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Solvent Assisted Low Temperature Dyeing. Part I: Results for Mohair (Angora Goat) Fibres

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Abstract
It is generally known that mohair tends to lose its luster when dyed for prolonged periods at the boil, and to preserve its luster it is generally necessary to use short dyeing cycles and low dyeing temperatures. Beyond preserving fibre luster, the dyeing of fibres at low temperature does have some advantages from an industrial point of view, namely saving energy, environmental control and fibre protection during dyeing. However, the problem of low temperature dyeing is that it hinders dye diffusion into the fibre, thus decreasing the colour yield of dyed samples. In this study, by using benzyl alcohol as a dyeing auxiliary at lower temperatures, it was possible to dye fibres at both at 90 °C and 80 °C for all dye classes without causing any decrease in colour yield. Dyeing kinetics were also studied, and it was demonstrated that in the presence of benzyl alcohol, the rate constant and Standard affinity increased.

Key words: *Mohair, low temperature dyeing, benzyl alcohol, solvent, kinetic.*

Introduction

Mohair fibres are "Luxury Fibers" [1] which have superiority in the domain of colour and luster, when compared to wool fibres [2]. As these fibres are also protein based, their dyeing characteristics are similar to those of wool. Mohair fibres can be dyed with conventional dyes used for wool dyeing and natural dyes, which can also be used to dye mohair fibres, but there could be some differences between wool and mohair [3].

It is generally the case that firms which treat mohair also treat wool, and hence similar machinery is used for the two fibres, although often under different conditions. The difference is that special precautions are taken with the aim of preserving the brightness of dyed sample colors, luster and other properties of fibres desired [4]. It is very important to dye mohair fibres at lower temperatures below the boiling point and to limit the time of dyeing at high temperatures so as to curtail any adverse effects on luster and other desirable properties. However, in the case of low temperature dyeing, the dye uptake and colour yield of samples will decrease and the effluent load increase as a result of increasing the amount of dye remaining in the liquor, which is not a desired situation in textile dyeing [3].

In recent years, many attempts have been made to improve fibre dyeability at lower temperatures or for shorter times. A technology which appears to hold considerable promise in this field is radio frequency (RF) dyeing. It was stated in literature that Barkhuysen and Maasdorp found that the dyeing time of mohair fibres could be drastically reduced from the conventional 90 minutes to 35 minutes using the radio frequency technique, with only 5 minutes of the 35 minutes representing actual exposure to the radio frequency field, showing that the luster of samples dyed using RF technology is higher [4].

Turpie (1985) quoted unpublished data in which the radio-frequency dyeing of tops produced better luster and enabled higher maximum spinning speeds than the conventional dyeing of mohair [5]. Smith (1988) claimed that owing to radio-frequency dyeing and bleaching, chemical damage is reduced because of the shorter exposure to 100 °C, leading to a decrease in energy and water consumption and effluent costs [6].

Carrion (1995) showed that dyeing at low temperatures without affecting dyeing diffusion and levelness is also possible with the aid of solvents. In his study, polyester was dyed in the presence of methylene chloride at 40 ºC, and wool was dyed at lower temperatures in the presence of n-butanol and benzyl alcohol [7]. Queirós Domingues et al (2003) showed that in the case of polyamide 6.6 microfibres, by using benzyl alcohol as a dyeing auxiliary, good diffusion could be obtained for metal complex dyes at temperatures below the normal dyeing temperature [8].

In this study we examined the usage of benzyl alcohol as a dyeing auxiliary for lowering the dyeing temperature without

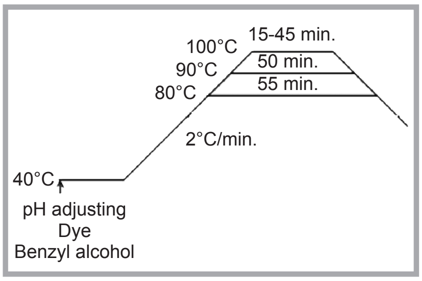

Figure 1. *Dyeing profile of mohair dyed with different dyes at either 80 °C, 90 °C or 100 °C.*

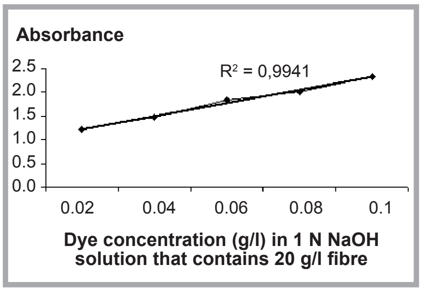

Figure 2. *Calibration curve of absorbance measured at 590 nm (λmax) against the dye concentration of Isolan Dark Blue 2S-GL.*

causing any decrease in the colour yields of dyed samples. This method of using benzyl alcohol is well known in the low temperature dyeing of wool fibre. It was first described by Peters et al [9, 10], and Beal et al investigated the influence of benzyl alcohol on the normal processes of wool dyeing and printing in detail [11]. The Ciba-Geigy company then proposed the Irga-solvent processes [12]. The new aspect of the present study is its usage in the dyeing of mohair fibres.

Material and Method

Mohair fibre of 31.47 μm mean fibre diameter was used in all experiments, supplied by IPLIKSAN A.S. / Turkey. The dyes used were Telon Blue BRL Micro (C.I. Acid Blue 324) (acid leveling dye), Telon Blue M-RLW (C.I. Acid Blue 204) (acid milling dye), Isolan Dark Blue 2S-GL (C.I. Acid Blue 193) (1:2 metal complex dye), and Realan Blue RC (reactive dye), kindly supplied by Dystar. All the experiments were carried out using soft mill water and repeated twice.

Determination of the effect of benzyl alcohol usage in the dyeing of mohair with different dye classes

In this study, the dye-uptake properties of mohair fibres with various dye classes at different temperatures in the presence (5-15-30 ml/l)) and absence of benzyl alcohol were tested. Dyeing procedures were carried out at a liquor to goods ratio of 30:1 and dyeing depth of 3% owf following the dye profile illustrated in ***Figure 1***. Mohair fibre was dyed with acid leveling dye (Telon Blue BRL Micro) at pH 3 using formic acid. Dyeing with acid milling (Telon Blue M-RLW) and reactive dye (Realan Blue RC) was carried out at pH 5 using acetic acid. Dyeing with 1:2 metal complex (Isolan Dark Blue 2S-GL) was carried out at pH 6 using acetic acid. It can be seen from ***Figure 1*** that dyeing was carried out at three different temperatures (100, 90 and 80 °C), where the total dyeing time was constant (90 min.). Furthermore, at 100 °C the dyeing time was chosen as 15 and 45 min. After dyeing, the liquor was cooled down to 60 °C at a rate of 2 °C/min., and the fibres were taken out. Then the dyed samples were rinsed with cold (5 min.) – warm (at 50 °C 5 min.) – cold (5 min.) water, respectively, and dried at 80 ºC. All experiments were carried out in a Termal HT type dyeing machine.

Reflectance values and CIE L*a*b* colour space values of the dyed samples were measured with a Minolta 3600d reflectance spectrophotometer with illumination/observer conditions set at D65/10°. Then Color yield K/S values were calculated from the reflectance values using the Kubelka-Munk equation. Color yield (K/S) Light fastness tests according to the ISO 105 B02 standard and washing fastness tests according to the ISO 105 C06 standard were also carried out.

Alkali solubility

After demonstrating the low temperature dyeability of mohair fibre using benzyl alcohol, alkali solubility values of fibres treated with water at different temperatures and times according to the graph given in ***Figure 1*** were determined in order to demonstrate the fibre preserving effect of low temperature dyeing. The IWTO-4-60 standard test method was used. They were calculated as a percentage of the original mass, according to the equation given below:

$$\text{Alkali solubility (\%)} = [(M_1 - M_2)/M_1]100$$

where M_1 is the mass of the oven-dried sample before sodium hydroxide treatment, and M_2 is the mass of the oven-dried sample after sodium hydroxide treatment.

Kinetic study

In order to explain the mechanism of the effect of benzyl alcohol, a kinetic study was also carried out. The kinetic study was realised using 1:2 metal complex dye (Isolan Dark Blue 2SGL (C.I. Acid Blue 193). For this aim fibres were isotherm dyed in the presence (30 ml/l) and absence of benzyl alcohol at 80 °C and 100 °C for 90 min. at a liquor to goods ratio of 50:1. After dyeing every 15 minutes, a fibre sample was taken out. Then these fibres were dissolved in 1 N NaOH solution at 100 °C for 30 min. at a liquor ratio of 50:1, and the absorbance values of these solutions were measured with a UV-1201 Schimadzu UV-Spectrophotometer. In order to determine the dye concentration in the liquor via absorbance measurement, a calibration curve was needed. For this aim dye solutions in 1 N NaOH containing 20 g/l of an un-dyed fibre sample were prepared. For the absorbance measurement test, 2 grams of the dyed sample was taken and put in 100 ml of 1 N NaOH solution. To obtain a calibration curve, dye solutions were prepared in 1 N NaOH solution containing 20 g/l fibre. For this aim 40 g. of NaOH and 20 g. of un-dyed fibre were put in 100 ml of water and boiled. Then after adding different amounts of dyes in this solution, absorbance values were measured. As a result, a calibration curve was obtained. Normally when there is no dye, the absorbance value should be 0, but in our study the calibration curve was prepared using dye solutions in 1 N NaOH containing 20 g/l of un-dyed fibre. For this reason the absorbance value of the solution, which does not contain dye, is not 0. Because dissolved un-dyed fibre gives a yellowish colour, the absorbance value of the solution which does not contain dye is 0.97.

By using the calibration curve (see ***Figure 2***), dye amounts present on the fibre (mol/kg) were calculated. The equation below gives the correlation between absorbance and dye concentration:

$$\text{Absorbance} = 13 \times \text{Dye Concentration} + 0.97$$

In order to calculate the rate constant (k), a logarithm of the dye concentration in the dyebath was drawn versus the dyeing time measured in minutes. The gradient of this graph gives the rate constant of dyeing;

$$[DL] = [DL]_0 e^{-kt} \Rightarrow$$
$$(\log [DL]_0 - \log[DL])/t = k(\log e) \Rightarrow$$
$$k = -(\text{gradient})(2.303)$$

[DL]: Dye concentration in the dyebath
$[DL]_0$: Initial dye concentration in the dyebath

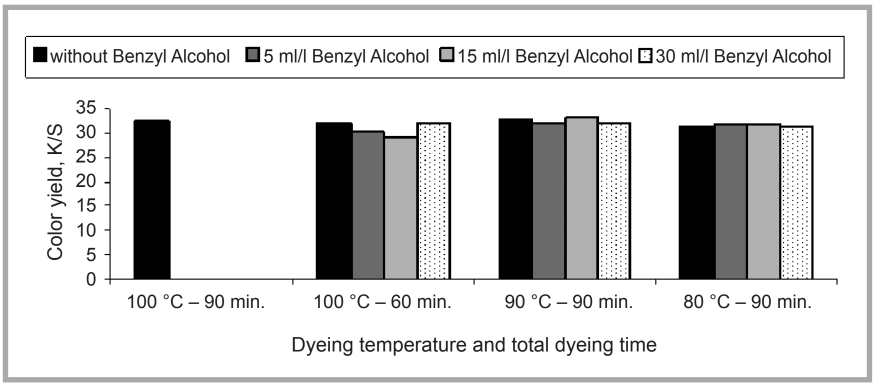

Figure 3. *Color yield results (K/S at 630 nm) of mohair dyed with 3% owf Telon Blue BRL Micro (acid leveling dye) at different temperatures.*

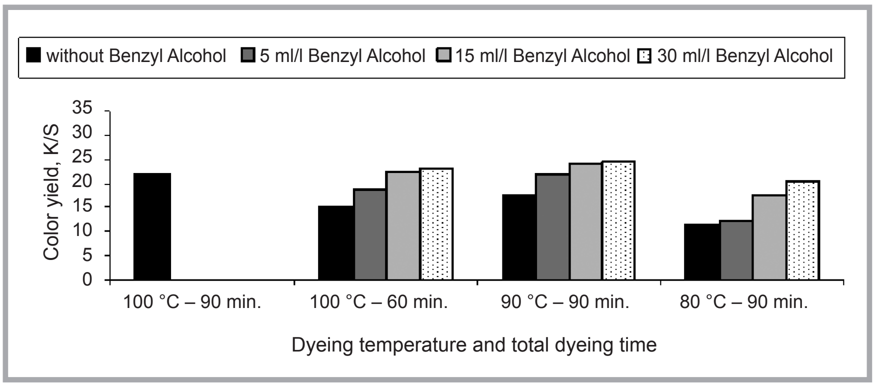

Figure 4. *Color yield results (K/S at 630 nm) of mohair dyed with 3% owf Telon Blue M-RLW (acid milling dye) at different temperatures.*

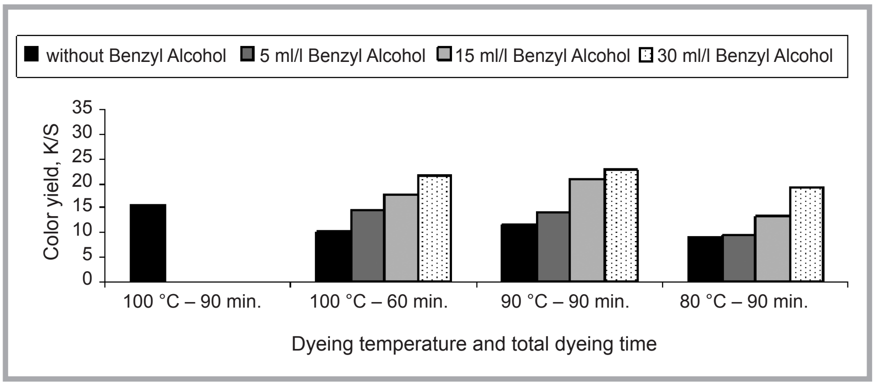

Figure 5. *Color yield results (K/S at 590 nm) of mohair dyed with 3% owf Isolan Dark Blue 2S-GL (metal complex dye) at different temperatures.*

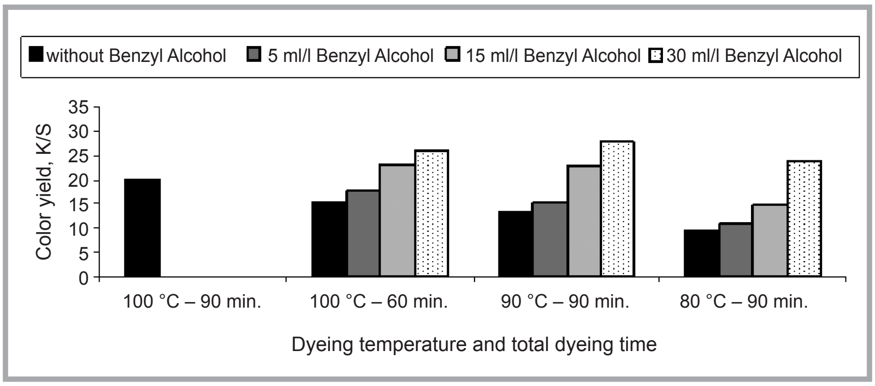

Figure 6. *Color yield results (K/S at 620 nm) of mohair dyed with 3% owf Realan Blue RC (reactive dye) at different temperatures.*

The standard affinity for the distribution of dye between the fibre and dyebath is proportional to the logarithm of the ratio of the absolute activities of the dye in the fibre and dyebath. Since the activity of the dye is assumed to be directly related to its concentration, one can write the equation as follows [12];

$$-\Delta\mu^{\circ} = RT[\ln([D]_F/[D]_L)]$$

$\Delta\mu^{\circ}$ - standart affinity in J mol^{-1}
R - gas constant in 8.317 J K^{-1} mol^{-1}
T - absolute temperature in K
$[D]_F$ - concentration of dye on the fibre at equilibrium in g/l
$[D]_L$ - concentration of dye remaining in the dyebath at equilibrium in g/l

By using the dye concentration on the fibre at the 90th min. ($[D]_F$), the dye concentration remaining in the dyebath ($[D]_L$) was calculated (as the initial concentration of dye in the dyebath was known). These values were put into the equation given above (T is the temperature of isotherm dyeing), and standard affinity values were calculated.

Results and discussion

Effect of benzyl alcohol usage in the dyeing of mohair with different dye classes

The colour yields of fibres dyed in the presence (5-15-30 ml/l) and absence of benzyl alcohol at various temperatures and for different periods of time are given in ***Figures 3 - 6***.

From these figures, it can be seen that the colour yield of the fibres dyed in the presence of benzyl alcohol was greater than that of fibres dyed in the absence of benzyl alcohol, except for leveling acid dye, where there is no difference. Leveling acid dyes are small molecular dyes which do not need high energy for diffusion, and for this reason they can be exhausted by fibre, also at lower temperatures than the boiling point. As a result, it can be suggested that acid dyes with a low molecular weight should be preferred for preventing colour yield losses at low temperature dyeing; however, it will not be a good solution when high wet fastness values are desired, as these dyes have fairly low wet fastness properties. However, if low temperature dyeing can be achieved with big molecular dyes without causing any decrease in the colour yield, it would result in improved dyeing characteristics, such as wet fastness, of the final product. As

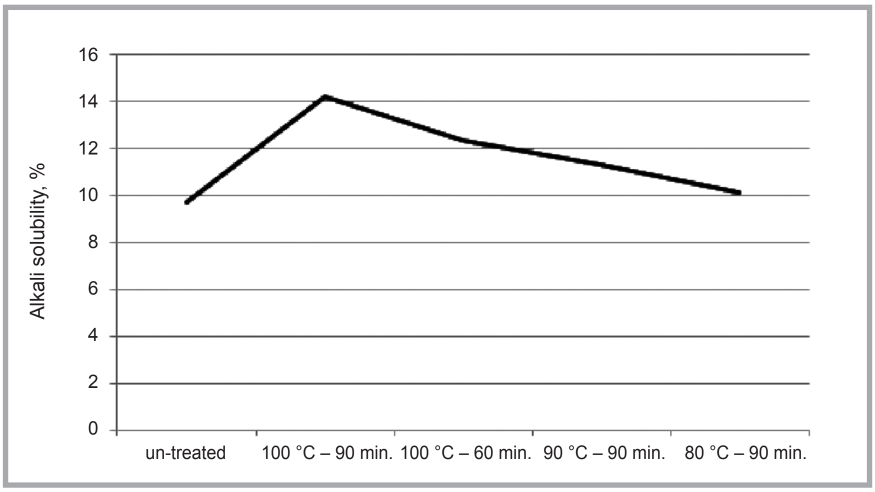

Figure 7. *Alkali solubility values of mohair fibre, untreated and treated at different conditions.*

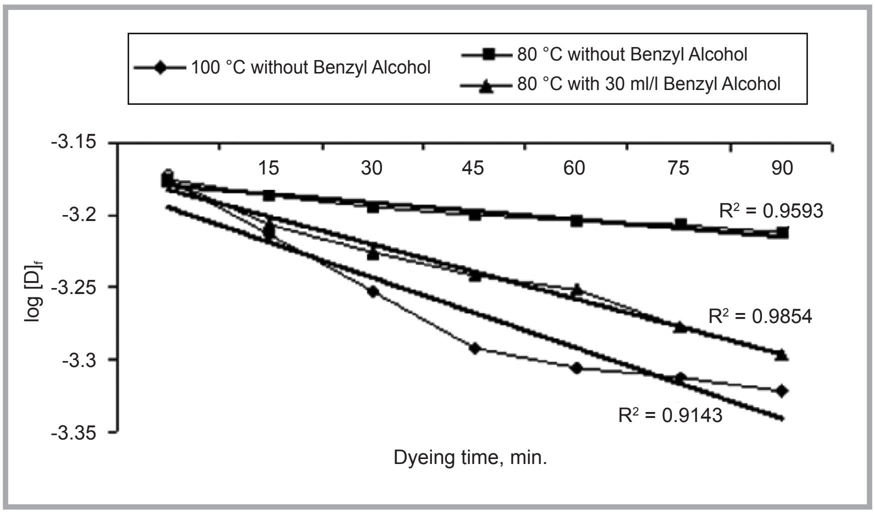

Figure 8. *Logarithm of dye concentration in the fibre versus the dyeing time of 3% owf Isolan Dark Blue 2S-GL on mohair*

Table 1. *CIEL*a*b* values of mohair dyed with Telon Blue BRL Micro.*

Dyeing Temp. – Time	Benzyl alcohol, ml/l	L*	a*	b*
100 ºC – 90 min.	–	17.80	4.63	-25.86
100 ºC – 60 min.	–	18.08	4.45	-26.23
	5	18.36	4.81	-25.56
	15	18.53	4.54	-25.38
	30	17.56	4.32	-24.79
90 ºC – 90 min.	–	18.56	2.43	-25.45
	5	18.34	2.55	-24.94
	15	18.49	2.17	-25.11
	30	18.87	2.67	-26.05
80 ºC – 90 min.	–	17.84	4.92	-25.50
	5	17.70	5.20	-25.85
	15	18.07	4.94	-26.32
	30	17.56	4.95	-25.02

can be seen from ***Figures 3 - 6***, at low temperatures or short dyeing cycles, the color yield increases with an increase in benzyl alcohol concentration in the liquor, and the colour yield of samples dyed in the presence of 30 ml/l benzyl alcohol at lower temperatures (90 °C or 80 °C) or for shorter times (100 °C 15 min.) is the same as that of samples dyed in the absence of benzyl alcohol at 100 °C. From these results, it can be generally concluded that mohair fibres can be dyed at lower temperatures (90 °C or 80 °C) or for shorter times (100 °C 15 min.) with all dye classes without causing any decrease in colour yield when benzyl alcohol is used as the auxiliary in dyeing.

There are two main reasons for the dye-uptake increase caused by benzyl alcohol. Benzyl alcohol, as an organic solvent, swells the fibre and loosens the fibre structure, which enhances dye diffusion into the fibre. Furthermore, in literature it was stated that benzyl alcohol decreases dye aggregation [13]. As is generally known, big aggregates have lower diffusion ability, and for this reason a decrease in dye aggregation will cause an increase in dye diffusion ability. Both these factors combine to make dye diffusion into fibre much easier [13]. Peters et al also stated that the solvent lowers the activation heat barrier and accelerates dyeing in some conditions [10].

One of the advantages of low temperature dyeing is the preservation of the fibre. From ***Figure 7*** it can be seen that when mohair fibre is treated at a boiling temperature (as in the dyeing process), its alkali solubility value increases, which means the fibre is damaged, but if fibre is treated at lower temperatures, such as 80 °C, fibre damage can be prevented.

From ***Table 1 - 4*** it can be seen that generally the differences in the a* and b* values of the colour obtained in the presence and absence of benzyl alcohol are smaller than those in the L* values. If the L* values are examined, it can be seen that the L* values of fibres dyed in the absence of benzyl alcohol are higher except for leveling acid dye (Telon Blue BRL Micro). L* is the value of lightness-darkness and an increase therein shows that the colour is getting lighter. Furthermore, the L* values of fibres dyed at 80 °C in the presence of 30 ml/l benzyl alcohol are similar to those of fibres dyed at 100 °C in the absence of benzyl alcohol, which means fibres can be dyed at 80 °C without any decrease in colour yield. From this point of view, the results obtained are parallel with K/S values.

As is generally known, wet fastness properties have great importance for dyers. For that reason, the washing and light fastness properties of fibres dyed at 100 ºC in the absence of benzyl alcohol and fibres dyed at lower temperatures (90 °C or 80 °C) or for shorter times (100 °C

*Table 2. CIEL*a*b* values of mohair dyed with Telon Blue M-RLW.*

Dyeing Temp. – Time	Benzyl alcohol, ml/l	L*	a*	b*
100 ºC – 90 min.	–	28.84	7.20	-40.06
100 ºC – 60 min.	–	34.09	4.16	-39.26
	5	32.41	5.59	-40.76
	15	30.62	7.34	-42.44
	30	29.78	8.05	-42.56
90 ºC – 90 min.	–	32.36	6.09	-40.98
	5	29.86	7.37	-41.43
	15	29.54	8.55	-43.14
	30	28.62	8.74	-42.41
80 ºC – 90 min.	–	36.49	2.50	-37.23
	5	36.49	2.54	-38.36
	15	32.64	5.32	-40.31
	30	30.96	6.70	-41.10

*Table 3. CIEL*a*b* values of mohair dyed with İsolan Dark Blue 2S-GL.*

Dyeing Temp. – Time	Benzyl alcohol, ml/l	L*	a*	b*
100 ºC – 90 min.	–	23.95	0.07	-10.60
100 ºC – 60 min.	–	28.90	-0.42	-11.87
	5	24.57	-0.06	-11.55
	15	21.88	0.35	-10.65
	30	18.98	0.63	-9.27
90 ºC – 90 min.	–	27.24	0.00	-11.77
	5	24.68	0.47	-11.76
	15	19.76	0.92	-10.49
	30	18.61	0.89	-9.89
80 ºC – 90 min.	–	30.47	0.08	-11.43
	5	30.05	0.01	-11.92
	15	25.27	0.50	-11.22
	30	20.85	0.69	-10.46

*Table 4. CIEL*a*b* values of mohair dyed with Realan Blue RC.*

Dyeing Temp. – Time	Benzyl alcohol, ml/l	L*	a*	b*
100 ºC – 90 min.	–	26.74	3.00	-33.08
100 ºC – 60 min.	–	29.78	2.12	-32.78
	5	28.82	2.57	-34.45
	15	24.84	4.70	-33.82
	30	23.40	6.90	-35.25
90 ºC – 90 min.	–	32.08	0.56	-33.20
	5	30.65	1.26	-33.88
	15	26.20	3.12	-34.62
	30	22.99	5.35	-34.13
80 ºC – 90 min.	–	36.78	-1.15	-32.49
	5	35.00	-0.67	-33.03
	15	31.94	0.88	-35.02
	30	25.98	4.17	-35.93

15 min.) in the presence of 30 ml/l benzyl alcohol, which have approximately the same colour yields, were tested.

When ***Table 5*** is examined, it can be seen that there are not any big differences in the washing and light fastness among the fibres dyed in the presence and absence of benzyl alcohol.

Effect of benzyl alcohol usage on dyeing kinetics

In order to explain why the dye-uptake increases in the presence of benzyl alcohol, a kinetic study was realised using 1:2 metal complex dye (Isolan Dark Blue 2S-GL). The dye-uptake amounts (mol dye/kg. fibre) versus the dyeing time are given in ***Figure 8***. By taking into consideration that the gradient of these curves gives the rate constant of dyeing, it can be said that the dyeing rate significantly diminishes when the temperature is decreased from 100 °C to 80 °C, and by adding benzyl alcohol into the dyeing liquor it rises again. These results could be understood better from the rate constant values (k) calculated from these experimental results (see ***Table 6***). Kinetic parameters are given in ***Table 6***, from which it can be seen that the rate constant and standard affinity diminishes significantly when the dyeing temperature is decreased, whereas the rate constant and standard affinity of samples dyed in the presence of 30 ml/l benzyl alcohol at 80 °C are very close to those of the sample dyed in the absence of benzyl alcohol at 100 °C. As is generally known, the rate constant is related to the kinetic energy, and when the temperature is decreased, it normally diminishes, but if benzyl alcohol is added into the dyeing liquor it enhances dye diffusion into the fibre (as explained before), and hence the dyeing rate constant increases. As a result, nearly the same dye-uptakes could be achieved at temperatures of 80 °C and 100 °C. These results explain why the dye-uptake of fibres increases in the presence of benzyl alcohol.

Conclusions

Attempts to dye wool at low temperature go back many years. Initially the prime motive was energy conservation, but more recently there has been an increasing awareness of the advantages to be gained from protecting the wool fibre by limiting the temperature, or the time at the top temperature, during dyeing [12]. Although low temperature dyeing is a very important concept for wool dyeing, it is gaining much more importance in the dyeing of more sensitive luxury fibres such as mohair.

According to the experimental results, it can be concluded that mohair fibres can be dyed at lower temperatures (90 °C or 80 °C) or for shorter times (100 ºC 15 min.) by adding adequate amount of benzyl alcohol into the liquor. Furthermore, benzyl alcohol usage in dyeing does not cause any negative effect on the washing and light fastness of dyed samples. According to the experimental results of alkali solubility tests, it can be

Table 5. Effect of benzyl alcohol usage on the washing and light fastness properties of dyed mohair samples (WO: Wool, PAC: Polyacrylonitrile, PES: Polyester, PA: Polyamide, CO: Cotton, CA: Cellulose Acetate).

	Dyeing Temp. – Time	Benzyl alcohol, ml/l	Washing fastness						Light fastness
			WO	PAC	PES	PA	CO	CA	
Telon Blue BRL Micro	100 ºC – 90 min.	–	3	4-5	4-5	2-3	2-3	4	7-8
	100 ºC – 60 min.	5	3	4-5	4-5	2-3	2-3	4	7-8
	90 ºC – 90 min.	15	3	4-5	4-5	2-3	2-3	4	7-8
	80 ºC-90 min.	30	3	4-5	4-5	2-3	2-3	4	7-8
Telon Blue M-RLW	100 ºC – 90 min.	–	5	5	5	4	4-5	5	7
	100 ºC – 60 min.	5	5	5	5	4	4-5	5	7
	90 ºC – 90 min.	15	5	5	5	4	4-5	5	7
	80 ºC – 90 min.	30	5	5	5	4	4-5	5	7
Isolan Dark Blue 2S-GL	100 ºC – 90 min.	–	5	5	5	4-5	4-5	5	7
	100 ºC – 60 min.	5	5	5	5	4-5	4-5	5	7
	90 ºC – 90 min.	15	5	5	5	4-5	4-5	5	7
	80 ºC – 90 min.	30	5	5	5	4-5	4-5	5	7
Realan Blue RC	100 ºC – 90 min.	–	5	5	5	5	4-5	5	6
	100 ºC – 60 min.	5	5	5	5	5	4-5	5	6
	90 ºC – 90 min.	15	5	5	5	5	4-5	5	6
	80 ºC – 90 min.	30	5	5	5	5	4-5	5	6

Table 6. Kinetic parameters of dyeing mohair with Isolan Dark Blue 2S-GL.

Conditions of dyeing process	k, min^{-1}	-Δμ, $KJ \cdot mol^{-1}$
100 °C – in the absence of benzyl alcohol	0,003685	-2,8636
80 °C – in the absence of benzyl alcohol	0,000871	-7,1724
80 °C – in the presence of 30 ml/l benzyl alcohol	0,002994	-3,3590

said that treating these fibres at lower temperatures will also decrease fibre damage. Furthermore it is thought that dyeing mohair fibres at lower temperatures/times could help to obtain bright colours (preserving fibre luster), which would also conserve energy.

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