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Efficient removal and separation of anionic dyes from aqueous medium by the application of reverse micelles of cationic surfactants

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ABSTRACT

A new protocol based on liquid/liquid extraction using reverse micelles is proposed. The recovery of solvent and reuse of dye and surfactant, after extracting the dye molecules from the core of reverse micelles of surfactant is related to the economic viability of the process. Experiments were conducted by mixing a known quantity of dye in aqueous phase and solvent-containing surfactants in a simple mixer. The separation of solvent phase, containing encapsulated dye in reverse micelles, from aqueous phase due to gravity results in separation of dye from water. The removal of different anionic dyes (methyl orange, congo red and aniline blue) from aqueous phase in amyl alcohol solvent using different cationic surfactants was studied. The percentage removal of dye depends upon the size of the reverse micelle of the surfactant. The solvent used for the dye removal can be recovered by distillation method and can be reused. The dye is separated from reverse micelles by lowering the temperature below the Kraft point by using the solvent depending upon the solubility of the two. Hence all the components of the experiment can be recovered and reused.

Keywords: Cationic surfactants; Solvent extraction; Dye removal; Reverse micelles; Anionic dyes

1. Introduction

Industrialization is an important part of the economy of every country. However, if not managed well, it plays a negative role in polluting the environment. Effluent water from various industries like carpet manufacturing, dyeing leather, distilleries, textile, pulp and paper industries, contain various types of dyes that should be removed before discharging it to the environment to avoid health hazards and destruction of the ecosystem. If released to the waste stream, without proper treatment, they could exert a great impact on the environment. Dyes released in the effluent can be natural as well as synthetic and fall into many types having a variety of properties, e.g. acidic, basic, disperse, mordant, direct, vat dyes, azo, diazo, anthroquinone based and metal complex dyes. There are more than 100,000 commercially available dyes with over 7×10^5 tonnes of dyestuff produced annually worldwide. Depending on their chemical complexity, structure and synthetic origin, dyes are

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resistant to fading on exposure to light, water, many chemicals and microbial attack [1,2]. They are very stable and many decompose only at temperature higher than 200˚C. Effluent containing dyes is responsible for water-borne diseases exhibiting symptoms such as haemorrhage, nausea, dermatitis, ulceration of the skin and mucous membranes, kidney damage and a loss of bone marrow leading to anaemia [3]. Thus, their presence in wastewater is unwanted and it is desirable to treat/remove the dyes from the effluents before their discharge to the environment. Several studies have been performed to find an effective and economical way of treating dye-containing wastewater. The three main categories of treatment are physical, chemical and biological methods. But the conventional biological treatments are less effective for wastewater containing azo dyes [4]. In the open literature, various techniques have been proposed to remove dyes from water, such as oxidation [5,6], adsorption [7–10], flocculation-precipitation [11], membrane separation [12], electro-coagulation [13–15], electrochemical destruction [16], photo catalysis [17,18] and ultrasound [19,20]. All the above methods have been compared but each displays advantages as well as disadvantages [21,22].

Oxidation to remove residual colorant is frequently practised. The oxidizing agents commonly used are chlorine or hydrogen peroxide and the latter is usually used together with iron (II) sulphate (Fenton's reagent), ozone or UV irradiation. Among the oxidation methods, UV/ozone or UV/H_2O_2 is one of the best technologies for the total removal of dye from wastewater [23,24]. These methods are only effective for those cases where low concentrations of organic matter are present in water. Heikkilal et al. [25] degraded some azo dyes, which were difficult to be degraded biologically, by decolorizing using lignin peroxidase isoenzyme. In biological processes, it was found that the chemical oxygen demand (COD) and the colour of dye wastewater could not be efficiently reduced/ removed. Bell et al. [26] treated and decolorized dye-wastewater using an anaerobic baffled reactor. In addition, physico-chemical processes, for example chemical coagulation, active-carbon adsorption and ozone oxidation studies showed that the decrease of COD and colour for dye wastewater was not efficient. These above-mentioned processes could not completely treat the dye wastewater. Flying ash was investigated [27] for its ability to adsorb dyes from aqueous solution. Batch pH, kinetics and isotherm studies were performed on a laboratory scale with synthetic dyes. Four concentrations of solutions with different compositions and particles size were treated with fly ash (granular activated carbon). The dye removal by using membrane technology has also been studied [28–31]. Membrane processes are very promising advanced treatment methods for colour removal as well as for reducing the volume of wastewater generated while recovering and recycling valuable components from the waste stream [12]. However, this method is expensive. Recently agricultural wastes are treated and used as low cost adsorbents for the wastewater treatment [32,33].

Due to the high cost and other disadvantages of the above methods, a new method for dye removal has been tried. This method is based on the solvent extraction method using reverse micelles of surfactants [34–36]. In this method, the dye is extracted into the reverse micelle of the surfactant soluble in a solvent by electrostatic attraction and is tightly held there. Reverse micelles are nanometer size aggregates of surfactant molecules surrounding a microscopic water core in apolar solvents. These inverted aggregates are drawn together by hydrogen bonding in the presence of minimal amounts of water and they are thermodynamically stable. The tendency of many water soluble solutes to partition into the aqueous inner core of reverse micelles present in the organic phase has spawned a great deal of interest in using such systems as continuous extractants for proteins [37–40], amino acids [41,42] and enzymes [43].

Keeping in view, the past work on and perception of solvent extraction method using reverse micelles of surfactants, the removal of anionic dyes was attempted with the laboratory synthesised cationic surfactants. The novelty of this method is that the surfactant and the dye used can be regenerated and reused.

2. Experimental section

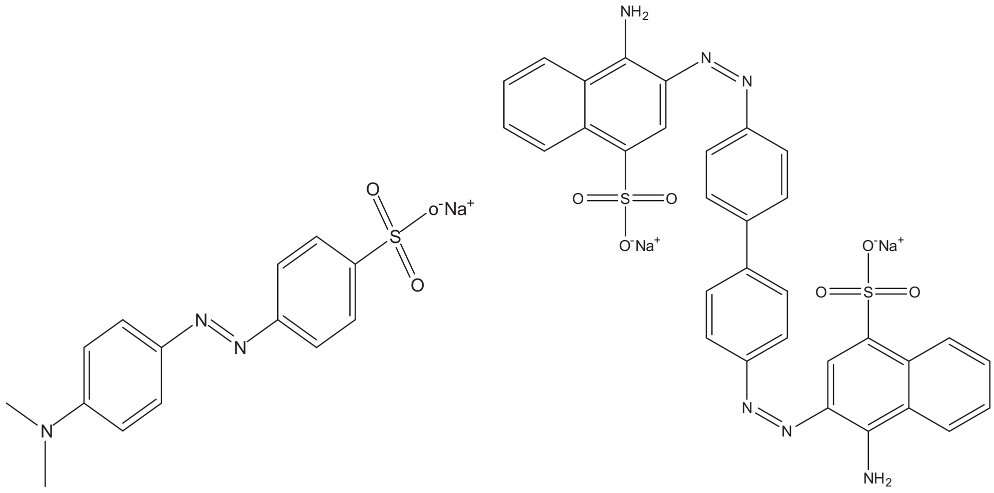
2.1. Materials

The surfactants used for the removal of the dyes from wastewater were synthesised in the laboratory [44] entitled as 1-(decyl oxy carbonyl methyl)-3-methyl imidazolium bromide, 1-(dodecyl oxy carbonyl methyl)-3-methyl imidazolium bromide, 1-(tetradecyl oxy carbonyl methyl)-3-methyl imidazolium bromide, 1-(decyl oxy carbonyl methyl)-3-methyl imidazolium chloride, 1-(dodecyl oxy carbonyl methyl)-3-methyl imidazolium chloride, 1-(tetradecyl oxy carbonyl methyl)-3-methyl imidazolium chloride. The physical properties have been given in Table 1.

The dyes used in testing the removal process are methyl orange (MO), congo red (CR) and aniline blue (AB). The solvent used is amyl alcohol (3-methyl butan-1-ol). The chemical formula and molecular weight of these dyes are given in Fig. 1.

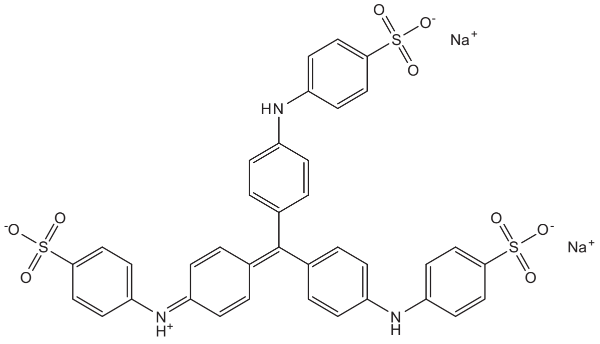
Table 1
Physical properties of laboratory synthesised cationic surfactants including critical micelle concentration (CMC)

S.No	Name	Formula	MW	CMC (mM)
1.	1-(tetradecyl oxy carbonyl methyl)-3-methyl imidazolium bromide **(C_{14}IB)**	$C_{20}H_{37}BrN_2O_2$	417.42	0.038
2.	1-(dodecyl oxy carbonyl methyl)-3-methyl imidazolium bromide **(C_{12}IB)**	$C_{18}H_{33}BrN_2O_2$	389.00	0.210
3.	1-(decyl oxy carbonyl methyl)-3-methyl imidazolium bromide **(C_{10}IB)**	$C_{16}H_{29}BrN_2O_2$	361.00	0.700
4.	1-(tetradecyl oxy carbonyl methyl)-3-methyl imidazolium chloride **(C_{14}IC)**	$C_{20}H_{37}ClN_2O_2$	373.00	0.041
5.	1-(dodecyl oxy carbonyl methyl)-3-methyl imidazolium chloride **(C_{12}IC)**	$C_{18}H_{33}ClN_2O_2$	344.50	0.310
6.	1-(decyl oxy carbonyl methyl)-3-methyl imidazolium chloride **(C_{10}IC)**	$C_{16}H_{29}ClN_2O_2$	316.50	0.790



Methyl Orange (MW = 327.00)
$C_{14}H_{14}N_3O_3SNa$

Congo Red (MW = 696.66)
$C_{32}H_{22}N_6Na_2O_6S_2$



Aniline Blue (MW = 799.80)
$C_{37}H_{27}N_3Na_2O_9S_3$

Fig. 1. The molecular structure, molecular formula and molecular weight of the three dyes.

2.2. Experimental set-up

A simple magnetic stirrer (Remi, India) was used for the mixing of the solvent and the aqueous phase. The speed of the stirrer can be measured accurately (rpm). A separating funnel was used to separate the solvent and aqueous phases by gravity. An UV spectrophotometer (Shimadzu, UV 1,800) was used to measure the removal of the dye from water.

To succeed as a viable alternative to the conventional extraction process, a solvent is used in the reverse micellar extraction technique, which is easily and cost effectively separated from solvent/aqueous phase dispersion in the presence of surfactant. The reverse micelles are formed in the solvent phase. Preliminary study showed that among butanol, ethyl acetate, amyl alcohol and octanol, amyl alcohol has been found to be the best solvent for the removal of dye from water using the reverse micelles technique. Amyl alcohol is sparingly soluble in water at 298 K and at atmospheric pressure. The dispersion of amyl alcohol in water is easy to separate into pure phases in a separating funnel by gravity. A 110 ml mixture of amyl alcohol/water dispersion separates in 2–5 h into pure phases within a range of the surfactant concentration.

2.3. Methods

2.3.1. Removal of the dye

Different concentrations of the dye were prepared in hundred milliliters of the aqueous solution. A known quantity of cationic surfactant above the cmc was added to 10 ml of amyl alcohol. The aqueous phase and amyl alcohol were mixed thoroughly using the magnetic stirrer for 5 min at 298 K. The whole solution was transferred to a separating funnel to separate the solvent and aqueous phases by gravity. The denser aqueous phase was collected at the bottom of the separating funnel, whereas the less dense amyl alcohol was collected at the top. The dye sample collected from aqueous phase was analysed in a UV spectrophotometer to determine the amount of dye extracted. The dye concentration in amyl alcohol was determined to check the mass balance. The experiments were repeated to check the accuracy and the data were found to be accurate within ± 5% error.

Calculation

$$\%\ \text{Removal} = \frac{D_i - D_{aq}}{Di} \times 100 \tag{1}$$

where D_i, initial concentration of dye taken; D_{aq}, concentration of dye left in the aqueous media after the two layers separates

2.3.2. Selective removal of the dye

For the selective removal of a given dye, the extraction of dyes from mixed dye solution by the solvent extraction method was tried. 100 ml of mixed solution of MO (0.1 mM, 1.63 mg/50 ml) was treated with (3 × cmc) of C_{10}IB and CR (0.1 mM, 3.54 mg/50 ml) with (3 × cmc) of C_{12}IB.

2.3.3. Recovery of solvent, surfactant and dye

The solvent amyl alcohol contains reverse micelles of surfactant encapsulating dye. The water obtained was free from the dye. The solvent amyl alcohol was distilled off and can be utilized again for the experiment. After distillation, the solid was left which contained the surfactant with the dye. This solid mass was dissolved in acetone and the temperature was lowered below 273 K. Depending upon the solubility both separate and are collected.

3. Results and discussion

3.1. Removal of dye

In the experiments the solvent: water ratio used was 1:10 instead of 1:2 as in the literature [29]. The solvent used has been reduced so that the method can be made more efficient and less costly. Transmission electron microscope (TEM) analyses were carried out to determine the size of reverse micelles and the shape of the clusters formed.

The concentrations of the dyes have been varied from 0.001 to 0.1 mM. For the lower concentrations, the dye removal was complete. Therefore, the results for removal of the dyes at 0.1 mM concentration have been discussed in this section. Data for removal of MO (0.1 mM) at surfactant concentration 3 and 15× cmc are reported in Fig. 2 and for CR (0.1 mM) has been reported in Fig. 3 where as the removal of AB (0.1 mM) was very poor by using reverse micelles of C_{10}IX and C_{12}IX below 10% hence not shown in the figure.

The data in Fig. 2 reveal that, among the range of surfactants tested, the surfactant C_{10}IX (X is Cl, Br) led to maximum removal of MO. Fig. 3 reveals the maximum removal of CR with application of the surfactant C_{12}IX. This may be due to the compatibility of the size of reverse micelle and the dye molecule. It was found that the surfactants with bromides as counter ions show better percentage removal than surfactants with chlorides as counter ions. This is likely due to the reason that bromides are more hydrophobic and facilitate the surfactant to form reverse micelles faster in comparison to chlorides. Hence the further studies were carried out with surfactants having bromides as counter ions. The results indicated that the dye removal process by reverse micelle and the solvent extraction method is selective. To further confirm

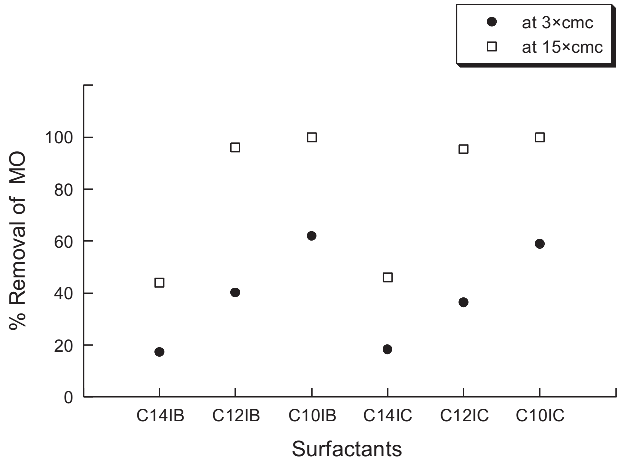


Fig. 2. Percentage removal of MO (0.1 mM) by the six synthesised cationic surfactants used at 3 × cmc and 15 × cmc.

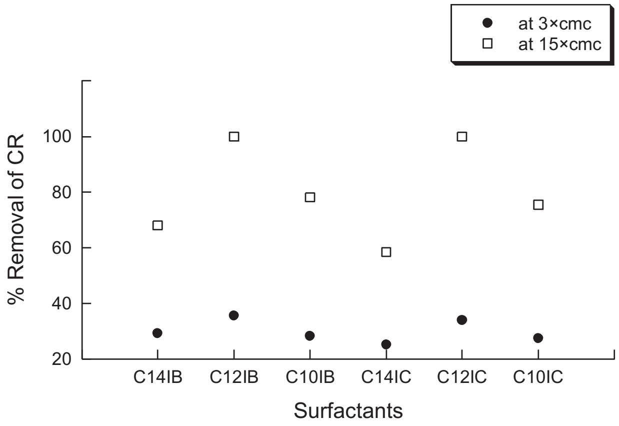


Fig. 3. Percentage removal of CR (0.1 mM) by the six synthesised cationic surfactants used at 3 × cmc and 15 × cmc.

the effect of size of reverse micelle on specific dye removal, studies were carried out by treating a particular surfactant with a mixture of dyes.

3.2. Selective removal of the dye

It was found that from the mixture, the surfactant selectively encapsulated the respective dye i.e. C_{12}IB selectively encapsulated CR, showing more removal of CR (37%) as compared to MO (34%) from the mixture, where as C_{10}IB selectively extracted MO (60%) from the mixture than CR (20%). This helped in concluding that the size of the reverse micelle plays an important role in the dye removal. TEM analysis has been carried out to know the size of the reverse micelles. Figs. 4(A)–6(A) show TEMs of clusters of surfactants encapsulating dye. Figs. 4(B)–6(B) show the sizes of the reverse micelles in nm.

3.2.1. TEM analysis

Among these three dyes MO has the smallest size. Fig. 4(B) shows the size of the C_{10}IB reverse micelle is small. The size, however, increases with an increase in chain length of the surfactant, as can be seen in Fig. 5 (B) and Fig. 6(B). Thus C_{10}IB was found to be selective in removing MO while C_{12}IB is selective in removing CR. The percentage removal of AB is very poor by both C_{10}IB and C_{12}IB as the size of reverse micelles of these surfactants are very small as compared to the size of dye. Thus, the application of C_{14}IB for the removal of AB was tested. Further the studies were carried out for the removal of the dyes over a range

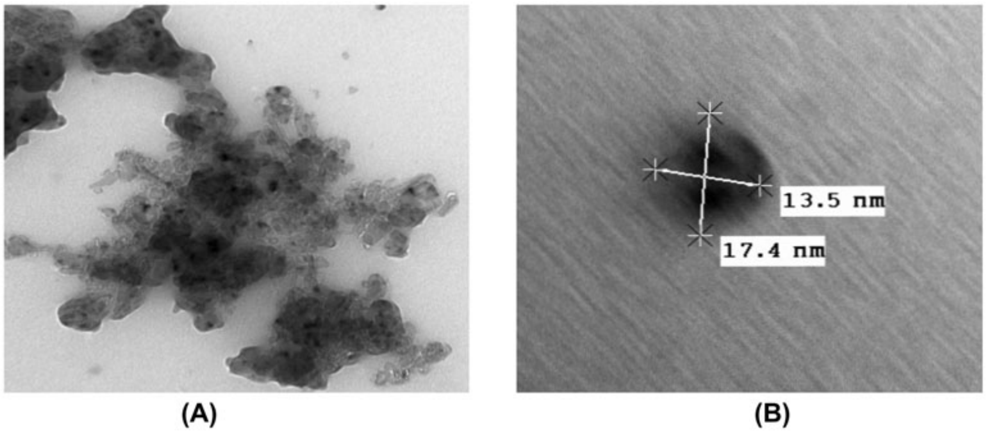


Fig. 4. (A) TEM showing the clusters of reverse micelles of C_{10}IB containing MO inside it. (B) TEM shows the reverse micelle of C_{10}IB encapsulating MO.

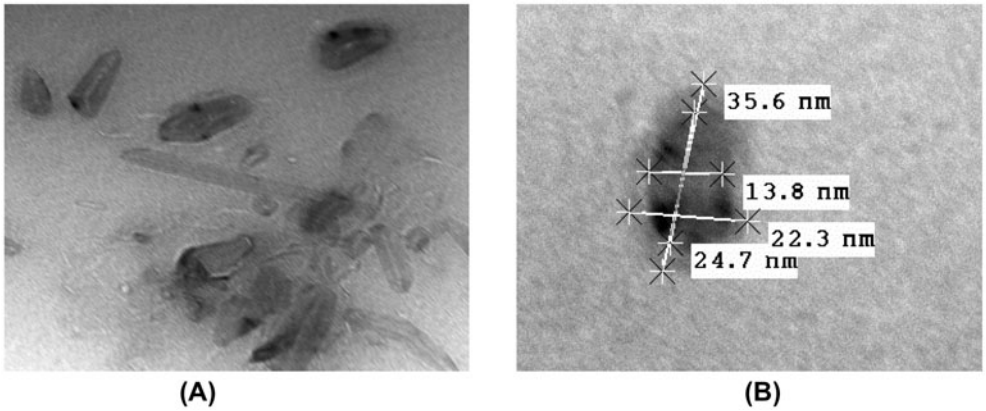


Fig. 5. (A) TEM showing the clusters of reverse micelles of $C_{12}IB$ containing CR inside it. (B) TEM shows the reverse micelle of $C_{12}IB$ encapsulating CR.

of concentrations with respective surfactants concentrations equivalent to 5, 10 and 15× cmc. The results for the removal of MO are depicted in Fig. 7. The data for CR are reported in Table 2.

As shown in Fig. 7 there is decrease in the percentage dye removal when the concentration of the dye is increased from 0.1 to 0.4 mM. Further it can be seen in Fig. 7 that with doubling the surfactant concentration there is an increase in the percentage dye removal even at high concentration of the dye in the original solution i.e. if the concentration of surfactant is doubled from 5 to 10× cmc, the removal of 0.4 mM MO increases from 69.52 to 98% and becomes 100% at 15× cmc. This also reveals the interaction of dye molecule with surfactant. It can also be seen that the percentage removal of dye decreases with increasing the concentration of the dye and at higher concentrations of the surfactants the higher percentage removal can be achieved. The dye absorption in the UV measurement is out of range so a dilution factor was considered.

3.3. Effect of surfactant concentration

To study the behaviour as the function of the concentration of the surfactant, the concentration of the dye was fixed and concentration of the surfactant was varied from 1.5 to 15× of cmc. The data for MO (0.4 mM) are reported in Fig. 8, for CR (0.1 mM) in Fig. 9 and for AB in Fig. 10.

Fig. 8 shows that percentage removal of the dye MO increases with increasing the surfactant concentration.

Fig. 9 represents the removal of CR (0.1 mM). It can be seen that the removal goes on increasing with increasing the concentration of surfactant. The maximum removal has been found to be at 13 × cmc while maximum removal of AB is at a surfactant

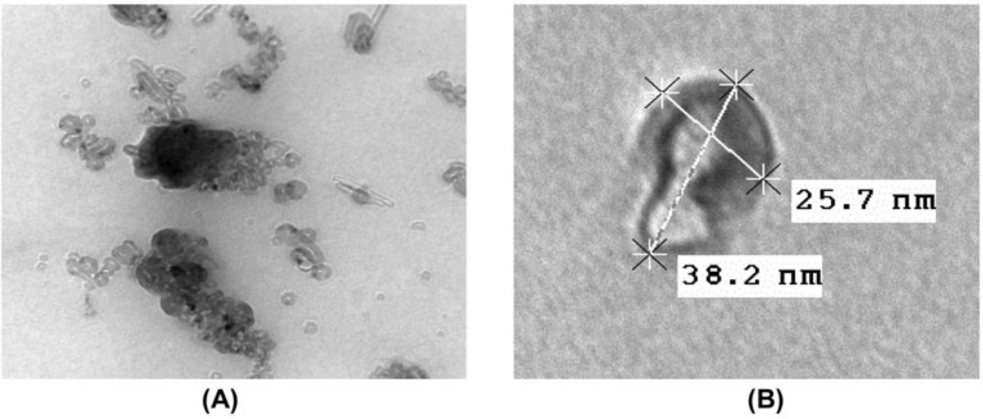


Fig. 6. (A) TEM showing the clusters of reverse micelles of $C_{14}IB$ containing AB inside it. (B) TEM shows the size of the reverse micelle of $C_{14}IB$ encapsulating AB.

Table 2
Data for different concentrations of CR red with two concentrations of surfactant

Concentration of (C_{12}IB)	(10 × cmc)			(15 × cmc)	
Concentration of CR	0.1 mM	0.15 mM	0.2 mM	0.2 mM	0.3 mM
% Removal	70.00	45.33	38.00	71.92	39.00

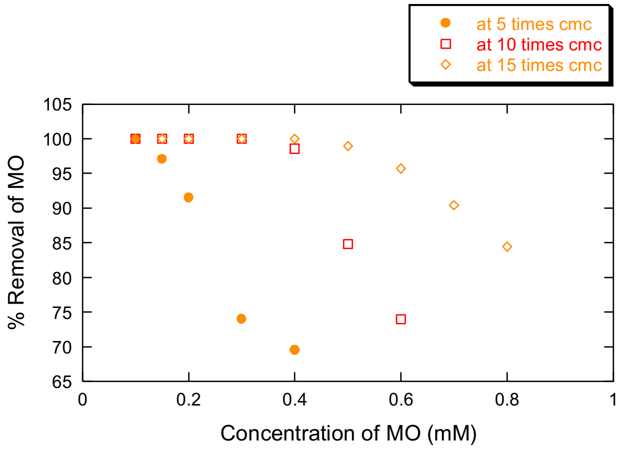


Fig. 7. represents percentage removal of different concentrations of MO ranging from 0.1 to 0.8 mM at 5, 10 & 15× cmc of C_{10}IB.

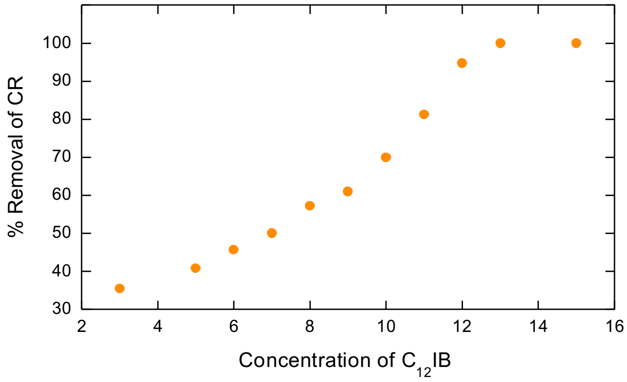


Fig. 9. Percentage removal of CR (0.1 mM) by different concentrations of C_{12}IB (mg).

concentration 15× of cmc. The graph is plotted as percentage removal of AB as a function of concentration of surfactant (Fig. 10).

The above data shows that with the increase in the surfactant concentration, the percentage removal of the dye increases. This is because as the amount of surfactant increases, the number of reverse micelles increases linearly. This increases the number of dye molecules that can be encapsulated. This has been explained by mass action kinetics [45].

For a pseudo reaction of dye molecule D with a number n of reverse micelle M.

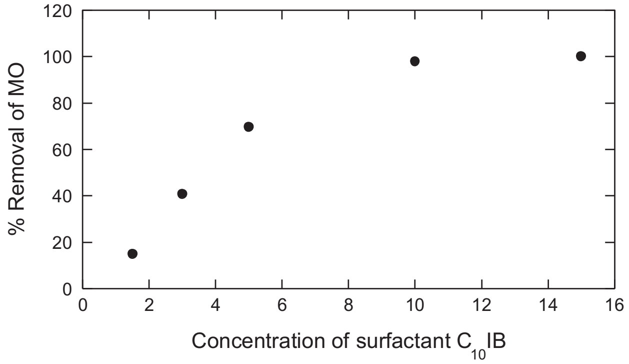


Fig. 8. Removal of MO (0.4 mM) by the C_{10}IB when using a range of 1.5–15× the cmc (mg).

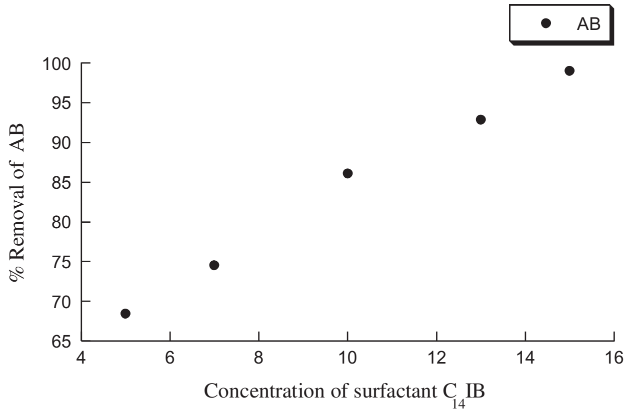


Fig. 10. Percentage removal of AB (0.1 mM) by C_{14}IB at different concentrations in terms of cmc (mg).

$$D + n[M] - DM + [(n-1)M] \tag{2}$$

where D = dye molecule, M = reverse micelle, DM = reverse micelle with entrapped dye molecule.

Reverse micelle concentration. M is related to the surfactant concentration $[S]$ as:

$$[M] = \frac{[S]}{N_{ag}} \tag{3}$$

where (N_{ag}) is the aggregation number, which is assumed to be independent of the surfactant concentration [35]. So the increase in the surfactant concentration leads to an increase in the number of reverse micelles, consequently more of the dye molecules get encapsulated, increasing the percentage removal of dye.

3.4. Recovery of solvent, surfactant and dye

Micelles are formed at the particular temperature called Krafft point temperature. The separation of dye from the reverse micelles depends upon Krafft point temperature and difference in the solubility of the dye and the surfactant in the chosen solvent, acetone. As the temperature was lowered below the Krafft temperature, the reverse micelles began to break-up. Depending upon the solubility, the dye goes into the solvent acetone and as the surfactant is insoluble in acetone, it is left behind and hence gets separated. By distillation, the solvent and the dye can be obtained and used further. To maximize the yield of the surfactant(range being 50–70%), the temperature has to be monitored closely to the required Krafft temperature.

4. Conclusions

In the present study, we have derived a new protocol for the removal of dye from water. They are defined as follows

- It is concluded that the use of reverse micelles of cationic surfactants in a solvent extraction method is more efficient than the usual direct adsorption method [46–47].
- It was found that the surfactants were selective in dye removal depending upon the size of their reverse micelles in relation to the molecular size of the dye.
- It is found that bromo analogues are more efficient than chloro analogues as the cmc of bromo analogues is lower than chloro analogues due to greater hydrophobic nature of bromide ions.
- The dye removal increases with the increase in concentration of surfactant because with the increasing amount of surfactant the number of reverse micelle is assumed to increase linearly which in turn increases the number of dye molecules that can be encapsulated.
- This method is novel as the surfactant used can be separated and thus the separated dye, surfactant and the solvent can be reused.
- Though the use of solvents is employed, which naturally require enclosure and recovery, this method is fundamentally eco friendly to nature. However to consider the full economic model and the important final carbon footprint, the energy usage in distillation and final cooling would need to be compared with the direct adsorption technique.

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