase in micellar size (increasing aggregation number) with temperature [23].

## 2.3. Mechanism of dye solubilization

The insoluble, sparingly soluble or highly soluble solute in water dissolve extensively in/on the micelles of surfactant. The extent of solubilization and the location of solubilization in the micelles of non-ionic surfactant are related to one another, but not clearly understood. Some authors have proposed that, for non-ionic surfactant, the core is surrounded by a mantle of aqueous hydrophilic chains, and solubilization may occur in both the core and the mantle [24]. The relative amount of solubilization in these two regions of non-ionic micelles depends on the polarity of solubilize. Non-ionic surfactants appear relatively more hydrophobic at higher temperatures, due to an equilibrium shift that favors dehydration of the ether oxygens. As the cloud point is approached, the solubilization of non polar solubilizes increases, probably due to an increase in the aggregation number of the micelles. For polar

solubilizes, solubilization decreases owing to dehydration of the polyoxyethylene chains accompanied by coiling more tightly. These observations demonstrate that non polar compounds are solubilized in the core of micelles, while polar solubilizes are located on the mantle. Both of the temperature effects cited here are consistent with variations in the space available for the solubilized molecules in the micelles [24].

## 3. Experimental

### 3.1. Material

The dye used in this study is congo red (CR) (FW: 696.67,  $\lambda_{\max}$ : 499 nm, dye content: 97%, supplied by Aldrich Chemical Company, USA). Triton X-100 (Iso-octyl phenoxy polyethoxy ethanol) containing approximately 10 ethoxy units per molecule (wt per ml at 20 °C is 1.06 g, mol. wt.: 628,  $\lambda_{\max}$ : 226 nm), supplied by Loba chemie, India, has been used as the non-ionic surfactant. The critical micellar concentration (CMC) of TX-100 is  $2.8 \times 10^{-4}$  M (175.8 ppm) at 25 °C [25]. The micellar molecular weight is in the range of 60,000–90,000 and a micelle has a radius of about  $4.3 \times 10^{-9}$  m [25]. Heptane and calcium chloride are procured from Merck (India) and S.D. Fine Chem. Ltd. (India).

### 3.2. Methods

#### 3.2.1. Cloud point extraction (CPE) of dye

In the CPE experiments, solution (50 ml) of TX-100 and congo red has been prepared by dissolving accurately weighed amount of surfactant and dye, respectively, in distilled water at different concentrations. The concentrations of dye in feed are 7, 35, 70, 139, 275 and 555 ppm. The concentrations of TX-100 in feed are 0.02, 0.03, 0.04, 0.05, 0.1, 0.2 and 0.25 M. To observe the effect of salt on CPE of dye and TX-100, the concentration of salt ( $\text{CaCl}_2$ ) has been selected as 0.05, 0.1, 0.2 and 0.5 M.

Each experiment is conducted by using a 50 ml measuring cylinder containing different concentrations of TX-100, dye and salt solution in

a constant temperature bath (supplied by Testing Instruments Manufacturing Company Ltd., India) for 20 min. After complete phase separation, measuring cylinder is removed from the temperature bath and cooled for 2 min. The volumes of coacervate phase and concentration of dilute phase have been measured. All the experiments have been conducted at four different temperatures (70 °C, 75 °C, 80 °C and 85 °C). The concentration of dye and TX-100 has been determined by a spectrophotometer (make: Thermo Spectronic, USA; model: GENESYS 2). Pure TX-100 and CR solution is initially calibrated separately for different concentration in terms of absorbance units, which is recorded at the wavelength of 226 nm and 499 nm, respectively, at which maximum absorption takes place. The standard technique is used to find out the concentrations of both dye and surfactant [26].

### 3.2.2. Solvent extraction (SE) for surfactant recovery

To meet the environmental standards and the economy of the process, surfactant concentration must be reduced from the dye free dilute phase before its discharge. The experimental investigation on separation of TX-100 at 30 °C from aqueous solution has been performed using heptane as extracting medium. All SE experiments are conducted in a cylindrical vessel. The composition of each set of experiment is prepared by adding different volume (2, 5, 10, 15, 20 and 25 ml) of heptane to a fixed volume (30 ml) of TX-100 solution of 243, 479, 2079 and 5157 ppm. Dye concentration is zero, since the concentration of dye in the dilute phase is zero for a wide range of feed dye–TX-100 system. The first two concentrations are in the range of dilute phase of CPE and the remaining concentrations are beyond the limit of dilute phase TX-100 concentration in the present experimental condition. The mixture of TX-100 solution and heptane is vigorously shaken for 20 min using a stirrer (Type-RQ-123, Remi Motors Ltd., India) at 450 rpm to achieve equilibrium. After that, the mixture is transferred into a separating funnel and kept for 3 h for complete separation of two phases. Volume and

concentration of aqueous and non-aqueous phase are measured. The experiments are conducted in batch mode.

## 4. Results and discussion

This section has been divided into two sub-sections. In first section, the effects of various operating conditions on the extent of both dye and surfactant separation have been discussed for CPE. The effect of heptane volume and concentration of TX-100 on the efficiency of SE has been discussed in the second section. For cloud point extraction, efficiency has been defined below,

$$\begin{aligned} &\text{Efficiency of extraction of dye or TX-100} \\ &= \frac{\text{amount of dye or TX-100 in coacervate phase}}{\text{initial amount of dye or TX-100 in feed}} \\ &\quad \times 100 \end{aligned}$$

### 4.1. Cloud point extraction

#### 4.1.1. Effect of surfactant and solute concentration

For successful CPE of dye, it is desirable to use minimum amount of surfactant for maximum extraction of dye. Fig. 1a and b shows the effect of concentration of TX-100 on extraction of dye of different initial concentration at 70 °C and 85 °C, respectively. It has been observed from both the figures that for dye concentration more than 35 ppm, extraction of dye increases sharply when TX-100 concentration increases from 0.02 to 0.1 M. Beyond 0.1 M, increase in extraction efficiency becomes gradual.

It may be observed from Fig. 1a, for dye concentration of 35 ppm, 100% dye extraction is possible with TX-100 concentration of 0.02 M. For 70 ppm of dye concentration, 0.1 M TX-100 may be an optimum concentration to achieve 100% extraction of dye. For higher dye concentrations, e.g., 139, 275 and 555 ppm, with 0.25 M TX-100, about 98.2, 97.2 and 92.3% extraction of dye is achieved, respectively. It has also been observed that for a particular TX-100 concentration,

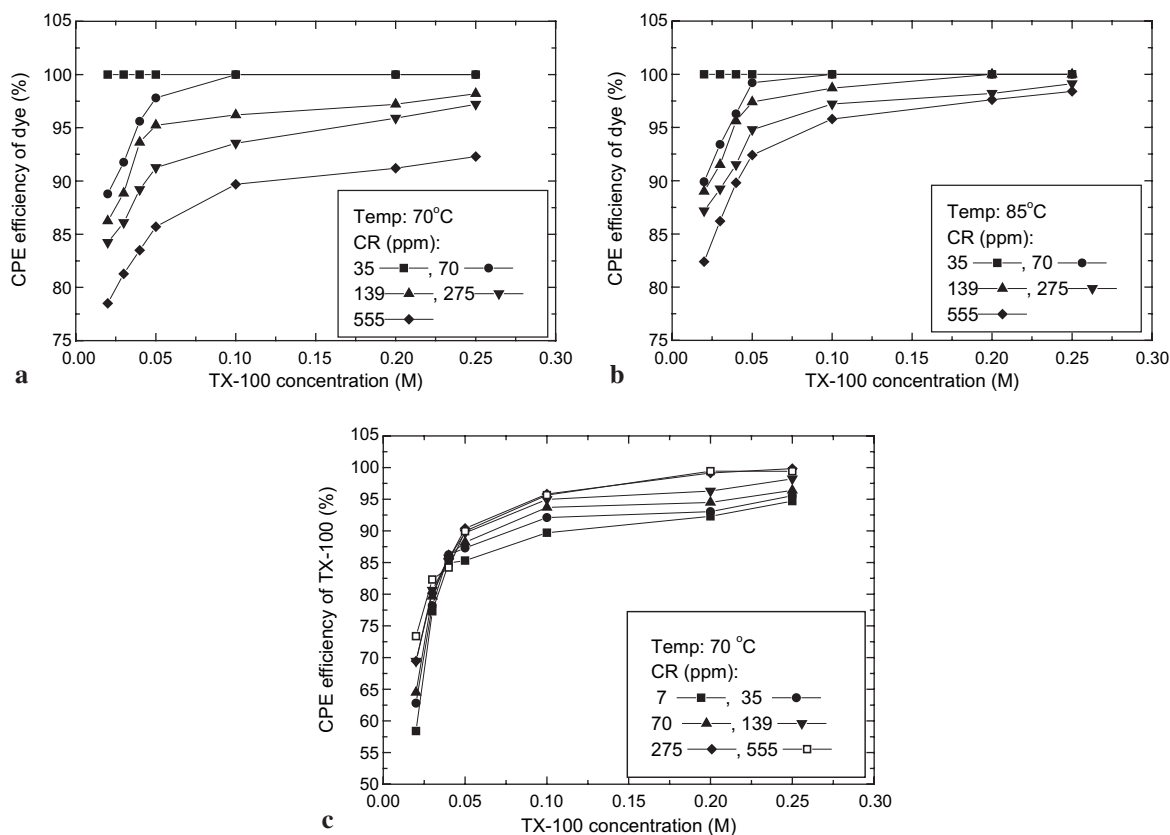


Fig. 1. (a) Effect of concentrations of TX-100 and dye on the efficiency of CPE of dye at 70 °C. (b) Effect of concentrations of TX-100 and dye on the efficiency of CPE of dye at 85 °C. (c) Effect of concentration of TX-100 on the efficiency of CPE of TX-100 at different feed dye concentration at 70 °C.

percentage extraction decreases with the dye concentration. With increase in surfactant concentration, the volume of coacervate phase increases to maintain the material balance, as the concentration of surfactant in dilute phase remains almost constant [16]. This increase in coacervate phase volume renders the higher solubilization of dye, which explains the higher extraction of dye at higher TX-100 concentration at constant temperature and initial dye concentration. Fig. 1b presents the efficiency of dye extraction with surfactant concentration at 85 °C. This figure clearly indicates that the extraction efficiency increases with temperature. For example, extraction efficiency of concentrated dye solution of 555 ppm is about 92.3% at 0.25 M surfactant concentration, at 70 °C (Fig. 1a), whereas it is

about 98.4% at the same concentration level at 85 °C (Fig. 1b). At surfactant concentration of 0.25 M, the extraction efficiency of all the dye concentration used in the present experiments increases from 92.3 to 100% (Fig. 1a) to about 98.4 to 100% (Fig. 1b) when temperature increases from 70 to 85 °C. As discussed earlier, increase in temperature leads to an increase in aggregation number of micelles thereby increasing the extent of solubilization of dyes at higher temperature. It may be noted from Fig. 1a and b that beyond a surfactant concentration of 0.2 M, the increase of extraction efficiency of dye concentration more than 70 ppm is gradual for all the feed dye concentrations used in this study. Therefore, 0.1 M TX-100 may be considered as the optimum surfactant dose for efficient CPE of CR dye up to

70 ppm and that of for dye concentration beyond 70 ppm is 0.2 M TX-100.

Variations of the efficiency of extraction of TX-100 with feed dye and TX-100 concentration have been shown in Fig. 1c at 70 °C. It may be observed from the figure that extraction efficiency of surfactant increases with the initial surfactant concentration for all the dye concentrations. For example, at feed surfactant concentration of 0.25 M, surfactant is extracted in the coacervate phase in the range of 94.7–99.41% for all the dye concentrations. Since, TX-100 concentration in dilute phase remains almost constant (slightly above the CMC) at constant temperature and dye concentration, the extraction efficiency of TX-100 increases with the increase in feed TX-100 concentration. Beyond concentration of 0.1 M, the

increase in extraction efficiency is gradual because of the fact that at higher surfactant concentration ( $>0.1$  M), the surfactant concentration in dilute phase increases with feed TX-100 concentration. At constant surfactant concentration, TX-100 extraction increases with the dye concentration. As more dyes are solubilized, inter micellar repulsion decreases, which increases the micellar size and hence the coacervate phase volume.

#### 4.1.2. Effect of temperature

The effect of temperature on the efficiency of CPE has been shown in Fig. 2a for the initial dye concentrations of 139, 275 and 555 ppm, respectively, using 0.04 and 0.05 M of TX-100. It is clear from the figures that the extraction of dye increases with temperature and feed TX-100

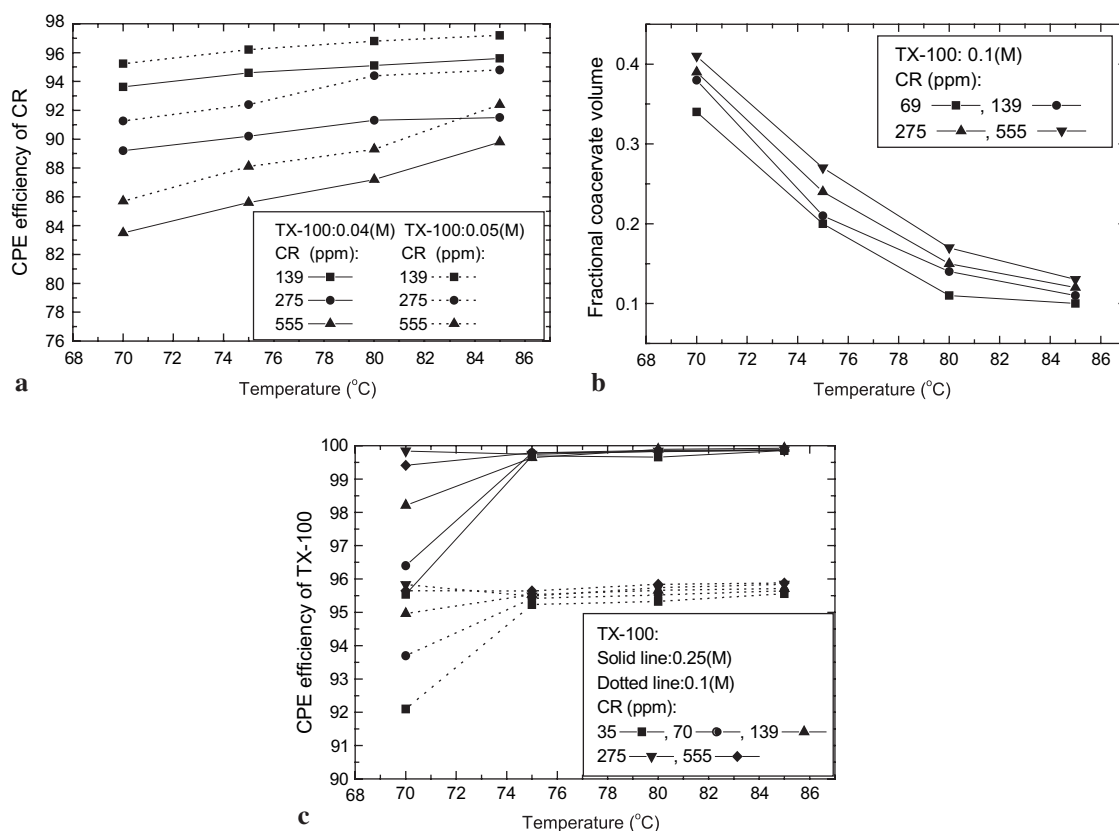


Fig. 2. (a) Effect of temperature on the efficiency of CPE for 139, 275 and 555 ppm of CR at TX-100 concentration of 0.04 and 0.05 M. (b) Effect of temperature on the fractional coacervate phase volume at different feed dye concentration and at TX-100 concentration of 0.1 M. (c) Effect of temperature on CPE efficiency of TX-100 at different feed dye and TX-100 concentration.



concentration. It may be observed that the extraction of dye (for 139 ppm of feed dye and 0.04 M of TX-100) increases from 93.62 to 95.6% when the temperature increases from 70 to 85 °C. Molecules of congo red is ionic, but due to the presence of three benzene rings it shows an amphiphilic nature. The increase in extraction efficiency or solubilization of dyes in the micelles with temperature is may be due to the fact that the polarity of CR molecule decreases with increase in temperature and shows preferentially hydrophobic nature at higher temperature. Therefore, CR molecules get solubilized in the micelles. At higher temperature, this is augmented by the increase in aggregation number as discussed earlier leading to an increase in the extraction efficiency or solubilization. Similar trend is observed for higher feed concentration of dye but extraction efficiency decreases with feed dye concentration.

Fig. 2b shows the variation of fractional coacervate phase volume with temperature at different feed dye concentration and at 0.1 M of TX-100. It is evident from Fig. 2b that the volume of coacervate phase decreases with temperature. For example, at 139 ppm of dye, fractional volume of coacervate phase decreases from 0.34 to 0.10 when temperature is raised from 70 to 85 °C. As discussed earlier, at elevated temperature, the interaction among the TX-100 micelles increases leading to dehydration from the external layers of micelles resulting in a decrease in volume of coacervate phase. It is also evident from Fig. 2b that the volume of coacervate phase increases with dye concentration at constant temperature. For example, at 70 °C temperature, fractional volume of coacervate phase increases from 0.34 to 0.41 when dye concentration increases from 139 to 555 ppm. This is because as dye concentration increases, more dyes will be solubilized leading to an increase in fractional coacervate phase volume.

The variation of the extent of extraction of the surfactant with temperature is presented in Fig. 2c for different feed dye concentration. It may be observed from the figure that the extraction efficiency of the surfactant increases initially with temperature and feed dye concentration. For example, at 139 ppm dye concentration, the efficiency of extraction of surfactant increases

from 95.54 to 99.79% when temperature is raised from 70 to 75 °C at 0.25 M of TX-100. On the other hand, at 70 °C extraction efficiency increases with dye concentration as discussed earlier. Beyond 75 °C, the efficiency of TX-100 extraction is almost independent of temperature and dye concentration. The increasing extraction efficiency at higher temperature is due to the more micellar attraction as discussed in Section 2.2. The same trend has been observed for TX-100 extraction with temperature and dye concentration at 0.1 M of surfactant. It may be observed from the figure that the extraction efficiency is more for higher feed TX-100 concentration. Since, all the feed TX-100 concentrations are much above the CMC and dilute phase remains slightly above the CMC, the ratio of the surfactant concentration of dilute phase to that of the feed decreases with increase in feed TX-100 concentration and hence extraction efficiency decreases with feed surfactant concentration.

#### 4.1.3. Effect of salt concentration

Fig. 3a and b shows the variation of extraction efficiency with  $\text{CaCl}_2$  concentration at TX-100 concentration of 0.03 M and at 85 °C for initial dye concentration of 400 and 600 ppm, respectively. It may be observed from both the figures that the extraction efficiency of both dye and TX-100 increases with  $\text{CaCl}_2$  concentration. From Fig. 3a it is found that the extraction of both dye and TX-100 increases sharply (from 89.45 to 93.99% for dye and 80.14 to 85.78% for TX-100) up to 0.2 M of  $\text{CaCl}_2$ , but beyond that, the increase in efficiency becomes gradual. The size and number of micelles in coacervate phase increases with salt concentration as discussed in Section 2.3. Therefore, the amount of solubilized dye increases with  $\text{CaCl}_2$  concentration. Hence, the percentage extraction of both dye and surfactant in the coacervate phase increases with salt concentration. A similar trend is observed in Fig. 3b for higher dye concentration.

#### 4.2. Solvent extraction

The performance of solvent extraction has been observed in terms of the percentage recovery of

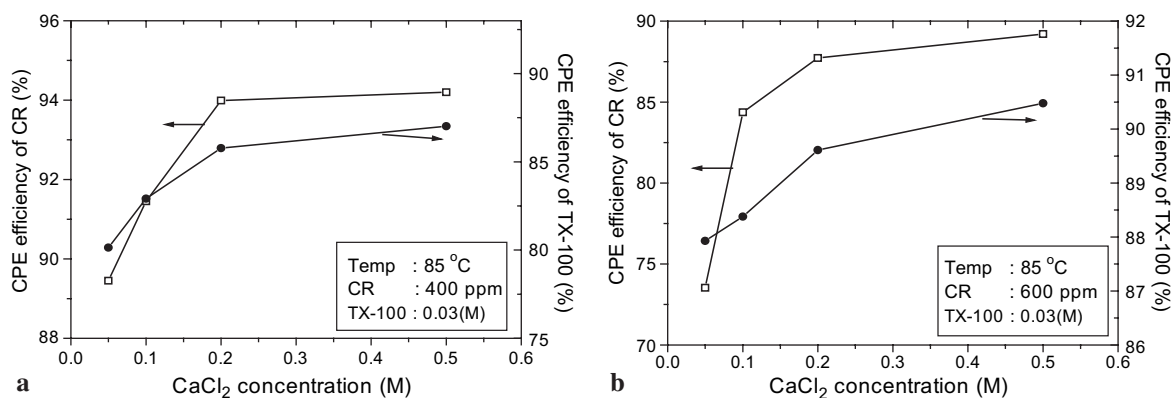


Fig. 3. (a) Effect of  $\text{CaCl}_2$  concentration on the efficiency of CPE for 400 ppm of feed dye at  $0.03\text{ M}$  of TX-100. (b) Effect of  $\text{CaCl}_2$  concentration on the efficiency of CPE for 600 ppm of feed dye at  $0.03\text{ M}$  of TX-100.

TX-100 with different volume of extracting medium at four different initial TX-100 concentrations and is presented in Fig. 4. It is clear from the figure that for a fixed TX-100 concentration (say  $243\text{ ppm}$ ), the surfactant recovery increases sharply from  $42.29$  to  $81.74\%$ , when the volume of heptane increases from  $2$  to  $15\text{ ml}$ . With further increase of heptane volume to  $25\text{ ml}$ , the surfactant recovery reaches up to  $90\%$ . The increase in recovery with the solvent volume is due to more distribution of TX-100 molecule in the heptane phase. The concentration distribution of the surfactant in both the solvent and dilute phase is constant at a fixed temperature (room temperature:  $32 \pm 2^\circ\text{C}$ ). Therefore, keeping the solvent

volume fixed, increase in feed surfactant concentration results in lower recovery of surfactant. On the other hand, at same surfactant concentration, extraction efficiency increases with solvent volume.

## 5. Conclusion

Cloud point extraction of congo red can be successfully used to remove color from wastewater using TX-100 as a non-ionic surfactant. The effects of temperature, concentrations of  $\text{CaCl}_2$ , TX-100 and congo red on the extraction efficiency have been studied in detail. It is observed that the extraction efficiency increases with temperature, TX-100 and  $\text{CaCl}_2$  concentration. From the experimental results, it is observed that for dye concentration up to  $35\text{ ppm}$ ,  $100\%$  congo red dye removal is possible using  $0.1\text{ M}$  TX-100 at  $70^\circ\text{C}$ . For higher dye concentration up to  $555\text{ ppm}$ ,  $97.5$ – $100\%$  dye removal can be achieved using  $0.2\text{ M}$  of TX-100 and  $0.2\text{ M}$  of salt ( $\text{CaCl}_2$ ) concentration at  $85^\circ\text{C}$ . To make the CPE process more economical, solvent extraction technique has been adopted using heptane as an organic solvent. From the dilute phase,  $90\%$  surfactant can be recovered using a volume ratio of heptane to dilute phase of  $1:1.2$ . For higher surfactant concentration (which simulates surfactant concentration in coacervate phase),  $65$ – $87\%$  surfactant recovery is possible for the above volume of the solvent.

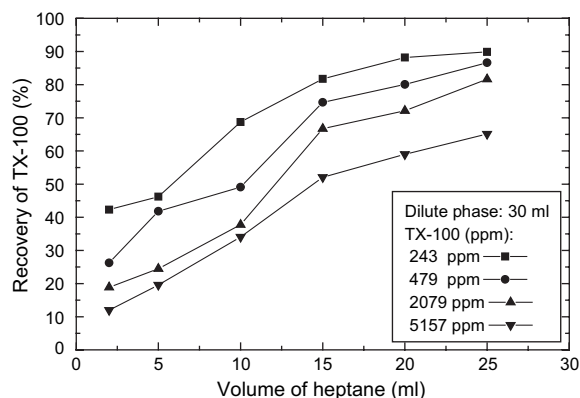


Fig. 4. Effect of heptane volume on the recovery of TX-100 at different feed TX-100 concentration.



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