



Removal of Acid Yellow 36 and Rhodamine B from single and binary systems by reverse micelles

Anindita Saha^{1*}, Prabhat Pandit¹

¹Department of Chemical Engineering, Malaviya National Institute of Technology, Jaipur 302017, India

Abstract: In the present study reverse micelles are employed to encapsulate ionic dyes such as, azo dye Acid Yellow 36 (AY36) also known as Metanil Yellow and cationic dye Rhodamine B (RhB). This method proved to be highly efficient in the removal of high concentrations of dyes as much as 40mg/100ml from aqueous solutions of single and binary systems. The effect of different parameters like initial dye concentration, surfactant concentration, pH and salt (KCl) concentration on the percentage removal of the dyes were studied. It was observed that the percentage removal for both anionic and cationic dye decreased with the increase in the initial dye concentration and increased with the increase in the surfactant concentration in case of both the dyes in single and binary systems. Further, it was noticed that for anionic dye the percentage removal increased with the increase of both pH and KCl concentration while the results were exactly the reverse in case of RhB dye which exists in both cationic and zwitterionic forms at pH above 4. In binary systems, it was found that the removal of RhB increased compared to its removal from single system, while the removal of AY36 decreased in binary system.

Keywords : azo dye, Rhodamine B, dye removal, binary system, zwitterionic, reverse micelle.

1. Introduction

The dyes which are released from different industries like textile, paint, leather, cosmetics, paper and pulp contribute as one of the major pollutants of fresh water and their presence even at very low concentration can affect the aesthetic conditions of the water bodies.¹ The non-biodegradable dyes apart from affecting the aquatic organisms by depleting the dissolved oxygen of water bodies also seriously harm the terrestrial animals upon its consumption and other uses due to their toxicity and carcinogenicity^{2,3}.

A typical azo dye Metanil Yellow or Acid Yellow 36 is also used in textile industries for colouring protein fibres⁴ like wool, silk, etc. Apart from its use in textile industries, Acid Yellow 36 is known to have been used as soap colorants, in shoe polish, laundry, cleaning agents, pigment manufacturing units and also in

different food such as sweet meat, juices and ice-creams, though its use in food industry is strictly prohibited because of its carcinogenic effects^{5,6}. Rhodamine B which can exist in both cationic and zwitterionic forms at different pH^{7,8} is widely used in printing and textile industries as a colorant for acrylic fibres^{4,6}.

Various types of methods have been employed in the removal of synthetic dyes from industrial effluents before being discharged into the surrounding environment. Methods such as adsorption, coagulation/flocculation, electro-Fenton process, catalytic oxidation, ultrafiltration, ozonation, biological activation, microbial fuel cells⁹⁻¹² have been reported to efficiently remove dyes from single and binary aqueous solutions. Adsorption of basic dyes using activated carbon is one of the most common methods in practice as reported in many studies¹³. Adsorption of dyes onto various agricultural products and by-products or agricultural wastes like tree fern¹⁴, bark, rice husk, cotton waste^{15,16}, sugarcane dust¹⁷ have also been studied in literature for their capacity to remove different basic and acid dyes.

In recent years removal of high concentration of acid and basic dyes using reverse micelle has proved to be a cost effective treatment¹⁸⁻²⁰. And the basic advantage of this method is the dye and the solvent can be recovered thus making the process environment friendly²¹. Reverse micelles are nano-sized aggregates which are dispersed in an organic solvent phase with ionic surfactant molecules surrounding the water core²². The hydrophilic head of the surfactant points towards the water core and the hydrophobic tail points outside to the organic phase thus forming a structure which is reverse to normal micelles. These thermodynamically stable reverse micelles encapsulate the dye molecules inside the water core, thus removing the dye from water¹⁹. The organic and the aqueous phase are separated by the action of gravity. Reverse micelle are extensively used in the biotechnological field for the extraction of proteins and different other biomolecules without degrading their conformation²²⁻²⁵. In this work, cationic surfactant Hexadecyltrimethylammoniumbromide (HTAB) and anionic surfactant Sodium dodecyl benzenesulphonate were used to form the reverse micelles in the organic solvent amyl alcohol for the removal of anionic Acid Yellow 36 and cationic Rhodamine B respectively. The solvent to the aqueous phase volume ratio was maintained to 1:2¹⁹ as studied in the literature where reverse micelles were used to remove dyes like Methyl orange and Methylene blue. In this study an attempt was made to investigate the simultaneous removal of both anionic and cationic dyes from binary aqueous solution by reverse micellar system.

2. Experimental

2.1 Materials

Azo dye Acid Yellow 36 and cationic dye Rhodamine B were used in this study to prepare the dyeing wastewater. The molecular structure and UV-visible absorption spectra of AY36 and RhB is given in Figure 1 and 2. Cationic surfactant (HTAB) and anionic surfactant (SDBS) were used to form the reverse micelles. Table 1 gives the physical properties of the cationic surfactant HTAB and anionic surfactant SDBS. Amyl alcohol was used as the organic solvent and 0.1N HCl and NaOH were used for adjusting the pH of the aqueous phase. KCl was used to study the effect of salt concentration on dye removal. All the chemicals were of analytical grade and were acquired from Loba Chemie, Mumbai, India.

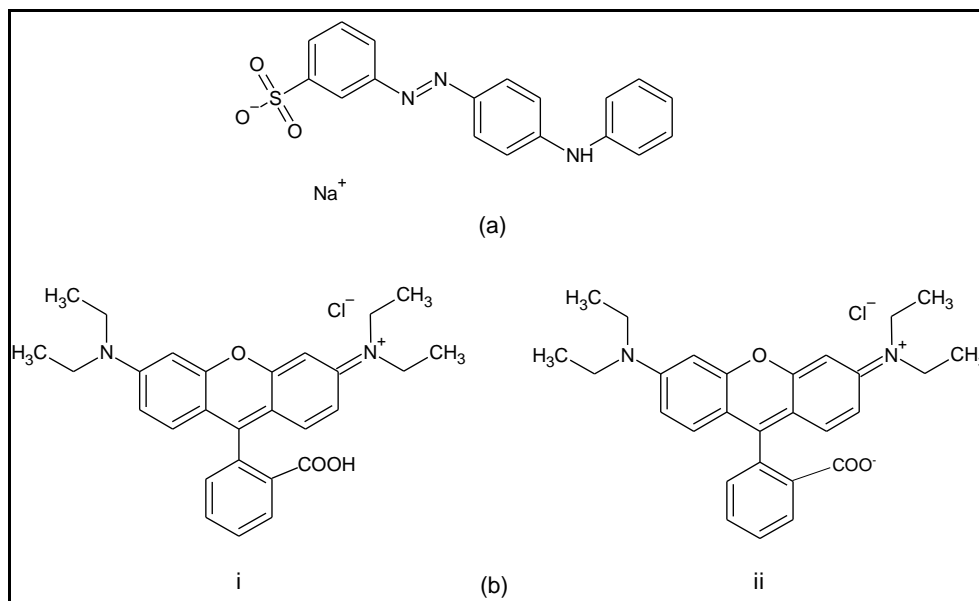


Figure 1 Chemical Structure of (a) Acid Yellow 36; (b) Rhodamine B, (i) Cationic (ii) Zwitterionic

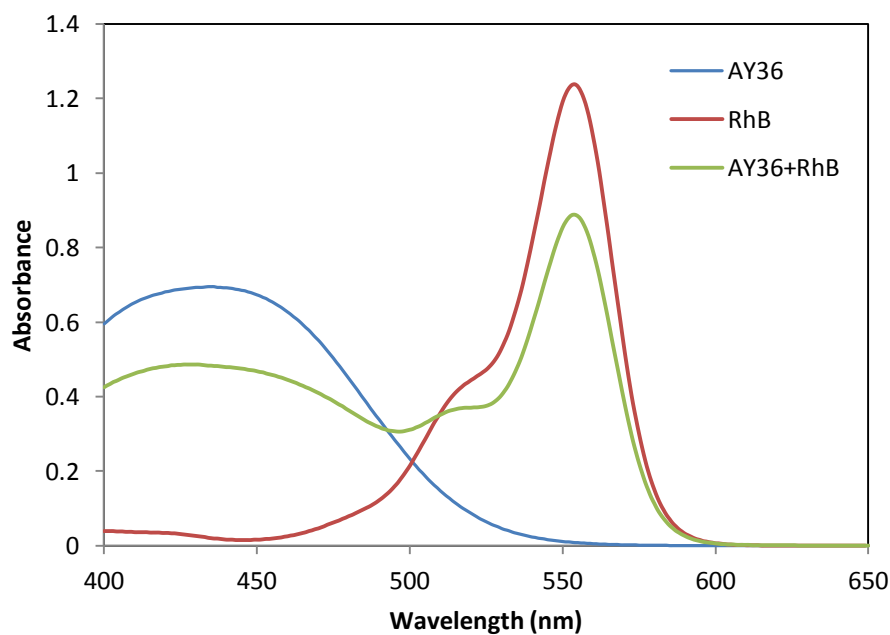


Figure 2 UV-Visible spectra of AY36 and RhB in single and binary aqueous solution

Table 1 Physical Properties of Surfactants

Sr. no	Name	Chemical Formula	Type	Molecular Weight	CMC (ppm)
1	Hexadecyltrimethyl ammonium bromide	C ₁₉ H ₄₂ BrN	Cationic	364.6	350
2	Sodium dodecyl benzene sulphonate	C ₁₈ H ₂₉ NaO ₃ S	Anionic	348.5	500

2.2 Experimental set-up

A simple magnetic stirrer (Remi, India) was used for the mixing of the aqueous phase containing the dye and the solvent phase containing the surfactant. The speed of the stirrer was kept at 350rpm. Separating

funnel was used to separate the aqueous and solvent phases by gravity. An UV spectrophotometer (Shimadzu, UV 1900) was used to measure the absorbance of each of these samples after extraction at 434nm for AY36 and 545nm for RhB. The unknown concentration of dye samples were measured from the standard calibration plot prepared.

For the binary dye mixture the extraction was done in two sequential steps. First to the binary mixture, solvent containing the cationic surfactant was added in order to remove the anionic dye first and then the leftover aqueous phase containing the cationic dye was mixed with solvent containing the anionic surfactant was added to it and then it was again transferred to a separating funnel where the two phases get separated by the action of gravity. A schematic set-up for the treatment of a binary dye solution by reverse micellar extraction is represented in Figure 3.

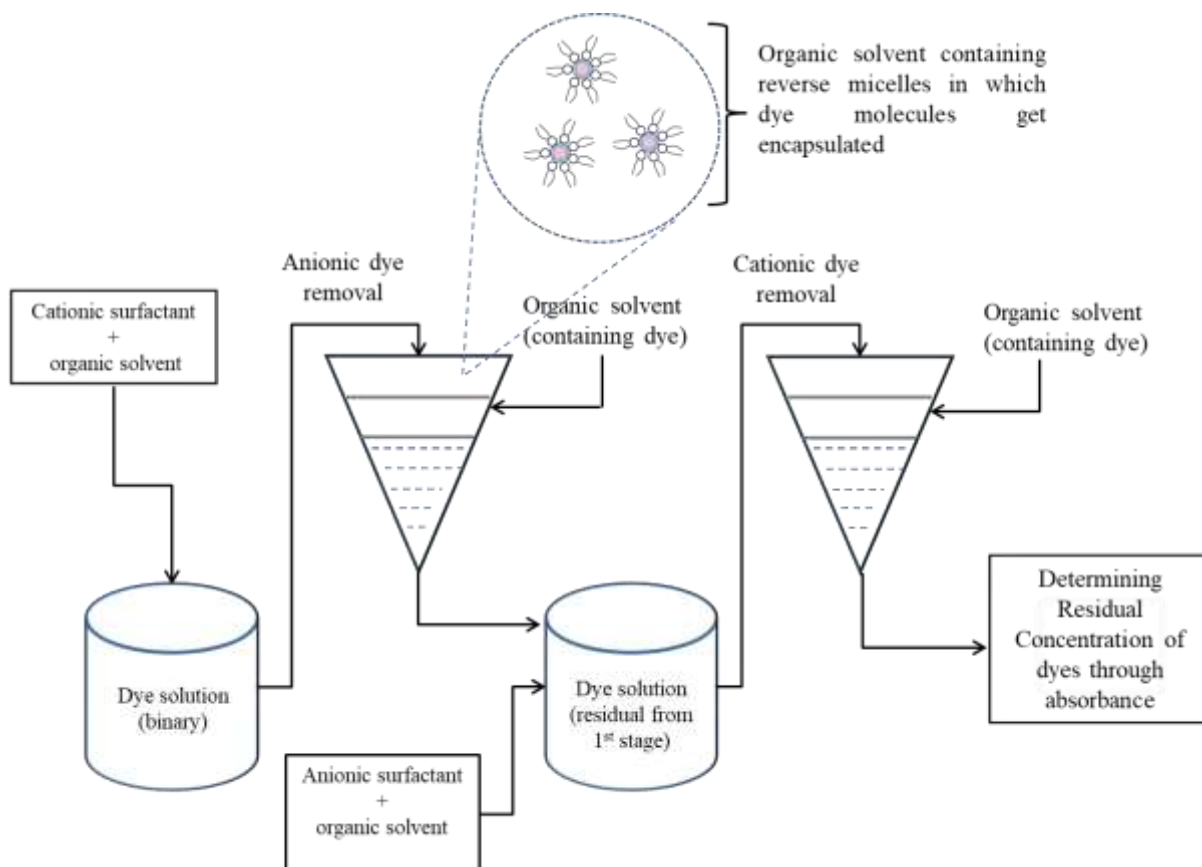


Figure 3 Schematic diagram of the experimental set-up for removal of dyes from binary system

2.3 Method

2.3.1 Dye sample preparation

A stock solution of 1000 mg l^{-1} for each dye was initially prepared. In order to obtain the dye solutions of required concentration successive dilutions were done to the stock solution using distilled water. In case of binary mixture a known concentration of each dye were mixed with each other in the required mass ratio. Reverse micellar extraction of AY36 and RhB in single and binary dye systems were carried out in a batch mode at room temperature.

2.3.2 Reverse micellar extraction for single dye system

For single dye system initial concentration of the dye was varied from 10-50mg/100ml by diluting the stock solution and the pH was varied from 4 to 10 and the KCl concentration was also varied between 2g to 8g/100 ml of the aqueous phase. The 100 ml of aqueous solution with known concentration of dye was added to 50 ml of the solvent containing a known quantity of HTAB or SDBS. The two phases were then mixed

thoroughly at a fixed rpm until a homogeneous mixture was formed. The solution mixture was then transferred to a separating funnel where the denser aqueous phase collected at the bottom while the lighter amyl alcohol phase containing the reverse micelles in which the dye molecules were encapsulated were collected at the top under the action of gravity. After separation the aqueous phase was collected from bottom and analysed in a UV spectrophotometer to determine the residual concentrations of dye. The amount of percentage removal of dyes was calculated using Eq. (1)

$$\% \text{ Removal} = \frac{C_i - C_f}{C_i} \times 100 \quad (1)$$

Where C_i , the initial concentration of dye; C_f , the final concentration of dye left in aqueous phase after the extraction.

2.3.3 Reverse micellar extraction for binary dye system

For binary dye systems, 100 ml of dye solutions were prepared with 50 ml of each dye in aqueous phase. The mass ratio of AY36 to RhB was kept at 1:1, 1:2 and 2:1 at a certain initial concentration of 20 mg/100 ml aqueous phase. The pH of the aqueous phase was maintained at 6 and the KCl concentration was kept at 4g/100 ml of aqueous phase. The concentration of HTAB and SDBS added to the dye solution was 30 mg/50 ml amyl alcohol and 40 mg/50 ml amyl alcohol respectively. The extraction was carried out sequentially in two steps, where in the first step the binary dye mixture was first added to 25 ml amyl alcohol containing a known concentration of HTAB and mixed thoroughly at a fixed rpm. This whole solution was then transferred to a separating funnel where the two phases get separated. Then in the second step the remaining aqueous phase was added to 25 ml of amyl alcohol containing a known concentration of SDBS and mixed well. This whole mixture is again separated using a separating funnel. The absorbance was measured at their respective wavelengths. The dye concentrations in aqueous phase were determined by Eqs (2) and (3).^{26,27}

$$C_{AY36} = \frac{K_{B_2}A_1 - K_{B_1}A_2}{K_{A_1}K_{B_2} - K_{A_2}K_{B_1}} \quad (2)$$

$$C_{RhB} = \frac{K_{A_1}A_2 - K_{A_2}A_1}{K_{A_1}K_{B_2} - K_{A_2}K_{B_1}} \quad (3)$$

Where K_{A_1} , K_{B_1} , K_{A_2} and K_{B_2} are the calibration constants for dyes AY36 and RhB at wavelengths $\lambda_{1,max}$, 434nm and $\lambda_{2,max}$, 554nm respectively. A_1 and A_2 are the absorbance at wavelengths $\lambda_{1,max}$ and $\lambda_{2,max}$ respectively.

3. Results and Discussions

3.1 Effect of Surfactant and initial dye concentration

The solubilisation of dye in the reverse micelles depends on both the surfactant and initial dye concentration. It can be seen from Figure 4, that with the increase in the AY36 concentration from 10 to 50mg/100ml of water, the percentage removal decreased from 96.93 to 92.20% with a HTAB concentration of 30mg/50ml amyl alcohol at pH 6 and 4g/100 ml aqueous phase of KCl. But at a feed concentration of AY36 of 40mg/100 ml aqueous phase, with the increase in the HTAB concentration from 15 to 30 mg/50 ml amyl alcohol, the percentage removal increased from 88.73 to 93.13%.

A similar trend can be observed in the case of RhB removal using SDBS as the surfactant from Figure 5. The percentage removal decreased from 99.03 to 96.56 when the initial RhB concentration increased from 10 to 50mg/100 ml aqueous phase at SDBS concentration of 40mg/50ml amyl alcohol. The percentage removal increased from 92.81 to 97.27 for 40 mg RhB/100 ml aqueous phase with the increase in SDBS concentration from 25 to 40 mg SDBS/50 ml amyl alcohol.

Increasing the surfactant concentration increases the number of reverse micelles^{20,28} and this leads to the uptake of more water and hence more number of dye molecules get encapsulated. But it can be observed from Figure 4 and 5 that increasing HTAB concentration above 30mg/50ml amyl alcohol and SDBS concentration above 40mg/50ml amyl alcohol did not increase the percentage removal of dye considerably. Since a constant

volume of solvent can accommodate only a fixed number of reverse micelles thus no more reverse micelles are formed after increasing the surfactant concentration above a certain level.

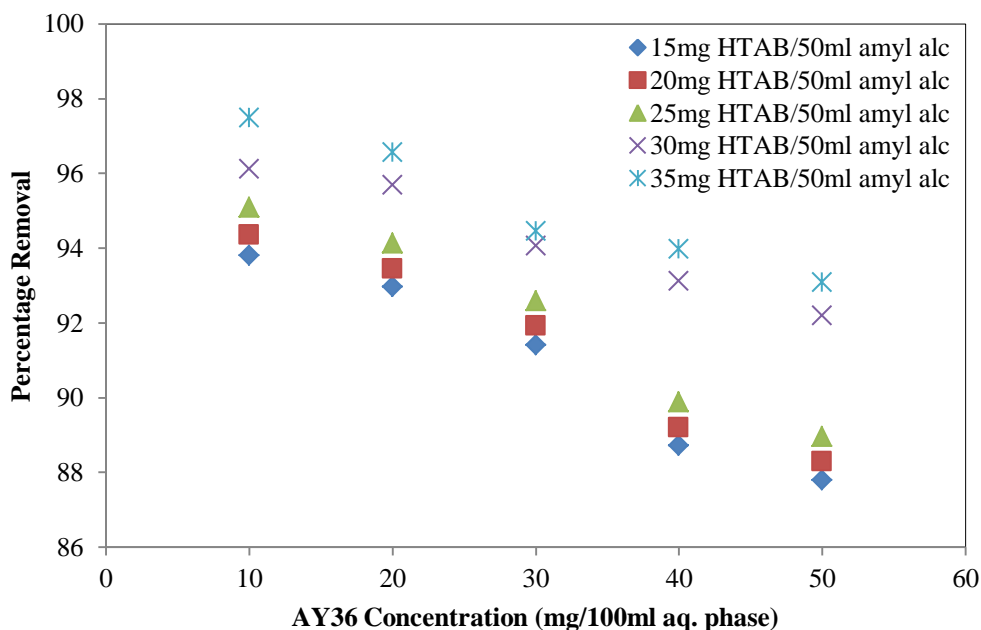


Figure 4 Effect of initial dye and surfactant concentration on the removal of AY36 from aqueous solution

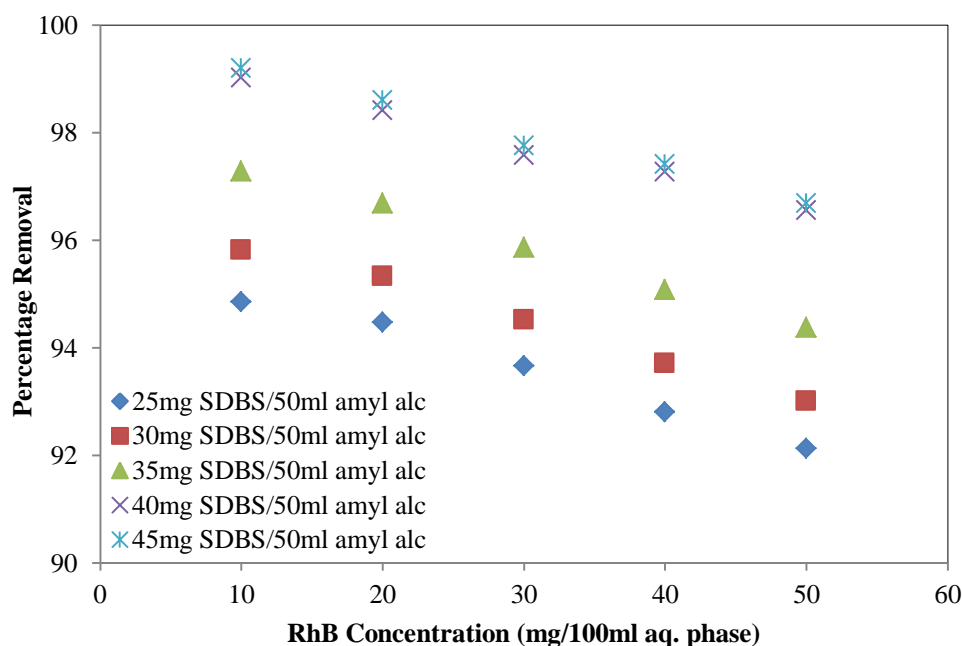


Figure 5 Effect of initial dye and surfactant concentration on the removal of RhB from aqueous solution

3.2 Effect of pH

From the results shown in Figure 6 and 7 it can be seen that the percentage removal of AY36 increased with the increase in pH, while the reverse trend is observed for RhB.

The decrease in the percentage removal of AY36 in acidic condition can be explained with Figure 8 which depicts the structural change in AY36 molecule. The AY36 molecule becomes less anionic in nature with decrease in pH and thus less electrostatic interaction prevails in between headgroup of HTAB and the dye molecule and thus the percentage removal decreases.

For RhB, the cationic group N^+ gets neutralized with OH^- at high pH. And thus removal of cationic RhB decrease with an increase in pH as the electrostatic interaction between the dye molecule and negatively charged headgroup of SDBS decreases.

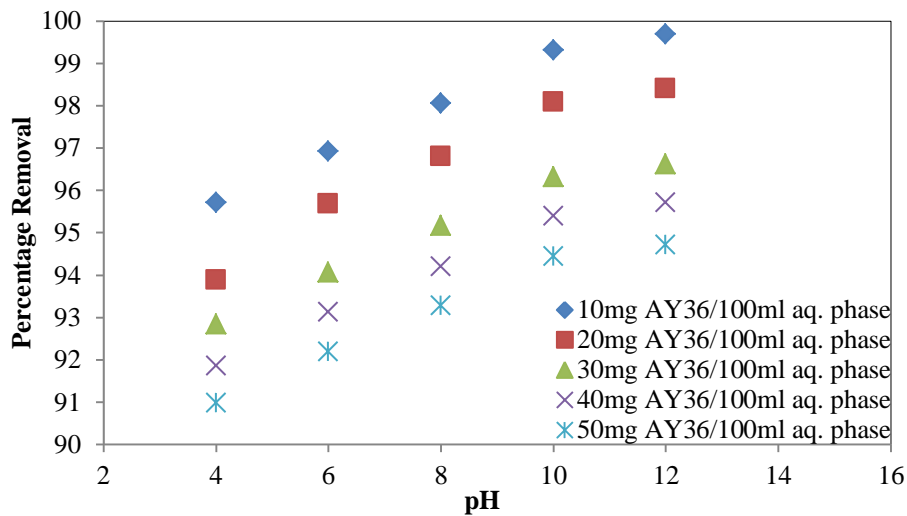


Figure 6 Effect of pH on the removal of AY36 from aqueous solution

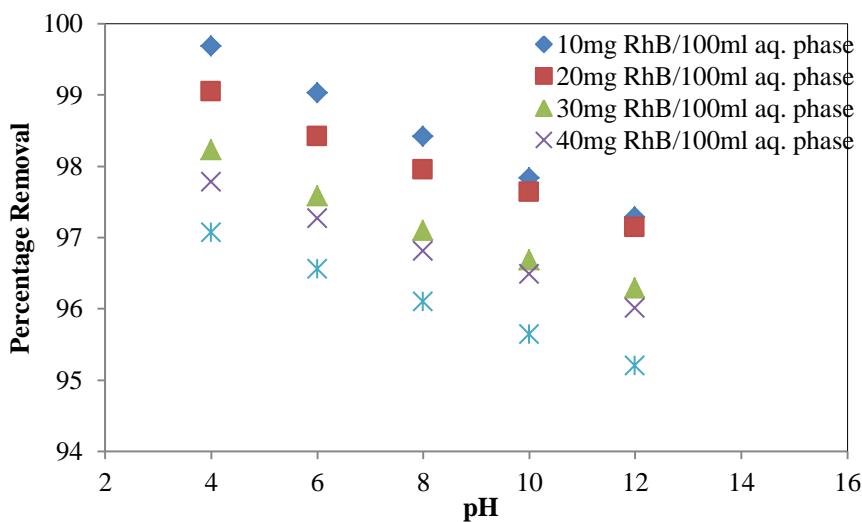


Figure 7 Effect of pH on the removal of RhB from aqueous solution

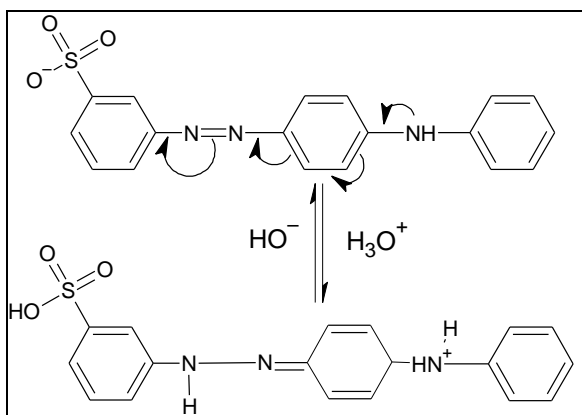


Figure 8 Structural changes in AY36 in acidic and basic conditions

3.3 Effect of salt concentration

The textile dye effluents contain considerable amounts of salts^{29,30} and thus determining their effect in percentage removal of dye is important. The salt concentration of the aqueous phase influences the percentage removal of AY36 and RhB to a great extent. The effect of salt concentration was investigated by varying the KCl concentration in aqueous phase at pH 6 from 2 to 8g/100ml aqueous phase. It can be seen from Figure 9 and 10 that the percentage removal of AY36 increased steadily with the increase in KCl concentration and the percentage removal of RhB decreased with the increase in KCl concentration.

In case of AY36 the percentage removal increased when the concentration of KCl increased above 2g/100 ml of aqueous phase. This is due to the reduction of CMC (critical micelle concentration) which leads to the early formation of reverse micelles and hence higher percentage removal of AY36 was achieved with lesser concentration of HTAB.

In presence of K^+ and Cl^- , a double layer is formed which shields the electrostatic interaction between the negatively charged headgroup and RhB molecule^{20,31}. Due to the formation of double layer the repulsion between head groups takes place and this leads to the reduction of the reverse micelles' size and thus the water content inside the reverse micelles is reduced which is known as desalting effect³² and this results in the reduced dye encapsulation by reverse micelles and hence the percentage removal of RhB was decreased.

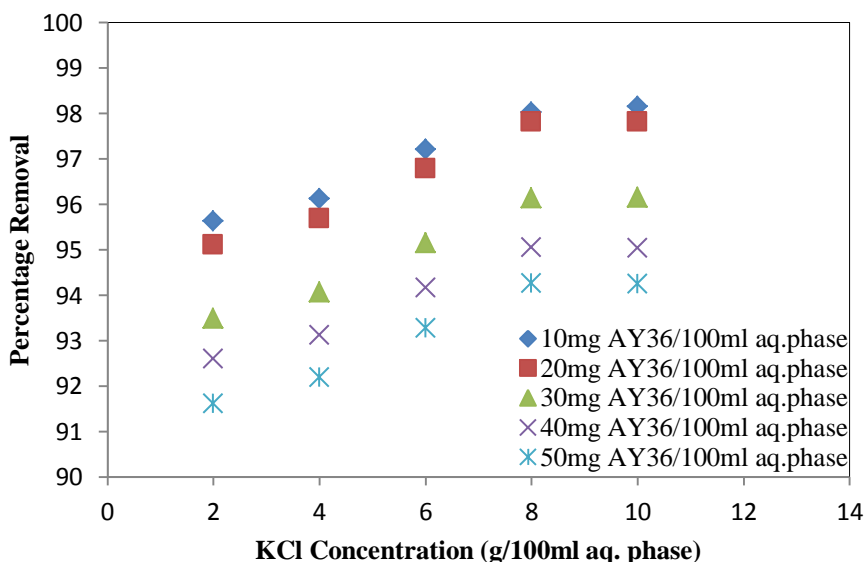


Figure 9 Effect of KCl concentration on removal of AY36 from aqueous solution

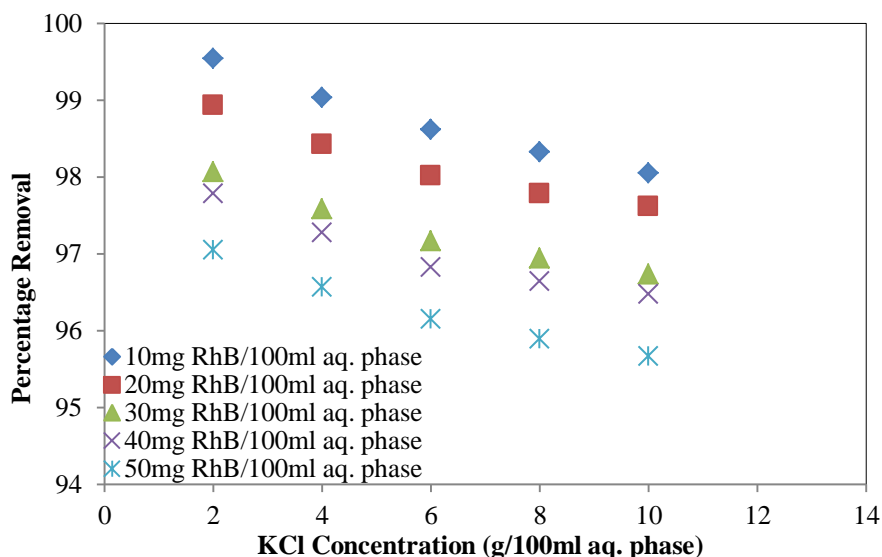


Figure 10 Effect of KCl concentration on removal of RhB from aqueous solution

3.4 Comparison in the removal of AY36 and RhB from single and binary system

The mass ratios of AY36 to RhB was maintained at 1:1, 1:2 and 2:1 with 1 dye concentration in single system as 20mg/100ml and 40 mg/100 ml aqueous phase and KCl concentration was kept at 4g/100 ml aqueous phase.

From Figure 11, it could be observed that the percentage removal of both the dyes from single system is slightly different than their removal from binary system. For AY36, it could be seen that the removal percentage of AY36 from single system of dye concentration 20 and 40 mg/100 ml aqueous phase is almost same as its removal percentage from binary systems of 1:1, 1:2 and 2:1 at pH 4.0. But the percentage removal of AY36 from binary system gradually decreased as compared to its removal from single system above pH 4.0.

However in case of RhB, a reverse trend could be observed. At pH 4.0 though the percentage removal of RhB from both single and binary system remained same, but the removal of RhB increased from binary system than in single system.

RhB can exist in two molecular forms, cationic and zwitterionic above pH 4,^{33,34} as shown in Figure 11. Thus pH of a solution has a significant role on the charge state of RhB. At pH 4 or lower, RhB is solely in cationic state and thus it was removed only by anionic surfactant SDBS. At higher pH the carboxylic group of RhB gets ionized and thus more was the removal of RhB because it got encapsulated in both cationic and anionic reverse micelles HTAB and SDBS respectively.

Due to this competitive interaction of AY36 and RhB dyes with cationic surfactant the solubilisation of AY36 in the HTAB reverse micelles decreased and hence the removal of AY36 was comparatively less in binary system in presence of RhB than in single system.

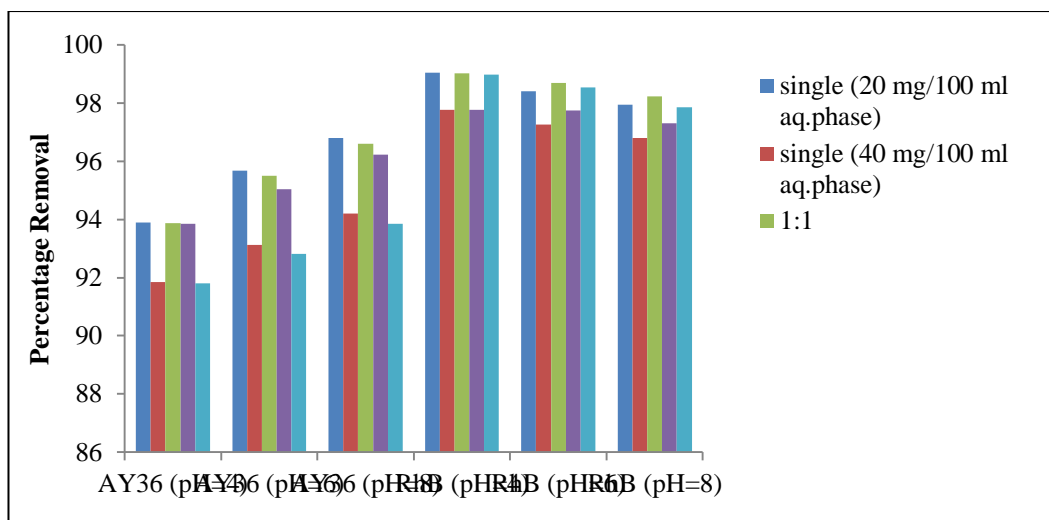


Figure 11 Dye removal efficiencies of AY36 and RhB in binary dye solutions (mass ratios of AY36 to RhB are 1:1, 1:2 and 2:1)

4. Conclusion

The study proved to be efficient in the removal of ionic dyes like AY36 and RhB from both single and binary aqueous solutions. The dyes AY36 and RhB were solubilized into the reverse micelles due to the electrostatic interaction between the dye molecules and the charged layer inside the reverse micellar core. The study also indicated that along with surfactant and initial dye concentration, pH and salt concentration had significant effects on the removal of the dyes.

The results indicated that the percentage removal of AY36 decreased in binary system in presence of RhB to some extent while the removal of RhB from binary system increased when compared to its removal

from single system. From the study it is established that RhB can exist in both zwitterionic and cationic forms above pH 4 and this leads to the better extraction of RhB in reverse micelles from binary system as it was encountered by both cationic and anionic surfactant. Due to this competitive interaction of both the dyes with cationic surfactant the percentage removal of AY36 decreased in binary system. The extraction of the dye from aqueous phase to the apolar solvent phase was solely dependent on the electrostatic interaction between the oppositely charged headgroup of the surfactant and the charge on the dye molecule. The results demonstrated that solvent extraction of dyes using reverse micelles can be effectively used in removal of high concentrations of ionic dyes in the industries at a large scale.

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