

## REVIEW ARTICLE

# A review on reverse micellar approach for natural fiber dyeing

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## Abstract

This study reviews the need and importance of non-aqueous dyeing systems in the chemical processing industry, especially using the reverse micellar system in the dyeing of natural fibres. When it comes to conventional dyeing systems, which use large quantities of water, chemical, and energy, cause a lot of effluent load to the environment. Again the effluent water has to be treated well to eliminate all the harmful substances in it. To overcome the issues, a lot of research has been carried out in this area to minimise the use of water and chemicals in the dyeing process. Like use of low material to liquor ratio (M:L) in dyeing, dyes with high fixation at lower temperatures, low/salt less dyeing in case of reactive dyeing with cotton, cottonseed oil dyeing, microwave-assisted dyeing, and use of supercritical carbon dioxide in case of polyester dyeing and many more. All the above said methods are aqueous-based and, after the completion of dyeing, results in wastewater generation, which requires further treatment to reduce the harmful chemicals. Therefore, to further minimise the use of water and chemicals in the dyeing of natural fibres reverse micellar system has been introduced with the help of surfactants of both Ionic and nonionic in nature. As of today, a lot of work has been carried out in the dyeing of natural fibres with this system by employing ionic, non-ionic, and mixed surfactants. Fascinating results were obtained in the dyeing with good levelness, high exhaustion, and fixation values, and results were compared with conventional dyeing. Computer colour matching studies were also done better to understand the applicability of these systems in the industry and found nearer results.

## 1 | INTRODUCTION

The textile chemical processing industry is one of the most significant contributors to environmental pollution and almost 70% of total pollution from the textile industry. At the same time, the use of chemicals will also harm the environment and human beings.<sup>1</sup> This is

**Abbreviations:** K/S, Reflectance versus scattering; CI number, Colour Index number; M:L, Material to liquor ratio; HLB scale, Hydrophilic-lipophilic balance scale; PEG, Polyethylene glycol; D5, Decamethyl cyclopentasiloxane.

mainly due to the use of large quantities of different chemicals like alkali, acids, surfactants, inorganic salts, and organic matters like dyes present in the effluent water.<sup>2</sup> The textile chemical processing industry consumes high energy to heat the dye solution, leading to severe environmental problems.<sup>3,4</sup> Various methods have been developed to reduce the use of energy and also water.<sup>5,6</sup>

In the case of the cotton processing industries where reactive dyes are mainly used, the effluent water coming out consists of highly polluted, heavy oxygen demand, salinity, and colour. So this needs to be treated properly before discharging to the nearby water sources. Since the treatment involves high investment cost, so many industries are unable to afford it, so the developments should come out in chemical processing technology like machinery design, eco-friendly auxiliaries, which will reduce the effluent load.<sup>7</sup> Cotton dyeing with reactive dyes consumes large quantities of water per kilogram of fabric compared to any other class of dye. During the application large quantity of salt is required for dye exhaustion which leads to salinity in the effluent water. Apart from this, it also consists of alkali and organic matter.<sup>8</sup>

Reactive dyes are most widely used for cotton dyeing because of their unique characteristic, i.e. they are able to react chemically with the hydroxyl groups of fibre and form covalent bonds. Because of this strong bond formation, dyed fabrics possess good fastness properties. There are chances of dye reacting with the hydroxyl groups present in the water leads to hydrolysis of dye.<sup>9</sup> Influence of nonionic surfactant on hydrolysis of reactive dyes were studied using high-performance liquid chromatography. The results revealed that, critical micellar concentration of surfactant plays an important role in hydrolysis of reactive dyes. Surfactant in below the critical micellar concentration acts as dispersant, further accelerating the dye hydrolysis. If it is above the critical micellar concentration, the dye will still be in its solubilised state in micellar phase.<sup>10</sup> This hydrolysed dye remains in the dye bath and on the fibre surface. In order to obtain good washing fastness, surface-level unfixed dye particles have to be removed by a process called washing off, where a series of soaping and rinsing with cold and hot water is involved. Around 50% of dyeing cost is due to this washing-off and effluent treatment.<sup>11</sup> The dye particles which are left with the fibre are said to be fixed and form covalent bonds with the fibre. If optimum conditions are maintained in dyeing, around 50-80% dye fixation will be there in the fibre. The remaining colour is discharged into the effluent water.<sup>8</sup> The reactive dyeing of cotton involves the usage of a large quantity of salt to assist the exhaustion process, depending on the depth of shade, dye structure, and method of application. The use of alkali depends on the type of dye, and pH required in the

dyeing process leading to the fixation of the dye in the fibre. Regardless of the dye, salt, and alkali used they are all relatively non-biodegradable and toxic.<sup>12</sup> After the dyeing process, the leftover water consists of all these chemicals which have high levels of biological oxygen demand, organic and inorganic solids.<sup>13</sup>

In order to overcome the ecological issues with this conventional dyeing system which uses large quantities of water and other chemicals, causing highly pollutant effluent water, researchers have carried out investigations in developing new technology in chemical processing of textiles for reduced use of chemicals and water.<sup>14-16</sup> Moreover, the aqueous dyeing system requires high heating energy to heat the dye solution.<sup>3</sup> This high amount of energy consumption can lead to various environmental issues.<sup>17</sup> Different approaches have been introduced by researchers in dyeing, such as use of reactive dyes without the salt,<sup>16</sup> pre-treating the cotton fabric with EDTA,<sup>12</sup> chitosan and its derivatives,<sup>18</sup> dendrimers (polyamidoamine),<sup>19</sup> grafting cellulose with cationic agent,<sup>20</sup> sericin modification of cellulose<sup>21</sup> and, reuse of wastewater for dyeing,<sup>22</sup> low material to liquor ratio (M:L) dyeing using microwave radiation,<sup>23</sup> use of natural dyes and mordant,<sup>24-26</sup> supercritical carbon dioxide in dyeing of synthetic fibres.<sup>27-29</sup> All these methods improve the present water-based dyeing system to reduce the use of water and chemicals without compromising on fixation and depth of colour. But there are certain limitations with these methods. Wide pre-treatment of a cotton fabric involves additional cost, and if the treatment is not uniform uneven dyeing will result, and effluent water consisting of more pollutants. Developing a new dyeing method in which the conventional aqueous-based dyeing is shifted towards a non-aqueous-based dyeing system with the help of surfactants and solvents.<sup>30</sup> The main objective of this review is to highlight the importance and need for the reverse micellar system and its use in the dyeing of natural fibres.

In the process of understanding the reverse micellar approach in the coloration of textiles, it is very important to understand the role and function of surfactants. Surfactants are organic compounds having two chemical parts with different polarities. The head group has an affinity towards polar phases, and the tail group has an attraction with non-polar phases.<sup>31-34</sup> Because of this uniqueness in its structure, it can be used in many applications to reduce interfacial and surface tension between two or more phases.<sup>35-37</sup> Surfactants have a tendency to form micelles since it generates self-assembled structures in the solutions from nanometre to micron range. It consists of both hydrophobic and hydrophilic parts which makes them suitable to use in various applications which includes medicine,<sup>38,39</sup> detergents,<sup>40</sup> wetting agents,<sup>41-43</sup> drug delivery,<sup>44,45</sup> anti-corrosive treatments.<sup>46-49</sup>

Surfactants are the main component in the formation of micelles. The nature of surfactant is amphiphilic, ie it consists of hydrophilic head and hydrophobic tail. In other words, a surfactant is made out of polar and non-polar groups. As per the nature of polar and non-polar groups, surfactants are classified as cationic, anionic, zwitter ionic and nonionic. In aqueous solutions, surfactants can form micelles and inorganic solvents produce the reverse micelles.<sup>50-52</sup> Micelle are said to be aggregate formation of molecules consisting of both polar and non-polar groups. The structure of micelles looks like a hydrophilic head oriented towards dispersing water, whereas hydrophobic tail forms the micellar core by arranging themselves in interior region. Substances dispersed in a polar solvent can get dissolved by this core.<sup>53</sup>

Reverse micelles are characterised as a nano sized aqueous medium in an organic phase they are stabilised by surface active agents. They are developed with the help of different aqueous phases, organic solvents and surfactants.<sup>54</sup> In order to prepare reverse micelles, it requires an immiscible organic phase (80-90%) containing surfactant (~10%) and a polar solvent (0-10%) to dissolve the dye. Therefore, a reverse micellar system is also called a water-in-oil (W/O) emulsion. As the main component in the preparation of reverse micelle, the surfactant which consists of a hydrophobic tail tries to arrange itself in the polar solvent and the hydrophilic head forms the water pool which is shown in Figure 1. This will be protected from the direct contact with organic solvent and dissolve dyes, enzymes, and protein in aqueous medium. This system can be characterised by its viscosity comparable with organic solvents, large surface area ( $10^2$ - $10^3$  m<sup>2</sup> cm<sup>-3</sup>), transparent nature (nanometre size range from 10 nm to 100 nm), thermodynamic stability, low interfacial tension ( $<10^{-2}$  mN m<sup>-1</sup>), and ability to dissolve solvents of a polar nature.<sup>55</sup>

Reverse micelle formation involves dissolving the surfactant in an organic solvent. Since most of the surfactants have less or partial solubility in organic solvents the use of co-surfactants helps to dissolve them in organic solvents and form the required reverse

micelle.<sup>56</sup> The co-surfactant acts as an agent to reduce the ion-ion interaction between the organic phase and the surfactant molecules. Thereby it allows the close packing of head groups of surfactant to form a hydrophilic inner core which is stable and forms the reverse micelle.<sup>57</sup> In spite of assisting in dissolving the surfactant in the organic phase it will also assist in adjusting the diameter and shape of the micelle either smaller or larger.<sup>58</sup> Along with using co-surfactant, the use of co-solvents also favours the reverse micelle formation and the molecular weight, water solubility and dielectric constant determine the suitability of using co-solvent in the reverse micellar system.<sup>54</sup> Lower water solubility is the primary requirement of co-solvent to be used in reverse micelle formation. Isooctane, *n*-octane, hexane and long-chain alcohols are some of the solvents preferred to be used as co-solvents.<sup>59</sup> Furthermore, *n*-octane and isooctane have been used as co-solvents for the formation of reverse micelle without modifying the size and structure of the cationic reverse micelle.<sup>60</sup>

## 2 | REVERSE MICELLAR SYSTEM IN TEXTILE DYEING

The reverse micellar system has gained a lot of interest in the chemical processing of natural fibres even though so many methods have been introduced to perform the dyeing process with low M:L because of the low effluents in the process. Table 1 shows the various surfactants used in the preparation of reverse micellar systems to textile coloration. This system aims at solubilising small amounts of water in the interior of the micelle there by resulting in the formation of a stable microenvironment.<sup>61</sup> The dyeing of textiles in this system is schematically shown in Figure 2. Reverse micelles were characterised by molar ratio of water to surfactant. This is given in Equation 1.

$$W_o = [H_2O]/[S] \quad (1)$$

where  $W_o$  is mole ratio of water to surfactant,  $H_2O$ , water and  $S$ , surfactant.

The radius of the micelle is related to moles of surfactant and water per micelle. The surfactant was mainly located at the interphase separating the water pool and organic phase employed.<sup>62</sup>

The reverse micellar system has great potential for use in textile pre-treatments and can also be extended to the coloration of textiles. Cotton and wool fabrics have been comfortably scoured using enzymes in ionic Aerosol-OT reverse micellar system. Bio scouring of cotton was

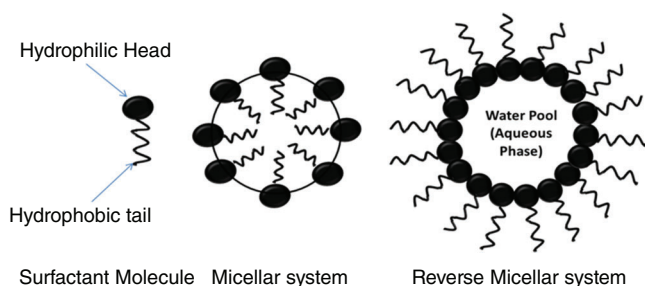


FIGURE 1 Micelles and reverse micelles formation

TABLE 1 Different reverse micellar systems employed in dyeing of textiles for natural fibres

S no	Reverse micellar system and Interaction forces	Surfactant molecule	Organic phase employed	Application	References
1	Ionic surfactant and electrostatic interactions	Sodium bis-2-ethylhexylsulphosuccinate (Aerosol-OT, AOT)	Isooctane	Pre-treatment of cotton, wool/dyeing of cotton with reactive dyes/wool with acid dyes	6
2	Ionic surfactant and hydrophobic	Sodium bis-2-ethylhexylsulphosuccinate (Aerosol-OT, AOT)	Isopropyl alcohol/ <i>n</i> -hexane mixture (1:5)	Simultaneous dyeing and enzymatic processing of wool fabrics with acid and reactive dyes	65
3	Nonionic and hydrogen bonding	Polyoxyethylene <i>tert</i> -octylphenyl ether (TX-100)	<i>n</i> -Octanol and isooctane (1:5)	Dyeing of cotton with reactive dyes	69
4	Nonionic and hydrophobic	Poly(oxyethylene glycol) (12) tridecylether and <i>n</i> -octanol was used as co-surfactant in the dyeing	Heptane and octane were used as organic solvent	Dyeing of cotton fabric with reactive dyes	61
5	Nonionic and hydrophobic	Poly(ethylene glycol) (12) tridecylether, <i>n</i> -octanol was used as co-surfactant in the dyeing process	Decamethylcyclopentasiloxane (D5)	Reactive dyeing on cotton fibre	74
6	Nonionic and hydrophobic	Poly(oxyethylene glycol) (12) tridecylether, <i>n</i> -octanol was used as co-surfactant in the dyeing process (surfactant to co-surfactant ratio 1:8)	Heptane (reagent grade with a minimum 99% <i>n</i> -heptane) and solvent volume to cotton weight ratio 8:1	Reactive dyeing on cotton fibre	77-78
7	Nonionic and Hydrophobic	Poly(ethylene glycol) (12) tridecylether, <i>n</i> -octanol was used as co-surfactant in the dyeing process (surfactant-to-co-surfactant molar ratio 1:8)	Octane and nonane (wool-to-solvent weight ratio [w/v] 1:10)	Dyeing of wool with reactive dyes	72
8	Nonionic mixed surfactant and hydrophobic	Polyoxyethylene <i>tert</i> -octylphenyl ether (TX-100), sorbitan monopalmitate (Span40)	<i>n</i> -Octanol and isooctane	Dyeing of cotton fabric with CI Reactive violet 2	73
9	Nonionic and hydrophobic	Polyoxyethylene sorbitan trioleate (Tween-85)	Isopropyl alcohol/ <i>n</i> -hexane (1:5)	Enzymatic hydrolysis of wool fabrics	65

performed with pectinase enzyme in the presence of a reverse micellar system. Even at a low concentration of pectinase enzyme (ie 0.1 g L<sup>-1</sup>), bio scouring performance was remarkably enhanced when compared to the aqueous

system, which uses nearly 100 times more enzymes. When it comes to wool fabric treated with protease enzyme in the presence of reverse micellar system, no significant effect was found when compared with the aqueous system.

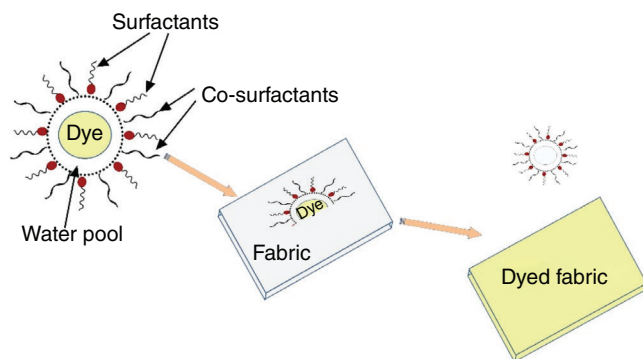


FIGURE 2 Dyeing of textiles in reverse micellar system

But interestingly Sawada and Ueda,<sup>6</sup> noticed that protease maintained its activity in the non-aqueous system. Minor work has been done on the dyeing behaviour of cotton, polyester and blends using direct, reactive and disperse dyes to know the effect of reverse micelles on the dye uptake by the fibre. Results revealed that, dye absorption rate is higher than the conventional water-based system due to the very low water bath ratio.<sup>6</sup>

In order to further explore the effect of reverse micellar system in dyeing Sawada and Ueda<sup>61</sup> have investigated the dyeing behaviour of cotton fabric with direct dyes in the presence of surfactant Aerosol-OT. Increasing the concentration of surfactant increases the solubility of the dye in the water pool formed in the reverse micelle. Dye uptake of cotton fabric in direct dyes was compared with aqueous and reverse micellar system. Colour depth was analysed with respect to amount of water content in the reverse micellar system. It was found that, low water content on cotton (ie  $w = 10 \text{ mol kg}^{-1}$ ) leads to lower dye uptake comparatively with water content around  $20\text{-}50 \text{ mol kg}^{-1}$ , this is due to lower swelling of cotton in the system with low water content. It was also found that colour depth in the reverse micellar system was much deeper compared with the aqueous system without the use of electrolytes due to the ionic head group of the surfactant acting as an auxiliary to assist the dyeing process. Therefore, the increased dye ability of dye in this system was due to high concentration of dye in the water pool created in the reverse micellar system at low water concentration.

Furthermore, to provide useful knowledge of the dyeing behaviour of protein fibres in the reverse micellar system Sawada and Ueda<sup>63</sup> made an attempt to dye silk fabric in the reverse micelles made out of bis-2-ethylhexylsulphosuccinate Aerosol-OT (AOT) (anionic) as a surfactant in the presence of iso-octane as a solvent with the potential to dissolve hydrophilic substances such as enzyme and dye. Therefore, in the dyeing process with this combination of dye and micelle the dye particle

adsorption on to the fibre would be more effective. Silk fabric was dyed with Colour Index (CI) Acid Orange 7 in the presence of aqueous, reverse micellar and acid reverse micellar system. Results of this study were found to be quite the opposite to earlier studies that is dyeability of silk in a reverse micellar system with and without acid was very low compared to aqueous system. This was due to the competitive adsorption between surfactant and dye molecules. Molecular size of the selected dye has an influence on this context, where the dye has an intermediate molecular size between surfactant and direct dye selected in earlier studies. Studies were also carried to dye silk fabric with direct dye (CI Direct Red 28) in the reverse micellar system and interestingly dye uptake was improved and it is comparable with aqueous-based dyeing using acid dyes. This may be due to the high concentration of dye in the water pool and the higher affinity of the dye in the reverse micellar system. Reactive dye is also used in this investigation to understand the adsorption of dye on to silk fabric in this system. Fixation of 80 to 90% was achieved with reactive dyes due to less amount of water and more concentrated dye available in the water pool.

## 2.1 | Nonionic reverse micellar system

Ionic head groups in the surfactant molecules have great impact on the water pool polarity.<sup>64</sup> To provide a suitable environment for the enzyme reaction in the water pool, nonionic surfactant was used in reverse micelle preparation. Polyoxyethylene sorbitan trioleate (Tween-85) a nonionic surfactant was used to prepare the reverse micellar system to modify wool fabrics with Bioprax 30L enzymes. The results obtained were most promising and the effective changes took place without much damage to the fibre. By increasing the enzyme concentration and treatment time in the nonionic reverse micellar system results in the increased weight loss of wool fabrics. Enzyme found to be still active in this system catalyses the protein component by hydrolysis in the wool fibre. It was found that enzyme treatment in the reverse micellar system did not influence the tear strength of wool fabrics as compared to aqueous system.<sup>65</sup>

To enhance the dye exhaustion in reverse micellar system an attempt was made to treat the protein fibres in nonionic surfactant in non-aqueous media. This system has superior properties and forms a stable aqueous microenvironment by solubilising water in its interior of the micelle that is the water pool. Tween-85 was used as a nonionic reverse micellar system for dyeing and enzymatic processing of wool fabrics. The small angle X-ray scattering results showed that the present system proved

to be good enough to incorporate enzymes and dyes in its interiors without changing the structure of the micelle. Reverse micellar system was prepared with acid/reactive dye and Bioprax 30L enzyme and wool fabric was kept in this solution at 40°C to initiate the reaction. Adsorption of acid and reactive dyes onto wool fabric was found to be more effective and shows colour strength ( $K/S$ ) of around 22-25 at 3 h treatment time. The effect of dye bath pH was also studied in this system and compared with aqueous-based system. Results of the study confirm that wool can also be dyed even in alkaline pH without much changes in the final  $K/S$  values compared to aqueous system. Wool can be effectively dyed in this system with deep shades compared to the same shade using conventional way of dyeing with acid dyes. But enzymes in this system were observed to have lower activity compared to aqueous dyeing.<sup>66</sup>

In preparing the reverse micelles anionic surfactant (Aerosol-OT) was used which has an influence on the polarity of the water pool and leads to an uneven micro-environment. Further this leads to having a negative impact on the system developed.<sup>67</sup> So to overcome this problem nonionic surfactant “polyoxyethylene *tert*-octylphenyl ether (TX-100)” was used to prepare the reverse micelles to obtain an even microenvironment. Dyeing of cotton fabrics was performed in this reverse micellar system with reactive dyes. To study the dye bath exhaustion dyeing was performed with and without the addition of salt. It was evident from the results that an increase in concentration of salt in the bath leads to a decrease in the exhaustion of dye particles on to fibre in the reverse micellar system due to aggregation of dye particles in the presence of sodium chloride (NaCl). The dyeing experiments were performed without addition of salt and interesting results were obtained by the authors. It was found that the dye uptake is higher in the proposed system that is for reactive RY145 at 1% dye concentration was found to have 70% exhaustion when compared to aqueous system with same conditions for same dye and concentration having 60% exhaustion. This was due to high concentration of large molecular weight reactive dye in the reverse micellar system which also minimises the competitive adsorption between dye and surfactant.<sup>68</sup> This is unlike the work done by Sawada and Ueda,<sup>63</sup> who showed that there was competitive adsorption between acid dye and surfactant, which resulted in low dye uptake. However, in depth analysis of adsorption studies on dyeing behaviour and fastness properties of dyed fabrics in reactive dyes in the proposed system was not reported by the authors.

Yi et al<sup>69</sup> have further carried out adsorption studies on application of reactive dyes on to cotton fabric in the nonionic Triton X-100 reverse micellar system. The

reactive dye after its solubilisation was injected into the micellar system. Colour strength and fixation rate of dye were studied in the present system and water-based system. The results found higher fixation rate of 80-90% and colour strength was achieved in the present micellar model when compared to bulk water system. Also the adsorption pattern was found to be monolayer and depends on molecular structure of dyes. A decreasing pattern of adsorption was observed with increasing NaCl concentration in the proposed micellar system due to dye aggregation in the presence of inorganic salt. Fastness properties of dyed fabrics were also measured and found that good fastness of washing grade 4-5 and rubbing grade 4-5 were achieved.

## 2.2 | Use of co-surfactant in nonionic reverse micellar system

A novel method of dyeing cotton in reverse micellar system was prepared to promote the dye absorption and fixation in the reverse micellar system by using nonionic surfactant and co-surfactants in required proportions which are shown in Figure 3. The use of co-surfactant in the reverse micellar system is to reduce the surface tension as minimum as possible and assist in well dispersed dye particles in the water pool. It was also found that the hydrophilic-lipophilic balance (HLB) value of the reverse micellar system decreased after addition of co-surfactant. The optimization of this ratio is very important in deciding the water pool stabilisation and effective incorporation of dye into this system.

The effect of using co-surfactant in reverse micellar system and its dye absorption by the fibre have been studied so far. Reverse micelles were prepared with nonionic surfactant along with a co-surfactant. The nonionic surfactant was prepared with polyethylene glycol (PEG)-

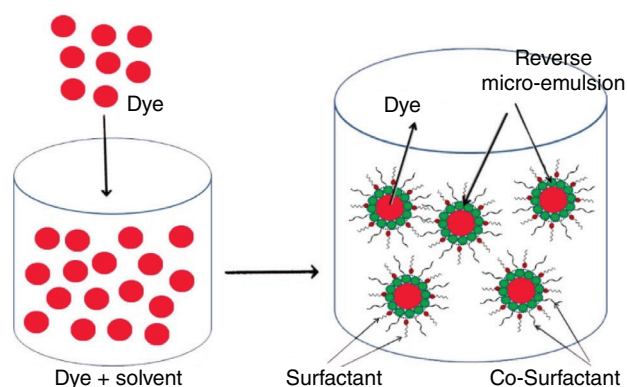


FIGURE 3 Surfactant and co-surfactant in reverse micelle formation and presence of dye in the water pool

12 tridecyl ether as shown in Figure 4, *n*-octanol (co-surfactant) in alkane (heptane and octane) based organic solvent and cotton fabrics were dyed with this combination after injecting reactive dye in to the system as shown in Figure 5.

The effect of co-surfactant on dye ability and fixation rate of reactive dye on to cotton fabrics was studied. Also the effect of alkali and possibility of dye fixation in the absence of salt was investigated. Results revealed that, the highest colour yield was obtained at solvent volume to cotton weight ratio of 1:8 due to proper dye distribution in the water pool. Increasing this ratio results in lower dye uptake, because of large water pool volume dye gets aggregate and difficult to diffuse into the fibre. Highest *K/S* value was reached at surfactant to water mole ratio of 0.05:1-0.04:1. Higher values results in stable water pool formation. There is a considerable effect of surfactant to co-surfactant ratio on the dyeability of cotton fabrics and its colour strength. It was found that surfactant to co-surfactant ratio of 1:8 shows the highest *K/S* value and also shows the increased dye-fibre interactions in the proposed system. Figure 4 shows the schematic representation of dye in water pool created by using surfactant and co-surfactant combination.

The effect of water pool volume on dye solubilisation in water pool and colour fixation was investigated. It was found that, water pool volume of 0.5 mL shows the highest colour yield in the range from 0.3 to 0.7 mL. A low volume of water pool results in inadequate swelling of cotton fibre, further reducing the dye uptake by the fibre. Excess water pool volume slightly weakens the dyeability of fibre since the fibre is swollen in water. Water pool volume at 0.3 mL shows the effective dissolution of soda ash for good colour depth. The influence of dyeing time on *K/S* value was also studied. It was found that 40 min dyeing time at 60°C shows maximum colour

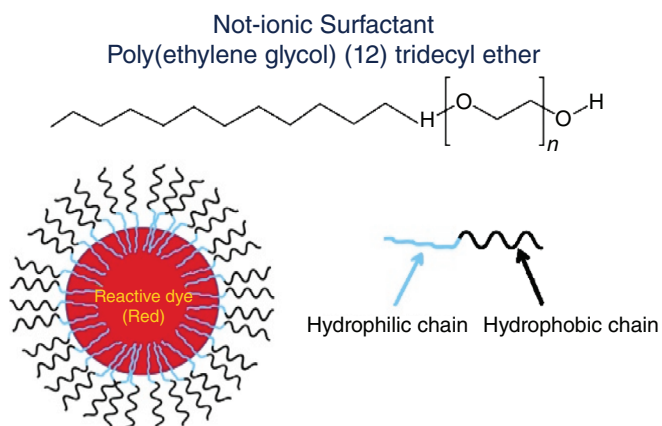


FIGURE 4 Nonionic surfactant – polyethylene glycol-12 tridecyl ether

strength. Colour fastness to laundering was studied and the colour retaining capacity of dyed fabrics in the reverse micellar system was found to be more or less similar to water-based system.<sup>62</sup> Dispersibility of dye in the reverse micellar system is also very important to obtain even dyeing. But in this study, dispersibility of dye in surfactant and co-surfactant and its resultant system was not determined.

Therefore, investigations were carried out in the agglomeration behaviour of dyes which are encapsulated in reverse micellar system. These studies are significant for the evaluation of quality of dyed fabrics. The dispersibility of reactive dye introduced in the PEG-based reverse micellar system under various surfactant and co-surfactant ratios has been studied. Raman spectroscopy was used to analyse the effective adsorption of reactive dyes on to cellulosic fibre in varying surfactant and co-surfactant molar ratios, surfactant to water mole ratios. As already stated in the previous studies, optimization of surfactant and co-surfactant ratio is very important in stabilisation and solubilisation of dye in the water pool formed in the reverse micellar system. Surfactant to co-surfactant molar ratio of 1:9 was found to have a high impact on the *K/S* sum value (414.53) of dyed fabrics compared to molar ratios of 1:6 and 1:8 (410.25 and 411.05) with better levelness measured in terms of relative unlevelness index ( $RUI = 0.137$ ) confined in smaller size that is <100 nm with high dispersed state. The results also revealed that, decreasing the molar ratio of surfactant to co-surfactant from 1:8 to 1:6 leads to increase in size of dye encapsulated reverse micelle from 80 nm to 1.5  $\mu\text{m}$  at constant surfactant concentration. It was understood that, the use of co-surfactant (*n*-octanol) was helpful in reinforcing the interface between outer and inner sides of the micellar structure and further reduces

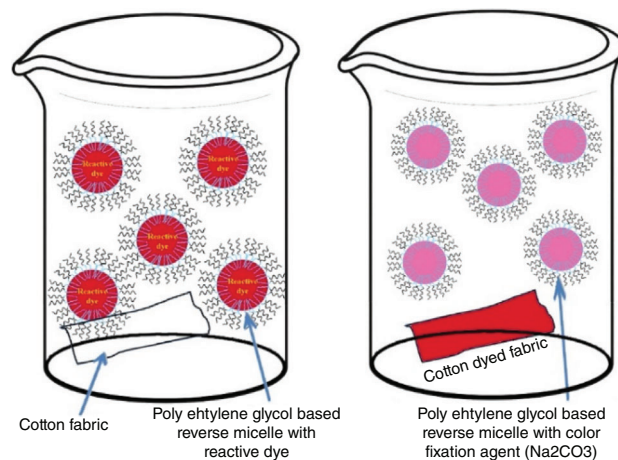


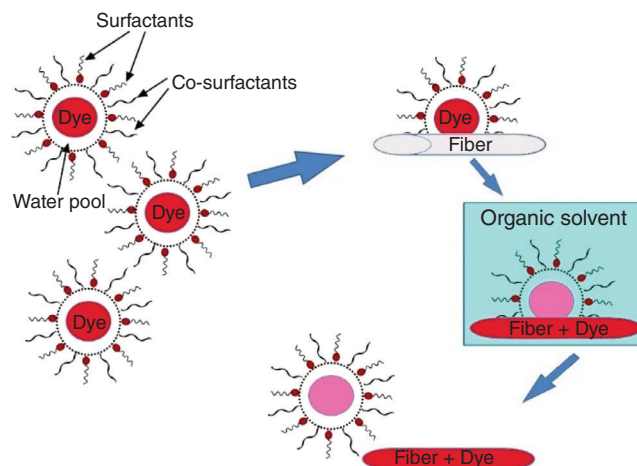
FIGURE 5 Dyeing of cotton fabric in PEG-based reverse micellar system

the aggregation of dye particles. Decrease in amount of *n*-octanol results in a decrease in rigidity of surfactant hydrophobic chains causing the morphological distortion of reverse micelle formed leading to aggregation of dye in the water pool. Results proved that increasing the ratio leads to an increase in dispersibility of dye particles. It was also found that the size of the dye aggregates was influenced by various surfactant parameters which suggest the distribution of dye on the fabric depends on the morphology of dye aggregates. Like the earlier studies, increasing molar ratio of water and surfactant results in an increased water pool volume, thereby increasing the reverse micellar size which favours dye clustering.<sup>70</sup>

Most of the investigations on reverse micellar system preparation and dyeing were carried out on cotton fabrics using reactive dyes. Octane and nonane assisted PEG-based reverse micellar system was developed and acid dye was injected in to the system to dye wool fabrics. Wool fabrics were dyed in this system and compared with the conventional water-based system. Computer colour matching and levelness of dyed fabrics were investigated. The results obtained were comparable with the water-based dyeing method. From the reflectance values measured no chromatic change was found. For both the dyed systems calibrations curves were almost linear and slightly higher for water-based system. But the colour values (*K/S*) obtained were higher than that of conventional dyed samples. The RUI values obtained depict that the obtained dyed samples achieved good to excellent levelness both subjectively and objectively.<sup>71</sup> The reverse micellar approach was successfully adopted for dyeing of wool with reactive dyes by using PEG as a building media. Three different colours were introduced in the core of the PEG-based reverse micellar system under optimum conditions. The dyeing process can be done with reactive dyes without salt in the proposed system which is comparable with the water-based dyeing. In addition, the dyed fabrics showed good fastness to washing compared to water-based dyeing.<sup>72</sup>

### 2.3 | Mixed reverse micellar systems

To further enhance the dyeing properties of cotton fabric in nonionic reverse micellar system the use of a mixed reverse micellar system was introduced instead of mono micellar system which is shown in Figure 6. Mixed surfactants wide nonionic TX-100, sorbitan monopalmitate (Span 40) was used as dye carrier medium. The particle size distribution, aggregation of micelles, electric conductivity, adsorption behaviour of reactive dyes on to cotton fabrics and colour fastness properties were measured. The results were found to be more interesting when



**FIGURE 6** Dyeing of cotton fabric in mixed surfactant reverse micellar system employing polyoxyethylene *tert*-octylphenyl ether (TX-100) and sorbitan monopalmitate (Span40)

maximum solubilisation capacity of water was obtained at equi-molar ratio of the two surfactants. Solubilisation capacity of the proposed mixed micellar system was improved by increasing the amount of Span 40 up to 50%. HLB value of used surfactants were reduced after addition of Span 40, resulting in an improved solubilisation capacity which in turn results in increased dye solubility. Water pool diameter and packing density of the reverse micellar system was improved by increasing the quantity of Span 40. The adsorption and dye movement towards the fibre was improved by adding Span 40 surfactant in to TX-100 which acts as a medium in lowering the repulsive interacting between dye and fibre. Finally, the proposed system improved the dye fixation percentage to 86.34% compared to 83.46% for the aqueous-based system. Colour strength was also found to be better in the proposed mixed micellar system.<sup>73</sup>

### 2.4 | Rate of hydrolysis of dye in reverse micellar system

So far the researchers focused on establishing the reverse micellar dyeing with different surfactants, co-surfactant combinations by employing suitable organic phase solvents systems. Studied related to hydrolytic kinetics of reactive dyes are very limited. This will give the prompt information about the dye uptake and fixation of dye inside the fibre. Hydrolysis rate of vinylsulphone (VS)-based reactive dyes was studied in the siloxane reverse microemulsion medium employing decamethyl cyclopentasiloxane (D5) solvent. Hydrolysis rate of VS reactive dye has been analysed using high-performance liquid chromatography. The results found that the rate of hydrolysis was slow when compared



to conventional water-based dyeing. It was also found that the dye hydrolysis rate was affected by ratio of aqueous solution to reverse microemulsion ratio as well as by the content of cellulose and temperature maintained in the dyeing.<sup>74</sup>

So far in the preparation of reverse micelles hydrocarbon solvents as a medium such as *n*-heptane, hexane, cyclohexane, which are not only eco-friendly but also toxic to humans. Further to reduce the effluent load in the waste water out of this system and to make this system acceptable, reverse micelle were employed with continuous phase medium of eco-friendly solvents along with the regular surfactants. One such organic phase employed is the use of D5. It is a clear, odour less, non-oily cyclic, colour less, and safe fluid mostly used in consumer and industrial applications. Dyeing of cotton fabric were carried out using this D5 solvent in a suitable dispersion system (HPIS). Superior results were found in terms of dye uptake (90-95%), dye fixation (90%) and *K/S* value (17@2% dye concentration) when compared with water-based system using salt (dye uptake 55-60%, dye fixation 50%, *K/S* value 5.5@2% dye concentration). A visual evaluation method was used to assess the levelness of dyeing and it was found that satisfactory dyeing levelness was obtained at a bath ratio of 1:30, controlled dyeing temperature and time of 20°C and 20 min, respectively, alkali concentration of 40 gpl, and alkali solution pick up of 160%. Colour fastness properties like wash, dry, and wet rubbing of dyed fabrics in both systems were measured and found to have similar results except for wet rubbing (3, 4) compared to water-based system (4).<sup>75</sup> By using this D5 solvent, reverse micellar system was prepared and dyeing was carried out in this system, which further reduces the harmful effects of hydrocarbon-based organic solvents.

## 2.5 | Computer colour matching studies

Once the dyeing is done it is important for the dyer to match the colour with the customer given sample. So far the reverse micellar system has been developed and has dyed the textile substrates but studies related to computer colour matching of dyed fabric has not been reported. PEG (nonionic surfactant)-based reverse micellar system comprising an eco-friendly medium of solvent assisted D5 was used to dye the cotton fabric with reactive dyes. Colour matching and levelness of fabrics dyed with reactive dyes in the system were studied. From the results it was found that the *K/S* values obtained were higher than that of conventional dyed samples. From the computer colour matching results it was shown that the measured concentrations were more or less similar to that of

expected concentrations for both methods. This indicates exact colour matching is obtained with the introduced system. RUI value was used to measure the levelness of dyed samples in both systems. It was found that the samples dyed in the water-based system had RUI values between 0.02 to 0.34, whereas the samples dyed in the proposed micellar dyeing system showed values in the range of 0.05 to 0.40. This clearly shows that in the D5 solvent assisted method there was good to excellent levelness when compared to water-based conventional method.<sup>76</sup> This was due to the fact that most of the dye particles were not in an aggregate state but were dispersed uniformly in the bath.

To further explore the computer colour matching of solvent-based reverse micellar dyeing and water-based dyeing of cotton fabrics, PEG-12 tridecyl ether and octane assisted reverse micellar system prepared and dyed the cotton fabrics with reactive dyes. The authors were mainly investigating the computer colour matching of dyed samples from the present system and the conventional water-based system. The results revealed that the measured concentrations were near to that of expected concentrations for both systems. Therefore this octane assisted reverse micellar system can be implemented in industrial dyeing with computer colour matching since the colour difference was observed to be less than one.<sup>77</sup> PEG assisted by heptane and nonane as solvents was developed and injected the known quantity of dye into the system. Results of the studies proved statistically that, the measured concentrations were similar to that of expected concentrations for both systems. This indicates that, the system developed can be conveniently applied to industries and colour matching can be achieved and large quantities of water can be saved. Nonane-based dyed samples achieved good to excellent levelness.<sup>78,79</sup> The effect of process parameters on the dyeability and final colour yield of dyed samples have not yet been reported.

A study was conducted on effect of pH and hardness on reverse micellar dyeing of reactive dyes in the presence of heptane assisted PEG-based nonionic surfactant system. The main focus was to study the colour yield, reflectance, unevenness and CIE *L*, *a*, and *b* values. A decrease in pH levels were shown to have higher colour yield and lower reflection. Furthermore, hardness in the water irrespective of pH causes unevenness in the dyed samples. However, changes in hardness and pH does not changes the CIE *L*, *a*, and *b* values due to small usage of water in the reverse micellar system.<sup>80</sup> Even though the reverse micellar system proved to be effective and more than 90% of dye is fixed, the use of water limited further fixation and the dyed fabric required several washings. Therefore, a non-aqueous-based dyeing system was introduced by Deng et al., a novel binary system based on

dimethyl sulphoxide (DMSO) and D5 which do not mix (immiscible) with each other in normal room temperatures. When the temperature is increased the solvents will mix with each other. By using this system, the results of dye fixation showed that more than 97% of dye fixed and only one stage washing process was required to obtain good fastness properties. Furthermore, consumption of energy and recycling of solvent becomes easy.<sup>81</sup>

## 2.6 | Advantages and disadvantages of different reverse micellar systems

Even though advantages are available with the proposed reverse micellar system when compared with the conventional water-based system in terms of reduced use of water, chemicals, and energy, the system is still not getting wide acceptance by the industries due to several reasons. The major drawback with this system is the high cost of surfactant, co-surfactants, and solvents used compared to existing chemicals used in conventional dyeing, the system is still in the laboratory stage and there is a lack of clearness about the various challenges that are going to be faced during its implementation in bulk production, issues with levelness in the dyed fabrics, controlling the process parameters and finally recovery and reuse of surfactants. If these issues can be addressed in a well-defined manner industries could benefit from this system.

## 3 | FUTURE SCOPE OF WORK

In view of raising demand for safeguarding the environment from harmful effluents, so many methods were introduced in the textile chemical processing industry. All are mainly focused on reduced use of chemicals, dyeing at low M:L, energy saving, use of super critical carbon dioxide, pre-treatment of textiles to minimise the chemical use, use of reused water in dyeing and reverse micellar dyeing. This review mainly focused on the various studies involved in the use of reverse micellar system in textile coloration carried out with various surfactants and solvents. It was observed that, compared to conventional dyeing which uses large quantities of water and chemicals, reverse micellar system developed which consumes very less amount of water and chemicals with higher dye fixation values. In fact, the use of water and other chemicals has been minimised or eliminated and the dyeing performed with required levelness. Studies were even carried out to improve the levelness and improve colour matching of dyed samples. Even after having all these benefits, the future research should focus

on development and use of effective and low cost surfactants and solvents for its wide acceptance in the industry for its commercial acceptance.

All the studies listed in this review have been predominantly discussed based on the experiments done at laboratory level. When it comes to industry the quantity of fabric used is huge, conditions to be maintained are completely different. Therefore, studies need to be focused in the directions to perform the dyeing process at less effluent load to the environment. Also a lot of investigation needs to be carried out on the dyeing kinetics in the proposed reverse micellar system which would help to understand the dye movement towards the fibre that would assist in controlling various process parameters in dyeing for uniform dyeing. Most of the work done so far has focused on the dyeing behaviour of cotton fibre in the reverse micellar medium and limited work has been done on wool fibres. This can be extended to other natural fibres and synthetic fibres also in view of reduced use of water, chemicals, and energy.

## 4 | CONCLUSION

This review critically focuses on the vast study conducted in the area of non-aqueous dyeing specially concentrated on reverse micellar dyeing of natural fibres especially on cotton and few studies on wool. Reverse micelles are prepared by using immiscible organic phase, surfactant and a polar solvent to dissolve the dye particles. This system is characterised by its thermodynamically stable, large surface area, low interfacial tension and capable of forming a stable microenvironment that is a water pool which can dissolve polar solvents. The role of surfactant is crucial in the formation of the reverse micellar system where the surfactant is mainly located at the interphase separating the water pool and organic phase employed. The use of co-surfactant also helps in the formation of the required micellar formation by allowing the close packing of head groups of surfactants to form a hydrophilic inner core. Ionic, nonionic and mixed surfactants were used in combination with organic solvents and co-solvents at different molar concentrations to prepare a stable micellar formation. Dye particles were dissolved in aqueous phase and introduced in the water pool formed by the surfactant. The dyeing of cotton fabrics carried out at different temperatures were found to have better colour uptake and fixation compared with aqueous-based dyed fabrics. Furthermore, computer colour matching and fastness studies were also carried out to compare the extent of colour matching of dyed fabric in the proposed system with aqueous-based samples. The results showed that there are no significant differences in the colour

value of the final dyed samples and even fastness properties were also found to have similar patterns.

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