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Simultaneous dyeing and antibacterial finishing for cotton cellulose using a new reactive dye

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ABSTRACT

Simultaneous dyeing and antibacterial finishing for cotton fabric using a new antibacterial reactive dye having a modified chemical structure to the commercial reactive dye CI Reactive Red 198 were studied. This modification was carried out by replacing metanilic acid in the commercial dye with 4-amino-N-(4,6-dimethylpyrimidin-2-yl)benzenesulfonamide (sulfadimidine). Optimum exhaustion and fixation values were achieved at 60 g/l sodium sulphate and 20 g/l sodium carbonate for both dyes. The modified dye exhibited higher substantivity, exhaustion and fixation efficiency compared to the commercial dye. Antibacterial activities of the dyed samples at different concentrations of both dyes were studied against gram positive (*Staphylococcus aureus*) and gram negative (*Escherichia coli*) bacteria. The cotton dyed with the modified dye shows higher antibacterial efficacy compared to the dyed cotton fabric using the commercial dye, especially on gram negative (*E. coli*) bacteria. All the reactive dyeings also exhibited high fastness properties.

1. Introduction

Cotton textiles in contact with the human body offer an ideal environment for microbial growth, antibacterial finishes are now highly desirable for textiles, especially textile materials used in hospitals and hotels are liable to control cross infection and disease transmission (Gupta, Khare, & Laha, 2004). Attempts have been made to incorporate antibacterial agents into cotton textiles before and/or after dyeing, for example the incorporation of poly(hexamethylene biguanide) as a bactericide for fabrics into undyed and dyed cotton with bis(monochlorotrizine) reactive dyes has been investigated, and reported that the presence of covalentaly bound reactive dye on cotton increases the capacity of the fibre to uptake poly(hexamethylene biguanide) (Kawabata & Taylor, 2004). The same meaning also has been investigated by examining the capacity of cotton treated with antimicrobial agent (Reputex-20) to uptake vinyl sulphone based reactive blue dye compared with that of untreated dyed cotton. It was concluded that the antimicrobial effect of the treated dyed cotton are found to be better compared to the untreated dyed cotton (Ali, Saleem, & Umbreen, 2009).

Reactive dyes are the only textile colourants designed to form covalent bond with the substrate during the application process, reactive dyes furnish a wide gamut of shades of good light fastness and excellent wash fastness on cotton. Such properties place this class of dyes at the quality end of the market (Renfrew & Taylor, 1990). For the importance of reactive dyes to cotton fabric, the idea of this work concentrates on how to develop a well-known reactive dye to increase its antibacterial effect by incorporating an antibacterial group to the dye structure, as sulfonamide moiety. Sulfonamides are an important class of antibacterial drugs used in medicine and veterinary practice. Comprehensive descriptions on the analytical aspects of some important sulfa drugs have been reviewed (Papastephanou & Frantz, 1978; Rudy & Senkowski, 1973; Stober & Dewitte, 1982; Woolfenden, 1977). The application of sulfonamides in the field of dyes has been previously reported. A series of sulfadimidine azo dyes has been synthesized via coupling of sulfadimidine diazonium salt (as one of sulfonamides) with N-substituted anilines, these dyes were embedded in a chitosan colloid and applied to the surface of cotton fabric by the pad-dry cure technique (Gaffer, Gouda, & Abdel-Latif, 2013).

In the present work we choose sulfadimidine group to be incorporated into a commercial reactive dye structure Remazol Red RB (CI Reactive Red 198) by replacing metanilic acid with Sulfadimidine group resulting a modified reactive dye **1**.

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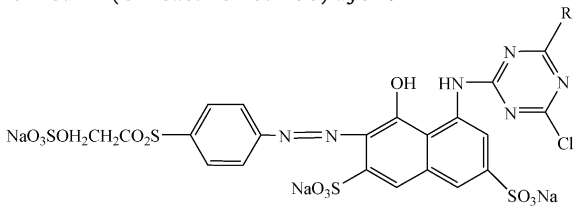
Studying the dyeing behaviour and the antibacterial activity on cotton fabric for the modified reactive dye **1** compared with Remazol Red RB (CI Reactive Red 198) dye **2**.



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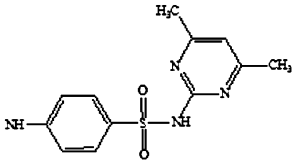
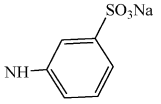


Modified Reactive Dye **1**

R =



Dye **2** (CI Reactive Red 198)

2. Experimental

2.1. Materials

Mill-scoured and bleached cotton fabric, 130 g/m^2 was obtained from Misr company for spinning and weaving El-Mahala El-Kobra, Egypt. Before dyeing, the fabric was treated with a solution containing 3 g/l non-ionic detergent (Hostapal CV, Hoechst) and 5 g/l sodium carbonate at liquor ratio 1:50 at boiling for 4 h, thoroughly washed in water and dried at room temperature.

Cyanuric chloride was obtained from Aldrich. 1-Aminobenzene-4-β-sulphatoethylsulphone was obtained from Amar Impex, Mumbai, India and sulfadimidine was obtained from Acros. All other chemicals and solvents used in this study were of laboratory reagent grade.

Remazol Red RB (CI Reactive Red 198) dye **2** was obtained from DyStar.

Antibacterial activity of the dyed cotton fabric was evaluated by a quantitative shake-flask method antibacterial test method. A gram-positive bacterium *Staphylococcus aureus* ATCC X6538P and a gram-negative bacterium *Escherichia coli* ATCC 25922 were used as the test organisms. They were provided from the Regional Center for Mycology and Biotechnology, AL-Azher University, Nasr City, Cairo.

2.2. Methods

2.2.1. Synthesis of modified reactive dye *1*

The synthesis of the modified reactive dye **1** was carried out by initial preparation of monochlorotriazinyl H-acid coupling component by dissolving cyanuric chloride (3.88 g; 0.02 mol; 95%) in acetone, poured on ice then supplied with H-acid solution (7.98 g; 0.02 mol; 80%) over 30 min, the mixture was stirred at 0–5 °C for 4 h while controlling the pH at 3.5 using 2 M aqueous solution of sodium carbonate. The reaction mixture was further supplied for a second condensation with sulfadimidine (5.68 g; 0.02 mol; 98%) and stirred at 30 °C, pH 5.5–6 for 5 h. The condensation product was then coupled with the diazonium salt of 1-aminobenzene-4-β-sulphatoethylsulphone (5.92 g; 0.02 mol; 95%) which was diazotized by the method previously described (Lewis, Renfrew, & Siddique, 2000). The produced dye was precipitated by adding 15% sodium chloride (w/v), filtered off and dried in a vacuum oven at 30 °C. The dye had a maximum absorption λ_{max} (H_2O) = 515.4 nm, its purity was determined by elemental analysis, and gave the following results:

$C_{33}H_{26}N_{10}O_{15}S_5Na_3Cl$ (1067.38) calculated: C, 37.13; H, 2.46; N, 13.12.

Found: C, 36.96; H, 2.28; N, 13.02.

M.p. > 300 °C, yield (74%); IR (KBr): υ/cm^{-1} = 3473 (OH), 1550 (—N=N—), 1166–1135 and 1084–1045 (—SO_2—). ^{1}H NMR: δH (ppm) in [$_2H^6$]DMSO: 2.2, 2.3 (s, 6H, 2CH_3), 3.59–3.61 (2H, *t*, *J* = 6.9 Hz, α-$CH_{2\,(SES)}$), 3.92–3.95 (2H, *t*, *J* = 6.9 Hz, β-$CH_{2\,(SES)}$), 6.69 (s, 1H, pyrimidine Ar—H), 7.46, 7.87, 7.88 (s, 3H, naphthoic Ar—H), 7.90–7.97 (dd, 4H, SO_2NHAr—H), 8.01–8.03 (dd, 4H, N=N—Ar—H), 14.11 (s, 2H, SO_2NH_2).

2.2.2. Dyeing procedure

A series of dyeings on cotton fabric was produced using both dyes **1** and **2** at a liquor ratio 40:1 at various dye concentrations (1–5% owf). The dyeing was started at 40 °C, for 45 min, during this period 20, 40 and/or 60 g/l sodium sulphate was added in three portions at an interval of 10 min. Then various amounts of sodium carbonate (5–25 g/l) were added portionwise while the temperature was raised to 60 °C over 30 min. After which time, the dyeing was continued at the desired fixation temperature for a further 60 min. After dyeing, all the dyed samples were rinsed with water and air dried.

2.3. Measurements

2.3.1. General

The infrared (IR) spectra were recorded on a Nexus 670 FT-IR Spectrometer (KBr; Thermo Nicolet, USA).

The ^{1}H NMR spectra were recorded on a JEOL 500 MHz spectrometer (Japan) using TMS as an internal standard and the chemical shift values are expressed in δ ppm and *J* values given in Hz.

The wavelength of maximum absorption (λ_{max}) was measured on a Shimadzu UV-2401PC UV/vis spectrophotometer (Shimadzu, Japan).

2.3.2. Dye exhaustion

For all dyeings, the dye exhaustion was measured by sampling the dye-bath before and after dyeing. The dye concentration (g/l) of the dye-bath was measured on Shimadzu UV-2401PC UV/vis spectrophotometer at λ_{max} for each dye. The percentage of dye-bath exhaustion (%*E*) was calculated using Eq. (1):

$$\%E = \left[1 - \left(\frac{C_2}{C_1}\right)\right] \times 100 \quad (1)$$

where C_1, C_2 are the concentrations of the dye-bath before and after dyeing, respectively.

2.3.3. Dye fixation

The dye fixation ratio (%*F*); the percentage of the exhausted dye chemically bound on the fibre, was measured by refluxing the dyed samples in 50% aqueous DMF (liquor ratio 20:1) for 10 min to extract the unfixed dye (Bredereck & Schumacher, 1993). 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*) was calculated using Eq. (2):

$$\%F = \frac{C_1 - C_2 - C_3}{C_1 - C_2} \times 100 \quad (2)$$

where C_3 is the concentrations of extracted dye.

From the results of the dye-bath exhaustion (*E*) and dye fixation ratio (*F*), the total dye fixation (*T*), which is the percentage of the

dye chemically bound relative to the total amount of dye used, was calculated for all dyeings using Eq. (3):

$$\%T = \frac{\%E \times \%F}{100} \quad (3)$$

2.3.4. Antibacterial test method

Tryptic soy agar and trypyic soy broth were used to grow the bacterial cultures of *E. coli* and *S. aureus*, respectively. Bacteria were cultivated at 35 °C for 24 h. Potassium hydrogen phosphate buffer solution (pH 7.2) used as a test medium. Sterile potassium hydrogen phosphate buffer solution (100 ml) was added to sterile Erlenmeyer flask (300 ml), which was the inoculated with 1.0 ml of a bacterial inoculum and a piece (1 cm × 1 cm) of dye free fabric. Time zero counts were made by removing 1.0 ml of aliquots from the Erlenmeyer flask, and triplicate withdraw of 0.1 ml of it was placed in a separate media and after 24 h of incubation at 37 °C, the initial number of bacterial colonies or the zero time counts of viable bacteria were determined. 1 g of sterile dyed fabric cut into pieces (1 cm × 1 cm) was put in the flask and shaked for 1 h. 1 h counts were made in accordance with the above described procedure.

The flask was shaked at 37 °C for the prescribed different times (1–6 h) and three repeats were needed for each sample.

The percentage of bacteria reduction (R, %) was calculated using the equation: $R = [(B - A)/B] \times 100$, where B is number of bacterial colonies from undyed cotton fabric, and A is number of bacterial colonies from dyed cotton samples (Ye et al., 2005).

2.3.5. Fastness testing

The dyed samples were tested, after washing-off using 2 g/l non-ionic detergent at 60 °C for 30 min, according to ISO standard methods (Bradford, 1990). The wash fastness test was carried out in accordance with ISO 105-C04 (1989). Other specific tests used were as follows: colour fastness to rubbing, ISO 105-X12 (1987); colour fastness to perspiration, ISO 105-E04 (1989) and colour fastness to light, ISO 105-B02 (1988).

3. Results and discussion

3.1. Identification of the modified reactive dye

The ^{1}H NMR spectrum of the modified dye **1** shows two triplets at 3.61 and 3.94 ppm, each integrated to two protons, assignable to the α- and β-methylene protons of the sulphatoethylsulphone (SES) group, respectively.

The IR spectra showed characteristic absorption band at 3473 cm^{-1}, corresponding to stretching vibration of OH. The band appeared at 1550 cm^{-1} was due to the symmetric vibrations of the azo group. Absorption bands within the m = 1166–1135 and 1083–1045 cm^{-1} were attributed to SO_2 of the coupling component.

3.2. Effect of salt concentration

Initially, the effect of salt concentration on the dyeing of cotton fabric with the modified reactive dye **1** and the commercial dye **2** was studied at 40 °C, using 2% owf. The exhaustion and the total fixation values of the dyes are given in Fig. 1. The figure clearly shows that the modified reactive dye **1** has higher exhaustion and total fixation values than dye **2**, even at low salt concentrations. This may be attributed to that the presence of the sulfadimidine group increases the molecular structure of the dye and reduces the number of sulphonic groups than those in dye **2**, which in turn reduces the repulsion force between the dye and the fabric, this in turn expected to have better substantivity to cotton under the neutral exhaust dyeing stage. It is believed that during the primary exhaustion stage, an equilibrium is established between the dye in the fibre and the dye in solution. This equilibrium is disturbed at the secondary exhaustion stage by the subsequent alkaline dyeing conditions at 20 g/l sodium carbonate and 60 °C. The dye-reactive groups could lead to further dye uptake and dye-fibre fixation under the alkaline conditions.

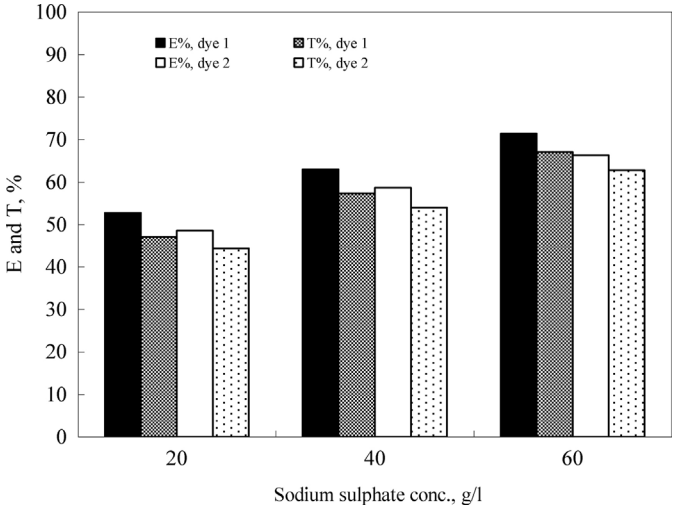


Fig. 1. Exhaustion (E%) and total fixation (T%) of modified dye **1** and Remazol Red RB dye **2** on cotton fabric at different sodium sulphate concentrations.

Also the results obtained reveal that the exhaustion of both dyes **1** and **2**, increase as the salt concentrations increase. Because, it is believed that the presence of salt allows overcoming the forces of repulsion between the dye anions and the negatively charged fibre surface. This in turn maximizes the dye substantivity to cotton fabric as the salt concentration increased.

3.3. Effect of alkali concentration on dye fixation

Dyeing behaviours of the modified reactive dye **1** and the commercial dye **2** on cotton were studied at different sodium carbonate concentrations (fixation stage) at 60 °C and at the same neutral exhaust dyeing conditions of 60 g/l sodium sulphate at 40 °C, using 2% owf. The results are shown in Fig. 2. However the results clearly show that the degree of exhaustion and fixation obtained from dye **1** are higher than those obtained from commercial dye **2**. It is clear that the optimum exhaustion and fixation on cotton was achieved at 20 g/l sodium carbonate for both dyes, because both dyes have similar reactive groups sulphatoethylsulphone/monochlorotriazine (SES/MCT) which would facilitate the dye exhaustion and fixation by virtue of the complimentary behaviour between vinylsulphone (VS) and MCT groups in both dyes after β-elimination of the SES group. This in turn maximizes the probability of dye/fibre interaction through both VS and MCT reactive groups with the fibre hydroxyl groups, resulting in high fixation yield.

3.4. Effect of dyeing time

The effect of dyeing time on the extent of exhaustion and total fixation yield of both dyes **1** and **2** was assessed. Fig. 3 shows the build-up of both dyes on cotton fabric. The results reveal that considerable exhaustion and fixation values are obtained along the range of dyeing time for both dyes even at shorter fixation time. These dyes have also the potential to react with the fibre via the reactive VS and MCT groups after β-elimination of the SES group. The results also clearly show high exhaustion and fixation levels

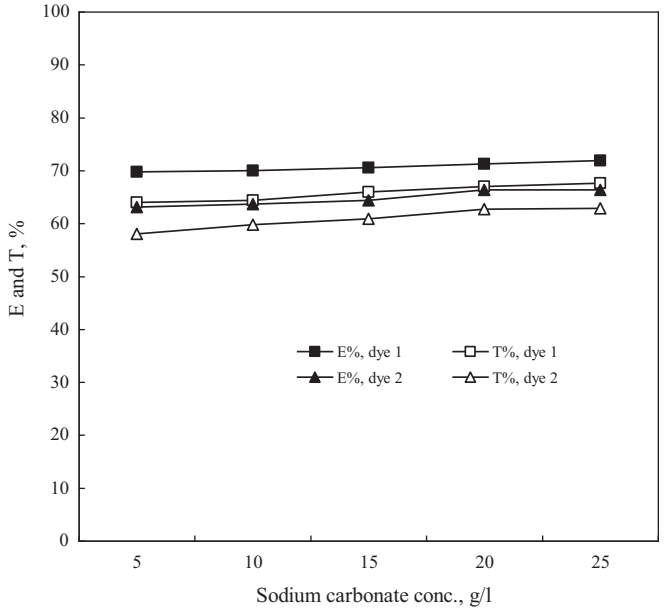


Fig. 2. Exhaustion (*E*%) and total fixation (*T*%) of modified dye **1** and Remazol Red RB dye **2** on cotton fabric at different sodium carbonate concentrations.

were achieved using dye **1** along the range of fixation time. Incorporation of sulfadimidine group in dye **1** instead of metanilic acid in dye **2** may results in increasing the molecular weight as well as reducing the number of sulphonic group of the modified dye. This in turn leads to higher substantivity of dye **1** towards the fabric under the neutral dyeing stage prior to its fixation stage.

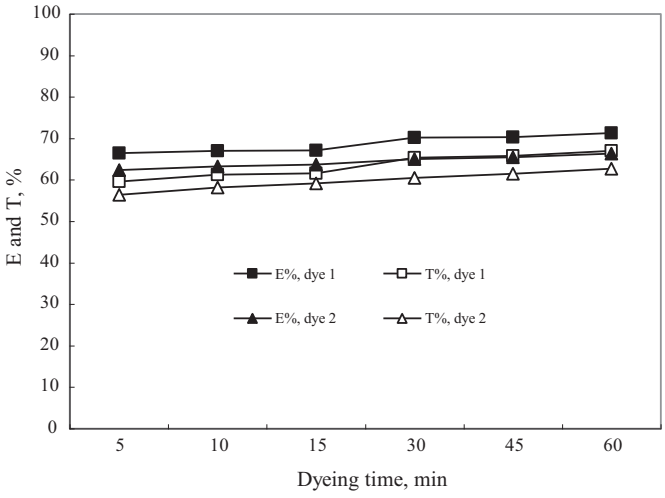


Fig. 3. Effect of dyeing time on the exhaustion and total fixation of dyes **1** and **2** (2% owf) on cotton fabric.

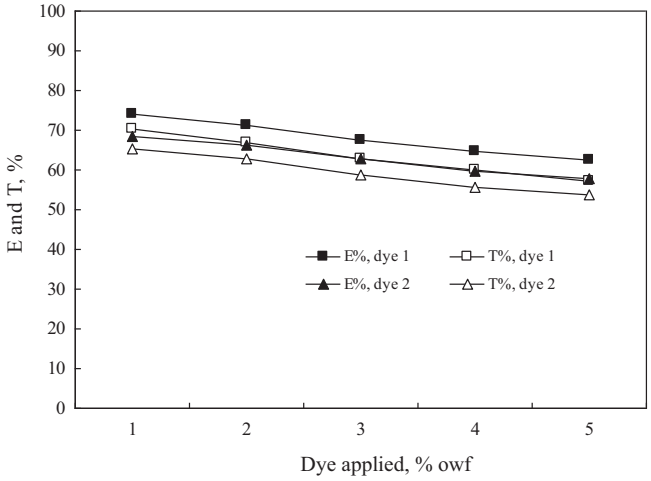


Fig. 4. Exhaustion (*E*%) and total fixation (*T*%) at different concentrations of modified dye **1** and Remazol Red RB dye **2** on cotton fabric.

3.5. Effect of dye concentration

The extent of the exhaustion and fixation of dyeing cotton with different concentrations of the reactive dyes (1–5% owf) was investigated and the results are shown in Fig. 4. From this figure, it is clear that the extent of exhaustion and fixation values secured using the modified dye **1** are higher than using the commercial dye **2**. The results also indicate that the extent of exhaustion and fixation values appear to decrease with increasing dye concentration from 1 to 5% owf for both dyes. This is believed to be due to the lowering of the dye substantivity at higher dye concentration as a result of increasing dye aggregation, which lowers dye penetration in the fibre. Additionally, at high dye concentrations the number of available dye sites on the fibre decreases and competitive hydrolysis increases, resulting in a lower extent of exhaustion and fixation yield of the dye.

3.6. Antibacterial activities of dyes

Antibacterial activities of dyes **1** and **2** on the dyed cotton fabric at different concentrations and at the optimum conditions for both dyes was studied against gram positive (*S. aureus*) and gram negative (*E. coli*) bacteria as shown in Tables 1 and 2. All dyed samples at different concentrations for both dyes gave antibacterial activity against gram positive and gram negative bacteria with different degrees, depending on the dye used. Antibacterial activity of dyed samples increases with increasing concentration and contact time till 6 h of contact. The cotton dyed with modified dye **1** shows higher antibacterial efficacy compared to those dyed with commercial dye **2**, especially on gram negative (*E. coli*) bacteria because of the presence of sulfadimidine group. Such a conclusion is also supported by the results obtained by (Balaz, Ilavsky, Sturdik, & Kovac, 1985; Singh, Gurusiddaiah, & Whalen, 1985).

Table 1
Results of antibacterial test for cotton fabric dyed with modified dye **1** and the commercial dye **2** against *E. coli* (gram negative).

Dye conc. (% owf)	Reduction %							
	Modified dye **1**				Commercial dye **2**			
	1 h	2 h	4 h	6 h	1 h	2 h	4 h	6 h
1	11.9	14.3	16.7	21.4	4.8	7.1	16.7	14.3
2	14.3	19.0	21.4	26.2	2.38	7.1	11.9	16.7
3	23.8	28.6	30.9	33.3	7.1	16.7	21.4	23.8
4	28.6	28.6	33.3	35.7	4.8	14.3	21.4	23.8
5	30.9	33.3	35.7	37.4	9.5	19.0	23.8	26.2

Table 2
Results of antibacterial test for cotton fabric dyed with modified dye **1** and the commercial dye **2** against *S. aureus* (gram positive).

Dye conc. (% owf)	Reduction %							
	Modified dye **1**				Commercial dye **2**			
	1 h	2 h	4 h	6 h	1 h	2 h	4 h	6 h
1	3.9	7.3	9.6	11.2	2.6	5.1	5.1	7.6
2	5.1	8.6	10.6	13.9	2.6	7.6	8.3	8.7
3	7.6	13.6	14.2	16.4	5.5	7.9	10.2	10.6
4	10.2	14.4	17.4	19.9	7.4	12.4	13.1	14.4
5	11.4	14.8	18.7	21.9	8.6	12.8	13.8	15.1

Table 3
Fastness properties of modified reactive dye **1** and commercial dye **2** on cotton fabric.

Dye	Dye conc. (% owf)	Fastness to rubbing		Washfastness			Fastness to perspiration						Light
							Alkaline			Acidic			
		Dry	Wet	Alt[a]	SC[b]	SW[c]	Alt	SC	SW	Alt	SC	SW	
1	2	3	3	4–5	4–5	5	4–5	4–5	5	4–5	4–5	5	6
	4	3	3	4–5	4	5	4–5	4	5	4–5	4	5	6
2	2	3	3	4–5	4	5	4–5	3–4	5	4–5	3–4	4–5	6
	4	3	3	4–5	3	5	4–5	3	4–5	4–5	3	4	6

[a] Alt, alteration.
[b] SC, staining on cotton.
[c] SW, staining on wool.

3.7. Fastness properties

The fastness properties of the dyed samples with 2 and 4% owf dye concentrations of both dyes at the same optimum dyeing conditions (60 g/l Na_2SO_4 and 40 °C and 20 g/l Na_2CO_3 at 60 °C) were investigated and given in Table 3. The results show that the fastness properties of the dyed samples of dye **1** for washing and perspiration are slightly better than those of the commercial reactive dye **2** depending on the percentage of dye fixed. The rubbing fastness appears to be the same at both shades for both dyes. Also, the light fastness was identical for both dyes. This seems reasonable as the dyes under investigation have the same chromophoric system.

4. Conclusion

A modification of a well-known commercial reactive dye (Remazol Red RB (CI Reactive Red 198)) to achieve a new antibacterial reactive dye was carried out by replacing metanilic acid in the commercial dye with sulfadimidine group. Simultaneous dyeing and antibacterial finishing of cotton fabric was investigated by the exhaust method. The dyes were applied at different dyeing conditions. Such modified dye **1** exhibited higher substantivity, exhaustion and fixation efficiency compared to the commercial dye **2**. By virtue of both dye structures, the presence of the sulfadimidine group instead of metanilic acid reduces the content of sulphonic acid groups as well as enlarges the structure of the target dye **1**, promoting a higher substantivity towards cotton under the neutral dyeing stage. The presence of SES/MCT reactive system would also maximize the dye exhaustion and fixation values under the alkaline dyeing conditions by virtue of the complimentary behaviour between VS and MCT groups for both dyes after β-elimination of the SES group. All the dyed samples exposed to antibacterial test exhibited antibacterial efficacy against gram-positive (*S. aureus*) and gram-negative (*E. coli*) bacteria, but the dye **1** showed higher activities than the Commercial dye **2**. All the reactive dyeings for both dyes exhibited high fastness properties.

Owing to the reduction in the number of sulphonic groups in dye **1** leads to better dye substantivity under neutral dyeing conditions coupled with higher exhaustion and fixation efficiency of being based on a bifunctional VS/MCT reactive system. Even more important, incorporation of the sulfadimidine group to the dye structure, such model of reactive dyes showed higher antibacterial efficacy compared to the conventional dye **2**. The satisfactory dyeing performance and good antibacterial properties on cotton fabric should lead to design of novel antibacterial reactive dyes with improved application properties.

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