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Surface chemical and colorimetric analysis of reactively dyed cellulosic fabric. The effect of ISO 105 CO9 laundering and the implications for waste cellulosic recycling

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ABSTRACT

Previous studies have established that the application of crosslinking dyes and easy care finishes to cotton can significantly reduce the dissolution of waste cotton in solvents, such as N-Methylmorpholine oxide, and limit the potential recycling of cellulosic materials through the Lyocell fibre regeneration process. In this investigation the surface chemical compositions of three reactive dyed Tencel fabrics were studied using X-ray Photoelectron Spectroscopy (XPS) and the presence of the dye at the fibre surface demonstrated. The effect of the ISO 105 C09 oxidative-bleach fading test on the azo and anthraquinone chromophoric species was established by both surface chemical and colorimetric analyses. At low dye application levels the C. I. Reactive Black 5 and C. I. Reactive Red 228 dyed fabrics (azo chromophore) exhibited obvious colour fade while the anthraquinone-based C. I. Reactive Blue 19 dyed fabric was resistant to colour fade. However it is apparent that although some of the covalently bound dye will be removed during “first life” usage, most of the reactive colorant will remain bound to the cotton and will therefore need to be stripped from the waste garments to produce a white cellulosic feedstock prior to reprocessing through Lyocell fibre regeneration. A sequential acid, alkali and peroxide treatment completely removed the azo-based C. I. Reactive Black 5 and C. I. Reactive Red 228 colorants from the dyed cotton, however, the anthraquinone-based C. I. Reactive Blue 19 was highly resistant to removal and will require alternative chemical processing to remove the colorant.

1. Introduction

Lyocell is a regenerated cellulose fibre, which has been developed in response to technical, environmental and economic challenges [1]. It is the first commercially solvent-spun cellulosic fibre which negates the necessity for the xanthate derivatisation step in viscose manufacturing, with the added benefit of the amine oxide solvent being recyclable [2].

Reactive dyes are reported to have a high affinity for Lyocell/Tencel and can form covalent bonds, particularly the polyfunctional dyestuffs, with the cellulosic substrate [3–5]. Reactive dye systems can be classified as mono-functional, bi-functional and tri-functional, with the extent of substrate fixation, hydrolysis and durability influenced by the nature of the reactive functionality and the application conditions [3–5]. With the multi-functional reactive dyes, there is also the opportunity for crosslinking reactions which can enhance the physical properties of the Lyocell fabric by reducing wet fibrillation, maintain the colour strength by more durable fixation and minimise diffuse reflectance at the fibre/fabric surface resulting from wet laundering [6–11].

The surface sensitive X-ray Photoelectron Spectroscopy (XPS) technique has been used to investigate the nature of the outer cellulosic fibre surface, characterising the surface functionalities and elemental composition [12–14]. In this study the application of three reactive dyestuffs was investigated in order to assess dye fixation in the fibre bulk and at the fibre surface and the build-up of dye within the outer 10 nm surface. In addition the dyed Tencel fabrics were washed under ISO 105 C09 test conditions with a view to establishing the durability of the surface bound dye to different washing regimes through the colour strength (K/S) and lightness

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(L^*) measurements, and exploring the dye fading mechanism. This diagnostic ISO 105 C09 oxidative-bleach fading test has now gained wide acceptance and can be used to predict the potential fading due to colorant destruction in domestic use [15–17].

Previous studies have established that the application of cross-linking dyes and easy care finishes to cotton can significantly reduce the dissolution of waste cotton in swelling solvents and accordingly can limit the potential recycling of cellulosic materials through the Lyocell fibre regeneration process [18–22]. Therefore it is vital in any commercial processing of waste cotton that colourless crosslinking easy care finishes and coloured dyestuffs are effectively removed prior to dissolution. This study investigated a method to 'strip' colourants from reactive dyed cotton in order to prepare the materials for new fibre regeneration. The relative efficiencies of sequential acid, alkali and oxidative hydrogen peroxide dye stripping treatments were assessed subjectively and quantified by determining the fabric colour strength (K/S) and lightness (L^*).

2. Experimental

2.1. Materials

Scoured plain woven Tencel fabric (150 g/m^2), generously donated by Lenzing, Austria, was further treated with deionised water twice at 90 °C for 20 min and then washed in acetone, rinsed in deionised water and finally air-dried prior to use in the laboratory. A bleached, mercerised, plain woven, 100% cotton fabric, (135 g/m^2) was supplied by Phoenix Calico Limited, Cheshire, United Kingdom and was used as received.

The ECE phosphate detergent and tetraacetylethylenediamine (92% active) were purchased from the Society of Dyers and Colourists (SDC) enterprise, Bradford, UK. Sodium perborate tetrahydrate (97%) active was purchased from Aldrich Chemicals Ltd., UK.

C. I. Reactive Blue 19 and C. I. Reactive Black 5 were kindly supplied by Dystar, Germany and C. I. Reactive Red 228 was kindly supplied by Ciba, UK. The dye structures are illustrated in Fig. 1.

3. Methods

3.1. Application of reactive dyes to tencel

The Tencel fabric (10 g) was dyed in a Mathis Labomat dyeing machine with 1%, 5% and 10% o.m.f. (on mass of fabric) of the specified dye following commercial application recommendations. Tencel fabrics were introduced into dyebaths containing 1%, 5% and 10% o.m.f. of the C. I. Reactive Blue 19 and C. I. Reactive Black 5, respectively, at 40 °C with a liquor to goods ratio of 15:1, and 50, 50 and 80 g/L sodium chloride, respectively, added over 15 min 5 g/L sodium carbonate was then added and the dyebath maintained at 40 °C for a further 30 min 2 g/L of sodium hydroxide was then added and the dyeing continued for a further 60 min at 40 °C. The fabric was removed from the dyebath and rinsed thoroughly in deionised water prior to after-soaping, final rinsing and air drying.

Tencel fabrics were introduced into the dye bath containing 1%, 5% and 10% o.m.f. of the C. I. Reactive Red 228, at 60 °C with a liquor to goods ratio of 15:1, and 50, 70 and 80 g/L sodium chloride, respectively, added over 30 min 15, 20 and 20 g/L sodium carbonate, respectively, were then added and the dye bath temperature maintained to 60 °C for a further 60 min. The dyebath was then dropped and the fabric rinsed thoroughly in deionised water prior to soaping, final rinsing and air-drying.

3.2. After-soaping treatment [23]

In order to remove any unfixed dye from the dyed Tencel, the dyed fabrics were after-soaped with a 1 g/L aqueous non-ionic detergent solution at 100 °C for 15 min at a liquor to goods ratio of 50:1. At the end of the wash-off, the dyed samples were rinsed in hot water at 80 °C, then in cold water and finally air-dried at room temperature.

3.3. Application of reactive dyes to cotton

Reactive dyeing of the cotton fabric was performed following the method described above. However, only the 4% o.m.f. dye applications were used with other parameters remaining the same.

3.4. Laundering process ISO 105 C09 wash test

The dyed fabrics were washed following the ISO 105 CO9 procedure, either with or without the perborate and TAED (tetraacetylethylenediamine) incorporated into the treatment solution [15]. The washed fabrics were rinsed in warm water and then air dried prior to further analysis.

The wash solution was prepared as recommended with 10 g/L of ECE (phosphate-based) detergent, 12 g/L sodium perborate tetrahydrate and 1.8 g/L TAED being dissolved in 1 L of deionised water. The wash pot containing the fabric and wash solution was rotated in a Washtec-P machine (Roaches) at 40 revolutions per minute, 60 °C for 30 min. At the end of the wash cycle the fabric sample was washed thoroughly in running cold water and deionised water, respectively, and air-dried prior to colour analysis.

3.5. Colour analysis [23]

The colour strength (K/S) was calculated from reflectance measurements using the single constant Kubelka-Munk equation (Equation (1)):

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

Where K is the coefficient of absorption, S is the coefficient of scatter, R is reflectance expressed as a proportional value and λ is the wavelength.

The K/S and CIE $L^*a^*b^*$ values were calculated from the mean of four reflectance measurements produced by a Datacolor Spectroflash 600. The CIE $L^*a^*b^*$ values were calculated under illuminant D65 using a 10° standard observer. The K/S values were calculated at the λ_{max} of the dyed fabric. Each fabric sample was folded twice in order to achieve opacity.

3.6. Dye exhaustion and fixation analysis [23]

Percentage dye exhaustion (%E) was calculated from Equation (2):

$$\%E = \frac{C_0 - C_1}{C_0} \times 100\% = \frac{A_0 - A_1}{A_0} \times 100\% \quad (2)$$

Where C_0 and C_1 are the concentrations of the dye before and after dyeing (g/L), respectively, and A_0 and A_1 are the absorbance values of the dyebath before and after dyeing at the λ_{max} of each dye, respectively.

For each dyeing, the total fixation efficiency (T), which is the percentage of the dye originally applied to the fabric becoming covalently bonded, was calculated using Equation (3):