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A Study of Color Fixation Agents in Secondary Alcohol Ethoxylate-Based Reverse Micellar Cotton Dyeing System with Reactive Dyes

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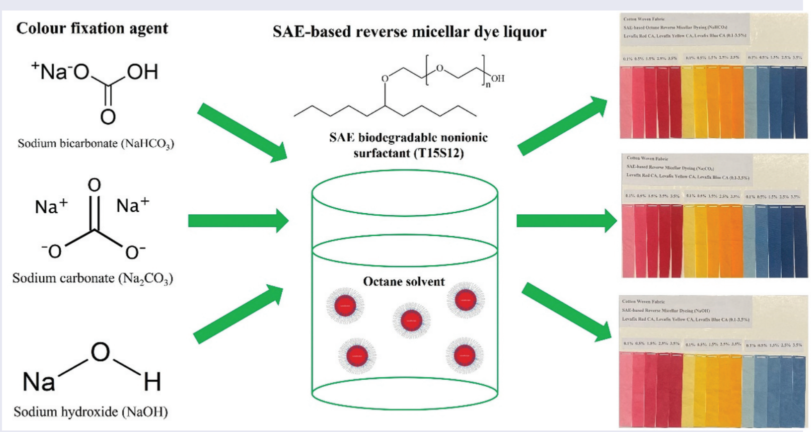
ABSTRACT

Influence of color fixation agent (CFA) and dyebath pH in secondary alcohol ethoxylate (SAE)-based reverse micellar dyeing system of cotton with reactive dyes was investigated and compared with water-based dyeing system using different alkali (i.e. CFA) such as: (i) sodium bicarbonate ($NaHCO_3$), (ii) sodium carbonate (Na_2CO_3) and (iii) sodium hydroxide (NaOH). The color, tensile strength, fastness and surface morphological properties of dyed samples were examined. Experimental results showed that samples dyed with Na_2CO_3 can achieve the highest color yield, followed by $NaHCO_3$ and NaOH in both water-based and reverse micellar dyeing system. The color yield and reflectance percentage of the dyed cotton samples were found to be closely related to the dyebath pH value. Relative unlevelness indices (RUI) also reflected that cotton samples dyed with NaOH in reverse micellar dyeing system have a higher chance of color unlevelness when compared with $NaHCO_3$ and Na_2CO_3. Tensile strength results affirm that higher alkalinity of dyebath could cause higher strength loss to the colored cotton samples. Both cotton samples dyed by water and SAE approach showed good to excellent color fastness properties while scanning electron microscopic (SEM) images exhibit that the use of different alkalis may cause some damage to the cotton fiber.



KEYWORDS
Cotton; reactive dye; alkali; secondary alcohol ethoxylate; reverse micelle; dyeing

关键词
棉花; 活性染料; 碱; 仲醇乙氧基化物; 反胶束; 染色

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摘要

研究了固色剂（CFA）和染浴pH值对基于仲醇乙氧基化物（SAE）的棉活性染料反胶束染色体系的影响，并与使用不同碱（即CFA）的水性染色体系进行了比较，例如:（i） 碳酸氢钠（NaHCO3），（ii）碳酸钠（Na2CO3）和（iii）氢氧化钠（NaOH）. 对染色样品的颜色、拉伸强度、牢度和表面形态特性进行了检测. 实验结果表明，在水性和反胶束染色体系中，用Na2CO3染色的样品可以获得最高的显色率，其次是NaHCO3和NaOH. 染色棉样品的显色率和反射率与染浴pH值密切相关. 相对不平度指数（RUI）也反映出，与NaHCO3和Na2CO3相比，在反胶束染色系统中用NaOH染色的棉花样品有更高的颜色不平度机会. 拉伸强度结果证实，染浴碱度越高，有色棉样品的强度损失越大. 用水和SAE方法染色的棉花样品都显示出良好到优异的色牢度性能，而扫描电镜（SEM）图像显示，使用不同的碱可能会对棉纤维造成一些损伤.

Introduction

Conventional dyeing of cotton fabric with reactive dye relies on three main phases: (i) exhaustion phase which involves the use of salt or electrolyte; (ii) fixation phase in which alkali is used to promote covalent bond fixation of dye on fibre under alkaline condition; and (iii) washing off phase in which soaping are used to remove residuals of salt, alkali, unfixed and hydrolysed dye (Aspland 1992; Broadbent 2001; Lewis 2014). However, this conventional method poses a detrimental threat to the ecosystem and living environment since it requires the use of substantial amount of water while producing colored effluents composed of residual dyes, electrolytes and alkalis (Hossain et al. 2021; Khatri et al. 2015; Uddin 2021).

To mitigate the adverse environmental effects of conventional water-based dyeing approach for cotton, extensive attempts have been made to switch dyeing to an environmentally friendly and sustainable method. Periyasamy (2022) and Rahman et al. (2023) reported natural dyeing of cotton using fruit and root extracts respectively. Xia et al. (2018) investigated the use of ethanol-water dye liquor for dyeing cotton fiber. Luo et al. (2018) and Abou Elmaaty et al. (2022) used supercritical carbon dioxide as a medium for dyeing cotton fabric. Pei et al. (2022), Wei et al. (2021), and A. Wang et al. (2023) dyed cotton in silicone oil system, hydrophobic deep eutectic solvent (HDES) system and ternary solvent system without the presence of salt. Work has also been done by using dioctyl sodium sulfosuccinate surfactant for dyeing of cotton with low liquor ratio (Mamun Kabir, Sk, and Koh 2021). Several attempts have also been made on using radiation tools for modifying cellulosic fibers to enhance their dyeability (Adeel et al. 2017, 2018; Bhatti et al. 2012).

Instead of using those dyeing methods, recent works systematically focus on the use of reverse micelles, which are nano-sized aggregates in spherical shape formed by self-assembly of surfactant in solvent medium, as a reactive dye carriers which would be a promising way to dye cotton in a sustainable salt-free and water-saving manner (Tang and Kan 2020; Wang et al. 2016). Our previous works explored the properties of cotton fabrics dyed by poly(ethylene glycol) (PEG)-based surfactant (Tang, Lee, et al. 2017; Tang et al. 2019b, 2023b) and alkyl polyglucoside (APG) surfactant-based (Lee et al. 2022; Tang et al. 2023, 2023) reverse micelles. In addition, when rhamnolipid biosurfactant-based reverse micelles was used, it was found that the dyeing of cotton fiber can be achieved under octane medium even in alkali-free manner with optimized parameters (Tang et al. 2023c) and this dyeing system can be applied in computer color matching (Tang et al. 2023a). Recently, we have optimized the use of secondary alcohol ethoxylate (SAE) surfactant for developing reverse micellar dyeing system for achieving a salt-free dyeing of cotton in octane medium with reactive dyes (Tang et al. 2023). However, further study concerning the influence of alkali concentration and pH value in this dyeing system is needed.

Alkali is used as color fixation agent (CFA) in dyeing cotton with reactive dye in water-based some novel non-aqueous-based dyeing systems in order to create and maintain proper dyebath pH value for dye-fiber covalent fixation to take place. It can help the acidic disassociation of hydroxyl groups of cellulose, forming cellulosate ions capable of reacting with dye molecules through nucleophilic addition or substitution (Tang et al. 2019a). Common CFA used for dyeing of cotton generally include: (a) sodium bicarbonate ($NaHCO_3$, weak alkali); (b) sodium carbonate (Na_2CO_3, medium alkali); and (c) sodium hydroxide (NaOH, strong alkali). The strength of alkali used in the dyeing process is based on reactivity of the selected dyestuff (Bhuiyan et al. 2013).

Since the effect of CFA and its influence on the SAE surfactant-based reverse micellar dyebath pH has not yet been found in the literature. Therefore, it is essential to study this important factor and being a continuation of our previous study (Tang et al. 2023), this work aims at examining the dyeing performance of cotton under previously optimized parameters with the use of different alkali as the CFA. The influence of the CFA alkalinity and dyebath pH value between SAE-based and water-based dyeing systems would be compared. The influence includes: (i) investigating the dyebath pH of SAE reverse micellar system with the use of different alkali ($NaHCO_3$, Na_2CO_3 and NaOH); (ii) comparing the properties of samples in water and reverse micellar system with different CFAs; (c) assessing the tensile strength, washing and crocking fastness properties of cotton samples dyed in these two systems; and (d) evaluating the surface morphology of cotton samples dyed in the two systems with the use of scanning electron microscopy.

Experimental

Cotton fabric sample

Pure cotton woven fabric (127 ends and picks per cm; and fabric weight of 139 g/m^2) was pre-washed with detergent (2 g/L) at 49°C for 45 minutes. The pre-washed cotton fabrics were then tumble-dried and subsequently conditioned (24 hours, 20 ± 2°C and 65 ± 2% relative humidity (RH)) prior to a series of dyeing experiments and measurements.

Reagents

Figure 1 shows the chemical structure of secondary alcohol ethoxylated (SAE) surfactant and it was purchased from Sigma Aldrich. Octane (98+% purity) and n-octanol (>99% purity) of reagent grade were purchased from Alfa Aesar. Sodium chloride (NaCl) was purchased from VWR. Sodium bicarbonate ($NaHCO_3$), sodium carbonate anhydrous (soda ash, Na_2CO_3), and sodium hydroxide (NaOH) were supplied by Sigma Aldrich. Reactive dyes, Levafix Red CA (RCA), Levafix Blue CA (BCA), and Levafix Yellow CA (YCA), were obtained from Dystar (Shanghai, China) and used directly without further purification.

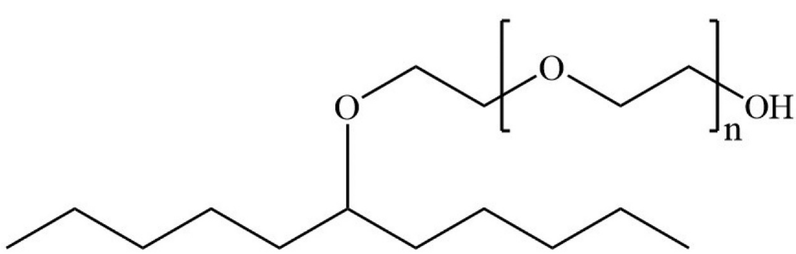

Figure 1. The chemical structure of tergitol type 15-S-12 biodegradable sae-based nonionic surfactant (n = 12).

Dyeing of cotton with different CFA with water

Water-based dyeing of cotton fabric was conducted as per recipe recommended by dye supplier (Table 1). Liquor-to-goods ratio (LR) of 20:1 was used to assure leveling of the dyed samples. NaCl was used as exhaustion agent while different types of CFAs ($NaHCO_3$, Na_2CO_3, or NaOH), in similar amounts of 5 g/L (Table 1) were used as CFA and for ease of comparison. The procedure of water dyeing of cotton is presented in Figure 2. The dye liquor was first prepared with relative concentration of reactive dye and relative amount of NaCl. Pre-washed and conditioned cotton fabric samples was then soaked in the dyeing liquor and put in the thermostatic water bath (at 30°C) for 10 minutes with shaking. The water bath temperature was then increased to 70°C to facilitate dyeing, for 40 minutes. After that, 5 g/L alkali ($NaHCO_3$, Na_2CO_3, or NaOH), serving as CFA, was added for dye fixation. Subsequently, rinsing was carried out to allow the dyed fabric to be washed with detergent solution twice (2 g/L at 50°C), cold rinsed with water, air-dried on a hanger and conditioned (20 ± 2°C and 65 ± 2% RH) for 24 hours.

Formation of SAE-based reverse micelle

The procedure followed for SAE-based reverse micelle formation of reactive dye in the interior water-pool was similar to our previous work (Tang et al. 2023). A solution mixture was first formed by pre-mixing SAE biodegradable nonionic surfactant with cosurfactant (n-octanol). Subsequently, octane solvent was added into the mixture with continuous stirring at room temperature to form reverse micelle with empty core (water-pool). Reactive dye, in aqueous form, was finally injected dropwise in the prepared reverse micellar solution and then being encapsulated in the water-pool of the SAE-based reverse micelles.

Table 1. Water dyeing recipe for cotton fabric.

Liquor ratio 50:1, 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	g/L	5	5	5	5	5

Remarks: Alkali = NaHCO3, Na2CO3, or NaOH.

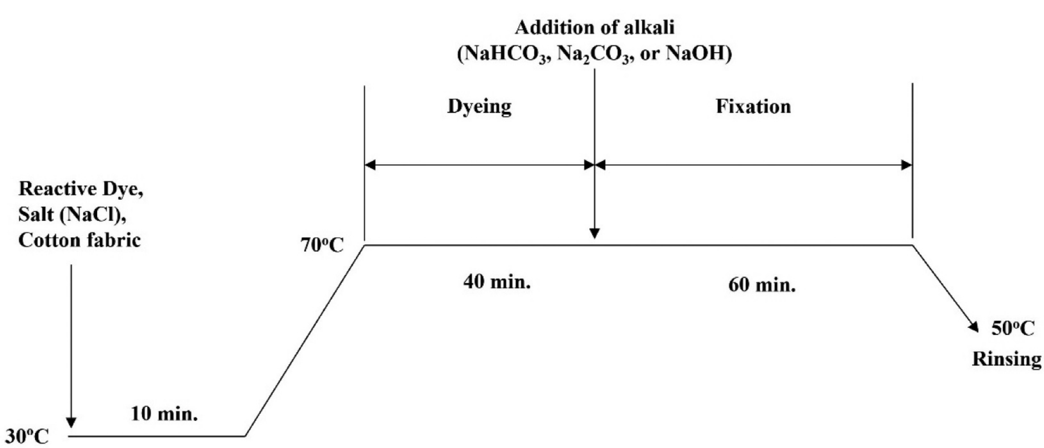

Figure 2. Workflow of water dyeing of cotton fabric (with NaCl).

Recipe for SAE-based reverse micellar salt free dyeing of cotton fabric

The dyeing parameters optimized in our previous work were directly used as the recipe for dyeing of cotton fabric without the use of salt (Tang et al. 2023). The dyeing medium is octane. The recipe details are displayed in Table 2.

SAE-based reverse micellar dyeing of cotton with different CFAs

The mechanism of SAE-based reverse micellar salt free dyeing of cotton was illustrated in Figure 3 accompanied with the dyeing profile as shown in Figure 4. Pre-washed cotton fabric sample was soaked in the SAE reverse micellar dyeing liquor and put in a thermostatic water bath for 10 minutes with shaking at 30°C. Dyeing was then implemented for 40 min when the bath temperature was increased to 70°C. Alkali ($NaHCO_3$, Na_2CO_3, or NaOH) of different amounts (0.03 to 0.07 g/g) corresponded to different dye concentrations (Table 2), serving as CFA, was then added to implement fixation for 60 min. Subsequently, the dyed fabric samples were washed with detergent solution twice (2 g/L at 50°C), rinsed with tape water, air-dried, and finally conditioned (20 ± 2°C and 65 ± 2% RH) for 24 hours.

pH value measurement

pH value of the water-based dye liquor and the SAE dye liquor after addition of different alkali ($NaHCO_3$, Na_2CO_3, or NaOH) was measured by pH meter (Eutech Instruments, Singapore).

Table 2. Dyeing recipe for SAE reverse micellar dyeing in octane medium.

SAE reverse micellar dyeing recipe					
Surfactant to water mole ratio				1:20	
Surfactant to co-surfactant mole ratio				1:8	
Solvent to cotton ratio (v/w)				10:1	
Water-pool volume for dye (mL)				0.5	
Water-pool volume for alkali (mL)				0.3	
Dyeing time (min)				40	
Fixation time (min)				60	
Dyeing and fixation temperature (°C)				70	
Dye concentration (% o.w.f.)	0.1	0.5	1.5	2.5	3.5
Alkali to cotton weight ratio (g/g)	0.03	0.04	0.05	0.06	0.07

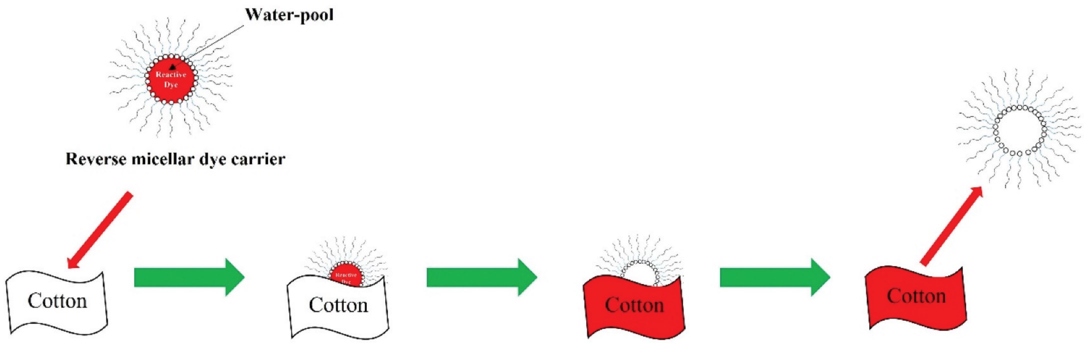

Figure 3. Dyeing mechanism of reverse micellar approach on cotton fabric.

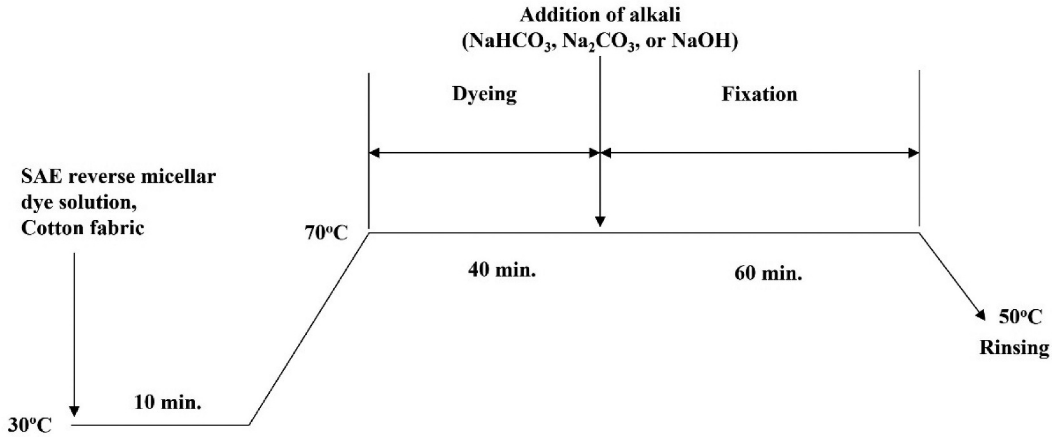

Figure 4. Workflow of salt-free SAE reverse micellar octane dyeing of cotton fabric.

Color yield

Cotton fabric samples dyed by different kinds of alkali in water and reverse micellar octane medium were examined by SF650 Spectrophotometer (DataColor International, USA) from 400 nm to 700 nm. The measuring parameters and conditions were: (i) medium aperture is 20 mm in diameter; (ii) light source is illuminant D_{65}; (iii) standard observer is 10°; (iv) sample was folded twice to guarantee opacity; (v) only face side of the cotton fabric samples was measured; (vi) date was collected within 400 nm to 700 nm at 10 nm; and (vii) four measurements of each sample were taken and averaged (Tang et al. 2023). The K/S value was then calculated by Equation (1) and summed from 400 nm to 700 nm to obtain K/S_{sum} value.

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

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

*CIE L*a*b* value*

The CIE $L^*a^*b^*$ values of the cotton fabric samples dyed by different kinds of alkali in water and octane medium was measured with the use of SF650 Spectrophotometer (DataColor International, USA) and the measuring parameters and conditions were same as "Color Yield" measurement (Tang et al. 2023).

Color levelness assessment

The color levelness of the cotton fabric samples dyed by different alkali in water and octane medium was assessed by Relative Unlevelness Indices (RUI) (Chong, Li, and Yeung 1992). Four spots from each dyed cotton fabric sample were randomly chosen for evaluation as per our previous study using same apparatus and parameters as mentioned in color yield section (Tang et al. 2023). Calculation of RUI value of each sample was done by using Equation (2).

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

where s_λ: standard deviation of reflectance value; $\overline{R}$: reflectance value; V_λ: photopic relative luminous efficiency function (Chong, Li, and Yeung 1992).

Scanning electron microscopy (SEM)

The fiber surface morphology and damage caused to cotton dyed samples by different kinds of alkalis was evaluated by VP-SEM model SU1510 scanning electron microscope (Hitachi, Japan).

Color fastness to washing

The color fastness to washing of the cotton fabrics dyed by different alkali in water and octane medium was tested by using AATCC Test Method 61–2013, Test Number 2A and rating was given by the use of gray scale according to the AATCC Evaluation Procedure 1–2012 and AATCC Evaluation Procedure 2–2012 for color change and staining respectively.

Color fastness to crocking

The color fastness to crocking (color staining) of the cotton fabrics, in wet and dry state, dyed by different alkali in water and octane medium was performed by AATCC Test Method 8–2013 using white cotton cloth and rated by gray scale in accordance with AATCC Evaluation Procedure 2–2012 for staining.

Tensile properties

Grab Test (ASTM standard D5034) was performed to evaluate tensile strength and elongation at break of cotton fabric samples dyed by different kinds of alkalis in water and octane medium (3.5%).

Results and discussion

Color yield

Color yields of the dyed cotton fabric samples with different CFAs are shown in Table 3. Cotton fabric samples dyed by using SAE-based system with Na_2CO_3 show the highest color yield, followed by SAE-based system with $NaHCO_3$, water-based system with Na_2CO_3, water-based system with $NaHCO_3$ and water-based system with NaOH, whereas SAE-based system with NaOH registers the lowest color yield (Sequence: SAE Na_2CO_3 > SAE $NaHCO_3$ > Water Na_2CO_3 > Water $NaHCO_3$ > Water NaOH > SAE NaOH).

Table 3. Color yield of the dyed fabric samples with different CFA.

K/S_{sum} value										
		Water			SAE					
Color	Dye conc.	$NaHCO_3$	Na_2CO_3	NaOH	$NaHCO_3$	%	Na_2CO_3	%	NaOH	%
BCA	0.1%	4.98	6.19	3.29	8.92	78.84	9.01	45.55	5.34	61.95
	0.5%	13.84	23.57	9.61	29.53	113.35	34.96	48.34	14.27	48.47
	1.5%	32.59	71.35	27.48	74.59	128.91	93.77	31.42	26.36	−4.04
	2.5%	48.53	121.10	46.11	115.29	137.58	161.80	33.61	33.53	−27.28
	3.5%	61.02	163.17	64.14	170.86	180.02	216.79	32.86	38.29	−40.31
RCA	0.1%	4.18	5.43	3.28	10.75	157.33	11.51	111.78	8.46	157.66
	0.5%	9.89	19.35	8.92	40.52	309.63	47.44	145.18	27.47	207.94
	1.5%	25.31	63.43	25.02	107.11	323.14	127.40	100.84	64.54	157.95
	2.5%	37.69	110.34	41.99	165.69	339.56	220.29	99.64	98.75	135.18
	3.5%	51.10	143.89	55.82	219.39	329.37	283.64	97.12	128.39	130.02
YCA	0.1%	6.15	8.25	4.42	9.79	59.18	10.21	23.74	6.92	56.70
	0.5%	17.54	30.07	13.40	34.96	99.34	38.26	27.23	19.14	42.79
	1.5%	45.12	88.90	38.16	91.29	102.34	111.73	25.68	37.29	−2.30
	2.5%	67.51	137.97	64.73	144.74	114.39	171.19	24.08	55.94	−13.58
	3.5%	89.84	177.10	91.21	183.57	104.34	212.80	20.16	63.86	−29.98

When using $NaHCO_3$ and Na_2CO_3 as CFA, SAE-based reverse micellar dyed cotton fabric samples (0.1% to 3.5% o.w.f.) can achieve higher K/S_{sum} value than the conventional water-based dyed cotton fabric samples. This can be explained by the advantages of using reverse micellar salt free dyeing system in which it can minimize the ionization between dye and fiber while enhancing swelling of cotton fiber (Tang, Wang, et al. 2017; Yi, Deng, and Sun 2014).

When NaOH is used as CFA, only cotton fabric sample dyed with (RCA) in reverse micellar salt free system can obtain a higher color yield while blue-dyed (BCA) and yellow-dyed (YCA) cotton fabric samples have poorer color yields when compared with the water-based system. This is probably related to the dyebath pH value which will be further discussed in this paper. It is also estimated that red reactive dye (RCA) may have relatively higher alkaline resistance in reverse micellar dyeing system than blue (BCA) and yellow (YCA) reactive dyes as it is less likely to be influenced by the alkalinity of the dyebath when a similar amount of NaOH was used during the dyeing process.

With respect to the percentage difference in color yield, cotton fabric samples dyed in SAE-based reverse micellar system with $NaHCO_3$ obtain the most significant percentage increase in color yield ranging from 59% to 329%, followed by cotton fabrics dyed with Na_2CO_3 (20% to 145%) when compared with the water-based system. However, in case of NaOH, the SAE-based dyed cotton fabric samples can obtain an increase in color yield only when RCA reactive dye and low concentrations (0.1% and 0.5%) of BCA and YCA reactive dye are used. The use of higher concentrations (above 0.5%) of BCA and YCA reactive dye results in a significant percentage decrease in color yield (-4% to -40% and -2% to -30% respectively). This indicates that the recipe (Table 2) is feasible for SAE-based dyeing system with $NaHCO_3$ and Na_2CO_3 only while modification of recipe is needed if NaOH is used as CFA. The increase in alkalinity of the dyebath may be the cause of the percentage decrease in color yield for SAE-based salt free dyeing of cotton in octane medium with NaOH.

pH value

The pH values of the dyebaths after the addition of different alkalis are listed in Table 4. The pH values of SAE-based dyebath are generally higher than that of the conventional dyebath. When the amount of alkali is fixed in the two systems, it is found that the dyebath in the presence of NaOH as CFA has the highest alkaline pH value (Water: 11.95 to 12.10; SAE: 12.30 to 13.25), followed by dyebath with Na_2CO_3 (Water: 10.39 to 10.50; SAE: 10.30 to 11.05), whereas dyebath in the presence of $NaHCO_3$ has the lowest alkaline pH value (Water: 7.85 to 8.05; SAE: 9.25 to 9.60). However, higher pH value of the dyebath may not increase the dye uptake and color yield strength of the dyed cotton fabric.

Table 3 shows that SAE-based reverse micellar dyed cotton fabric with NaOH obtains the lowest color yield (K/S_{sum} value and there is decrease in color yield at high dye concentration even though the pH value of the dyebath is the highest (Table 4). The possible reason is that high alkalinity of dyebath (above 11) causes an increase of anionic dissociated cellulose. More negatively charged and deprotonated hydroxyl groups may lead to an increase of negative charge on cellulose. This may increase the electrostatic repulsion between dye molecules and cotton fiber, causing lesser dye uptake and a significant decrease in color yield of the dyed cotton fabric (Broadbent 2001; Tayade and Adivarekar 2013). Therefore, optimal dyebath pH

Table 4. pH value of the dyebath after addition of different CFA.

	pH value	
	Water	SAE
$NaHCO_3$	7.85–8.05	9.25–9.60
Na_2CO_3	10.39–10.50	10.30–11.05
NaOH	11.95–12.10	12.30–13.25

value is essential for maximizing the uptake of reactive dye molecules by cotton fabric during the dyeing process.

As listed in Tables 3 and 4, dyebath pH values and color yield of the dyed cotton fabrics in water-based and SAE-based system with $NaHCO_3$ are lower than of Na_2CO_3. This means that the recipes for both systems (Tables 1 and 2) for dyeing of cotton with $NaHCO_3$ can be improved by adding more $NaHCO_3$ to further increase the alkalinity of the dyebath. While the recipes for dyeing cotton with Na_2CO_3 is the most effective since the dyed cotton fabrics can achieve the highest color yield with dyebath pH value between 10 and 11 which is believed to be the closest to the optimal pH value.

Reflectance

The reflectance curves of the dyed cotton samples with different alkalis are presented in Figure 5. Cotton fabric samples dyed by SAE-based system with three different alkalis ($NaHCO_3$, Na_2CO_3, and NaOH) at five color depths (0.1% to 3.5%) can obtain lower reflectance percentage than samples dyed by conventional system except when the samples are dyed with high concentrations of BCA and YCA (above 0.5%) in the presence of NaOH. This reflects that most of the SAE-based dyed cotton fabrics can achieve darker shades than water-based dyed cotton fabrics, consolidating the supremacy of using SAE-based salt free non-aqueous dyeing approach for dyeing of cotton fabrics.

Among the three different alkalis used as CFA, cotton fabric samples dyed with Na_2CO_3 achieve the lowest reflectance percentage, followed by samples dyed with $NaHCO_3$ which have slightly higher reflectance percentage than that of Na_2CO_3. Fabric samples dyed with NaOH gain the highest reflectance percentage since the dyebath pH is highly alkaline (pH value section) which leads to a higher chance of alkaline hydrolysis of the dye molecules, lowering the final dye exhaustion and fixation.

The reflectance curves of the dyed cotton fabric samples in both water-based and SAE-based systems are generally identical in shape, without the peak shift. There is no chromatic change on the dyed samples, verifying that the use of $NaHCO_3$, Na_2CO_3 and NaOH as CFA only affects the shade, but does not cause alternations of the color properties of the dyed cotton fabric samples.

Relative unlevelness indices (RUI)

Table 5 and Figure 6 present the color levelness, in the form of relative unlevelness indices (RUI); and the visual images of cotton samples dyed in both water-based and SAE-based systems with different CFAs. Samples dyed in conventional water-based system using $NaHCO_3$, Na_2CO_3, and NaOH can achieve excellent levelness with RUI values of below 0.2. High liquor-to-goods ratio of 20:1 may be the result of low RUI value (excellent levelness) of the water-based dyed samples. In case of SAE salt free dyed cotton samples, most of them can obtain good to excellent levelness with RUI value less than 0.5 when $NaHCO_3$ and Na_2CO_3 are used as CFA. However, when NaOH is applied in the SAE-based dyeing system, samples dyed with BCA reactive dye at high concentrations (2.5% and 3.5%) lead to poor color levelness (RUI: 0.55–0.58). The worst case is those samples dyed with RCA reactive dye at high concentrations (2.5% and 3.5%) in which the RUI values after calculation are in the range from 0.87 to 1.06, indicating poor to bad color levelness. It may be owing to high alkalinity of dyebath and strong electrostatic repulsion between dye and fiber.

CIE L*a*b* value

The CIE L^*a^{**} values of the dyed cotton fabric samples with different CFAs are represented in Table 6. L^* values (lightness) of the dyed cotton fabric samples in water and octane medium decreased when dye concentration of three reactive dyes increases from 0.1% to 3.5%, although different alkalis were used as CFA. It means that the dyed cotton fabric samples become darker

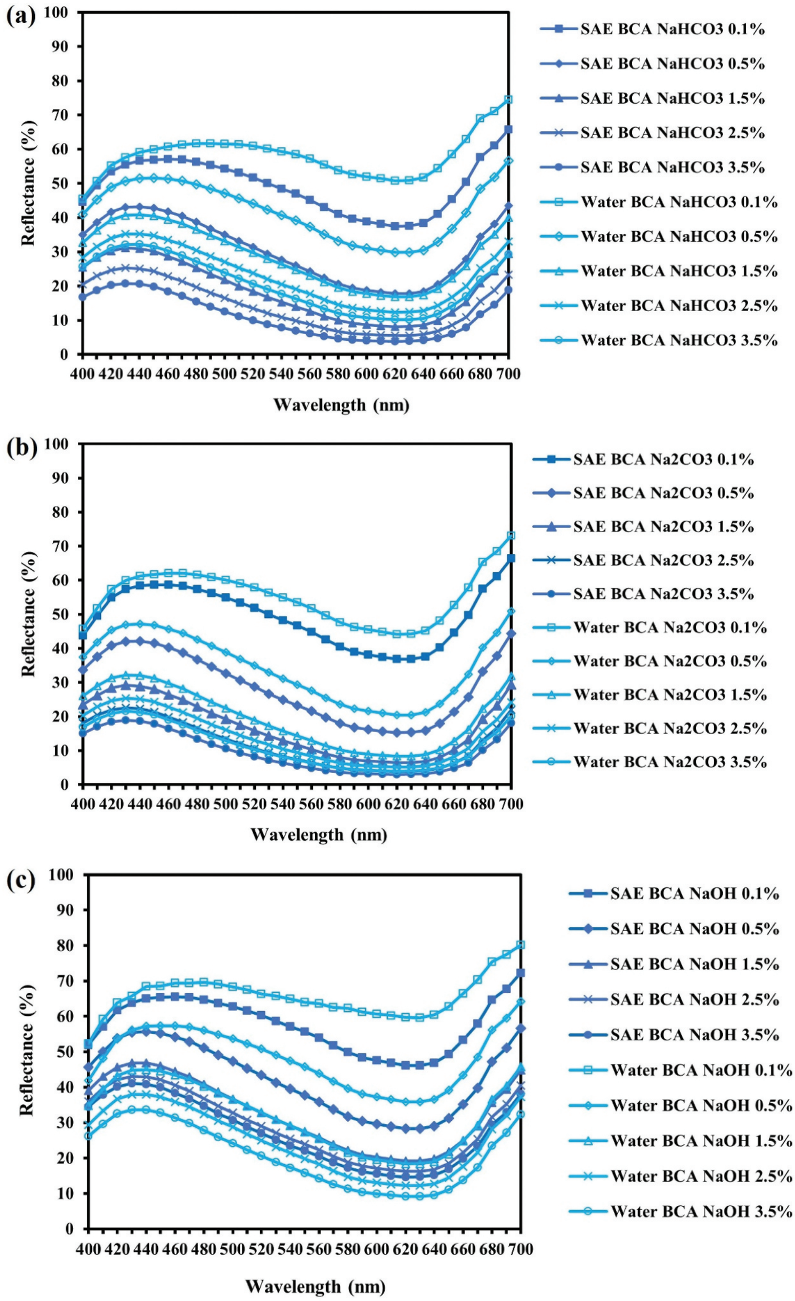

Figure 5. Reflectance curves of the dyed cotton fabrics: (a) BCA $NaHCO_3$; (b) BCA Na_2CO_3; (c) BCA NaOH; (d) RCA $NaHCO_3$; (e) RCA Na_2CO_3; (f) RCA NaOH; (g) YCA $NaHCO_3$; (h) YCA Na_2CO_3; and (i) YCA NaOH.

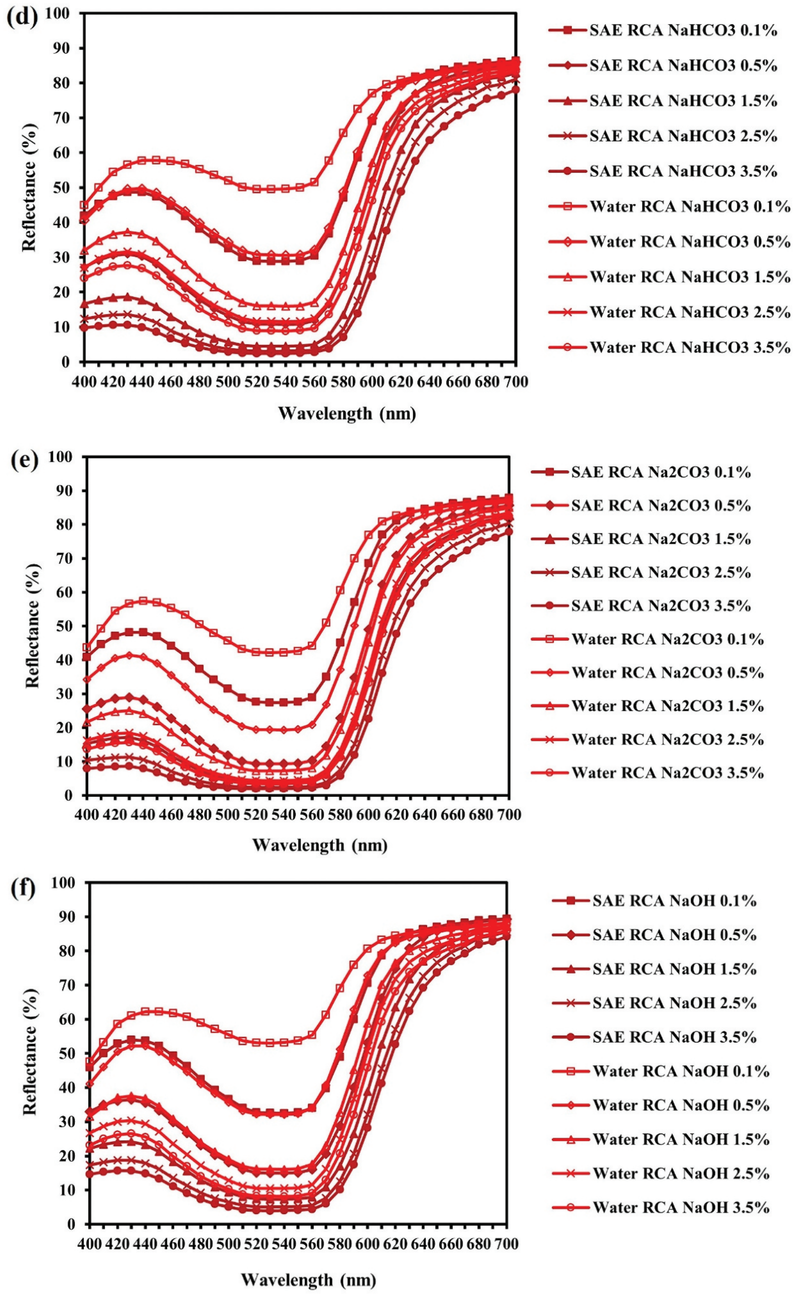

Figure 5. (Continued).

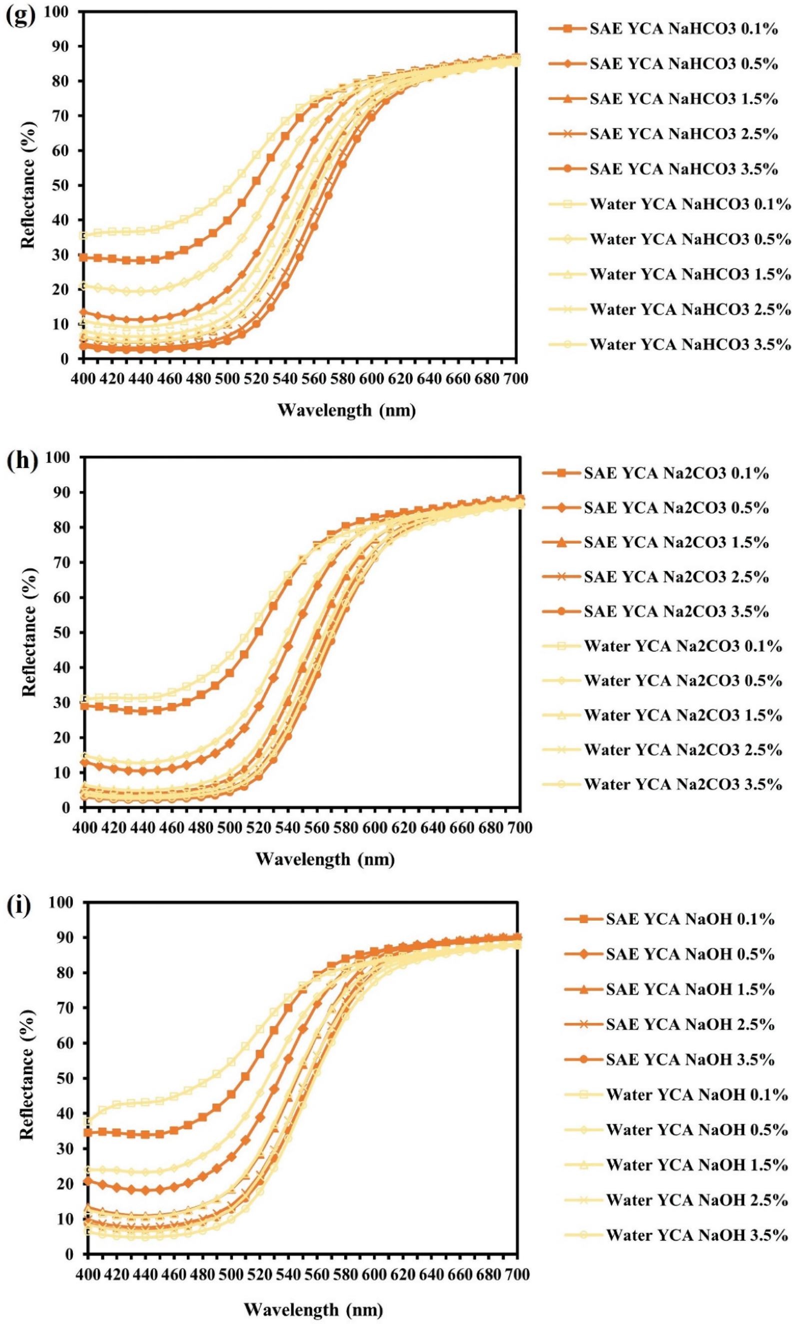

Figure 5. (Continued).

Table 5. Levelness of the dyed samples with different CFA.

		RUI											
		Water						SAE					
Color	Dye conc.	$NaHCO_3$	Visual	Na_2CO_3	Visual	NaOH	Visual	$NaHCO_3$	Visual	Na_2CO_3	Visual	NaOH	Visual
BCA	0.1%	0.04	Excellent	0.03	Excellent	0.03	Excellent	0.05	Excellent	0.05	Excellent	0.14	Excellent
	0.5%	0.03	Excellent	0.05	Excellent	0.03	Excellent	0.14	Excellent	0.23	Good	0.18	Excellent
	1.5%	0.07	Excellent	0.13	Excellent	0.07	Excellent	0.19	Excellent	0.12	Excellent	0.14	Excellent
	2.5%	0.08	Excellent	0.24	Excellent	0.15	Excellent	0.24	Good	0.46	Good	0.58	Poor
	3.5%	0.17	Excellent	0.15	Excellent	0.05	Excellent	0.28	Good	0.33	Good	0.55	Poor
RCA	0.1%	0.03	Excellent	0.03	Excellent	0.17	Excellent	0.21	Good	0.12	Excellent	0.17	Excellent
	0.5%	0.03	Excellent	0.04	Excellent	0.07	Excellent	0.12	Excellent	0.04	Excellent	0.40	Good
	1.5%	0.10	Excellent	0.19	Excellent	0.08	Excellent	0.23	Good	0.26	Good	0.30	Good
	2.5%	0.14	Excellent	0.27	Excellent	0.08	Excellent	0.14	Excellent	0.20	Good	1.06	Bad
	3.5%	0.17	Excellent	0.22	Excellent	0.13	Excellent	0.43	Good	0.29	Good	0.87	Poor
YCA	0.1%	0.02	Excellent	0.03	Excellent	0.01	Excellent	0.04	Excellent	0.05	Excellent	0.05	Excellent
	0.5%	0.02	Excellent	0.06	Excellent	0.02	Excellent	0.05	Excellent	0.06	Excellent	0.11	Excellent
	1.5%	0.03	Excellent	0.09	Excellent	0.10	Excellent	0.13	Excellent	0.14	Excellent	0.28	Good
	2.5%	0.03	Excellent	0.05	Excellent	0.05	Excellent	0.06	Excellent	0.08	Excellent	0.09	Excellent
	3.5%	0.09	Excellent	0.12	Excellent	0.07	Excellent	0.11	Excellent	0.15	Excellent	0.08	Excellent

Remark: RUI <0.2 = Excellent levelness; 0.2 to 0.49 = Good levelness; 0.5 to 1 = Poor levelness; >1 = Bad levelness (Chong, Li, and Yeung 1992).

in shade (lower lightness) when high concentration of dye is used. Within comparison to water-based dyed cotton fabric samples, SAE-based dyed cotton fabric samples generally show lower L^* values, validating the benefit of using SAE-based dyeing approach for cotton fabrics. Among different alkalis, samples dyed with Na_2CO_3 generally gain the lowest L^* values, followed by cotton fabric samples dyed with $NaHCO_3$ whereas cotton fabric samples dyed with NaOH have the highest L^* values, showing the inferior performance of NaOH as a CFA.

When RCA and YCA reactive dyes are used, both a^* and b^* values of the samples dyed in water and octane medium are positive and increase with increasing dye concentrations, indicating the dyed samples are located in red-yellow region and become redder and yellower. Samples dyed with Na_2CO_3 generally obtain the highest a^* and b^* values while samples dyed with NaOH reveal the lowest a^* and b^* values among the three alkalis. In case of BCA reactive dye, both water-based dyed and SAE-based dyed samples generally show decreasing a^* and b^* values when higher dye concentrations are used. This reveals that both water-based dyed and SAE-based dyed samples are located in blue-green region and become bluer and greener. Both water-based dyed and SAE-based dyed samples have similar a^* values but different b^* values when $NaHCO_3$, Na_2CO_3, and NaOH are used. Among the three alkalis, samples dyed with Na_2CO_3 generally exhibit the lowest b^* value whereas samples dyed with NaOH receive the highest b^* value. This indicates that samples dyed with Na_2CO_3 are bluer in shade than $NaHCO_3$ and NaOH.

SEM image

Figure 7 shows SEM images of undyed (Figure 7a), water-based dyed and SAE-based dyed cotton fabric samples with 3.5% of RCA reactive dye using different alkalis as CFA. It is observed that the use of $NaHCO_3$ and Na_2CO_3 as CFA (Figures 7b,c,e,f) does not cause significant alkaline damage on cotton fiber surface. However, when NaOH is the CFA (Figures 7d and g), significant damage occurs on the fiber surface with the presence of many protruding cotton fibrils in random directions, probably owing to high pH value (alkalinity) of the dyebath during the dyeing process. Therefore, using NaOH of similar amount as $NaHCO_3$ and Na_2CO_3 is not recommended in both water-based and SAE-based dyeing systems.

Table 6. CIE L*a*b* value of the dyed cotton fabric with different CFA.

		Conventional									SAE								
		$NaHCO_3$			Na_2CO_3			NaOH			$NaHCO_3$			Na_2CO_3			NaOH		
Dye	Dye (%)	L*	a*	b*	L*	a*	b*	L*	a*	b*	L*	a*	b*	L*	a*	b*	L*	a*	b*
BCA	0.1	79.60	-4.89	-3.66	76.61	-6.87	-9.87	83.87	-3.54	-0.65	72.50	-7.84	-12.27	72.27	-8.33	-14.07	77.90	-7.01	-10.83
	0.5	67.02	-8.99	-15.87	59.29	-10.54	-23.18	71.63	-8.46	-13.95	56.20	-10.81	-23.90	53.55	-11.30	-26.67	66.14	-9.34	-20.43
	1.5	54.94	-10.79	-23.30	43.05	-12.02	-30.70	57.31	-11.27	-23.97	42.47	-12.03	-30.24	39.10	-12.12	-32.53	57.69	-10.27	-24.80
	2.5	49.00	-11.23	-25.85	35.46	-11.47	-32.63	49.69	-12.28	-27.90	36.17	-11.46	-31.56	31.53	-11.09	-34.00	54.24	-10.57	-26.09
	3.5	45.60	-11.47	-27.12	31.32	-10.59	-32.73	44.81	-12.65	-30.04	30.73	-10.56	-32.49	27.60	-9.79	-33.60	52.30	-10.78	-26.76
RCA	0.1	83.38	17.95	6.27	81.19	23.92	4.77	85.23	17.30	5.54	74.94	33.22	4.99	74.41	35.01	4.79	76.81	31.46	2.92
	0.5	75.66	31.50	6.85	69.94	41.61	7.28	77.01	31.60	6.10	63.02	49.49	11.27	61.46	51.00	11.61	65.89	45.61	7.64
	1.5	66.73	43.48	7.47	59.11	53.25	14.71	67.50	44.48	8.95	54.20	55.55	18.75	53.18	56.68	20.33	57.36	52.20	12.64
	2.5	62.92	47.46	9.58	54.44	56.32	19.59	62.61	49.72	11.72	50.25	56.55	23.16	48.63	57.70	26.74	53.27	53.95	15.96
	3.5	60.04	50.09	11.36	52.19	56.95	22.14	59.82	51.72	13.36	47.23	55.98	25.78	45.88	56.90	30.00	50.69	54.57	18.10
YCA	0.1	88.29	9.08	32.79	87.79	10.88	39.00	90.12	7.73	28.66	87.20	12.30	42.33	87.79	13.26	44.76	89.85	11.24	39.31
	0.5	84.86	16.89	53.04	83.79	21.39	65.61	87.02	15.42	50.01	82.71	23.28	67.67	82.97	24.48	70.76	86.40	19.93	58.60
	1.5	80.64	25.51	70.49	78.78	31.73	83.97	82.93	24.61	70.35	77.90	32.21	82.93	77.84	34.01	87.62	83.35	26.46	70.58
	2.5	78.57	29.41	76.97	75.99	35.96	88.84	80.53	29.26	79.54	74.79	36.68	87.74	75.07	38.34	91.76	81.18	30.30	77.15
	3.5	76.73	32.25	80.55	73.98	38.62	90.53	78.64	32.43	84.40	73.03	39.23	89.60	73.32	40.68	93.00	80.05	31.96	78.37

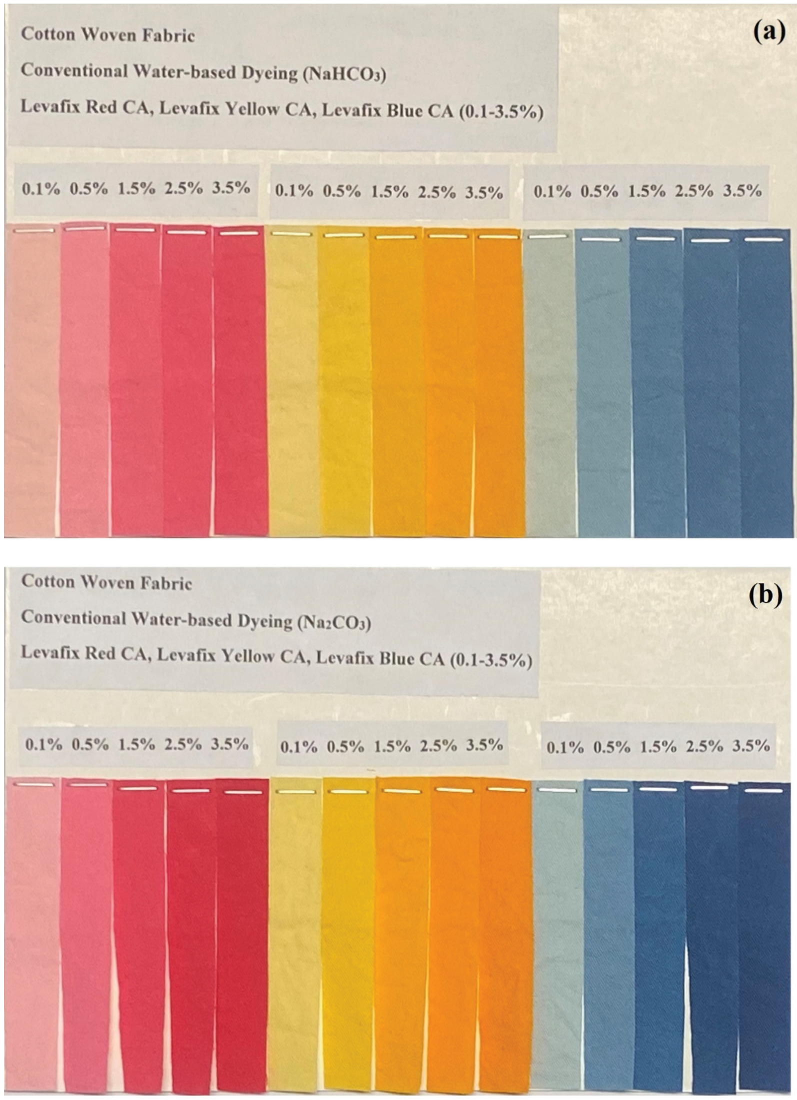

Figure 6. Visual images of the dyed cotton fabrics: (a) water $NaHCO_3$; (b) water Na_2CO_3; (c) water NaOH; (d) SAE $NaHCO_3$; (e) SAE Na_2CO_3; (f) SAE NaOH.

Color fastness to washing

The color change and staining of dyed cotton fabrics with the use of different alkali as CFA, after the washing fastness test, are depicted in Table 7. It is observed that both water-based dyed and SAE-based dyed samples can achieve excellent ratings against color change (rating 4–5). In case of color staining, most of the dyed samples in two different systems obtain excellent ratings against color staining (rating 4–5) except SAE-based dyed samples dyed with BCA and RCA reactive dyes of high concentrations (2.5% and 3.5%) of which have the rating of 4. These results prove that the dyed samples in two different dyeing systems are adequately washed after the rinsing process in which unfixed and hydrolyzed dye, and unwanted chemicals and auxiliaries are removed, ensuring the accuracy of the data obtained from color properties measurement. In addition, these results

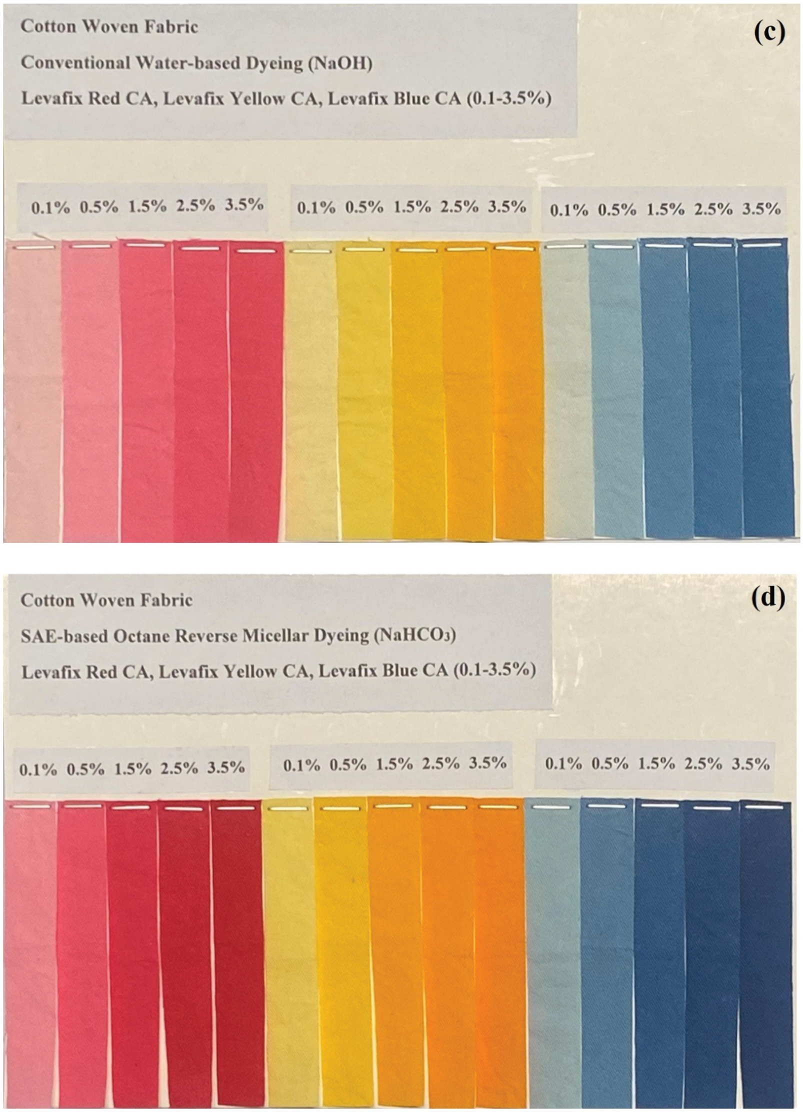

Figure 6. (Continued).

also affirm that the use of different alkalis as CFA cause no effect on the washing fastness of the dyed cotton fabric samples.

Color fastness to crocking

Table 8 shows the crocking fastness results of the dyed cotton fabric samples. It is clear that water-based dyed samples and most of the SAE-based dyed samples (dye conc. 0.1% to 1.5%) can achieve excellent crocking fastness (rating 4–5) while only a few SAE-based dyed samples dyed with high concentrations (2.5% and 3.5%) have slightly lower crocking fastness (rating 4). This reveals that the dye molecules are well bonded or fixed in the fiber matrix of the cotton fabric samples and the unfixed dye residues are sufficiently removed after the rinsing process. The results also make it evident that the use of different alkalis as CFA does not have any effect on the crocking fastness of the dyed samples in two different systems.

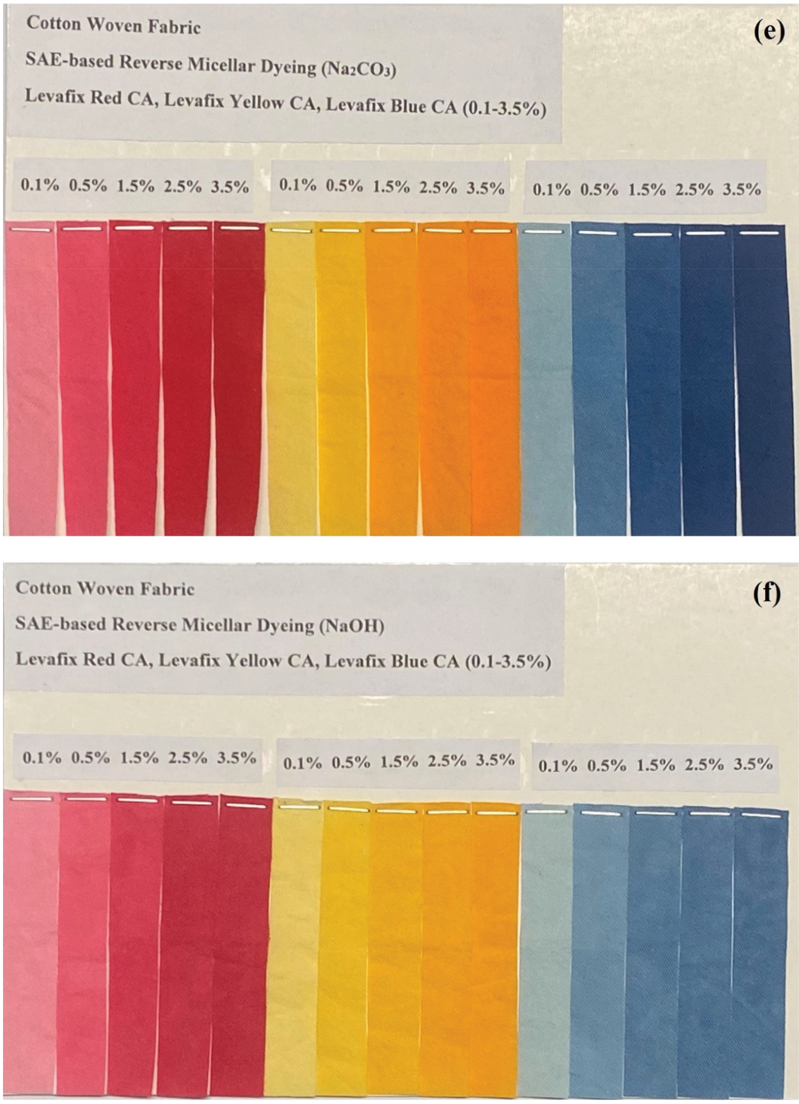

Figure 6. (Continued).

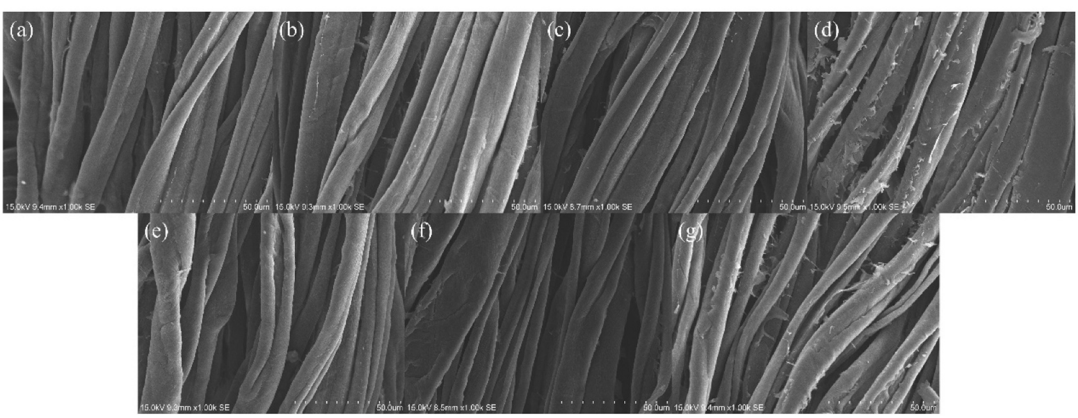

Figure 7. SEM images of (a) undyed; and red-dyed cotton samples (3.5% dye concentration) using different dye fixation agents: (b) water $NaHCO_3$; (c) water Na_2CO_3; (d) water NaOH; (e) SAE $NaHCO_3$; (f) SAE Na_2CO_3; (g) SAE NaOH.

Table 7. Washing fastness result of dyed cotton fabrics.

	Dye conc. (%)	Color change	Color staining	
			Wool	Cotton
BCA	0.1	4-5/4-5/4-5/4-5/4-5/4-5*	4-5/4-5/4-5/4-5/4-5/4-5	4-5/4-5/4-5/4-5/4-5/4-5
	0.5	4-5/4-5/4-5/4-5/4-5/4-5	4-5/4-5/4-5/4-5/4-5/4-5	4-5/4-5/4-5/4-5/4-5/4-5
	1.5	4-5/4-5/4-5/4-5/4-5/4-5	4-5/4-5/4-5/4-5/4-5/4-5	4-5/4-5/4-5/4-5/4-5/4-5
	2.5	4-5/4-5/4-5/4-5/4-5/4-5	4-5/4-5/4-5/4-5/4-5/4-5	4-5/4-5/4-5/4/4/4
	3.5	4-5/4-5/4-5/4-5/4-5/4-5	4-5/4-5/4-5/4-5/4-5/4-5	4-5/4-5/4-5/4/4/4
RCA	0.1	4-5/4-5/4-5/4-5/4-5/4-5	4-5/4-5/4-5/4-5/4-5/4-5	4-5/4-5/4-5/4-5/4-5/4-5
	0.5	4-5/4-5/4-5/4-5/4-5/4-5	4-5/4-5/4-5/4-5/4-5/4-5	4-5/4-5/4-5/4-5/4-5/4-5
	1.5	4-5/4-5/4-5/4-5/4-5/4-5	4-5/4-5/4-5/4-5/4-5/4-5	4-5/4-5/4-5/4-5/4-5/4-5
	2.5	4-5/4-5/4-5/4-5/4-5/4-5	4-5/4-5/4-5/4-5/4-5/4-5	4-5/4-5/4-5/4/4/4
	3.5	4-5/4-5/4-5/4-5/4-5/4-5	4-5/4-5/4-5/4/4/4	4-5/4-5/4-5/4/4/4
YCA	0.1	4-5/4-5/4-5/4-5/4-5/4-5	4-5/4-5/4-5/4-5/4-5/4-5	4-5/4-5/4-5/4-5/4-5/4-5
	0.5	4-5/4-5/4-5/4-5/4-5/4-5	4-5/4-5/4-5/4-5/4-5/4-5	4-5/4-5/4-5/4-5/4-5/4-5
	1.5	4-5/4-5/4-5/4-5/4-5/4-5	4-5/4-5/4-5/4-5/4-5/4-5	4-5/4-5/4-5/4-5/4-5/4-5
	2.5	4-5/4-5/4-5/4-5/4-5/4-5	4-5/4-5/4-5/4-5/4-5/4-5	4-5/4-5/4-5/4-5/4-5/4-5
	3.5	4-5/4-5/4-5/4-5/4-5/4-5	4-5/4-5/4-5/4-5/4-5/4-5	4-5/4-5/4-5/4-5/4-5/4-5

Remark: 1 = most color change and staining; 5 = least color change and staining. *Rating indication: water-dyed $NaHCO_3$ sample/water-dyed Na_2CO_3 sample/water-dyed NaOH sample/SAE-dyed $NaHCO_3$ sample/SAE-dyed Na_2CO_3 sample/SAE-dyed NaOH sample.

Tensile strength and elongation at break

Tensile strength values (N) of pristine cotton fabric samples and cotton fabric samples dyed with different CFAs, are presented in Table 9. Undyed cotton sample originally has tensile strength of 415 N in warp direction. After the dyeing process with the use of different alkalis, both water-based dyed (315 N to 411 N) and SAE-based dyed (292 N to 375 N) cotton samples generally exhibit a decrease in tensile strength in warp direction. This decrease in tensile strength may be because of the bath temperature, and the dyebath pH value during the dyeing process (Zhang et al. 2021). In addition, water-based dyed cotton fabric samples generally have higher tensile strength (strength loss only between −1% and 23.9%) in warp direction compared with SAE-based dyed cotton fabric samples (strength loss ranged from −9.51% to −29.48%). This may be because of use of non-aqueous solvent which may pose relatively higher damage to the tensile strength of the cotton fiber when compared with samples dyed in water medium. Tensile strength of the undyed sample in weft direction (Table 9)

Table 8. Crocking fastness result of dyed cotton fabrics.

	Dye conc. (%)	Color staining	
		Dry	Wet
BCA	0.1	4-5/4-5/4-5/4-5/4-5/4-5	4-5/4-5/4-5/4-5/4-5/4-5
	0.5	4-5/4-5/4-5/4-5/4-5/4-5	4-5/4-5/4-5/4-5/4-5/4-5
	1.5	4-5/4-5/4-5/4-5/4-5/4-5	4-5/4-5/4-5/4-5/4-5/4-5
	2.5	4-5/4-5/4-5/4/4/4	4-5/4-5/4-5/4/4/4
	3.5	4-5/4-5/4-5/4/4/4	4-5/4-5/4-5/4/4/4
RCA	0.1	4-5/4-5/4-5/4-5/4-5/4-5	4-5/4-5/4-5/4-5/4-5/4-5
	0.5	4-5/4-5/4-5/4-5/4-5/4-5	4-5/4-5/4-5/4-5/4-5/4-5
	1.5	4-5/4-5/4-5/4-5/4-5/4-5	4-5/4-5/4-5/4/4/4
	2.5	4-5/4-5/4-5/4/4/4	4-5/4-5/4-5/4/4/4
	3.5	4-5/4-5/4-5/4/4/4	4-5/4-5/4-5/4/4/4
YCA	0.1	4-5/4-5/4-5/4-5/4-5/4-5	4-5/4-5/4-5/4-5/4-5/4-5
	0.5	4-5/4-5/4-5/4-5/4-5/4-5	4-5/4-5/4-5/4-5/4-5/4-5
	1.5	4-5/4-5/4-5/4-5/4-5/4-5	4-5/4-5/4-5/4-5/4-5/4-5
	2.5	4-5/4-5/4-5/4-5/4-5/4-5	4-5/4-5/4-5/4/4/4
	3.5	4-5/4-5/4-5/4/4/4	4-5/4-5/4-5/4/4/4

Remark: 1 = most color staining; 5 = least color staining. *Rating indication: water-dyed $NaHCO_3$ sample/water-dyed Na_2CO_3 sample/water-dyed NaOH sample/SAE-dyed $NaHCO_3$ sample/SAE-dyed Na_2CO_3 sample/SAE-dyed NaOH sample.

Table 9. Tensile strength of undyed and dyed cotton samples with different CFA.

Strength (N)	Undyed		Water												SAE											
			$NaHCO_3$				Na_2CO_3				NaOH				$NaHCO_3$				Na_2CO_3				NaOH			
Sample	Warp	Weft	Warp	(%)	Weft	(%)	Warp	(%)	Weft	(%)	Warp	(%)	Weft	(%)	Warp	(%)	Weft	(%)	Warp	(%)	Weft	(%)	Warp	(%)	Weft	(%)
BCA 3.5	415	216	411	−1.00	235	8.71	346	−16.64	215	−0.38	315	−24.14	216	0.06	375	−9.51	239	10.55	358	−13.72	236	9.17	357	−13.85	227	4.95
RCA 3.5			403	−2.88	210	−2.49	317	−23.46	211	−2.27	316	−23.90	218	1.06	352	−15.11	221	2.36	321	−22.49	216	0.25	292	−29.48	214	−0.62
YCA 3.5			373	−9.97	230	6.72	337	−18.63	209	−3.31	328	−20.97	198	−8.40	372	−10.34	234	8.21	336	−19.04	219	1.50	323	−22.02	205	−5.22

Table 10. Elongation at break of undyed and dyed cotton samples with different CFA.

Breaking extension	Undyed				Water												SAE											
					$NaHCO_3$				Na_2CO_3				NaOH				$NaHCO_3$				Na_2CO_3				NaOH			
	Warp		Weft		Warp		Weft		Warp		Weft		Warp		Weft		Warp		Weft		Warp		Weft		Warp		Weft	
Sample	(mm)	(%)	(mm)	(%)	(mm)	(%)	(mm)	(%)	(mm)	(%)	(mm)	(%)	(mm)	(%)	(mm)	(%)	(mm)	(%)	(mm)	(%)	(mm)	(%)	(mm)	(%)	(mm)	(%)	(mm)	(%)
BCA 3.5	16.65	22.2	9.42	12.56	18.41	24.55	10.66	14.21	16.68	22.24	8.85	11.8	17.4	23.2	9.92	13.23	16.43	21.91	9.18	12.24	16.6	21.96	8.72	11.64	17.19	22.92	10.35	13.8
RCA 3.5					17.91	23.88	9.91	13.21	16.61	22.15	8.61	11.48	17.67	23.56	10.67	14.23	16.17	21.56	8.93	11.91	16.56	21.93	8.45	11.36	17.17	22.89	9.08	12.11
YCA 3.5					19.17	25.56	10.67	14.23	16.18	21.57	9.16	12.21	17.93	23.91	9.86	13.15	16.68	22.24	8.35	11.13	16.04	21.25	8.89	11.97	16.92	22.56	8.85	11.8

is 216N. Strength gain and strength loss vary in case of both water-based dyed (198N to 235N) and SAE-based dyed (205 N to 239 N) samples. SAE-based dyed cotton fabric samples generally achieve relatively higher tensile strength in weft direction.

Among the three alkalis (Table 9), cotton fabric samples dyed with $NaHCO_3$ in water-based and reverse micellar system have the lowest strength loss in both warp and weft directions, followed by Na_2CO_3, while cotton fabric samples dyed with NaOH incur the highest strength loss. This may be because of pH value (alkalinity); NaOH (strong alkali) has the highest dyebath pH value whereas dyeing with $NaHCO_3$ (weak alkali) reveals the lowest dyebath pH value, as shown in Table 4 in the previous section.

Elongation at break, in terms of length (mm) and percentage (%), of undyed and dyed cotton fabric samples is displayed in Table 10. The elongation at break along warp direction for undyed, and dyed cotton fabric samples in different dyeing systems is generally higher than that along their weft direction. Meanwhile, SAE-based dyed cotton fabric samples, using three alkalis, can achieve elongation at break which is similar to that of undyed and water-based dyed cotton fabric samples, indicating that those alkalis do not lead to severe variation in elongation at break of the dyed cotton fabric samples.

Conclusion

The influence of different CFAs and dyebath pH in SAE-based reverse micellar dyeing system for cotton fabrics was investigated. Experimental results reveal that cotton fabric samples dyed with Na_2CO_3 as CFA can achieve the highest color yield, followed by $NaHCO_3$, whereas cotton fabric samples dyed with NaOH have the lowest color yield in both water-based and SAE-based reverse micellar dyeing system. When compared with water-based dyed cotton fabric samples, SAE-based dyed cotton fabric samples generally acquire higher color yield when $NaHCO_3$ and Na_2CO_3 are used. However, dyeing cotton fabric samples with NaOH in SAE-based reverse micellar system results in significantly lower color yield, particularly when BCA and YCA reactive dyes are used.

The dye uptake and color yield are found to be closely related to the dyebath pH value. Too high (above 12) or low (7 to 9) dyebath pH may lead to lower color yield. The optimum dyebath pH (alkalinity) is thus in the range of 10 to 11 with the use of Na_2CO_3 as CFA, especially for surfactant-based reverse micellar salt free dyeing system. Since the alkali amount was fixed in this study, it is estimated that improvement can be made on current dyeing recipe when using $NaHCO_3$ and NaOH as CFA in which higher amount of $NaHCO_3$ can be added to increase the dyebath pH whereas the amount of NaOH can be reduced in order to lower the pH value of the dyebath.

Reflectance curves further confirm that too high or low dyebath pH may also cause higher reflectance percentage while the RUI values reflect that cotton fabric samples dyed with NaOH have higher chance of color unlevelness when compared with $NaHCO_3$ and Na_2CO_3 probably owing to the too high alkalinity of the dyebath. CIE $L^*a^*b^*$ values of samples dyed with different alkalis are also studied. Both cotton fabric samples dyed in two systems show good to excellent fastness against washing and crocking, validating adequate removal of residues composed of dye and chemical auxiliaries. Tensile strength results affirm a positive relationship between dyebath pH and strength loss, suggesting higher alkalinity of dyebath may lead to higher strength loss while SEM images show that the use of different alkalis may cause different degrees of damage to the surface morphology of the cotton fiber.

Since there are parameters, other than CFA and dyebath pH, that may affect the dyeing performance of SAE surfactant-based reverse micellar salt free dyeing approach on cotton fabric, future works concerning SAE-based surfactant series and co-surfactant series, as well as the applicability of this approach on computer color matching with combination of reactive dye of three primary colors are necessary to further develop knowledge and understandings about this non-aqueous waterless dyeing approach.

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