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ORIGINAL RESEARCH

Non-aqueous salt-free reverse micellar dyeing of cotton fabrics with reactive dyes: a study of secondary alcohol ethoxylate (SAE)-based non-ionic surfactants with different properties

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Abstract The suitability and dyeing performance of SAE-based biodegradable surfactants of different properties as the building block for reverse micelle formation and functioning as dye carrier for dyeing of cotton fabric in non-aqueous octane medium are investigated. Morphology of the dye encapsulated reverse micelles formed by these surfactants is examined by transmission electron microscopy. The images show that SAE-based surfactants of different properties form reverse micelles of different shapes. Dyeing of cotton fabrics is conducted with the use of reverse micelles of different morphologies as dye carrier. The dyeing, colourfastness and tensile properties of the dyed samples are evaluated and compared with fabrics dyed in conventional water-based system. Samples dyed with Tergitol 15-S-12 (TS12) surfactants achieved the highest colour Yield and the lowest reflectance percentage while Tergitol 15-S-20 (TS20)-dyed samples obtained excellent colour levelness. Poor fabric levelness was found when Tergitol 15-S-7 (TS7) and Tergitol 15-S-9 (TS9) surfactants were used. SAE-based surfactants with optimal moles of ethylene oxide and hydrophilic-lipophilic balance value achieve the best dyeing effect on cotton fabric.

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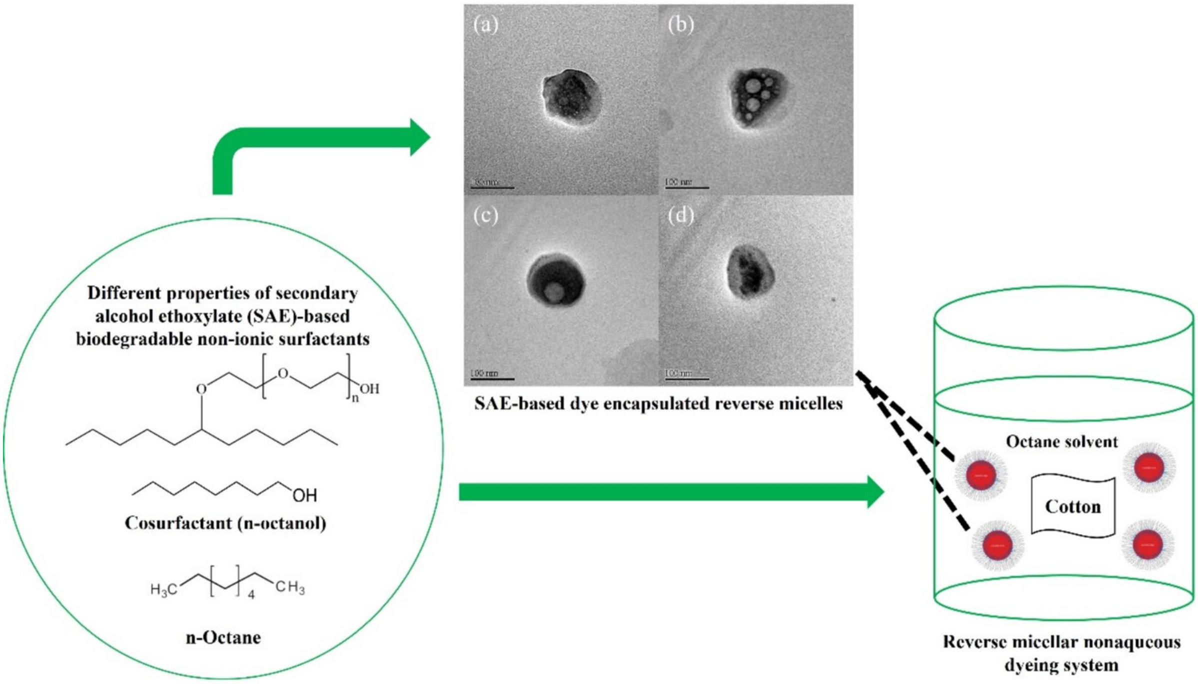
Graphical Abstract



Keywords Nonionic surfactant · Secondary alcohol ethoxylate · Cotton fabric · Reactive dyes · Non-aqueous dyeing · Reverse micelle · Octane · Salt-free

Introduction

Textile industry has long been described as a manufacturing industry with one of the longest and complex supply chains in the world (Hynes et al. 2020; Niinimäki et al. 2020). It generally begins with fibres which can be found naturally in the environment or produced synthetically from petroleum derivatives. By transforming the fibres into yarns through spinning and from yarns to fabrics through weaving and knitting, the cotton fabrics undergo a series of wet processes, including scouring, bleaching, mercerising, dyeing and finishing, which not only requires a significant amount of water, but also produce large quantities of wastewater (Fan et al. 2024; Holkar et al. 2016; Lu et al. 2024; Paździor et al. 2019; Shu et al. 2024).

To improve the textile dyeing process and make it a more environment-friendly and sustainable process, many attempts have been reported in recent literature. Kumbhar et al. (2019) and Jiang et al. (2022) reported the use of natural dye extracted from leaves and seeds for dyeing of cotton fabric. Several studies have been done on salt-free dyeing of cotton with chemical modifications (Dong et al. 2020; Niu et al. 2020; Tang et al. 2021) or through the use of cationic polyelectrolyte complex (Wang et al. 2022). Instead of using water as dyeing medium, different attempts have been made on dyeing of cotton in non-aqueous medium. Abou Elmaaty et al. (2022) and Zaghloul et al. (2021) used supercritical carbon dioxide as medium for dyeing of cotton. In addition, cotton has also been dyed by using binary solvent system (Deng et al. 2019), ternary solvent system (Wang et al. 2020), silicone oil system (Pei et al. 2022), liquid paraffin system (An et al. 2020) and spent cooking oil (Liu et al. 2019).

The use of surfactants as building blocks for formation of reverse micelle to be used as the dye carrier is one of the alternative greener ways for non-aqueous dyeing of cotton with low add on technology (Tang

& Kan 2020; Wang et al. 2016). Surfactants, also known as amphiphiles or tensides, is abbreviation of surface active agents (Dave & Joshi 2017). They are substances in which they can adsorb at surfaces or interfaces, decreasing the surface or interfacial tensions between two immiscible phases with the formation of molecular clusters named as micelles (polar water phase) or reverse micelles (non-polar oil phase) when their concentration is above the critical micelle concentration (CMC) (Holmberg et al. 2003). They can be ionic or nonionic in nature. Ionic surfactants can be subdivided into anionic, cationic or amphoteric depending on the dissociation of type of ions in aqueous solutions. They are generally hydrophilic surfactants and soluble in water (Rath & Srivastava 2021).

Nonionic surfactants can be either hydrophilic or lipophilic, depending on the balance between the hydrophilic or lipophilic portions of the nonionic surfactants (Hydrophilic-Lipophilic Balance, HLB) (Housaindokht & Nakhaei Pour 2012). The concept of HLB was first proposed by William C. Griffin with a series of formulae for calculation of HLB of several nonionic surfactants (Pasquali et al. 2009). HLB is one of the key factors in the reverse micellar system in which the change of HLB value may affect the size and polar core of the reverse micelles (Gacek & Berg 2015; Michor et al. 2016; Parent et al. 2011). In addition, nonionic surfactant solutions may cloud when temperature increases. Cloud point is the temperature at which this transition occurs. It also indicates the solubility of nonionic surfactants since a higher cloud point denotes a higher hydrophilicity of the surfactant (Nakama 2017).

Our previous works mainly focused on the use of nonionic polyethylene glycol (PEG) surfactant as a building block for formation of reverse micelles as dye carriers for dyeing of cotton fabrics in non-aqueous medium (Tang et al. 2017). An attempt has been made to study the effect of HLB value of nonionic surfactants, including PEG and Brij series, on reverse micellar dyeing of cotton fabric (Tang et al. 2018). Recently, we have developed and optimised the parameters of a novel reverse micellar system based on the use of nonionic secondary alcohol ethoxylated (SAE) surfactant (Tang et al. 2023a). However, only SAE surfactant with 12 mol of ethylene oxide (EO) is studied whereas the influence of other SAE-based surfactants of different properties on reverse micellar dyeing of cotton fabric has not yet been reported in the literature. Therefore, it is worthwhile to further investigate this aspect.

Herein, SAE-based surfactants of different properties are selected as the building blocks for formation of reverse micelles as reactive dye carriers for dyeing of cotton fabric in non-aqueous octane medium. The objectives are: (a) to investigate the feasibility of using SAE-based surfactants of different properties for formation of reverse micelles; (b) to examine the morphology of reverse micelles formed by different SAE surfactants as dye encapsulants in non-aqueous octane medium; (c) to compare the properties of reverse micellar dyed cotton fabrics with those of conventional water-dyed fabrics; (d) to assess the colour fastness of the dyed fabrics; and (e) to evaluate the degree to damage to surfaces of cotton fabrics dyed by different SAE-based surfactants.

Experimental

Materials and reagents

Ready-for-dyeing 100% pure cotton woven fabric (density: 127 ends and picks per cm; weight: 139 g/m^2) was first subjected to a pre-washing treatment for removal of dirt and other unwanted substances. The pre-treatment condition was stated as follows: (i) 2 g/L domestic laundering detergent; (ii) 49 °C washing temperature; (iii) treatment time of 45 min; and (iv) tumble-dried. Then it was conditioned at 20 ± 2 °C and relative humidity (RH) of 65 ± 2% for 24 h. Several secondary alcohol ethoxylate (SAE)-based nonionic biodegradable surfactants with different properties, including Tergitol series of 15-S-7 (TS7), 15-S-9 (TS9), 15-S-12 (TS12), and 15-S-20 (TS20) surfactants, were purchased from Sigma Aldrich (Table 1). The general chemical structure of SAE-based surfactants is shown in Fig. 1. Octane (98 + % purity) and n-octanol (> 99% purity) were purchased from Alfa Aesar. They were of reagent grade. Sodium chloride (NaCl) was purchased from VWR. Sodium carbonate (soda ash, Na_2CO_3) was purchased from Sigma Aldrich. Three reactive dyes, namely Levafix Red CA (RCA), Levafix Blue CA (BCA) and Levafix Yellow CA (YCA), were bought from Dystar (Shanghai, China) and used directly as received.

Table 1 Properties of SAE-based non-ionic surfactants

	TS7	TS9	TS12	TS20
Moles of ethylene oxide (EO)	7	9	12	20
Cloud point (°C)	37	60	89	> 100
Critical micelle concentration (CMC) (ppm) (25 °C)	38	52	104	315
Surface tension (dynes/cm)	30	30	33	38
Hydrophilic-lipophilic balance (HLB)	12.1	13.3	14.5	16.3

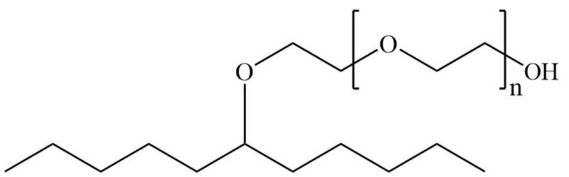


Fig. 1 Chemical structure of SAE-based nonionic biodegradable surfactant (n = 7, 9, 12 and 20)

Aqueous dyeing of cotton fabric

Cotton fabric was dyed with reactive dyes in conventional water-based medium with liquor-to-goods ratio of 10:1 for five colour depths (0.1%, 0.5%, 1.5%, 2.5%, and 3.5% on weight of fibre, o.w.f.). NaCl and Na_2CO_3 of the corresponding amount (Table 2) were added to promote exhaustion and fixation. Cotton fabric was first submerged in dye liquor prepared with dye and NaCl and placed in water bath (10 min). The bath temperature was then raised from 30 to 70 °C (Fig. 2). After 40 min of dyeing, Na_2CO_3 was then added to promote fixation and the fabric was kept in the dye for a further 60 min. The dyed fabric was then rinsed at 50 °C twice with 2 g/L of detergent solution, cold rinsed with tap water, drip-dried and conditioned at 20 ± 2°C and 65 ± 2% relative humidity for 24 h before subsequent measurements.

Dye-encapsulated reverse micelle formation with SAE-based surfactants

Table 3 shows the parameters used for formation of SAE-based reverse micelles with reactive dye molecules encapsulated in the water-pool. SAE-based surfactants of different mole EO and properties were mixed with n-octanol (co-surfactant) to form the solution. Octane was added to the mixture to facilitate the formation of reverse micelles. Reactive dye, in the form of aqueous solution, was finally added dropwise in the reverse micelle solution with continuous stirring to obtain a well-dispersed reverse micellar dye liquor.

Table 2 Recipe for aqueous dyeing of cotton fabric

Liquor ratio 10:1, 70 °C						
Reactive dye	% o.w.f	0.1	0.5	1.5	2.5	3.5
NaCl	g/L	10	20	42.5	55	65
Alkali (Na_2CO_3)	g/L	5	5	5	5	5

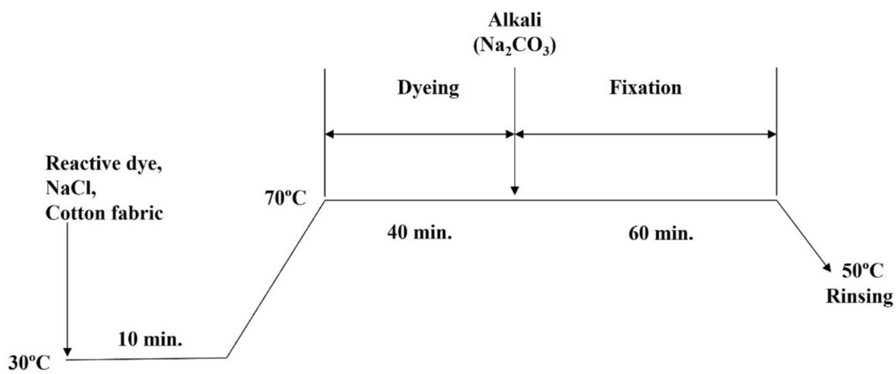


Fig. 2 Aqueous dyeing profile of cotton fabric

Table 3 Parameters for SAE-based dye-encapsulated reverse micelle formation

Parameters	
Surfactant to water mole ratio	1:20
Surfactant to co-surfactant mole ratio	1:8
Octane solvent to cotton ratio (v/w)	10:1
Water-pool volume for dye (mL)	0.5
Water-pool volume for soda ash (mL)	0.3

Table 4 Recipe for SAE-based reverse micellar dyeing of cotton fabric

Solvent ratio 10:1, 70 °C					
Dye concentration (% o.w.f.)	0.1	0.5	1.5	2.5	3.5
Soda ash to cotton weight ratio (g/g)	0.03	0.04	0.05	0.06	0.07

Salt free SAE surfactants-based reverse micellar dyeing of cotton

Cotton fabric was dyed by using reverse micelles, formed by different mole EO and properties of SAE-based surfactants, as reactive dye carrier in octane non-aqueous solvent medium (Fig. 3). The fabric was first submerged in reverse micellar dye liquor and placed in water bath for 10 min at 30 °C. Then the bath temperature was raised to 70 °C and the fabric was dyed for 40 min. Alkali (Na_2CO_3) (Table 4) was added in the dye liquor to allow dye fixation for 60 min. After that, the dyed fabric was rinsed at 50 °C twice with 2 g/L of detergent solution, cold rinsed with tap water, drip-dried and conditioned at 20±2 °C and 65±2% RH environment for 24 h before subsequent measurements.

Colour yield (K/S$_{sum}$ value) measurement

Colour yield (K/S_{sum} value) of both conventional aqueous-dyed and SAE surfactants-based reverse micellar dyed cotton fabric specimens was measured by DataColor SF650 Spectrophotometer (DataColor International, USA) based on Eq. (1), according to the Kubelka–Munk theory. The specimens were folded twice and then measured by using illuminant D_{65} and 10° observer in specular included condition with the use of 20 mm diameter aperture. Each specimen was measured four times, and the average value was taken.

$$K/S = (1 - R)^2/2R \tag{1}$$

where K: absorption coefficient; S: scattering coefficient; and R: reflectance factor of sample.

CIE L*a*b* value measurement

CIE L*a*b* value of both conventional aqueous-dyed and SAE surfactants-based reverse micellar dyed cotton specimens was measured by using similar instruments and parameters, as stated in colour yield measurement section.

Colour levelness assessment

Colour levelness of water-dyed and SAE surfactants-based reverse micellar dyed fabric samples was evaluated by the use of Relative Unlevelness Indices (RUI) (Chong et al. 1992). Four random spots in each dyed sample were measured by apparatus and parameters

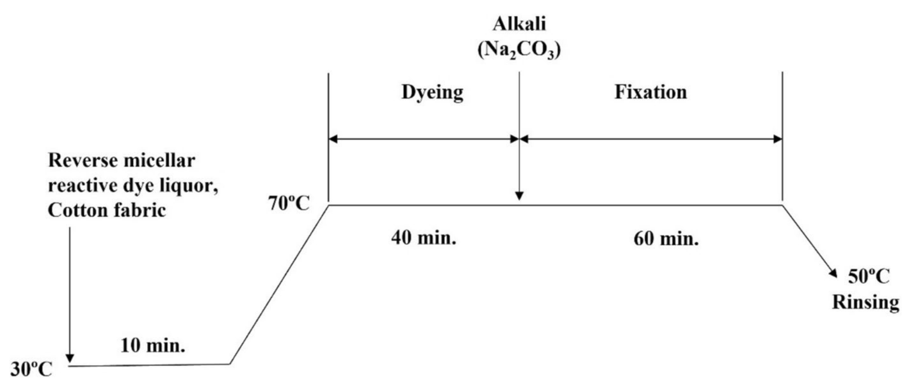


Fig. 3 Salt-free SAE-based reverse micellar dyeing profile of cotton fabric

the same as stated in colour yield section. RUI value of each dyed sample was then calculated by using Eq. (2).

$$\mathrm{RUI} = \sum_{\lambda=400}^{700} (s_\lambda/\overline{R})V_\lambda \quad (2)$$

where s_λ: standard deviation of reflectance value; $\overline{R}$: reflectance value; V_λ: photopic relative luminous efficiency function.

Scanning electron microscopy (SEM)

Hitachi VP-SEM SU1510 scanning electron microscope (Hitachi, Japan) was used to assess the surface morphology and potential damage caused by reverse micellar dyed cotton sample with different SAE surfactants.

Transmission electron microscopy (TEM)

Dye encapsulated morphologies of reverse micelles self-assembled by different SAE surfactants were examined by using JEM 2010 transmission electron microscope (JEOL Co. Tokyo, Japan). The accelerating voltage and beam current for TEM observation were set at 120 kV and 69 μA respectively.

Washing fastness

Washing fastness of water-dyed and SAE surfactants-dyed cotton fabric samples was measured according to AATCC Test Method 61–2013 (Colorfastness to Laundering: Accelerated) Test no. 2A.

Crocking fastness

Dry and wet crocking fastness of water-dyed and SAE surfactants-dyed cotton fabric samples were evaluated according to AATCC Test Method 8–2013 (Colour fastness to Crocking: Crockmeter Method).

Tensile properties

Breaking strength and elongation of water-dyed and SAE surfactants-based reverse micellar dyed cotton fabric samples (3.5% o.w.f. dye concentration) were measured according to ASTM standard D5034-21 (Standard Test Method for Breaking Strength and Elongation of Textile Fabrics: Grab Test).

Results and discussion

TEM images

TEM images of dye-encapsulated morphology of the reverse micelles formed by biodegradable SAE-based non-ionic surfactants with different properties are shown in Fig. 4. It is observed that SAE-based surfactants of different properties and moles EO can form reverse micelle and encapsulate reactive dye in different morphologies. When TS7 surfactant is used as the building block for encapsulation of dye molecules (Fig. 4a), the morphology of reverse micelle is generally spherical but irregular in shape. Diameter of the reverse micelle is about 131 nm while thickness of the TS7 surfactant layer ranges from 8 to 31 nm. The encapsulated dye has an egg-like shape and structure.

With regard to the dye encapsulated structure of reverse micelle formed by TS9 (Fig. 4b), morphology of the reverse micelle and the encapsulated dye is rather triangular in shape. The base of the triangular TS9 reverse micelle is about 153 nm whereas its height is about 131 nm. Thickness of the TS9 surfactant layer is between 8 and 23 nm. The distinct characteristic of TS9 reverse micelle is that the encapsulated dye has many circular bubble-like structures when compared with other SAE-based surfactants.

As stated in our previous work (Tang et al. 2023a), the TS12 surfactant-based reverse micelle (Fig. 4c) is spherical in shape with the diameter approximately 135 nm and the outer surfactant layer thickness between 6 and 23 nm. The reactive dye is being encapsulated as circular or round shape with diameter ranging from 100 to 112 nm.

Regarding TS20 SAE-based surfactant (Fig. 4d), morphology of the formed reverse micelle is elliptical in shape with dye encapsulated being elongated in the interior water-pool region. The major axis and the minor axis of ellipse-shaped reverse micelle are approximately 135 nm and 108 nm respectively. The thickness of the outer TS20 surfactant layer varies from 8 to 31 nm.

Table 5 shows the area and average surfactant layer thickness of reverse micelle in relation to moles EO

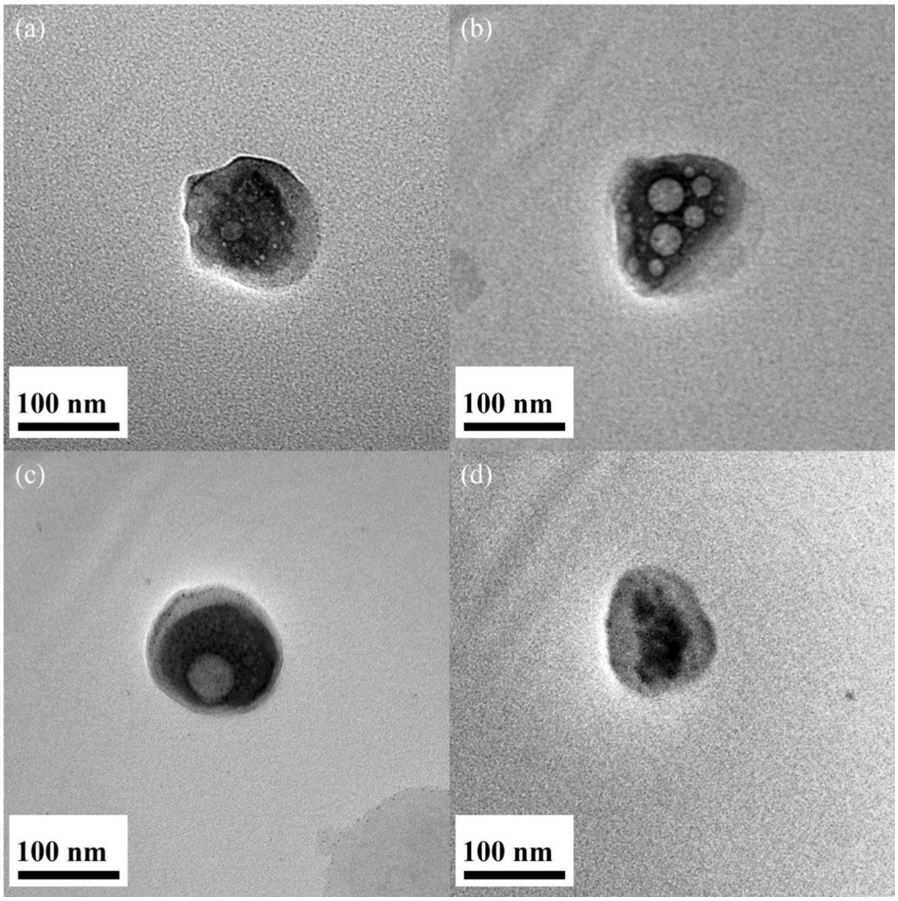


Fig. 4 TEM images of dye encapsulated morphology of reverse micelles with reactive red dye using different SAE-based surfactants: **a** TS7; **b** TS9; **c** TS12; and **d** TS20 (magnification: 40,000x)

Table 5 Area and average surfactant layer thickness of reverse micelle

Surf.	Moles EO	HLB	Area (μm^2)	Average thickness (nm)
TS7	7	12.1	13.5	19.5
TS9	9	13.3	10	15.5
TS12	12	14.5	14.3	14.5
TS20	20	16.3	11.4	19.5

and HLB value. HLB value of the surfactant generally increases with increasing moles EO. However, it is observed that the area of reverse micelle fluctuates from 10 to 14.3 μm^2 and does not have exact relationship with moles EO and HLB value. The fluctuation of the area of reverse micelles is possibly related to their shape. Unlike TS7 and TS12 which forms reverse micelles in almost a round shape, reverse micelles of TS9 and TS20 are in triangular and ellipse shape respectively. Nevertheless, it is found that the average surfactant layer thickness generally decreases from 19.5 to 14.5 nm when the moles EO (7–12) and HLB (12.1–14.5) increase while further increase in EO (20) and HLB value (16.3) results in increase in surfactant layer thickness (19.5 nm). It is worth noting that TS12, compared with other surfactants, can achieve the largest area of the reverse micelles while having the smallest average surfactant layer thickness. This indicates that the TS12-based reverse micelles have larger capacity capable of encapsulating more small reactive dye molecules or larger sizes of dye molecules, optimising the dyeing performance.

These TEM images (Fig. 4a–d) validate the presence of different shapes of reverse micelles and different encapsulation morphologies of reactive dye in non-aqueous octane medium formed by using SAE-based surfactants with different properties and these morphologies of reverse micelles, to a large extent, affect the dyeing properties, especially colour yield and levelness, of the dyed cotton samples. This is discussed in the subsequent sections. In addition, these findings, to a certain extent, show the evolution of the shape of the reverse micelles, from irregular form to ellipse form, when the moles EO and HLB value of the biodegradable SAE-based surfactant increases.

Colour yield

Colour yield of conventional water-dyed cotton samples and non-aqueous octane-dyed cotton samples with moles EO of SAE-based non-ionic surfactants of different properties is presented in Table 6. Generally speaking, cotton fabrics dyed by SAE surfactants with moles EO ranging from 7 to 12 in octane medium with reactive dyes can achieve higher colour Yield than fabrics dyed in conventional aqueous water medium. However, when the moles EO of the SAE-based surfactant further increase from 12 to 20, colour yield of the dyed cotton fabric drops significantly, i.e. colour yield of the TS20-dyed fabric is poorer than that of the conventional water-dyed fabric.

The highest colour yield was found when TS12 surfactant was used for dyeing with BCA, RCA and YCA reactive dyes, probably owing to the reduced ionisation of reactive dyes, which improved swelling of cotton fibres (Wang et al. 2016) as reported in our previous optimisation work (Tang et al. 2023a). Reactive blue-dyed and yellow-dyed fabrics, using TS7 and TS9 surfactants, obtain the second highest and almost the same colour yield while reactive red-dyed fabrics, using TS9 surfactant, reveal higher colour yield than that of TS7 surfactant. The worst colour yields are found when cotton fabric is dyed by using TS20 surfactant with both BCA, RCA and YCA reactive dyes in which K/S_{sum} values of the dyed fabrics are worse than those of the water-dyed fabrics. Poorest colour yield of TS20-dyed cotton fabrics may be the result of high surface tension and HLB value (16.3) of TS20 surfactant (Tang et al. 2018; Yi et al. 2015) compared with other SAE-based surfactants. High surface tension may hinder or resist the penetration of dye into the fabric, leading to uneven dye distribution and lowering the colour yield of the dyed fabric. Too high HLB value may lead to phase separation and agglomeration of dye molecules. This may reduce the solubility and stability of dye in the non-aqueous medium and prevent even dispersion and penetration of dye into the fabric, leading to poor dye uptake and resulting in lower colour yield of the dyed fabric. Another possible reason is related to the moles EO of the SAE-based non-ionic surfactant in which longer EO chain of non-ionic surfactant favours the hydrolysis of reactive dyes (Wang et al. 2018). Hydrolysis is undesirable in the dyeing process in which it leads to the formation of hydrolysed dye

Table 6 Colour yield of different SAE surfactants-dyed samples

K/S_{sum} value										
Colour	Dye conc. (%)	Water	TS7	%	TS9	%	TS12	%	TS20	%
BCA	0.1	6.19	9.10	47.13	9.30	50.29	9.01	45.55	4.33	−0.00
	0.5	23.57	31.41	33.27	31.24	32.57	34.96	48.34	11.18	−52.58
	1.5	71.35	85.65	20.04	78.05	9.40	93.77	31.42	24.99	−64.97
	2.5	121.10	128.21	5.87	132.64	9.53	161.80	33.61	41.61	−65.64
	3.5	163.17	194.95	19.48	192.18	17.78	216.79	32.86	54.74	−66.45
RCA	0.1	5.43	10.82	99.12	11.01	102.57	11.51	111.78	6.64	22.27
	0.5	19.35	37.39	93.25	41.18	112.83	47.44	145.18	19.34	−0.04
	1.5	63.43	102.38	61.40	118.73	87.16	127.40	100.84	47.42	−25.25
	2.5	110.34	171.33	55.27	184.66	67.35	220.29	99.64	79.90	−27.59
	3.5	143.89	218.39	51.77	247.08	71.71	283.64	97.12	112.04	−22.13
YCA	0.1	8.25	9.36	13.52	10.33	25.28	10.21	23.74	6.13	−25.72
	0.5	30.07	35.96	19.57	37.43	24.49	38.26	27.23	17.35	−42.31
	1.5	88.90	95.93	7.90	102.58	15.38	111.73	25.68	39.30	−55.80
	2.5	137.97	137.18	−0.57	156.10	13.14	171.19	24.08	64.12	−53.52
	3.5	177.10	201.12	13.56	194.47	9.81	212.80	20.16	82.38	−53.48

molecules that cannot form covalent bonds with the cotton fibres. This reduces the overall fixation of the dye to the fabric, resulting in lower colour yield and less vibrant colours. In addition, hydrolysed dye may increase the washing requirements with the use of more water and energy and cause poor colour fastness of the dyed fabric. When the hydrolysed dye is discharged as the effluent, it may increase the environmental burden as well as the waste treatment costs.

Regarding critical micelle concentration (CMC), it refers to the concentration at which surfactant molecules start to form reverse micelles. Frankly speaking, reverse micelles can solubilize dyes more effectively once the surfactant concentration is above CMC, enhancing the colour yield. If the surfactant concentration is below the CMC, reverse micelles are insufficient to solubilize the dye, leading to lower colour yield.

Surfactants with high CMC, TS20, require a higher surfactant concentration to form reverse micelles. This means that at lower concentrations, fewer reverse micelles are available to solubilize the dye, resulting in lower colour yield of the TS20-dyed samples when compared to that of the samples dyed by SAE-based surfactants with lower CMC (TS7, TS9 and TS12). In contrast, low CMC surfactants, such as TS7, TS9 and TS12, form reverse micelles at relatively lower surfactant concentrations. As more reverse micelles are available in lower concentrations, it favours effective dye solubilization, achieving better colour yield than that of TS20.

Although lower CMC surfactants are more efficient in solubilizing dyes at lower concentrations and provide better colour yield, surfactants of low CMC, such as TS7 and TS9, may lead to excessive reverse micelle formation, causing aggregation and uneven dye distribution and resulting in poor colour levelness which will be discussed later. Therefore, surfactant of optimum CMC is more beneficial to strike a balance between colour yield and levelness.

Compared with colour yield of conventional water-dyed fabrics, TS7 surfactant-dyed fabrics gain 5.9 to 47.1% increase for BCA reactive dye, 51.8 to 99.1% for RCA reactive dye and −0.6 to 19.6% for YCA reactive dye. In case of TS-9 surfactant-dyed fabrics, the percentage increase in colour Yield for BCA, RCA and YCA reactive dyes is 9.4 to 50.3%, 67.4 to 112.8% and 9.8 to 25.3% respectively. The highest percentage increase in colour yield for the three reactive dyes is TS12 surfactant-dyed fabrics in which increase is 31.4 to 48.3%, 97.1 to 145.2% and 20.2 to 27.2% for BCA, RCA and YCA reactive dyes, respectively. However, when moles EO increases to 20 (TS20 surfactant), a drastic reduction of 30 to 66.6%, 0.04 to 27.6% and 25.7 to 55.8% is observed in colour yield for BCA, RCA and YCA reactive dyes respectively. This finding indicates that the increase of moles EO and HLB value may not absolutely contribute to an increase in colour yield since too high or too low moles EO and HLB value may lead to higher surface tension (Yi et al. 2015), unstable reverse micelle formation (Tang et al. 2018) and hydrolysis of the reactive dyes (Wang et al. 2018) whereas SAE surfactant with 12 mol EO (TS12 surfactant) is believed to be the most suitable surfactant that optimises the dyeing performance of cotton fabric in SAE-based reverse micellar dyeing system.

Reflectance

Figure 5 depicts the reflectance curve of cotton samples dyed by three reactive dyes using conventional water-based and TS surfactant non-aqueous reverse micellar-based methods. Generally speaking, TS7, TS9 and TS12 surfactant-dyed samples reveal lower reflectance percentage than conventional water-dyed samples when BCA, RCA and YCA reactive dyes are used (Figs. 5a–c, e–g, i–k). This indicates that samples dyed by TS7, TS9 and TS12 surfactant can obtain darker shades than samples dyed by conventional water-based method, highlighting the benefit of using SAE-based biodegradable surfactant for non-aqueous dyeing of cotton in octane solvent medium.

Nevertheless, when TS20 surfactant and higher moles EO and HLB value are used (Figs. 5d, h and l), the dyed cotton samples exhibit higher reflectance percentage, and thus a paler shade compared with conventional water-dyed samples. This further validates the importance of moles EO and HLB value as one of the parameters for choosing the most suitable SAE-based biodegradable surfactant for non-aqueous reverse micellar dyeing of cotton fabric.

Besides the reflectance percentage, it can be observed that reflectance curves of both conventional water-dyed cotton samples and SAE-based surfactant-dyed samples are identical in shape, as is evident from the distinct colour characteristics of reactive

Fig. 5 Reflectance curves of the dyed cotton fabrics: **a** BCA TS7; **b** BCA TS9; **c** BCA TS12; **d** BCA TS20; **e** RCA TS7; **f** RCA TS9; **g** RCA TS12; **h** RCA TS20; **i** YCA TS7; **j** YCA TS9; **k** YCA TS12; **l** YCA TS20

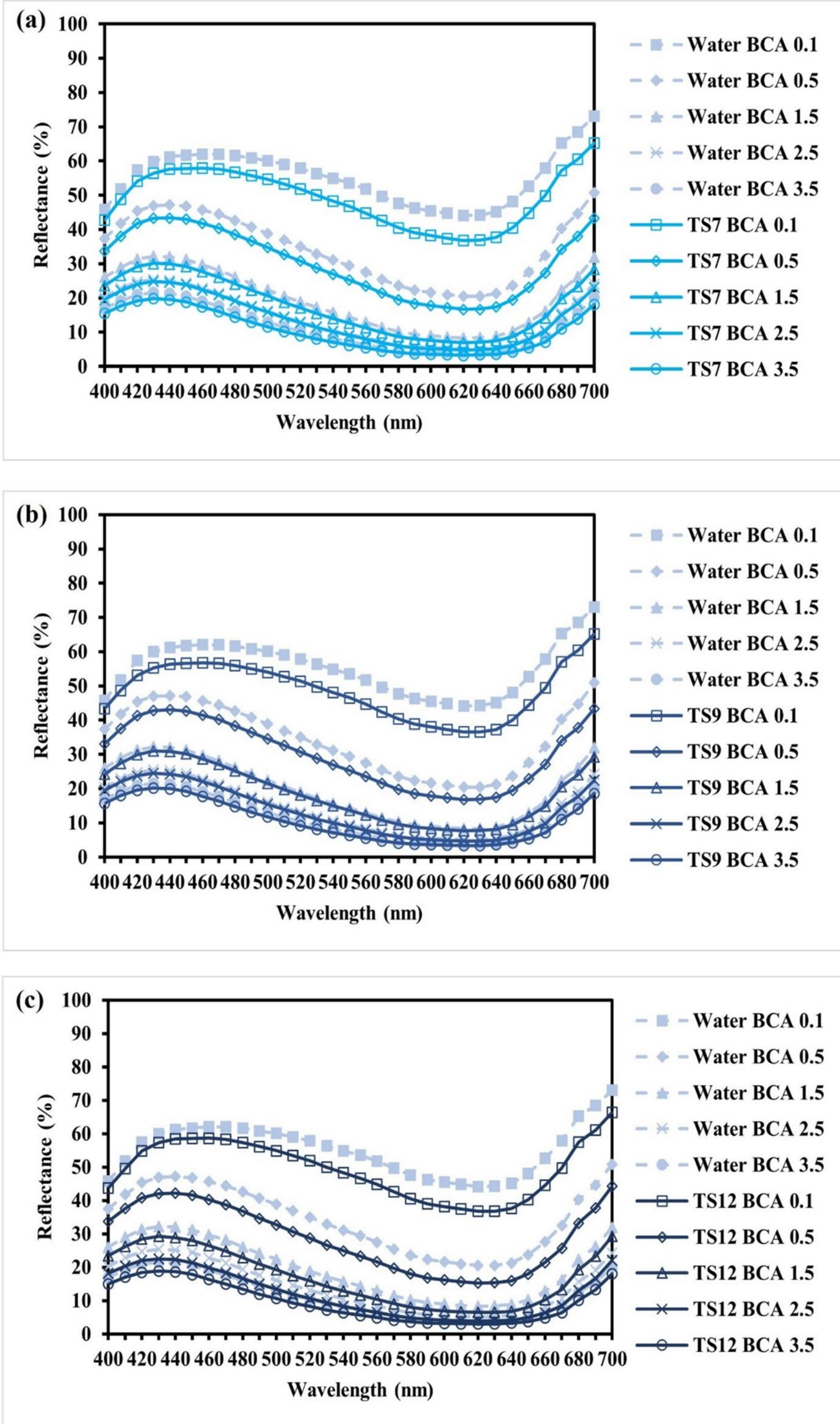


Fig. 5 (continued)

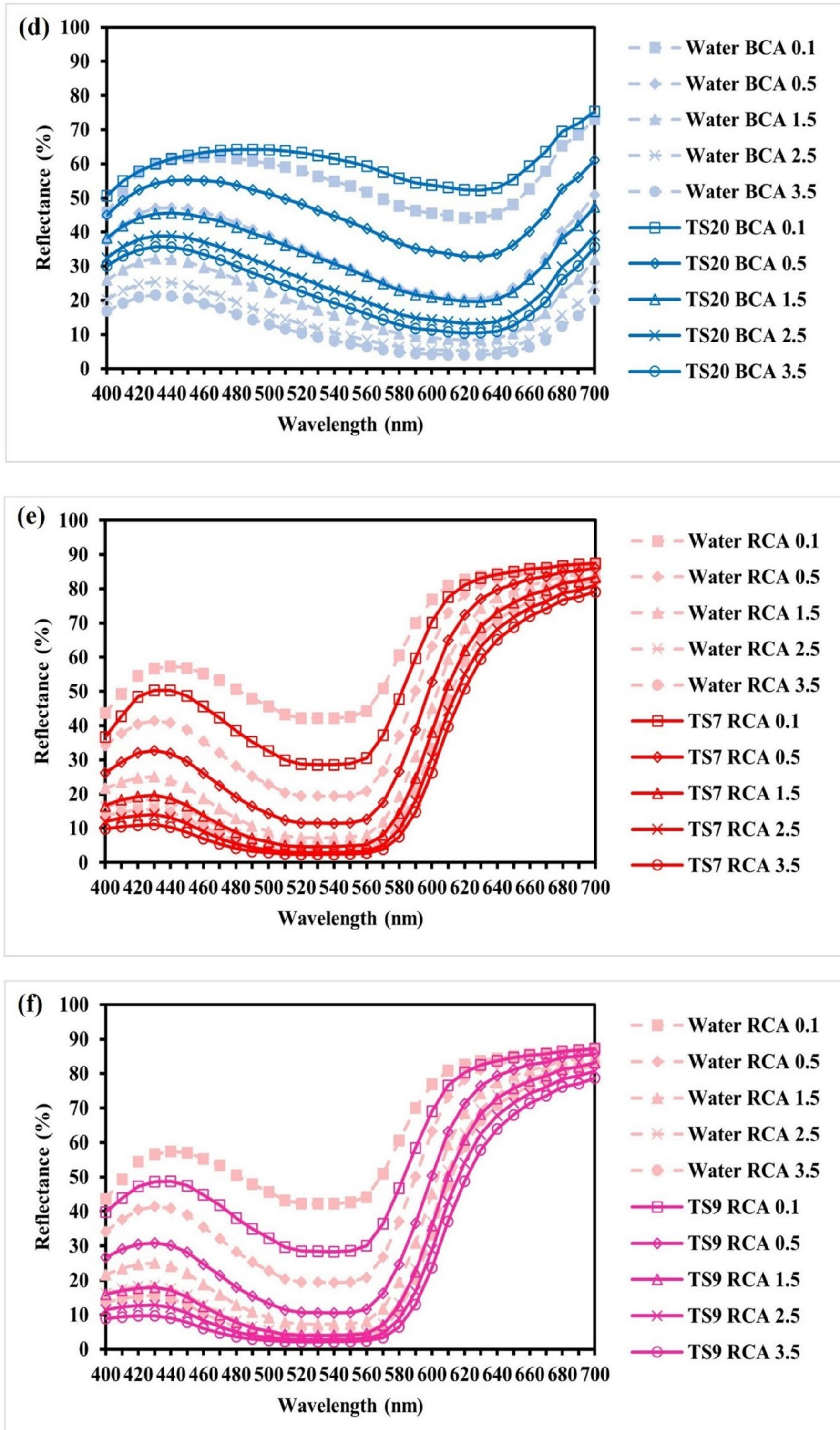
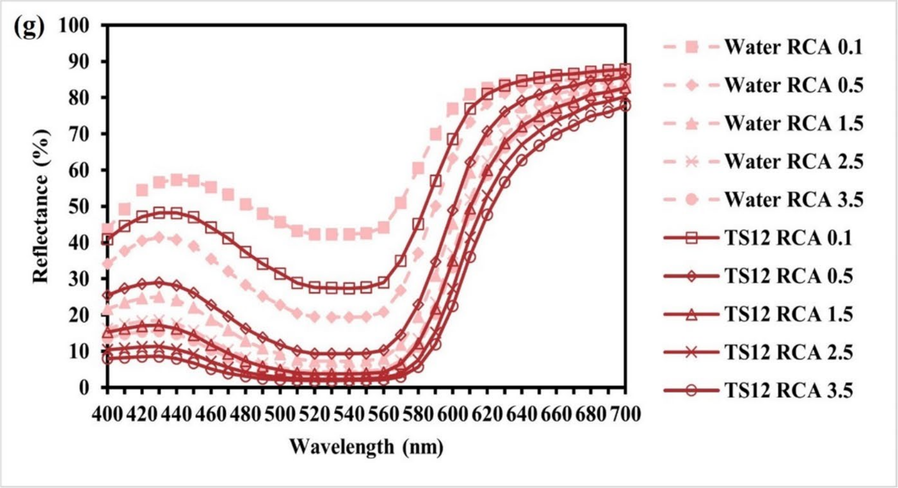
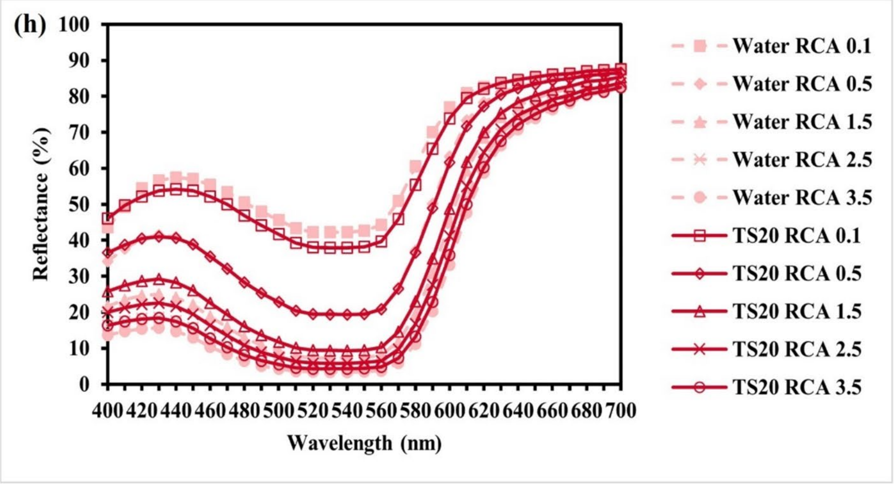
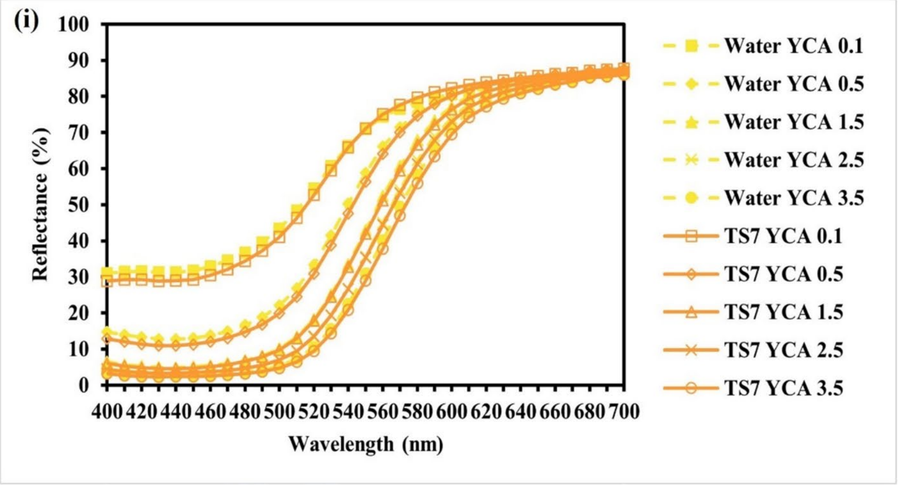


Fig. 5 (continued)







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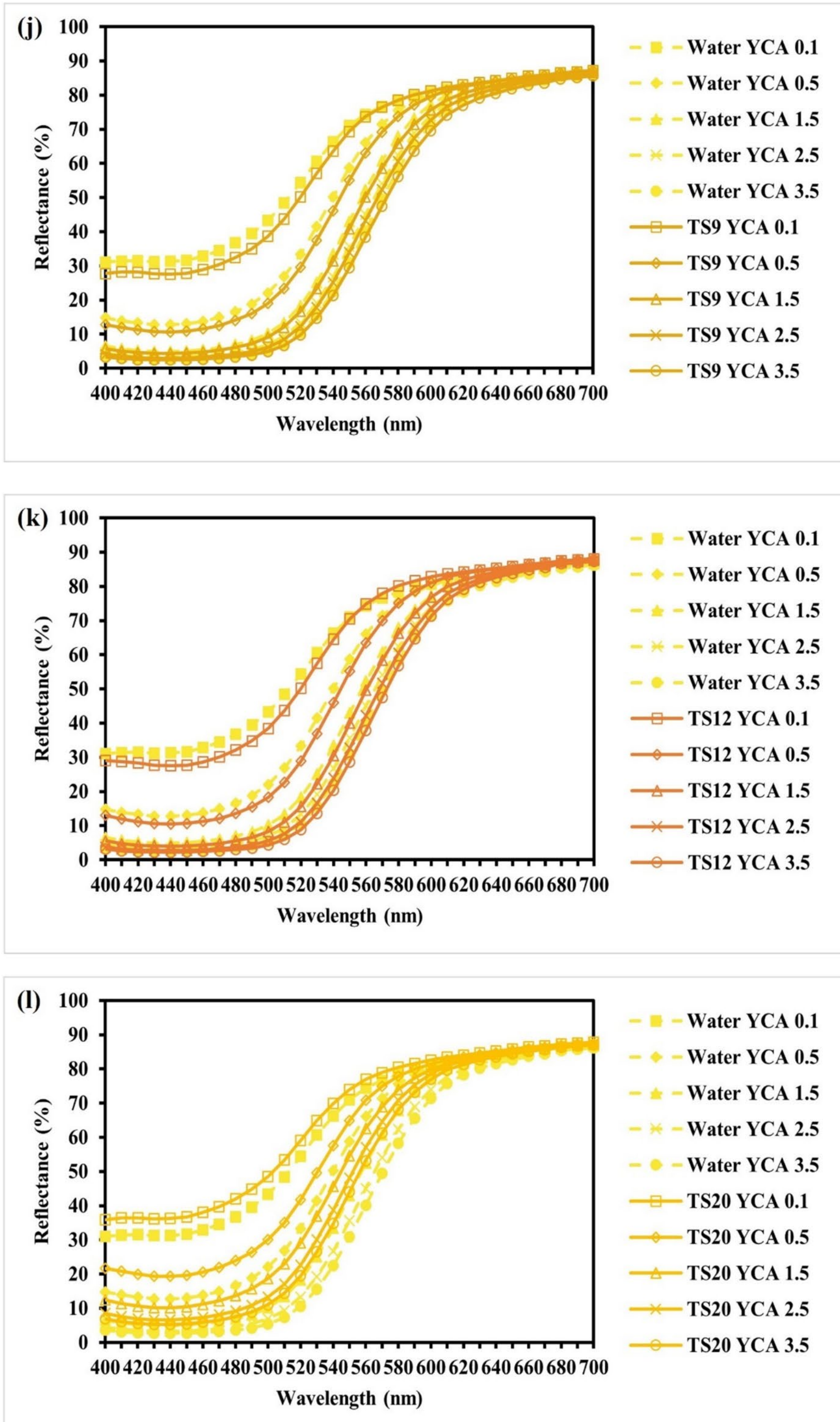


Table 7 RUI of water-dyed and surfactant-dyed samples

Relative unlevelness indices (RUI)												
Colour	Dye conc. (%)	Water	Visual	TS7	Visual	TS9	Visual	TS12	Visual	TS20	Visual
BCA	0.1	0.03	Excellent	0.33	Good	0.07	Excellent	0.05	Excellent	0.02	Excellent
	0.5	0.05	Excellent	0.71	Poor	0.09	Excellent	0.23	Good	0.06	Excellent
	1.5	0.13	Excellent	0.61	Poor	0.62	Poor	0.12	Excellent	0.06	Excellent
	2.5	0.24	Good	0.28	Good	0.15	Excellent	0.46	Good	0.16	Excellent
	3.5	0.15	Good	0.91	Poor	0.28	Good	0.33	Good	0.21	Good
RCA	0.1	0.06	Excellent	0.41	Good	0.11	Excellent	0.12	Excellent	0.09	Excellent
	0.5	0.07	Excellent	0.28	Good	0.51	Poor	0.04	Excellent	0.10	Excellent
	1.5	0.08	Excellent	0.61	Poor	0.63	Poor	0.26	Good	0.11	Excellent
	2.5	0.09	Good	0.62	Poor	0.75	Poor	0.20	Good	0.17	Excellent
	3.5	0.06	Good	0.27	Good	0.68	Poor	0.29	Good	0.21	Good
YCA	0.1	0.03	Excellent	0.05	Excellent	0.09	Excellent	0.05	Excellent	0.03	Excellent
	0.5	0.06	Excellent	0.12	Excellent	0.09	Excellent	0.06	Excellent	0.04	Excellent
	1.5	0.09	Excellent	0.27	Good	0.24	Good	0.14	Excellent	0.10	Excellent
	2.5	0.05	Excellent	0.15	Excellent	0.22	Good	0.08	Excellent	0.08	Excellent
	3.5	0.12	Excellent	0.36	Good	0.20	Good	0.15	Excellent	0.06	Excellent

Remark: RUI < 0.2 = Excellent levelness; 0.2–0.49 = Good levelness; 0.5–1 = Poor levelness; > 1 = Bad levelness (Chong et al. 1992)

dyes (Fig. 5). No peak shift is observed. This verifies that the use of SAE-based non-ionic biodegradable surfactant does not alter the colour properties of the dyed cotton samples as compared to the water-dyed cotton samples.

Colour levelness

Colour levelness of water-dyed and SAE-based surfactant-dyed cotton samples is displayed in Table 7 in terms of relative unlevelness indices (RUI). Both water-dyed, TS12-dyed and TS20-dyed cotton samples can achieve good to excellent levelness (RUI value below 0.5). Although TS20-dyed samples obtain the lowest colour yield (poorer than water-dyed samples as shown in Table 6), these samples gain the best colour levelness (RUI between 0.03 and 0.21) when compared with other dyed samples. The possible reason is stable and good encapsulation of reactive dyes in the water-pool region of TS20 reverse micelle (Fig. 4d) in which the dye molecules are evenly distributed on fabric surface, diffusing into the fibre matrix. Good to excellent levelness (RUI between 0.05 and 0.46) of TS12-dyed samples is attributed to optimisation for stable microenvironment for dye molecule encapsulation having been assured (Fig. 4c), striking a balance between colour yield and levelness of the dyed fabric samples (Tang et al. 2023a).

In case of TS7 and TS9-dyed fabric samples, poor colour levelness is observed (RUI ranged from 0.5 to 1). The worst colour levelness is observed when fabrics were dyed by using SAE-based surfactant with lowest moles EO (TS7) in which the RUI value reaches as high as 0.91. TS9-dyed samples obtained the second poorest colour levelness with RUI values between 0.51 and 0.75. Poor colour levelness of TS7 and TS9-dyed samples may be the result of poor and unstable encapsulation of dye molecules by TS7 and TS9 reverse micelles, illustrating morphologies of irregular shape (Fig. 4a) and triangular shape (Fig. 4b) as discussed in previous sub-section. In addition, it can be seen that poor colour levelness of the TS7-dyed samples is mainly found in case of blue reactive dye whereas TS9-dyed samples have poor colour levelness when red reactive dye is used. This is possibly related to the compatibility and chemistry of the reactive dye in non-aqueous dyeing system (Tang et al. 2023b, 2019).

CIE L*a*b* value

Table 8 presents CIE L*a*b* values of water-dyed and SAE-based surfactant-dyed fabric samples. With

Table 8 CIE L*a*b* value of water-dyed and surfactant-dyed cotton samples

CIE L*a*b*																
Dye	Dye (%)	Water			TS7			TS9			TS12			TS20		
		L*	a*	b*	L*	a*	b*	L*	a*	b*	L*	a*	b*	L*	a*	b*
BCA	0.1	76.61	−6.87	−9.87	70.70	−8.58	−13.00	73.59	−8.81	−10.09	72.27	−8.33	−14.07	81.50	−5.87	−6.87
	0.5	59.29	−10.54	−23.18	53.00	−11.56	−25.28	57.95	−11.10	−21.18	53.55	−11.30	−26.67	71.37	−9.54	−18.18
	1.5	43.05	−12.02	−30.70	38.12	−12.03	−31.40	44.96	−12.08	−26.75	39.10	−12.12	−32.53	61.00	−11.02	−25.70
	2.5	35.46	−11.47	−32.63	32.45	−10.96	−32.75	37.52	−11.02	−28.77	31.53	−11.09	−34.00	53.89	−10.47	−27.63
	3.5	31.32	−10.59	−32.73	28.10	−9.72	−32.41	32.36	−9.76	−29.45	27.60	−9.79	−33.60	49.96	−10.03	−26.73
RCA	0.1	81.19	23.92	4.77	74.97	33.72	4.60	74.92	34.97	4.69	74.41	35.01	4.79	81.12	25.92	4.77
	0.5	69.94	41.61	7.28	62.50	49.57	10.18	62.31	50.09	10.71	61.46	51.00	11.61	69.62	39.58	7.08
	1.5	59.11	53.25	14.71	54.35	55.62	19.89	53.96	55.61	20.00	53.18	56.68	20.33	64.86	47.25	13.71
	2.5	54.44	56.32	19.59	50.05	56.14	24.62	49.64	56.65	25.12	48.63	57.70	26.74	59.55	50.32	17.59
	3.5	52.19	56.95	22.14	47.23	56.74	27.44	46.60	56.70	28.53	45.88	56.90	30.00	56.21	50.95	19.14
YCA	0.1	87.79	10.88	39.00	87.90	12.00	42.28	87.87	12.22	43.51	87.79	13.26	44.76	89.63	9.88	36.00
	0.5	83.79	21.39	65.61	83.84	23.39	69.30	83.45	23.89	69.28	82.97	24.48	70.76	87.79	18.39	55.61
	1.5	78.78	31.73	83.97	78.34	32.70	85.83	78.23	32.45	86.16	77.84	34.01	87.62	80.30	27.73	73.97
	2.5	75.99	35.96	88.84	75.54	37.08	89.64	75.35	37.29	89.58	75.07	38.34	91.76	77.55	31.96	78.84
	3.5	73.98	38.62	90.53	74.17	39.63	91.36	74.67	39.07	90.74	73.32	40.68	93.00	75.77	33.62	80.53

regard to BCA reactive dye, it is apparent that both TS7 and TS12-dyed samples obtained the lowest L* value, followed by water-dyed and TS9-dyed samples, while TS20-dyed samples acquired the highest L* value, indicating the lightest of TS20-dyed samples. For the a* value, water-dyed samples possess more green elements than TS20-dyed samples whereas TS7, TS9 and TS12-dyed samples are generally redder than water-dyed and TS20-dyed samples. For the b* value, TS12-dyed samples are bluer than the water-dyed, TS7-dyed and TS-9-dyed samples while TS-20 contains fewer blue elements compared with the others.

When RCA reactive dye was used, TS12-dyed samples had the lowest L* value (darkest), followed by TS9-dyed samples, TS7-dyed samples and water-dyed samples whereas TS20-dyed samples had the highest L* value (lightest). For the a* value, TS12-dyed samples generally are redder than TS7, TS9 and water-dyed samples which have similar a* values. Among all the samples, TS20-dyed samples have the least red elements. For the b* value, TS12-dyed samples have the highest b* value (yellowest), followed by TS9-dyed samples, TS7-dyed samples and water-dyed samples while TS20-dyed samples had the lowest b* value (bluest).

When fabrics dyed with YCA reactive dye were examined, TS12-dyed samples had the lowest L* value (darkest). Both TS7, TS9 and water-dyed samples had similar L* values while TS20-dyed samples exhibit the highest L* value (lightest). TS12-dyed samples have a higher a* value (redder) than TS7, TS9 and water-dyed samples whereas TS20-dyed samples acquire the lowest a* value (greenest). TS12-dyed samples exhibit the highest b* value (yellowest). Both TS7, TS9 and water-dyed samples denote similar a* values while TS20-dyed samples have the lowest b* value (bluest).

Visual colour appearance

Figure 6 illustrates the visual colour appearance of the water-dyed and SAE surfactant-dyed fabric samples. Generally speaking, TS7-dyed, TS9-dyed and TS12-dyed fabric samples possess similar colour shades, without noticeable shade difference. They generally have the darkest shade when compared with water-dyed and TS20-dyed samples. The visual appearance of water-dyed samples, though they exhibit lighter shades than TS7, TS9 and TS12-dyed samples, show slightly darker shades than TS20-dyed samples. Apparently, TS20-dyed samples possess the lightest shade among all the dyed samples. This finding verifies the colour yield of the dyed samples as discussed in colour yield section and validates that SAE surfactant of optimum moles EO, chain length and HLB value are essential to achieve the best dyeing properties of the cotton fabrics and the best dyeing performance in reverse micellar dyeing system.

SEM image

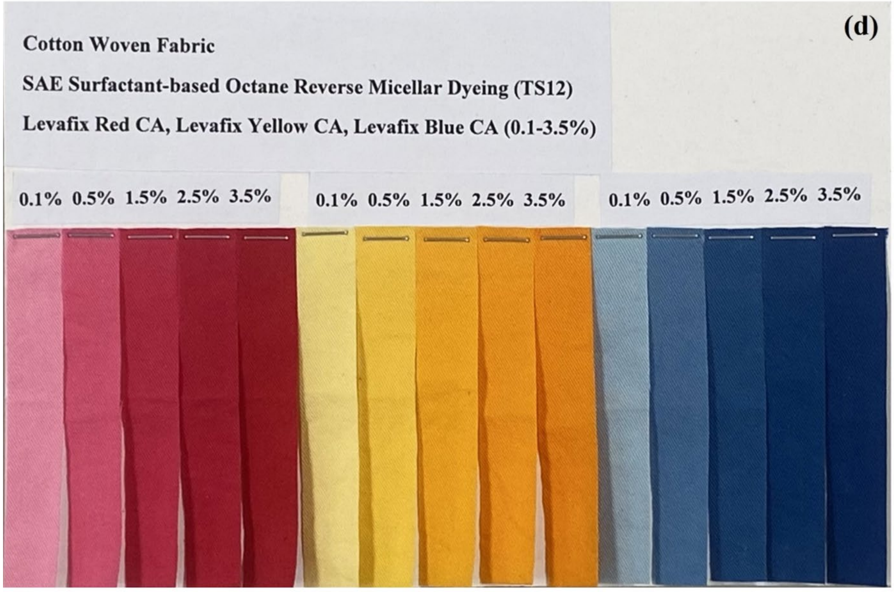
Figure 7 shows the SEM images of pristine, water-dyed and SAE surfactants-based reverse micellar dyed cotton samples. It is clear that both water-dyed and SAE surfactants-based reverse micellar dyed cotton samples do not have significant fibre surface damages after the dyeing process (Fig. 7b–f), when compared with pristine cotton sample (Fig. 7a). This indicates that the use of different properties of SAE surfactants does not cause severe damage to the surface of the cotton fibre. Among the samples dyed by SAE surfactants with different properties, TS12 and TS20 surfactants (Fig. 7e–f) cause noticeably lower degree of fibre damages than TS7 and TS9 surfactants with the presence of small amount of microfibrils on cotton fibre surface as evidenced in Fig. 7c–d.

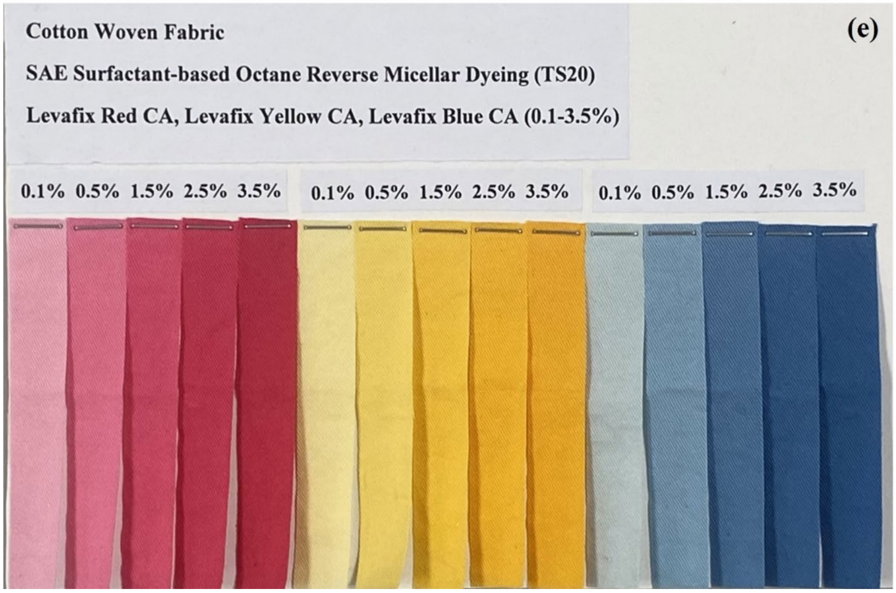
Washing fastness

The colour change and staining of water-dyed and surfactant-dyed samples after AATCC washing fastness test is depicted in Table S1. With regard to the colour change, both water-dyed and SAE-based surfactant-dyed samples obtain excellent rating of 4–5 as colour properties of the washed samples, to a large extent, are the same as they were after the dyeing and rinsing process. Concerning colour staining, water-dyed samples reveal excellent rating of 4–5 while SAE-based surfactant-dyed samples show good to excellent rating of 4–4.5. TS7-dyed and TS9-dyed samples with BCA and RCA reactive dyes have slightly lower rating (rating 4) than TS12-dyed and TS20-dyed samples (rating 4–5). These findings indicate that both water-dyed and SAE-based surfactant-dyed samples are thoroughly rinsed without severe colour bleeding and fading, guaranteeing the removal

Fig. 6 Visual images of the dyed cotton fabrics: **a** water; **b** TS7; **c** TS9; **d** TS12; and **e** TS20

Fig. 6 (continued)





of unfixed dye and chemical residues and the accuracy of the colour measurement results of the dyed samples.

Crocking fastness

The colour staining results of water-dyed and SAE-based surfactant-dyed samples after crocking fastness test are presented in Table S2. It can be seen that water-dyed samples have excellent and constant fastness against dry and wet crocking with rating of 4–5. SAE-based surfactant-dyed samples gained excellent fastness rating of 4–5 against dry and wet crocking when they were dyed at low dye concentrations from 0.1% to 1.5%. Samples dyed at higher dye concentrations, except TS20-dyed samples, result in slightly lower, but still good crocking fastness rating of 4. These findings indicate that both water-dyed and SAE-based surfactant-dyed samples have good colour resistance against the imposed rubbing action.

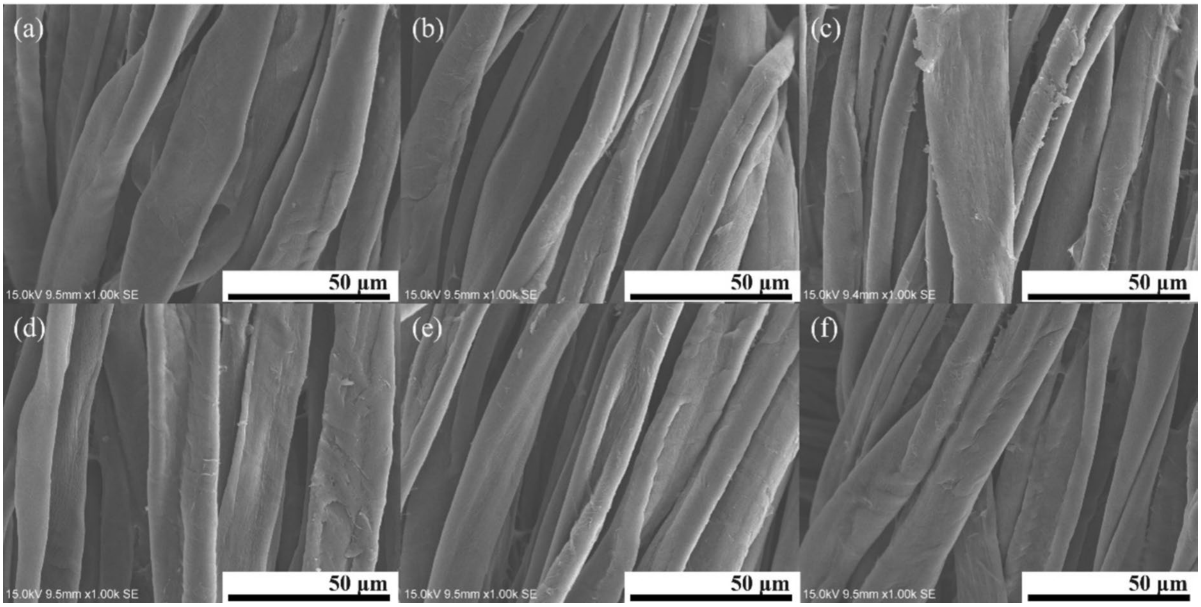


Fig. 7 SEM images of undyed and 3.5% red-dyed cotton samples: **a** undyed; **b** water-dyed; **c** TS7; **d** TS9; **e** TS12; and **f** TS20 (magnification: 1000×)

Tensile strength and breaking extension

Tables S3 and S4 show the tensile strength (N) and breaking elongation (mm and %) of undyed, water-dyed and SAE-based surfactant-dyed cotton samples. As Table S3 shows, both water-dyed (317–346 N) and SAE-based surfactant-dyed (304–381 N) samples suffer certain degree of strength loss after the dyeing process compared with the undyed samples (415 N) in warp direction. Among the dyed samples, TS20-dyed samples have the least strength loss (363–381 N) which is even smaller than that of water-dyed samples (317–346 N). This indicates that the samples dyed by TS20 SAE-based surfactant may undergo a dyeing process which is milder than the process samples dyed by water are subjected to. In the weft direction, most of the surfactant-dyed samples generally gain strength after the dyeing process, except TS9-dyed samples with 3.5% BCA reactive dyes while water-dyed samples generally lose strength loss after the conventional dyeing process. The degree of strength loss, to a large extent, is related to dyeing temperature, pH value and the chemicals used during the dyeing process (Zhang et al. 2021).

Regarding the breaking extension of the cotton samples (Table S4), both water-dyed and SAE-based surfactant-dyed samples have breaking extension similar to undyed samples in warp as well as weft direction. Undyed samples have breaking elongations of 16.65 mm (22.2%) and 9.42 mm (12.56%) in warp and weft direction respectively. The breaking elongations of water-dyed samples in warp and weft directions are 16.18–16.68 mm (21.57–22.24%) and 8.85–9.16 mm (11.48–12.21%) respectively. In case of SAE-based surfactant-dyed samples, breaking elongations of the dyed samples are 16.04–18.93 mm (21.25–25.24%) and 8.45–10.18 mm (11.36–13.57%). This finding validates that neither conventional nor non-aqueous dyeing process causes adverse effect to the breaking elongation of the dyed samples.

Conclusion

The suitability and dyeing performance of various SAE-based biodegradable surfactants for forming reverse micelles and acting as dye carriers for cotton fabric dyeing in a non-aqueous octane medium are investigated in this work. Reactive dye molecules are encapsulated in the water-pool of these reverse micelles. TEM images reveal that different

SAE-based surfactants form reverse micelles of varying shapes.

Dyeing of cotton fabrics is conducted using different morphologies of reverse micelles as dye carriers. The properties of the dyed samples are evaluated and compared to those dyed in conventional water-based system. Results reveal that TS12 surfactant-dyed samples achieve the highest colour yield, followed by TS7 and TS9 surfactant, whereas TS20 surfactant-dyed samples obtain the lowest colour yield. Surfactant of optimum moles EO and HLB value can achieve the best dyeing effect on cotton fabric.

The reflectance curves of water-dyed and surfactant-dyed samples are identical, without any peak shift, indicating that SAE-based surfactants do not alter the colour properties of the fabric. TS20 surfactant-dyed samples achieve excellent levelness, followed by TS12, while TS7 and TS9 show variable levelness from excellent to poor based on the colour and concentration of the reactive dyes. CIE L*a*b* values of the dyed samples are also examined.

Both water-dyed and SAE-based surfactant-dyed samples show good to excellent washing and crocking fastness. While most dyed samples lose strength in the warp direction, they gain strength in the weft direction after both conventional and non-aqueous dyeing processes. SEM images reveal that different SAE-based surfactants can cause varying degrees of surface damage to cotton fibres. TS12 surfactant is identified as the most suitable SAE-based surfactant for reverse micelle formation, optimizing dyeing properties in a non-aqueous medium in this study.

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Author contributions C.K. conceptualised and designed the study. Material preparations, experiments and data collections were performed by Y.T., H.L., C.L., J.Y. and Y.W. SEM was performed by S.J. and Y.T. TEM was performed by Y.T. All authors engaged in data analysis and investigation. Y.T. and C.L. prepared the first draft of the manuscript. C.K. and Y.T. reviewed and edited the manuscript. Y.T. contributed figures and tables of the manuscript. C.K. allocated all the resources and supervised the whole process of the study. All authors have read and agreed to the final version of the manuscript.

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Data availability No datasets were generated or analysed during the current study.

Declarations

Conflict of interest The authors declare no competing interests.

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