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Dye Migration Influences on Colour Characteristics of Wool Fabric Dyed with Acid Dye

Abstract

The influence of non-ionic auxiliaries on absorption of the acid milling CI Acid Blue 80 dye on wool fibre and on the colour changes of dyed fabric has been investigated. The kinetics of dye absorption and the colour characteristics of dyed fabric, including washing fastness, were determined. A non-ionic surfactant based on poly-ethoxylated alkylphenol enables dyeing at low temperatures, colour characteristics and wash fastness to be improved, whereas the alkylaminoethoxylated product enhances the dyeing rate at higher temperatures. The variation of colour yield values in the dyed fabric which occur due to the use of non-ionic auxiliaries or the difference in dyeing temperatures is explained and discussed.

Key words: *dye migration, wool, auxiliaries, colour difference.*

◼ Introduction

Higher-affinity dyes, such as acid milling dyes with larger molecules, do not penetrate into the wool fibre at lower temperatures, and dyeing unlevelness or ring dyeing becomes a major problem. Ring dyeing can give problems such as reduced fastness, shade change, or poor rubbing fastness. Localised unlevelness is primarily related to the physical and/or chemical non-uniformity of the substrate; a typical example of this is skitteriness in wool dyeing. One of the fundamental mechanisms that can contribute to level dyeing is the migration of dye after initially unlevel sorption into the fibre [1].

As a fibre, wool is heterogeneous, both chemically and physically. In the case of wool without the damage in cuticle cells, it is assumed that the dye uptake in the early stage of adsorption is subject to the dyeing behaviour of the intercellular regions of the cuticle-cuticle (surface) layer [2]. Dye most readily enters the fibres by diffusion through the intercellular region between the scale cells of the wool fibre, penetrates fairly rapidly into the non-keratinous endocuticle region of the surface layer, and soon reaches equilibrium with the dye in the outer solution. Within the cuticle cells, the endocuticle and then the exocuticle become coloured, as the dye travels through the intercellular cement and penetrates into the cells from their undersides [2].

It has been established that an increase in dye bath exhaustion and the improved levelness of dyeing with selected milling acid dyes could be obtained when sparingly water-soluble organic compounds are added to the dye bath, or wool fibres are pre-treated before dyeing [3-8]. Riva et al have investigated the influence of pre-treating wool with a non-ionic auxiliary on dye sorption, and assumed that the enhancement of the increase in dye bath exhaustion and the faster rate of dyeing is due to the sorption of the auxiliary by the wool [4]. The partial solubilisation of free fatty acids of a lipid layer of cellular membrane complex (CMC) due to the treatment of the wool with non-ionic polyglycol ethers and the modification of the hydrophobic domains of the CMC in wool fibres were proved by Coderch et al [5].

In a previous paper, we reported on the influence of applied surfactants on wool fibre dyeing with acid milling dye at a low temperature, considering its chemical and morphological heterogeneity. In that study, we have found that the presence of non-ionic auxiliaries in the dye bath causes a reduction of the surface barrier to the dye diffusion into the fibre [9]. It was suggested that when 1.5% of dyeing with acid-milling Acid Blue 80 (Figure 1) is proceeded, the dye is transferred from the endocuticle to the sulphur-rich exocuticle rather than into the cortex. As the non-ionic nonylphenol ethoxylate (NPE) used as the dyeing auxiliary tends to be strongly and more quickly adsorbed than the dye in the inner layers of wool fibre, we hypothesised that it may cause blocking of the hydrophobic sites of fibre, and thus prevent the immobilisation of dye during the initial stage of dyeing.

In the present work we examine the influence of how acid dye is taken up by wool fabric on the changes in colour characteristics that occur thanks to applied non-ionic auxiliary dyeing agents and temperature.
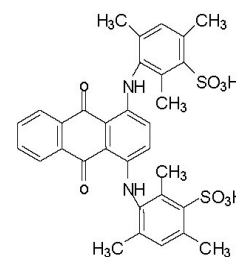

Figure 1. *Chemical formula of C. I. Acid Blue 80 dye.*

■ Experimental

Materials

The initial merino wool fabric corresponds to ISO 105/F: 1985 (E). The dye used in this study was CI Acid Blue 80 (Sandolan Milling Blue N-BL 150), supplied by Clariant and purified by recrystallisation three times: dissolving in hot dimethylformamide, precipitation by acetone, and then filtered.

The dyeing auxiliaries non-ionic products, ethoxylated alkylnonylphenol and the dispersing agent mixture Lanasan LT (NPE), are recommended for use in low-temperature wool dyeing (Clariant); alkylaminoethoxylate and the glycolic ether mixture (AAE) Rucogal MSC (Rudolf Chemie) are recommended for use in wool and polyamide levelling dyeing. The other analytical-grade reagents were used without purification.

Dyeing

Before dyeing, the fabric samples were immersed for one hour in 0.1M acetic acid buffer adjusted to a pH of 4.2±0.05. We measured the dyeing rates in a dye bath with an initial concentration of dye of 1.7×10^{-4} mol/l or 2.3% owf. The dyeing took place in a laboratory apparatus with a magnetic bar stirrer and cylinder to girdle the 10×20 cm fabric sample with dyeing liquor at a pH of 4.2±0.05. After dyeing, the dye bath's pH was unchanged. In all dyeing, we adopted the liquor-to-fibre ratio of 200:1 and a temperature of 60 °C or 85 °C. Dyeing was carried out both in the absence and presence of auxiliaries (1.8% owf) up to the dye absorption equilibrium. At the end of the dyeing process, the samples were rinsed thoroughly in water and dried in open air. Conventional dyeing in the presence of electrolyte was carried out using 10% owf sodium sulphate and 4% owf ammonium sulphate. Dye concentration in the dye bath was measured at the start, after 5, 10, 15, 20, 30, 45, 60, 90 minutes, and up to the dye's exhaustion.

Dyeing characteristics

Dye absorption was calculated from the difference in dye concentration before and after dyeing time t. Dye concentration in liquor was measured in a 10-mm cell using the Spectronic Unicom Genesys TM spectrophotometer. On completion of dyeing, the wool fabric samples were removed from the liquor, rinsed in water to remove any loosely adsorbed dye, and dried in air.

The percentage dye bath exhaustion E was calculated according to Equation 1:

$$E = \left(\frac{A_0 - A_d}{A_0} \right) * 100 \qquad (1)$$

where A_o and A_d are the adsorbances at 590 λ_{max} of Acid Blue 80 dye, initially in the dye bath and of residual dye in the dye bath respectively.

For the dyed fabric samples, the light diffuse reflectance measurements were performed with a Spektroflash SF 450 Plus spectrophotometer (Datacolour), and the colour yield values were calculated according to the Kubelka-Munk function (K/S) by Equation 2 [10]:

$$K/S = \frac{(1-R)^2}{2R} \qquad (2)$$

where:
  K - absorbance,
  S - scattering,
  R - reflectance.

Evaluating the colour parameters

Samples for colour measurements were prepared by folding the dyed wool fabric five times to give samples measuring about 3×3cm. Five points on each trial sample were measured using a spectrophotometer. The colour parameters were evaluated by means of the Cielab system and the modified CIE L* C* h* (D65/10º) system.

The following colour parameters for the dyed samples were obtained by the digital Cielab system: L* – lightness, a* – redness if positive coordinate, or greenness if negative coordinate, b* – yellowness if positive coordinate, or blueness if negative coordinate, C* – chromaticity, h – hue of the colour, X – coordinate x, Y – coordinate y, Z – coordinate z [10].

The values of colour difference (ΔE) for the dyed samples after relative treatments were calculated by Equation 3 [10]:

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

where:
$\Delta L^* = L^*_{trl} - L_{in}$,
$\Delta a^* = a^*_{trl} - a^*_{in}$,
$\Delta b^* = b^*_{trl} - b^*_{in}$
L^*_{trl}, a^*_{trl}, and b^*_{trl} are parameters of trials, i.e. after blank dyeing (treatment of undyed fabric under dyeing conditions, but without using dye) or after washing the dyed fabric samples.

L_{in}, a^*_{in}, and b^*_{in} are parameters of initial species before the relevant treatments.

The ΔE deviation of all treated samples was no higher than 0.2. The coordinates of the colours obtained and the results of the ΔE value determinations are summarised in Tables 1-3.

The yellowness index YI of the wool fabric samples in the blank dyeing trials was calculated using equation 4 [10]:

$$YI = 100 \cdot \frac{1.28X - 1.06Z}{Y} \%, \qquad (4)$$

where: X, Y, and Z are the C/2 CIE tristimulus values of the specimen.

The level of wool fibre damage occurring during the blank dyeing of the wool fabric was determined by means of tryptophan fluorescence intensity changes. The fluorescence spectra were measured with an MPF-4 fluorescence spectrophotometer (HITACHI). Samples of the blank dyed wool fabric signals (λ_{ex}=295 nm, λ_{ex}=346 nm) were measured and quantified by comparison with those of the initial untreated fabric.

The dyeing rate constants were calculated using a modified Cegarra-Puente equation (5) suitable for exhaustion baths [11]:

$$\ln\left[-\ln\left(1 - \frac{C_t^2}{C_\infty^2} \right) \right] = a \ln t + a \ln K \qquad (5)$$

where a is a coefficient depending on exhaustion, C_t is the dye concentration in the fibre at time t, C_∞ is the dye concentration at the equilibrium, K is the absorption rate constant, and t is the dyeing time.

Determining fastness

The samples were washed using an Isal AG 4006 Basel/Schweiz apparatus with rotating stainless-steel hermetic autoclaves, according to standard ISO 105/C06 A1S at 40 °C for 30 min. Colour difference (ΔE) and colour yield (K/S) data were obtained with a Datacolour SF450 Plus apparatus under illumination D65 using 10° standard observation angle. Each sample was measured five times and the average values were calculated.

■ Results and discussions

Dyeing rate

Figures 2 and 3 show the plots of the dyeing rate and relative dye uptake

Table 1. *Effect of auxiliaries and temperature on dye exhaustion (E, %), kinetic constant (K)and on colour yield of wool fabric; Colorimetric measurements data of fabric dyed using auxiliaries;Abbreviations given in Figure 1.*

Fabric	Dyeing time, min	Exhaustion, E, %	$t_{1/2}$, min	$K10^{-3}$, min^{-1}	K/S value	L*	a*	b*	C*
Dyed at 60 ºC									
A	300	98.70	48.4	1.86	21.95	28.45	4.83	-38.74	39.04
NPE	240	100.00	32.3	2.45	26.63	25.90	7.49	-39.76	40.46
AAE	270	97.05	46.0	1.56	22.16	28.85	4.77	-39.49	39.77
EL	600	77.30	150.0	0.35	19.34	30.55	3.49	-39.11	39.27
Dyed at 85 ºC									maž.
A	150	99.00	9	10	28.25	24.82	5.85	-37.42	37.87
NPE	90	99.40	6	16	28.42	25.05	6.77	-38.74	39.33
AAE	90	99.70	4	26	29.31	24.68	7.71	-39.70	40.44
EL	360	97.10	19	2.7	26.18	25.96	3.80	-36.27	36.47

C_t/C_∞ against the square root of dyeing time $t^{1/2}$ for dyeing in solution without any auxiliaries (A) and in the presence of dyeing organic auxiliaries (NPE or AAE) or electrolyte (EL) respectively. The values for the kinetic constant K in the absence and in the presence of auxiliaries are presented in Table 1. It is clear from these results that the application of the non-ionic surface active agent NPE for dyeing at 60 °C leads to a considerable increase in the apparent dye uptake, while in the presence of weakly cationic alkylaminoethoxylate AAE, the rate of dye absorption was even lower compared with that for dyeing without auxiliary. The application of the dyeing auxiliary NPE as well as AAE makes the whole quantity of dye to penetrate the fibre at 85 °C more readily as suggested by the dye exhaustion and dyeing rates.

From Figure 2 and Table 1, the NPE at 60 °C temperature can also be seen to cause the higher increase in total dye ab-

sorption and the rate of dyeing, compared with the figures for AAE. As mentioned above, alkoxyethylated nonylphenol has a significant effect on the cellular membrane complex (CMC) of wool fibre [5], while the alkylaminoethoxylates, which combine non-ionic and cationic properties, are capable of forming complexes with acid dyes, facilitate solubilisation, and provide level dyeing of wool [3]. Non-ionic alkylphenolethoxylate-type surfactants, in contrast to alkylaminoethoxylates, are not considered to have a structural effect on the levelling agent, but are known to be adducts which form complexes with wool fibre by means of hydrophobic interaction between hydrophobic sites [12]. Mieusset et al have shown that when dyeing with a 1:2 metal complex dye, a quick initial surfactant absorption by wool occurred regardless of the manner of surfactant application. Surfactant absorption is followed by pronounced desorption, with an increase of temperature and dye exhaustion [13].

Figure 3 shows that the intercept on the dye absorption axis, which proves the existence of a barrier of diffusion, is significantly reduced in the case of NPE; with AAE it did not reduce, compared with that during dyeing in the absence of an auxiliary. The results presented in Figure 1 and Table 1 also show that when dyeing was carried out at 85 °C, the AAE produced a higher increase in the total dye absorption and dyeing rate, particularly in the early dyeing stage.

It is known from the relevant literature [1] that the gradual decomposition of complexes between the levelling agent and the dye can occur when the dyeing temperature increases. Therefore it is assumed that at a higher temperature, the decomposition of AAE and anionic dye complexes could be a factor influencing the renewal of acid milling dye's affinity for wool fibre. The Blockage of electronegative sites of wool, due to the interaction with AAE containing amino-
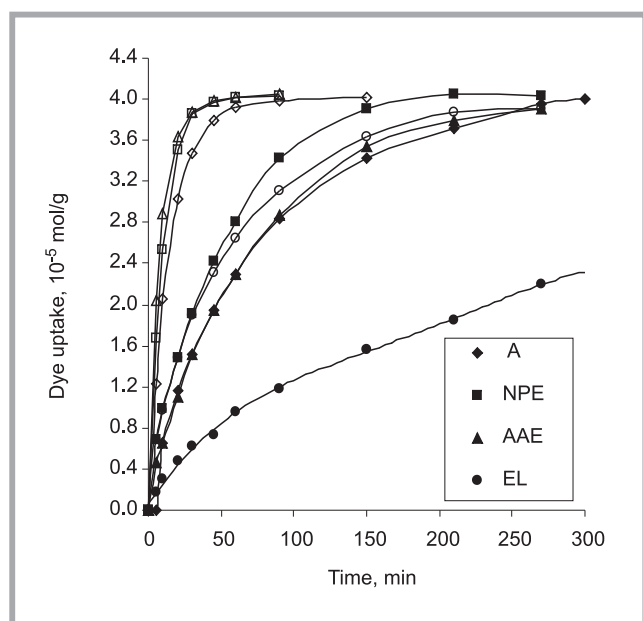

Figure 2. *Dye absorption kinetics; A – Dyeing in the absence of auxiliary ; NPE – Dyeing in the presence of auxiliaries: nonylphenolethoxylate; AAE – alkylamine ethoxylate; EL – Electrolyte; ◆■▲● – 60°C temperature; ◇□△○– 85 °C temperature.*
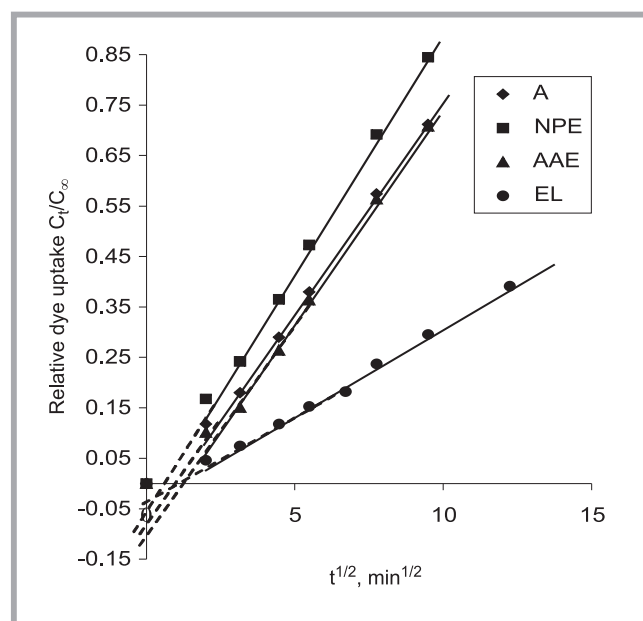

Figure 3. *Relationship of relative dye uptake versus dyeing time ($t_{1/2}$) at 60°C. A – Dyeing in the absence of auxiliary ; NPE – Dyeing in the presence of auxiliaries: nonylphenolethoxylate; AAE – alkylamine ethoxylate; EL – Electrolyte.*

groups could take place in this case, and an increase in the amount of the net positive charge on the protein accessible to dye should therefore also be an important factor in dye absorption.

Colour yield and colour characteristics

In general, the colour yield for dyed wool should be related to the amount of dye in a fibre, albeit only up to a certain dye concentration [10]. The K/S of dyed samples in this study has been measured, and the results are presented in Table 1. The results show that when the fabric samples were dyed at 60 °C, there was a significant variation in the ratio of K/S values and dye exhaustion, which is proportional to the amount of dye in the fibre. The variation of this ratio was small or disappeared completely when dyeing occurred at 85 °C. A comparison of dye exhaustion and colour yield data for the dyeings obtained at 60 °C and 85 °C temperature respectively shows that at the same level of dye exhaustion, for example dyeings at 60 °C and 85 °C temperature respectively, higher K/S values are obtained for samples dyed at higher temperature. This allows us to assume that when the dye absorption on the fibre is faster and exhaustion tends to be completed, the dye is transferred from the endocuticle to the sulphur-rich exocuticle rather than into the cortex, and therefore for dyeings at 85°C, the colour yield was higher in all the cases studied. Our assumption is in agreement with the conclusion made by Wolf et al [14]. Summarising the study of the results by TEM of morphological changes within the fibre of wool during the sorption and diffusion of 1:2 metal complex dyes, these authors have stated that the sorption of dyes or surfactants is accelerated by morphological changes within the cuticle-cellular membrane complex, which is the adsorption zone, or within the main diffusion path endocuticula, and did not depend on the cortex penetration capacity [14].

For the samples dyed at 60 °C, we have studied the effect of auxiliaries on the rate of dyeing and on changes to colour yield (K/S) values. The ratio of K/S values were compared with the ratio of kinetic constant values (K) for corresponding dyeings. The result was surprising, because the ratio of kinetic constant values taken from Table 1 K_{NPE}/K_A and the ratio of respective colour yield values $(K/S)_{NPE}/(K/S)_A$ were equal to 1.3 and 1.2 respectively. In other words, this comparison shows that the colour yield of dyed wool fibre depends also on the rate of dye absorption, and not only on the amount of dye in the fibre. The comparison of K and K/S values for NPE with respect to the corresponding values for dyeing, in the absence of auxiliary (K_{AAE}) and $(K/S)_{AAE}$ respectively, show that $K_{NPE}/K_{AAE} =1.5$ and $(K/S)_{NPE}/(K/S)_{AAE} =1.2$. Thus, the results presented above allow us to assume that the increase in the rate of dye uptake should be considered to be due to the facility of increase on the amount of dye in the surface layer cuticle of wool fibre. The approximate calculation of the amount of active sites represented by the basic groups of cuticle proteins should be less than $8.3×10^{-5}$equiv/g. In the cellular-membrane complex and endocuticle jointly, the amount of basic groups should be less than $3.8×10^{-5}$ equiv/g, while the amount of dye used in this study was $4.3×10^{-5}$ mol/g. Thus, the dye absorbed in the wool fibres should correspond to to approximately half the amount of the basic groups considered in the cuticle.

Our results as presented and discussed above are in agreement with the general statement made by Rippon, that for non-reactive dyes, no thermodynamic equilibrium with wool is established until the dye transfer into the keratinous regions is complete. If the dye remains largely in the non-keratinous regions, rapid diffusion out of the fibre can occur, and poor wet fastness properties are obtained [3].

The results presented in Table 1 show that dyeing at 60 °C both without the auxiliary and in the presence of AAE results in lower values of both the Cielab coordinates and the K/S values, compared with those for samples dyed in the presence of NPE. These results are consistent with the values of dye exhaustion and dyeing rate constants for the corresponding dyeings presented in Table 1. Increasing the dyeing temperature up to 85 °C significantly enhanced the redness (a*) of samples dyed in the presence of AAE, while for the samples dyed in the presence of NPE as well as in the absence of the auxiliary, chroma (C*) values were reduced.

Colour difference

The colour differences of dyed wool samples could have been influenced by the increase in wool yellowness or the reduction in the amount of melanin occurring due to damage to the fibre during dyeing. The migration of dye into or out of the fibre during washing or thermal and hydrothermal treatment are also noted to be very important factors in colour change [15]. We therefore attempted to determine whether the colour difference of wool fabric dyed with milling Acid Blue 80 could depend on the extent of fibre damage occurring during dyeing, on the change in the yellowness index value, or on dye migration during dyeing or the washing of dyeings. In this study, we introduced trials with benzyl alcohol, which is known as a very efficient auxiliary for wool dyeing at low temperature, but which unfortunately damages fibres. We expected that benzyl alcohol should be used to determine whether a relationship is reliably predictable between the colour change or the yellowing of fabric and the level of fibre damage occurring during blank dyeing.

Table 2 shows the colour difference and yellowness index values of the blank dyed fabric samples, which have been prepared while maintaining all the other selected conditions of dyeing, with the exception that the dye was not applied in these trials. As could be seen, the extent of colour change was higher for dyeings obtained without the auxiliary. The most significant colour change was when dyeing occurred in the presence of benzyl alcohol, which was used in this study for comparison. The data of fluorescence emission spectroscopy of the blank dyed wool fabric samples shows a marked

***Table 2.** Colour difference and fibre damage by means of fluorescence emission intensity changes in blank dyed wool fabric; Abbreviations given in Figure 2. • 3% v/v of benzyl alcohol was added into the bath of blank dyeing.*

Dyeing auxiliary and temperature	Time of treatment, min	ΔE	Yellowness index YI	Tryptophan emission, %
A	150	0.43	9.75	91.8
NPE	90	0.26	10.33	94.5
AAE	90	0.25	10.50	100.0
Benzyl alcohol*	150	0.74	11.30	83.7
EL	150	0.29	10.23	81.3
Initial untreated fabric	0	-	10.12	100.0

reduction in the fluorescence emission of tryptophan; that is, a significant degree of the wool fibre damage was also caused in the sample which had been dyed in the absence of the auxiliary, but longer exposed to dyeing conditions , than those with NPE or AAE. The samples dyed in the presence of benzyl alcohol showed an especially marked lowering in the fluorescence emission of tryptophan, an increase in the yellowness index value and in colour difference. However, the colour difference was not dependent upon the wool fibre damage occurring during blank dyeing in the presence of the electrolyte.It can thus be concluded that neither the increase in yellowness index value of wool fibre nor the fibre damage occurring during dyeing was responsible for the general colour difference of the dyeings due to the auxiliaries applied in this study.

In the light of the findings shown in Table 3, an attempt was made to explain the beneficial effect of temperature and dyeing auxiliaries on colour wash fastness, colour change and colour yield change during the treatment of dyed wool fabric. The corresponding colorimetric data for the dyeings after wash fastness testing are displayed in Table 3. The comparison of the changes in redness (Δa^*), blueness ($\Delta(-b^*)$), chroma (ΔC^*) and colour difference ΔE values for dyed fabric samples show the extent to which each of the different dyeings after washing was changed in terms of colour loss from the original dyeings. Table 3 shows that the extent of changes for the samples dyed while applying NPE at 60 °C were generally small, compared with those for AAE or for dyeings without an auxiliary. The dyeing at 85 °C resulted in an increase of the degree of colour change for AAE, but did not do so for the electrolyte, or for dyeings prepared without an electrolyte. Theoretically, the ability of a surface active levelling agent such as AAE to form

dye water-soluble complexes is an important factor within the fibre. The stability of the AAE-dye complexes which are formed under selected dyeing conditions could be responsible for AAE sorption into the inner layers of the fibre, either alone or together with dye, and therefore a relatively greater colour change due to washing the dyed fabric could occur. As seen from Table 3, the blueness (-b) of dyeings after washing for AAE was significantly higher; that is to say, we could observe the result of dye migration from the inner to the outer layers of the fibre. In the case of control dyeing without auxiliary or dyeing in the presence of electrolyte at a temperature 85 °C, the colour washing fastness by means of colour difference was higher due to the absence of the auxiliary, which can modify the dye solubility and hence leads to a lower dye affinity for wool fibre.

■ Conclusions

The dyeing behaviour of wool at low temperature using acid milling dyes and non-ionic auxiliary agents have been investigated. Our work shows the effect of temperature and auxiliaries on dyeing on acid milling dye equilibrium, rate of dye uptake, colour yield and colour coordinates of dyed wool fabric. The use of a non-ionic auxiliary based on alkylphenolethoxylate enhances both the rate of dyeing and the colour yield of fabric at 60 °C, while the increase in dye substantivity, dyeing rate and colour yield for alkylaminoethoxylate is most important at higher temperatures. Increasing dyeing temperature from 60 °C to 85 °C, or using auxiliaries, leads to a change in the colour yield of the wool fibre, which in general depends on dyeing rate when dyeing is extended up to the equilibrium. From colour coordinate measurement data, it is apparent that the colour changes of the dyed fabric sam-

ples which occur due to the variation of dyeing temperature are most important, presumably due to a variation of the dye-auxiliary complexes' stability and the change of dye affinity for wool, when alkylaminoethoxylate is used as a dyeing auxiliary.

The results of our investigation allow us to suggest that uneven dyeing and colour difference of wool fabric arises from a difference in the rate of dye diffusion into the whole volume of the cuticle cells of the wool fibre. Therefore the capability of selected dyes to migrate from the inner, low-sulphur protein layer of the endocuticle into the outer, exocuticle layer of the wool fibre should be estimated when selecting wool dyeing conditions.

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Table 3. *Changes of colour characteristics after wash fastness testing of fabric dyed using auxiliaries; Abbreviations given in Figure 2.*

Fabric [a]	Δ a	Δ (-b)	Δ C	Δ E
Dyed at 60 °C				
A	0.55	1.09	1.14	1.23
NPE	-0.07	-0.03	-0.04	0.78
AAE	0.55	0.92	0.97	1.09
EL	-0.12	0.24	0.23	1.26
Dyed at 85 °C				
A	0.72	0.25	0.35	0.76
NPE	-0.08	0.73	0.70	0.90
AAE	-1.11	0.91	0.72	2.33
EL	0.01	-0.21	-0.21	0.24