



Disperse Dyes Based on Pyrazolopyrimidinones Part 2: Synthesis and Dyeing Performance of Novel Biologically active Arylazopyrazolopyrimidinones dyes

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Abstract

Disperse dyes are considered important dyes in the dye industry and their use has increased significantly in recent years. Therefore, our goal is to prepare a new series of these dyes and use them in dyeing polyester fabrics, and also to demonstrate the added value of these dyes, as they have activity against many microbes. Making a number of disperse dyes based on pyrazolopyrimidinones is the aim of this investigation. Arylhydrazonopyrazolones **2a-f** [3-amino-4-(2-(4-substitutedphenyl)hydrazono)-1H-pyrazol-5(4H)-one] and enaminone **1** [(E)-1-(4-bromophenyl)-3-(dimethylamino)prop-2-en-1-one] reacted in an acidic environment to create these dyes. FTIR, mass, UV, NMR, and elemental studies were used to determine their structures. The fastness characteristics of these disperse dyes were evaluated after they were applied to polyester fabrics. The potential, biological activities of these novel disperse dyes against different cultures of bacteria were also carried out.

Keywords: Disperse dyes, Polyesters, Dyeing, Fastness properties.

Introduction

The clothing sector, especially textile sportswear, has been a major driver of the disperse dye market's notable expansion over the last five years [1-25]. In order to satisfy consumer demand for eco-friendly products, one significant trend is the shift towards more sustainable dyeing procedures, such as low-temperature dyeing.

A significant class of synthetic dyes based on the pyrazolo[1,5-a]pyrimidine ring system are disperse pyrazolopyrimidinone dyes [1-11].

It is well-known that nitrogen-containing heterocyclic compounds called pyrazolopyrimidinones work as a basic scaffold in a variety

of physiologically active substances [15, 17, 25]. In this work, we synthesized a new series of disperse dyes based on pyrazolopyrimidinones and used them to dye polyester fabrics. We also aimed to show the dyes' additional value by examining their anticipated biological activity on several bacterial species.

Experimental

General

All melting points are uncorrected and were measured using an electrothermal digital melting point device. The Pye Unicam SP3-100 spectrophotometer at Assiut University and the Shimadzu IR-470 infrared spectrophotometer were used to record the infrared spectra using the KBr pellet technique. Tetramethylsilane (TMS) was used

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as an internal reference while measuring ¹H-NMR spectra in deuterated dimethylsulphoxide (DMSO-d₆) on a Varian 300 MHz.

The chemical shifts are reported in parts per million (ppm) at Cairo University. Cairo University's HP model MS-5988 was used to perform mass spectra. At Cairo University, microanalyses for C, H, N, and halogen were carried out using a Vario El Elementar analyser.

Synthesis of azo Disperse Dyes **4a-e**

Seven millilitres of glacial acetic acid were used to dissolve a combination of arylhydrazonopyrazolones (0.01 M) and enaminones (0.01 M), which were then refluxed for four hours. To create dyes **4a-e**, the resulting solid was gathered and crystallised from DMF/water [1].

7-(4-bromophenyl)-3-(phenyldiazenyl)pyrazolo[1,5-a]pyrimidin-2(1H)-one (**4a**)

M.p. 125 °C, the IR spectra of dye **4a** shows absorption bands at $\nu = 3441\text{ cm}^{-1}$ (NH), 1629 cm^{-1} (C=O of amid group). ¹H-NMR (DMSO-d₆) 7.34 (triplet for 1H, $J = 7.2\text{ MHz}$, arom-H), 7.49-7.54 (multiplet for 4H, arom-H), 7.75 (doublet for 2H, $J = 8.1\text{ MHz}$, arom-H), 7.83 (doublet 2H, $J = 8.7\text{ MHz}$, arom-H), 8.04 (doublet 2H, $J = 8.4\text{ MHz}$, arom-H), 8.73 (doublet 1H, $J = 4.8\text{ MHz}$, NH).

(7-(4-bromophenyl)-3-(p-tolyldiazenyl)pyrazolo[1,5-a]pyrimidin-2(1H)-one (**4b**).

M.p. 105 °C the IR spectra of **4b** shows absorption bands at $\nu = 3446\text{ cm}^{-1}$ (NH), 1629 cm^{-1} (C=O of amid group). ¹H-NMR (DMSO-d₆) 2.36 (singlet for 3H, CH₃), 7.31 (doublet for 2H, $J = 6.9\text{ MHz}$, arom-H), 7.47 (doublet for 2H, $J = 7.8\text{ MHz}$, arom-H), 7.67 (doublet for 2H, $J = 6.9\text{ MHz}$, arom-H), 7.82 (doublet for 2H, $J = 8.7\text{ MHz}$, arom-H), 8.03 (doublet for 2H, $J = 8.4\text{ MHz}$, arom-H), 8.69 (doublet for 1H, $J = 4.8\text{ MHz}$, NH).

7-(4-bromophenyl)-3-((4-methoxyphenyl)diazenyl)pyrazolo[1,5-a]pyrimidin-2(1H)-one (**4c**).

M.p. 112 °C the IR spectra of **4c** shows absorption bands at $\nu = 3424\text{ cm}^{-1}$ (NH), 1632 cm^{-1} (C=O of amid group). ¹H-NMR (DMSO-d₆) 3.84 (singlet for 3H, OCH₃), 7.07 (doublet for 2H, $J = 9\text{ MHz}$, arom-H), 7.42 (doublet for 2H, $J = 4.5\text{ MHz}$, arom-H), 7.79-7.86 (multiplet for 4H, arom-H), 8.03 (doublet for 2H, $J = 8.7\text{ MHz}$, arom-H), 8.71 (doublet for 1H, $J = 4.8\text{ MHz}$, NH).

(7-(4-bromophenyl)-3-((4-chlorophenyl)diazenyl)pyrazolo[1,5-a]pyrimidin-2(1H)-one (**4d**).

M.p. 120 °C the IR spectra of **4d** shows absorption bands at $\nu = 3443\text{ cm}^{-1}$ (NH), 1633 cm^{-1} (C=O of amid group). ¹H-NMR (DMSO-d₆) 7.49 (doublet for 2H, $J = 4.5\text{ MHz}$, arom-H), 7.54 (doublet for 2H, $J = 8.4\text{ MHz}$, arom-H), 7.79-7.85 (multiplet for 4H,

arom-H), 8.02 (doublet for 2H, $J = 8.7\text{ MHz}$, arom-H), 8.73 (doublet for 1H, $J = 4.5\text{ MHz}$, NH).

7-(4-bromophenyl)-3-((4-bromophenyl)diazenyl)pyrazolo[1,5-a]pyrimidin-2(1H)-one (**4e**).

M.p. 140 °C, the IR spectra of **4e** shows absorption bands at $\nu = 3446\text{ cm}^{-1}$ (NH), 1674 cm^{-1} (C=O of amid group). ¹H-NMR (DMSO-d₆) 7.50 (doublet for 2H, $J = 4.8\text{ MHz}$, arom-H), 7.67-7.75 (multiplet for 4H, arom-H), 7.83 (doublet for 2H, $J = 8.7\text{ MHz}$, arom-H), 8.02 (doublet for 2H, $J = 8.7\text{ MHz}$, arom-H), 8.73 (doublet for 1H, $J = 4.8\text{ MHz}$, NH).

7-(4-bromophenyl)-3-((4-nitrophenyl) diazenyl)pyrazolo [1,5-a]pyrimidin-2(1H)-one (**4f**)

M.p. 180 °C, the IR spectra of **4f** shows absorption bands at $\nu = 3438\text{ cm}^{-1}$ (NH), 1674 cm^{-1} (C=O of amid group). ¹H-NMR (DMSO-d₆) 7.65 (doublet for 2H, $J = 4.8\text{ MHz}$, arom-H), 7.84-7.95 (multiplet 4H, arom-H), 8.03 (doublet for 2H, $J = 8.4\text{ MHz}$, arom-H), 8.34 (doublet for 2H, $J = 8.4\text{ MHz}$, arom-H), 8.78 (doublet for 1H, $J = 4.8\text{ MHz}$, NH).

Dyeing method

All dyes were used as pure powder in the same form as when they were manufactured without milling. At 130 °C with a 1:50 liquor ratio for one hour, two gm. of fabric samples were added to a flask containing a dyebath of 2% (o.w.f.) dye shade and Matexil DA-N (supplied by ICI Company, UK) as a dispersion agent. The dye was mixed with ten drops of DMF, the dispersing agent, and water to produce uniform dye dispersion. The pH was lowered to 4.5 using acetic acid and. After dyeing, the samples were removed, rinsed with warm water, and then placed in a solution containing 2 g/L sodium hydrosulphite and 2 g/L sodium hydroxide (caustic soda) for 10 minutes at 60 °C with a liquor ratio of 1:40. After the sample had been cleansed by reduction, it was thoroughly rinsed with water. The dyed samples were removed, cleaned with tap water, and allowed to air dry.

Antimicrobial Activities Test

The agar diffusion technique was used to test the antibacterial qualities of all prepared disperse dyes against Gramme positive bacteria, such as *Bacillus cereus*, *Micrococcus luteus*, and *Staphylococcus aureus*, and Gramme negative bacteria, such as *Escherichia coli*, *Pseudomonas aeruginosa*, and *Serratia marcescens*. A solution of dimethylformamide (DMF) containing 1 mg/ml was used. The bacteria are kept alive by nutrient agar. In DMF, no inhibitory zones were discernible. A number of microorganism cultures were injected into the agar media after a 24-hour inoculation period for bacteria at 37 °C. The diameter of the inhibitory zone (mm) was measured.

Fabrics

El-Mahalla El-Kobra Company, Egypt, provided the 100% polyester fabric that had been bleached and

scoured. To remove waxes and impurities, the fabrics were scoured in an aqueous solution with a liquor ratio of 1:50, 2 g/L of nonionic detergent solution (Hostapal, Clariant, Swiss), and 2 g/L of Na₂CO₃ at 50 °C for 30 minutes. They were then completely washed in cold tap water and allowed to dry at room temperature.

Color Measurements

A reflectance spectrophotometer was used to measure the colorimetric properties of the polyester fabrics that had been dyed. Using the light reflectance technique on an UltraScan PRO D65 UV/VIS Spectrophotometer, the colour yields of the dyed samples were ascertained. The colour strengths were calculated using the Kubelka-Mink equation and shown as K/S values [24].

$$K/S = (1 - R)^2 / 2R$$

Where, R is the reflectance of colored samples and K and S are the absorption and scattering coefficients, respectively.

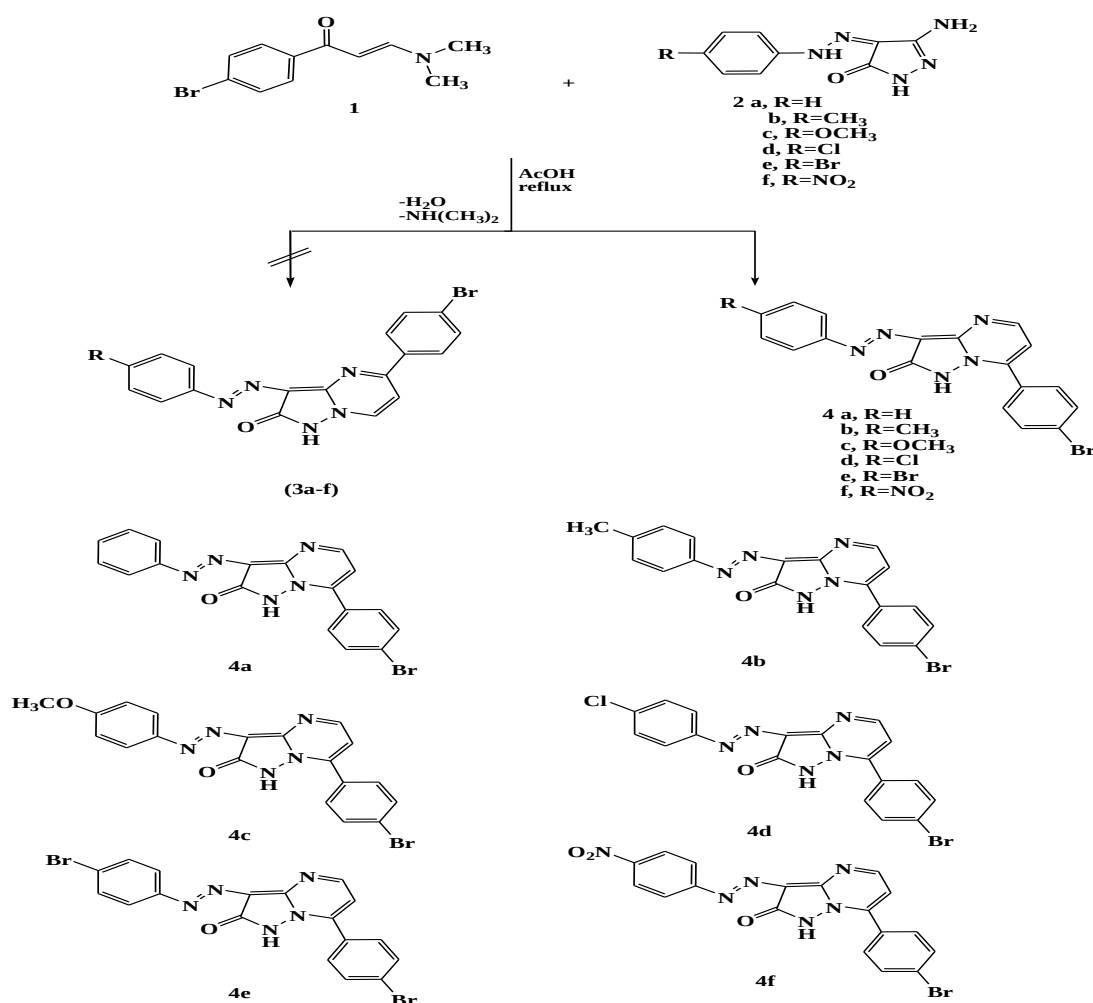
Color Fastness tests

The ISO 105-C02:1989 technique was used to determine colour fastness to washing. Using the ISO 105-X12:1987 test technique, colour fastness against

rubbing was assessed. The ISO 105-E04:1989 test technique was used to prepare the colour fastness to sweat. Using the ISO 105-B02:1988 test technique, colour fastness to light was evaluated [19-22].

Results and Discussion

Scheme 1 shows the method we developed for making the novel azo disperse dyes based on pyrazolopyrimidinone. The novel disperse dyes were synthesized when enaminone **1** [(E)-1-(4-bromophenyl)-3-(dimethylamino)prop-2-en-1-one] reacts with arylhydrazonopyrazolones **2a-f** [3-amino-4-(2-(4-substitutedphenyl)hydrazono)-1H-pyrazol-5(4H)-one]. The identities of the pyrimidinone-dispersed dyes **4a-f** were determined using elemental analysis, mass spectral data, ¹H-NMR spectroscopy, as well as the infrared and ultraviolet spectrums. Al-Etaibi *et al.*'s X-ray crystallographic structure does rule out the possibility of regioisomers **3a-f** forming in interactions of enaminone **1** with compounds **2a-f** [1, 6, 8].



Scheme 1: Synthesis of novel disperse dyes.

3.1 Dyeing Performance

Polyester fabrics could be dyed using disperse dyes **4a–f** as a substrate (2% shade). The dyeing substrate has a varied K/S and overall colour difference ΔE , according to the data obtained and shown in Table 1. The presence or location of the replacement in the dye molecules determines the difference in K/S and

ΔE . The K/S of dye **4b** (15.39) and **4c** (21.04) are much greater than that of dyes **4d**, **4e** and **4f** (14.22, 13.22 and 8.46), according to the results shown in Table 1. Additionally, dye **4c**'s total colour difference (ΔE) of 81.22 is significantly greater than dyes **4d**, **4e** and **4f**'s (76.57, 76.20 and 70.12, respectively).

TABLE 1. Optical measurements of prepared dyes.

Dye No	ΔE	<i>L</i>	<i>a</i>	<i>b</i>	$\lambda_{\max}$	<i>C</i>	<i>h</i>	K/S
4a	85.14	63.53	8.90	74.03	440	76.02	83.15	22.80
4b	77.45	61.85	9.11	61.85	440	67.31	82.14	15.39
4c	81.22	56.35	25.41	65.05	440	69.86	68.66	21.04
4d	76.57	64.82	4.73	66.55	435	67.12	85.94	14.22
4e	76.20	65.73	3.07	66.57	440	66.98	87.36	13.22
4f	70.12	66.47	6.68	60.19	445	61.01	83.66	8.46

3.2 Fastness Properties

This is consistent with the data that was gathered, which indicates that the dyes **4d**, **4e** and **4f**'s fastness to light is improved to very good (6, 5, 5-6) by the presence of a Cl, Br and NO₂ substituent. As an alternative, dyes **4b** and **4c** to (4-5 and 4-5) exhibit a gradual increase in fading, which could be explained by the presence of CH₃ and OCH₃ groups. We can say that the groups that donate electrons, such as CH₃ and OCH₃, are less stable against light than those

groups that withdraw electrons, such as Cl, Br and NO₂, according to the data obtained and shown in Table 2. Good intra-fiber diffusion of the dye molecules within the polyester dyed fabrics, as well as the assumed relatively large dye molecule particle size, may be the cause of the very good fastness for both rubbing & washing and excellent perspiration fastness of the polyester dyed fabrics, according to the data obtained and shown in Table 2.

TABLE 2. Fastness properties of the prepared dyes

Dye Number	Light fastness	Rubbing fastness		Wash fastness			Perspiration fastness					
		Dry	Wet	Alt	SC	SW	Acidic			Alkaline		
							Alt	SC	SW	Alt	SC	SW
4a	5	5	5	5	5	5	5	5	5	5	5	5
4b	4-5	4	4	4-5	4	4	5	5	5	5	5	5
4c	4-5	5	5	4-5	4-5	4-5	5	5	5	5	5	5
4d	6	4-5	4-5	4-5	4	4	5	5	5	5	5	5
4e	5	4	4-5	4-5	4	4	5	5	5	5	5	5
4f	5-6	4	4	4-5	4	4	5	5	5	5	5	5

Where: Alt = Alteration, SC = Staining on cotton, SW = Staining on wool.

3.3 Antimicrobial Activity

The data showed that all dyes **4a–f** had good effectiveness against *Escherichia coli*, but none of them had any effect on *Serratia marcescens*. Dyes **4b**, **4c**, **4e**, and **4f** showed positive antibacterial activity against three pathogens, while disperse dyes

4a and **4d** showed positive antimicrobial activity against four microbes. Consequently, the dyes that were created demonstrated promising outcomes regarding their potential for application in medical fields.

TABLE 3. Antimicrobial results of prepared dyes

Dye No	G ⁺ Bacteria (inhibition zone in mm)			G ⁻ Bacteria (inhibition zone in mm)		
	<i>Staphylococcus aureus</i> (cont.34)	<i>Micrococcus luteus</i> (cont.24)	<i>Bacillus cereus</i> cont.18	<i>Serratia marcescens</i> (cont.28)	<i>Escherichia coli</i> (cont.26)	<i>Pseudomonas aeruginosa</i> (cont.42)
4a	8	7	0	0	8	7
4b	0	9	0	0	8	7
4c	0	8	8	0	7	0
4d	8	7	8	0	7	0
4e	0	7	8	0	7	0
4f	8	0	0	0	8	7

Conclusions

We prepared a number of new dyes as part of our plan to develop new disperse dyes. These dyes were employed to dye polyester garments, and the results showed great sweat resistance as well as extremely good fastness against rubbing, light, and washing. The antibacterial properties of these novel dyes against both Gram-positive and Gram-negative bacteria were examined in an effort to highlight their added value, and encouraging findings were found for usage these dyes in medical domains.

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Conflicts of interest

There is no conflict of interest in the publication of this article.

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