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Dyes and Pigments, Vol. 28, No. 2, pp. 123–130, 1995

0143-7208/95 $9.50 + 0.00

0143-7208(95)00007-0

Dyeing of Polyester with CI Disperse Yellow 42 in the Presence of Various UV-Absorbers. Part II

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(Received 18 November 1994; accepted 6 January 1995)

ABSTRACT

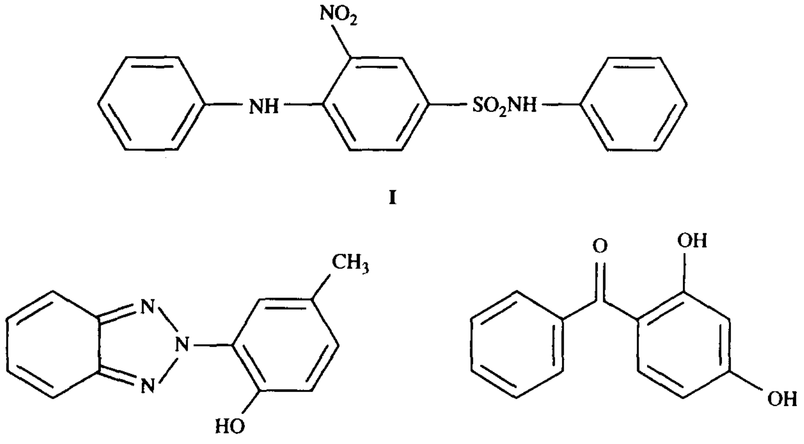
The effect of various amounts (0·5–1·2 owf) of two benzotriazole (Tinuvin P and Tinuvin 320) and one benzophenone (Ultrafast 800) type UV-absorbers applied directly in the dyeing of polyester fibres with CI Disperse Yellow 42 at various depths (0·5, 1·0 and 2·0% owf), or by after-treatment of the dyed fibre, was studied. Combinations of Tinuvin 320 with various amounts of an antioxidant (BHT) in the dye liquor were also used and results compared.

1 INTRODUCTION

In a previous paper[1] results from the application of the UV-absorber (UV-abs) Tinuvin P [2-(2-hydroxy-5-methylphenyl)-2H-benzotriazole] by different processes (pretreatment, addition to the dyebath, or after-treatment) on the light fastness of polyester fabrics dyed with CI Disperse Blue 85 and a red monoazo disperse dye were presented. An introduction referring to the application of UV-absorbers to automobile coloured upholstery to improve their light fastness, and relevant literature and patents, were also reported.[1]

In this paper, CI Disperse Yellow 42 (**I**), having a higher light fastness than the two previously studied dyes,[1] was used at various depths (0·5, 0·1, 2·0% owf). Dye **I** meets the requirements of automobile manufacturers only after an improvement of its light fastness.

In the present work, such an improvement is studied by applying the UV-abs Tinuvin P (**II**), Tinuvin 320 (**III**, benzotriazole of unknown



I

II

IV

Fig. 1. Dyes and UV-absorbers used.

formula) and Ultrafast 800 (**IV**, 2,4-dihydroxybenzophenone) in various amounts (0·3–12·0% owf) in the dye-liquor (process a), or by after-treatment of the dyed fabrics (process b).

An examination of the effect of the commonly used antioxidant BHT (**V**, 2,6-di-*tert*-butyl-4-methylphenol) in various combinations with the UV-abs **III** on the light fastness of the dyed fabrics by process a (dyeing depths 0·5 and 1·0% owf) was also carried out. The structures of the dyes and UV-absorbers are shown in Fig. 1.

A study of the improvement of the light fastness of dye **I** has been also reported[2] by incorporating moieties of various UV-abs into the backbone of the parent dye.

2 EXPERIMENTAL

2.1 Materials

Dye **I** was purified by extraction with ethanol and column chromatography of the extract on Silica Gel 60 (Merck) using toluene: ethylacetate (10–15% v/v) as eluant.

The commercial UV-abs **II** and **III** were purified by recrystallisation from aqueous acetone (m.p. 131–132°C and 153–154°C, respectively). Suspensions were obtained by adding an acetone solution to the dyebath and evaporating the solvent.

The amounts of dye **I** and of UV-abs **II** and **III** quoted in this paper correspond to 100% of pure compound, and those of **II** to dry solid matter (constant weight at 130°C).

2.2 Methods

2.2.1 Dyeing

Dyeings of polyester fabric were carried out with a liquor ratio of 30 : 1 in a Rotadyer apparatus, as described previously.[1]

2.2.2 Application of the UV-abs

This was effected using two different processes.

Process a. Addition of the appropriate amount of the UV-abs to the dye liquor, and subsequent dyeing.[1]

Process b. After-treatment of the dyed fabrics in an aqueous suspension of the appropriate amount of each UV-abs, by raising the temperature to 130°C over 30 min, and maintaining it at this level for 1 h.

All dyed fabrics were rinsed with 10 ml of distilled water, squeezed and air dried.

2.2.3 Application of antioxidant

This was made by addition of 0·5, 1·0 or 2·0% owf BHT to the dye liquors, in combination with the UV-abs Tinuvin 320 as above.

2.2.4 Determination of dye and UV-abs uptake

Calibration curves were obtained using chloroform or methanol solutions of dye, chloroform solutions of the UV-abs **II** and **III**, and methanol solutions of the UV-abs **IV**. The dye and UV-abs uptakes were determined spectrophotometrically by evaporating the bath liquors, dissolving the dry residues in chloroform or methanol (**IV**), and measuring the absorbances at the λ_{max} of the dye (408 mm) and of the UV-abs **II** (341 mm), **III** (343 mm) and **IV** (290 mm). Calculations of the UV-abs content in the presence of dye were done after subtraction of the absorbances of the dye at the λ_{max} of the UV-abs **II**, **III** and **IV**, which were determined by correlating the absorbance of the dye at its λ_{max} (408 mm) and at the above wavelengths. All the determinations were made in duplicate, and the results given are the mean values.

2.2.5 Light fastness tests

These were carried out according to BS 1006: 1992 BO2 (Ref. 3) at 50 ± 2°C.

3 RESULTS

3.1 Extinction coefficients

CI Disperse Yellow 42 (**I**), Tinuvin P (**II**), Tinuvin 320 (**III**) and Ultrafast 800 (**IV**) obeyed the Beer–Lambert law at the concentrations applied.

$E^{1\%}_{\lambda_{max}}$ (or the molar extinction coefficients $E_{\lambda_{max}}$) for the purified **I-IV** were found to be:

I: 150 (5650 l mol^{-1} cm^{-1}) in methanol (408 nm), and 160 (5904 l mol^{-1} cm^{-1}) in chloroform (412 nm),
II: 751 (16895 l mol^{-1} cm^{-1}) in chloroform (341 nm),
III: 453 in chloroform (343 nm) and
IV: 520 (10914 l mol^{-1} cm^{-1}) in methanol (290 nm).

3.2 Dyeing results

The dyeing results (dye and UV-abs uptake and % exhaustion) in the absence and presence of various amounts of the UV-abs **II-IV** for various dyeing depths, and the light fastness of the dyed fabrics by process a (addition of UV-abs in the initial dyeing liquor), are given in Table 1.

Comparison of the dye uptake in Table 1 shows that the three UV-abs have a different effect on the dye uptake, depending on the dyeing depth and UV-abs/dye molar ratio.

In particular, the presence of a benzotriazole type UV-abs, Tinuvin P (**II**) or Tinuvin 320 (**III**), at all three dyeing depths, slightly decreased or did not affect the dye uptake or exhaustion (Table 1). Such a reduction of the dye uptake or exhaustion could be attributed to an association of the UV-abs with the dye through the basic amino groups of dye **I** and the acidic phenolic hydroxyl groups of the UV-abs, resulting in larger dye particles and a lower absorption–diffusion. Similar results were also observed for analogous dyeings with two other dyes in the presence of **II**.

In contrast, the presence of a benzophenone type UV-abs (**IV**) resulted in a slight increase of dye uptake and exhaustion in all amounts applied. This different effect relative to that of the above two UV-abs (powders) might be due to the emulsifying agent of the Ultrafast 800 (emulsion) acting as carrier.

A high exhaustion of Tinuvin P and Tinuvin 320 was observed, higher for the former, in all dyeings shown in Table 1, whereas Ultrafast 800 showed significantly lower exhaustion values for corresponding amounts or UV-abs/dye molar ratios. Hence, a lower UV-abs/dye molar ratio on the dyed fabric was found for Ultrafast 800 than for the Tinuvins.

Light fastness values for the reference samples (absence of UV-abs) increased with increasing dyeing depth, as was anticipated. However, such an increase was not always observed for samples dyed at different depths and in the presence of the same initial UV-abs/dye molar ratio (Table 1). Thus, a one molar ratio of Tinuvin P/dye resulted in the same light fastness value (6–7) for all the three dyeing depths.

Addition of the UV-abs in the dyebaths (process a) resulted, in almost

TABLE 1
Dye and UV-abs Uptake and Light Fastness of Fabrics Dyed at Various Depths and Amounts of UV-abs (Process a)

Depth of dyeing (% owf)	*UV-abs*	*UV-abs (g) per 100 g fabric in dyeing liquor (UV-abs/dye molar ratio)*	*Dye (g) per 100 g dyed fabric (% exhaustion)*	*UV-abs (g) per 100 g dyed fabric (% exhaustion)*	*UV-abs/dye molar ratio on dyed fabric*	*Light fastness*
0·5	None	—	0·43 (86·9)	—	—	5–6
	Tinuvin P	0·3 (1·0)	0·34 (68·0)	0·25 (84·8)	1·21	6–7
	(II)	0·6 (2·0)	0·39 (78·9)	0·55 (92·4)	2·32	6–7
		1·5 (5·0)	0·44 (89·0)	1·47 (97·8)	5·48	7
	Tinuvin 320	0·5 (1·65)[a]	0·39 (78·2)	0·41 (81·3)	1·73[a]	6–7
	(III)[a]	1·0 (3·29)[a]	0·43 (85·1)	0·91 (90·9)	3·48[a]	7
		2·0 (6·58)[a]	0·42 (84·9)	1·86 (93·0)	7·24[a]	7
	Ultrafast 800	1·0 (3·46)	0·46 (92·2)	0·62 (62·2)	2·31	6–7
	(IV)	2·0 (6·92)	0·45 (91·1)	1·03 (51·7)	3·94	7
1·0	None	—	0·86 (86·4)	—	—	6
	II	0·6 (0·98)	0·80 (80·2)	0·58 (96·6)	1·18	6–7
		1·2 (1·97)	0·83 (83·5)	1·17 (97·7)	2·31	7
		3·0 (4·95)	0·81 (81·5)	2·90 (96·6)	5·88	7
		6·0 (9·80)	0·80 (80·4)	5·24 (87·4)	10·73	7–8
	III	0·5 (0·82)	0·83 (83·3)	0·45 (89·3)	0·89[a]	6–7
		1·0 (1·65)	0·85 (85·3)	0·92 (92·0)	1·77[a]	7
		2·0 (3·29)	0·84 (84·0)	1·90 (94·8)	3·70[a]	7
		1·0 (1·73)	0·94 (93·6)	0·60 (60·0)	1·09	7
		2·0 (3·46)	0·93 (93·1)	1·01 (50·6)	1·87	7–8
2·0	None	—	1·69 (84·5)	—	—	6–7
	II	1·2 (0·99)	1·68 (83·9)	1·18 (98·8)	1·15	6–7
		2·4 (1·97)	1·70 (85·4)	2·38 (99·0)	2·29	7
		6·0 (4·95)	1·75 (87·6)	5·98 (99·6)	5·66	7
		12·0 (9·90)	1·67 (83·5)	10·79 (89·9)	10·61	7–8
	III	0·5 (0·41)[a]	1·64 (81·8)	0·39 (78·7)	0·39[a]	6–7
		1·0 (0·82)[a]	1·68 (83·8)	0·66 (66·2)	0·64[a]	6–7
		2·0 (1·64)[a]	1·65 (82·7)	1·47 (73·3)	1·46[a]	7
	IV	1·0 (0·86)	1·89 (94·6)	0·56 (55·6)	0·51	7–8
		2·0 (1·73)	1·89 (94·4)	0·97 (48·3)	0·88	7–8

[a] Approximate value for a molecular weight equal to that of **II**.

all cases, in an improvement in light fastness ranging from 0·5–1·5 units on the blue standard scale. No improvement of light fastness was observed for samples dyed at the highest depth (2% owf) and a UV-abs/dye molar ratio lower than 1. The highest light fastness improvements (1·5 units) were observed for the depths of 0·5 and 1·0% owf in the presence of the highest amounts of the UV-abs (3–6% owf), or 3–9·8

UV-abs/dye molar ratio (Table 1). Nevertheless, the highest light fastness values (7–8 units) were observed for the samples of depth 1% and 2% owf in the presence of the highest molar ratio of Tinuvin P (9·9), and for a much lower molar ratio of Ultrafast 800 (3·5 and 0·9 respectively) (Table 1).

Comparing the effectiveness on the light fastness of the three UV-abs, at similar initial molar ratio to the dye, it is evident (Table 1, dyeing depths 1 and 2% owf) that Ultrafast 800 showed the best results, and Tinuvin 320 the worst. Moreover, considering the much lower molar ratios of Ultrafast 800/dye on the fabric in relation to the corresponding values of the Tinuvins, an even higher activity is concluded for the former.

The reference samples dyed in the absence of UV-abs, after treatment with the corresponding amounts of the UV-abs reported in Table 1, gave the results shown in Table 2. In all cases of the after-treatment (130°C, 1 h) with the three UV-abs, desorption of the dye (1·2–44·2%) was observed, resulting in some cases in a significant reduction of the final dye content on the fabric. The highest dye desorption resulted with the use of Tinuvin P, and the lowest with Ultrafast 800 (Table 2). Thus, the dye content on the final dyed fabrics by process b appeared to be lower in all cases than the corresponding value obtained with process a.

However, such a desorption was not observed[1] with other dyes during after-treatment with Tinuvin P under the same conditions (dyeing depth, amount of UV-abs owf, etc.). On the contrary, the UV-abs content on the final dyed fabrics by processes a and b was found to be substantially the same for the corresponding samples (Tables 1 and 2). Hence, the final dyed fabrics from process b generally showed a lower UV-abs/dye molar ratio content than the corresponding dyeings by process a, when applying the same amounts (% owf) of dye and UV-abs.

Most fabrics dyed by process b also showed an improvement in light fastness by treatment with all three UV-abs. Exceptions or even decreases of light fastness were observed for the samples which showed high desorption of dye during the after-treatment, mainly in the case of the more commonly used amounts of Tinuvin P. Nevertheless, treatment by a higher amount of Tinuvin P (6% owf) caused an improvement in light fastness. The improvements shown by process b were equal to, or lower than, those obtained by process a (Tables 1 and 2).

A different order for the three UV-abs as regards their light fastness effect was observed between processes a and b when using a similar UV-abs/dye molar ratio. Thus, in process b, Ultrafast 800 and Tinuvin 320 had a similar effect on the light fastness, and Tinuvin P had the lowest effect, in contrast to process a. However, as in process a, the same light fastness value was obtained using a lower final UV-abs/dye molar ratio on the fabric for Ultrafast 800 than for Tinuvin 320.

TABLE 2

Dye and UV-abs Content on Polyester Fabric Dyed and After-treated with UV-abs (Dyeing Process b)

Depth of dyeing (% owf)	*UV-abs*	*UV-abs (g) per 100 g fabric in treatment liquor*	*Dye (g) per 100 g dyed fabric after treatment (% dye loss by treatment)*	*UV-abs (g) per 100 g dyed fabric after treatment (% exhaustion)*	*UV-abs/dye molar ratio on dyed fabric after treatment*	*Light fastness*[a]
0·5	II	0·3	0·24 (44·2)	0·26 (86·5)	1·77	4–5
		0·6	0·30 (30·3)	0·57 (96·1)	3·11	5
		1·5	0·32 (25·6)	1·46 (97·5)	7·21	5–6
	III	0·5	0·38 (11·7)	0·40 (80·4)	1·72	6–7
		1·0	0·34 (21·0)	0·84 (80·7)	4·05	6–7
		2·0	0·33 (24·3)	1·79 (89·8)	8·09	7
	IV	1·0	0·39 (9·3)	0·61 (61·1)	2·56	6–7
		2·0	0·41 (4·7)	1·06 (52·8)	4·24	6–7
1·0	II	0·6	0·57 (33·7)	0·57 (94·6)	1·64	5–6
		1·2	0·59 (31·4)	1·18 (97·6)	3·28	5–6
		6·0	0·62 (27·9)	5·84 (96·1)	15·45	6–7
	III	0·5	0·78 (9·3)	0·40 (80·7)	0·84	6–7
		1·0	0·71 (17·4)	0·87 (86·9)	2·01	6–7
		2·0	0·71 (17·4)	1·80 (90·9)	4·16	7
	IV	1·0	0·83 (3·5)	0·58 (58·3)	1·14	6–7
		2·0	0·82 (4·7)	1·07 (53·3)	2·14	6–7
2·0	II	1·2	1·04 (38·5)	1·18 (97·2)	1·86	5–6
		2·4	0·96 (43·2)	2·39 (98·5)	4·55	5
		12·0	1·04 (38·5)	11·91 (98·1)	18·78	6–7
	III	0·5	1·26 (25·5)	0·45 (90·2)	0·83	6–7
		1·0	1·30 (23·1)	0·88 (88·0)	1·11	6–7
		2·0	1·31 (22·5)	1·79 (89·3)	2·24	7
	IV	1·0	1·64 (3·0)	0·58 (58·3)	0·54	7
		2·0	1·67 (1·2)	1·06 (52·8)	1·04	7

[a] Light fastness values for reference samples at depths of dyeing 0·5, 1·0 and 2·0% owf were 5–6, 6 and 6–7 respectively (Table 1).

Comparison between samples from processes a and b, having the same UV-abs and similar UV-abs/dye molar ratio on the fabric, showed that the light fastness of the former was higher than or equal to that of the latter. This does not agree with previous results,[1] where a higher improvement was obtained by process b; this might be due to the different behaviour (desorption) of dye **I** during the after-treatments, relative to that of previously reported dyes.[1]

Although a similar final UV-abs/dye molar ratio seems to result in a similar light fastness, in all cases no proportionality between them was

TABLE 3
Dye and UV-abs Uptake, and Light Fastness of the Dyed Samples (Process a) in the Presence of a Combination of Tinuvin 320 and Antioxidant BHT

Depth of dyeing and Tinuvin P (% owf)	*BHT (g) per 100 g fabric*	*Dye (g) per 100 g dyed fabric (% exhaustion)*	*UV-abs (g) per 100 g dyed fabric (% exhaustion)*	*Light fastness*
0·5	—	0·39 (78·2)	0·41 (81·3)	6–7
	0·5	0·38 (76·2)	0·40 (80·4)	6–7
	1·0	0·36 (72·4)	0·40 (80·4)	6–7
	2·0	0·35 (70·7)	0·37 (73·3)	6–7
1·0	—	0·85 (85·3)	0·92 (92·0)	7
	0·5	0·83 (82·7)	0·85 (84·9)	7
	1·0	0·82 (82·5)	0·78 (77·8)	7
	2·0	0·76 (75·6)	0·66 (66·2)	7

observed (Tables 1 and 2). The results of dyeing by process a in the simultaneous presence of various weight ratios (0·5–2) of Tinuvin 320 and antioxidant BHT are given in Table 3. In all cases, combination of Tinuvin 320 with the antioxidant BHT in the dye liquor caused a decrease of the dye and UV-abs uptake. This decrease was higher with increasing amounts of the antioxidant for the same dyeing depth and the same amount of UV-abs. However, the resultant reduction of the dye and UV-abs content on the fabric did not cause a measurable change of light fastness with respect to the samples dyed in absence of the antioxidant. This is in agreement with other results, where combinations of different antioxidants and benzo-phenone UV-absorbers were studied.[4]

ACKNOWLEDGEMENTS

The authors thank VIOCHROM S.A. (Aghia Varvara, Attiki, Greece) for the supply of materials.

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