# Dyeing Properties of Cotton with Reactive Dye in Nonane Nonaqueous Reverse Micelle System

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**ABSTRACT:** In this study, we explored the dyeing behavior of cotton with reactive dyes in poly(ethylene glycol)-based reverse micelle system in nonaqueous alkane medium of nonane ($C_9H_{20}$). Calibration of dyeing databases for both conventional aqueous-based dyeing method and nonaqueous nonane reverse micellar dyeing method was initially established, along with simulated dyeing of standard samples with known concentrations. Several color difference formulae were used to conduct computer color matching (CCM), by matching the color between batch samples and the standard samples, for both dyeing methods. Excellent color matching results were achieved as both dyeing methods showed that the CCM-predicted concentrations were nearly the same as the known concentrations. It indicates that utilizing nonane as a solvent to facilitate reverse micellar dyeing of cotton can achieve good color matching when compared with that of the conventional aqueous-based dyeing system. Relative unlevelness indices were used to evaluate the evenness of the dyed samples. Good to excellent levelness results were obtained, comparable to that of the conventional aqueous-based dyeing system. Color fastness to laundering of nonane reverse micellar dyed samples was found to be good for industrial applications. In addition, during the reverse micellar dyeing process, only very low level of volatile organic compound content was detected and 98% nonane could be recovered simply by fractional distillation. The results in this study explored the practical usage of nonane nonaqueous reverse micellar approach on the dyeing of cotton with the use of reactive dyes.

Calibration Dyeing
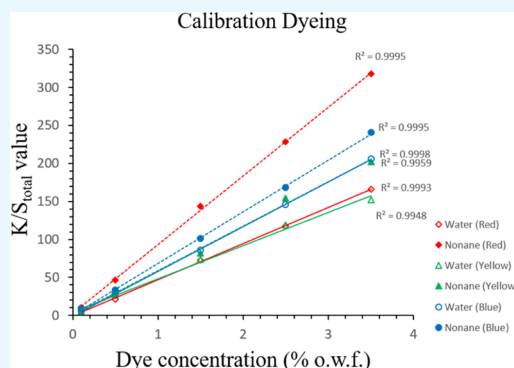
## 1. INTRODUCTION

Cotton dyeing with the use of reactive dyes requires enormous amounts of salt to facilitate dye exhaustion and absorption[1] and after dyeing, large amounts of water are required for cleaning the dyed fabrics, which increases the need for effluent treatment before discharging.[2,3] As the effluents discharged affect the ecological system adversely and the environmental cost of effluent treatment is huge, many alternative ways, such as dyeing with low-salt or salt-free content,[4−6] natural mordants dyeing,[7−10] reusing wastewater as one of the water source for dyeing,[11] and supercritical fluid dyeing using carbon dioxide,[8,12−14] have been proposed. Despite these proposed methods, the use of reverse micelles as the medium for nonaqueous textile dyeing has been considered for achieving ecological friendliness, energy saving, and safety in the working environment.[15,16] Reverse micelles, spherical aggregates in nanoscale, are self-assembled and formed by the use of certain surfactants in nonpolar medium.[17,18] They are able to solubilize small amounts of water in their interior regions, providing a stable aqueous microenvironment, named water pool, in nonaqueous dyeing medium. Although many research studies have investigated the dyeing of cotton fiber with the use of nonionic reverse micelles in nonaqueous medium,[16,19−21] all of them focused on primary colors. In industrial production, a variety of colors are produced with the use of primary colors (red, yellow, and blue) as the base, and the aesthetic

appearance of most textile products is a mixture of colors rather than a single color. Thus, color matching is an important aspect in industrial dyeing applications.[22,23]

In our previous research, we successfully dyed cotton fabric with reactive dyes of primary colors (red, blue, and yellow)[16] with poly(ethylene glycol) (PEG)-based reverse micelle system in nonaqueous alkane medium of heptane and octane. Computer color matching (CCM) has also been conducted with the use of decamethyl cyclopentasiloxane[24] and octane.[25] In this study, we explore the PEG-based reverse micelle system in nonane (which has less harmful effect than heptane and octane) for dyeing cotton with reactive dyes of primary colors. To meet the requirements of the industrial application, we also study the possibility of applying CCM system on nonane reverse micellar dyeing system of cotton and finally assess the levelness of dyed samples. Color fastness of the nonane reverse micellar dyed samples is investigated. To investigate the environmental issues of the reverse micellar dyeing process, volatile organic compound (VOC) content and the solvent recovery efficiency would be studied.
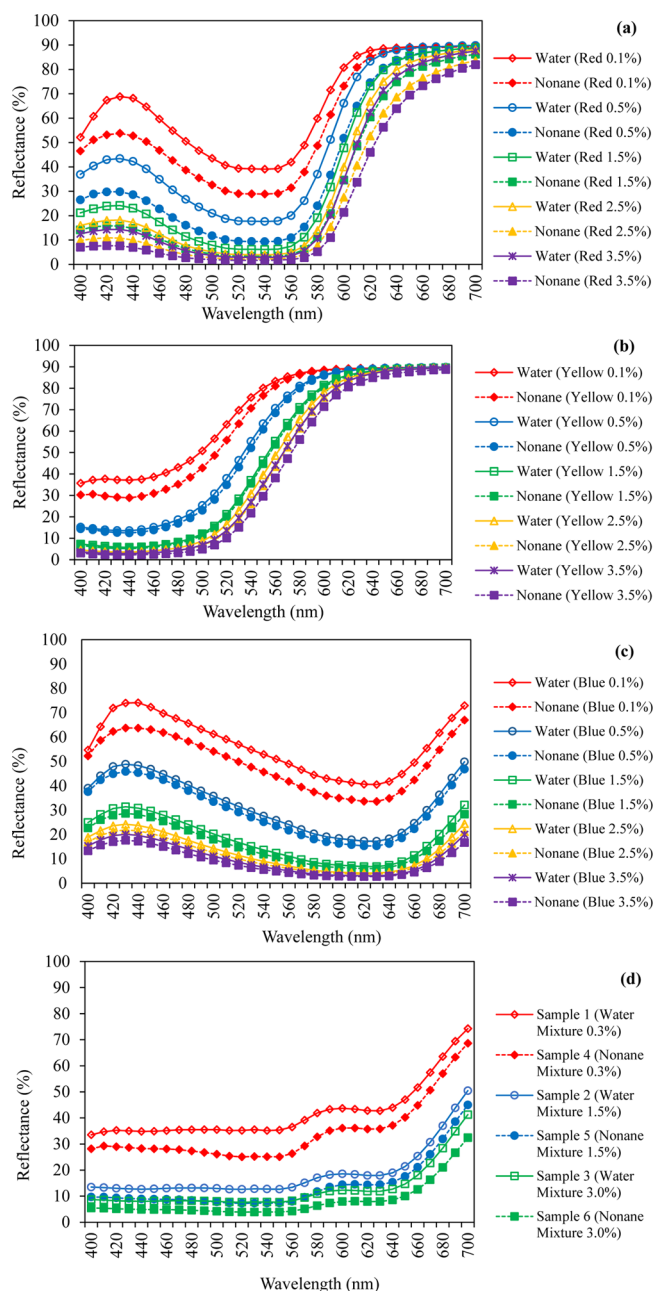
## 2. RESULTS AND DISCUSSION

### 2.1. Reflectance Curves of Batch and Standard Samples.
Figure 1 shows the reflectance curves of red (Figure



**Figure 1.** Reflectance curves of (a) red; (b) yellow; (c) blue; and (d) mixed color.

1a), yellow (Figure 1b), and blue (Figure 1c) color batch samples, respectively. The results show that reflectance values, from 400 to 700 nm wavelength, of the batch samples dyed in nonane medium at each dye concentration (0.1, 0.5, 1.5, 2.5, and 3.5%) are lower than batch samples dyed in water medium. This indicates that in nonane reverse micellar dyeing system, the absorption of dye in the fiber and the color yield of the dyed batch samples are higher with darker shade in appearance than that of the dyed batch samples in water medium. Concerning reflectance curves of three colors (red, blue, and yellow), the red color at each dye concentration had the most

significant reflectance value difference, whereas the differences in reflectance values of yellow color and blue color are less significant. This indicates that using nonionic PEG-based reverse micelle nonane-assisted dyeing approach can enhance color yield of fabrics even though same dye concentration is used.

Figure 1d shows the reflectance curves of color mixture of the standard samples. The reflectance values of the nonane-dyed standard samples at dye concentration from 0.3 to 3.0% are also lower than that of the water-dyed standard samples. No overlap is found in reflectance curves. This indicates that the color yield of the nonane-dyed standard sample is higher, with higher dye absorption and fixation in fiber, and the shade thus appears darker in comparison with the water-dyed standard samples.

The results illustrate that the shape of the reflectance curves of the water-dyed and nonane-dyed batch and standard samples are similar to each other. No peak-shifting of the curves is observed even though reflectance values of the dyed samples are not the same. This indicates that the use of nonane as the medium for dyeing of cotton fiber caused no chromatic change to the color of the dyed samples, and it also proves that using nonane solvent to facilitate the preparation and formation of PEG-based reverse micelles and as the medium for nonaqueous dyeing of cotton can be an alternative way for achieving water-saving and salt-free dyeing of cotton fiber and for computerized color matching with no chromatic shift.

### 2.2. Calibration Results.
The calibration curves of three primary colors (red, yellow, and blue) of water-dyed and nonane-dyed batch samples are shown in Figure 2. The
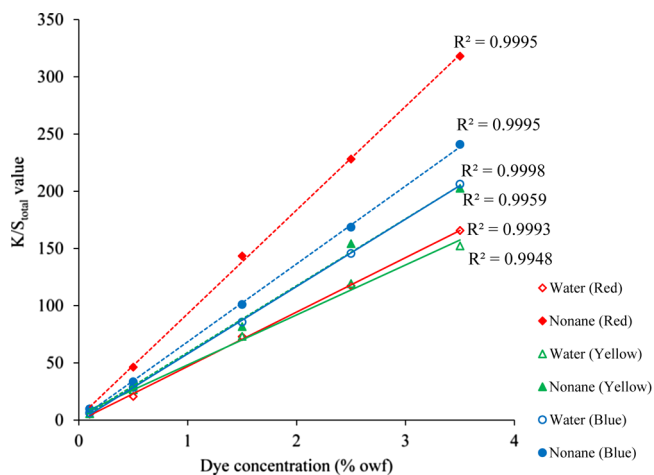


**Figure 2.** Plotted calibration curves of three colors of water and nonane-dyed batch samples.

calibration curve is plotted with $K/S_{total}$ value against the concentration of dye (% owf). The results from Figure 2 reveal that $K/S_{total}$ values of three colors of the nonane-dyed batch samples in reverse micelle approach are higher than that of the water-dyed batch samples. This indicates that using reverse micelle nonane nonaqueous dyeing approach can gain a better color yield than using conventional aqueous-based dyeing approach, and the use of nonane solvent does not have any adverse effects on covalent bonding between reactive dye and cotton fiber. The possible reason to explain is the utilization of PEG-based reverse micelle in nonaqueous dyeing, reducing the ionization effect between the dye molecules and the fiber. It

**Table 1. CCM-Predicted Recipes of Standard Samples with Different Color Difference Formulae (with Two Decimal Place)**

| formulae | color | conventional water-based dyeing | | | reverse micellar nonane-assisted dyeing | | |
|---|---|---|---|---|---|---|---|
| | | standard sample 1 (0.3%) | standard sample 2 (1.5%) | standard sample 3 (3%) | standard sample 4 (0.3%) | standard sample 5 (1.5%) | standard sample 6 (3%) |
| CIEL*a*b* | yellow | 0.10 | 0.50 | 0.88 | 0.10 | 0.52 | 1.02 |
| | blue | 0.09 | 0.44 | 0.76 | 0.11 | 0.49 | 1.03 |
| | red | 0.08 | 0.44 | 0.83 | 0.09 | 0.52 | 1.03 |
| CIEL*u*v* | yellow | 0.10 | 0.42 | 0.88 | 0.10 | 0.52 | 1.02 |
| | blue | 0.09 | 0.48 | 0.76 | 0.11 | 0.49 | 1.03 |
| | red | 0.08 | 0.50 | 0.83 | 0.09 | 0.52 | 1.03 |
| ANLAB | yellow | 0.10 | 0.50 | 0.88 | 0.10 | 0.52 | 1.02 |
| | blue | 0.09 | 0.44 | 0.76 | 0.11 | 0.49 | 1.03 |
| | red | 0.08 | 0.44 | 0.83 | 0.09 | 0.52 | 1.03 |
| HunterLab | yellow | 0.10 | 0.50 | 0.88 | 0.10 | 0.52 | 1.02 |
| | blue | 0.09 | 0.44 | 0.76 | 0.11 | 0.49 | 1.03 |
| | red | 0.08 | 0.44 | 0.83 | 0.09 | 0.52 | 1.03 |
| FMC2 | yellow | 0.10 | 0.42 | 0.78 | 0.07 | 0.52 | 1.02 |
| | blue | 0.09 | 0.48 | 0.81 | 0.08 | 0.49 | 1.03 |
| | red | 0.08 | 0.50 | 0.84 | 0.09 | 0.52 | 1.03 |
| JPC79 | yellow | 0.10 | 0.42 | 0.88 | 0.10 | 0.52 | 1.02 |
| | blue | 0.09 | 0.48 | 0.76 | 0.11 | 0.49 | 1.03 |
| | red | 0.08 | 0.50 | 0.83 | 0.09 | 0.52 | 1.03 |
| CMC 1.0 | yellow | 0.10 | 0.42 | 0.88 | 0.10 | 0.52 | 1.02 |
| | blue | 0.09 | 0.48 | 0.76 | 0.11 | 0.49 | 1.03 |
| | red | 0.08 | 0.50 | 0.83 | 0.09 | 0.52 | 1.03 |
| BFD 1.0 | yellow | 0.10 | 0.42 | 0.88 | 0.10 | 0.52 | 1.02 |
| | blue | 0.09 | 0.48 | 0.76 | 0.11 | 0.49 | 1.03 |
| | red | 0.08 | 0.50 | 0.83 | 0.09 | 0.52 | 1.03 |
| CIE94 1.0 | yellow | 0.10 | 0.50 | 0.88 | 0.10 | 0.52 | 1.02 |
| | blue | 0.09 | 0.44 | 0.76 | 0.11 | 0.49 | 1.03 |
| | red | 0.08 | 0.44 | 0.83 | 0.09 | 0.52 | 1.03 |

also enhances the swelling of the cotton fiber.[16,20] Another reason is the utilization of PEG-based reverse micelle in nonane-assisted dyeing approach, which minimizes the effect of hydrolysis of reactive dye in the dyeing process and enhances the adsorption behavior of the reverse micelles onto the fiber surface.[27−29]

The linearity of the calibration curves is expressed in terms of $R$-square $(R^2)$, a commonly used method for the measurement of goodness-of-fit of a regression.[30] $R^2$ values are between 0 and 1, in which 0 means nonlinear in structure and 1 means linear in structure. Figure 2 revealed that $R^2$ values of fabrics dyed in water medium were between 0.9948 (water yellow) and 0.9998 (water blue), whereas $R^2$ values of fabrics dyed by reverse micelle in nonane medium were between 0.9959 (nonane yellow) and 0.9995 (nonane red and blue). This indicates that calibration curves of water-dyed and nonane-dyed samples are linear in structure, which is ideal for CCM.[22,23]

**2.3. CCM-Predicted Recipes.** The CCM-predicted recipes of water-dyed and nonane-dyed standard samples were illustrated in Table 1, and the results reveal that the CCM-predicted recipes generated by using different color difference formulae for standard sample 1 are nearly the same (yellow: 0.10, blue: 0.09, and red: 0.08). The CCM-predicted recipes generated for standard sample 3 are also constant (yellow: 0.88, blue: 0.76, and red: 0.83) except FMC2 (yellow: 0.78, blue: 0.81, and red: 0.84). However, the CCM-predicted recipes generated by standard sample 2 are divided into two groups. One group includes CIEL*a*b*, ANLAB, HunterLab, and CIE94 1.0 (yellow: 0.50, blue: 0.44, and red: 0.44), whereas results of the others are yellow: 0.42, blue: 0.48, and red: 0.50.

These results indicate that CCM-predicted recipe variations are higher for standard sample 2 (water color mixture 1.5%), followed by standard sample 3 (water color mixture 3.0%) and standard sample 1 (water color mixture 0.3%).

Table 1 shows the CCM-predicted recipes of standard samples dyed by nonane reverse micellar approach with the use of different color difference equations. The CCM recipes generated for standard sample 5 (yellow: 0.52, blue: 0.49, and red: 0.52) and standard sample 6 (yellow: 1.02, blue: 1.03, and red: 1.03) are consistent, although different color difference formulae are used. For standard sample 4 (0.3%), the CCM-predicted recipes generated by using different color difference equations are also constant (yellow: 0.10, blue: 0.11, and red: 0.09) except FMC2 (yellow: 0.07, blue: 0.08, and red: 0.09). These results indicate that the CCM recipes predicted for samples dyed in nonane solvent medium have smaller variations than in conventional water-based medium.

**2.4. Levelness.** Table 2 summarizes the relative unlevelness indices (RUIs) value and the visual assessment of the levelness of cotton fabrics dyed in water aqueous medium and in nonane nonaqueous medium. The RUI of fabrics dyed in water medium varies from 0.02 to 0.34, whereas the RUI of fabrics dyed in nonane medium varies from 0.02 to 0.38. It means that both water-dyed and nonane-dyed standard and the batch samples can gain good to excellent levelness, which indicates that in reverse micellar nonane-assisted dyeing system, most dye molecules are in nonaggregate form and are well-dispersed in the fabric to avoid unlevelness. Figure 3 shows a comparison of the water-based and reverse micelle dyed samples, and it is

**Table 2. Levelness of Water-Dyed and Nonane-Dyed Batch and Standard Samples Based on RUI Value and Visual Assessment**

| sample | water | | nonane | |
|---|---|---|---|---|
| | RUI | visual assessment | RUI | visual assessment |
| red 0.1% | 0.17 | excellent | 0.16 | excellent |
| red 0.5% | 0.34 | good | 0.28 | good |
| red 1.5% | 0.24 | good | 0.16 | excellent |
| red 2.5% | 0.33 | good | 0.38 | good |
| red 3.5% | 0.16 | excellent | 0.14 | excellent |
| yellow 0.1% | 0.03 | excellent | 0.03 | excellent |
| yellow 0.5% | 0.08 | excellent | 0.02 | excellent |
| yellow 1.5% | 0.01 | excellent | 0.06 | excellent |
| yellow 2.5% | 0.06 | excellent | 0.07 | excellent |
| yellow 3.5% | 0.15 | excellent | 0.12 | excellent |
| blue 0.1% | 0.14 | excellent | 0.35 | good |
| blue 0.5% | 0.04 | excellent | 0.23 | good |
| blue 1.5% | 0.03 | excellent | 0.35 | good |
| blue 2.5% | 0.09 | excellent | 0.15 | excellent |
| blue 3.5% | 0.17 | excellent | 0.05 | excellent |
| mixture 0.3% | 0.02 | excellent | 0.23 | good |
| mixture 1.5% | 0.08 | excellent | 0.30 | good |
| mixture 3.0% | 0.09 | excellent | 0.35 | good |

obvious that the color developed in both types of samples has good levelness.
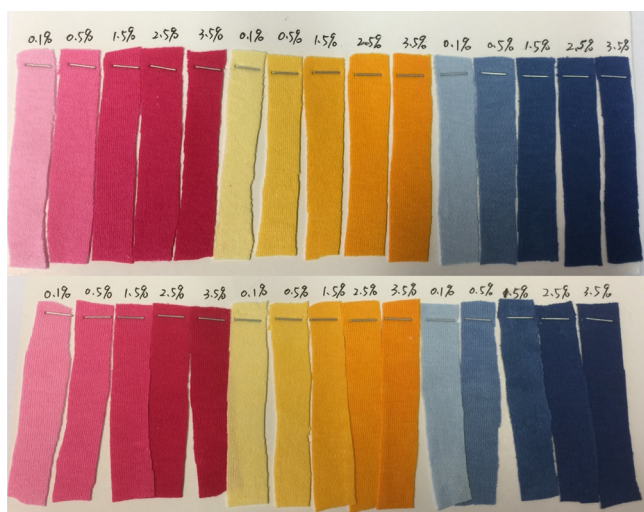


**Figure 3.** Comparison of water-based dyed fabric (above) and reverse micellar dyed fabric (below).

**2.5. Color Fastness.** The washing wetfastness results of color change and staining of dyed samples are examined. It shows that the color-retaining effect of nonane-reverse micellar dyeing is comparable to that of water-based dyeing in all color depth with a rating of 4−5. In the case of staining, nonane-reverse micellar dyed fabrics have the color fastness similar to laundering as water-based dyed fabrics in all color depth with a rating of 4−5. On the basis of the color fastness to laundering results, it can be concluded that nonane-reverse micellar dyeing system can provide dyeing properties similar to the conventional water-based dyeing system.

**2.6. VOC Content.** The detection of VOC leakage during the reverse micellar dyeing process was performed every 20 min. The VOC content was detected from 0 to less than 2 ppm

in the first 20 min. Until approximately 105 min, the VOC content reaches a steady level (2.4 ppm). The nonane leakage is in the range of 2−3 ppm during the reverse micellar dyeing process, which is at the low and acceptable level.

**2.7. Recovery of Solvent.** From the collection of distillate, the percentage of solvent recovery is based on the ratio of the amount of recycled solvent to the theoretical prediction of solvent volume used before dyeing. The nonane (98%) can be recovered for further use after the reverse micellar dyeing progress, and the percentage of recovery of nonane can be high in efficiency.

**2.8. Surface Morphology.** Scanning electron microscopy (SEM) has been applied to observe the surface characteristics of cotton fibers. Observation by SEM helps in distinguishing between reverse micellar dyed and water-based dyed samples at the fiber level. One can demonstrate the existence of a coating through pictorial evidence and obtain further structural information at micrometer and submicrometer length scales.[31] Figure 4 shows the SEM images before and after water-based and nonane-based dyeing processes. The surface of the untreated cotton (Figure 4a) reveals its characteristic natural and regular ridges with smooth surface. The water-based dyed samples under both 0.1 and 3.5% dyes do not show any fine structure on the fiber surface (Figure 4b,c). For cotton samples dyed in reverse micellar dyeing system (Figure 4d,e) under both 0.1 and 3.5% dyes, some filamentous structures are observed on the surface of most fibers in large dimension. The surfaces appear rougher overall with fiber surface dyed in water. For both cases, the surface features of reverse-micellar dyed cotton are clearly different from those of the water-based dyed and untreated cotton. The regularity of natural stripes along cotton fabric surface had almost vanished in the reverse micellar dyed cotton. This indicates that, after treatment with reverse micelle encapsulated dye in nonane, irregular stripes and rough surfaces can significantly increase the surface energy of cotton fabric, which is favorable for better diffusion into cotton matrix and interaction of dye with the cotton surface. The results of the reflectance curves indicated that the lower reflectance values obtained from reverse micellar dyed cotton reveal relatively more dye molecules adsorbed on the fiber surface than that in water-based dyeing process.

## 3. CONCLUSIONS

In this study, we investigated the possibility of using PEG-based reverse micelle in nonane medium for dyeing cotton with reactive dyes with three primary colors as well as mixed colors. The reflectance percentage ($R$ %) of the reverse micellar dyed samples in nonane medium was measured. No chromatic change was noted when compared with water-based dyed samples. The calibration dyeing databases for both dyeing approaches were setup, and the calibration curves of nonane-reverse micellar dyed samples are linear. The values of $R^2$ are between 0.9959 and 0.9995. The values of $K/S_{total}$ of nonane-dyed samples were generally higher than that of water-dyed samples for each primary color. CCM was initiated with the use of several color difference equations. The results revealed that the predicted concentrations were similar to the known concentrations for both approaches. These indicates that dyeing of cotton using reverse micelle approach in nonane medium can achieve excellent color matching. RUI has been used to evaluate the levelness of the dyed samples. The results indicate that nonane-dyed samples can achieve good to excellent levelness, comparable to the conventional aqueous-
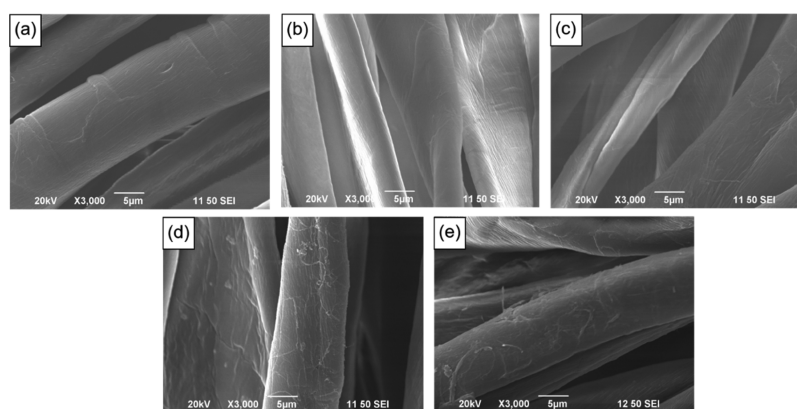
**Figure 4.** SEM micrographs of cotton fabric surface (a) undyed; (b) 0.1% dye in water; (c) 3.5% dye in water; (d) 0.1% in nonane; and (e) 3.5% in nonane.

based dyeing approach. In addition, color fastness to laundering results of the nonane-reverse micellar dyed samples was found acceptable in comparison with the water-dyed samples. Meanwhile, low VOC content but high solvent recovery efficiency could be achieved for the proposed reverse micellar dyeing process in nonane nonaqueous medium.

## 4. EXPERIMENTAL SECTION

**4.1. Fabric.** Pure-cotton interlock-knitted fabrics with fabric structure of 40 wales per in. (wpi) and 40 courses per in. (cpi) were initially cleaned with a solution, which contains 2 g/L soda ash and 2 g/L soap in a shaking water bath at temperature of 70 °C for 45 min. The cleaned fabrics were then cold-rinsed, tumble-dried, and conditioned in a room set at relative humidity of 65 ± 2% and temperature of 20 ± 2 °C for approximately 24 h before further experiments.

**4.2. Reagents.** PEG (12) tridecylether $(C_{13}H_{27}(OCH_2CH_2)_nOH)$ ($n \approx 12$) (PEG-12), a nonionic PEG-based surfactant, and *n*-octanol, a cosurfactant, were used to facilitate the formation of reverse micelles. Nonane $(C_9H_{20})$, an alkane solvent, was used as the nonaqueous dyeing medium for reverse micellar dyeing, whereas deionized water was used as the aqueous dyeing medium in conventional dyeing process. Sodium chloride (NaCl), an inorganic salt, was used as an exhausting agent in conventional aqueous-based dyeing only, whereas soda ash $(Na_2CO_3)$, an alkali commonly used in conventional dyeing, was used as a color fixation agent for both dyeing methods. They were of reagent grade. Reactive dyes of three primary colors (Levafix Yellow CA, Levafix Red CA and Levafix Blue CA) used were provided by DyStar from China, and they were directly used with no further purification.

**4.3. Cotton Dyeing in Water for Establishing Calibration Curves.** To ensure good levelness, liquor-to-goods ratio of 50:1 was used for calibration dyeing, and the dyeing recipe (dye concentration, amount of auxiliaries) was shown in Table 1. On the basis of the dye concentration (percentage on weight of fabric, % owf), the corresponding amount of NaCl was added during the dyeing process. To carry out the calibration dyeing in aqueous medium, cotton fabric was initially immersed in the dye liquor containing salt and dye. The prepared dye liquor was then put into the shaking water bath set at 30 °C. The dyeing process was launched at 60 °C for 40 min, followed by color fixation by adding the corresponding amount of $Na_2CO_3$ (Table 3) into the dye liquor at 60 °C for an hour. To remove the unfixed dye and

**Table 3. Dye Recipe for Calibration Dyeing**

| dye concentration used (% owf[a]) | auxiliaries for water-based dyeing | | auxiliaries for reverse micellar dyeing |
|---|---|---|---|
| | NaCl (g/L) | Na₂CO₃ (g/L) | Na₂CO₃ (color fixing agent to cotton weight ratio, g/g) |
| 0.1 | 10 | 5 | 0.06 |
| 0.5 | 20 | 5 | 0.06 |
| 1.5 | 42.5 | 5 | 0.08 |
| 2.5 | 55 | 5 | 0.08 |
| 3.5 | 65 | 5 | 0.09 |

[a]% owf means percentage of dye on weight of fabric.

auxiliary residues in the fabrics, soaping treatment, which immerses the dyed cotton fabric in soap solution (2 g/L) and rinses for 15 min at 60 °C for 2 times, was conducted after the fixation process. The cleaned fabrics were then cold-rinsed, tumble dried, and conditioned in a room set at a relative humidity of 65 ± 2% and a temperature of 20 ± 2 °C, for approximately 24 h before further experiments.[24,25] The dyed fabrics are termed as batch samples for establishing the calibration curves.

**4.4. Cotton Dyeing in Reverse Micellar System for Making Calibration Curves.** PEG-based nonionic reverse micelles were simply prepared by injection at room temperature. The PEG-12 nonionic surfactant and cosurfactant (*n*-octanol) were first premixed in a surfactant to cosurfactant ratio (1:7 in mole ratio) with agitation. The mixture was then dissolved in nonane to facilitate the self-assembly of PEG-based reverse micelles. Solvent-to-cotton weight ratio (v/w) used was 8:1. Reactive-dye aqueous solutions of controlled amount were injected dropwise into the PEG-based reverse micellar system and were then stirred vigorously for 2 min until the solutions with reverse micelle, encapsulated reactive dye, were well-dispersed[24,25] (Table 4).

Calibration dyeing was initiated at surfactant to water ratio of 1:20 mole ratio. The reverse micellar dye liquors were first

**Table 4. Recipe for Preparation of Reverse Micelle Solution per gram of Cotton**

| | amount |
|---|---|
| surfactant (g) | 1.56 |
| cosurfactant (g) | 2.03 |
| solvent (mL) | 8.00 |

prepared by the addition of corresponding amounts of surfactant, cosurfactant, dye solutions, and nonane solvent. The whole dyeing and fixation processes were conducted without addition of electrolytes [salt, (NaCl)]. To carry out the dyeing, the cotton fabric was first immersed in the dye liquor containing reverse micelle encapsulated reactive dye (in a stoppered flask) and was then put into the shaking water bath. The dyeing of cotton was conducted at 70 °C for 40 min. After that, fixation of dye was conducted by the addition of soda ash (Table 1) into the dye liquors at 70 °C for an hour. After fixation, soaping treatment, which immerses the dyed cotton fabric in soap solution (2 g/L) and rinses for 15 min at 60 °C for two times, was conducted after the fixation process. The cleaned fabrics were then cold-rinsed, tumble dried, and conditioned in a room set at relative humidity of 65 ± 2% and temperature of 20 ± 2 °C for approximately 24 h before further experiments.[24,25] The dyed fabrics are termed as batch samples for establishing the calibration curves.

**4.5. Calibration Curves Setup.** The color yield of the batch samples was obtained by using a Macbeth Color-Eye 7000A spectrophotometer (X-Rite, USA) to measure the reflectance ($R$ %) and $K/S$ values on the face side of the dyed fabrics. The measurement was set according to the following parameters: (i) specular included; (ii) large aperture (30 mm); (iii) folding the samples two times to ensure opacity; (iv) illuminant $D_{65}$ light source; and (v) 10° standard observer. The calibration curves were plotted with $K/S_{total}$ value against concentration of dye on the weight of fiber (% owf). $K/S$ values at specific wavelengths were calculated by eq 1. In this study, the color yield, expressed in terms of $K/S_{total}$ value, was calculated by the summation of each $K/S$ values between 400 and 700 nm wavelength, within the visible spectrum with data taken at 10 nm intervals. The higher the $K/S_{total}$ value, the more will be the dye uptake by the dyed fabrics and the better will be the color yield. After inputting $K/S_{total}$ values and the related concentration into the spectrophotometer, calibration curves could be established in the spectrophotometer for computerized color matching.

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

where $K$ is the absorption coefficient depending on colorant concentration; $S$ is the scattering coefficient caused by the dyed sample; and $R$ is the reflectance of the dyed sample at a specific wavelength.[22,23]

**4.6. Simulated Dyeing with Known Dye Concentration.** To prepare samples for CCM, simulated dyeing with a known concentration of dye was launched to produce color mixture for both conventional aqueous-based and nonane-assisted reverse micellar methods. The cotton fabrics were dyed as the standard samples. The mixture of color of the standard samples was produced based on the dye concentration presented in Table 5.

**4.7. Dye Recipe Prediction with Color Difference Formulae.** Different color difference formulae, which include CIEL*u*v*, CIEL*a*b*, HunteLab, ANLAB, FMC2, JPC79, BFD 1.0, CMC 1.0, and CIE94 1.0, were used for predicting the dye recipe for the standard samples; the standard samples were prepared by mixing the known concentration of dye in the dye recipe (Table 3). The $K/S_{total}$ of the dyed standard samples was measured by Color-Eye 7000A spectrophotometer (X-Rite, USA) using light source illuminant $D_{65}$ under specular included condition. Standard observer of 10° with a large aperture (30 mm) was used. By obtaining the $K/S_{total}$ value and using the

**Table 5. Dye Concentration (% owf) for the Preparation of Color Mixture**

| | | red (% owf) | yellow (% owf) | blue (% owf) | total (% owf) |
|---|---|---|---|---|---|
| conventional water-based dyeing | standard sample 1 | 0.1 | 0.1 | 0.1 | 0.3 |
| | standard sample 2 | 0.5 | 0.5 | 0.5 | 1.5 |
| | standard sample 3 | 1.0 | 1.0 | 1.0 | 3.0 |
| reverse micellar dyeing | standard sample 4 | 0.1 | 0.1 | 0.1 | 0.3 |
| | standard sample 5 | 0.5 | 0.5 | 0.5 | 1.5 |
| | standard sample 6 | 1.0 | 1.0 | 1.0 | 3.0 |

established calibration curves in the spectrophotometer, the concentration of dye in the recipe to produce the standard samples could be predicted by different color difference formulae. As a result, the known dye concentration (termed as "known concentration") and predicted dye concentration (termed as "predicted concentration") obtained from CCM in conventional aqueous-based and nonane-reverse micellar dyeing methods could be compared for accuracy.

**4.8. Levelness Evaluation.** The RUIs were used for the evaluation of the levelness of the dyed batch and standard sample and were calculated through the use of reflectance values of four randomly selected spots on the dyed standard and batch samples within the visible spectrum ($\lambda = 400-700$ nm) with data taken at 10 nm intervals. The apparatus used was Macbeth Color-Eye 7000A spectrophotometer (X-Rite, USA) using the following parameters as the condition of measurements: (a) illuminant $D_{65}$ light source; (b) specular included; (c) standard observer of 10°; and (d) large aperture (30 mm). The RUI was composed of four main equations. It includes (a) the standard deviation ($s_\lambda$) of reflectance measured at a specific wavelength (eq 2); (b) the uncorrected RUI ($RUI_u$) derived by the sum of standard deviations for all wavelengths (eq 3); (c) corrected RUI ($RUI_c$) derived by the sum of coefficients of variation of reflectance values (eq 4); and (d) RUI derived by multiple coefficients of variation of reflectance by the photopic relative luminous efficiency function ($V_\lambda$) (eq 5).[26] The interpretation of RUI values as suggested is (i) excellent levelness (unlevelness not detectable) (RUI < 0.2); (ii) good levelness (noticeable unlevelness under close examination) (RUI = 0.2−0.49); (iii) poor levelness (apparent unlevelness) (RUI = 0.5−1.0); and (iv) bad levelness (conspicuous unlevelness) (RUI > 1.0).

$$s_\lambda = \sqrt{\frac{\sum_{i=1}^{n}(R_i - \overline{R})^2}{n-1}} \tag{2}$$

$$(RUI_u) = \sum_{\lambda=400}^{700} s_\lambda \tag{3}$$

$$(RUI_c) = \sum_{\lambda=400}^{700} s_\lambda/\overline{R} \tag{4}$$

$$RUI = \sum_{\lambda=400}^{700} (s_\lambda/\overline{R})V_\lambda \tag{5}$$

**4.9. Color Fastness.** The color fastness to laundering of the batch samples was evaluated by AATCC Test Method 61-1A (2013) (color fastness to Laundering: Accelerated). The color change of the dyed fabric and staining of adjacent multifiber fabric were evaluated, and the rating of 5 is the excellent rating whereas 1 is the poorest rating.

**4.10. VOC Content.** A GX6000 gas detector (range 0−6000 ppm) was used for detecting the VOC content during the reverse micellar dyeing process.

**4.11. Recovery of Solvent.** After reverse micellar dyeing, 40 mL of the dyeing solution was placed into 100 mL round-bottom flask with magnetic stirring for distillation. The round-bottom flask was immersed in an oil bath and connected with a thermometer and a condenser tube via a three-way adapter. The distillation process was controlled at a rate of 1 drop per s under appropriate heating power. The temperatures measured at the intersection region of the three-way adapter was 149 °C for nonane. The distillate was completely collected when the temperature starts to drop and no further distillates were collected after condensation. The collected distillates were then transferred into a separatory funnel and two-phase layers can be observed. A small amount of water was at the bottom layer, and the upper layer was the recycled solvent.

**4.12. Surface Morphology.** SEM was used to observe the surface textures of untreated, water-treated, and nonane-treated cotton fiber samples. The SEM used is a JSM-6490 field-emission scanning electron microscope (JEOL, Japan), accompanied with an energy-dispersive X-ray spectroscopy system at 20 kV accelerating voltage. The samples were sputtered with a layer of thin conductive gold, prior to the observation.

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**Notes**
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