Influence of Urea on the Coloring Ability of a Low-Temperature Coloring Method of Keratin Fibers Using Polyethyleneimine

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Received 12 May 2003; accepted 11 August 2003

ABSTRACT: For the purpose of improving the coloring ability of keratin fibers at a lower temperature, we investigated the influence of urea on the coloring ability of a low-temperature coloring method using polyethyleneimine (PEI) as a counter ion reagent (the human hair was previously treated with a PEI solution, and then was colored with acid dye). The coloring and color fastness to shampooing of the hair pretreated with a PEI solution containing urea clearly improved compared with those pretreated with a PEI solution not containing urea. Also, we prepared cross-sectional samples of the treated hair and investigated the penetration of PEI and Orange II into human hair by optical microscopy. The results showed that the penetration of PEI and Orange II into human hair pretreated with a PEI solution that contained urea clearly increased compared with those pretreated with a PEI solution that did not contain urea. From these experiments, we concluded that urea acts as a penetration accelerator for PEI; and PEI, which penetrates deeper into human hair by adding urea, exerts counter ionization on Orange II, thus increasing the penetration of Orange II into human hair and thereby improving the coloring ability. © 2004 Wiley Periodicals, Inc. J Appl Polym Sci 91: 3827–3834, 2004

Key words: fibers; dyes/pigments; diffusion; color fastness; optical microscopy

INTRODUCTION

Urea has been known and used as a dyeing assistant of wool for low-temperature dyeing. Numerous reports in the literatures have been published on the interaction of urea with dyes and keratin fibers.[1–8] It is considered that the function of urea in low-temperature dyeing is the disaggregation of anionic dyes,[1] the swelling of the wool,[2,3] and removal of a portion of the hydrophobic material from the surface of the cuticle layer.[4,5] However, the actual function of urea cannot be clarified because of the complexity of its mechanism. Of all of these functions, an identical opinion concerning the swelling of the wool has not been presented.[6] Also, urea has been known not only to improve the whiteness of wool but also for the reduction effect for human hair when using urea with a bleaching agent or a reduction agent.[7,8] On the other hand, polyethyleneimine (PEI) can be adsorbed into hair, and almost completely remain in the hair even after washing in distilled water. Because PEI can be strongly adsorbed into damaged hair, PEI has been used in shampoos and rinses to improve the effects of hair conditioning and hair styling.[9] In a previous study, we devised a low-temperature coloring method using polyethyleneimine (PEI) as a counter ion reagent (the human hair was previously treated with PEI solution, and then was colored with acid dye).[10]

In this work, for the purpose of improving the coloring ability of keratin fibers at a lower temperature, we investigated the influence of urea on the coloring ability of a low-temperature coloring method using PEI. Next, we investigated the influence of urea on the penetration of PEI and Orange II into human hair by optical microscopy.

EXPERIMENTAL

Materials

Virgin Chinese white hair (average diameter: 70.6 μm) as a keratin fiber was purchased from Staffs Co. The hair was cleaned with a 0.5 wt % sodium lauryl sulfate solution (SLS). After washing in distilled water, the hair was dried at room temperature. Polyethyleneimine (PEI: number-average molecular weight: 600, 1200, 20,000) was supplied by Nippon Shokubai Co. (Japan). Tissue-Tek O.C.T.4583 Compound (Sakura Fine Technical Co.) was used as an embedded resin to make up the fiber cross section. Also, urea, Orange II as an acid dye, *N*-methyl-2-pyrrolidon (MP), 25 wt % ammonia solution, 35 wt % hydrogen peroxide, and citric acid were purchased from Wako Pure Chemical

Journal of Applied Polymer Science, Vol. 91, 3827–3834 (2004)

Industries (Osaka, Japan). Sodium polyoxyethylene lauryl ether sulfate (3 E.O.) (25 wt %) was purchased from Kao Co.

Preparation of bleached human hair

Bleached human hair was prepared according to Tate's method.[11] The virgin white hair was immersed in a solution of 6.0 wt % hydrogen peroxide (adjusted pH 10.1; ammonia water) at a hair : solution ratio of 1 : 50. The hair was soaked for 60 min at room temperature. After sufficiently washing in distilled water, the bleached hair was dried at room temperature.

Preparation of hair treated with PEI and its hair coloring method

The treatment steps of the human hair are shown in Table I. The treatment of the human hair treated with PEI is the first step only. The coloring procedure is the next step after the pretreatment procedure. The detailed procedures are as follows.

The above virgin human hair and bleached human hair were immersed in a solution of 10 wt % PEI with varying molecular weights (number-average molecular weight: 600, 1200, 20,000) and 5.0 wt % *N*-methyl-2-pyroridon (MP) at a hair : solution ratio of 1 : 15. The hair samples were soaked at 50°C and pH 11.1 for 15 min (PEI treatment procedure). After washing in distilled water for 1 min, the colored hair samples were prepared by the following procedures. The hair samples were immersed in a solution of 0.1 wt % Orange II and 4.0 wt % citric acid at a hair : solution ratio of 1 : 15. The hair was soaked at 26°C and pH 2.53 for 20 min. After washing in distilled water for 1 min, the hair samples were dried at room temperature (coloring procedure). Here, the PEI-treated hair samples were prepared to examine the penetration of the PEI by washing in distilled water for 1 min and drying at room temperature, after doing the PEI treatment procedure.

Also, as a control, samples 1 and 7 treated with MP were prepared by the following procedures. The above virgin human hair and bleached human hair were immersed in a solution of 5.0 wt % MP at a hair : solution ratio of 1 : 15. The hair samples were soaked at 50°C for 15 min, and then washed in distilled water for 1 min (MP treatment procedure). Finally, the coloring procedure was done. Here, the MP-treated hair samples (control) were prepared by washing in distilled water for 1 min and drying at room temperature, after completing the MP treatment procedure.

Preparation of hair treated with PEI containing urea and its hair coloring method

The treatment steps of the human hair are shown are Table I. The treatment of the human hair treated with PEI containing urea was done in two steps. The col-

TABLE I
Treatment Steps of Hair Samples

Sample	No.	Step 1	Step 2
Virgin hair	1	MP treatment (Control)[a]	—
	2	Urea treatment (Control)[b]	—
	3	PEI (M_w = 600) treatment[c]	—
	4	Urea treatment	PEI (M_w = 600)–urea treatment[d]
	5	PEI (M_w = 20,000) treatment	—
	6	Urea treatment	PEI (M_w = 20,000)–urea treatment
Bleached hair	7	MP treatment (Control)	—
	8	Urea treatment (Control)	—
	9	PEI (M_w = 600) treatment	—
	10	Urea treatment	PEI (M_w = 600)–urea treatment
	11	PEI (M_w = 1200) treatment	—
	12	Urea treatment	PEI (M_w = 1200)–urea treatment
	13	PEI (M_w = 20,000) treatment	—
	14	Urea treatment	PEI (M_w = 20,000)–urea treatment

[a] Treated with a solution of 5.0 wt % *N*-methyl-2-pyroridon (MP) at 50°C and at a hair : solution ratio of 1 : 15 for 15 min.
[b] Treated with a solution of 20 wt % urea and 5.0 wt % MP at 50°C and at a hair : solution ratio of 1 : 15 for 60 min.
[c] Treated with a solution of 10 wt % PEI and 5.0 wt % MP at 50°C and at a hair : solution ratio of 1 : 15 for 15 min.
[d] Treated with a solution of 10 wt % PEI, 20 wt % urea, and 5.0 wt % MP at 50°C and at a hair : solution ratio of 1 : 15 for 15 min.

oring procedure follows the treatment procedure. The detailed procedures are as follows.

The above virgin human hair and bleached human hair were immersed in a solution of 20 wt % urea and 5.0 wt % MP at a hair : solution ratio of 1 : 15. The hair samples were soaked at 50°C for 60 min. After washing in distilled water for 1 min, the hair samples were immersed in a solution of 10 wt % PEI with varying molecular weights (number-average molecular weight: 600, 1200, 20,000), 20 wt % urea and 5.0 wt % MP at a hair : solution ratio of 1 : 15. The hair samples were soaked at 50°C and pH 11.1 for 15 min (PEI–urea treatment procedure). After washing in distilled water for 1 min, the colored hair samples were prepared by the above coloring procedure. Here, the hair samples treated with PEI containing urea were prepared to examine the penetration of the PEI by washing them in distilled water for 1 min and drying at room temperature, after completing the above PEI–urea treatment procedure.

Also, as a control, samples 2 and 8 treated with urea were prepared by the following procedures. The virgin human hair and bleached human hair were immersed in a solution of 20 wt % urea and 5.0 wt % MP at a hair : solution ratio of 1 : 15. The hair samples were soaked at 50°C for 60 min, and then washed in distilled water for 1 min (urea treatment procedure). Finally, the coloring procedure was done. Here, the urea-treated hair samples (control) were prepared by drying at room temperature, after completing the above urea treatment procedure.

Evaluation of coloring ability

The difference of the lightness (ΔL^*) (undyed and dyed) and the difference of the total color change (ΔE^*ab) for the colored hair samples were obtained using the spectrocolorimeter (CM-6310D, Minolta). Next, the colored hair samples were immersed in a solution of 2.5 wt % sodium polyoxyethylene lauryl ether sulfate (3 E.O.) at a hair : solution ratio of 1 : 15. The hair samples were soaked at 40°C for 3 h. After washing in distilled water for 1 min, the hair samples were dried at room temperature. Finally, the coloring fastness to shampooing of the hair samples was evaluated by measuring ΔL^* and ΔE^*ab using the spectrocolorimeter. Next, the degree of elusion of Orange II from the colored hair samples was evaluated by measuring the Orange II absorbancy at 487 nm of the eluate solution that was created by immersing the colored hair samples in the sodium polyoxyethylene lauryl ether sulfate solution. This elute solution was diluted to a ratio of 1 : 30 [elute: 2.5 wt % sodium polyoxyethylene lauryl ether sulfate (3 E.O.)], before measuring absorbancy using a UV-2200 spectrophotometer (Shimadzu Co., Kyoto, Japan).

Evaluation of the penetration of Orange II and PEI into human hair

White human hair fibers dyed with Orange II, as described in the previous section, were embedded in a resin (Tissue-Tek O.C.T.4583 Compound) and frozen. The frozen blocks were microtomed on a Leica CM1800 to 10 μm thickness, and the penetration of Orange II into the cross-sectional samples was examined by optical microscopy.

White hair fibers treated with PEI, as described in the previous section, were embedded in a resin (Tissue-Tek O.C.T.4583 Compound) and frozen. The frozen blocks were microtomed on a Leica CM1800 to 10 μm thickness, and mounted on a glass slide. Next, the PEI-penetrated part of the cross-sectional samples were stained with a solution of 0.1 wt % Orange II at room temperature with a syringe. Finally, the penetration of PEI into the cross-sectional samples was examined by optical microscopy.

RESULTS AND DISCUSSION

Influence of urea on coloring ability

A characteristic of PEI is to have many cationic charges, attributed to the large amount of nitrogen in the molecule, different from other cationic compounds and cationic polymers.[9] Thereby, an improvement in the coloring ability can be expected by introducing PEI into human hair before ion binding formation with acid dye. By using this excellent characteristic of PEI, we developed a low-temperature coloring method, in which human hair was previously treated with a PEI solution, and then was colored with acid dye.[10] In this work, for the purpose of further improvement in the coloring ability, we devised a new low-temperature coloring method, in which urea was added into the PEI solution.

The coloring and color fastness to shampooing of the virgin human hair and the bleached human hair dyed with Orange II, after being treated with PEI, with and without urea, are shown in Table II. On varying molecular weights, the coloring and color fastness to shampooing of the hair (samples 3, 5, 9, 11, and 13), pretreated with PEI, clearly improved compared with those of the hair (samples 1 and 7) pretreated with MP. Amazingly, the coloring and color fastness to shampooing of the hair (samples 4, 6, 10, 12, and 14), pretreated with PEI containing urea, improved significantly. On the other hand, the coloring and color fastness to shampooing of the hair (samples 2 and 8), pretreated with only urea, did not significantly improve compared with those of the hair (samples 1 and 7) pretreated with MP. Also, under the same pretreatment conditions, the coloring of the bleached human

TABLE II
Coloring and Color Fastness to Shampooing of the Colored Hair Under Different Conditions

Sample	No.	Coloring ΔE^*ab (ΔL)	Fastness ΔE^*ab (ΔL)	Elusion of Orange II (487 nm)
Virgin hair	1	39.1 (−11.8)	17.0 (−2.9)	0.085
	2	43.7 (−11.1)	23.6 (−3.5)	0.107
	3	50.7 (−13.9)	38.5 (−9.7)	0.184
	4	55.0 (−15.3)	52.1 (−13.2)	0.199
	5	56.4 (−14.6)	42.8 (−10.0)	0.498
	6	57.8 (−15.6)	52.5 (−12.5)	0.451
Bleached hair	7	56.7 (−18.5)	38.3 (−10.8)	0.174
	8	58.9 (−19.7)	40.1 (−11.9)	0.136
	9	62.4 (−20.2)	54.0 (−15.8)	0.155
	10	66.2 (−21.8)	62.8 (−18.8)	0.104
	11	63.3 (−20.0)	53.1 (−15.5)	0.124
	12	65.3 (−21.2)	62.3 (−18.3)	0.130
	13	66.6 (−22.0)	56.6 (−16.9)	0.351
	14	72.0 (−24.5)	69.3 (−20.0)	0.422

hair clearly improved compared with that of the virgin human hair.

Next, the degree of elusion of Orange II from the colored hair samples is compared in Table II. In the case of the virgin human hair, samples 1 and 2 showed no significant difference in the elusion of Orange II. In samples 3 and 4, no significant difference in the elusion was seen. However, in samples 5 and 6 (MW = 20,000), the elusion of Orange II was greatly increased. In the case of the bleached human hair, samples 7 and 8 showed no significant difference in the elusion of Orange II. In samples 9, 10, 11 and 12 (MW = 600 and 1200), no significant difference in the elusion was seen. However, in samples 13 and 14 (MW = 20,000), the elusion of Orange II was greatly increased. Also, the degree of elusion of Orange II from the colored virgin human hair and the bleached human hair treated with PEI containing urea did not change compared with the colored sample treated with PEI not containing urea. Furthermore, the degree of elusion of Orange II from the colored virgin human hair samples 3 and 4 treated with PEI (MW = 600) and PEI (MW = 600) containing urea, respectively, increased compared with the colored samples 1 and 2 (control). On the other hand, the degree of elusion of Orange II from the colored bleached human hair samples 9, 10, 11, and 12 treated with PEI (MW = 600 and 1200) and PEI (MW = 600 and 1200) containing urea, respectively, decreased compared with the colored samples 7 and 8 (control) (the opposite effect was seen in the virgin human hair). It is believed that this phenomenon results from the strong electrostatic interaction of PEI and the bleached human hair.

From this experiment, the coloring and color fastness to shampooing of the hair pretreated with PEI solutions containing urea can be clearly improved compared with those pretreated with PEI solutions not containing urea.

Penetration of Orange II into human hair

To study the influence of urea on a low-temperature coloring method using PEI, we investigated the penetration of Orange II into virgin human hair and bleached human hair by optical microscopy.

The virgin human hair and bleached human hair were treated with 10 wt % PEI solutions, which had varying molecular weights and contained 20 wt % urea after being previously treated with 20 wt % urea, and then dyed with Orange II. The penetration of Orange II was then evaluated by observing the cross-sectional hair samples using optical microscopy. The cross-sectional photomicrograph of the bleached white human hair dyed with Orange II, which was previously treated with 5.0 wt % *N*-methyl-2-pyroridon at 50°C for 15 min (sample 7), is shown in Figure 1. The cross-sectional photomicrograph of the bleached white human hair dyed with Orange II, which was previously treated with 20 wt % urea at 50°C for 60 min (sample 8), is shown in Figure 2. The cross-sectional photomicrograph of the bleached white human hair dyed with Orange II, which was previously treated with 10 wt % PEI (MW = 600) at 50°C for 15 min (sample 9), is shown in Figure 3. The cross-sectional photomicrograph of the bleached white human hair dyed with Orange II, which was previously treated with 10 wt % PEI (MW = 600) containing 20 wt % urea at 50°C for 15 min, after being treated with 20 wt % urea at 50°C for 60 min (sample 10), is shown in Figure 4. The penetration of Orange II

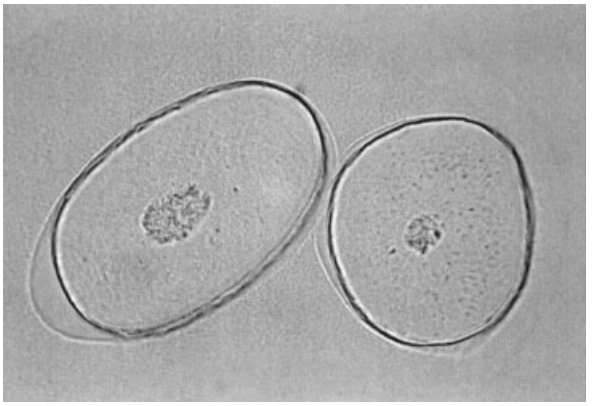

Figure 1 Cross-sectional photomicrograph of bleached white human hair dyed with Orange II, which was treated beforehand with 5.0 wt % N-methyl-2-pyroridon at 50°C for 15 min (sample 7).

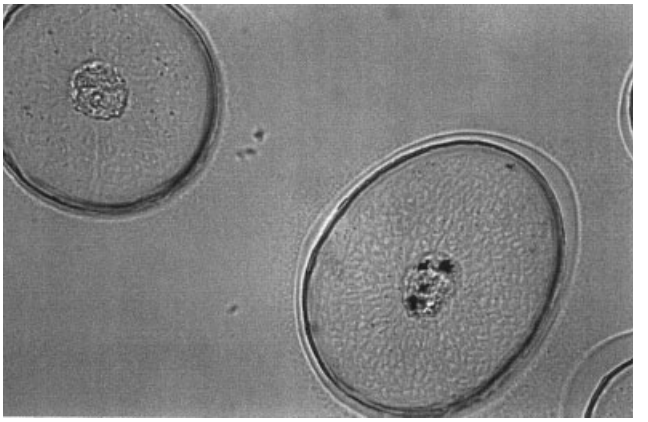

Figure 3 Cross-sectional photomicrograph of bleached white human hair dyed with Orange II, which was previously treated with 10 wt % PEI (Mw = 600) at 50°C for 15 min (sample 9).

into the human hair was increased by treating it beforehand with PEI. On the other hand, the penetration of Orange II into the human hair was not increased by treating it beforehand with urea. However, the penetration of Orange II into the human hair dyed with Orange II, which was previously treated with PEI containing urea, after being treated with urea, was clearly increased compared with that of the hair samples dyed with Orange II, which were previously treated with PEI.

The penetration and the diffusion coefficient of Orange II into the human hair pretreated under different conditions are shown in Table III.

Tate et al.[11] reported the appearance of erosion and a hole in the cuticle face and the increase in the rate of dye (Uranin) diffusion by treating it with hydrogen peroxide. In this study, the penetration of Orange II into the virgin human hair and the bleached human hair dyed with Orange II, which were previously treated with PEI (MW = 600), are compared in Table III. The penetration of Orange II into the human hair clearly increased by treating it with hydrogen peroxide. Also, in the case of the human hair dyed with Orange II, which was treated with PEI only or with PEI containing urea after being treated with urea, the penetration of Orange II into the hair showed an inverse relationship to the molecular weight of the PEI solution used.

Moreover, the diffusion coefficient of Orange II was estimated by eq. (1),[12] assuming that the diffusion of Orange II is a non-Fickian type (Table III):

$$L^2 = 2Dt \tag{1}$$

where L is the distance of penetration, D is the diffusion coefficient, and t is the diffusion time. The diffu-

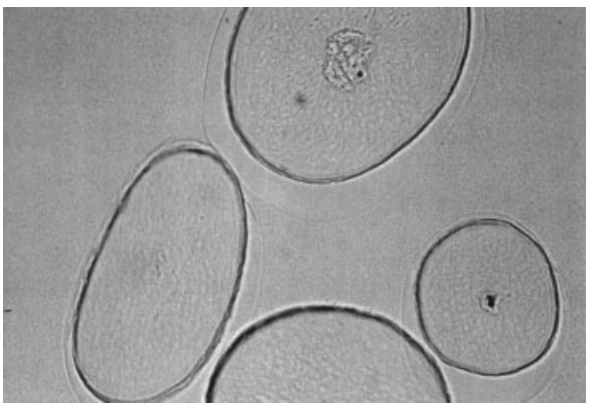

Figure 2 Cross-sectional photomicrograph of bleached white human hair dyed with Orange II, which was previously treated with 20 wt % urea at 50°C for 60 min (sample 8).

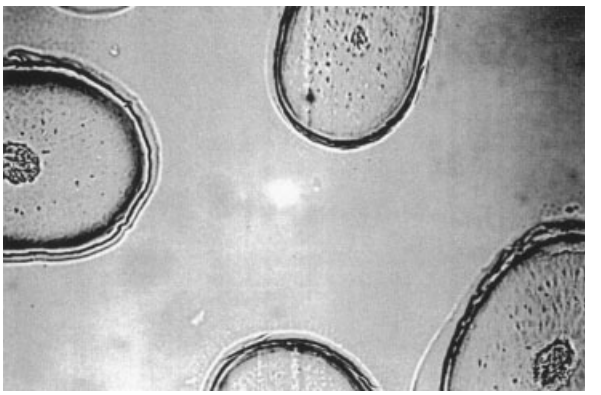

Figure 4 Cross-sectional photomicrograph of bleached white human hair dyed with Orange II, which was previously treated with 10 wt % PEI (Mw = 600) containing 20 % urea at 50°C for 15 min, after being treated with 20 wt % urea at 50°C for 60 min (sample 10).

TABLE III
Penetration and Diffusion Coefficient of Orange II Under Different Conditions

Sample	No.	Distance of penetration L (μm)	Diffusion coefficient D (cm^2/s)	Diffusion coefficient $D_{Average}$ (cm^2/s)
Virgin hair	3	1.5 ~ 1.9	$9.38 \times 10^{-12} \sim 1.50 \times 10^{-11}$	1.22×10^{-11}
	4	2.2 ~ 3.7	$2.02 \times 10^{-11} \sim 5.70 \times 10^{-11}$	3.86×10^{-11}
Bleached hair	7	0.93 ~ 1.9	$3.60 \times 10^{-12} \sim 1.50 \times 10^{-11}$	0.93×10^{-11}
	8	0.93 ~ 1.9	$3.60 \times 10^{-12} \sim 1.50 \times 10^{-11}$	0.93×10^{-11}
	9	1.9 ~ 3.7	$1.50 \times 10^{-11} \sim 5.70 \times 10^{-11}$	3.60×10^{-11}
	10	5.7 ~ 9.3	$1.35 \times 10^{-10} \sim 3.60 \times 10^{-10}$	24.8×10^{-11}
	11	1.9 ~ 3.7	$1.50 \times 10^{-11} \sim 5.70 \times 10^{-11}$	3.60×10^{-11}
	12	2.6 ~ 7.4	$2.81 \times 10^{-11} \sim 2.29 \times 10^{-10}$	12.9×10^{-11}
	13	1.5 ~ 2.8	$9.38 \times 10^{-12} \sim 3.27 \times 10^{-11}$	2.10×10^{-11}
	14	2.2 ~ 4.1	$2.02 \times 10^{-11} \sim 7.00 \times 10^{-11}$	4.51×10^{-11}

sion rate of Orange II into the human hair dyed with Orange II, after being treated with PEI containing urea, was improved 2.1–6.9 times compared with that of the colored hair treated with PEI only.
From this experiment, Orange II was clearly observed to penetrate deeper into the human hair by treating the hair beforehand with PEI containing urea.

Penetration of PEI into the human hair

PEI is a branched polymer that has many cationic charges attributed to the large amount of nitrogen in the molecule.[9] So, the penetration of PEI into the human hair can be observed by dyeing PEI-penetrated parts with Orange II.[13]

It has been suggested that adding urea into a PEI solution plays an important role in the increase in penetration of Orange II into human hair. With this relationship in mind, we investigated the influence of urea on the penetration of PEI into human hair. Here, urea sufficiently penetrated into human hair by treating it beforehand with 20 wt % urea at 50°C for 60 min. Next, we prepared the cross-sectional samples of human hair treated with 10 wt % PEI containing 20 wt % urea. Finally, the penetration of PEI for the cross-sectional samples dyed with Orange II was estimated by optical microscopy. The photomicrograph of the bleached white human hair treated with 5.0 wt % MP at 50°C for 15 min, then cross-sectioned, and finally dyed with Orange II is shown in Figure 5. The photomicrograph of the bleached white human hair treated with 20 wt % urea at 50°C for 60 min, then cross-sectioned, and finally dyed with Orange II, is shown in Figure 6. Both the bleached human hair sample treated with 5.0 wt % MP and the bleached human hair sample treated with 20 wt % urea slightly adsorbed Orange II into the cuticle surface, but did not adsorb Orange II into the cortex. This suggests that the penetration of PEI into the human hair can be observed without the influence of urea by using this method.

The penetration of PEI estimated by optical microscopy for the cross-sectional samples dyed with Orange II is shown in Table IV. The penetration of PEI into virgin human hair and bleached human hair clearly increased by adding 20 wt % urea in all cases, despite varying PEI molecular weights (600, 1200, 20,000), except for the virgin human hair sample treated with PEI (MW = 20,000).

From this experiment, urea clearly acts as a penetration accelerator for PEI. Also, the penetration of PEI into the bleached human hair treated with PEI (MW = 600) clearly increased compared with that of PEI into the virgin human hair treated with PEI (MW = 600). The increase in penetration of PEI by treating it with hydrogen peroxide could be attributable to a breakdown of the barrier function of the cuticle layers, or to a change in the structure of the cortex by decreas-

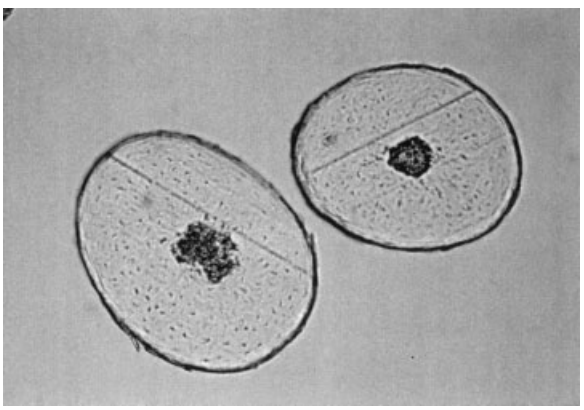

Figure 5 Photomicrograph of bleached white human hair treated with 5.0 wt % N-methyl-2-pyroridon at 50°C for 15 min, then cross-sectioned, and finally dyed with Orange II.

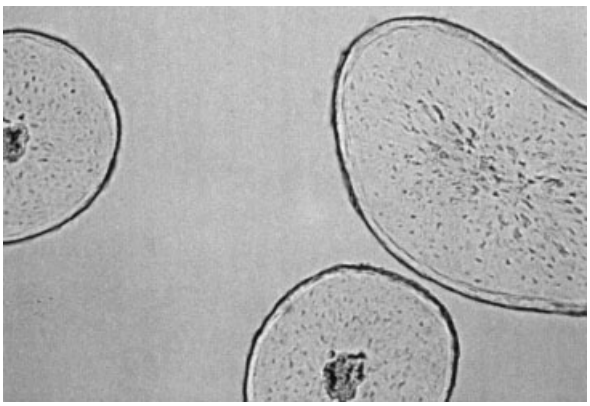

Figure 6 Photomicrograph of bleached white human hair treated with 20 wt % urea at 50°C for 60 min, then cross-sectioned, and finally dyed with Orange II.

ing the crosslink density in the matrix and increasing fiber swelling. Further, the penetration of PEI did not change by increasing the molecular weight of PEI to 20,000 despite the above structural changes observed by treating it with hydrogen peroxide. However, the penetration of PEI having a molecular weight of 20,000 into the bleached human hair increased by adding 20 wt % urea into the PEI solution. This suggests that the change in structure of the keratin fibers was significantly influenced by treating it with PEI containing urea.

Coloring mechanism of keratin fibers using PEI and urea

The penetration of PEI into the virgin human hair and the bleached human hair samples treated with 10 wt % PEI (MW = 600) and PEI containing 20 wt % urea at 50°C for 15 min is shown in Table V. Also, the penetration of Orange II for the virgin human hair and the bleached human hair dyed with 0.1 wt % Orange II at 26°C for 20 min, after being treated with 10 wt % PEI (MW = 600) only and PEI containing 20 wt % urea at 50°C for 15 min, is shown in Table V. It is seen that the penetration of PEI and Orange II into the virgin human hair and the bleached human hair increased by adding 20 wt % urea.

TABLE IV
Penetration of PEI Estimated by Optical Microscopy for Cross-Sectional Samples Dyed with Orange II

	Penetration (μm)			
	Virgin hair		Bleached hair	
M_w of PEI[a]	N/C[b]	Containing urea	N/C	Containing urea
600	4.63	6.48	6.02	10.2
1200	—	—	3.25	6.94
20,000	2.78	2.78	2.78	4.63

[a] PEI and PEI–urea treatment: 50°C, pH 11.1, 15 min. Here, in the case of samples treated with 10 wt % PEI containing 20 wt % urea, they were previously treated with 20 wt % urea at 50°C for 60 min.
[b] Not containing.

TABLE V
Penetration of PEI (M_w = 600) and Orange II into Virgin and Bleached Human Hair

			Penetration (μm)	
Sample	No.	Containing 20 wt % urea	PEI[a]	Orange II[b]
Virgin hair	3	N/C[c]	4.63	1.70
	4	Containing	6.48	2.95
Bleached hair	9	N/C	6.02	2.80
	10	Containing	10.2	7.50

[a] PEI and PEI–urea treatment: 50°C, pH 11.1, 15 min. Here, in the case of samples treated with 10 wt % PEI containing 20 wt % urea, they were previously treated with 20 wt % urea at 50°C for 60 min.
[b] Treated with 10 wt % PEI (M_w = 600) at 50°C for 15 min, and then 0.1 wt % Orange II at 26°C for 20 min. Here, in the case of samples treated with 10 wt % PEI containing 20 wt % urea, they were previously treated with 20 wt % urea at 50°C for 60 min.
[c] Not containing.

We reported that the PEI molecular weight had a slight influence on the penetration of Orange II (6.95 μm) compared with the significant influence on the penetration of PEI (MW = 300; 23.2 μm).[10] On the other hand, the penetration of Orange II into the virgin human hair and the bleached human hair increased by increasing the penetration of PEI (Table V). This suggests that the electrostatic interaction between PEI and Orange II was strengthened by increasing the molecular weight of PEI.

From these experiments, we concluded that urea acts as a penetration accelerator for PEI; and PEI, which penetrates deeper into human hair by adding urea, exerts counter ionization on Orange II, thus increasing the penetration of Orange II into human hair and thereby improving the coloring ability.

CONCLUSIONS

The coloring and color fastness to shampooing of the hair pretreated with a PEI solution containing urea were clearly improved compared with those pretreated with a PEI solution not containing urea. To clearly identify the cause of this, we investigated the penetration of PEI and Orange II into human hair by optical microscopy. The results showed that the penetration of PEI and Orange II into human hair pretreated with a PEI solution that included urea clearly increased compared with those pretreated with a PEI

solution that did not contain urea. From these experiments, we concluded that urea acts as a penetration accelerator for PEI; and PEI, which penetrates deeper into human hair by adding urea, exerts a counter ionization effect on Orange II, thus increasing the penetration of Orange II into human hair and thereby improving the coloring ability.

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