



## Dyeing of Polyester Fabrics with Disperse Dyes in the Presence of a UV-Absorber

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### ABSTRACT

*The adsorption kinetics of the UV-absorber Tinuvin P, 2-(2-hydroxy-5-methyl-phenyl)-2H-benzotriazole on polyester (PET) fabrics have been studied.*

*Polyester fabrics were dyed with CI Disperse Blue 85 and a new red monoazo disperse dye in different depths (0.5, 1 and 2% o.w.f.) by application of the UV-absorber by different processes (pretreatment, simultaneous addition and aftertreatment) and various UV-absorber/dye molar ratios (0.5–3.5).*

*The application of the UV-absorber (0.1–2.5% o.w.f.) resulted in a slight decrease in dye uptake by the pretreatment and simultaneous addition processes, and in most cases in an improvement of light fastness (0–2 units of the Blue Standard scale) of the dyed fabrics. The light fastness improvement was generally higher for the aftertreatment process and for the blue dye.*

### 1 INTRODUCTION

UV-absorbers (UV-abs) have been utilized to protect polymeric materials from photodegradation<sup>1,2</sup> caused by sunlight or ultra-violet rich artificial light. Recently, interest has developed in the application of UV-abs in the dyeing of textile materials to protect their colour from fading. High light fastness of dyed materials is important especially for automobile coloured upholstery, which can be exposed to direct sunlight and temperatures above 50°C. In this context, several papers<sup>3–6</sup> and patents,<sup>7–18</sup> have made reference to the application of UV-abs in textile coloration processes,

An improvement in light fastness has been achieved either by addition

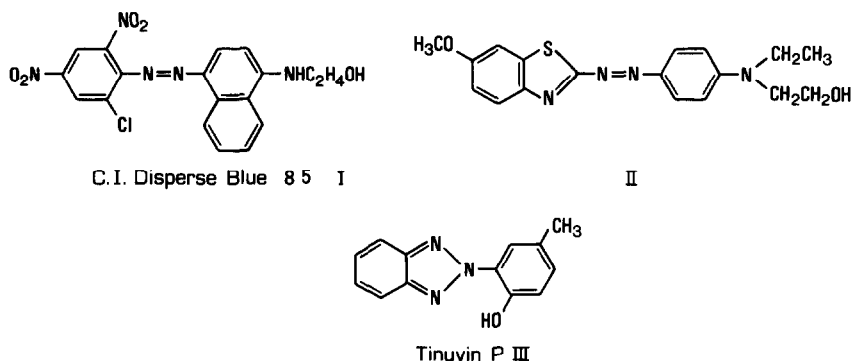


Fig. 1. Dyes and UV-absorber used in the present study.

(1–2%)<sup>3–18</sup> of a UV-abs in the dyeing process or by incorporating a UV-abs moiety in the dye molecule.<sup>19–22</sup>

Various commercial UV-abs, mainly of the benzophenone<sup>4,5,12–15</sup> and benzotriazole<sup>5–9,14,18</sup> and less of other classes (benzoic acid esters, hindered amines)<sup>14,17</sup> used directly in the dye bath up to 2% o.w.f., for polyester,<sup>4,5,8,9,12,14,18</sup> wool,<sup>6,13</sup> polyamide<sup>7</sup> and polypropylene<sup>11</sup> fibres caused a light fastness improvement ranging from 0 to 2 units of the blue standard scale<sup>23</sup> for various dyes.

Research has mainly focused on high light fastness disperse dyes applied on polyester fabrics on account of their use for automobile interiors. Nevertheless an investigation of light fastness improvement by applying UV-abs for dyes of lower light fastness is also interesting, especially when these are of low cost, brightness, etc., or applied to fabrics for other uses.

In this paper, a study of the dyeing of polyester (PET) fabrics with CI Disperse Blue 85 (I) and red dye (II) having bright colour, both of medium light fastness, was carried out in the presence and absence of the commercial benzotriazole UV-abs Tinuvin P (III) (Fig. 1), the light fastness of the fabrics dyed under different conditions was investigated. Thus various depths of dyeing (0.5–2% o.w.f.), molar ratios of UV-abs/dye (0.5–3.5) and three different methods of application of the UV-abs (pre- or aftertreatment of the fabrics or by addition of the UV-abs in the dye bath) were applied and the results, in comparison with the reference samples dyed in the absence of the UV-abs, are given and discussed.

## 2 EXPERIMENTAL

### 2.1 Materials

The dyes I (MW 415) and II (MW 356) used in this study were purified by extraction with ethanol and column chromatography of the extracted

material on Silica Gel 60 (Merck), applying the dyes from solution in chlorobenzene and eluting with a mixture of toluene: ethyl acetate (20–30% v/v).

The commercial yellowish powder of III (m.p. 128–132°C), after two recrystallizations from acetone, by addition of a small amount of water, afforded crystals m.p. 131–132°C (MW 225).

The amounts of dyes and UV-abs quoted in this paper correspond to 100% of pure materials. Commercial polyester (PET) fabrics were used.

## 2.2 Methods

### 2.2.1 Dyeing

The bath of the disperse dye (and/or of UV-abs) was prepared by adding an acetone solution of the appropriate amount of the dye (and/or of UV-abs) to the appropriate volume of water and then evaporating the acetone. No dispersing agent was used in order to avoid its interference in the spectrophotometric quantitative determinations of dye and UV-abs in the bath-liquor.

The polyester fabrics (5 × 10 cm) were dyed in a Rotadyer apparatus (Goodbrand Jeffrey's) at a liquor ratio of 1:30 with 0.5, 1 and 2% o.w.f. dye and 3% o.w.f. glacial acetic acid. The temperature was raised to 130°C within 30 min and maintained at this level for 1 h. The dyed fabrics were rinsed with 10 ml of distilled water and squeezed.

### 2.2.2 Application of the UV-absorber

This was done using three different processes:

- (a) pretreatment of the fabrics in a dispersion of the UV-abs (1% o.w.f.) under the same conditions to those of dyeing;
- (b) addition of the appropriate amount of the UV-abs in the dyeing liquor to obtain UV-abs/dye molar ratios of 0.5, 1 or 2 for each depth of dyeing;
- (c) aftertreatment of the dyed fabrics in a liquor (1:30) containing an appropriate amount of UV-abs corresponding to a UV-abs/dye molar ratio of 0.5, 1 or 2 for each depth of dyeing.

Adsorption kinetics of the UV-abs on polyester fabrics were carried out for three concentrations (0.2, 0.5 or 1% o.w.f.) under the dyeing conditions in the absence of dye.

### 2.2.3 Determination of dye and UV-abs uptake

Chloroform solutions of dye or UV-abs and a Pye Unicam SP 8000 spectrophotometer were used to obtain calibration curves. The dye and UV-abs

uptakes were determined spectrophotometrically, by evaporating the bath liquors, dissolving the dry residues in chloroform and measuring the absorbances at 341 nm for the UV-abs and 578 and 497 nm for the dyes I and II respectively. Calculations of the UV-abs content in the presence of dye were done after subtraction of the corresponding absorbance of the latter at 341 nm, which was determined by correlating the absorbance of the dyebath at its  $\lambda_{\max}$  and at 341 nm.

Equimolar mixtures of the dyes and the UV-abs did not show a shift in  $\lambda_{\max}$  and absorbance of the dyes, nor a shift in  $\lambda_{\max}$  of the UV-abs, indicating no formation of a complex between the dyes and the UV-abs.

#### 2.2.4 Light fastness tests

These were carried out in a Suntest Hanau apparatus with a Xenon lamp at temperature  $50 \pm 2^\circ\text{C}$  according to BS 1006: 1978 B02.<sup>23</sup>

## 3 RESULTS AND DISCUSSION

### 3.1 Molar extinction coefficients

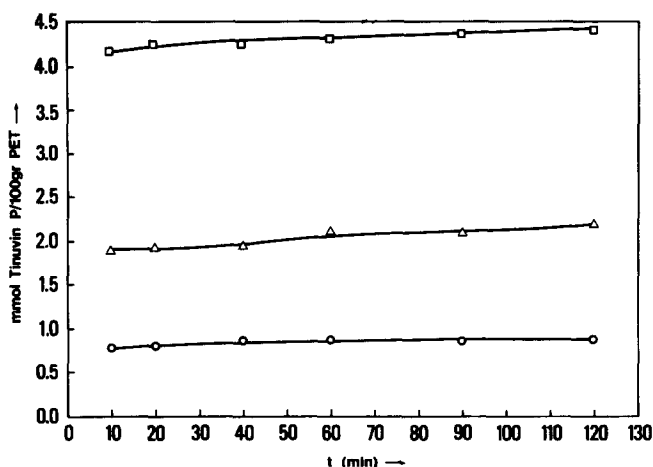
The dyes and UV-abs (Tinuvin P) used obeyed the Beer–Lambert law at the concentrations applied. The molar extinction coefficients for the purified I, II and III in chloroform solutions were found to be 27 680 l/(mol cm) (at 578 nm), 25 632 l/(mol cm) (at 497 nm) and 18 585 l/(mol cm) (at 341 nm) respectively and these values were used for their quantitative determinations. Thus, the purity of the commercial I, II and III was found to be 45, 53 and 92% w/w respectively.

### 3.2 Adsorption kinetics of the UV-abs III on PET fabrics

Adsorption isotherms at  $130^\circ\text{C}$  for III on polyester at different concentrations (0.2, 0.5 and 1% o.w.f.) are illustrated in Fig. 2. This figure shows that the UV-abs has been adsorbed almost quantitatively (99.0, 98.5 and 98.7%) from the three solutions (0.2, 0.5 and 1% o.w.f. respectively), equilibrium being achieved after 60 min.

### 3.3 Dyeing results

The dye uptake in the absence of UV-abs for the three dyeing depths (0.5, 1 and 2% o.w.f.) are given in Table 1. Fibres dyed in the absence of the UV-abs were further used as reference samples for the light fastness tests. Dyes I and II both showed small uptake differences for the different depths of dyeing used, while the uptake for I was generally somewhat higher than that of II.



**Fig. 2.** Absorption of the UV-absorber Tinuvin P on polyester (PET) fabric at 130°C from chloroform solutions of various concentrations ( $\circ \circ \circ$ , 25% w/v;  $\triangle \triangle \triangle$ , 0.5% w/v;  $\square \square \square$ , 1.0 w/v).

The results for the three dyeing processes were: (a) after pretreatment of the fabrics with 1% o.w.f. of UV-abs (130°C, 1 h); (b) by simultaneous addition of the UV-abs in the dyeing liquor and (c) by aftertreatment of the dyed fabrics with UV-abs dispersions for three dyeing depths (0.5, 1 and 2% o.w.f.) and various UV-abs/dye molar ratios are given in Tables 2, 3 and 4 respectively. All the fabrics used in dyeing process (a) were pretreated with 1% o.w.f. UV-abs (resulting in a UV-abs content of 4.34 mmol or 977 mg/100 gr fabric).

Comparison of the dye uptake values of Table 2 with those of Table 1 shows that the presence of the adsorbed UV-abs resulted in a decrease in dye

**TABLE 1**  
Dye Uptake of I and II for Various Depths of Dyeing

Dye	Depth of dyeing (% o.w.f.) (corresponding mmol dye/100 gr fabric)	Dye uptake (%)
I	0.5 (1.2)	99.4
	1.0 (2.4)	98.2
	2.0 (4.8)	98.0
II	0.5 (1.4)	97.5
	1.0 (2.8)	96.3
	2.0 (5.6)	95.5

TABLE 2

Dye Uptake, UV-abs Content and UV-abs/Dye Molar Ratios on the Dye after Pretreatment of Fabrics (Dyeing Process (a)) for Various Depths of Dyeing

Dye	Depth of dyeing (% o.w.f.) (corresponded mmol dye/100 gr fabric)	UV-abs molar ratio		Dye uptake (%)	UV-abs on 100 gr dyed fabric)	
		Before dyeing	On the dyed fabric		mmol	g
I	0.5 (1.2)	3.57	3.45	98.3	4.08	0.92
	1.0 (2.4)	1.80	1.80	90.8	3.93	0.88
	2.0 (4.8)	0.90	0.92	87.1	3.85	0.87
II	0.5 (1.4)	3.10	3.44	89.7	4.32	0.97
	1.0 (2.8)	1.54	1.77	86.9	4.32	0.97
	2.0 (5.6)	0.77	0.95	81.2	4.31	0.97

<sup>a</sup> Initial UV-abs content: 4.34 mmol or 977 mg/100 gr fabric.

TABLE 3

Dye and UV-abs Uptake for Various Depths of Dyeing and UV-abs/Dye Molar Ratios by Dyeing Process (b)

Dye	Depth of dyeing (% o.w.f.)	UV-abs/dye molar ratio in dyeing liquor	Dye uptake (%)	g UV-abs/100 g dyed fabric (UV-abs uptake (%))	UV-abs/dye molar ratio on the dyed fabric
I	0.5	0.5	97.1	0.12 (90.4)	0.46
		1.0	96.4	0.25 (94.5)	0.98
		2.0	96.4	0.52 (97.3)	2.01
	1.0	0.5	95.8	0.24 (91.3)	0.47
		1.0	95.1	0.49 (91.1)	0.96
		2.0	94.3	1.04 (97.4)	2.06
	2.0	0.5	94.1	0.50 (94.9)	0.50
		1.0	93.2	1.04 (97.5)	1.04
		2.0	88.6	2.07 (96.6)	2.15
II	0.5	0.5	96.4	0.15 (99.8)	0.51
		1.0	94.9	0.31 (99.7)	1.04
		2.0	92.4	0.63 (99.7)	2.16
	1.0	0.5	94.6	0.31 (99.8)	0.52
		1.0	93.4	0.63 (99.9)	1.06
		2.0	92.8	1.25 (99.5)	2.14
	2.0	0.5	92.7	0.63 (99.8)	0.53
		1.0	93.2	1.25 (99.6)	1.06
		2.0	93.1	2.50 (99.4)	2.13

uptake for both dyes I and II and its decrease is higher for higher depths of dyeing. A low desorption of UV-abs occurred during dyeing process (a) with dye I, while with dye II the desorption was insignificant (Table 2).

The determined UV-abs/dye molar ratio on the dyed fabric was found to be the same as that initially applied in the case of dye I, and slightly higher in the case of dye II (Table 2). This difference is explained by the relative lower dye uptake and UV-abs desorption of dye II compared with I.

The dye uptake by process (b) for both dyes I and II decreased with (i) increase in the dyeing depth for the same UV-abs/dye molar ratios and (ii) increase of UV-abs/dye molar ratio for the same dyeing depth (Table 3).

Comparison of the dye uptake between processes (a) and (b) (Tables 2 and 3) for the same dye and dyeing depths and approximately equal UV-abs/dye molar ratio (or equal UV-abs/dye content o.w.f.), shows that higher uptake values are obtained with process (b) (e.g. I: 94.3 versus 90.8% for depth 1%, UV-abs/dye 2 mol/mol and II: 93.2 versus 81.2% for depth 2%, UV-abs/dye, 0.9 mol/mol etc.).

The UV-abs uptake values under the conditions of process (b) (Table 3) compared to those obtained by process (a) (absence of dye) (Fig. 1) were found to be lower in the presence of dye I and slightly higher in the presence of dye II.

Comparison of the UV-abs contents on the dyed fabrics between dyeing processes (a) and (b) for the same depth of dyeing and initial UV-abs amounts (Tables 2 and 3) indicates higher (approx. 10%) values with process (b) for dyes I and II.

The UV-abs/dye molar ratios on the dyed fabric, compared to the corresponding ones in the bath liquor of process (b), were found to be practically the same for dye I, and slightly higher for dye II (Table 3).

No desorption of the dye was observed during aftertreatment of the fabrics with the UV-abs. Thus the dye uptake and dye content on the dyed fabrics by process (c) were the same as those of the reference sample for the same depth of dyeing, and independent of the applied UV-abs/dye molar ratio (Table 4). Consequently the dye uptake obtained by aftertreatment process (c) was higher than that of the dyeing processes (b) and (a) for the same dyeing depths for both dyes.

The UV-abs uptake values under the conditions of the aftertreatment process (c) with respect to process (a) (absence of dye) (Fig. 1) were found to be the same or slightly higher in the case of dye I and lower for dye II, in contrast to the corresponding results (referred to previously), for dyeing process (b).

The UV-abs/dye molar ratios on the dyed fabrics by process (c) were found to be practically the same as the applied corresponding ones (0.5, 1 and 2) for both dyes I and II (Table 4).

TABLE 4

UV-abs Content and UV-abs/Dye Molar Ratios on Dyes at Various Depths of Dyeing and Aftertreated with UV-abs at Various Concentrations of Polyester Fabrics (Dyeing Process (a))

<i>Dye</i>	<i>Depth of dyeing (% o.w.f.)</i>	<i>UV-abs/dye molar ratio in dyeing liquor</i>	<i>g UV-abs/100 gr dyed fabric (% UV-abs uptake)</i>	<i>UV-abs/dye molar ratio on the fabric</i>
I	0.5	0.5	0.13 (99.5)	0.49
		1.0	0.26 (97.5)	0.98
		2.0	0.53 (99.5)	2.01
	1.0	0.5	0.26 (99.8)	0.50
		1.0	0.53 (98.8)	1.01
		2.0	1.07 (98.7)	2.01
	2.0	0.5	0.53 (98.9)	0.50
		1.0	1.07 (99.4)	1.01
		2.0	2.15 (99.6)	2.03
II	0.5	0.5	0.14 (87.2)	0.45
		1.0	0.30 (96.3)	1.00
		2.0	0.60 (96.4)	2.07
	1.0	0.5	0.28 (90.0)	0.47
		1.0	0.59 (94.6)	0.98
		2.0	1.19 (94.6)	1.97
	2.0	0.5	0.68 (95.0)	0.50
		1.0	1.24 (98.6)	1.03
		2.0	2.51 (99.4)	2.09

### 3.4 Light fastness of the dyed fabrics

Light fastness values for the polyester fabrics dyed by the three dyeing processes ((a), (b), (c)) using various amounts of UV-abs (Tinuvin P) and dyeing depths, as well as those for the corresponding reference samples (R) dyed without the use of the UV-abs are given in Table 5. The reference samples dyed in absence of UV-abs, with CI Disperse Blue 85 I showed a slightly higher increase in light fastness with increasing depth of dyeing than those dyed with the red dye II, which showed no difference in light fastness between 0.5 and 1% depth of dyeing (Table 5). However, an increase in depth of dyeing in the presence/with addition of the same UV-abs/dye molar ratio (or the same UV-abs content) on the fabrics did not always result in an increase in the corresponding light fastness improvement, which in many cases appeared greater for lower than for higher depths of dyeing (Tables 5, 2-4).

Similarly, an increase in the UV-abs/dye molar ratio (or of the UV-abs/dye content) on the dyed fabrics (0.12-2.5% o.w.f., Tables 2-4) for the same



TABLE 5

Light Fastness Values of Polyester (PET) Fabrics Dyed in the Absence of UV-abs (R: reference samples) and with application of the UV-abs by pretreatment (a), simultaneous addition (b) and aftertreatment (c) processes. Black panel temperature  $50 \pm 2^\circ\text{C}$

Depth of dyeing (% o.w.f.)	UV-abs/dye molar ratio	I				II			
		R	(a)	(b)	(c)	R	(a)	(b)	(c)
0.5	0.5	2	—	2-3	3	2	—	2	2
0.5	1.0	—	—	2-3	3	—	—	2	2
0.5	2.0	—	—	3	3	—	—	2-3	2-3
0.5	3.1	—	—	—	—	—	2	—	—
0.5	3.5	—	3	—	—	—	—	—	—
1.0	0.5	2-3	—	3-4	4	2	—	2	3
1.0	1.0	—	—	3-4	4	—	—	2-3	3
1.0	1.5	—	—	—	—	—	2-3	—	—
1.0	1.8	—	3	—	—	—	—	—	—
1.0	2.0	—	—	3-4	4-5	—	—	3	3-4
2.0	0.5	3-4	—	3-4	4	3	—	3	3-4
2.0	0.8	—	—	—	—	—	3	—	—
2.0	0.9	—	4	—	—	—	—	—	—
2.0	1.0	—	—	3-4	4	—	—	3-4	4
2.0	2.0	—	—	4	4-5	—	—	4	4

depth of dyeing did not always result in an increase in the corresponding light fastness improvement (Table 5). No increase of light fastness improvement by increasing the amount of UV-abs (1-2%) has also been observed in several cases in the literature.<sup>4</sup>

The highest light fastness improvement (2 units of the blue standard scale relative to the reference sample) was obtained by dyeing process (c) using the blue dye I at 1% depth of dyeing and a UV-abs/dye molar ratio 2 (Table 5). The final UV-abs content was 1.07% o.w.f. (Table 4). The red dyeings showed the highest light fastness improvement by 1.5 units, also with the above dyeing conditions (process (c), 1% depth, UV-abs/dye = 2 mol/mol) with a final UV-abs content of 1.19% o.w.f. (Table 4).

Comparison of the light fastness improvement between the red and blue fabrics dyed under the same conditions showed that the application of the UV-abs Tinuvin P was more effective for the blue dyeings than the red ones at 0.5 and 1% depth of dyeing (Table 5). Particularly, no light fastness improvement was shown for the red samples dyed at 0.5% depth of dyeing by processes (a), (b) and (c) carrying a UV-abs/dye molar ratio  $\leq 1$  ( $\leq 0.31\%$  o.w.f. UV-abs) or at 1% depth of dyeing carrying the same UV-abs content (0.31% o.w.f.) (Table 5).

A relative comparison of the dyeing processes (a), (b) and (c) showed that the light fastness of the dyed fabrics increased in the order (a) → (b) → (c) for nearly all the cases of the blue dyeings and most cases of the red ones (Table 5).

Considering substantially the same UV-abs content (0.12–2.51% o.w.f) of the corresponding samples dyed by processes (b) and (c) (Tables 3 and 4) and the relatively lowest light fastness improvement of the samples dyed by process (a) (UV-abs content  $\approx$  0.9% o.w.f., Table 2), the highest light fastness improvement obtained by process (c) could be explained, in addition to its higher dye uptake (Tables 1–3), mainly by the assumption that this aftertreatment process results in a surfacial protective UV-abs 'coating' of the dye. Analogously, the lowest light fastness improvement for the samples dyed by the pretreatment process (a) and the intermediate results by the process (c) might also be explained.

#### 4 CONCLUSIONS

The UV-abs Tinuvin P was adsorbed almost quantitatively on the fabric under the dyeing conditions applied. The presence of the UV-abs in dyeing processes (a) and (b) resulted in a slightly lower dye uptake ((a) < (b)) than that of the aftertreatment process (c), which remained the same as initially. The UV-abs uptake values in the case of the blue dye were higher using process (c), whereas the red dye, they were higher with process (b). Both processes (a) and (b) showed higher UV-abs uptake for the red than the blue dyeings.

The UV-abs content on the fabrics (g/100 g) was higher for the red dyeings than for the corresponding blue dyeings by all processes (a), (b) and (c) (Tables 2–4). This is due to the higher initial amount of UV-abs used in the red dyeings, on account of the lower molecular weight of the red dye (356) than that of the blue dye (415) under the same depth of dyeing and UV-abs/dye molar ratios.

The UV-abs contents on the dyed fabrics under the same depth of dyeing and UV-abs/dye molar ratio were substantially the same by processes (b) and (c) and higher than the corresponding ones by process (a).

Application of the UV-abs Tinuvin P resulted in an improvement in light fastness ranging from 0 to 2 and 0 to 1.5 units of the blue standard scale for the samples dyed with dyes I and II respectively, with respect to the reference samples at the same depth of dyeing. The light fastness improvement with the use of UV-abs Tinuvin P was generally higher for the blue than for the red dyed fabrics and for process (c) relative to (b) and (a) (lowest). The highest light fastness improvement was obtained by the aftertreatment process (c)

for 1% depth of dyeing, and a final UV-abs content approx. 1–12% o.w.f. for both dyes.

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