



Synthesis, fastness properties, color assessment and antimicrobial activity of some azo reactive dyes having pyrazole moiety



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ABSTRACT

Some new azo dye derivatives having pyrazole and trichlorotriazine moieties were synthesized and were evaluated as reactive dyes for the exhaust dyeing of cotton fiber. The title dyes **5a–c** were synthesized from reactions of diazonium salts of 3-substituted-1-phenyl-5-aminopyrazoles **2a–c** with 6-amino-7-hydroxy-2-naphthalenesulfonic acid, 4-amino-5-hydroxynaphthalene-2,7-disulfonic acid and/or cyanuric chloride. Other reactive dyes **7a–f** were synthesized from reaction of cyanuric chloride and 4-aryloxy-3-substituted-1-phenyl-5-aminopyrazoles **6a–f**. The synthesized dyes were applied to cotton fiber and their color fastness to light, washing, perspiration, and rubbing were also measured. The dyes showed good to moderate affinity to cotton fiber. The biological screening showed that most of the tested compounds display promising antimicrobial activities against the tested microorganisms.

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1. Introduction

Reactive dyes are extensively used in the textile industry not only for cotton, but also wool and polyamides because of their wide variety of color shades, high wet fastness, ease of application and brilliant colors. A steady increase in the reactive dye usage has been observed as a result of the increased cotton use worldwide [1]. Reactive dyes for cellulose resemble acid dyes in their basic structure, but additionally possess one or more reactive groups. Their names were derived from their abilities to interact chemically with the fiber functional groups. Dyes containing a dichlorotriazinyl reactive group are capable of reacting with cellulosic fiber, in the presence of alkali, to form covalent bond between the dye and the fiber [2]. 5Aminopyrazoles are very important class of heterocycles due to their biological and pharmacological activities [3,4]. These compounds often exhibit anti-inflammatory, herbicidal, fungicidal, bactericidal and antipyretic activities [3,5–10]. As a part of our continuous interest in the reactivity of 5-aminopyrazole derivatives we have synthesized some novel reactive dyes having pyrazole moiety and investigated their applications as reactive dyes to cotton fiber [11–14]. Here we report the synthesis of some new azo dyes and their dyeing and biological properties.

2. Experimental

2.1. Materials

Commercially available cotton fibers were used for dyeing. The chemicals and reagents were obtained from Sigma–Aldrich and used without further purification. The solvents were of spectroscopic grade.

2.2. Characterization

Melting points were recorded on a Gallenkamp melting point apparatus and are reported uncorrected. The infrared spectra (IR) were recorded on Perkin–Elmer FTIR 1430 spectrophotometer using KBr disk technique. The ¹H NMR spectra were recorded on a Bruker AC spectrometer (300 MHz) at 25 °C in DMSO-d₆ with TMS as an internal standard. Chemical shifts were reported in ppm and the results are expressed as δ values. Mass spectra were measured on a Finnigan MAT 8222 EX mass spectrometer at 70 eV. Electron spray ionization (ESI) mass spectra were recorded on a Shimadzu LCMS-2010 eV spectrometer or Bruker Daltonics micro TOF-15 focus. Ultraviolet–visible (UV–vis) absorption spectra were recorded on an SHIMADZO UV-3101PC spectrophotometer using 1.0 cm matched silica cell. Microanalyses were performed on Perkin–Elmer 2400 Elemental Analyzer at Microanalytical Center at Cairo University. Reaction progress was monitored by the use of thin layer

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chromatography (TLC) using benzene/acetone (2/1 by volume) as eluent.

2.3. Visible absorption spectra of dyes

The dyes **3a–c**, **5a–c** and **8a–f** in DMF were prepared (0.001 M) and the absorbance of each solution was measured at the λ_{\max} , using 1 cm cells and a SHIMADZO UV-3101PC spectrophotometer. The molar extinction coefficients (ϵ max) of the dyes were determined and are shown in Table 1.

2.4. Dyeing and fastness determinations

2.4.1. Dyeing procedure

The applications and fastness properties of the synthesized dyes have been performed at Misr Spinning and Weaving Company, Central Q.C. Laboratories, Mehalla El-Kubra, Egypt. The synthesized azo dyes were applied to cotton fiber. The dyes were applied to bleached unmercerized five depths, viz. 1, 2, 4, 6 and 9% dye owing the mass of woven cotton at fiber at 60 °C and a liquor ratio of 10:1, using the quantities of Glauber's salt (exhaustion for 30 min) and soda ash (fixation for 60 min). After dyeing, the fabric was rinsed well with cold and hot water before soaping at a boiling temperature for 10 min. Finally the fiber was rinsed with cold water and air dried at room temperature [15].

2.4.2. Fastness determination

Color fastness to light, washing, perspiration, and rubbing of the prepared dyes on the cotton fabric were studied using the standard methods for the assessment color fastness of textile [16]. The obtained results were collected in Table 2.


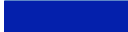










2.4.2.1. Fastness to washing. The test assessed using the laundero-metersponsored by the American Association of Textile Chemists and Colorists (A.A.T.C.C.). A test specimen (10 cm × 4 cm) of the dyed fiber was taken and samples (5 cm × 4 cm) of the white cotton and polyester fibers were placed in the container of the washing machine, with the necessary amount of soap solution (5 g/l) previously heated at 50 °C. The specimen was rinsed twice in cold water for 10 min, squeezed, and the composite specimen was opened out and dried in air. The color alteration of the uncovered portion of the specimen and the staining for both undyed fabrics was assessed using the international Grey scale.

2.4.2.2. Fastness to perspiration. The test specimen (6 cm × 6 cm) was placed between 2 species of undyed fabrics (cotton and polyester) and sewed along one side to form the test specimen. Testing fabrics were immersed into the solution of at room temperature for

Table 1
Absorption spectra of dyes **3a–c**, **5a–c** and **7a–f**.

Dye no	Absorption λ_{\max} (nm)	Log ϵ
3a	356	0.04
3b	358	3.5
3c	359	3.3
5a	611	4.5
5b	514	4.3
5c	593	4.9
7a	378	4.6
7b	380	4.3
7c	376	4.5
7d	382	4.2
7e	375	4.0
7f	381	3.7

Table 2
Fastness properties of dyes **3a–c**, **5a–c** and **7a–f** on Cotton fiber.^a

Dye no	Color	Washing		Perspiration		Rubbing		Light
		PES	Cotton	PES	Cotton	Wet	Dry	
3a		3–4	4	3–4	3–4	3–4	4–5	5–6
3b		3–4	4	4	3–4	4	3	5–6
3c		4	3	3–4	3–4	4–5	4–5	5–6
5a		4	3–4	3–4	4	4–5	4–5	5–6
5b		4	4	3–4	4–5	4–5	4–5	5–6
5c		4	3–4	4	4	4–5	4–5	6
7a		3–4	4	3–4	3–4	4	4	6
7b		3–4	4	3–4	3–4	3–4	4–5	6
7c		4	3	4	3–4	4–5	4–5	6
7d		4	4	4	4	4–5	4–5	5–6
7e		4	4	4	4	4	4–5	5–6
7f		4	4	4	4	4–5	4–5	6

^a Rate for light fastness: 4–8 (acceptable), 1–3 (not acceptable); rate for different fastness: 3–5 (acceptable), 1–2 (not acceptable).

30 min at a pH of 4. The solution was poured off and the sample was placed between two plastic plates (7.5 cm × 6.5 cm) under a force of about 4.5 kg. The plates containing the composite samples are kept in an oven at 30 °C for 4 h. The specimen was then separated from the undyed samples. Colors alteration of dyed material and staining of the undyed samples were assessed using the international Grey scale.

2.4.2.3. Fastness to rubbing. Test assessment was made according to the Grey scale using Crockmeter of atlas electronic type. Dyed fabrics to be tested were placed on the base of the Crockmeter. A square of white testing cloth was mounting over the end of the finger which protects downward on the specimen sliding back, and force to make ten complete turns of the crank at the rate of one turn per a second. For wet rubbing test, the testing squares were thoroughly wet in distilled water and squeezed between filter papers through hand wringer under standard conditions.

2.4.2.4. Fastness to light. The specimen of the dyed textiles were exposed, in a well ventilated exposure chamber to light from a xenon arc, along with dyed wool standards. The air temperature in the chamber was maintained at 30 °C. The effective humidity was maintained at 45 ± 5%. The variation of light intensity over the area covered by specimen and standards should not exceed 20%. The samples and standards were exposed simultaneously under the same conditions for the same time. The samples were viewed in the light from a day-light fluorescent lamp and given a degree in comparison with the relative to Blue scale (1–8) standards of A.A.T.C.C.

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