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The use of sodium edate in dyeing: II. Union dyeing of cotton/wool blend with hetero bi-functional reactive dyes

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Abstract

Union dyeing of cotton/wool blend with hetero bi-functional reactive dyes, namely CI Reactive Yellow 145, CI Reactive Red 194, and CI Reactive Red 195 is obtained using one-bath and two-bath dyeing processes. The results obtained are compared with those obtained by conventional two-bath dyeing process. Factors affecting the dyeability, such as the amount of sodium edate, sodium sulphate, temperature and dye concentration are investigated. The dyed fabrics were evaluated with respect to the dye exhaustion and fixation, levelling and the fastness properties. Excellent to good fastness was obtained for all samples irrespective of the dyeing method. The result of investigation offers a new viable method for union dyeing by using sodium edate two-bath and one-bath processes with a better levelling and dye uptake.

Keywords: Textile auxiliary; Sodium edate; Union dyeing; Cotton/wool blend; Bi-functional reactive dyes

1. Introduction

Cotton/wool fabrics are rapidly increasing in consumer interest for value, comfort, versatility, and styling. These blends are found in sport and active wear, light to bottom weights, and home furnishings. It is a comfortable, durable and attractive fabric. The complementary properties of these fibers include wool's high elongation and weakness at low stress and cotton's resistance to elongation and strength at high stress. Wool's low thermal conductivity and hygroscopicity, leading to apparent dryness, complement cotton's propensity to dissipate heat and absorb moisture with easy wettability. While cotton maintains high thermal conductivity, wool retains low thermal conductivity up to moisture regains of about 15% [1].

The conventional method of union dyeing (uniformly dyeing or solid shade dyeing) requires reactive or acid dyes for wool and reactive or direct dyes for cotton in a two-bath process. However, the selection of compatible dyes with a better colour match for union dyeing of the blend, the preferential dye uptake by wool and the alkaline sensitivity of wool remains of much concern. In this regard, Ciba–Geigy have produced Cibacron F dyes based on monofluorotriazine that fixes well with cotton at low fixation temperature (60 °C) and pH (9.5) and have applied these dyes in combination with Lanasol wool fiber reactive dyes in a two-bath dyeing process [2].

Alternatively, one-bath dyeing method using one dye would be of interest as it offers the desired union shade together with energy saving. It has been reported that Cibacron F dyes could also be applied for dyeing the

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blend in a one-bath process by firstly dyeing the cotton component and then adjusting temperature and pH to dye the wool component with the residual hydrolysed dye that behaves like acid dyes [2]. On the other hand, union dyeing of the blend with anionic dyes in a one-bath dyeing process has been obtained after selective chemical modification by pre-treatment of the cotton to render it aminised or cationised so as to behave like wool in the dyebath [1,3–11].

Bi-functional reactive dyes with sulphatoethylsulphone (SES) group and monochlorotriazine (MCT) group have been developed and introduced to the market on a commercial basis as the Sumifix Supra (Fig. 1) range since 1979. The combination of both warm and hot dyeing (60 °C, vinyl sulphone (VS) and 80 °C, MCT, respectively) reactive groups within the same molecule allows higher fixation to be obtained with very good ‘temperature range’ properties, i.e. a similar colour yield is obtained when dyeing at temperatures between 60 and 80 °C. Furthermore, this type of dyes has a wide pH range (9.5–11.5) of fixation onto cotton that would give the optimum levelness [12–14]. Optimum dyebath pH, respectively, is reported to be between 4 and 5 [15] and between 5 and 6 [16] for the reaction of wool with MCT and VS groups present in mono functional reactive dyes. These combined properties in Sumifix Supra dyes have led to a conventional application of these dyes in a two-bath process for union dyeing of cotton/wool blends [12].

In the previous part of this paper [17], which examined the use of sodium edate in the dyeing of cotton with reactive dyes, it was found that sodium edate substantially acted as an exhausting and fixing agent for reactive dyeing of cotton. Sodium edate is majorly used in cosmetic formulations as it is environmentally safe and non-toxic biodegradable organic salt [18]. As a part of our ongoing interest in exploring the viability of using sodium edate in dyeing, we wish to report in this work the dyeability of cotton/wool blend fabrics with Sumifix Supra dyes. Different factors affecting dyeability using both two-bath and one-bath dyeing processes and fastness properties were thoroughly investigated.

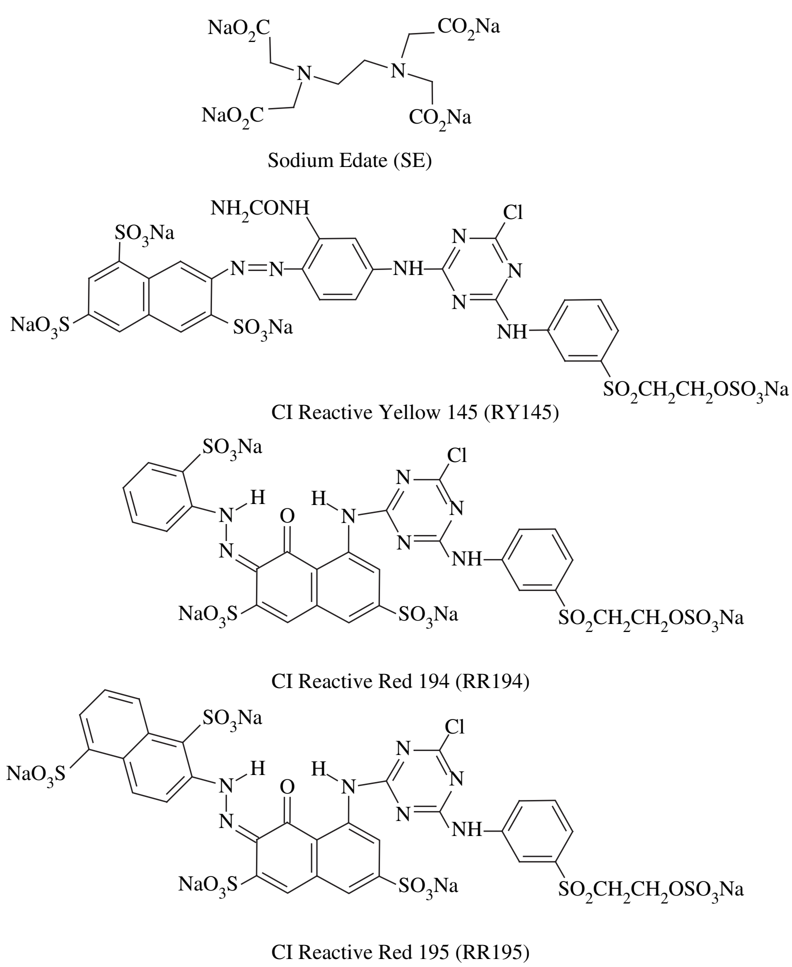

Fig. 1. Structures of dyes and sodium edate.

2. Experimental

2.1. Materials

2.1.1. Cotton/wool blend fabric

Knitted cotton/wool, 70/30, blended fabric of 310 g/m^2, supplied by Golden Tex Co., Tenth of Ramadan-Egypt, was initially treated in an aqueous solution with a liquor ratio 50:1 containing 0.5 g/l sodium carbonate and 2 g/l non-ionic detergent (Hostapal CV, from Clariant–Egypt) at 60 °C for 30 min, after which time it was thoroughly rinsed and dried at room temperature.

2.1.2. Dyestuffs and chemicals

The hetero bi-functional MCT/SES reactive dyes used (Fig. 1) were Sumifix Supra Yellow 3RF (CI Reactive Yellow 145), Sumifix Supra Brilliant Red 3BF (CI Reactive Red 195), and Sumifix Supra Brilliant Red 2BF (CI Reactive Red 194), from Sumitomo. All dyes were of commercial grade and were used as received. Sodium edate (SE) was purchased from Fluka (Germany). Albegal A, an amphoteric levelling agent was supplied by Ciba. All other chemical reagents, namely sodium sulphate (SS), ammonium sulphate and sodium carbonate, were of laboratory grade.

2.2. Dyeing procedure

All dyeings were carried out using CI Reactive Yellow 145 of 3% owf unless otherwise specified in distilled water with a liquor ratio 20:1. The selected dyeing conditions were then applied for all dyes. The reactive dyes were applied to cotton/wool blend using two-bath and one-bath dyeing methods as indicated in Fig. 2.

2.2.1. Two-bath dyeing

2.2.1.1. Wool dyeing. Dyes were first applied to wool component (2 g samples) using 1% owf Albegal A, 5% owf ammonium sulphate and 3% dye. Samples were introduced into the dyebath at 50 °C and the temperature was raised to 98–100 °C over 40 min, then the pH of the dyebath was adjusted to 4.5 by diluted acetic acid and the dyeing continued at the boil for a further 60 min. After which time, the dyebath temperature was reduced to 60 °C, and then the samples were removed from the dye pots, rinsed in cold water and warm water for 10 min. The dyed sample (2 g) was divided into two pieces, each of 1 g sample. One piece of the dyed sample was stripped under refluxing with 50% aqueous dimethylformamide (DMF) solution at a liquor ratio of 20:1 for 15 min until all the unfixed dye was removed. The other piece was subjected for dyeing cotton.

2.2.1.2. Cotton dyeing. In a dyebath containing different amounts of SS (0–50 g/l) and 3% dye, cotton/wool samples (1 g) of a pre-dyed wool component was introduced at 35 °C and the dyeing was carried out at this temperature for 15 min, then various amounts of SE (0–50 g/l) were added while the temperature was raised to 60 °C unless otherwise specified over 20 min. After which time the dyeing was continued at 60 °C for a further 60 min. On other hand, the recommended conventional dyeing (CM) procedure [12] was employed as a control for the experiments, i.e. dyeing cotton in the presence of 50 g/l SS at 35 °C for 15 min, then 10 g/l sodium carbonate was added in portions while the temperature was raised to 60 °C. The dyeing continued for a further 60 min, then the samples were rinsed and striped with 50% aqueous DMF as mentioned above.

2.2.2. One-bath dyeing

CI Reactive Yellow 145 was applied to cotton/wool blend at a liquor ratio 20:1 with 50 g/l SS and 1% owf Albegal A. The dyebath was set at 50 °C. The temperature was raised to the boil, then the pH was adjusted to 4.5 using acetic acid and the dyeing continued for 30 min. The temperature was then reduced to the desired temperature of 50–80 °C at which 0–50 g/l SE was added in portions over 15 min. The dyeing was continued at the desired temperature for further 45 min. The dyed samples were rinsed and striped with 50% aqueous DMF as mentioned above.

2.3. Measurements and analyses

2.3.1. Dye exhaustion

The uptake of the reactive dye by cotton or wool in the blend fabric was measured by sampling the dyebath before and after the dyeing. The concentration (g/l) of the dyebath was measured on Shimadzu UV-2401PC UV/visible spectrophotometer at λ_{max} of the dye. The percentage of the individual dye exhaustion on wool ($\%E_w$) and cotton ($\%E_c$) was calculated using Eqs. (1) and (2), respectively:

$$\%E_w = \left(1 - \frac{C_2}{C_1}\right) \times 100 \tag{1}$$

$$\%E_c = \left(1 - \frac{C_4}{C_3}\right) \times 100 \tag{2}$$

where C_1 and C_2 are the concentrations of dye in the dyebath before and after dyeing wool component in the blend fabric, respectively, and C_3 and C_4 are the concentrations of dye in the dyebath before and after dyeing

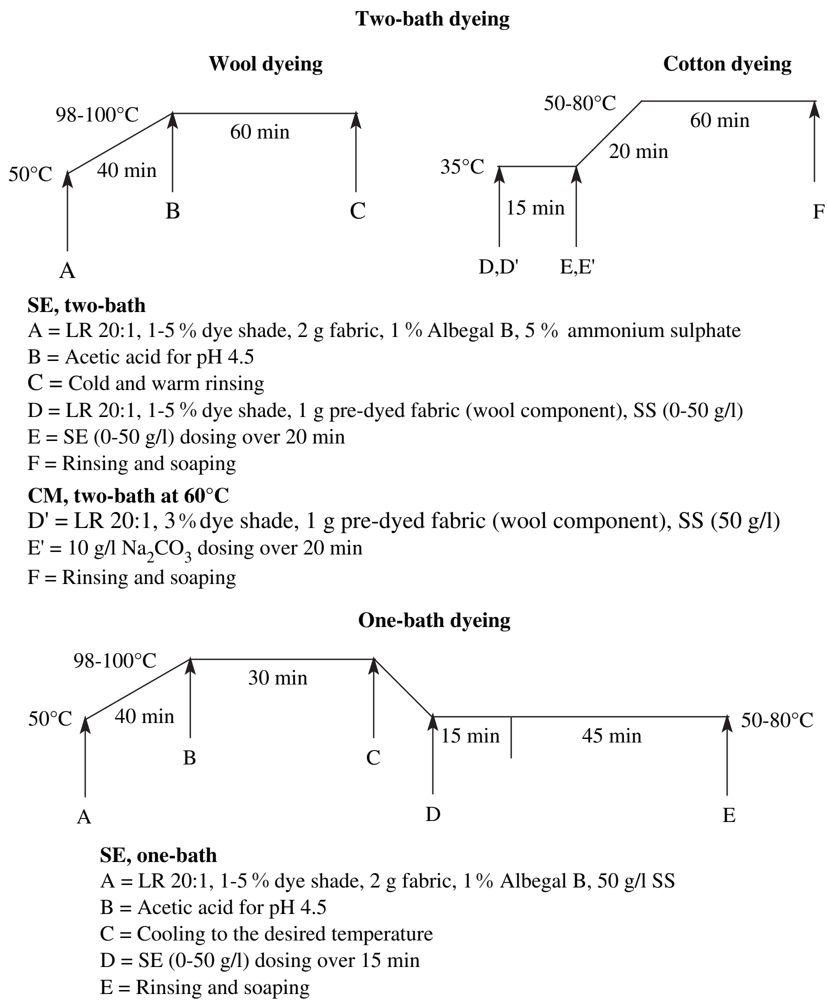

Fig. 2. Dyeing profile methods for reactive dyes used.

cotton component in the blend fabric, respectively.

By measuring the concentrations of the dye used in the dyeing process, the percentage of the dye exhaustion on the blend ($\%E_{c/w}$) at the end of dyeing could be calculated using Eq. (3):

$$\%E_{c/w} = \frac{(C_1 + C_3) - (C_2 + C_4)}{(C_1 + C_3)} \times 100 \quad (3)$$

2.3.2. Dye fixation

The dye fixation ratio ($\%F$), the percentage of the exhausted dye chemically bound on the blend fabric, was measured by refluxing the dyed samples in 50% DMF (liquor ratio 20:1) for 15 min to extract the unfixed dye. This procedure was repeated until the extract was clear. The concentration of the extract was then measured spectrophotometrically at λ_{max} and the percentage dye fixation ratio ($\%F$) on wool and cotton components was calculated using Eqs. (4) and (5), respectively [19,20]:

$$\%F_w = \frac{C_1 - C_2 - C_5}{C_1 - C_2} \times 100 \quad (4)$$

$$\%F_c = \frac{C_3 - C_4 - (C_6 - C_5)}{C_3 - C_4} \times 100 \quad (5)$$

where C_5 is the concentration of the extracted dye after dyeing wool, and C_6 is the concentration of the extracted dye after complete dyeing of cotton/wool blend.

The percentage of the dye fixation ratio on the blend fabric after extracting the dye could be calculated using Eq. (6):

$$\%F_{c/w} = \frac{(C_1 + C_3) - (C_2 + C_4) - C_6}{(C_1 + C_3) - (C_2 + C_4)} \times 100 \quad (6)$$

From the dyebath exhaustion (E) and dye fixation ratio (F), the total dye fixation (T), which is the percentage of dye chemically bound relative to the total amount of dye used, was calculated for dyeing wool ($\%T_w$) and cotton ($\%T_c$) components using Eqs. (7) and (8), respectively [19,20].

$$\%T_w = \frac{(\%E_w \times \%F_w)}{100} \quad (7)$$

$$\%T_c = \frac{(\%E_c \times \%F_c)}{100} \quad (8)$$

After complete dyeing process, the total dye fixation on the blend fabric ($\%T_{c/w}$) could be calculated using Eq. (9):

$$\%T_{c/w} = \frac{(\%E_{c/w} \times \%F_{c/w})}{100} \quad (9)$$

2.3.3. Levelling properties

Levelling properties of dyed blend fabrics using 3% owf dye applied at the selected dyeing conditions for all dyes were assessed on a GretagMacbeth Colour-Eye 7000A spectrophotometer by measuring the colour differences within each sample at five separate points and the average colour difference (ΔE) between these points was determined [21,22].

2.3.4. Fastness testing

The dyed samples were tested, after washing-off using 2 g/l non-ionic detergent (Hostapal CV) at 85 °C for 15 min, according to ISO standard methods. The specific tests were: ISO 105-X12 (1987), colour fastness to rubbing; ISO 105-C02 (1989), colour fastness to washing; and ISO 105-E04 (1989), colour fastness to perspiration.

3. Results and discussion

It is of interest to dye cotton/wool blend fabrics using hetero bi-functional reactive dyes with union shade. It is anticipated from the structure of SE (Fig. 1) that this compound in addition to its confirmed viability as exhausting and fixing agent in reactive dyeing of cotton [17], it may have the feature of being a mild organic base with a capacity of buffering action in the dyebath owing to its content of tetra sodium acetate derivatives. Additionally, SE structure is somewhat similar to wool structure regarding the amino acids content of wool and thus intimate interaction would be expected. Furthermore, the sequestering effect of SE by capturing metal ions, such as calcium or magnesium [23] that could be otherwise absorbed by wool component during the dyeing process prevents dye precipitation leading to better union dyeing of the blend. This structural feature would help fixation of reactive dyes onto cotton/wool fabrics with a better fixation rate that helps better for level dyeing. In this regard, different factors that may affect the dyeability of the blend using two-bath as well as one-bath dyeing techniques are investigated.

3.1. Effect of SE concentration in the presence of 50 g/l SS

The effect of SE concentration on the dyeability of the blend fabrics with CI Reactive Yellow 145 was conducted at different concentrations (0–50 g/l). Fig. 3 shows the exhaustion and total fixation on cotton component and the comparative results of the cotton/wool blend using two-bath and one-bath dyeing methods. It is clear that increasing the salt concentration from 0 to 10 g/l leads to a pronounced effect on the dye uptake for both dyeing methods and further increases, however, leads to a marginal increase. This result reflects the findings that SE acts as exhaustive and fixing agent.

3.2. Effect of SS concentration in the presence of 50 g/l SE

It is known that dye migration from the dyebath into the fiber is an entropy favorable process and adding

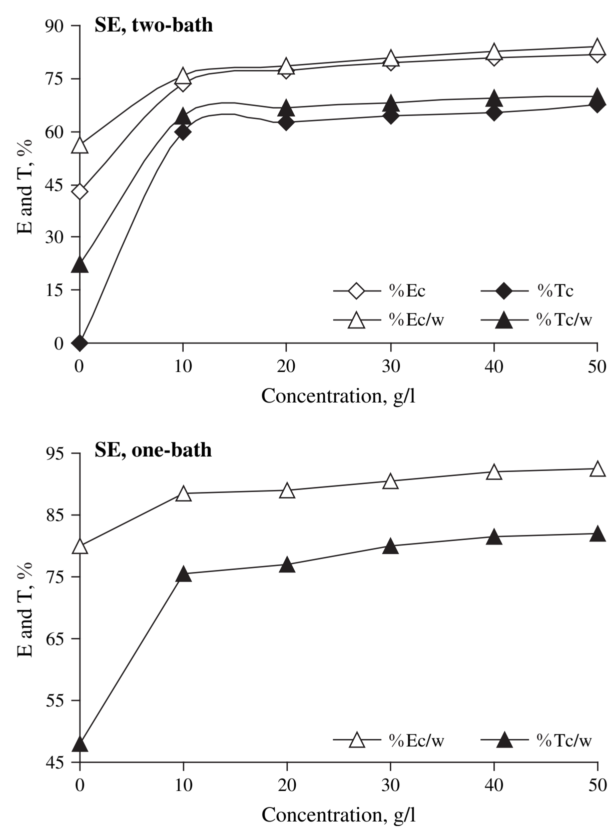

Fig. 3. Effect of sodium edate on the exhaustion and total fixation of CI Reactive Yellow 145 on cotton/wool blend fabrics.

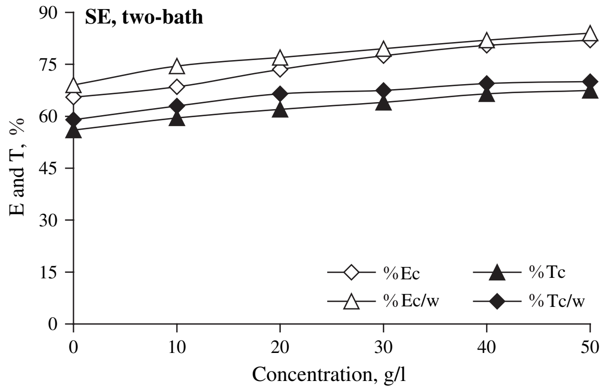

Fig. 4. Effect of sodium sulphate concentration on the exhaustion and total fixation of CI Reactive Yellow 145 on cotton/wool blend fabrics.

neutral electrolyte, such as SS, would further assist this process for a better dye uptake. Fig. 4 shows that the dye exhaustion and total fixation values for the dyed samples obtained using a two-bath dyeing method are slowly increasing with the increase in SS concentration. In part one [17] of this work, it was concluded that the colour strength values of dyed cotton samples with mono and homo bi-functional reactive dyes using SE are independent of the concentration of SS added due to the high exhausting effect of SE and its alkalinity

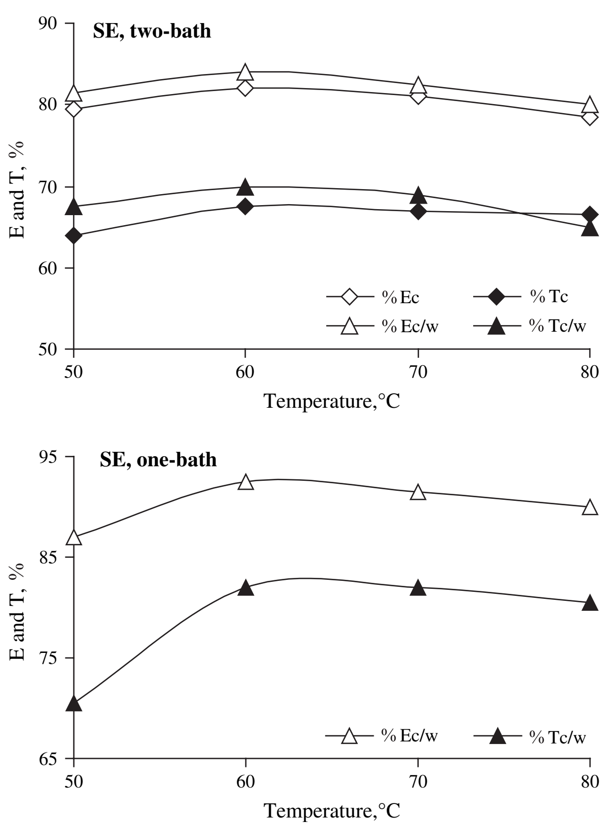

Fig. 5. Effect of temperature on the exhaustion and total fixation of CI Reactive Yellow 145 on cotton/wool blend fabrics.

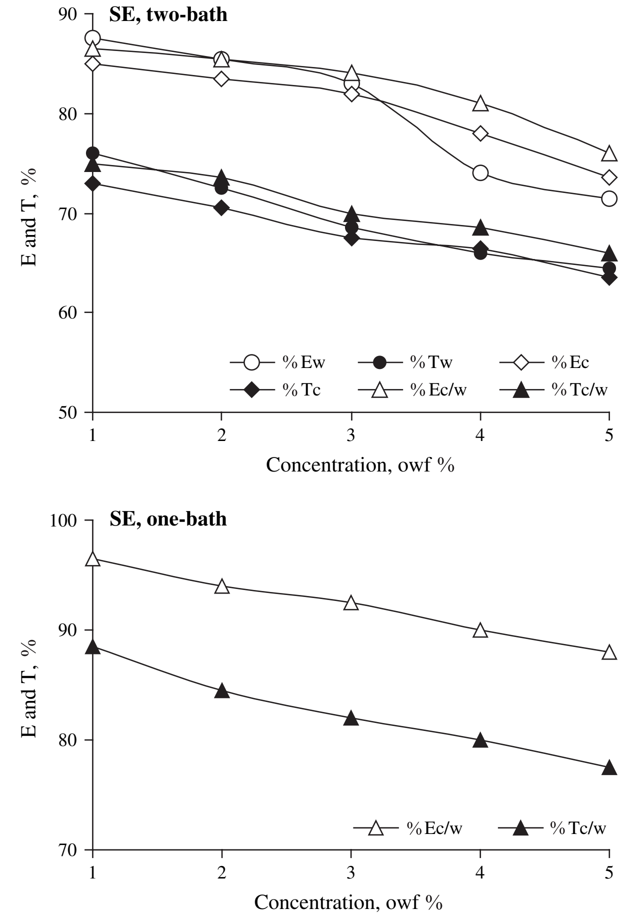

Fig. 6. Effect of CI Reactive Yellow 145 concentration on the exhaustion and total fixation on cotton/wool blend fabrics.

which as well favor more dye uptake on cotton. However, in the present work it seems that the slight improvement in the dye uptake upon increasing SS concentration may be attributed to the strong overall negative charge on the pre-dyed wool present in close proximity to cotton component. The anionic character of dye molecules seems to suppress the exhausting effect of SE. Thereby, an additional SS is necessary for modifying the equilibrium and favoring the movement of dye molecules from the solution to the cotton component in the blend fabrics.

3.3. Effect of temperature

Fig. 5 shows the effect of dyeing temperatures (50, 60, 70, 80 °C) on the dye uptake using two-bath and one-bath processes in the presence of 50 g/l SS and 50 g/l SE. An increase in the exhaustion of CI Reactive Yellow 145 on the blend fabrics is observed as the dyeing temperature increased from 50 to 60 °C. However, increasing the temperature from 60 to 80 °C was accompanied by a successive decrease in the exhaustion and total fixation values for both methods. It is known that

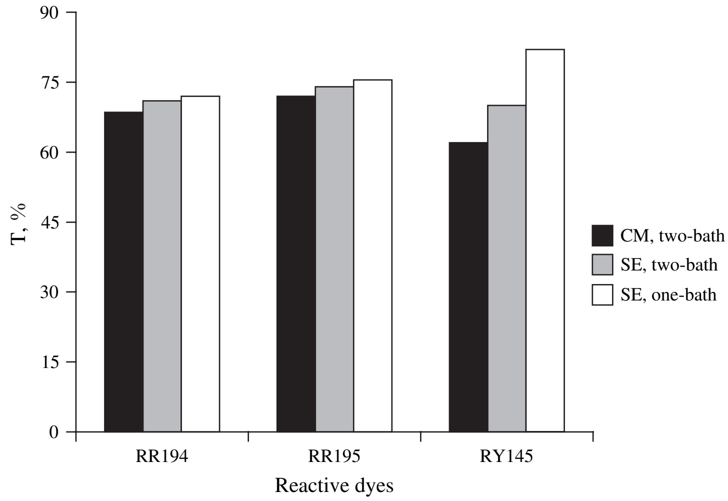

Fig. 7. Comparative total fixation of different dyes on cotton/wool blend fabrics using different dyeing methods.

increasing the temperature allows the swelling of the blend fibers, which leads to a higher dye uptake. The results indicate that 60 °C is the suitable temperature for dyeing, at which temperature a proper reaction between the dye bi-functional VS/MCT reactive system with cellulose through both VS and MCT groups is performed. On the other hand, increasing the dyeing temperature from 60 to 80 °C would be expected to reduce the reactivity level of the VS group by hydrolysis, and therefore the exhaustion and fixation yield would also decrease.

3.4. *Effect of dye concentration*

Exhaustion and total fixation of CI Reactive Yellow 145 at different concentrations (1–5% owf) were investigated in a one-bath and two-bath dyeing methods at 60 °C and in the presence of 50 g/l SS and 50 g/l SE. Fig. 6 shows that exhaustion and total fixation decrease with increasing dye concentration. This result can be explained as follows. As more dye becomes fixed to the fabric, the net negative charge on the fabric increases with the increasing concentration of sulphonate groups present on the dye. At higher concentration of dye the electrostatic repulsion between the negatively charged dye and the dyed fabric increases, resulting in a lower proportion of dye fixation. Additionally, the number of available sites present on the fibers reduces and competitive hydrolysis increases.

3.5. *Application with different dyes*

Having the selected edate dyeing conditions of 3% owf dye, 60 °C, 50 g/l SS and 50 g/l SE, it was desirable to apply other Sumifix Supra dyes, namely CI Reactive Red 194 and CI Reactive Red 195 together with CI Reactive Yellow 145 for union dyeing of cotton/wool blend fabrics to have a comparative dye uptake values between SE two-bath, SE one-bath, and CM two-bath methods. Comparison of the dye uptake values for the three dyes using CM two-bath with those obtained using SE two-bath dyeing method reveals that SE two-bath method is giving the best results in all parameters of dye uptake for all dyes. This result emphasizes the viability of using SE in place of sodium carbonate for better union dyeing.

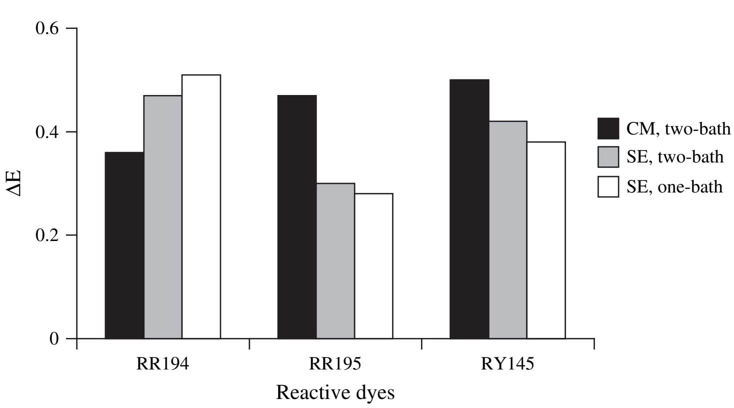

Fig. 8. Average colour differences (ΔE) of dyed cotton/wool fabrics using different dyeing methods.

Table 1
Exhaustion and total fixation values for CI Reactive Yellow 145, CI Reactive Red 194, and CI Reactive Red 195 using 3% owf dye applied at the selected dyeing conditions of each dyeing method

Reactive dyes[a]	Two-bath dyeing method	Exhaustion and total fixation (%)					
		E_w	T_w	E_c	T_c	$E_{c/w}$	$T_{c/w}$
RY145 (λ_{max}, 419 nm)	CM	83.0	68.5	73.5	60.0	75.5	62.0
	SE	83.0	68.5	82.0	67.5	84.0	70.0
RR194 (λ_{max}, 518 nm)	CM	85.5	75.0	70.5	62.5	78.0	68.5
	SE	85.5	75.0	76.0	67.0	81.5	71.0
RR195 (λ_{max}, 519 nm)	CM	86.0	75.5	75.5	67.0	81.5	72.0
	SE	86.0	75.5	78.5	69.5	83.5	74.0

[a] Molar extinction coefficients are 0.0155, 0.0234 and 0.0224 $mg^{-1} L cm^{-1}$ for RY145, RR194, and RR195, respectively.

Furthermore, performing union dyeing for the blend using SE by one-bath process also affords good results of the dye uptake relative to SE two-bath and CM two-bath methods, as shown in Fig. 7.

3.6. Levelling properties

The levelling properties of the blend fabrics dyed with the three dyes using 3% owf shade are given in Fig. 8, from which it is clear that the average colour differences (ΔE, calculated from the CIE $L^* a^* b^*$ coordinates) of the dyed blend fabrics show good levelling properties in all cases. However, it is indicated that samples obtained by SE one-bath method has the best levelling properties relative to those obtained by SE two-bath and CM two-bath methods for CI Reactive Yellow 145 and CI Reactive Red 195. Special comment about the anomalous levelling results of CI Reactive Red 194 may be as follows. The structural feature of CI Reactive Red 194 and CI Reactive Red 195 as they exist predominantly in hydrazone forms [24] makes them different in affinity toward the blend fabrics especially its cotton component. It is anticipated that hydrazone form would make these dyes more rigid with a better coplanarity relative to CI Reactive Yellow 145. Such properties make these dyes more substantive toward cotton component. Also, comparing the coplanarity of CI Reactive Red 195 with that of CI Reactive Red 194 would suggest that CI Reactive 195 is more coplanar and thus could be more substantive to cotton component of the blend relative to CI Reactive Red 194. It has been reported that substantivity tends to be incompatible with the diffusibility of the dye [25]. In this context, CI Reactive Red 194 seems better in diffusibility than CI Reactive Red 195 owing to its smaller size. On the other hand, the apparent tinctorial strength as indicated in the footnote of Table 1 for the three dyes may in part indicate that the dye content of CI Reactive Red 194 is higher than the dye content of CI Reactive Yellow 145. Therefore, the apparent higher diffusibility and higher dye content of CI Reactive Red 194 have led to lower levelling results in comparison with other dyes.

3.7. Fastness properties

As shown in Table 2, the fastness tests of washing, rubbing and perspiration of samples that had been dyed with the three dyes are excellent to good and are the same irrespective of the dyeing method and depends on the proportion of the dye fixed.

Table 2
Fastness properties of CI Reactive Yellow 145, CI Reactive Red 194, and CI Reactive Red 195 hetero bi-functional reactive dyes applied on cotton/wool blend at 3% owf dye concentration using different dyeing methods

Dye	Dyeing method	Rubbing		Washfastness			Perspiration					
		Dry	Wet	Alt	SC	SW	Alkaline			Acidic		
							Alt	SC	SW	Alt	SC	SW
RY145	CM two-bath	4–5	4	4–5	4–5	4–5	4–5	4–5	4–5	4–5	4–5	4–5
	SE two-bath	4–5	4	4–5	4–5	4–5	4–5	4–5	4–5	4–5	4–5	4–5
	SE one-bath	4–5	3–4	4–5	4–5	4–5	4–5	4–5	4–5	4–5	4–5	4–5
RR194	CM two-bath	4	3–4	4–5	4	4–5	4–5	4–5	4–5	4–5	4	4–5
	SE two-bath	4–5	3–4	4–5	4	4–5	4–5	4–5	4–5	4–5	4–5	4–5
	SE one-bath	4	3–4	4–5	4	4–5	4–5	4–5	4–5	4–5	4	4–5
RR195	CM two-bath	4	3–4	4–5	4	4–5	4–5	4–5	4–5	4–5	4–5	4–5
	SE two-bath	4	3–4	4–5	4–5	4–5	4–5	4–5	4–5	4–5	4–5	4–5
	SE one-bath	4	3–4	4–5	4–5	4–5	4–5	4–5	4–5	4–5	4–5	4–5

4. Conclusion

The dyeability of cotton/wool blend fabrics with hetero bi-functional reactive dyes using sodium edate in the dyebath was investigated using three different dyeing methods. The results indicate that sodium edate two-bath and one-bath techniques were the best relative to the two-bath conventional dyeing method. This success in process optimization for better union dyeing of cotton/wool blend would suggest the viability of its application in industrial scale. Future work is being in progress for further exploration for the use of sodium edate in dyeing.

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