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Investigation into the Effect of a Plant Derived Stabiliser on the Light and Wash Fastness of Sulphur Dyed Cotton and Nylon Fabrics

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Abstract

In this study both cotton fabric and nylon fabric were dyed with a range of commercial Sulphur dyes and the light and wash fastness of the coloured fabrics investigated. The effect of aftertreating the coloured cotton and nylon fabrics with a tannin-based commercial product, Bayprotect Cl, in the presence or absence of sodium sulphate in the treatment bath, was found to significantly improve the light fastness of the Sulphur dyed cotton and the photoprotective effect was partially stable to ISO 105 CO6 washing. In addition the tannin aftertreatment also improved the colour stability of the dyed fabrics to the perborate-based ISO 105 CO6 washing. The possible mechanisms for the improved fastness properties are also discussed.

The application of sulphur dyes to nylon is potentially commercially useful but has been limited because of the reported poor light fastness of the dyeings. The photoprotective effect of the tannin-based aftertreatment was investigated with a view to providing the necessary commercial performance. However, it was established that on this fibre the light fastness improvement was marginal and the associated wash fastness to oxidative bleach-based ISO 105 CO6 washing limited.

Introduction

Sulphur dyes are a major class of textile dyes that are typically applied to cellulosic textiles (Nguyen and Juang 2013; Shore 1995). A key commercial feature of sulphur dyes is their moderate cost which makes their application to cellulosic textile materials attractive for dark shades. In addition they possess moderate to good wash and light fastness although cellulosic fabrics dyed with sulphur dyes are susceptible to percarbonate or perborate-based washing powders where the oxidative bleaching increases the wash fading of the dyed goods. The effect of laundering with an oxidative bleaching agent (sodium perborate tetrahydrate) and a bleach activator (TAED) on the wash fastness of C. I. Leuco Sulphur Black 1 dyed cotton fabric has been studied previously and it was concluded that the activated perborate bleaching cleaved the dye disulphide bonds causing surface oxidation, surface dye loss and fabric colour change (Soliman et al 2013).

The wet fastness of sulphur dyes has been reported to be improved through the application of alkylating agents (Burkinshaw and Collins 1995; Cook 1982; Preston 1986; Wood 1976), quaternary ammonium alkyl compounds (Cook 1982; Heid et al. 1973), dicyandiamide-formaldehyde condensates (Burkinshaw and Collins 1995) and crease resist finishing agents (Burkinshaw and Collins 1997; Preston 1986; Venkataraman 1974). In addition the effect of treatments with several cationic fixing agents have also been assessed and found to be beneficial (Burkinshaw et 1997; Burkinshaw and Collins 1995; Burkinshaw and Collins 1996; Burkinshaw and Collins 1997; Burkinshaw and Collins 1998). More recently, the application of lanthanum triacetate to sulphur dyed cotton has also been explored and found to substantially reduce the oxidative bleaching of sulphur dyed textiles after repeated launderings (Zhou and Yang 2010).

Burkinshaw has also reported on the potential use of sulphur dyes on nylon with a view to achieving deep shades with good wash fastness and extending the commercial applicability of sulphur dyes to fibres other than cellulosics (Burkinshaw et al. 2000; Burkinshaw et al. 2003a; Burkinshaw et al. 2003b; Burkinshaw et al. 2006). However their commercial application was limited by the relatively low light fastness of the sulphur dyed nylon and this extension of sulphur dye usage has not been pursued further.

Natural antioxidants can be divided into the enzymatic and non-enzymatic antioxidants (Rahman 2007; Kirschweng et al 2017). Enzymatic antioxidants, such as catalase, superoxide dismutase, glutathione reductase or glucose-6-phosphate dehydrogenase, either catalyze directly the decomposition of reactive oxygen species to harmless compounds (Mates et al 1999) or regenerate non-enzymatic antioxidants (Gamble and Burke 1984). In contrast non-enzymatic antioxidants can remove pro-oxidative transition metal contaminants, scavenge alkoxy and peroxy radicals, or quench singlet oxygen species (Martysiak-Zurowska and Wenta 2012).

Within the non-enzymatic antioxidant class are the tannins which consist of the hydrolysable tannins, condensed tannins and the phlorotannins (Oroian and Escriche 2015; Prabhu and Bhute 2012; Ramakrishnan et al. 2006). Hydrolysable tannins are esters of gallic acid (gallotannins), ellagic acid (ellagitannins) or other phenolic acids and a polyol, such as D-glucose, fructose or xylose (Ky et al. 2016; Pizzi 2008); they have a molecular weight range from 500 to 5000 Daltons (Arapitsas 2012). Hydrolysable tannins usually occur in leaves, galls and wood bark, while the condensed tannins can be obtained from the skin, seed or stems of grapes (Ky et al. 2016). The oligomeric condensed tannins, or proanthocyanidins, are flavonoid-based derivatives with a molecular weight up to 30,000 Daltons (Oroian and Escriche 2015).

Bayprotect Cl is an anionic tannin-based protective stain-release agent for nylon 6,6 and nylon 6 and acts as an antioxidant, providing protection against cleaning agents, chlorine-based bleaches, in particular hypochlorite, and other oxidative substances such as benzoyl peroxide (Tanatex Chemicals product information, 2018). In this study the effect of this plant derived tannin-based protective agent on the fastness of sulphur dyed cotton and dyed nylon is investigated with a view to demonstrating useful beneficial improvements in light and wash fastness performance.

Experimental

Materials

A plain woven, bleached, 100% cotton fabric, 135g/m², was used throughout this study and was supplied by Phoenix Calico Limited, UK. The scoured 100% nylon 6,6 fabric, 159g/m², used throughout this study was supplied by Phoenix Calico Limited, UK. The dyes used were Diresul Black RDT-LS (C. I. Sulphur Black 1), Diresul Blue RDT-G (C. I. Sulphur

Blue 7), Diresul Green RDT-N (C. I. Sulphur Green 2), Diresul Bordeaux RDT-6R (C. I. Sulphur Red 10) and Diresul Olive RDT-B (C. I. Sulphur Green 16).

The reducing agents used were sodium sulphide or sodium hydrosulphite (Laboratory grade, Aldrich Chemicals Ltd, UK.). The oxidation bath comprised of hydrogen peroxide and sodium bicarbonate (Laboratory grade, Aldrich Chemicals Ltd, UK.). The fabric was soaped with a non-ionic detergent, Hostapal NIN (Clariant International Ltd.). The ECE non-phosphate detergent, ECE phosphate detergent and tetraacetylethylenediamine (92% active) were purchased from the Society of Dyers and Colourists (SDC), Bradford, UK. Sodium perborate tetrahydrate (97% active, Laboratory grade) was purchased from Aldrich Chemicals Ltd., UK.

Dyeings

All dyeings were carried out in sealed stainless steel dye pots of 1000 cm³ capacity, housed in a Mathis Laboratory dyeing machine. The dyeing of cotton fabric with sodium sulphide was carried out with 4% o.m.f. (on mass of fabric) dye, 5 g/L sodium sulphide, 5 g/L sodium carbonate and 15 g/L sodium chloride, with liquor to goods ratio of 10:1.

The fabric was first heated to a temperature of 40-50°C in the presence of half of the quantities of water, sodium sulphide and soda ash. The liquor was kept at this temperature for 5 minutes, then the dyestuff and balance of water, sodium sulphide and soda ash were added. The dyebath was further kept for 10 minutes at the same temperature and then the sodium chloride was added. The temperature was then increased to 90°C at a gradient of 2°C/min and kept at 90°C for 30 minutes. The solution was then cooled to 50°C at a gradient of 5°C/min.

The dyed fabrics were then washed thoroughly with cold water to remove any unfixed surface dye. The dyed fabric was then oxidised with 5 g/L hydrogen peroxide and 1 g/L soda ash at 40-45°C for 15 minutes. The fabric was then finally soaped with 1g/L of non-ionic detergent at the boil for 20 minutes and then rinsed with cold and warm water. The liquor to goods ratio employed for oxidation and soaping was 10:1.

Nylon 6,6 fabric was dyed in dyebaths containing 4% o.m.f. sulphur dye and 1 g/L sodium hydrosulphite at pH 7 and a liquor to goods ratio of 20:1. The fabrics were introduced into the dyebaths heated to 50°C and the dyebaths were raised to 98°C at 2°C/min, maintained at this

temperature for 45 minutes, and finally cooled to 40°C at a gradient of 5°C/min at which point the dyed fabrics were removed, rinsed with cold water and squeezed to remove any remaining water. The dyed fabrics were added to an aqueous solution of Diresul Entwickler BRI.ES (a stabilised oxidising agent for sulphur dyes) (5g/L) at pH 4.5 and heated at 60°C for 15 minutes; a liquor to goods ratio of 50:1 being employed. The dyed fabrics were finally removed, rinsed thoroughly with cold water and then dried at room temperature.

Aftertreatment with Bayprotect Cl

Aqueous treatment baths containing either 2, 4, 6, 8 or 10% o.m.f. Bayprotect Cl were prepared and adjusted to pH 3 with 10% w/v citric acid solution; the liquor to goods ratio of the treatment bath being adjusted to a ratio of 10:1. Cotton and nylon 6,6 fabrics, pre-dyed with the specified sulphur dye (4% o.m.f.), were added to the treatment baths at 20°C and the temperature of the bath then raised to 98°C at 2°C/minute. After 20 minutes at 98°C the treated fabrics were removed, rinsed with warm water, then with cold water, and finally allowed to dry at room temperature.

The process was repeated, but with either 20, 50 or 100g/L sodium sulphate added to the Bayprotect Cl treatment bath.

Colour Measurement

The colour of the dyed and aftertreated fabrics were measured using a Datacolor Spectroflash 600 spectrophotometer, with a 10° standard observer and D65 illuminant, and were the average of four measurements. The colour strength (K/S) was determined using the Kubelka-Munk equation, Scheme 1.

$$\left(\frac{K}{S}\right)_{\lambda} = \frac{(1-R_{\lambda})^2}{2R_{\lambda}}$$
 Scheme 1

Where K is the absorption coefficient, S is the scattering coefficient, R is the reflectance expressed as a fractional value at wavelength of maximum absorption λ . The colour difference, ΔE was calculated and presented as an indicator of visual colour change.

Determination of Fastness Properties

The colour fastness of the untreated and aftertreated sulphur dyed fabrics were tested according to ISO standard methods, including fastness to washing (ISO 105 CO6/C2S and ISO 105 CO9) and light (ISO 105 B02).

Results and Discussion

The effect of light exposure on all the sulphur dyed cotton fabrics was to produce colour fading, with the light stability of the coloured fabrics being dye dependent, Table 1. Treatment of the sulphur dyed fabrics with Bayprotect Cl resulted in an increase in the light stability, typically an improvement of 1.0 - 1.5, but again the effect was dependent on the dye and the tannin application level.

Dyed Cotton	Exposure time				Colour Difference, ∆ E	
+ Bayprotect Cl	16 Hrs	32 Hrs	16 Hrs*	32 Hrs*	No Salt ¹	Salt ²
Sulphur Red 10 Control	4	3	4	3	4.6+	1.1+
+ 4% o.m.f. BCl	4-5	3-4	4-5	3-4	6.7	4.1
+ 6% o.m.f. BCl	4-5	4	4-5	4	2.6	2.4
Sulphur Blue 7 Control	3	2	3	2	5.3+	1.0+
+ 4% o.m.f. BCl	3-4	2-3	3-4	2-3	5.4	2.9
+ 6% o.m.f. BCl	3-4	2-3	3-4	2-3	6.3	2.3
+ 10% o.m.f. BCl	4-5	3-4	4-5	3-4	6.6	2.1
Sulphur Black 1 Control	4-5	3-4	4-5	3-4	1.5+	1.3+
+ 6% o.m.f. BCl	4-5	4	4-5	4	0.7	0.5
+ 10% o.m.f. BCl	4-5	4-5	4-5	4-5	0.8	0.8
Sulphur Green 2 Control	3-4	2-3	3-4	2-3	3.9+	0.7+
+ 2% o.m.f. BCl	4-5	4	4-5	4	3.3	4.1
+ 10% o.m.f. BCl	4-5	4	4-5	4	1.5	2.0
Sulphur Green 16 Control	4	3	4	3	4.4+	1.5+
+ 2% o.m.f. BCl	4-5	4	4-5	4	4.3	4.3
+ 4% o.m.f. BCl	4-5	4-5	4-5	4-5	3.5	3.0

Table 1 Light-fastness of sulphur dyed cotton fabrics (4% o.m.f.) aftertreated with Bayprotect Cl (BCl), with and without sodium sulphate addition.

* 20g/L sodium sulphate incorporated with Bayprotect Cl addition.

⁺ No Bayprotect Cl addition.

¹ Colour difference between untreated dyed fabrics and tannin aftertreated dyed fabrics without sodium sulphate present. Fabrics not exposed to simulated sunlight.

² Colour difference between untreated dyed fabrics and tannin aftertreated dyed fabrics with sodium sulphate present. Fabrics not exposed to simulated sunlight.

In a previous study examining the light fastness of cotton fabrics dyed with plant derived dyes and aftertreated with a range of photostabilisers it was found that gallic acid and vitamin C were the most effective of the plant derived antioxidants (Cristea and Vilarem 2006). It has been proposed that vegetable polyphenols are radical trapping antioxidants while ascorbic/citric acid function as metal chelating antioxidants (Cristea and Vilarem 2006). Therefore it is probable that the Bayprotect Cl tannin used in this study produces the observed improved light fastness in the sulphur dyes by trapping the free radicals produced by UV radiation. Tannins have also been reported to improve the light fastness of other natural dyed cotton fabrics (Hong 2018).

Dyed Cotton	Exposure time				
+ Bayprotect Cl	16 Hrs	32 Hrs	16 Hrs*	32 Hrs*	
Sulphur Red 10 Control	4	3-4	3-4	3	
+ 4% o.m.f. BCl	4-5	4	4	3-4	
+ 6% o.m.f. BCl	4-5	4	4	3-4	
+ 10% o.m.f. BCl	5	4-5	4-5	4	
Sulphur Blue 7 Control	3-4	3	3-4	3	
+ 4% o.m.f. BCl	4-5	3-4	3-4	3	
+ 6% o.m.f. BCl	4-5	4	3-4	3	
+ 10% o.m.f. BCl	4-5	4	4	3-4	
Sulphur Black 1 Control	4-5	4	4-5	4	
+ 6% o.m.f. BCl	4-5	4-5	4-5	4-5	
+ 10% o.m.f. BCl	5	4-5	5	4-5	
Sulphur Green 2 Control	3-4	3	3-4	3	
+ 2% o.m.f. BCl	4	3-4	3-4	3	
+ 10% o.m.f. BCl	4-5	4	3-4	3	
Sulphur Green 16 Control	2-3	2-3	3	2-3	
+ 2% o.m.f. BCl	3-4	3	3	2-3	
+ 4% o.m.f. BCl	3-4	3	3-4	3	
+ 10% o.m.f. BCl	4	3-4	4	3-4	

Table 2 Light-fastness of the ISO 105-C06 pre-washed, sulphur dyed (4% o.m.f.) cotton fabrics aftertreated with Bayprotect Cl (BCl), with and without sodium sulphate addition.

* 20g/L sodium sulphate incorporated with Bayprotect Cl addition.

Following the application of the Bayprotect Cl to the dyed cotton substrates it was observed that there was some loss of colour from the sulphur dyed fabrics, Table 1. Therefore in order to minimise the loss of dye from the cotton the effect of incorporating salt into the tannin aftertreatment liquor was evaluated and it was found that in general the addition of sodium sulphate does indeed partially reduce the colour loss, Table 1. In addition the incorporation of the salt into the tannin aftertreatment formulation does not adversely affect the observed beneficial improvement in light fastness.

Dyed Cotton	Colour Change Rating			
+ Bayprotect Cl	No Salt Addition	Salt Addition*		
Sulphur Red 10 Control	3	2-3		
+ 2% o.m.f. BCl	3	3-4		
+ 4% o.m.f. BCl	3-4	3-4		
+ 10% o.m.f. BCl	3-4	3-4		
Sulphur Blue 7 Control	2-3	2-3		
+ 2% o.m.f. BCl	2-3	3-4		
+ 4% o.m.f. BCl	4	4		
Sulphur Black 1 Control	2	2		
+ 6% o.m.f. BCl	2-3	2		
+ 10% o.m.f. BCl	2-3	2-3		
Sulphur Green 2 Control	3	3		
+ 6% o.m.f. BCl	3-4	3		
+ 10% o.m.f. BCl	3	3		
Sulphur Green 16 Control	2-3	4		
+ 2% o.m.f. BCl	3	4		
+ 10% o.m.f. BCl	3	4		

Table 3. ISO 105-CO6 Wash-fastness of sulphur dyed cotton fabrics (4% o.m.f.) aftertreated with Bayprotect Cl (BCl), with and without sodium sulphate addition.

* 20g/L sodium sulphate incorporated with Bayprotect Cl addition.

A key performance criterion for any protective treatment for textiles is the wash fastness durability of the applied effect chemical. Therefore the Bayprotect Cl treated sulphur dyed fabrics were pre-washed under ISO 105-C06 wash fastness conditions and then subjected to simulated sunlight exposure. Table 2 shows that light exposure again resulted in colour fading of the fabrics but that the tannin treatment was still effective in reducing the colour fade. However the protective effect was reduced probably due to the partial loss of the photoprotective agent.

Dyed Cotton +	Exposure time				
Bayprotect Cl	16 Hours	32 Hours	16 Hours*	32 Hours*	
Sulphur Red 10 Control	4	3	3-4	2-3	
+ 2% o.m.f. BCl	4-5	3-4	5	4	
+ 5% o.m.f. BCl	4	3-4	4	3-4	
Sulphur Blue 7 Control	2	1-2	2-3	2	
+ 2% o.m.f. BCl	2-3	2	2	1-2	
+ 5% o.m.f. BCl	2-3	2	2-3	2	
+ 10% o.m.f. BCl	3	2-3	3	2-3	
Sulphur Black 1 Control	2	1-2	1-2	1-2	
+ 2% o.m.f. BCl	2	1-2	2-3	2	
+ 5% o.m.f. BCl	2	1-2	2	2	
+ 10% o.m.f. BCl	3-4	2	2-3	2	
Sulphur Green 2 Control	1	1	1-2	1	
+ 2% o.m.f. BCl	1-2	1	1-2	1	
+ 5% o.m.f. BCl	1-2	1	2	1-2	
+ 10% o.m.f. BCl	2	1-2	2	1-2	

Table 4 Light-fastness of sulphur dyed nylon fabrics (4% o.m.f.) aftertreated with Bayprotect Cl (BCl), with and without sodium sulphate addition.

* With 20g/L sodium sulphate incorporated with Bayprotect Cl addition.

The effect of the Bayprotect Cl on the wash fastness of the sulphur dyed cotton fabrics was variable, Table 3. For C. I. Sulphur Red 10 and C. I. Sulphur Blue 7 the benefits are clear, both without and with the salt present during application of the tannin reagent, but for the other sulphur dyes examined there is little improvement. The mechanism by which the wash fastness of sulphur dyed fabric is improved by the aftertreatment with a tannin may possibly be related to a number of factors. According to a study (Ontiveros-Ortega et al. 1998), the negative zeta potential value of cotton decreases with the treatment of the bleached cotton

fabric with tannic acid attributed to the H⁺ ion adsorption on the fabric surface. The adsorption of tannic acid probably has arisen due to electrostatic interaction and hydrogen bond formed between the polar carboxyl end groups of oxycellulose and phenolic hydroxyl groups of tannic acid. Similarly the polar sulphonate groups in the oxidised sulphur dyes will hydrogen bond to the hydroxyl functionalities on the cellulose as well as the tannin. Hence providing greater substantivity, reducing dye desorption and increasing colour fastness to laundering.

Dyed Cotton	Exposure time				
+ Bayprotect Cl	16 Hours	32 Hours	16 Hours*	32 Hours*	
Sulphur Red 10 Control	3	2-3	4	3	
+ 2% o.m.f. BCl	3	2-3	3-4	2-3	
+ 5% o.m.f. BCl	3	2-3	3-4	3	
Sulphur Blue 7 Control	2	1-2	2-3	1-2	
+ 2% o.m.f. BCl	2-3	1-2	2-3	2	
+ 10% o.m.f. BCl	2-3	2	2-3	2	
Sulphur Black 1 Control	2	1-2	2	1-2	
+ 2% o.m.f. BCl	2	2	2	1-2	
+ 10% o.m.f. BCl	2-3	2	3	2-3	
Sulphur Green 2 Control	1	1	1	1	
+ 2% o.m.f. BCl	1	1	1	1	
+ 5% o.m.f. BCl	1-2	1	1-2	1	
+ 10% o.m.f. BCl	1-2	1	1-2	1	

Table 5 Light-fastness ISO 105-C06 pre-washed, sulphur dyed (4% o.m.f.) nylon fabrics aftertreated with Bayprotect Cl (BCl), with and without sodium sulphate addition.

* With 20g/L sodium sulphate incorporated with Bayprotect Cl addition.

The effect of light exposure on the sulphur dyed nylon fabrics was to produce obvious colour fading and the light stability of the coloured fabric was dependent on the dye applied, Table 4. Similar to the work of Burkinshaw et al. we have found that the light fastness of sulphur dyes on nylon was relatively low (Burkinshaw et al. 2000; Burkinshaw et al. 2003a; Burkinshaw et al. 2003b; Burkinshaw et al. 2006). Aftertreatment of the sulphur dyed fabrics with the tannin reagent only resulted in a marginal improvement in the light stability with

some of the sulphur dyed nylon. Similarly for the dyed nylon fabrics after treated with the tannin in the presence of salt little improvement in the light fastness performance was observed, Table 4.

The effect of ISO 105-C06 pre-washing the Bayprotect Cl treated sulphur dyed nylon fabrics on the light fading of the sulphur dyes on exposure to simulated sunlight was to clearly reduce the effectiveness of the tannin treatment in reducing the colour fade, Table 5. Again the protective effect was reduced probably due to the partial loss of the photoprotective agent.

Dyed Cotton +	Colour Change Rating			
Bayprotect Cl	No Salt Addition	Salt Addition*		
Sulphur Red 10 Control	4-5	4-5		
+ 2% o.m.f. BCl	5	4-5		
+ 5% o.m.f. BCl	4	4-5		
+ 10% o.m.f. BCl	4-5	4-5		
Sulphur Blue 7 Control	3-4	3		
$\pm 2\%$ o m f BCl	3-4	3		
	3-4	3		
+ 5% o.m.t. BCl	3-4	3		
+ 10% o.m.f. BCl	4	3-4		
Sulphur Black 1 Control	4-5	4-5		
+ 2% o.m.f. BCl	4-5	4-5		
+ 5% o.m.f. BCl	4-5	4-5		
+ 10% o.m.f. BCl	4-5	4-5		
Sulphur Green 2 Control	4-5	4-5		
+ 2% o.m.f. BCl	4-5	4-5		
+ 5% o.m.f. BCl	4-5	4-5		
+ 10% o.m.f. BCl	4-5	4-5		

Table 6. ISO 105-CO6 Wash-fastness of sulphur dyed nylon fabrics (4% o.m.f.) aftertreated with Bayprotect Cl (BCl), with and without sodium sulphate addition.

* With 20g/L sodium sulphate incorporated with Bayprotect Cl addition.

The wash fastness of acid dyes on nylon can be improved by a back-tanning process in which the dyed fabric is first treated with tannic acid and then tartar emetic (Broadbent 2001). A more environmentally acceptable and cheaper treatment involves the use of polymeric Syntans (synthetic tanning agents) which reportedly function by complexing with the dye in the outer fibre surface so preventing migration and loss of the dye from the fibre during laundering (Broadbent 2001; Schindler and Hauser 2004). In this study the effect of the tannin application alone was minimal on the wash fastness, Table 6.

Conclusions

The effect of aftertreating the sulphur dyed cotton fabrics with a tannin-based product, Bayprotect Cl, in the presence or absence of sodium sulphate in the treatment bath, was to significantly improve the light fastness of the sulphur dyed cotton, with the photoprotective effect being partially stable to ISO 105 CO6 washing. The light protective effect imparted by the tannin-based reagent was probably due to scavenging of the degradative free radicals.

The tannin aftertreatment also improved the colour fastness of some of the sulphur dyed fabrics to perborate-based ISO 105 CO6 washing probably through complexation of the polyphenol of Bayprotect Cl to the sulphur dye.

The application of sulphur dyes to nylon has previously been proposed to be potentially commercially useful but has been little used due to the reported poor light fastness of the dyed nylon. Therefore the photoprotective effect of the tannin-based aftertreatment on sulphur dyed nylon was evaluated but unfortunately the light fastness improvement was marginal and the associated wash fastness to oxidative bleach-based ISO 105 CO6 washing was also limited.

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