Fastness and Dyeability Improvement of Wool Fabric Pretreated with Oxalate Derivatives

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ABSTRACT: Wool fabrics were pretreated with calcium and sodium oxalate in acidic and alkaline pH media. The pretreated and untreated fabric samples were then dyed in the same bath with acid dyes by the exhaustion technique. The pretreated fiber sample surfaces were observed using a scanning electron microscope. The color strength and fastness properties of the fabrics were investigated. The results of the study showed that pretreatment with oxalate derivatives can be used as a means of improving the dyeability of wool fibers. As the dyed, pretreated wool fabrics had higher color strength and fastness results than the untreated wool fabrics, the mechanical properties were affected negatively. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 122: 3440–3445, 2011

Key words: wool; pretreating; oxalate; dyeing; fastness

INTRODUCTION

Wool, the fibrous coating from sheep, is the most important animal fiber used by the textile industry. Considerable amounts of wool fibers are used in textiles, apparel, and carpets.[1] The acquired dyeing and fastness properties of woolen fabric are very important characteristics in terms of the user.[2] The interaction of certain compounds with wool fibers results in some changes in the polymeric structure of the fibers. These effects on the fiber properties can be expected to strongly affect the dyeing results. Improving the dyeing and fastness properties of textile fibers have been subject of various studies.[3–5] Allam and Bendak[6] reported that improvement of the dyeability of wool, silk, and polyamide fabrics with cationic dye was achieved by their pretreatments with saccharin sodium salts. Ali and Khatip[7] studied wool fabric pretreated with chitosan and some natural constituents, and their pretreatments effectively enhanced the dyeing properties, color fastness, whiteness, and wettability. Periolatto et al.[8] examined the application of enzymes in wool dyeing process; study of their enzymatic pretreatment decreased dyeing temperature with good mechanical properties. Yuan et al.[9] reported that a new class of green solvent ionic liquid improved the dyeability of wool, and the ionic liquid treatment increased exhaustion and color depth. In another different study, calcium oxalate in alkaline medium had been applied to wool fiber, feathered-leather, and cotton as a pretreatment process. Onal et al.[10] applied the calcium oxalate to wool fibers and dyed them with *Rubia Tinctorum* L. natural dye. The calcium oxalate pretreatment process improved dyeing yield and the washing and light fastness properties of wool fiber.

This study evaluates the effects of calcium and sodium oxalate pretreatment on wool fabric in acidic and alkaline media. The novelty of this work is effective in improvement of acid dyeing and fastness performance of wool fabric.[11]

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EXPERIMENTAL

Fabrics

The wool fabric was supplied by Bahariye Textile Company (Istanbul, Turkey). The characteristics of the fabric were as follows: woven twill 1/1, 344 g/m^2. The warp and weft yarns are identical (45 tex), and the density of these yarns are 34 and 34 threads per cm, respectively. The fabrics were washed with a solution of nonionic detergent (1 g/L) for 30 min at 50°C, thoroughly rinsed, and dried at room temperature.

Chemicals and dyes

Calcium oxalate, disodium oxalate, urea, ammonia, and acetic acid used in this study are of commercial grade. The seven commercial acid dyes used in this study were shown in Table I. The dyes were used as obtained from the dye manufacturer (Alfa Chem., Turkey) without further purification. However, the

TABLE I
Dye Code and λ_{max} of Dyes Used

Dye code	Commercial name of used dyes	The wavelength of maximum absorbance, λ_{max} (nm)
D1	Synacid Bordeaux BW	525
D2	Synacid N.Blue RL	578
D3	Synacid Brown ABL	471
D4	Synacid Blue BRL	591
D5	Synacid Cyanine M5R	565
D6	Synacid Black MSRL 180%	573
D7	Synacid Yellow 3GLM	428

TABLE II
Pretreatment Type, Chemicals, and pH
(+, Used and –, Unused)

Chemicals	Pretreatment type				
	P1 untreated	P2	P3	P4	P5
Calcium oxalate	−	+	+	−	−
Disodium oxalate	−	−	−	+	+
Urea	−	+	+	+	+
Ammonia	−	+	−	+	−
Acetic acid	−	−	+	−	+
Pretreatment pH	–	11–11.5	3.5–4	11–11.5	3.5–4

exact chemical structure of these dyes cannot be obtained as they are commercial products.

Scanning electron microscope analysis

A scanning electron microscope (SEM) LEO 440 was used to characterize the surface morphology of the wool fibers after oxalate pretreatment. The samples were prepared by the standard preparative technique of applying a gold layer to produce a conductive surface.

Fabric pretreatment

The wool fabric samples were treated with 3%, over weight for fiber (owf) of chemicals in glass bottles using a liquor ratio of 1 : 20 at 80°C for 1 h with continuous mixing. The pretreatment diagram is shown in Figure 1(a). Table II shows pretreatment type and pH of the pretreating bath.

Dyeing methods

Dyeing was carried out in a laboratory dyeing machine Termal (Istanbul, Turkey) using the following solution: 1% owf dye and 1 mL/L acetic acid (15%). The all-in exhaust dyeing method was used for dyeing. Four different pretreated and untreated wool fabrics were dyed in the same dyeing bath. The dyeing process was started at 35°C; after working at this temperature for 10 minutes, the temperature was then raised to 95°C and dyeing was continued at the same temperature for a further 45 minutes.

After dyeing, the wool fabrics were removed from the bath and rinsed with tap water. The dyed fabrics were washed within a 1 g/L nonionic detergent at 80°C for 10 min until any excess dye was removed and then rinsed. The fabric samples were air dried. The dyeing diagram is shown Figure 1(b).

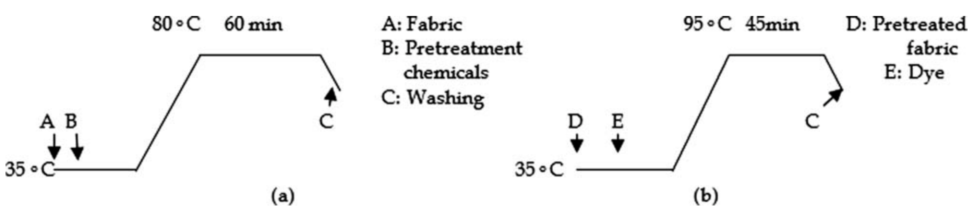

Figure 1 Pretreatment (a) and dyeing (b) diagrams of wool fabric.

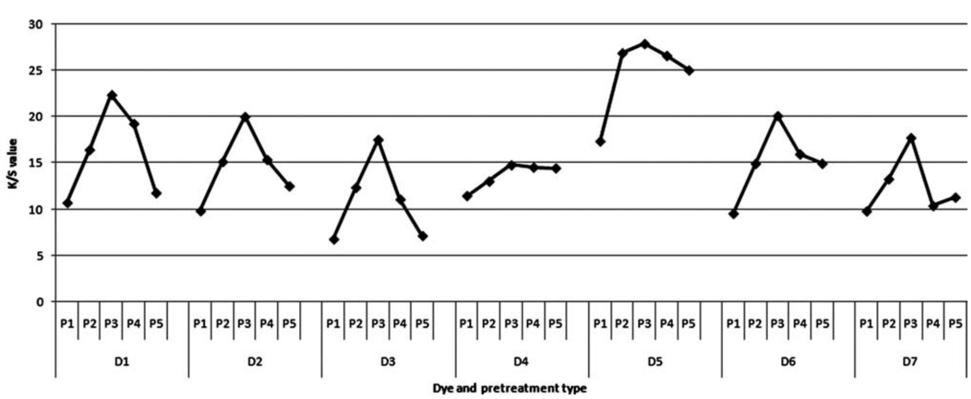

Figure 2 Dependence of the color intensity (K/S) of chemically pretreated wool fabrics (D: dye, P: pretreatment type).

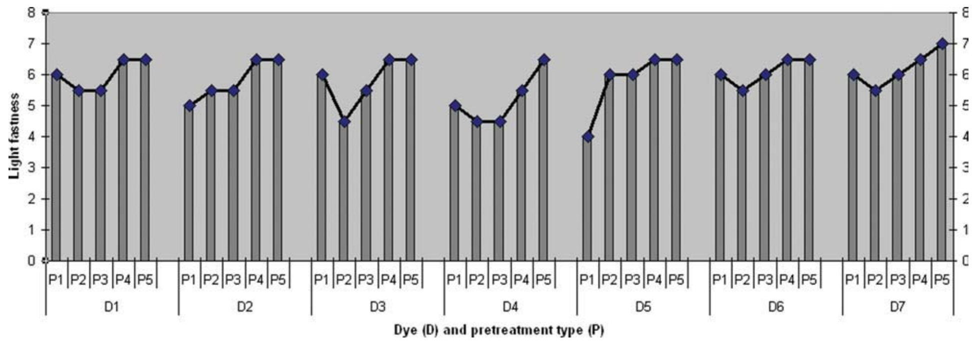

Figure 3 Light fastness of dyed wool fabrics. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Color measurement, fastness, and mechanical properties

The spectral reflectance measurements of the dyed fabrics were carried out using a Konica Minolta 3600d spectrophotometer. Color intensity was expressed as K/S values of the dyed samples using the Kubelka–Munk equation:

$$K/S = (1 - R)^2/2R \quad (1)$$

TABLE III
Washing and Rubbing Fastness of Dyed Wool Fabrics

Dye	Pretreatment	Washing fastness		Rubbing fastness	
		Changing	Staining	Dry	Wet
D1	P1	5	4	4	3
	P2	2/3	2/3	4/5	4/5
	P3	5	3/4	4/5	4
	P4	4/5	3/4	4/5	3/4
	P5	4/5	1/2	3/4	3/4
D2	P1	5	4	4	4
	P2	5	4/5	4/5	4/5
	P3	5	3/4	4	4
	P4	4/5	3/4	4/5	3/4
	P5	5	4/5	4/5	1/2
D3	P1	5	4	4	4
	P2	5	3/4	4/5	4/5
	P3	4/5	3/4	4	4
	P4	5	4/5	4/5	3/4
	P5	4/5	4	4/5	2/3
D4	P1	2	1/2	4	4
	P2	1/2	1/2	4/5	4/5
	P3	1/2	1	5	5
	P4	3/4	2/3	4/5	4/5
	P5	2	1/2	2/3	2/3
D5	P1	4	3	4	5
	P2	4/5	2/3	4/5	4/5
	P3	4	3/4	4	5
	P4	4/5	1/2	3/4	4/5
	P5	4/5	1/2	1/2	4/5
D6	P1	5	4	5	4
	P2	4/5	3/4	4/5	4/5
	P3	6	2/3	4	4
	P4	6/7	3/4	4/5	3/4
	P5	6/7	4	4/5	2/3
D7	P1	4	3	5	4
	P2	4/5	3/4	4/5	4/5
	P3	3/4	1/2	4	4
	P4	4/5	3/4	4/5	3/4
	P5	4/5	3/4	4/5	2/3

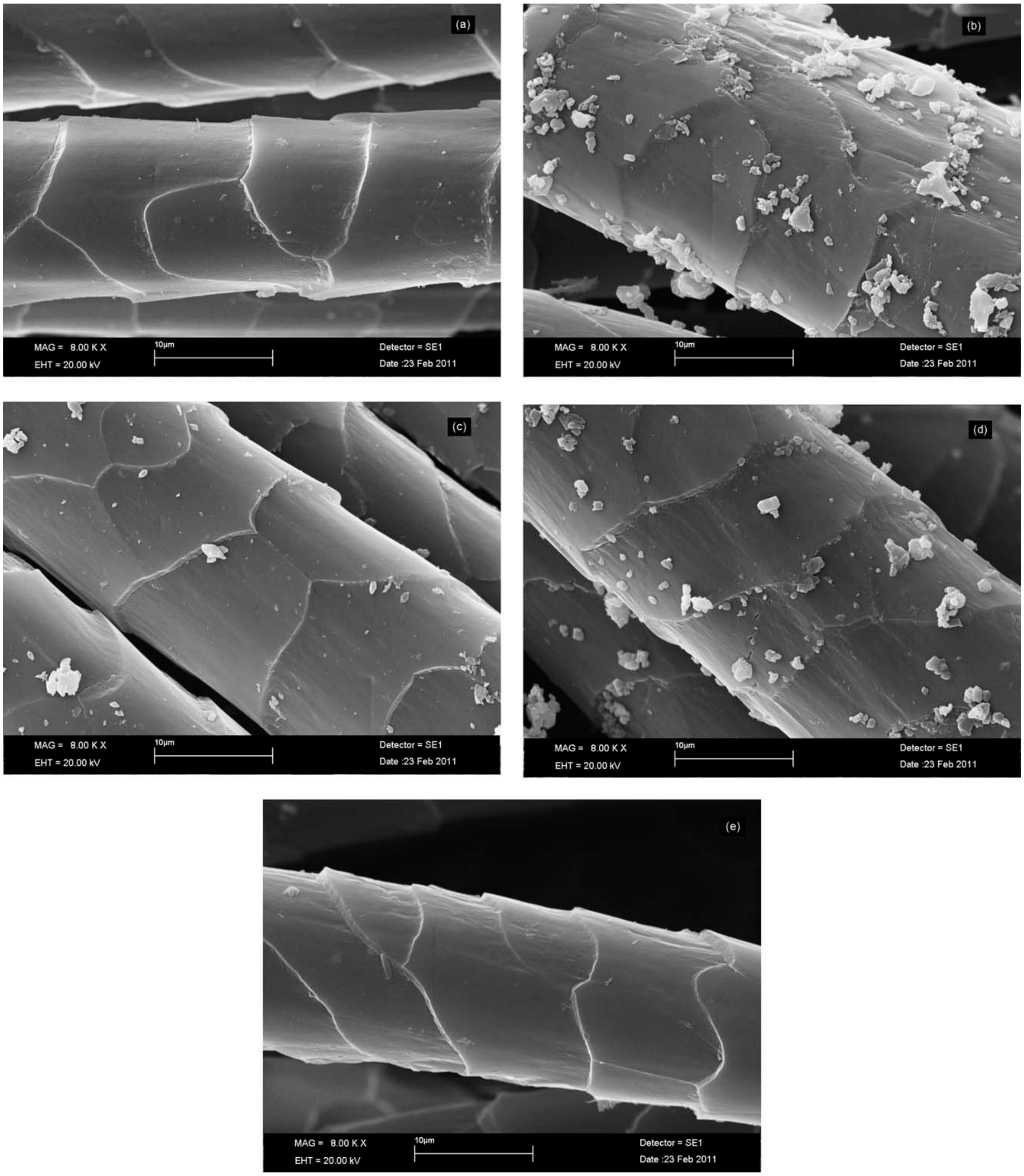

Figure 4 SEM pictures of the wool fibers after different treatments. (a) P1 untreated; (b) P2 treated; (c) P3 treated; (d) P4 treated; and (e) P5 treated.

where R is the decimal fraction of the reflectance of the dyed sample, K is the absorption, and S is the scattering coefficient.[12]

The fastness values against light, washing and rubbing of dyed samples were determined according to EN ISO 105-B02, EN ISO 105-C06, EN ISO 105-X12, and EN ISO 105-E04 standards. The tensile strength of wool fabrics was determined using Instron 4411 universal testing machine (USA). Performance properties of the wool fabric samples were evaluated using standard procedure EN ISO 13934-1. The samples were balanced at 25°C and at a relative humidity of 60% for 4 h before testing.

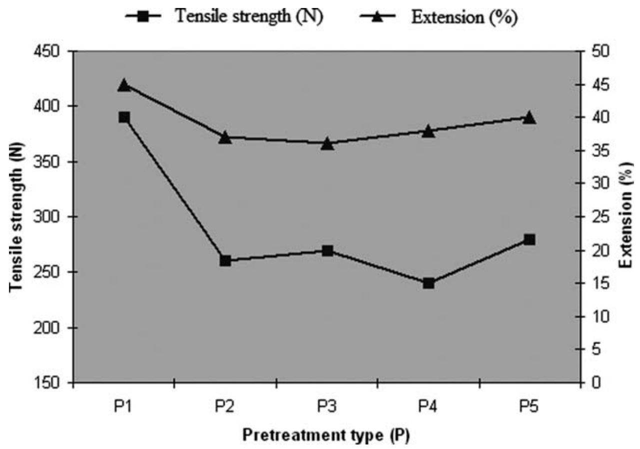

Figure 5 Changes in tensile strength (*N*) and extension (%) of pretreated (P1, P2, P3, P4, and P5) wool fabric.

RESULTS AND DISCUSSION

Build-up

The wool fabrics were pretreated chemically at 80°C by exhaustion technique for 1 h. The treated samples (P2, P3, P4, and P5), along with the untreated (P1) one were dyed using acid dyes as described in Experimental section. The extent of dyeing is expressed as color intensity (K/S).

Figure 2 shows the dependence of color intensity of the pretreated wool fabric on dyeing time of at 95°C with 1% D1, D2, D3, D4, D5, D6, and D7 acid dyes. It was observed that pretreatment type changed the color strength of the dyed wool fabrics. It can be seen from Figure 2 that in all acid dyes, K/S values increased with calcium oxalate (P2, P3) and then decreased with sodium oxalate (P4, P5). In general, the highest K/S values were obtained from the P3 pretreatment (wool fabric pretreated with calcium oxalate in acidic medium) type in all acid dyes. The high color strength of dyed pretreated wool fabrics orginates from the cation ions (Ca^{+2}, Na^{+2}) which is situated on the fiber. The positive ions in the fiber increased exhaustion of anionic acid dye molecules to the fiber from the dyeing bath.

Effect of light fastness

Light fastness tests were carried out on wool fabrics dyed with acid dyes. The results are shown in Figure 3.

Figure 3 shows the dependence of light fastness of the pretreated wool fabric on pretreatment type. It was observed that pretreatment type changed the light fastness of dyed wool fabrics. It can be seen from Figure 3 that there was an increase in light fastness with disodium oxalate pretreatment (P4, P5), and then a decrease was observed with calcium oxalate (P2, P3) in D1, D3, D4, and D7 acid dyes, while all types of pretreatment improved light fastness in D2 and D5 dyes. In general, the greatest improvement in light fastness was obtained from the P5 pretreatment type in D4 acid dyes.

Washing and rubbing fastness

Table III shows the dependence of washing and rubbing fastness of the pretreated wool fabric on pretreatment type. It was observed that pretreatment type changed the fastness properties of the dyed wool fabrics. The results show that washing and rubbing fastnesses increased or decreased depending on the pretreatment and fastness type. For example, in D1 dyes, a decrease was observed in washing fastness with calcium oxalate (P2, P3), and then an increase was observed with sodium oxalate (P4, P5). However, in D5 dyes, an increase was observed in washing fastness with calcium oxalate (P2, P3), and then a decrease was observed with sodium oxalate (P4, P5).

The build-up and fastness properties of dyed wool fabrics were similar; it can be concluded that, calcium and disodium oxalate pretreatment is effective in wool fiber dyeing with acid dyes. For example, all dyeing performances were improved using P4 pretreatment in D4 dyes.

Surface morphology

The SEM images [Fig. 4(a–e)] show no distinguishable physical modification of the wool fiber surface. The surface of the untreated wool fiber [Fig. 4(a)] is smooth and without damage when compared with the calcium oxalate-pretreated wool surface [Fig. 4(b,c)]. The mineral deposits are probably due to the low solubilty of calcium oxalate. There are mineral deposits on the surface of pretreated wool fiber with sodium oxalate in alkaline medium [Fig. 4(d)], but there are no mineral deposits on the fiber surface in acidic medium.

Mechanical properties

We investigated the properties related to tensile strength and extension in terms of the role of pretreatment types. Figure 5 shows the changes on tensile strength and extension of wool fabrics pretreated with calcium and sodium oxalate in acidic and alkaline media. When the tensile strength of the pretreated wool fabric is compared with that of an untreated fabric, the tensile strength of the pretreated wool fabric seriously decreases. However, decrease on extension of the pretreated fabric is limited. Most chemical treatments generally cause significant decrease in mechanical performance of fabric. The oxalate pretreatment of wool fabrics reduced tensile strength and extension in our experimental study.

CONCLUSIONS

As a result of experimental study, changes in the dyeing performance of chemically treated wool fibers were comparatively investigated by evaluation of their color strength, fastness, and mechanical properties. Calcium and disodium oxalate chemicals were applied to wool fabrics in acidic and alkaline pH media. Pretreatment processes did not change the surface of the wool fibers but decreased the mechanical properties such as tensile strength and extension. It is probably owing to pretreatment processes which could damage the chemical structure of the wool fiber. The dyeability of treated wool fabrics with acid dye was found to be better than that of the fabric untreated one at 95°C. Higher color strength was obtained in the treated wool fiber from dyeing with acid dyes.

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