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Synthesis of Bifunctional Monochlorotriazine Reactive Dyes Increasing UV-Protection Properties of Cotton Fabrics

Abstract

Several yellow and red bis-monochlorotriazine reactive dyes with built-in UV-absorber residues were synthesised. Their application properties were determined during the coloration of cotton, and the UV-protection factor (UPF) of the dyed fabrics was measured. The results of these measurements were compared with those obtained for coloration of the same fabrics carried out with the use of dyebaths containing a composition of a model dye and colourless fibre-reactive UV absorber.

Key words: chlorotriazine, UV-absorbers, ultraviolet protection factor (UPF).

Introduction

In the last decade increasing attention has been paid to protective properties of textiles against UV radiation originating from sunlight. In view of the practical and aesthetic points, in summer we usually wear clothes which are made of cotton, but this natural fibre comprises only a small barrier to ultraviolet radiation, especially when the textile garment is white or dyed with light colours. A typical industrial practice used in order to overcome this problem is the application of a colourless water-soluble auxiliary which shows an affinity to cellulose fibre and acts as a UV absorber [1-5]. Especially interesting from a practical point of view are fibre-reactive absorbers which, when fixed to the fabric, are able to maintain its protective properties for extended periods. Examples of such UV-absorbers – derivatives of triazine [6] and oxalic acid diarylide [7] – are presented in **Figure 1**.

In recent reviews of the possible development of synthetic dyes for textile applications, it has been suggested that a modern colorant could also perform additional functions beside its specific colouristic and fastness properties, such as antibacterial action, increased UV- or IR- absorption, or water repellence [8, 9]. These dual dye properties would allow the number of chemicals used in the dye-house to be reduced.

A synthesis of a dyes with built-in UV-absorber moiety was earlier reported in groups of disperse [10-11], acid [12], direct [12] and reactive dyes for cotton [13]. The aim of this work was to increase the lightfastness of newly-prepared color-

ants or to prevent the photodegradation of dyed fibres.

In our previous work on the synthesis of reactive dye derivatives of C.I. Reactive Red 198 containing built-in UV-absorber residue (2'-hydroxyphenylbenzotriazole) in their molecules, it was found that cotton fabric dyed with new colorants exhibited approximately 10% higher UPF-factor values in comparison with those of the same fabric coloured with model dye. A disadvantage of the dyes discussed above was their moder-

ate affinity to cellulose fibre, which limited their application in typical exhaust methods [14].

The purpose of the present work is to examine the synthesis of more substantive symmetric homobifunctional monochlorotriazine yellow and red dyes of a general structure, as presented in **Figure 2**.

While preparing these dyes, three water-soluble aromatic diamines were used as diamine bridges: 4,4'-diaminostilbene-2,2'-disulphonic acid (D), 4,4'-diamino-

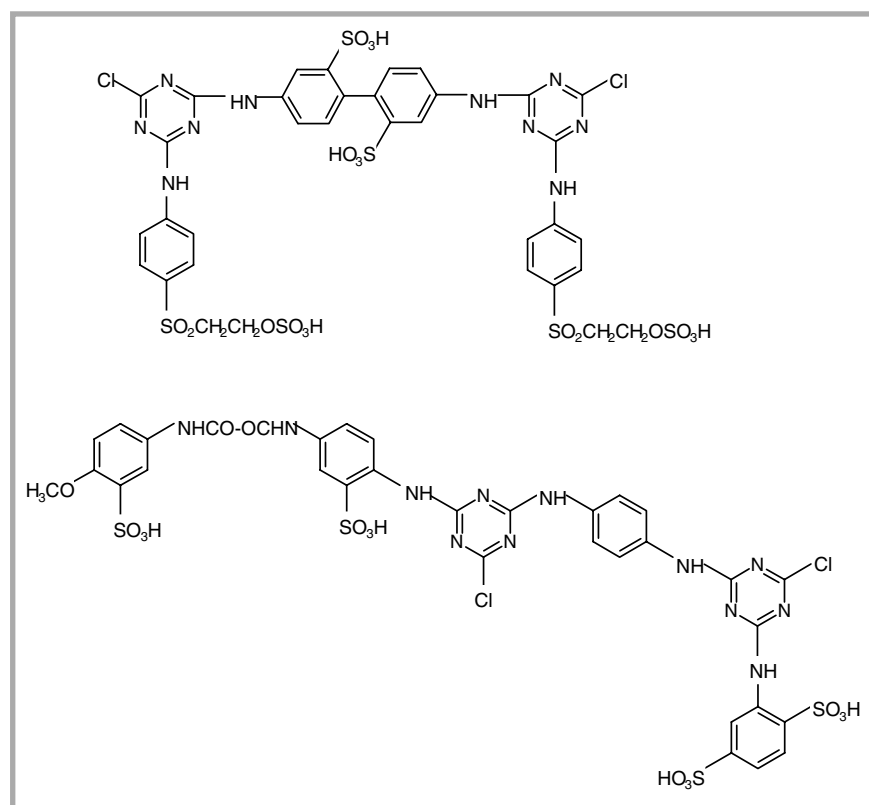


Figure 1. Examples of fibre-reactive, water-soluble colourless UV-absorbers used for increasing the protective properties of cotton fabrics.

3,3'-disulpho-diphenylurea (D1) and 2-methoxy-1,3,5-triazynyl-4,6-bis-N1,N1'-(1,4-diaminophenyl-3-sulphonic) acid (D2) (**Figure 3**). The use of 4,4'-diaminostilbene-2,2'-disulphonic acid in the preparation of UV-absorber derivatives of *s*-triazine was earlier reported [6, 7, 15]. The two latter diamines synthesised in our laboratory were used as intermediates in the preparation of colourless bis-monochlorotriazine cellulose-reactive UV absorbers of a similar type [16, 17].

Several UV-absorbers based on diamines D1 and D2 prepared during our previous work [16], such as A1 and A8 presented in **Figure 4**, were characterised by good light stability, whose UV-protection properties increased after their application in cotton fabrics. These products may be classified in the group of light stabilisers without hydrogen bridges, which owe their properties to high molar absorption in the 300-400 nm range [18].

It might be expected that introducing the same diamine bridges to the dye structure would increase its UV-absorption in the desired range. Due to the high level of wash-fastness caused by the fact that a dye molecule is covalently bonded to the fibre, it might also be expected that the UV-protection properties of the dyed fabric should persist for an extended period.

The application and protective properties of the newly synthesised yellow and red dyes were compared with a model yellow dye Y1 and model red dye R1 (C.I. Reactive Red 120), in which, according to industrial practice, simple derivatives of phenylenediamine were used as diamine bridges.

In order to estimate the possibility of decreasing the number of chemicals used in the dyehouse by using the newly synthesised dyes, additional application tests were carried out. In these tests, samples of the same textile fabric were dyed with a composition of a model reactive dye and fibre-reactive UV-absorber auxiliary. For this purpose, two colourless fibre-reactive UV-absorbers synthesised during our earlier work [16], A1 and A8 (presented in **Figure 4**), were used. A model reactive yellow (Y1) and reactive red (R1) were used as the reference dyes.

Experimental

Characterisation of products

Mass spectra of the prepared dyes were taken in a dihydroxybenzoic acid matrix with a Voyager Elite spectrometer using

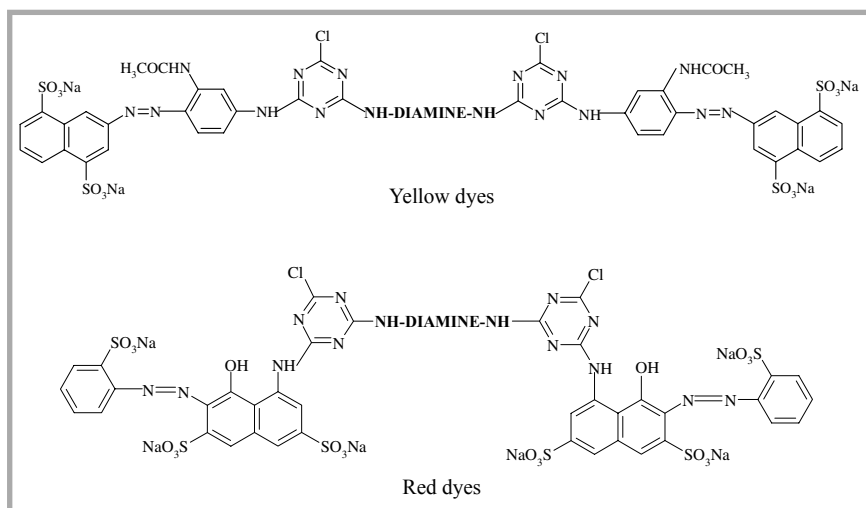


Figure 2. General structure of the homobifunctional monochlorotriazine reactive dyes synthesised in present work.

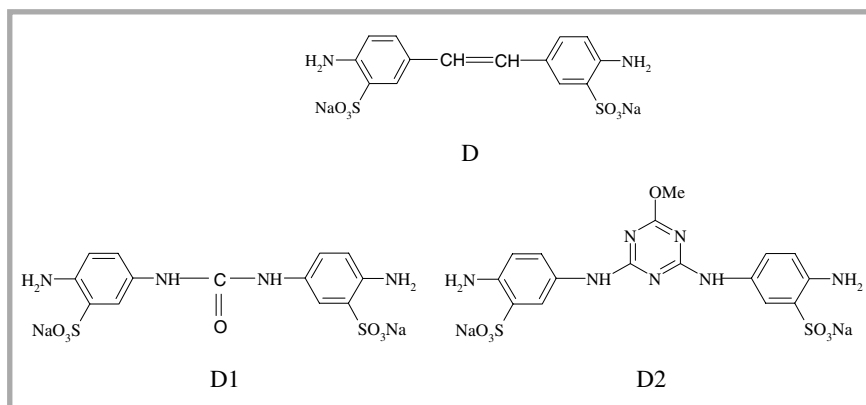


Figure 3. Structure of diamine bridges used.

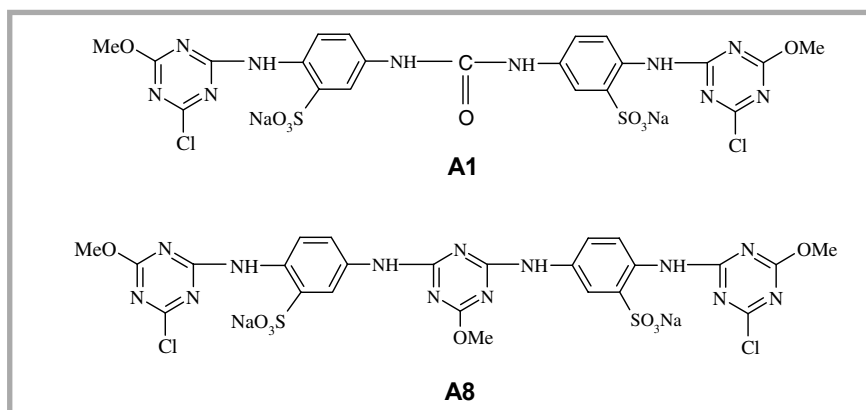


Figure 4. Structure of colourless reactive UV-absorbers used in the present work.

the MALDI (matrix assisted laser desorption ionisation) technique. Spectrophotometric measurements were taken on a Perkin Elmer Lambda 40 spectrophotometer in the UV-VIS range. The measurements of UV transmittance through the fabric samples were carried out with a Beckman DK-2A double-beam spectrophotometer equipped with an Ulbricht sphere and powered by a hydrogen lamp.

HPLC analysis was performed using a DAD 200 instrument with a Zorbex SB-Phenyl column, eluent 50% phosphate buffer pH = 6.9 (0.575g $\text{NH}_4\text{H}_2\text{PO}_4$ + 0.7 g Na_2HPO_4 in 900 ml water and 100 ml methanol mixture) + 50% methanol.

Synthesis of the reactive dyes

Synthesis of the required intermediates was carried out as previously de-

scribed: diamine D2 [17], diamine D1 [19], absorbers A1 and A8 [16]. All the other intermediates used, including 4,4'-diaminostilbene-2,2'-disulphonic acid (D), were of technical grade, obtained by courtesy of Boruta S.A. Dyestuff Industry Works, Zgierz (Poland).

The bi-functional reactive yellow dyes Y1-Y4 (**Figure 2**) were synthesised by coupling diazotised 2-aminonaphthalene-4,8-disulphonic acid ('C-acid') with 3-aminoacetanilide (temperature 10-12 °C, pH = 3.5-4.0 from NaHCO₃). After completing the process, the reaction mixture was alkalisied to pH = 8.0-8.2 with sodium carbonate, and the monoazo dye was separated by salting out with sodium chloride. After filtration, the dye was dispersed in water and condensed with cyanuric chloride (3-5 °C, pH = 6.5-7.0), and after that with appropriate amounts of diamine (30-40 °C, pH = 6.5-7.0). Both condensation processes were controlled by TLC chromatography (Merck Kiesegel 60 F₂₅₄ plates, ethyl acetate, n-propanol, water in the ratio of 1:6:2 as the eluent; e.g. for dye Y4; TLC data – dichlorotriazine dye R_f = 0.46, monochlorotriazine dye R_f = 0.05). HPLC analysis of dye Y4 gave one single peak with a retention time of 6.55 min, for 87.8% of the area. The structure of the final products was also confirmed by MALDI mass spectroscopy in negative mode. For the same Y4 dye, molecular ions [M-COCH₃ + H]⁻ m/z = 1644, [M-2COCH₃-Na + H]⁻ m/z = 1579, [M - C acid residue + 2H]⁻ m/z = 1345 were detected, among others.

Reactive red dyes (R2-R4) and derivatives of C.I. Reactive Red 120 (R1) were prepared by the usual methods, i.e. condensation of cyanuric chloride with 1-amino-8-hydroxynaphthalene-3,6-disulphonic acid ('H-acid') in an aqueous medium at pH = 1.2-1.5 at a temperature of 0-5 °C followed by coupling with diazotised orthanilic acid (5-10 °C, pH = 5-6 from trisodium phosphate). Dichlorotriazine dye was then condensed with an appropriate diamine at 30-40 °C and pH = 6.5 ± 02 (10% NaOH). The process was controlled by TLC chromatography (Merck Kiesegel 60 F₂₅₄ plates, ethyl acetate, n-propanol, water in the ratio of 1:6:2 as the eluent; e.g. for dye R3 TLC data – dichlorotriazine dye R_f = 0.32, monochlorotriazine dye R_f = 0.12). The dye was separated from the solution by salting out with the use of 7% sodium chloride and 9% potassium chloride (by volume), then

filtered and buffered to a pH of 7.0 ± 0.2, and finally dried. The main product area determined (as in the above example) by HPLC analysis was 90.3%. The structure of this dye, which was a mixture of sodium and potassium salts from the parent product, was also confirmed by mass spectroscopy in negative mode. Molecular ions such as pentasodium, tripotassium salt (M1) m/z = 1938 and trisodium pentapotassium salt (M2) m/z 1974 and [M1-Na+H]⁻ m/z = 1914, [M1-2Na+2H]⁻ m/z = 1894, [M1-2K-2Na-2H]⁻ m/z = 1812, [M2-3Na+H]⁻ m/z = 1902, [M2-2K+2H]⁻ m/z = 1894 were detected, among others.

■ Results and discussion

Application tests

Application tests of the cotton fabrics were carried out on a Rotec (Roaches) laboratory dyeing machine using a process typical for monochlorotriazine reactive dyes. Bleached cotton with a linen weave, with a weight of 104 g/m² (Fabric 1), and bleached cotton satin, with a weight of 119 g/m² (Fabric 2), were used. Dyeing was carried out for 70 minutes at a temperature of 85 °C (2 g fabric samples, 1:10 fabric-bath ratio, 50 g/dm³ of crystalline sodium sulphate and 20 g/dm³ of sodium carbonate). Model yellow Y1 and red R1 dyes were used at concentrations of 1% of the weight of fabric. The amounts of the R2-R4 and Y2-Y4 dyes used were calculated on the basis of their spectrophotometric measurements in order to receive the same dyebath strengths as in the case of the model dyes. The percentage degrees of the dyes and UV-absorber exhaustion were calculated from spectrophotometric measurements according to the following formula:

$$E = \left(1 - \frac{D_2}{D_1}\right) \times 100\%$$

where: D₁ – absorption of the dyebath at λ_{max} before dyeing, D₂ – absorption of the dye bath at λ_{max} after dyeing

The dye fixation of the fibre was measured with the use of two samples of dyed fabric of the same weight. One of them was subjected to repeated hot extraction with pyridine-water azeotrope in order to strip away any unfixed dye. After rinsing in water and drying, the sample was dissembled into single threads and dissolved in cold (0-5 °C) concentrated sulphuric acid. After pouring this solution on a water-ice mixture and diluting it with distilled water to a constant volume,

the concentration of dye was measured spectrophotometrically. The same procedure was repeated with the sample which had not been extracted with the water-pyridine system. The percentage degree of fixation was then calculated from the following formula:

$$F = \frac{C_2}{C_1} \times 100\%$$

where: C₁ – absorption of the dissolved sample with pyridine-water azeotrope without extraction, C₂ – absorption of the dissolved sample extracted with pyridine-water azeotrope.

The total dyeing process efficiency was calculated from the following formula:

$$T = \frac{E \times F}{100} \%$$

However, the above-described method was not applicable in measurements of the UV-absorber fixation of the fibre due to intense absorption of the dissolved fabric samples in the near UV-region.

Structural data of the prepared dyes, accompanied by their application properties for Fabric 2, and UPF values for both dyed fabric samples are listed in **Table 1**.

It can be noted that all the examined colorants exhaust well in the cotton fabric used, and the substitution of typical intermediates used in industrial practice, such as *p*-phenylenediamine or *o*-phenylenediamine, with diamines of a more extended structure results in practically no changes in the behaviour of dye exhaustion. In the case of yellow dyes, a slight increase in the exhaustion values can even be observed. All the dyes gave very similar levels of fixation and dyeing efficiency when compared with those of the model dyes, although in the case of red dyes these values are somewhat lower than might have been expected. The same observations apply to the colour yield of the dyeings, where only a slight decrease in this parameter can be noted in the case of the yellow dyes Y2 and Y3. Practically the same application results were obtained while dyeing Fabric 1.

Measurements of the protective properties of the dyed fabrics

All the textiles used were examined for their protective properties by *in vitro* measurements of their UPF value, according to the method laid down in the appropriate European Standard [20]. The

Table 1. Structure of synthesised dyes, their application properties for cotton and measured UPF values (1% dyeings owf).

Dye	Diamine Bridge	Fabric 2				UPF	
		Exhaustion, %	Fixation, %	Dyeing efficiency, %	K/S ³	Fabric 1	Fabric 2
Y1 (R. Yellow)	o-PDA ¹	85.3	74.5	63.5	4.53	10.9	36.6
Y2	D	91.6	72.3	66.2	3.55	9.9	50.9
Y3	D2	91.1	74.5	67.8	3.81	14.7	69.6
Y4	D1	93.7	76.0	71.2	4.90	12.3	96.6
R1 (R. Red 120)	p-PDA ¹	84.8	75.4	64.0	5.53	13.7	56.7
R2	D	89.7	76.5	68.6	5.10	11.9	70.9
R3	D2	83.9	74.2	62.2	5.86	15.8	55.5
R4	D1	86.0	78.0	67.1	5.80	11.5	70.7
A1	D1	83.0	–	–	81.6 ²	12.8	41.8
A8	D2	85.2	–	–	76.8 ²	14.0	42.4

¹ o-PDA – o-phenylenediamine, p-PDA – p-phenylenediamine,

² whiteness, measured on a Gretag Macbeth SpectroEye spectrophotometer with illuminate D₆₅ and 10° additional normal observer,

³ at λ_{max} of prepared dyes, – yellow dyes 410 nm, – red dyes 540 nm.

UPF factor was calculated as follows:

$$UPF = \frac{\sum_{\lambda=290}^{\lambda=400} E(\lambda)\epsilon(\lambda)\Delta\lambda}{\sum_{\lambda=290}^{\lambda=400} E(\lambda)T_i(\lambda)\epsilon(\lambda)\Delta\lambda}$$

where E(λ) is the solar spectral irradiance [W/m²nm], ε(λ) the erythema action spectrum, T_i(λ) the spectral transmittance of sample i at the wavelength λ, and Δλ is the interval of the wavelength in nm. A mean value was taken from three measurements for UPF calculation. The results of these measurements are given in **Table 1**.

It was already known [4] that the structural characteristics of a particular textile garment (thickness, porosity etc) are the main factors determining its UV-protecting properties, can once again be observed in the results presented. As expected, both of the textiles used exhibited improved UV-protecting properties after coloration, compared with the blank fabric samples. The introduction of special UV-absorbing residue to the dye mol-

ecule usually results in an increase in the measured UPF value. This phenomenon was especially noticeable in the case of Fabric 2, which was less translucent to UV radiation. After dyeing with the yellow dyes Y2-Y4, this fabric was characterised by UPF values above 40, which according to Standard [20] ensure sufficient UV-protection. The highest blocking of ultraviolet radiation by the fabric was observed in the case of dyes Y3 and Y4. It is worth noting that despite the lower colour yield of the resulting dyeing, dye Y3 gives a much higher UPF value than the model dye Y1.

Fabric 2, coloured with all the examined red dyes, exhibited sufficient protective properties (UPF > 40). This was probably partially caused by the different chromophoric system of these dyes. However, the use of new diamine bridges in the case of dyes R2 and R4 gave an increase of about 20% in the UPF values, compared with those of the model C.I. Reactive Red

120; this might be important when dyeing at lower colorant concentrations.

At present no data is available concerning how the protective properties of the dyed fabrics discussed above would change after repeated laundering. It may be expected that with the high level of wash-fastness specific to reactive dyes, the UV protection properties of dyed fabric would persist for an extended period. The assumption is the good light-resistance of UV-absorbers based on diamines used in this research and determined in the earlier work [16]. It would, however, require separate research, during which other factors such as the effect on UPF measurements of fabric shrinkage, the influence of detergents or optical brighteners present in the washing medium would also have to be considered.

The protective properties of the new dyes were compared with results obtained using traditional methods, in which samples of cotton fabrics were dyed with compositions of model dyes (Y1 and R1) and the colourless fibre-reactive UV absorbers A1 and A8, shown in **Figure 4**. As previously, 1% model dye concentrations per weight of the fabric were used, accompanied by 0.25%, 0.5% and 1% wt concentrations of the UV absorber applied. The UPF values of dyed fabrics are graphically presented in **Figures 5 and 6**.

As expected, the simultaneous application of the dye and UV-absorber auxiliary in the same dyeing bath increased the protective properties of the coloured textiles in comparison with the single use of the model dye. Nevertheless, for the more UV-transparent Fabric 1, the UPF values measured are still below the desired level. While dyeing Fabric 2, it was noted that the measured UPF values were

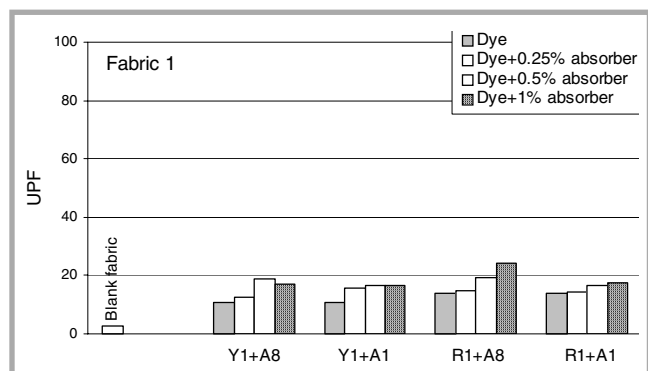


Figure 5. Protection properties of cotton (Fabric 1) coloured with model dyes with the addition of colourless UV-absorbers. (1% dyeings, UPF rating over 40 – adequate protection properties).

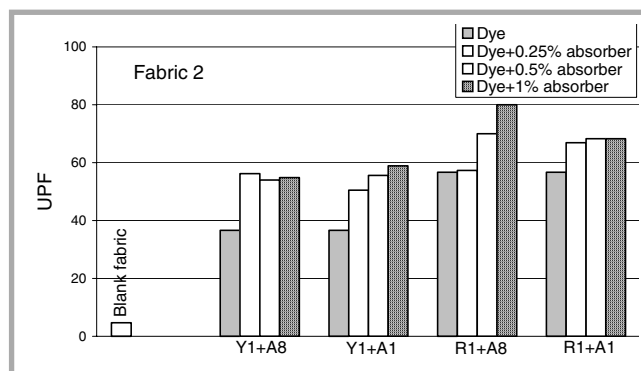


Figure 6. Protection properties of cotton (Fabric 2) coloured with model dyes with the addition of colourless UV-absorbers. (1% dyeings, UPF rating: over 40 – adequate protection properties)

influenced by both the nature of the UV-absorber auxiliary used and the structure of the model dye. The competition between dye and absorber during the sorption process in the cellulose fibre might be the cause of the different behaviour of the absorbers A1 and A8 when applied with yellow and red model dyes. In the case of the yellow dye Y1, the use of a 0.25-0.5% UV-absorber auxiliary per weight of the fabric was necessary to obtain a product characterised by adequate UV-protecting properties.

Conclusions

From the analysis of the results described above, it may be concluded that introducing appropriate residues to the molecule of a reactive dye can increase its UV-protection properties. The coloration of cellulose fabrics with the use of specially tailored dyes could eliminate the necessity of using additional UV-absorber auxiliaries, and therefore decrease the number of chemicals used in the dyehouse. This fact should be considered when designing new reactive dyes; although introducing UV-absorbing residues into dyes characterised by a different structure than that presented in **Figure 1**, would probably require more pronounced efforts. In the case of currently manufactured dyes, the 'colorant + UV-absorber auxiliary' method might still be the most convenient way of improving the UV-protection properties of dyed textiles.

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Kaunas University of Technology (KTU) Kaunas, Lithuania



The roots of the university go back to 1920 when the first centre of university-type higher courses was established. In 1922 the Government of Lithuania accepted a resolution establishing Kaunas University, which was then given the name of Vytautas the Magnus in 1930. In 1950 Kaunas University was reorganized into Kaunas Polytechnic Institute (KPI) and Kaunas Medical Institute. In 1990 KPI changed its name to the present name of Kaunas University of Technology (KTU). Now KTU is the largest technological University not only in Lithuania, but also in all the Baltic States. Many Prime Ministers, Ministers, Members of the Lithuanian Parliament and even the President of the Republic of Lithuania have graduated from KTU.

The University is the only one in Lithuania engaged in textile engineering. The Department of Textile Technology dates back to 1929, when the decision to begin education in textile engineering at the University was accepted, and in 1932 the first lectures started. The Department of Textile Technologies, as an organisational unit, was established in 1940, due to the founding of a Laboratory of Fibre Technology in 1936. The creator and first Head of the Department was Professor J. Indriunas. Up to now more than 2500 students have graduated in textile technology (including more than 200 from Latvia and Estonia) and more than 80 postgraduate students have been awarded a Ph. D. degree. The majority of textile engineers of the Lithuanian textile industry graduated from this department. Senior Professors of the Department, such as A. Matukonis, V. Milašius, and A. Vitkauskas are well known not only in Lithuania, but also in Poland and the whole of Europe.

Today the Department's staff includes 7 professors, 4 associate professors, 7 lecturers and 12 doctoral students. Up to now more than 2000 scientific articles have been published in Lithuanian and international journals as well as in conference proceedings. The main fields of research activity carried out by the Department of Textile Technology are as follows:

- *the rheological properties of textiles,*
- *the flammability and heat transfer of textiles,*
- *the wettability of textiles,*
- *computerised structural design,*
- *the development of textile manufacturing technologies, and*
- *the manufacturing of nanofibres, among others.*

The department is a member of the international Association of Universities for Textiles (AUTEX). The Professors of the Department have given lectures at various European universities regarding the activities of AUTEX and the European Masters Studies Programme in Textile Engineering (E-TEAM).

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