Dyeing Transition Temperature of Wools Treated with Low Temperature Plasma, Liquid Ammonia, and High-Pressure Steam in Dyeing with Acid and Disperse Dyes

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ABSTRACT: Wool fibers treated with oxygen low-temperature plasma, liquid ammonia (NH_3), and high-pressure (HP) steam were dyed with two acid and three disperse dyes. Rate of dyeing, saturation dye uptake, and dyeing transition temperature were measured. Rate of dyeing of the O_2 plasma, NH_3, and HP steam-treated wools increased with acid dyes, whereas it did not increase with disperse dyes. Although dyeing transition temperature for acid dyes was decreased by the plasma, NH_3, and HP steam treatments, the temperature for disperse dyes was not changed by the treatments. Therefore, it seems that acid dyes penetrate by the intercellular diffusion through the interscale Cell Membrane Complex (CMC) of wool, whereas disperse dyes penetrate by the intracellular diffusion through the intrascale cuticle surface independently with CMC relaxation by the treatments. © 2001 John Wiley & Sons, Inc. J Appl Polym Sci 80: 1058–1062, 2001

Key words: wools; liquid ammonia; high-pressure steam; dyeing transition temperature

INTRODUCTION

Several studies on wool dyeing have already been carried out in relation to dye penetration into the fiber. Leeder et al. demonstrated that metal containing acid mordant dye penetrated through the Cell Membrane Complex (CMC) of the wools.[1,2] We also reported the same behavior from the dyeing properties of the plasma, liquid ammonia, and high-pressure steam-treated wools with acid dyes.[3–7] The rate of dyeing and saturation dye exhaustion of the plasma and HP steam-treated wools increased for acid dye, especially for milling-type acid dye and direct dyes. It was considered that the pretreatments cause a relaxation of CMC structure and lead to an increase of dyeing properties with acid dyes. Recently, we reported that rate of dyeing of the plasma-treated wool increased considerably with ionic-type natural dyes such as cochineal and Chinese cork tree, whereas disperse-type gromwell was not changed the dyeing properties.[8]

In this article, oxygen low-temperature plasma, liquid ammonia, and high-pressure steam-treated

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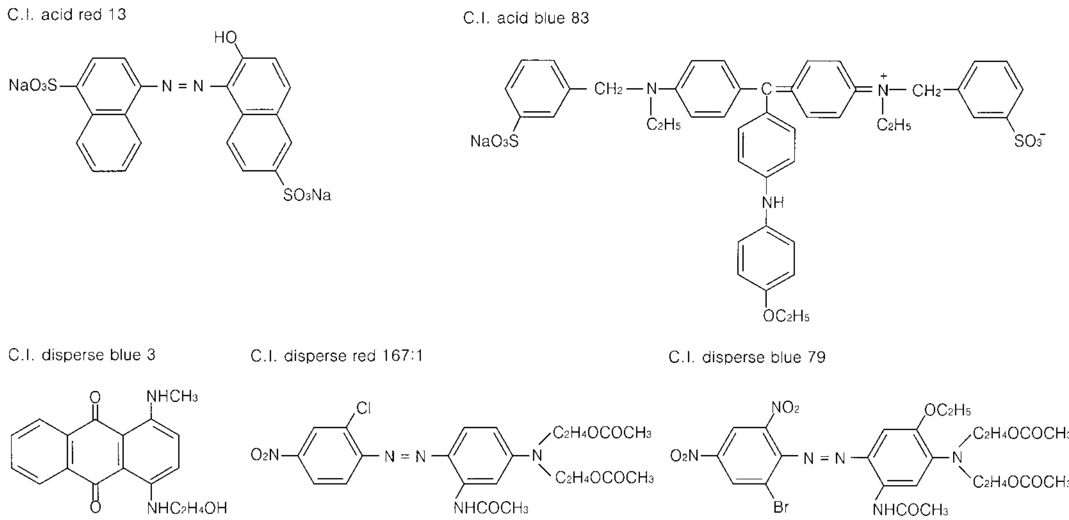
Table I Dyes Used



wool fibers were dyed with disperse and acid dyes, and the dyeing property, rate of dyeing, equilibrium dye uptake, and dyeing transition temperature was investigated for both dyes.

EXPERIMENTAL

Materials

Scoured merino wool fiber was treated with oxygen low-temperature plasma for 1 min under a pressure of 133 Pa, at a power of 300 W using a Yamato Plasma Reactor PR 501A and with high-pressure (HP) steam for 10 min at 130°C using a laboratory high-pressure steamer and liquid ammonia (NH_3) for 2 s using a practical range of Nisshinbo Co., Japan.

Dyeing

Table I shows the chemical structure of the dyes used in this experiment. The treated fibers were dyed with two acid dyes, leveling type C.I. acid red 13 and milling type C.I. acid blue 83, and three disperse dyes, C.I. disperse blue 3, C.I. disperse red 167 : 1 and C.I. disperse blue 79.

Dyebath was adjusted to pH 4.5 for C.I. acid red 13, pH 5.5 for C.I. acid blue 83, and pH 5.0 for disperse dyes with potassium dihydrogen phosphate and disodium hydrogen phosphate buffers (1 : 1).

Initial dye concentration in dyeing rate was prepared to 4×10^{-4} mol/L for acid dyes and 5×10^{-4} mol/L for disperse dyes. Dyeing was carried out at 80°C up to 20 h with acid dyes and 3 h for disperse dyes. The dye concentration in equilibrium dye uptake was adjusted to $0.5–6 \times 10^{-4}$ mol/L for acid dyes and $0.8–12 \times 10^{-4}$ mol/L for disperse dyes, and dyed up to 120 h. Dyeing transition temperature was obtained after dyeing for 10 min at temperatures of 20°C to 120°C.

After dyeing, the fibers were extracted with 25% pyridine aqueous solution for acid dyes and 100% DMF for disperse dyes.

RESULTS AND DISCUSSION

Dyeing Rate

Previously we reported that oxygen low-temperature plasma treatment of wool incorporates many oxygen atoms on the fiber surface in the form of —OH, —C═O, and —COOH,[3] liquid ammonia and high-pressure steam treatments did not change the nitrogen content in the fiber,[5] and the chemical composition.[7] But the treatments induced an increase not only the rate of dyeing but also equilibrium dye uptake of the acid dyes, especially milling acid dyes and direct dyes.[3,4] Therefore, we suggested that the treatments cause a relaxation of the CMC structure of wool

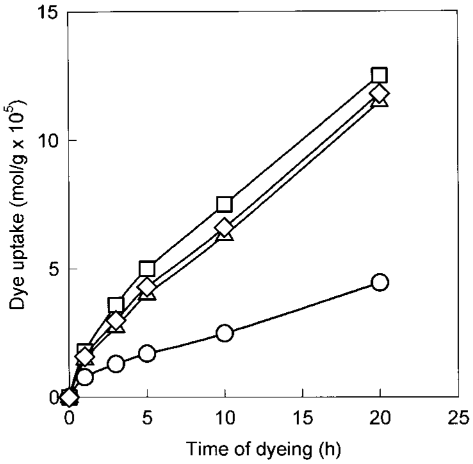


Figure 1 Dyeing rate of acid blue 83 on treated wools: (○) untreated, (□) O_2 plasma, (△) NH_3, (◇) HP steam.

and contribute to an increase of dyeing properties.

An example of dyeing rate for acid and disperse dyes was shown in Figures 1 and 2. Although the rate of dyeing was extensively increased for the plasma, NH_3, and HP steam-treated fibers with acid blue dye, it was not changed with disperse red dye. From these results, it supposes that dyeing behavior of the treated wools is wholly different for both kinds of dyes. That is, the plasma, NH_3 and HP steam treatments were greatly effective to improve the dyeability of wools toward the water soluble dyes such as acid and direct dyes,[3–7] while entirely not influenced for disperse dyes. Therefore, it assumes that dye penetration

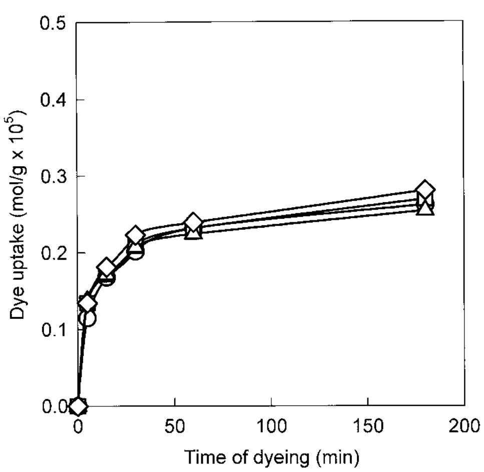


Figure 2 Dyeing rate of disperse red 167 : 1 on treated wools: (○) untreated, (□) O_2 plasma, (△) NH_3, (◇) HP steam.

Table II Equilibrium Dye Uptake of Treated Wools with Acid and Disperse Dyes

Dye	Equilibrium Dye Uptake ($mol/g \times 10^5$)			
	Untreated	O_2 Plasma	NH_3	HP Steam
Acid red 13	14.83	16.31	18.74	15.08
Acid blue 83	18.94	27.12	27.70	27.28
Disperse blue 3	1.20	1.24	1.26	1.23
Disperse red 167 : 1	0.30	0.30	0.33	0.33
Disperse blue 79	0.15	0.14	0.12	0.15

of acid dyes into wool brings about through CMC of the interscale. Namely, relaxation of CMC structure by the plasma, NH_3, and HP steam treatments are effective to improve the interscale diffusion of the dyeing with acid dyes. On the other hand, disperse dyes diffuse by the intrascale diffusion through the compact and hydrophobic cuticle surface without regarding CMC relaxation by the pretreatments.

Equilibrium Dye Uptake and Time of Half Dyeing

Table II shows equilibrium dye uptake for acid and disperse dyes for 120 h at 80°C of the plasma, NH_3, and HP steam-treated wools. Equilibrium dye uptake for acid dye was increased a little for leveling type, and considerably for milling type acid dyes by the pretreatments, whereas equilibrium dye uptake for disperse dyes was almost no change. Time of half dyeing was shown in Table III. Although time for the plasma, NH_3, and HP steam-treated wools decreased greatly for acid dyes because of considerable increase in early dye

Table III Time of Half Dyeing of Treated Wools with Acid and Disperse Dyes

Dye	Time of Half Dyeing (min)			
	Untreated	O_2 Plasma	NH_3	HP Steam
Acid red 13	811	95	116	454
Acid blue 83	5390	1563	1821	1690
Disperse blue 3	19	18	17	18
Disperse red 167 : 1	13	14	15	12
Disperse blue 79	55	53	54	48

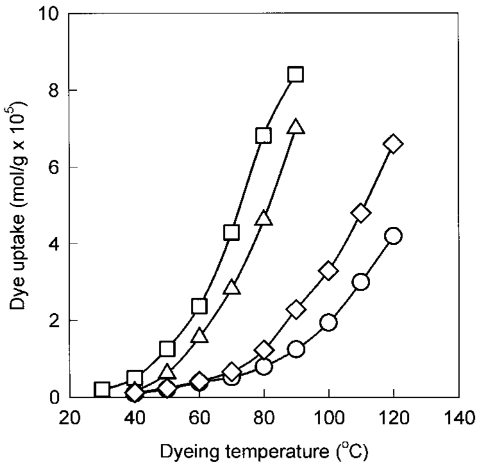


Figure 3 Relationship between dye uptake and dyeing temperature of acid red 13 on treated wools: (○) untreated, (□) O_2 plasma, (△) NH_3, (◇) HP steam.

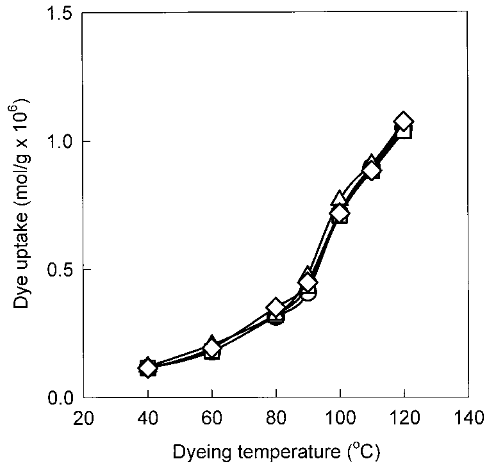


Figure 5 Relationship between dye uptake and dyeing temperature of disperse blue 3 on treated wools: (○) untreated, (□) O_2 plasma, (△) NH_3, (◇) HP steam.

exhaustion, whereas the treatments were not effective for disperse dyes.

From these results, it seems that dye penetration into wool fiber with both type dyes is obviously different. So, we investigate that CMC relaxation of wool by the treatments is effective at improving the dye diffusion of water soluble acid dyes, whereas CMC relaxation is quite no effective for water insoluble disperse dye that diffuse through intrascale. Although wool fiber is commonly dyed with acid dye by ionic bonding with amino group in the fiber, the fiber also contains a hydrophobic group and dyed with disperse dyes by hydrophobic bonding just like a nylon fiber

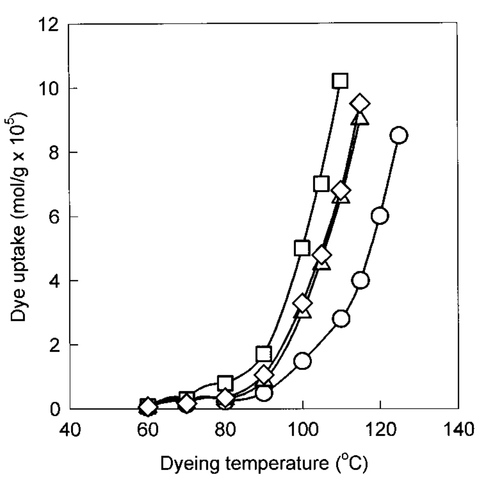


Figure 4 Relationship between dye uptake and dyeing temperature of acid blue 83 on treated wools: (○) untreated, (□) O_2 plasma, (△) NH_3, (◇) HP steam.

that dyed with acid and disperse dyes. Therefore, it seems that dyeing of wool with disperse dyes shows same behaviors with that of polyester fiber.

Dyeing Transition Temperature

In dyeing of polyester fiber with disperse dyes, it is commonly known that the relationship between dye exhaustion and dyeing temperature shows a sigmoid curve. It has been considered that the temperature at which dyeing increases rapidly corresponds to the dyeing transition temperature.[8,9] Therefore, with regard to polyester dyeing, it is well known that heat set, carrier, and organic solvent treatments cause a change of internal structure of the fiber and influence the dyeing transition temperature. This kind of behavior is observed for all dyeing systems, independently of dyes and fibers.

Relationship between dye uptake and dyeing temperature showed a sigmoid curve both for acid and disperse dyes as shown in Figures 3, 4, 5, 6 and 7, respectively. The plasma, NH_3, and HP steam-treated wools lowered the dyeing transition temperature with acid dyes. From the results, it seems that the treatment brings about a relaxation of CMC structure and increases dyeing property with acid dyes. On the other hand, in dyeing of the plasma, NH_3, and HP steam-treated wools with disperse dyes, dyeing transition temperature was not affected regardless CMC relaxation.

Dyeing transition temperatures for both dyes are shown in Table IV. The temperature of the

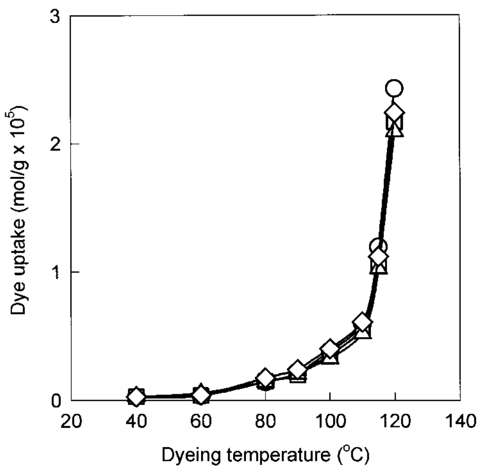


Figure 6 Relationship between dye uptake and dyeing temperature of disperse red 167 : 1 on treated wools: (○) untreated, (□) O_2 plasma, (△) NH_3, (◇) HP steam.

plasma, NH_3, and HP steam-treated wools was decreased with acid dyes considerably, whereas this temperature of disperse dyes has not changed by the pretreatment. It is supposed that water-soluble acid dyes penetrate through the interscale CMC, while water-insoluble disperse dyes diffuse from the compact cuticle surface independently of CMC relaxation.

Table IV Dyeing Transition Temperature of Treated Wools with Acid and Disperse Dyes

Dye	Dyeing Transition Temperature (°C)			
	Untreated	O_2 Plasma	NH_3	HP Steam
Acid red 13	83.0	42.0	50.5	75.0
Acid blue 83	103.5	87.0	93.0	92.7
Disperse Blue 3	77.0	74.0	74.0	73.5
Disperse red 167 : 1	107.2	107.0	107.2	106.9
Disperse Blue 79	106.0	106.0	106.0	105.5

CONCLUSIONS

Dyeing rate, equilibrium dye uptake of the O_2 plasma, NH_3, and HP steam-treated wools increased for acid dyes, but not changed for disperse dyes compared with an untreated wool. Dyeing transition temperature of wool was decreased by the O_2 plasma, NH_3, and HP steam treatments with acid dyes, whereas the temperature was not changed with disperse dyes independent of the pretreatments. Therefore, it seems that acid dye penetrates from the interscale CMC, and disperse dye diffuses through the cuticle surface, regardless relaxation of CMC by the O_2 plasma, NH_3, and HP steam treatments.

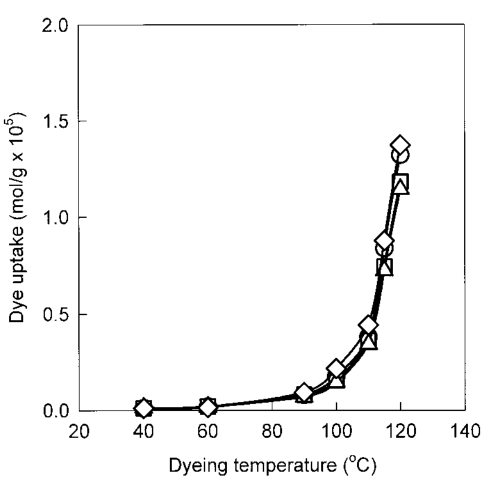


Figure 7 Relationship between dye uptake and dyeing temperature of disperse blue 79 on treated wools: (○) untreated, (□) O_2 plasma, (△) NH_3, (◇) HP steam.

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