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Spun-dyed lyocell

Avinash P. Manian [a], Hartmut Ruef [b], Thomas Bechtold [a,*]

[a] *Christian-Doppler Laboratory for Textile and Fiber Chemistry in Cellulosics, Institute of Textile Chemistry and Textile Physics, University of Innsbruck, Hoechsterstrasse 73, 6850 Dornbirn, Austria*
[b] *Textile Innovation, Lenzing AG, 4860 Lenzing, Austria*

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Abstract

Cellulose pulp was dyed with a vat dye in an exhaust dyeing process. The dyed pulp, mixed with undyed pulp, was dissolved in NMMO and spun to obtain 'spun-dyed' lyocell fibers. The dyeing behavior of pulp and fastness properties of the spun-dyed fibers were compared with that of different cellulosic substrates dyed with the same vat dye. The cellulose pulp exhibited the highest degree of dye exhaustion as compared to lyocell fabrics (woven and knit), yarns, fibers, and woven cotton. The spun-dyed lyocell fibers exhibited superior light fastness as compared to conventionally dyed cotton and lyocell substrates. The spun-dyeing process is believed to involve lower costs than conventional dyeing and to be more eco-friendly. The fiber spinning process did not appear to be detrimental to the color in spun-dyed fibers.

Keywords: Lyocell; Spun-dyeing; Mass Coloration; Vat dye

1. Introduction

Spun-dyeing, mass coloration or dope dyeing maybe defined as "a method of coloring manufactured fibers by incorporation of the colorant in the spinning composition before extrusion into filaments" [1]. The technique has the advantages of cost efficiency, uniformity of coloration, superior colorfastness, and is the method of choice for imparting color to polymers that are otherwise difficult to dye, e.g. polypropylene.

One of the primary considerations in any mass coloration process is to ensure the chemical and physical stability of the polymer–colorant mixture. Regenerated cellulosics present unique challenges in this regard, because their manufacturing processes often involve treatment of the cellulose with strong reducing and/or oxidizing agents, which may militate against the stability of colorants. But mass coloration of regenerated cellulosics has been found possible and various methods for its achievement are reported in literature.

Many of the methods involve the addition of a vat dye to the spinning dope [2–7]. The vat dye is reduced to its leuco form prior to addition in spinning dope, or in the spinning dope, or even in the formed filament, with oxidation back to their parent form being accomplished generally during or after regeneration. In some techniques, vat acids or the ester derivatives of the leuco compounds of vat dyes are added to the spinning dope and the final color is developed by oxidation treatments, generally of the formed filament [8,9]. Mass coloration may also be achieved by adding to spinning dopes colorants dissolved in polar water-miscible solvents or by dissolving colorants directly in the spinning dope [10–17]. The colorants used in these methods are selected dyes, dye derivatives, or pigments. The dispersion of certain finely milled organic or inorganic pigments in spinning dopes prior to filament spinning has been suggested as a possible route [18–26], with additives being recommended in some cases to improve pigment dispersability [21–23,27]. Other proposed techniques involve the suspension of sulfur dye intermediates in spinning dopes [28], or utilizing waste cotton textiles dyed with reactive dyes by mixing them with fresh cellulose, subjecting

* Corresponding author. Tel.: +43 5572 28533, fax: +43 5572 28629.
E-mail address: textilchemie@uibk.ac.at (T. Bechtold).

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the mixture to xanthation, and spinning colored filaments there from [29].

The techniques listed above pertain to mass coloration of viscose or cuprammonium rayon, wherein cellulose pulp is treated with strong reducing agents under alkaline or neutral conditions. In this communication we report results from an investigation into the mass coloration of lyocell, a regenerated cellulosic fiber manufactured in a closed-loop process involving the dissolution of wood pulp in *N*-methyl-morpholine-*N*-oxide (NMMO), which is a strong oxidant. The objective of this study was to determine the feasibility of mass coloration of lyocell and to compare the properties of spun-dyed lyocell fibers with other cellulosic substrates dyed by a conventional exhaust process.

2. Experimental

2.1. Lyocell spun-dyeing

In essence, the spun-dyeing process consisted of dyeing cellulose pulp with C.I. Vat Green 5 (Indanthrene Brilliant Green FFB), and mixing the dyed pulp with undyed pulp at a ratio of 1:4. Fibers were spun by dissolving the pulp mixture in NMMO and extruding the cellulose dope via an air gap into an aqueous spinning bath. A schematic illustration of the spun-dyeing process is shown in Fig. 1, and is described elsewhere [30]. From the amounts of dye used in dyeing pulp and the ratio of undyed pulp mixed with dyed pulp during fiber spinning, it was estimated that the spun-dyed lyocell fibers were dyed to a 2.5% shade.

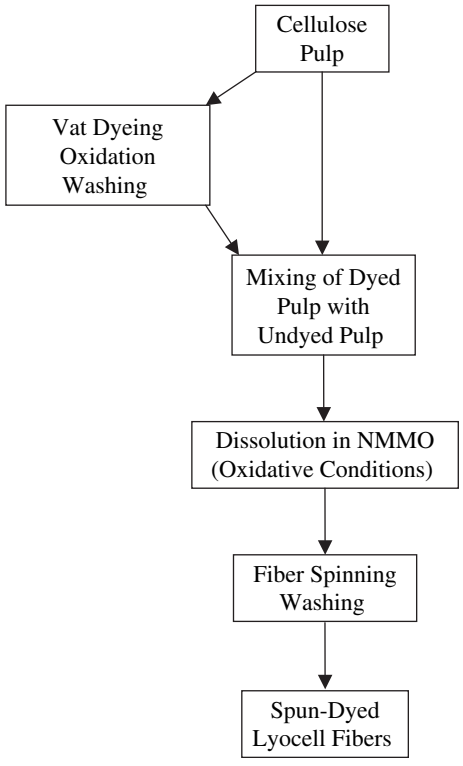

Fig. 1. Schematic illustration of the spun-dyeing process.

2.2. Comparison of dyeing behavior

In a series of dyeing experiments, 1.2–1.8 g of different lyocell substrates with increasing degrees of internal structural order and binding complexity: pulp, fiber, yarn, and fabrics (knit and woven) described in Table 1, were dyed in an exhaust process with C.I.Vat Green 5. A woven cotton fabric was also included in the dyeing experiments. The dye exhaustion and resulting color depth observed in fiber, yarn and fabric substrates were compared with those obtained in pulp and spun-dyed lyocell.

The woven fabrics and pulp were used for dyeing as obtained, but the samples of knit fabric, yarn, and fiber were scoured in a 2 ml/L solution of a detergent in deionized water at 40 °C with a liquor ratio of 1:20 for 45 min. The detergent used was a neutral formulation made up of cocoa fatty acids (58.8 g/L), KOH (16.5 g/L), a secondary alkane sulfonate (117.6 g/L), a fatty alcohol ethoxylate (352.8 g/L), citric acid (2.9 g/L), ethanol (132.3 g/L), and isopropanol (95.6 g/L) in deionized water. At the end of scouring, samples were washed thoroughly with running cold water and centrifuged at 4700 rpm for 10 min in a laboratory centrifuge. To avoid the possibility of hornification that is known to occur in lyocell due to wetting and drying [31,32], the scoured samples were used directly for dyeing after centrifugation without an intermediate drying step.

Samples were dyed to a series of shades ranging from 0.1 to 3.0% (on weight of substrate) in a Mathis Labomat dyeing machine at a material to liquor ratio of 1:40 from formulations containing 0.025–0.75 g/L dye, 6.17 g/L NaOH, and 5.0 g/L $Na_2S_2O_4$. The dyeing temperature profile is shown in Fig. 2.

In formulating dye liquors, dye and alkali were added from stock solutions that were prepared and used for the whole dyeing experiment, while the reducing agent was added from solutions prepared fresh before each individual set of dyeings. The dye liquors were added to dyepots and pre-heated to 47–50 °C in a water bath before introducing samples. The dyepots were then loaded onto the dyeing machine and the dyeing program was initiated. At the end of dyeing, the spent dye liquor was drained and the samples were oxidized by treatment with a 4 g/L solution of a commercial formulation of the sodium salt of *m*-nitro benzene sulfonic acid (Ludigol, BASF AG) at a material to liquor ratio of 1:40 at 60 °C for 15–20 min. The dyed samples were then washed in running cold water and dried.

Dye exhaustion was calculated from the difference in dye concentration between dye liquors before and after dyeing, expressed as a percentage of dye concentration in liquor before dyeing. Dye concentrations in spent liquors were determined from their optical density values measured, after diluting liquors as required with a stock vat solution, on a Hitachi U-2000 Double Beam Spectrophotometer at a wavelength of 614.0 nm, which was determined to be the wavelength of maximum absorbance of the reduced dye solution. A calibration curve of optical densities (at 614.0 nm) of reduced dye solutions of known concentration was used to compute dye concentrations from optical density values.

Table 1
Description of substrates

Substrate	Code	Construction	Yarn count (Nm)	Fiber fineness (dtex)	Processing history	Mass/area (g/m^2)
Cotton (woven)	Co-W	4/1 Satin weave, ring spun yarns	68/1	–	Desized, scoured, and bleached.	171.40
Lyocell (woven)	CLY-W	1/1 Plain weave, ring spun yarns	50/1	1.3	Desized and scoured	136.39
Lyocell (knit)	CLY-K	Single jersey, ring spun yarns	50/1	1.3	Unscoured	128.95
Lyocell (yarn)	CLY-Y	Ring spun yarn	68/1	1.3	Unscoured	–
Lyocell (fiber)	CLY-F	–	–	1.3	Unscoured	–
Cellulose pulp	CLY-P	–	–	–	–	–

The color depth in dyed samples was evaluated with the Kubelka–Munk function, shown in Eqs. (1) and (2).

$$\left(\frac{K}{S}\right) = \frac{(1-R)^2}{2R} \tag{1}$$

$$\left(\frac{K}{S}\right)_{\text{Corr}} = \left(\frac{K}{S}\right)_{\text{D}} - \left(\frac{K}{S}\right)_{\text{U}} \tag{2}$$

where, R = fraction of light reflected by substrate at wavelength of maximum absorbance, $(K/S)_{\text{D}} = (K/S)$ value of dyed substrate, $(K/S)_{\text{U}} = (K/S)$ value of undyed substrate, $(K/S)_{\text{Corr}} = (K/S)$ values corrected for reflectance from undyed substrate.

The reflectance of substrates, at an 8° angle of incidence and inclusive of specular reflection, was measured at 637.0 nm in a Specord® 50 UV–VIS Spectrometer equipped with an integrating sphere. Due to its construction, there was a distinct difference in reflectance between the two faces of the cotton satin fabric, and one face exhibited more gloss than the other. In order to achieve better reproducibility in results, all reflectance measurements for the cotton fabric were conducted on the face with lower gloss.

The color difference between spun-dyed fibers and the other conventionally dyed substrates was determined from their CIELAB color coordinates, measured on a Minolta Chroma Meter CR-200 with D_{65} illuminant at a 2° field of observation, as shown in Eq. (3).

$$\Delta E^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \tag{3}$$

Where, ΔE^* = color difference between spun-dyed fiber and other dyed substrate; ΔL^*, Δa^*, Δb^* = difference in mean L^*, a^*, and b^* values, respectively, between spun-dyed fibers and conventionally dyed substrate.

As in measurements of reflectance, all colorimetric measurements for cotton fabric samples were conducted on the face with lower gloss. A total of nine measurements were conducted on specimens from each combination of substrate and liquor dye concentration.

2.3. Fastness properties

Three specimens from each combination of substrate and liquor dye concentration were tested for colorfastness to light and water, according to the procedure described in ISO-105 BO2 and AATCC 107-1997, respectively [33,34]. The fastness ratings were assigned independently by three different observers.

3. Results and discussion

3.1. Dye exhaustion

The dye exhaustion observed in substrates is shown in Fig. 3. The data were analyzed in a two-way ANOVA procedure at a 0.05 level of significance to determine the influence of substrate type and dye liquor concentration on dye exhaustion.

There was a pairing among substrates with respect to dye exhaustion, with close similarities in dye exhaustion trends within substrate pairs and significant differences between them. At any given level of initial dye concentration in bath, fiber and pulp exhibited the highest dye exhaustion, followed

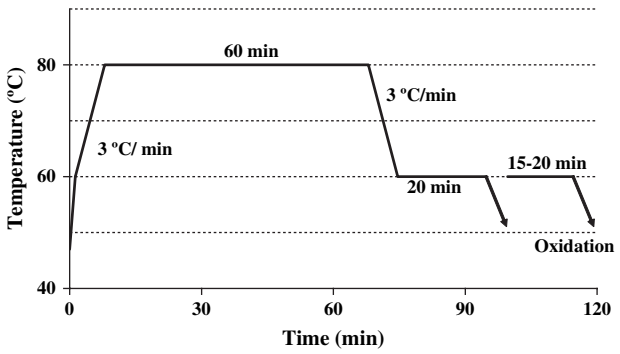

Fig. 2. Dyeing temperature profile.

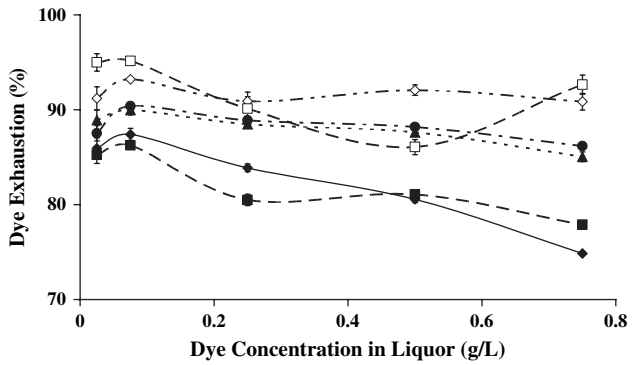

Fig. 3. Dye exhaustion in substrates: (◆) Co-W, (■) CLY-W, (▲) CLY-K, (●) CLY-Y, (◇), CLY-F, (□) CLY-P. Error bars represent ±1 standard deviation value ($n = 3$). Refer Table 1 for substrate descriptions.

by knit fabric and yarn, while the woven fabrics exhibited the lowest dye exhaustion among all substrates studied.

The dye exhaustion in all substrates decreased with increasing initial dye concentration in liquor, but the rate of decrease in dye exhaustion differed between substrate pairs. The greatest rate of decrease was observed in the pair of woven fabrics, while those in the knit fabric and yarn, and fiber and pulp were more gradual. Although dye exhaustion in all substrates appeared to pass through a peak at a dye concentration of 0.075 g/L in liquor, it was not statistically significant. The dye exhaustion in pulp passed through a minimum with increasing initial dye concentration in liquor, but the reasons for this are not immediately evident and maybe due to extraneous experimental errors.

The differences observed in dye exhaustion between substrates maybe attributed to their differences in ratio of mass to surface area (*m*/*a*), the degree of internal structural order, and binding complexity, since these factors govern dye accessibility in substrate and thereby influence both dyeing rate and equilibrium dye uptake. In general, among the substrates studied, dye exhaustion at any given level of initial dye concentration in bath increased with decreasing *m*/*a* ratio and decreasing degrees of internal structural order and binding complexity. As an example, one of the highest dye exhaustion values was observed in pulp which possessed the highest surface area per unit mass and the lowest degrees of internal structural order and binding complexity, while the lowest degree of dye exhaustion was observed in woven fabrics where the *m*/*a* ratio, internal structural order and binding complexity were the highest.

However, there appeared some anomalies in the correlation between substrate accessibility and dye exhaustion. There were no significant differences in dye exhaustion between fiber and pulp, between knit fabric and yarn, and between the woven fabrics, although there were distinct differences in *m*/*a* ratio, binding complexity and/or internal structural order within these substrate pairs. Lyocell exhibits a strong swelling tendency in alkaline solutions leading to changes in substrate internal structure and morphology [35,36]. The dyeing liquors were alkaline and hence it is possible that some degree of lyocell swelling occurred during dyeing leading to changes in structure and accessibility in substrates, which may explain the similarities observed in dye exhaustion within substrate pairs despite their initial structural differences.

3.2. Color depth

The color depth exhibited by the different dyed substrates as a function of their percent shade is shown in Fig. 4. The data were analyzed in a two-way ANOVA procedure at a 0.05 level of significance to determine the influence of substrate type and percent shade on color depth.

There were no significant differences in color depth between fabrics (woven and knit), yarn, and fiber at any level of percent shade. The dyed pulp exhibited the lowest color depth among all substrates and the build up in color depth with increasing percent shade was also comparatively lower in pulp. The color depth in spun-dyed lyocell fibers appeared

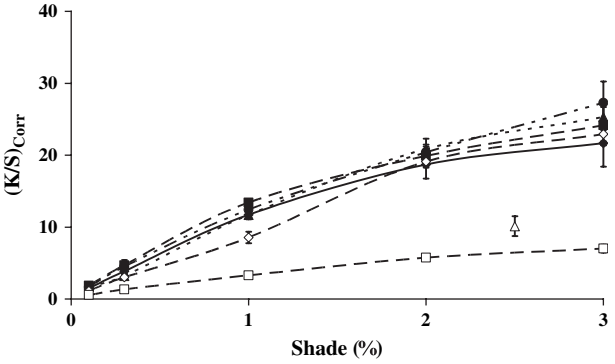

Fig. 4. Color depth [$(K/S)_{Corr}$] in dyed substrates: (◆) Co-W, (■) CLY-W, (▲) CLY-K, (●) CLY-Y, (◇), CLY-F, (□) CLY-P, (△) spun-dyed lyocell. Error bars represent ±1 standard deviation value ($n = 9$). Refer Table 1 for substrate descriptions.

to be higher than that exhibited by the dyed pulp but lower than that exhibited by the other substrates, when compared at similar shade depths.

The perceived color depth in any textile substrate is influenced by the amount of dye present near its surface and its light scattering propensity. In general, for a given level of dye exhaustion, substrates that have a higher surface concentration of dye and/or a lower propensity to scatter light appear to have greater color depth. The low color depth in dyed pulp, in spite of its relatively high dye exhaustion, maybe attributed to a highly uniform distribution of dye within the pulp and a higher light scattering propensity due both to its high surface area per unit mass and small particle size.

It is possible that a higher uniformity in dye distribution is also a reason for the low color depth observed in spun-dyed fibers. However, as the dye exhaustion in spun-dyed fibers was only estimated and not measured, and since the degree of dye exhaustion strongly influences color depth, it may not be possible to attribute the low color depth in spun-dyed fibers solely to uniformity in dye distribution.

3.3. Color difference

The color difference between spun-dyed fibers and the other conventionally dyed substrates was determined to assess whether the fiber spinning process influenced the color in spun-dyed fibers. A partial list of the ΔE^* values obtained is shown in Table 2, along with the color coordinates of individual substrates. In the interest of conserving space, only those values are listed for each substrate type where the ΔE^* values, with respect to spun-dyed fibers, were the lowest: woven fabrics and yarns dyed to 1.0% shade, fibers dyed to 2.0% shade, and pulp dyed to 3.0% shade.

However, ΔE^* values are a composite measure of differences along three coordinates in the CIELAB color scale, and do not elaborate on the nature of the color difference. Hence, the CMC formula was used to calculate the values of chroma (C^*_{ab}) and hue (h^*_{ab}) for substrates from their CIELAB values, as described in the AATCC Test Method 173-1998 [37], and are also listed in Table 2. Although there were

Table 2
Color difference between spun-dyed fibers and other dyed substrates, and mean color coordinates of individual substrates ($n = 9$)

Substrate	Shade (%)	ΔE^*[a]	L^*	a^*	b^*	C^*_{ab}	h^*_{ab}
Co-W	1.0	5.12	46.20	−31.54	−11.82	33.68	180.36
CLY-W	1.0	3.18	42.64	−30.14	−11.79	32.37	180.37
CLY-K	1.0	2.48	43.39	−29.37	−11.35	31.49	180.37
CLY-Y	1.0	3.21	44.75	−30.18	−12.39	32.63	180.39
CLY-F	2.0	4.66	41.98	−31.51	−13.21	34.16	180.40
CLY-P	3.0	5.97	48.15	−29.90	−9.77	31.47	180.32
Spun-dyed fiber	~2.5	–	43.59	−27.19	−12.52	29.93	180.43

Refer Table 1 for substrate descriptions.
[a] ΔE^* values calculated with respect to spun-dyed fibers.

distinct differences in lightness and chroma between the spun-dyed fibers and conventionally dyed substrates, the differences in hue were not significant. In fact, no significant differences in hue were observed between the spun-dyed and conventionally dyed substrates at any level of liquor dye concentration, indicating that the fiber spinning process was not detrimental to the color in spun-dyed fibers in case of C.I. Vat Green 5.

3.4. Colorfastness

The light and water fastness ratings of the dyed substrates, averaged across liquor dye concentrations and observer, are given in Table 3. In the test for colorfastness to water, samples of dyed substrates were sandwiched between strips of undyed wool and lyocell fabrics instead of multi-fiber fabric as prescribed in the test method [34]. There was little difference in staining between the wool and lyocell strips, and hence the two stain ratings were also averaged. While there were no differences in colorfastness to water between the different substrates, the light fastness in spun-dyed lyocell fibers was superior to that observed in the other conventionally dyed substrates.

The high dye exhaustion observed in pulp and the fact that dyed pulp maybe mixed with undyed pulp to obtain desired shade depths could lead to substantial savings in production costs and could also prove to be more eco-friendly. For illustration, the chemical and energy requirements for conventional dyeing are compared with those for mass coloration in Table 4.

The amounts shown in Table 4 are calculated for exhaust dyeing with a vat dye from the IN class with a liquor ratio of 1:10 at 80 °C and are based on recommendations in literature [38]. The values quoted for the conventional dyeing process are for a 100 kg batch of substrate dyed to a 2.0% shade and those quoted for the mass coloration process are for a 20 kg batch of pulp dyed to a 10.0% shade wherein the dyed pulp maybe diluted with 80 kg of undyed pulp in the fiber spinning process to obtain a final shade of 2.0% in the spun-dyed fibers. The dyeing time of 30 min listed for the mass coloration process as compared to the 90 min for conventional dyeing is due to the fact that a leveling step is not required in pulp dyeing processes.

Table 3
Colorfastness of dyed substrates

Substrate	Fastness ratings	
	Light	Water
Cotton (woven)	6	5
Lyocell (woven)	5	5
Lyocell (knit)	4	5
Lyocell (yarn)	6	5
Lyocell (fiber)	6–7	5
Spun-dyed fiber	7	5

Table 4
Projected savings in producing spun-dyed lyocell fibers

Requirement	Conventional dyeing		Mass coloration		Savings (%)
	Per liter	Per batch[a]	Per liter	Per batch[b]	
Hydrosulfite	5 g	5 kg	16 g	3.2 kg	36
NaOH (38° Bé)	20 ml	20 L	52 ml	10.4 L	48
Auxiliaries (wetting/dispersing agents)	1–2 g	1–2 kg	0.5 g	0.1 kg	90–95
Energy		250.8 MJ		50.2 MJ	80
Time		90 min		30 min	67

[a] For a batch of 100 kg substrate dyed to 2.0% shade at a liquor ratio of 1:10.
[b] For a batch of 20 kg pulp dyed to 10.0% shade at a liquor ratio of 1:10.

4. Conclusions

It was demonstrated that mass coloration of lyocell is a feasible technique and that the fiber spinning process does not influence the color in spun-dyed fibers. The advantages of mass coloration processes over conventional dyeing processes for lyocell are many: the savings in energy and chemicals are expected to make mass coloration substantially more cost effective and eco-friendly than conventional dyeing; the problems associated with vat dyeing of lyocell due to its strong swelling propensity in alkaline solutions are obviated; and spun-dyed fibers maybe imparted treatments without fear of impairing their potential for coloration. In addition, the high fastness to light and water in spun-dyed lyocell fibers may permit their use in both woven and non-woven technologies, for applications in such specialty fields as medical textiles.

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