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Synthesis of Some Azo Disperse Dyes Based on Pyridone Moiety and Their Application on Polyester Fabrics

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THE AIM of this work was to prepare a series of some azo disperse dyes based on pyridones moiety. The prepared dyes were used for dyeing polyester fabric. The chemical structure and dyeing performance of these dyes were investigated.

Introduction

The wide spreading of Disperse dyes has a good impact in the field of textile industry while the discovery of synthetic fabrics especially polyester fabric. Disperse dyes are a kind of dyes which have impossibility to solve in water and these make their affinity for hydrophobic fabric such as cellulose acetate, nylon, polyester [1]. The particles of disperse dyes appear in suspension states in the water bath in the presence of dispersing agent [2]. Pyridones derivatives become a major area of research and an important branch in preparing dyes. Disperse dyes which are based on pyridone moiety as a coupling component have a great impact in dyestuff industry [3] because of their excellent fastness properties like perspiration, good light fastness and wash fastness, also their physico-chemical properties make them play an important role in manufacturing photoactive materials. Pyridone derivatives are used as antibacterial agent which has a great effect in pharmaceutical sciences [4]. Different anilines disperse dyes were prepared and used in dyeing polyester fibers with different shades starting from, yellow to greenish yellow [5]. In conjunction to our previous effort for synthesis new disperse dyes [6], we report herein the synthesis of some new disperse dyes based on pyridone moiety and study their dyeing performance for dyeing polyester fabrics with different dyeing methods.

Materials and Methods

Materials

Scoured and bleached 100% polyester fabric (149 g/m^2) was supplied by El-Mahalla El-Kobra Company. The fabric was treated before dyeing with a solution containing nonionic detergent (Hostapal CV, Clariant-Egypt, 5 g/L) and sodium carbonate (2 g/L) in a ratio of 50:1 at 60 °C for 30 min, thoroughly washed with water, and air dried at room temperature.

General procedure for the synthesis of azo disperse dyes 8a-f

A cold solution of aryldiazonium salt (10 mmol), (prepared by adding a solution of sodium nitrite (1.00 g in 10 mL H_2O) to a cold solution of arylamine hydrochloride (10 mmol) with stirring as described earlier) (3). The resulting solution of the aryldiazonium chloride was then added to a cold solution of compound **6a,b** (10 mmol) in ethanol (20 mL) containing sodium acetate (2.00 g). The mixture was stirred at room temperature for one hr and the solid product so formed was collected by filtration and re-crystallized from proper solvent. Dyes 8a-d were recently prepared [6, 7].

1,4-Diethyl-5-[(2-hydroxy-phenyl)-hydrazono]-2,6-dioxo-1,2,5,6-tetrahydro-pyridine-3-carbonitrile (8e)

Yield 2.14 g (68%), m.p. 148-150 ºC. IR (KBr): υ = 3370 cm^{-1}(OH), 3298 cm^{-1}(NH), 2100 cm^{-1} (CN), 1704, 1635 cm^{-1} (2CO). MS: m/z = 311 (M^{-1},100%), 297(11%), 267(7%), 241(5%), 107(14%), 79(11%).

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1,4-diethyl-5-(2-(3-nitrophenyl)hydrazono)-2,6-dioxo-1,2,5,6-tetrahydropyridine-3-carbonitrile (8f).

Yield 2.12 g (63%), m.p. 292-294 ºC. IR (KBr): υ = 3265 cm^{-1} (NH), 2219 cm^{-1} (CN), 1673, 1630 cm^{-1} (2CO). 340 (M^{-1} 100%), 326(17%), 393(18%), 267(12%), 250(16%), 138(18%), 91(12%).

Dyeing

Conventional dyeing method (Carrier method)

Samples (2 g) were introduced into a flask containing a dyebath of 2% (w.o.f) dye shade and (Avolan IS) as dispersing agent 1-4%, commercial HC carrier (Liquid) supplied by Egyptian Turkish Co. (Cairo, Egypt) for auxiliaries with ratio 2% (w.o.f) at 100 ºC with a 1:50 liquor ratio. During dyebath preparation, the dye was mixed with 10 drops of DMF and then mixed with dispersing agent, and water was added to prepare a homogeneous dispersion of the dye. The pH was adjusted to 4.5 by using acetic acid. At the end of the dyeing process after 1 h, the dyed samples were removed, rinsed in warm water, and subjected to reduction clearing in a solution comprising 2 g/L of sodium hydrosulphite and 2 g/L of sodium hydroxide (caustic soda) for 10 min at 60 ºC, with a liquor ratio of 1:40, and the reduction-cleared sample was rinsed thoroughly in water. The dyed samples were removed, rinsed in tap water, and allowed to dry in the open air [6].

High temperature dyeing method (HT)

A dispersion of the dye was produced by dissolving the appropriate amount of dye (2% shade) in 10 drops of DMF and then added dropwise with stirring to the dye bath (Liquor ration 50:1) containing sodium lignin sulfonate as dispersing agent 1-4%. The ratio of dispersing agent to dyestuff is 4 to 1. The pH of the dye bath was adjusted to 4.5 using acetic acid and the wetted polyester fabrics were added. Dyeing was performed by raising the dye bath temperature to 130 °C under pressure in a dyeing machine at a rate of 15 °C/min, holding at this temperature for 60 min and then cooling to 50 °C. After dyeing, the fabrics were thoroughly washed and subjected to surface reduction clearing ((2 g NaOH + 2 g sodium hydrosulphite)/L The samples were washed and air-dried.

Colour measurements and analyses

Colour measurements

The color yields of the dyed samples were determined by using the light reflectance technique performed on a Perkin-Elmer (Lambda 3B) UV/VIS Spectrophotometer. The color strengths, expressed as K/S values, were determined by applying the Kubelka-Mink equation [8,9].

Color Fastness tests

Fastness properties of the dyed samples were tested to washing, rubbing, perspiration, and light fastness according to AATCC standard tests [10-12].

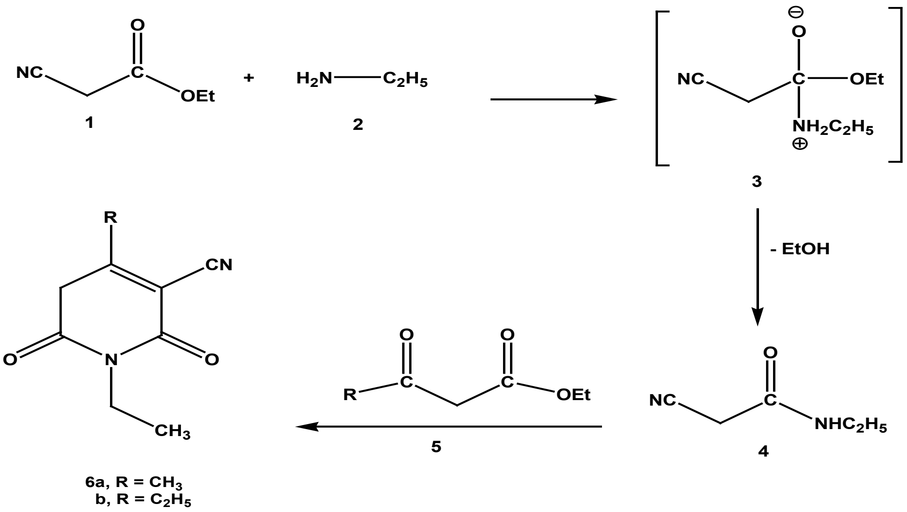
Results and Discussion

Pyridones **6a,b** are prepared by reacting ethyl cyanoacetate, with ethyl amine to produce amide derivatives **4**, which reacts with ethylacetoacetate or methyl propionylacetate to afford the target compounds (*cf.* Scheme 1). Coupling of compounds **6a,b** with aromatic diazonium salts afforded the arylhydrazono disperse dyes **8a-f** based on pyridone moiety (*cf.* Scheme 1).

Disperse dyes **8a-f** were applied to polyester fabrics using both high temperature dyeing method (HT) at 130 °C and/or carrier dyeing method. Yellow, dark yellow, and brown color shades were obtained. The dyeing properties on the polyester fabrics were evaluated in terms of their fastness properties (e.g., fastnesses to washing, rubbing, light, and perspiration). Tables 1 and 2 show the colour of dyeing on polyester fabrics is expressed in terms of CIELAB coordinates as: lightness (L*); *a** (red - green); and *b**, (yellow - blue). The data listed in Tables 3 and 4 show that the high temperature dyeing method displayed very good fastness levels to washing, rubbing and perspiration, while, the carrier dyeing method has good fastness levels to washing, rubbing and perspiration. The light fastness properties are fair in both different dyeing methods.

Effect of dispersing agent concentration on the K/S using carrier

Table 5 shows the relationship between K/S as a parameter for dye uptake and the concentrations of the dispersing agent used from 1 to 4 %, generally, the values K/S decrease with increasing amount of dispersing agent applied at both 100 and 130 °C.



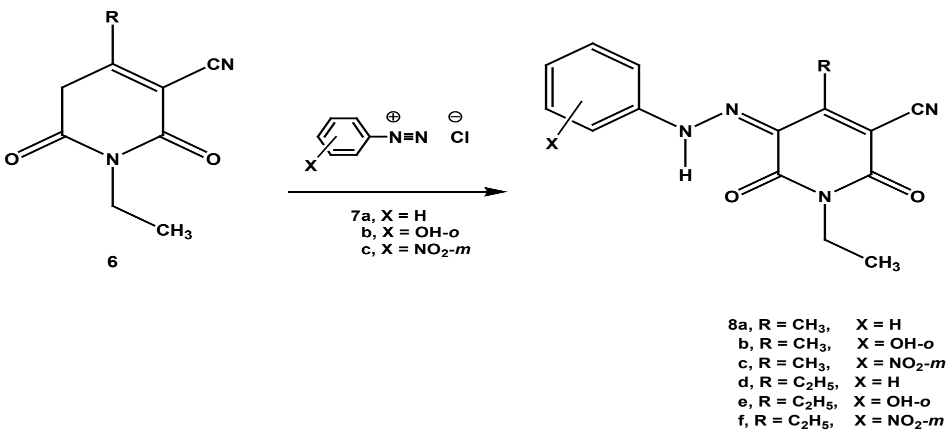


TABLE 1. Optical measurements of the synthesized disperse dyes 8a-f on polyester fabric at100 °C.

Dye NO.	Absorption λ_{max}/nm	L*	*a**	*b**	K/S
8a	440	75.88	-6.66	90.47	25.98
8b	470	67.57	16.72	55.98	6.36
8c	435	76.13	-8.77	71.94	10.48
8d	440	74.26	-3.23	88.48	25.84
8e	440	63.79	14.23	51.21	6.88
8f	430	64.09	14.46	72.53	26.91

TABLE 2. Optical measurements of the synthesized dyes disperse dyes 8a-f on polyester fabric at 130 °C.

Dye NO.	Absorption λ_{max}/nm	L*	*a**	b*	K/S
8a	440	72.24	1.80	90.82	27.39
8b	470	54.19	25.20	56.95	17.33
8c	435	73.96	-1.42	83.03	20.77
8d	445	74.42	1.80	93.82	28.22
8e	440	49.64	18.09	44.75	15.22
8f	430	64.92	12.75	72.84	28.36

In both dyeing methods disperse dyes 8c and 8f (having electron-attracting nitro group) show better light fastness (6) than disperse dyes 8b and 8e (having electron donating hydroxyl group).

TABLE 3. Fastness properties of the synthesized disperse dyes 8a-f on polyester fabrics at 100°C.

Dye No.	Washing			Perspiration						Rubbing		Light (40h)
				Acid			Alkali					
	ALT	SC	SW	ALT	SC	SW	ALT	SC	SW	Wet	Dry	
8a	4-5	4-5	4-5	4-5	4-5	4	4-5	4-5	4	4	4-5	6
8b	4-5	4-5	4	4	4	4	4-5	4	4	3-4	3-4	4-5
8c	4	4-5	4	4-5	4-5	4-5	4-5	4-5	4	3-4	3-4	6
8d	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4	4-5	6
8e	4	4-5	4	4	4	4	4-5	4	4	3-4	4-5	4-5
8f	4-5	4-5	4-5	4	4	4	4	4	4	3-4	4-5	6

TABLE 4. Fastness properties of the synthesized disperse dyes 8a-f on polyester fabrics at 130°C.

Dye No.	Washing			Perspiration						Rubbing		Light (40h)
				Acid			Alkali					
	ALT	SC	SW	ALT	SC	SW	ALT	SC	SW	Wet	Dry	
8a	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4	4-5	4-5	6
8b	4	4	4	4-5	4-5	4	4-5	4	4	4-5	4-5	4-5
8c	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	5	6
8d	4-5	4-5	4-5	4-5	4-5	4	4-5	4-5	4	4-5	4-5	6
8e	4-5	4-5	4-5	4-5	4-5	4	4-5	4-5	4	4-5	4-5	4-5
8f	4-5	4-5	4-5	4-5	4-5	4	4	4	4	4-5	4-5	6

TABLE 5. Effect of concentration of dispersing agent on the K/S using carrier 2% g/l.

Dye No.	Dyeing at 100 °C			Dyeing at 130 °C		
	Concentration of dispersing agent	Absorption λ_{max}/nm	K/S	Concentration of dispersing agent	Absorption λ_{max}/nm	K/S
8a	1	440	21.35	1	440	32.84
	2		19.82	2		32.27
	3		20.39	3		32.55
	4		20.70	4		32.62
8b	1	470	5.47	1	470	18.96
	2		4.92	2		15.36
	3		5.05	3		15.53
	4		5.13	4		18.96
8c	1	435	10.19	1	435	23.31
	2		8.74	2		21.95
	3		9.15	3		22.67
	4		9.91	4		23.13
8d	1	360	21.18	1	360	23.34
	2		17.81	2		16.87
	3		21.06	3		17.74
	4		21.13	4		22.00
8e	1	470	6.10	1	470	20.79
	2		5.39	2		19.26
	3		5.64	3		20.02
	4		5.74	4		20.31
8f	1	430	23.15	1	430	28.41
	2		22.77	2		26.66
	3		22.93	3		27.86
	4		23.09	4		27.95

Conclusion

Coupling of 4-methy pyridone-3-carbonitrile or 4-ethyl pyridone-3-carbonitrile with aromatic diazonium salts affords a series of disperse dyes. Disperse dyes having electron withdrawing group show better light fastness than dyes having electron donating group. Also, for both dyeing methods, the dye uptake expressed as K/S decrease with increasing the amount of dispersing agent.

References

1. Osman H. and Khairy M., Optimization of polyester printing with disperse dyes nano printing, *India Journal Of Fibre And Textile Research,* 202 (2013).

2. Arthur D. Broadbent, *Basic Principles of Textile Coloration,* Society of Dyers and Colourists, 307 (2001).

3. Alya Al-Etaibi, Morsy Ahmed El-Apasery, Huda Mahmoud, and Nouria Al-Awadi, Synthesis, characterization and antimicrobial activity, and applications of new azo pyridone, *European Journal of Chemistry*, **321** (2014).

4. Ismail Ajaj, Fathi H. Assaleh, Jasmina Markovski, Milica Ranc, Danijela

Brkovic, Milos Milc and Aleksandar D. Marinkovic, Solvatochromism and azo–hydrazotautomerismof novel arylazopyridone dyes, *Arabian Journal of Chemistry*, **2** (2015).

5. Jasmina Dostanic, Natasa Valentic, Gordana Uscumlic and Dusan Mijin, Synthesis of 5-(substituted phenylazo)-6-hydroxy-4-methyl-3-cyano-2-pyridones from ethyl 3-oxo-2-(substituted phenylazo)butanoates. *J. Serb. Chem. Soc.* **76** (4), 499–504 (2011).

6. Alya Al-Etaibi, Morsy A. El-Apasery and Nouria Al-Awadi. The effect of dispersing agent on the dyeing of polyester fabrics with disperse dyes derived from 1, 4-diethyl-2, 6-dioxo-1, 2, 5, 6-tetrahydropyridine-3-carbonitrile. *European Journal of Chemistry,* **4** (3), 240-244 (2013)

7. Kurenkaka Johnson Sakoma, Kasali Ademola Bello and Mohammed Kabir Yakubu*,* synthesis of some azo disperse dyes from 1-substituted 2-hydroxy-6-pyridone derivatives and their colour assessment on polyester fabric. *Open Journal of Applied Sciences*, **2**, 54-59 (2012).

8. Arcoria A., Cerniani A., De Giorgy R., Longo M.L. and Toscano R.M., Carrier dyeing of polyester fibre with some disperse azo dyes , *Dyes Pigments,* **11** (4), 269-276 (1989).

9. Al-Etaibi A.M., El-Apasery M.A., Huda H.M. and Al-Awadi, N.A., *Molecules* **17,** 4266-4280 (2012).

10. Al-Mousawi S.M., El-Apasery M.A. and Mahmoud H. M., *Molecules*, **18,** 7081-7092 (2013).

11. Chrysler L.P., *Methods of Test for Color Fastness of Textiles and Leather,* 7th ed., Bradford, London, 89-94 (1990).

12. El-Apasery M.A., Al-Qalaf F., Almohammad K. and Mahmoud H., *Eur. J. Chem*, **4** (3), 211-215 (2013).

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تشييد بعض الصبغات الازية المشتتة المعتمدة على حلقة البيريدون و أستخداماتها على أقمشة البولى إستر

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