

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



R. Farouk*, H.E. Gaffer

Textile Research Division, National Research Centre, 12622 Dokki, Cairo, Egypt

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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.

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1. Introduction

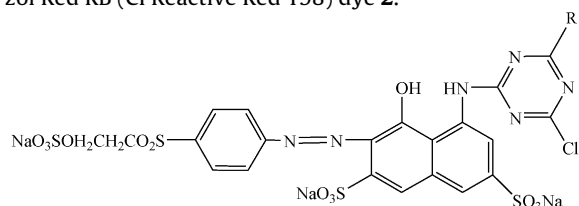
Cotton textiles in contact with the human body offer an ideal environment for microbial growth, antibacterial finishes are now highly desirable for textiles, especially textile materials used in hospitals and hotels are liable to control cross infection and disease transmission (Gupta, Khare, & Laha, 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(monochlorotriazine) reactive dyes has been investigated, and reported that the presence of covalently 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).

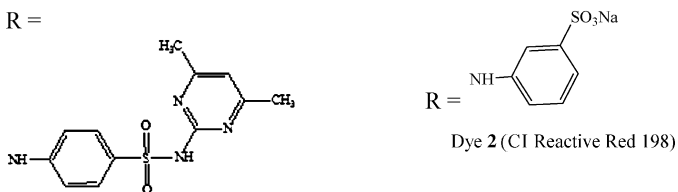
In the present work we choose sulfadimidine group to be incorporated into a commercial reactive dye structure Remazol Red RB (CI Reactive Red 198) by replacing metanilic acid with Sulfadimidine group resulting a modified reactive dye **1**.

* Corresponding author. Tel.: +20 1061030532; fax: +20 233370931.
E-mail address: rfabdelhady@hotmail.com (R. Farouk).

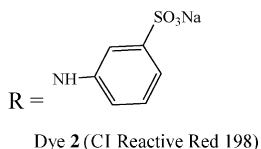
Studying the dyeing behaviour and the antibacterial activity on cotton fabric for the modified reactive dye **1** compared with Remazol Red RB (CI Reactive Red 198) dye **2**.



R =



Modified Reactive Dye **1**



2. Experimental

2.1. Materials

Mill-scoured and bleached cotton fabric, 130 g/m² 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 (Hostopal 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} (H₂O) = 515.4 nm, its purity was determined by elemental analysis, and gave the following results:

C₃₃H₂₆N₁₀O₁₅S₅Na₃Cl (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): ν/cm⁻¹ = 3473 (OH), 1550 (–N=N–), 1166–1135 and 1084–1045 (–SO₂–). ¹H NMR: δH (ppm) in [₂H⁶]DMSO: 2.2, 2.3 (s, 6H, 2CH₃), 3.59–3.61 (2H, t, J = 6.9 Hz, α-CH₂(_{SES})), 3.92–3.95 (2H, t, J = 6.9 Hz, β-CH₂(_{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, SO₂NHAr–H), 8.01–8.03 (dd, 4H, N=N–Ar–H), 14.11 (s, 2H, SO₂NH₂).

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 ¹H 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 C₁, C₂ 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 C₃ 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

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