Extraction of Hazardous Direct Green b Dye with Application of Reverse Micelles of Gemini Surfactants

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Abstract:- **Removal of dyes from the water is hot cake in the science world. Different methods have been employed for the satisfactory removal of dyes. Current methods for their removal largely rely on adsorption techniques which are costly and produce another waste to be disposed off, whereas the concept of reverse micelles acting to encapsulate the dye in aqueous micro pool in solvent environment provides a useful chemistry. The removal of the direct dye DG-6 from aqueous phase in amyl alcohol solvent using cationic surfactants was studied. 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 effect of dye and surfactant concentration, pH, solvent, salts like KCl and MgCl₂ were 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.**

Keywords: **Reverse Micelles, Cationic Gemini Surfactant, Liquid – Liquid extraction method, salt effect, partition coefficient**

I. INTRODUCTION

Color has long been an important part of society. Color can denote class, economic status, and style. It is no wondering then that every civilization, dating back even to ancient times has developed different dye techniques and processes. These processes range from using dyestuffs found in nature, to the chemicals that are sometimes used today. No matter which dyes are used, natural or synthetic, water is required to complete the process. The ever-developing dye industry has adversely affected the environment. As with any industry, production creates a considerable amount of waste. Textile manufacturers generate about 3, 84,000 tons of waste each year. Efforts should be focused on managing resources, recycling, and proper disposal of waste. Earlier natural dyes were used hence were degradable. Things began to change around 1856 when the synthetic dyes came into existence. They are cheaper to produce, brighter, more color-fast, and easy to apply to fabric, these new dyes changed the playing field. Scientists raced to formulate gorgeous new colors and before long, dyed fabric was available to all, and natural dyes started becoming obsolete for most applications. This brightly colored, changed new world was not without a down side however. The chemicals used to produce dyes today and their breakdown products are often highly toxic, carcinogenic and mutagenic to life. Dyes cause a lot of problems to the environment. Depending on exposure time and dye concentration, dyes can have acute or chronic effects on exposed flora and fauna and even its presence in small quantities (less then 1 ppm) is highly visible in water due to their brilliance. It affects the aquatic life as the light absorption by dyes diminishes photosynthetic activity of algae and seriously influence on the food chain. Dyes can remain in the environment for an extended period of time, because of high thermal and photostability. Dyes are mostly introduced into the environment through industrial effluents.

There is ample evidence of their harmful effects. Triple primary cancers involving kidney, urinary bladder and liver of dye workers have been reported. Mathur et. al. studied the mutagenicity of textile dyes and the effluents containing these dyes, and the influence on the health of textile dyeing workers and the environment [1]. The degradation product of dyes could be carcinogenic. Textile dyes can cause allergies such as contact dermatitis and respiratory diseases, allergic reaction in eyes, skin irritation, and irritation to mucous membrane and the upper respiratory tract. Depending on their chemical complexity, structure and synthetic origin, dyes are resistant to fading on exposure to light, water, many chemicals and microbial attack [2-3]. 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 [4]. Public perception of water quality is greatly influenced by the colour. Hence, their presence in wastewater is unwanted, and it is mandatory to remove dyes from effluents before their discharge to the environment. It has become an aim of many researchers to find an effective and economical way of treating the dye-containing wastewater, for protecting the environment. Several studies have been performed for the treatment of effluents. The three main categories are chemical, physical and biological methods. The conventional biological treatments are less effective for treating wastewater containing

azo dyes [5]. In literature, various techniques have been proposed to remove dyes from water, such as oxidation [6-7], adsorption [8-11], flocculation-precipitation [12], membrane separation [13], electro-coagulation [14-16], electrochemical destruction [17], photo catalysis [18-19] ultrasound [20-21]. All the methods have been compared but these methods have advantages as well as disadvantages [22-23]. 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 [24-25]. 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 [26–29], amino acids [30-31] and enzymes [32].

Keeping in view, the past work and perception on solvent extraction method using reverse micelles of surfactants, the removal of anionic dyes was attempted with the laboratory synthesised cationic surfactants.

II. EXPERIMENTAL SECTION

2.1 materials

The cationic gemini surfactant used to prepare reverse micelles was 1,1'-(1,1'-(ethane-1,2-diylbis-(sulfanediyl))bis(hexadecane-2,1-diyl))dipyridinium bromide (CMC = 0.03 mM and Molecular weight = 859.04) [33]. Direct Green B (DG-6) is anionic in nature with sulfate and amino groups attached to the aromatic moieties. The structure and the properties are given in Figureure 1.

Direct Green B (MW = 812.7)
$C_{34}H_{22}N_8Na_2O_{10}S_2$
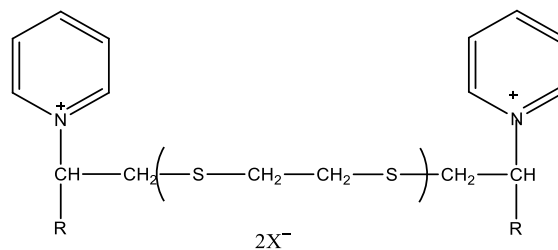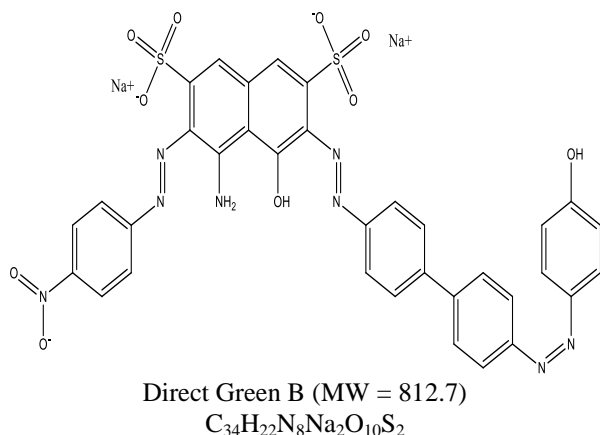
16-2-16 (MW = 859.04)
CMC = 0.03 mM
$C_{44}H_{78}Br_2N_2S_2$

Figure1 Structure of the dye DG-6 and the Surfactant

The solvent used for dye removal from water was amyl alcohol (3-methyl butan-1-ol). Freshly prepared distilled water was used in all the experiments. The pH was varied by adding analytical grade HCl and NaOH. Analytical grade KCl and $MgCl_2$ were used to study the effect of salt. These chemicals and the dye were acquired from E. Merck, India.

2.2 Experimental Setup

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. A UV-Vis spectrophotometer (Shimadzu, UV 1800) has been used to measure the color intensity after and before dye removal in the aqueous phase.

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. Process optimization was carried out with 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 in 1:10 ratio separates in 2-5 hour into pure phases within a range of the surfactant concentration.
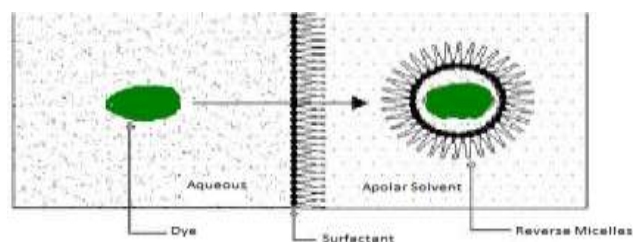
Schematic Representation

Figureure 2 Schematic representation of the liquid liquid extraction of the dye between an aqueous phase and an apolar phase containing reversed micelles

2.3 Methods

2.3.1 Removal of the dye

Different concentrations of the dye were prepared. A known quantity of cationic surfactant above the critical micelle concentration (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 $25^{o}C$. The whole solution was transferred to a separating funnel to separate the solvent and aqueous phases by gravity. The heavier aqueous phase was collected at the bottom of the separating funnel, whereas the lighter amyl alcohol was collected at the top. The residual concentration of dye in aqueous phase was analysed by UV-Vis 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.

2.3.2 Calculation

The extraction performance was determined with the partition coefficient (K) and % removal (E), given by equation 1 and 2

$$E = \frac{D_i - D_{aq}}{D_i} \times 100 \quad (1)$$

$$K = \frac{D_{org}}{D_{aq}} \quad (2)$$

Where

D_i = initial concentration of dye taken

D_{aq} = concentration of dye left in the aqueous media after the two layers separates

D_{org} = concentration of dye encapsulated in the reverse micelles in the organic phase

III. RESULTS AND DISCUSSION

The percentage removal of the dye with the application of surfactant is probably due to the formation of reverse micelle of surfactant in organic layer with entrapment of dye anion in its core. As the efficient removal of the dye, depends upon the size compatibility of the dye with the size of core of the reverse micelle [34]. Depending upon this the surfactant chosen for the removal of DG-6 is 16-2-16. In the first experiment the 0.1 mM of DG-6 was treated with 1 mg of surfactant 16-2-16 and the percentage removal was 72.04 %. It is shown in Figure. 3 that complete removal of the dye can be achieved by the solvent extraction process using the reverse micelles. Effect of various parameters on the percentage removal of the dye is discussed below:

3.1 Effect of Solvent:

To study the effect of the solvent, the concentration of the dye DG-6 (20 mg) and the surfactant (4 mg) is kept constant. The

different solvents viz. amyl alcohol, benzyl alcohol, methyl benzoate, octanol, decanol are studied for the removal of DG-6. The results are reported in Table 1.

Table 1: Effect of the solvent on the percentage removal (E) of DG-6

S.No	Solvent	E
1.	Amyl Alcohol	94.05
2.	Benzyl Alcohol	61.70
3.	Octanol	87.60
4.	Decanol	88.88
5.	Methyl Benzoate	55.68

The result in the table shows that the best solvent for the removal of the dye is amyl alcohol. Hence all the experiments were done using amyl alcohol.
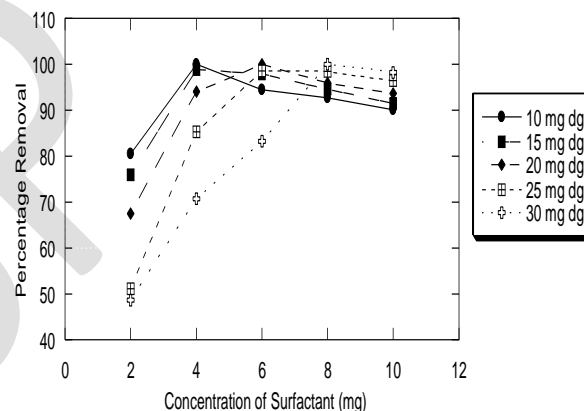

Figure. 3 Effect of the surfactant concentration on the percentage removal of DG-6

3.2 Effect of surfactant concentration:

In this section dye concentration and solvent to water mass ratio were kept constant and the effect of the surfactant concentration has been studied by increasing surfactant concentration from 2 mg to 10 mg. The percentage removal of the dye is studied as the function of surfactant concentration. The results are shown in the Figure. 3 and partition coefficient is given in Table 2. It is verified that an increase in the surfactant concentration causes an increase in the partition coefficient K leading to increase in the percentage removal of the dye (E). This can be attributed to the fact that by increasing the amount of surfactant, the number of reverse micelles increase linearly which increases the number of dye molecules to be encapsulated in the miceller core. This can be further supported by thermodynamic model based on the mass action kinetics:

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

$$D + n [M] \longrightarrow DM + [(n\text{-}1) M] \qquad (3)$$
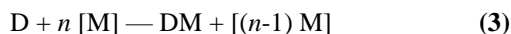
where D = Dye molecule, M = Reverse micelle, DM = Reverse micelle with entrapped dye molecule. Reverse micelle concentration. M is related to the surfactant concentration. [S] as

$$[M] = \frac{[S]}{N\text{ag}} \qquad (4)$$

Aggregation number (Nag) is assumed to be independent of surfactant concentration. So an increase in the surfactant concentration leads to enhanced reverse micelle formation in solvent. Consequently more number of dye molecules gets encapsulated increasing the solubility of the dye in the organic phase. This causes a great reduction of dye concentration in aqueous phase thereby explaining the higher E values and increase in the K with surfactant concentration. After certain concentration of the surfactant the percentage removal decreases after it reaches 100 % due to saturation. the Figure shows that 10 mg of DG-6 is removed 100% at 4 mg of the surfactant where as 15 mg, 20 mg, 25 mg and 30 mg of the dye DG-6 is removed 100 % at the concentration of the surfactant 5 mg, 6 mg, 7 mg, 8 mg respectively. After these concentrations there is decline in the percentage removal of the dye. Hence it is recommendable that the concentration of the dye and the surfactant must be in proper proportions for the better results.

Table 2: partition coefficient (K) and % Removal (E) by varying the concentration of the surfactant

			Before LLE		After LLE			
S.No	D_i	D_{sur}	D_{aq}	D_{org}	K	E
1.	20	2	6.49	13.51	2.081	67.50
2.	20	4	1.19	18.81	15.806	94.05
3.	20	6	0.00	20.00	α	100.00
4.	20	8	0.80	19.20	24.000	96.00
5.	20	10	1.24	18.76	15.129	93.84

3.3 Effect of concentration of dye

The effect of the dye concentration has been studied by increasing the concentration of the dye from 10 mg to 30 mg/ 100 mL by keeping the surfactant concentration constant in the range of 2 mg to 10 mg. Partition Coefficient and percentage removal (E) is shown in Figure. 4 and Table 3. The partition coefficient K and the percentage removal (E) of MO decreases with the increase in the concentration of dye at constant concentration of surfactant i.e. at 2 mg and 4 mg as it is shown in Figure. 4. It may be due to the fact that the number of reverse micelles formed remains constant at particular concentration of the surfactant. With the increase in MO concentration the intake of the dye in the reverse micelles does not take place i.e. organic phase tends to become saturated of the dye leading to the enhanced dye concentration in the water

phase. Howe ever at 6 mg of the surfactant, there is first increase in the percentage removal from 10 mg to 20 mg of the dye concentrations. But at higher concentration of the dye i.e. at 25 mg and 30 mg, the percentage removal decreases. The results for higher concentrations of the surfactant are different. At the concentration of 8 mg and 10 mg of the surfactant, the percentage removal of the dye increases gradually. This can be explained on the basis that at higher concentrations of surfactant the reverse micelles are not saturated and are available for the encapsulation of the dye molecules. Hence shows an increase in the percentage removal of the dye.
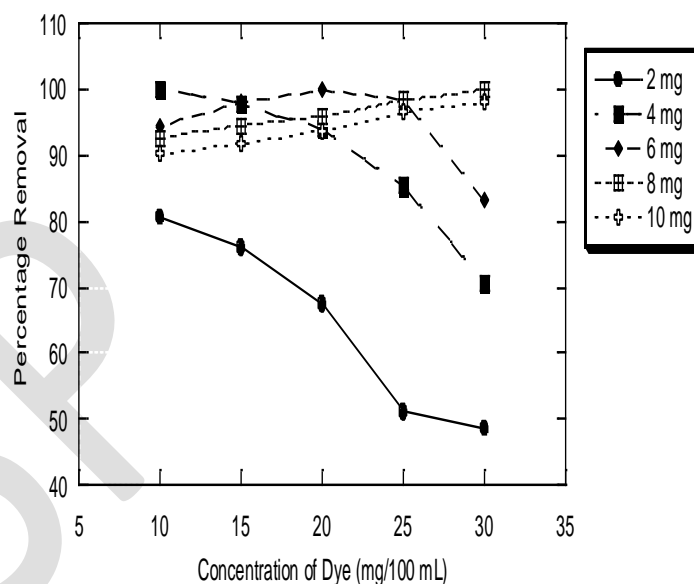

Figure 4 Effect of the dye concentration on the percentage removal of DG-6 as function of surfactant concentration (2 mg – 10 mg)

3.4 Effect of pH:

It has been found that pH of aqueous solution significantly influences the extraction efficiency; especially for pH sensitive dyes as pH of the aqueous phase affects the degree of ionization of a dye molecule. But this is a general characteristic of direct dyestuffs that possess a good ability to dye cotton (cellulose) over a wide pH range. Hence there is little or no effect of pH on direct dyes. The effect of pH on the removal of DG-6 is investigated in the pH range of 2-8 (by adding 0.1 N hydrochloric acid or 0.1 N sodium hydroxide solutions), while initial DG-6 concentration, volume of solvent, and surfactant amount were 20 mg 100mL^{-1}, 10.0 mL and 2, 4, 6, 8, 10 mg, respectively. The results shown in Figure. 5 reveal that the extraction efficiency decreases when the concentration of surfactant is 2 mg. At the higher concentrations, there is no effect of pH and at all pH, the effect of surfactant concentration also perishes.

Table 3: Partition Coefficient (K) and % Removal (E) by varying the concentration of dye

S.No	D_i	2 mg		4 mg		6 mg		8 mg		10 mg	
		K	E	K	E	K	E	K	E	K	E
1.	10	4.15	80.60	α	100	17.18	94.50	12.70	92.72	9.20	90.15
2.	15	3.17	76.04	44.45	97.86	48.67	97.98	17.40	94.56	11.32	91.88
3.	20	2.08	67.50	15.81	94.05	α	100	24.00	96.00	15.13	93.84
4.	25	1.05	51.11	5.83	85.36	67.12	98.53	62.13	98.41	27.38	96.47
5.	30	0.95	48.76	2.42	70.73	4.95	83.23	α	100	55.07	98.23

removal of DG-6 increases gradually. The difference in the decrease in DG-6 concentration in water and the increase in DG-6 concentration in amyl alcohol suggests the possibility of the formation of surfactant–methyl orange complex in the presence of salt at the amyl alcohol–water interface. Rabie *et al* proposed a similar view for low protein extraction in the presence of NaBr. However, it should be noted that the dye molecule might not behave in a fashion similar to that of the protein molecule, since they are complicated in nature.
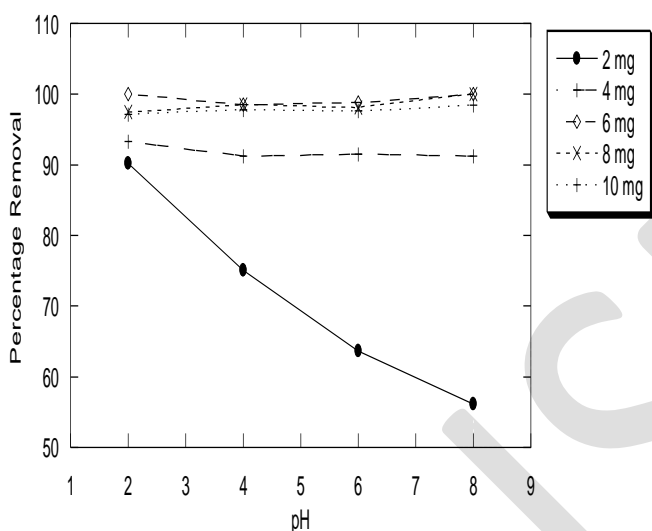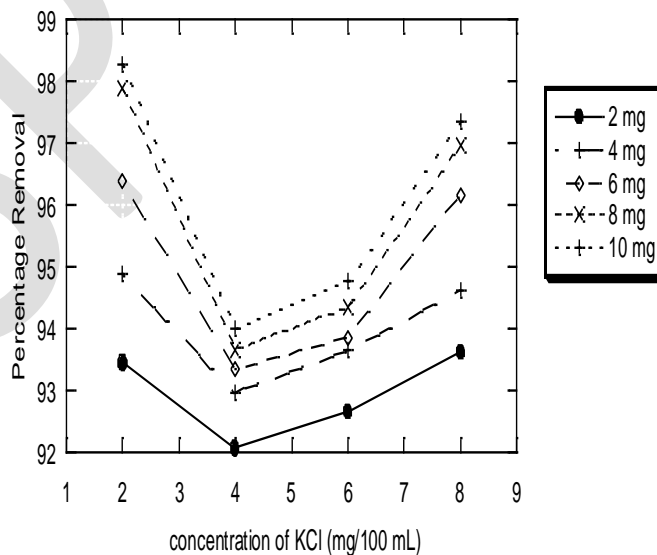

Figure.5 Effect of pH on the percentage removal of DG-6 as function of surfactant concentration (2 mg – 10 mg)

Figure.6 The effect of salt KCl on the removal of DG-6 (20 mg/100 ml) as function of surfactant concentration (2 mg – 10 mg)

3.5 Effect of salts

The effect of salt on the removal of the dye with the application of the reverse micelle by solvent extraction method has been studied. In this study the experiments were carried out using two salts viz. KCl and $MgCl_2$. Both the salts have their on effect on the percentage removal depending upon their effect on the CMC of the surfactant. The results of the experiments of the salt effect are given below.

3.5.1 Effect of KCl

The effect of the salt KCl has been studied by adding salt in the range from 2 gm to 8 gm to the dye solution. The results of the effect of KCl on the percentage removal of DG-6 from water in the presence of surfactant are shown in Figure. 6. It can be seen that the percentage removal of DG-6 is decreased with the increase in KCl concentration from 2 mg to 4 mg for a given dye concentration (20 mg 100^{-1} ml). On further increasing the salt concentration, the percentage

3.5.2 Effect of salt $MgCl_2$

The effect of the salt $MgCl_2$ has been studied by adding salt in the range from 2 gm to 8 gm to the dye solution. The results of the effect of $MgCl_2$ on the percentage removal of DG-6 from water in the presence of surfactant are shown in Figure. 7. The Figure shows that the effect of $MgCl_2$ on the removal of DG-6 from the solution is different from that of effect of KCl. There is decrease in the percentage removal on adding 2mg to 6 mg of the salt. Futher addition of salt $MgCl_2$ enhances the percentage removal of DG-6.
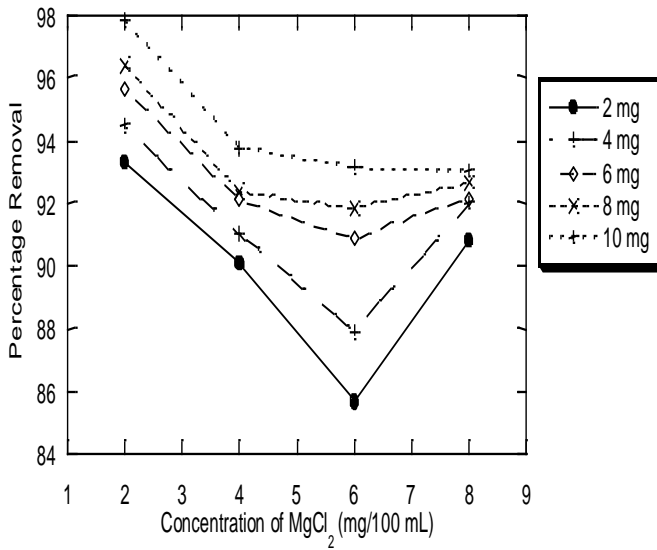
Figure. 7 Effect of $MgCl_2$ on the percentage removal of DG-6 (20 mg/100 ml) as function of surfactant concentration (2 mg – 10 mg)

3.5.3 Effect of anion on the removal of MO

In order to study the effect of anion on the removal of the dye, experiments were carried out using bromide salts. In addition to Cl^-, dye removal by reverse micelles is also affected by Br^-, but the extent of their effect on removal is very small. Hence the basic reason of the increase in the dye removal can be the cations K^+ and Mg^{2+}. The effect of the salts can be seen in Figure. 8, 9 and Figure. 10.
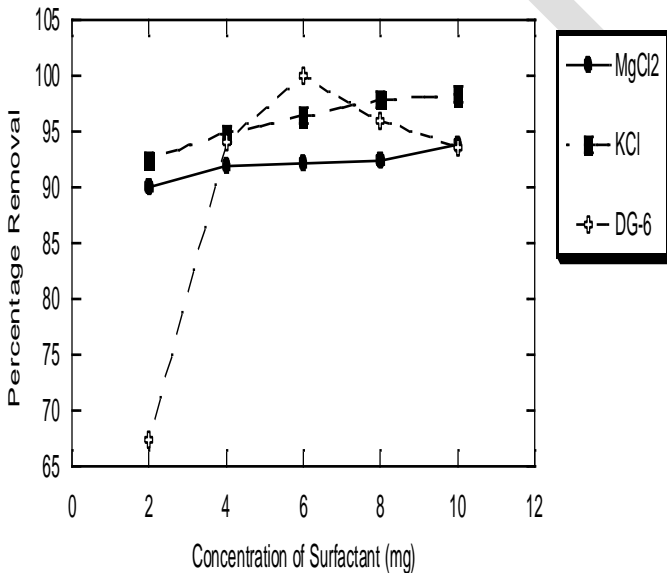

Figure.8 Percentage removal of DG-6 (20mg/100 ml) at different concentrations of Surfactant in the presence of 0.2 mol of KCl and 0.2 mol of $MgCl_2$ separately
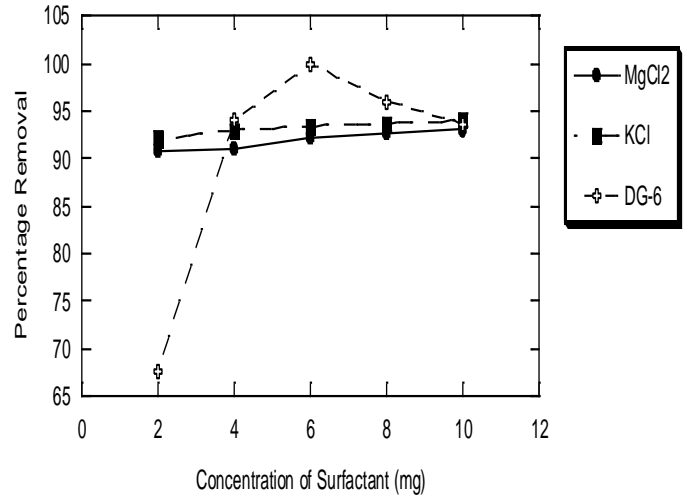

Figure.9 Percentage removal of DG-6 (20mg/100 ml) at different concentrations of Surfactant in the presence of 0.4 mole of KCl and $MgCl_2$ and without salt separately
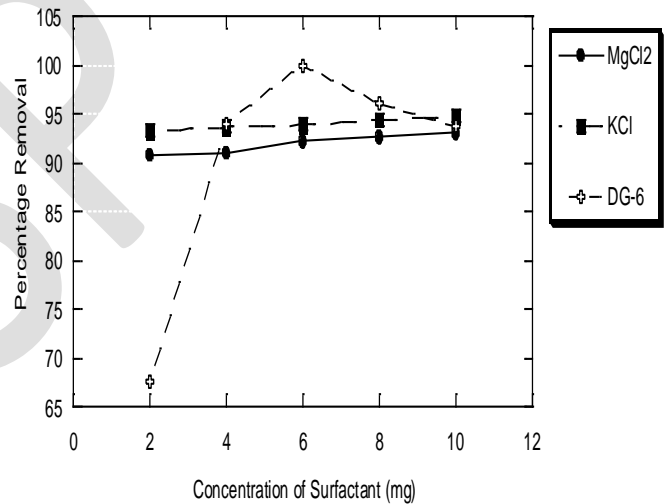

Figure.10 Percentage removal of DG-6 (20mg/100 ml) at different concentrations of Surfactant in the presence of 0.8 N of KCl and $MgCl_2$ and without salt separately

The above data shows that the cation plays important part in the removal of the dye. The removal of DG-6 in the presence of both the salts increased though they follow a different trend. The percentage removal of DG-6 is more in the presence of K^+ than in the presence of Mg^{2+}. Less the charge on the ion more it facilitates the removal of the dye. Positive charge facilitates the early formation of the surfactant-methyl orange complex as their presence lowers the CMC of the surfactant. The extent of lowering the CMC of the surfactant is different in both the cases. KCl lowers the CMC of the surfactant to higher extent hence shows better removal of the dye than $MgCl_2$.

IV. CONCLUSION

Dye removal via solvent extraction method with the application of Reverse Micelles shows promising and excellent results. 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. The effect of pH and salt concentration is explained based on charge transfer mechanism and electrostatic interactions and dye-surfactant complex formation. 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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