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Abstract

In this study it was aimed to determine the effect of ultrasound usage on the colour yield in dyeing angora fibres, and it was found that dyeing in the presence of ultrasound energy increases the dye-uptake of angora fibres, hence higher colour yield values were obtained. According to experimental results, it can be said that at all points the colour yield values were higher in the presence of ultrasound than in its absence. The difference between them was greater for darker shades and for dyeing carried out in an acidic medium (pH 5), and also for moderate dyeing periods (90 min.). Furthermore there is no important difference between washing fastness, alkali solubility and strength values of fibres dyed in the presence and absence of ultrasound.

Key words: ultrasound, dyeing, colour, angora, fibre, fastness.

■ Introduction

Angora is a keratinous textile material produced by the long-haired Angora rabbit [1]. Angora rabbit hairs are derived from domesticated, highly bred animals, and cut or combed from the living species [2]. Angora fibre production is the third largest animal fibre industry in the world after wool and mohair [3]. Substantial quantities are also produced in France and smaller quantities in Eastern Europe and South America [4]. Because of its fineness, softness, lightness and good insulating properties, Angora rabbit hair is used in the production of high quality yarns for hosiery and knitting garments [5].

As these fibres are protein based, dyeing characteristics are similar to wool. In general, except the dyes used for special and aesthetic purposes, acid, 1:2 metal complex and partially reactive dyes are of great importance in angora dyeing [6]. The chemical structure of angora fibres has some similarities to the other animal fibres, but because of their more sensitive structure, during processes like washing, bleaching and dyeing, these fibres must be processed cautiously [7]. For this reason, it is very important to dye angora fibres at lower temperatures below boiling point and to limit the time of dyeing at high temperatures, so as to curtail any adverse effects on fibre properties; but in the case of low temperature dyeing, the dye-uptake and colour yield of samples will decrease and the effluent load will increase as a result of increasing the amount of dye remaining in the liquor. It is not a desirable situation in textile dyeing [6].

Leveling acid dyes are small molecular dyes that do not need high energy for dif-

fusion, 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 of low molecular weight should be preferred for preventing colour yield losses at low temperature dyeings, but it will not be a good solution when high wet fastness values are desired as these have fairly low wet fastness properties. However, if low temperature dyeing can be achieved with dyes of higher molecular weight without causing any decrease in colour yield, it would result in improved dyeing characteristics, such as the wet fastness of the final product [8].

In recent years, many attempts have been made to improve the various aspects of dyeing, and new technologies have been, and are being developed to reduce fibre damage, decrease energy consumption and increase productivity [9]. One of these new technologies that improve the dyeability of proteinous fibres is ultrasound.

Ultrasound is sound at a frequency that is above the threshold of human hearing [10]. The lowest audible frequency for humans is about 18Hz and the highest is normally around 18-20 kHz for adults, above which it becomes inaudible and is defined as ultrasound [11]. In recent decades the use of ultrasound technology has established an important place in different applications such as the medical field, and has started to revolutionize environmental protection. The idea of using ultrasound in textile wet processes is not a new one. On the contrary there are many reports from the 1950s and 1960s describing the beneficial effects of ultrasound in textile wet processes [12].

Ultrasound allows for process acceleration and the attainment of the same or better results than those offered by existing techniques under less extreme conditions, i.e., lower temperature and lower chemical concentrations. For this reason textile wet processes assisted by ultrasound are of great interest for the textile industry [12]. 3 main phenomena explaining the effect of ultrasound are as follows:

- **Dispersion:** breaking up of micelles and high molecular weight aggregates into uniform dispersions in a dye bath,
- **Degassing:** expulsion of dissolved or entrapped gas or air molecules from fibre into liquid and removal by cavitation, thus facilitating dye-fibre contact, and
- **Diffusion:** accelerating the rate of dye diffusion inside the fibre by piercing the insulating layer covering the fibre and accelerating the interaction or chemical reaction, if any, between dye and fibre [11].

Some of the benefits of the use of ultrasonics in dyeing can be listed as follows;

- energy savings by dyeing at lower temperatures and reduced processing times,
- environmental improvements by reduced consumption of auxiliary chemicals,
- lower overall processing costs, thereby increasing industry competitiveness [13]

Power ultrasound can enhance a wide variety of chemical and physical processes, mainly due to the phenomenon known as cavitation in a liquid medium, that is the growth and explosive collapse of microscopic bubbles. The sudden and explosive collapse of these bubbles can

generate “hot spots”, i.e., localised high temperature, high pressure, shock waves and severe shear force capable of breaking chemical bonds [14]. Improvements observed in ultrasound-assisted dyeing processes are generally attributed to the cavitation phenomena [12].

In literature there are several articles related to the usage of ultrasound in dyeing [10, 11, 13 - 18]. In our previous study we investigated the effect of ultrasound in the dyeing of mohair fibres [18]. Although there are many articles related to the usage of ultrasound in dyeing, there is only one study on angora, carried out by Perincek et al. [19]. They investigated the effects of novel treatment techniques (ozone and ultrasound) on the dyeability of Angora rabbit fibre. For this purpose, the effects of the ozonation time and fibre moisture during ozonation on the dyeing properties of Angora rabbit fibre were researched. Also dyeing was performed conventionally and with the use of ultrasound techniques after ozonation, which were then compared in terms of the colour yields. It was found that the ozonation process and dyeing with power ultrasound improved the dyeability of Angora rabbit fibre significantly. Nevertheless in this study it was aimed to determine the effect of ultrasound in dyeing of angora fibres with all dye classes that are used in angora dyeing such as acid, 1:2 metal complex and reactive. Apart from the dye class, the efficiency of ultrasound in angora dyeing was determined depending on the dyeing depth, dyeing pH, dyeing period and dyeing temperature. Moreover it was clarified if ultrasound assisted dyeing could be a good choice for low temperature dyeing of angora fibres or not.

Materials and method

In the experiments; angora fibres were used. Physical properties of the fibres are given in *Table 1*.

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. An ultrasound device of 36 kHz frequency produced by the AGS Company was used. Before dyeing, fibre samples were fully wetted. Both ultrasonic assisted and convention-

al dyeings were carried out in an ultrasonic bath. During conventional dyeings, the ultrasonic function of the device was turned off. A glass beaker with the fibre and liquor was placed in an ultrasonic bath filled with water at the temperature desired. The liquor to good ratio was 30:1 in all experiments.

To determine the effect of ultrasound on the dye exhaustion of angora fibre, fibres were isothermally dyed with milling acid dye in 2% owf at 70 °C for 1 h in the presence and absence of ultrasound. The angora fibre was dyed with acid levelling dye (Telon Blue BRL Micro) at pH 3 using formic acid. Dyeing with the acid milling (Telon Blue M-RLW) and reactive (Realan Blue RC) dyes was carried out at pH 5 using acetic acid. Dyeing with the 1:2 metal complex dye (Isolan Dark Blue 2S-GL) was carried out at pH 6 using acetic acid. In order to avoid affecting the dyeing properties of fibres, the usage of auxiliaries (levelling agent etc.) or salts was avoided. After dyeing, the dyed samples were rinsed with cold (5 min.) - warm (at 50 °C 5 min.) - cold (5 min.) water, respectively, and dried. Then reflectance (R%) and CIE L*a*b* values of the dyed samples were measured with a Minolta 3600d reflectance spectrophotometer with illumination/observer conditions set at D65/10°. Colour yield (K/S) values were calculated from reflectance values using the Kubelka-Munk equation.

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

R - Reflectance value at maximum absorption wave length in nm,
K - Absorption coefficient,
S - Scattering coefficient.

By taking the K/S value of material which was dyed in the absence of ultrasound as 100, the Relative in % Colour Yield values of samples dyed in the presence of ultrasound were calculated as follows;

$$\text{Relative Color Yield in \%} = \frac{[(K/S)_u \times 100]}{(K/S)} \quad (2)$$

(K/S)_u:- K/S value of material dyed in the presence of ultrasound,

(K/S):- K/S value of sample dyed in the absence of ultrasound.

Washing fastness tests according to the ISO 105 C06 standard were also carried out. Furthermore the alkali solubility test was applied to fibres dyed with milling acid dye in 2% owf at pH 5 and 70 °C

for 1 h in the presence and absence of ultrasound.

Test method IWTO-4-60 was followed for the alkali solubility test (0.1 N NaOH, 65 °C, 1 h), calculated as a percentage of the original mass, according to the equation given below:

$$\text{Alkali solubility in \%} = \frac{(M_1 - M_2)}{M_1} \times 100 \quad (3)$$

where, M₁ is the mass of the oven-dried sample before sodium hydroxide treatment and M₂ is that of the oven-dried sample after sodium hydroxide treatment.

In order to determine whether ultrasound treatment has an effect on fibre damage, fibre strength and scanning electron microscope analysis (SEM) were also conducted. Quanta FEG A 250 scanning electron microscope (FEI, the Netherlands) was employed for imaging of fibre samples at 4000× magnification.

In order to observe how the increase in colour efficiency provided by ultrasonic dyeing depends on the dyeing depth, dyeing pH, dyeing period, dyeing temperature and liquor ratio, dyeing treatments in four different concentrations (0.5% - 1.5% - 2.5% - 3.5%), four different pH values (3 - 5 - 7 - 9), four different periods (30 - 60 - 90 - 120 min.), three different temperatures (50 - 60 - 70 °C) and three different liquor ratios (30:1 - 60:1 - 90:1) in the presence and absence of ultrasound were carried out. These experiments were carried out using acid milling (Telon Blue M-RLW) dye. Colour yield values of dyed samples were measured and compared with each other.

Results and discussion

The relative in % colour yield increase values of angora fibres dyed with various dye classes in the presence of ultrasound are given in *Figure 1*.

When *Figure 1* is investigated, it is understood that in the presence of ultrasound, while the dye-uptake increases for milling acid and reactive dyes, there is no significant change in the dye-uptake of levelling acid dye and 1:2 metal complex dye. The increase in colour yields obtained in dyeing in the presence of ultrasonic waves can be attributed to the 3 main phenomena previously explained: dispersion, degassing and diffusion effects of ultrasound [11].

When all these factors are taken into consideration, the reason for the dyeings carried out in the presence of ultrasound being darker could be understood. However, ultrasound did not improve the dye-uptake for levelling acid and 1:2 metal complex dyes. As leveling acid dyes have small molecules, during dyeing with these at both low and high temperatures (when dyeing period is constant) approximately all dye molecules are taken up by fibres. So that all dye molecules are exhausted in the absence of ultrasound, in the presence of ultrasound no more dye could be exhausted, and there would be no difference in dye-uptake. For 1:2 metal complex dyes, the situation is opposite. These dyes have molecules even bigger than those of milling acid dyes, and for these dyes to diffuse into the fibre, high energy is needed. In experiments, dyeings were carried out at 70 °C, where dye diffusion is quite difficult, and hence even in the presence of ultrasonic energy sufficient dye-uptake could not be obtained.

CIEL*a*b* values of angora fibres dyed with various dye classes in the presence and absence of ultrasound are given in **Table 1**.

If L* values are examined, it can be observed that L* values of samples dyed in the presence of ultrasound are lower than for the fibre dyed in the absence of ultrasound for milling acid. The reactive dyes. L* value is that of lightness-darkness and a decrease in the L* value shows that the colour gets darker. On the other hand, there is no significant change in the L* values of samples dyed with leveling acid and 1:2 metal complex dye. These results are in parallel with K/S values.

When **Table 1** is examined, it is seen that for all dye classes, generally the differences in a* and b* values of the colour are smaller compared to that in L* values. From **Table 1**, it can be seen that for the sample dyed in the presence of ultrasound, the a* value is generally bigger and the b* value generally smaller. From these results it can be understood that the nuance of colour obtained in the presence of ultrasound will be less greenish and less yellowish.

Washing fastness values of angora fibres dyed with various dye classes in the presence and absence of ultrasound are given in **Table 2**, from which it can be seen that there is no negative effect of ul-

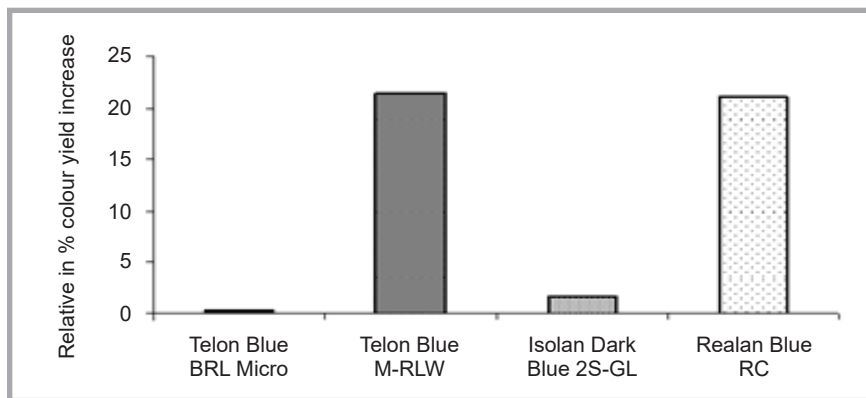


Figure 1. Relative (%) colour yield increase values of angora fibres dyed with various dye classes in the presence of ultrasound (at 70°C for 1 hour at 2% depth)

Table 1. Physical properties of fibres used in the experiments.

Diameter, micron	dtex	Average length, cm	Strength, cN	Relative strength, cN/tex	Elongation at break, %
16.82	2.58	6.47	3.55	13.76	27.55

Table 2. CIE L*a*b* values of angora fibres dyed with various dye classes (at 70 °C for 1 hour at 2% depth) in the presence and absence of ultrasound.

Dye	Ultrasound	L*	a*	b*
Telon Blue BRL Micro	+	29.55	-0.88	-37.51
	-	29.71	-1.58	-35.61
Telon Blue M-RLW	+	61.49	-4.18	-30.50
	-	64.38	-3.98	-28.56
Isolan Dark Blue 2S-GL	+	64.76	-1.80	-8.76
	-	65.18	-1.86	-9.65
Realan Blue RC	+	59.91	-4.80	-27.62
	-	62.26	-5.31	-25.60

Table 3. Washing fastness values of angora fibres dyed with various dye classes (at 70°C for 1 hour at 2% depth) in the presence and absence of ultrasound (WO: Wool, PAC: Polyacrylonitrile, PES: Polyester, PA: Polyamide, CO: Cotton, CA: Cellulose acetate).

Dye	Ultrasound	Washing fastness					
		WO	PAC	PES	PA	CO	CA
Telon Blue BRL Micro	+	4 - 5	5	4 - 5	3	4	4 - 5
	-	4 - 5	5	4 - 5	3	4	4 - 5
Telon Blue M-RLW	+	4 - 5	5	4 - 5	4 - 5	4 - 5	5
	-	4 - 5	5	4 - 5	4 - 5	4 - 5	5
Isolan Dark Blue 2S-GL	+	5	5	4 - 5	4	4 - 5	5
	-	5	5	4 - 5	4	4 - 5	5
Realan Blue RC	+	5	5	5	4 - 5	4 - 5	5
	-	5	5	5	4 - 5	4 - 5	5

trasound on the washing fastness values of the dyed samples. In order to observe whether ultrasound causes damage to fibre or not, alkali solubility tests of samples dyed with 2% owf milling acid dye (at pH 5 and 70 °C for 1 h) in the presence and absence of ultrasound were realised. Alkali solubility values of fibres dyed in the presence and absence of ultrasound were found to be 5.88% and 5.67% respectively. These results indicate that ultrasound does not have a negative effect on fibre.

In order to determine whether ultrasound treatment has an effect on fibre damage, fibre strength and scanning electron microscope analysis (SEM) were also conducted. Strength values of undyed angora fibres treated at 70 °C for 60 min. at pH 5 in the presence and absence of ultrasound are given in **Table 4** (see page 140).

As can be seen from **Table 4**, there is no significant difference between the strength values of angora fibres treated in the presence and absence of ultrasound.

Table 4. Strength values of undyed angora fibres after 60 min treatment at 70 °C in the presence of ultrasound and after 60 min treatment at 70 °C in the absence of ultrasound

Sample	Strength, cN
Treated in the presence of ultrasound	2.71
Treated in the absence of ultrasound	2.87

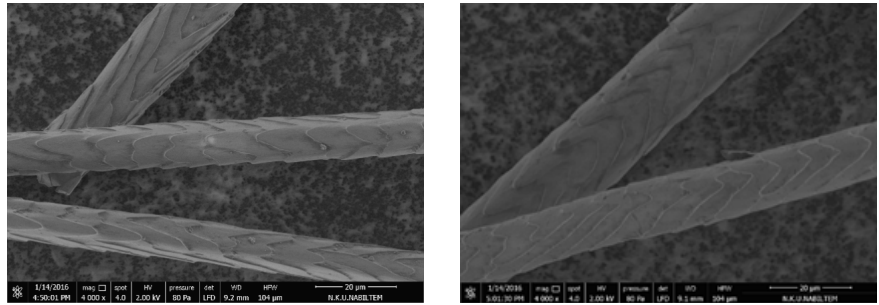


Figure 2. SEM images of undyed angora fibres at magnification of 4000× after 60 min treatment at 70 °C in the presence of ultrasound (on the left) and after 60 min treatment at 70 °C in the absence of ultrasound (on the right).

Images of SEM analysis performed on undyed angora fibres treated at 70 °C for 60 min. at pH 5 in the presence and absence of ultrasound are given in **Figure 2**.

When **Figure 2** is examined, it can be seen that the treatment of angora fibres at 70 °C both in the presence and absence of ultrasound does not give any damage. These results are consistent with earlier reports that were carried out on wool fibres [20]. However, it is important to note that at higher temperatures or prolonged treatment times some detachment

of scales from the fibres could happen, as observed in previous studies [21].

The effect of ultrasound usage on colour efficiency in dependence on the dyeing depth, dyeing pH, dyeing period, dyeing temperature and liquor ratio is given in **Figures 3 - 7**.

As shown in **Figure 3**, it is clear that the colour yield values are higher in the presence of ultrasound, especially for

darker shades. This situation can be explained by the ultrasound's effect of lowering the association degree, as previously mentioned. The aggregation increases with increasing dyeing depth, because the dye concentration per unit volume increases. For this reason the effect of ultrasound could be observed more clearly in darker shades.

When **Figure 4** is examined, it can be understood that the color yield value in-

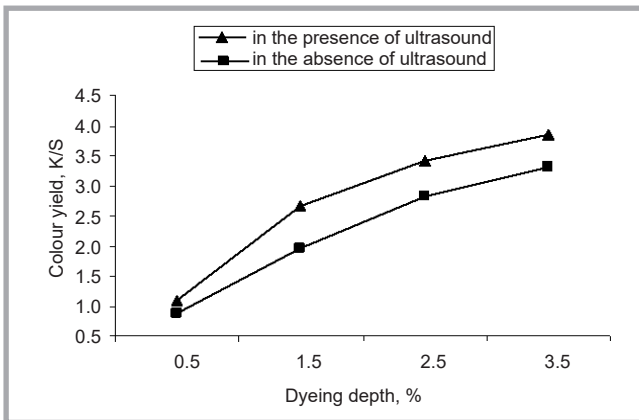


Figure 3. Effect of ultrasound on colour efficiency depending on dyeing depth during dyeing with Telon Blue M-RLW (dyeing conditions: 70 °C - 60 min. - pH 5 - 30:1).

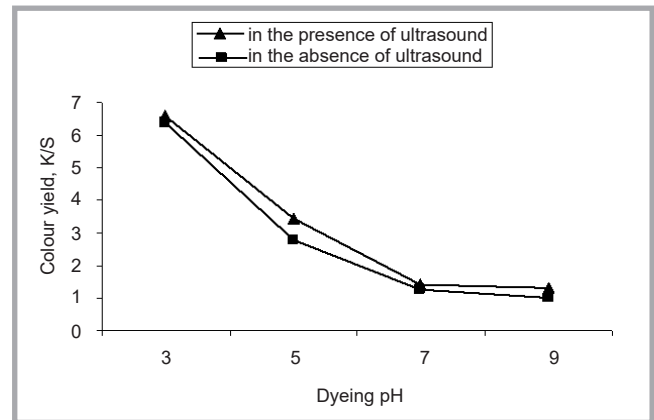


Figure 4. Effect of ultrasound on colour efficiency depending on dyeing pH during dyeing with Telon Blue M-RLW (dyeing conditions: 70 °C - 60 min. - 2.5% - 30:1).

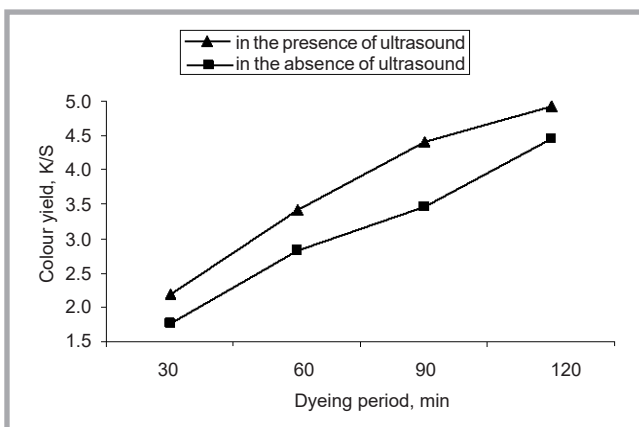


Figure 5. Effect of ultrasound on colour efficiency depending on dyeing period during dyeing with Telon Blue M-RLW (dyeing conditions: 70 °C - 2.5% - pH 5 - 30:1).

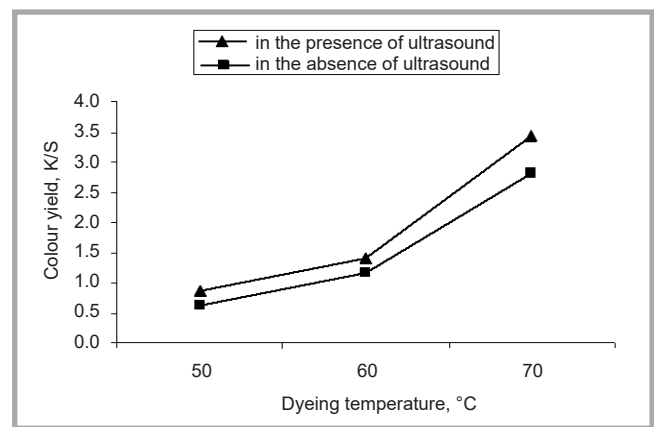


Figure 6. Effect of ultrasound on colour efficiency depending on dyeing temperature during dyeing with Telon Blue M-RLW (dyeing conditions: 60 min. - 2.5% - pH 5 - 30:1).

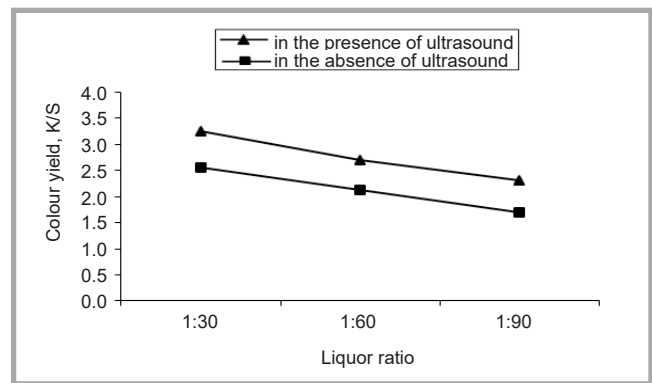
creases with an decrease in the dyeing pH (with the increase in acidity), with both dyeings carried out in the presence and absence of ultrasound and colour yields of samples dyed in the presence of ultrasound always being higher. This difference is more obvious in the mild acidic medium, while it is less obvious in the strong acidic and basic medium, due to the fact that as acid is added to the system, hydrogen ions from the acid react with the carboxylate anions to form carboxylic acid groups, leaving the positively charged ammonium groups available to act as "dye sites" for acid dyes [22]. For this reason, the benefit in dye-uptake caused by ultrasound is not so evident.

Under alkaline conditions, on the other hand, hydrogen ions are abstracted from the positively charged amino groups. The carboxyl anions then confer a negative charge on the substrate [22]. In this case, the (-) charged dye is repulsed by fibre and the dye-uptake decreases dramatically. For this reason the ultrasound energy also cannot be enough to increase the dye-uptake.

From these results, it can be stated that ultrasound technology will be more useful for dyes that require mild acidic conditions (such as pH 5) for application. Otherwise it cannot be suggested to carry out dyeing treatments at mild acidic conditions for all dye classes in order to utilise the positive effect of ultrasound, because for each dye class the pH values recommended by the manufacturers are the optimum pH values for dyeing fibres with the respective dye. Using liquors with a higher or lower pH value would lead to a lower dye uptake or the unlevelness problem (due to fast dye absorption by the fibre), respectively.

Figure 5 shows the effect of the duration of dyeing. Colour yields obtained in dyeings carried out both in the presence and absence of ultrasound increase with a prolonged dyeing time. Furthermore it can be seen that the difference between dyeings in the presence and absence of ultrasound decreases with a rise in the dyeing time (for 120 min.), because with the prolongation of the dyeing time, fibres have enough time to take up dye molecules, and for this reason even if the dye-uptake is slower when ultrasound energy does not exist, differences in the dye-uptake amount between dyeings carried out in the presence and absence

Figure 7. Effect of ultrasound on colour efficiency depending on liquor ratio during dyeing with Telon Blue M-RLW (dyeing conditions: 70 °C - 60 min. - 2.5% - pH 5).



of ultrasound decrease. On the other hand, when the dyeing period is too short (30 min.), fibres do not have enough time to take up dye molecules even in the presence of ultrasonic energy, and hence it does not affect the dyeing result whether ultrasound exists or not.

As shown in **Figure 6**, it is clear that the colour yield increases as the dyeing temperature increased in dyeings carried out both in the presence and absence of ultrasound, and at all points the colour yield values were higher in the presence of ultrasound. This increase in dye uptake can be explained by fibre swelling and, hence, enhanced dye diffusion. Also the ultrasonic power provides an additional factor of de-aggregation of dye molecules, thus leading to further enhancement of dye diffusion and better dyeability than dyeing carried out in the absence of ultrasound [11].

From **Figure 7**, it can be understood that colour yields obtained in dyeings carried out both in the presence and absence of ultrasound decrease with an increase in the liquor ratio. On the other hand, the colour yields of samples dyed in the presence of ultrasound are always higher. This result again shows that ultrasonic power improves dye exhaustion.

Conclusions

In this study it was determined that dyeing in the presence of ultrasound energy increases the dye-uptake of angora fibres during dyeing with milling acid and reactive dyes, and hence higher colour yield values are obtained. According to experimental results, it can be said that at all points the colour yield values were higher in the presence than in the absence of ultrasound. The difference between them was greater for darker shades and for dyeing carried out in acidic medium (pH 5), and also for moderate dyeing pe-

riods (90 min.). Furthermore there is no important difference between washing fastness, alkali solubility and strength values of fibres dyed in the presence and absence of ultrasound. In the light of these experimental results, it can be said that ultrasound energy could be used in the dyeing of angora fibres in order to improve their dyeability. On the other hand, it could be an alternative for decreasing dyeing temperature during dyeing with milling acid and reactive dyes, because the potential dye-uptake decrease which will be caused by lowering the dyeing temperature would be compensated by the dye-uptake increase supplied by ultrasound energy.

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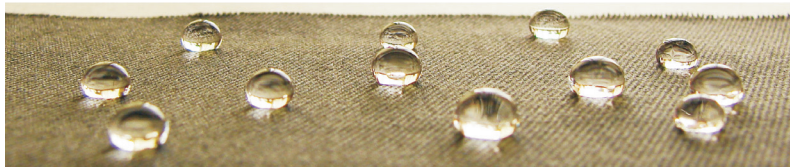
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The Scientific Department of Unconventional Technologies and Textiles specialises in interdisciplinary research on innovative techniques, functional textiles and textile composites including nanotechnologies and surface modification.

Research are performed on modern apparatus, *inter alia*:

- Scanning electron microscope VEGA 3 LMU, Tescan with EDS INCA X-ray microanalyser, Oxford
- Raman InVia Reflex spectrometer, Renishaw
- Vertex 70 FTIR spectrometer with Hyperion 2000 microscope, Brüker
- Differential scanning calorimeter DSC 204 F1 Phenix, Netzsch
- Thermogravimetric analyser TG 209 F1 Libra, Netzsch with FT-IR gas cuvette
- Sigma 701 tensiometer, KSV
- Automatic drop shape analyser DSA 100, Krüss
- PGX goniometer, Fibro Systems
- Particle size analyser Zetasizer Nano ZS, Malvern
- Labcoater LTE-S, Werner Mathis
- Corona discharge activator, Metalchem
- Ultrasonic homogenizer UP 200 st, Hielscher

The equipment was purchased under key project - POIG.01.03.01-00-004/08 Functional nano- and micro textile materials - NANOMITEX, co-financed by the European Union under the European Regional Development Fund and the National Centre for Research and Development, and Project WND-RPLD 03.01.00-001/09 co-financed by the European Union under the European Regional Development Fund and the Ministry of Culture and National Heritage.



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