

New Disperse Dyes for Polyester Microfibres

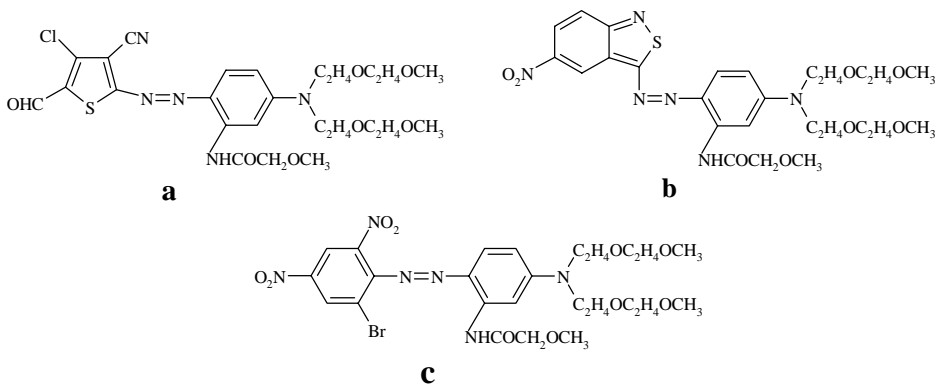
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Abstract: A series of disperse dyes bearing ether groups have been synthesized. The visible absorption spectra of them were studied, their fastness on polyester microfibres were investigated.

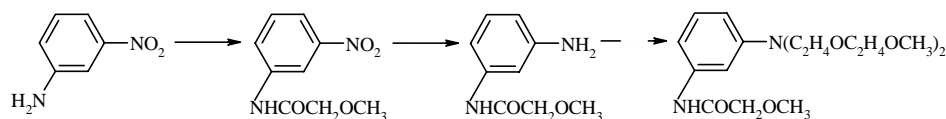
Keywords: Disperse dye, ether group, microfibres.

With the rapid development of polyester microfibres, there is a urgent need to develop suitable disperse dyes¹. Although many works have been done in this field, most of the dyes are selected from the conventional disperse dyes, such as C.I. Disperse Blue 257, C.I. Disperse Red 135². Here we report a series of new disperse dyes bearing $-N(C_2H_4OC_2H_4OCH_3)_2$ group, they are found to have good fastness on polyester microfibres.



The dyes were prepared according to the literature respectively³⁻⁵, the coupling component was prepared as described in **Scheme 1**.

Scheme 1



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Reagents and conditions: i) $\text{CH}_3\text{OCH}_2\text{COCl}/\text{CH}_3\text{COONa}/\text{CH}_3\text{COOH}$, 20°C, 2h, 95%, ii) $\text{Fe}/\text{CH}_3\text{COOH}/\text{H}_2\text{O}$, 90-95°C, 5h, 89%, iii) $p\text{-CH}_3\text{C}_6\text{H}_4\text{SO}_3\text{C}_2\text{H}_4\text{OC}_2\text{H}_4\text{OCH}_3/\text{methyl isobutyl ketone}/\text{MgO}$, reflux, 15h, brown viscous oil and was used without isolation further.

The spectroscopic data showed that the dye **a** and **b** were more bathochromic than **c**, the factor are associated with the thienyl or the isothiazole residues. Since the heterocyclic residues are more polarizable than the benzene ring, so the electronic effects of substituents (*e.g.* $-\text{NO}_2$, $-\text{CN}$, $-\text{CHO}$) are transmitted more readily to the rest of the chromogen. All the dyes showed good fastness properties on polyester microfibrils having diameter of 0.4 denier, especially in sublimation fastness. It might be explained by the introduction of ether group which increased the dyes' molecular weight and their compatibility with the fibres.

Table 1 Visible absorption spectra of **a**, **b** and **c** in DMF

Dye	λ_{max} (nm)	ϵ ($\times 10^{-4}$)	Shade on fabrics
a	618	6.79	blue
b	620	5.15	blue
c	552	3.78	violet

Table 2 Fastness of **a**, **b** and **c** on polyester microfibrils (0.4 denier)

Dye	Sublimation 180 /30sec			Sublimation 190 /30sec			Washing					Rubbing		
	1	2	3	1	2	3	W	R	S	N	C	A	wet	dry
a	4-5	4-5	5	4-5	4-5	5	3-4	5	3	3-4	5	4-5	5	4
b	4-5	4	4	4-5	3-4	4-5	3-4	5	3-4	4	5	4	5	5
c	4-5	5	5	4-5	4	4-5	3-4	5	4	5	5	4-5	5	5

*1, change of shade on dyed fabric; 2, staining polyester; 3, staining cotton; W, wool; R, Rayon; S, silk; N, nylon; C, cotton; A, acetate

References and Notes

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6. **a**: $^1\text{H-NMR}$ (400MHz, CDCl_3 , δ ppm): 3.36 (s, 6H, $-\text{CH}_2\text{CH}_2\text{OCH}_3$), 3.50-3.53 (m, 4H, $-\text{CH}_2\text{CH}_2\text{OCH}_3$), 3.61-3.63 (m, 4H, $-\text{CH}_2\text{CH}_2\text{OCH}_3$), 3.63 (s, 3H, $-\text{COCH}_2\text{OCH}_3$), 3.76-3.78 (m, 4H, $-\text{NCH}_2\text{CH}_2\text{O}-$), 3.82-3.84 (m, 4H, $-\text{NCH}_2\text{CH}_2\text{O}-$), 4.06 (s, 2H, $-\text{COCH}_2\text{OCH}_3$), 6.64-6.67 (d, 1H, Ar-H), 7.89 (s, 1H, Ar-H), 8.04 (s, 1H, Ar-H), 9.97 (s, 1H, $-\text{CHO}$), 10.24 (s, 1H, $-\text{NHCO}-$); IR ($\text{KBr}/\text{cm}^{-1}$): 2226, 1697, 1653, 1612; API-ES (m/z): 581.1. **b**: $^1\text{H-NMR}$ (400MHz, CDCl_3 , δ ppm): 3.38 (s, 6H, $-\text{CH}_2\text{CH}_2\text{OCH}_3$), 3.53-3.55 (m, 4H, $-\text{CH}_2\text{CH}_2\text{OCH}_3$), 3.63 (s, 3H, $-\text{COCH}_2\text{OCH}_3$), 3.63-3.65 (m, 4H, $-\text{CH}_2\text{CH}_2\text{OCH}_3$), 3.79-3.82 (m, 8H, $-\text{NCH}_2\text{CH}_2\text{O}-$), 4.11 (s, 2H, $-\text{COCH}_2\text{OCH}_3$), 6.66-6.69 (d, 1H, Ar-H), 7.66-7.69 (d, 1H, Ar-H), 7.84-7.94 (br, 1H, Ar-H), 8.13-8.16 (br, 2H, Ar-H), 9.12 (s, 1H, Ar-H), 10.15-10.35 (br, 1H, $-\text{NHCO}-$); IR ($\text{KBr}/\text{cm}^{-1}$): 1697, 1606, 1550, 1313; API-ES (m/z): 590.2. **c**: $^1\text{H-NMR}$ (400MHz, CDCl_3 , δ ppm): 3.36 (s, 6H, $-\text{CH}_2\text{CH}_2\text{OCH}_3$), 3.49 (s, 3H, $-\text{COCH}_2\text{OCH}_3$),

3.51-3.53 (m, 4H, $-\text{CH}_2\text{CH}_2\text{OCH}_3$), 3.60-3.62 (m, 4H, $-\text{CH}_2\text{CH}_2\text{OCH}_3$), 3.75-3.79 (m, 8H, $-\text{NCH}_2\text{CH}_2\text{O}-$), 4.03 (s, 2H, $-\text{COCH}_2\text{OCH}_3$), 6.60-6.63 (dd, 1H, Ar-H), 7.82-7.84 (d, 1H, Ar-H), 8.14 (s, 1H, Ar-H), 8.47-8.48 (d, 1H, Ar-H), 8.66-8.67 (d, 1H, Ar-H), 9.95-10.15 (br, 1H, $-\text{NHCO}-$); IR ($\text{KBr}/\text{cm}^{-1}$): 3350, 3084, 1699, 1612, 1525, 1329; API-ES (m/z): 656.1.

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