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ORIGINAL ARTICLE

Colorimetric studies of some newly synthesized bisazo reactive dyes

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KEYWORDS

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Abstract A series of cold brand bisazo reactive dyes (**4a**–**h**) were obtained by the coupling of tetrazotised 4,4′-methylene-bis(2-methyl-5-nitro aniline) (**2**) with various cyanurated coupling components (**3a**–**h**) in good yield. Their dyeing performances as reactive dyes have been assessed on silk, wool and cotton fabrics. These dyes were characterized by UV–Vis, FTIR, ^{1}H NMR spectroscopic techniques elemental analysis. The percentage dye bath exhaustion and fixation on different fibers were found to be very good. The dyed fabric showed moderate to very good light fastness and good to excellent washing and rubbing fastness properties. Spectral properties and colorimetric data (L^*, a^*, b^*, C^*, H^*, K/S) have also been studied in detail.

1. Introduction

For the last 50 years many fibre reactive dyes have been developed which form covalent bond between dye and fibre particularly cellulosic fibre (Renfrew, 1999). Reactive dyes containing the triazine ring have an electrophilic site which undergoes nucleophilic substitution reaction by react with hydroxyl group of cellulose unit. Reactive dyes are mainly applied to cotton fibre for achieving high wash fastness properties. The high wash fastness is obtained by the covalent bond formation between dye and fibre in the fixation step (Waring, 1990).

The advantage of bisazo reactive dyes over monoazo reactive dyes is that they possess two reactive groups which give higher fixation yield then monoazo dyes which possess one reactive group because if one of the two dye fibre bonds is hydrolyzed, another is still left for fixation (Bredereck and Schumacher, 1993; Matsi et al., 1988). Additionally, most of the reactive dyes used in exhaust dyeing employ at least two reactive groups in order to maximize fixation (Taylor, 2000).

In continuation of our previous studies (Patel et al., 2009, 2010a,b,c) on the synthesis of a variety of bisazo reactive dyes, we now report on the successful synthesis of several new cold brand bisazo reactive dyes based on 4,4′-methylene-bis(2-methyl-5-nitro aniline) and their application as reactive dyes for dyeing silk, wool and cotton fabrics. The fastness properties, spectral properties and colorimetric data (L^*, a^*, b^*, C^*,

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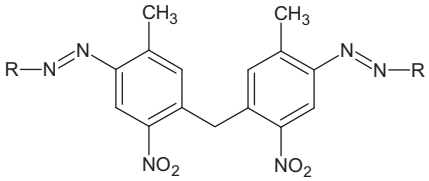
H^*, K/S) were also discussed. The general structure of these dyes (**4a**–**h**) is shown in Fig. 1.

2. Experimental

2.1. General

All the chemicals used in the dye synthesis were of commercial grade and were further purified by crystallization and distillation. Melting points were determined by the open capillary method. The purity of all the dyes was checked by TLC (Fried and Sharma, 1982). The visible absorption spectra were measured using a Shimadzu UV-1700 spectrophotometer. A Perkin-Elmer model 881 recording infrared spectrophotometer was used for recording the FTIR spectra of the dyes as KBr pellets in the range between 4000 and 400 cm^{-1} and ^1H NMR spectra on a Bruker Avance II 400 instrument using TMS as internal standard and DMSO as solvent. Elemental analysis of C, H and N were carried on Carlo Erba 1108 instrument. The light fastness was assessed in accordance with BS: 1006-1978 (Standard Test Method, 1978). The rubbing fastness test was carried out with a Crockmeter (Atlas) in accordance with AATCC-1961 (AATCC Test Method, 1961) and the wash fastness test in accordance with IS: 765-1979 (Indian Standard, 1979). Colorimetric data (L^*, a^*, b^*, C^*, H^*, K/S) were recorded on Reflectance Spectrophotometer Gretag Macbeth CE: 7000.



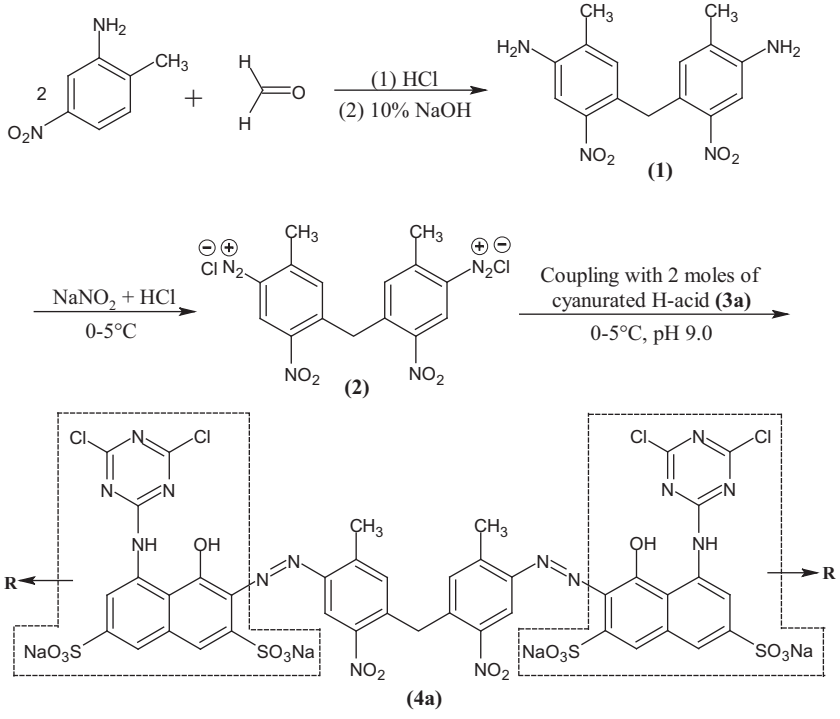
(4a-h)

Where R = various cyanurated coupling components (**3a-h**) (Table-1)

Figure 1 General structure of reactive dyes (**4a**–**h**).

2.2. Synthesis of 4,4′-methylene-bis(2-methyl-5-nitroaniline) (*1*) (Patel and Patel, 2011)

2-Methyl-5-nitroaniline (15.2 g, 0.1 mol) was first mixed with sufficient HCl to reach pH 4 of the medium. Then, the pale yellow, colored solution was put into a three-necked flask fitted with a stirrer and was mixed at 50 °C temperature. Formaldehyde (35 ml, 3%, v/v) was added at interval of 10 min over a period of 1 h and the stirring was continued for a further 10 h maintaining temperature at 60 °C. Then, the stirring was stopped and the product was washed with NaOH (2%, w/v) first and then with water till the mixture became neutral. Finally, the yellowish colored product (**1**) was dried in an oven at 90 °C for about 4 h (13.61 g, 86.04%). Yellow powder, m.p. 202 °C (recrystallized from acetic acid). R_f = 0.75 (PhMe:EtOAc, 3:1, v/v). IR (KBr): ν_{max} (cm^{-1}): 3455, 3363 (N–H), 2925, 2845 (C–H), 1568, 1356 (N═O). ^1H NMR (400 MHz, DMSO-d_6) δ ppm: 2.58 (2H, s, CH_2), 2.75 (6H, s, N–CH_3), 6.67 (4H, s, NH_2), 6.95–7.25 (4H, m, Ar–H). Anal. Calcd. for $C_{15}H_{16}O_4N_4$ (316.31 g/mol): C, 56.96; H, 5.10; N, 17.71%. Found: C, 56.78; H, 4.93; N, 17.60% (Scheme 1).



Where R = Various cyanurated coupling components (**3a-h**)

Scheme 1 Synthesis of reactive dye **4a** by using cyanurated H-acid (**3a**) as coupling component.