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Ultrasonic assisted dyeing
III. Dyeing of wool with lac as a natural dye

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Abstract

The dyeing of wool fabrics using lac as a natural dye has been studied in both conventional and ultrasonic techniques. The extractability of lac dye from natural origin using power ultrasonic was also evaluated in comparison with conventional heating. The results of dye extraction indicate that power ultrasonic is rather effective than conventional heating at low temperature and short time. The effects of dye bath pH, salt concentration, ultrasonic power, dyeing time and temperature were studied and the resulting shades obtained by dyeing with ultrasonic and conventional techniques were compared. Colour strength values obtained were found to be higher with ultrasonic than with conventional heating. The results of fastness properties of the dyed fabrics were fair to good. Dyeing kinetics of wool fibre with lac dye using conventional and ultrasonic conditions was compared. The time/dye-uptake isotherms are revealing the enhanced dye-uptake in the second phase of dyeing (diffusion phase). The values of dyeing rate constant, half-time of dyeing and standard affinity and ultrasonic efficiency have been calculated and discussed.

Keywords: Ultrasound; Sonochemistry; Dyeing; Wool; Natural dye; Kinetic; Lac

1. Introduction

Recently, a revival interest in the use of natural dyes in textile coloration has been growing. This is a result of the stringent environmental standards imposed by many countries in a response to the toxic and allergic reactions associated with synthetic dyes. Conventional wisdom leads to the belief that natural dyes are friendlier to the environment than their synthetic counterparts. Natural dyes can exhibit better biodegradability and generally have a higher compatibility with the environment. Considerable research work is being undertaken around the world on the application of natural dyes [1–9]. A review on the use of natural dyes in textile coloration has been published [10].

Lac is a unique dye material of animal origin being the secretion of a tiny insect *Kema lacca* (Kerr) thriving on certain host plants such as, Palas (*Butea monosperma*), Kusum (*Scheichera oleosa*) and Ber (*Zizyphus mauritania*). The use of lac dye (CI Natural Red 25) in the dyeing of silk and leather seems to have been known to the Chinese some 4000 years ago. Lac is a mixture of at least five closely related laccaic acids (Fig. 1), which are water soluble red dyes of anthraquinoid type structure [11,12].

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. Sudden and explosive collapse of these bubbles can generate "hot spots" [13,14], i.e., localised high

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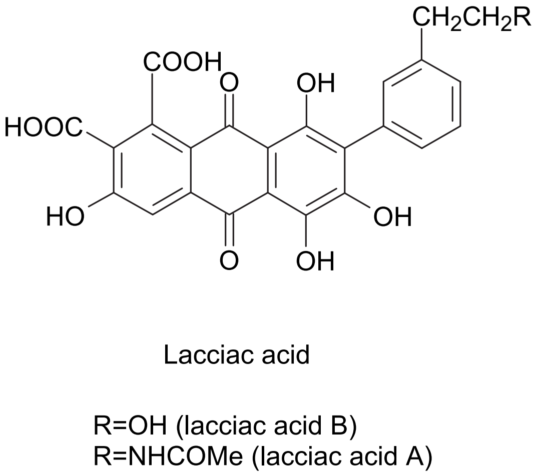

Fig. 1. Main colorant species present in aqueous extract of lac.

temperature, high pressure, shock waves and severe shear force capable of breaking chemical bonds.

In continuation of our interest in using power ultrasonic in dyeing [15,16], we conducted the present study to investigate the dyeing and fastness properties of wool fibres using lac as a natural dye. Different factors affecting dyeability and fastness properties were thoroughly investigated. Comparative results of the dyeing kinetics of wool fibres using both ultrasonic and conventional dyeing methods are also presented.

2. Experimental

2.1. Materials

A commercial material of lac dye was used and its properties are listed in Table 1. Scoured and bleached wool twill fabric with the following characteristics was purchased from Misr for Spinning and Weaving Company, Mahalla El-Kobra, Egypt: weight 205 gm^{-2}, 72 ends per inch, 64 picks per inch. Before using, the fabric was treated with a solution containing 5 $g\,L^{-1}$ non-ionic detergent (Hostapal CV, Clariant), at 50 °C for 30 min. Then, the fabric was thoroughly washed with water and air dried at room temperature.

Ultrasonic extraction and dyeing were conducted in a thermostated CREST benchtop 575 HT ultrasonic cleaner of capacity 5.75 L, frequency 38.5 kHz and with a maximum 500 W output. The output power levels are from 100 to 500 W.

2.2. Methods

2.2.1. Conventional extraction

Conventional extraction was carried out in 100 ml boiled distilled water using varying amounts of the dye materials (2–12%) for different time intervals (20–120 min). After filtration and certain dilution, the optical density of the dye liquor at 535 nm was measured [17,18].

2.2.2. Ultrasonic extraction

Ultrasonic extraction was carried out as described above in 100 ml distilled water using varying amounts of the dye materials (2–12%) at different temperatures (50–80 °C) using different sonic powers (100–500 W) and for different time intervals (20–120 min). After filtration and certain dilution, the optical density of the dye liquor at 535 nm was measured.

2.2.3. Dyeing procedure

In a dye bath containing different amounts of sodium chloride (0–20 $g\,L^{-1}$) and 8% lac dye with liquor ratio 50:1, wool fabric was dyed using conventional heating (CH) at different pH values (1.5–9) for different durations (12–120 min) and at different temperatures (30–80 °C). For comparison, the same condition of dyeing was made using ultrasonic dyeing (US) with sonic power of 500 W. The optimum value of sonic power was obtained after studying the effect of sonic power in the dyeability of wool. Thus, in a dye bath containing 8% lac dye with liquor ratio 50:1 and pH 2.5, wool fabric was dyed at 80 °C for 60 min using different sonic power levels (100–500 W). The effect of the number (1–5) of dye bath reuse was also conducted in a comparative manner for both US and CH methods.

The dyed samples were rinsed with cold water, washed in a bath of liquor ratio 60:1 using 3 $g\,L^{-1}$ non-ionic detergent (Hostapal CV, Clariant) at 50 °C for 30 min, then rinsed and finally dried at ambient temperature.

The pH values were recorded with Hanna pH meter and adjusted with dilute solutions of sodium carbonate, as the dye extract was acidic (pH 1).

2.2.4. Dyeing rate

Wool fabric samples were cut into pieces approximately 1 cm^2 and dyed at pH 2.5 in a beaker with 200 ml aqueous solution composed of 1 $g\,L^{-1}$ non-ionic wetting agent (Hostapal CV, Clariant) and 8 $g\,L^{-1}$ lac dye with

Table 1
Characteristics of lac dye used

Botanical name	CI name	Class	Part used	λ_{max} (nm) in water	Molar extinction coefficient ($mg^{-1}\,L\,cm^{-1}$)
Kerria lacca	Natural Red 25	Anthraquinone	Whole insect	535	0.0009

liquor ratio 50:1 at 80 °C with frequent shaking. Dyeing was conducted using both ultrasonic power at 500 W and conventional heating conditions. After selected time intervals, 0.5 ml of the dye bath was pipetted into test tubes and diluted with water to 5 ml to measure its absorbance at 535 nm.

2.3. Colour strength

The reflectance of the soaped samples was measured on a Perkin–Elmer Lambda 3B UV/Vis spectrophotometer. Relative colour strengths (K/S values) were determined using the Kubelka–Munk equation [19].

2.4. UV/Vis absorption spectra

The UV/Vis absorption spectra in water were recorded using a Shimadzu UV/Vis spectrophotometer. The quantity of dye-uptake was estimated using the following equation:

$$Q = (C_0 - C_f)V/W \tag{1}$$

where Q is the quantity of dye-uptake (mg g^{-1}), C_0 and C_f are the initial and the final concentrations of dye in solution (mg L^{-1}), respectively, V is the volume of dye bath (L) and W is the weight of fibre (g). The concentrations of dye solutions were determined after reference to the respective calibration curve of the lac dye using Lambert–Beer law.

2.5. Fastness testing

The dyed samples were tested according to ISO standard methods. The specific tests were: ISO 105-X12 (1987), colour fastness to rubbing; ISO 105-C02 (1989), colour fastness to washing; ISO 105-E04 (1989), colour fastness to perspiration; and ISO 105-B02 (1988), colour fastness to light (carbon arc).

3. Results and discussion

It has been recognised for many years that power ultrasound has great potential for application in a wide variety of industrial processes as it offers potential cost savings in time, chemicals, energy and reduced effluent [20–22]. In this context, applying power ultrasound in textile coloration was of interest. Thus, exploiting power ultrasonic in the dyeing of wool fabrics using lac as a natural dye was made in two steps, i.e. ultrasonic extraction of the dye followed by ultrasonic dyeing. Therefore, comparative study between CH and US as well as the different factors that may affect these processes are investigated.

3.1. Dye extraction

3.1.1. Effect of dye amount

Comparative extraction of lac dye using US (at 80 °C) and CH (at 100 °C) methods and the extent of extractability were monitored spectrophotometrically at λ_{max} 535 nm. Fig. 2 shows that as the dye amount % (amount of dye used per 100 ml distilled water) increases, the absorption of the dye extract also increases when using either US or CH with much higher values at all points in US case as expected. The maximum extractability was attained with 8 g/100 ml water.

3.1.2. Effect of extraction time

US and CH extractions of lac (8 g/100 ml water) were carried out for different times. Fig. 3 shows that the rate of dye extraction by US is higher than that of CH to reveal maximum dye extraction after 40 min for the US. It is worth noting that the absorption of the extract using US was 1.59 after 40 min whereas on using CH it was only 1.33 after 80 min. Prolonged extraction time using US, however, may lead to dye degradation as revealed by decreasing the absorbance of the extract.

3.1.3. Effect of ultrasonic power

The effect of ultrasonic power on the extractability of lac was conducted at different power levels (100–500). As shown in Fig. 4, the absorption of the extract increases with increasing power level up to 500 W at which the maximum extraction was attained. The results of dye extraction emphasize the enhancement effect of power ultrasonic, which helps much dye extraction from natural source by virtue of its dispersion, degassing and diffusion (accelerating the rate of dye desorption from the bark) capability.

3.1.4. Effect of extraction temperature

Preliminary studies have shown that conventional extraction of lac dye from the bark into water is temperature dependant and directly proportional with

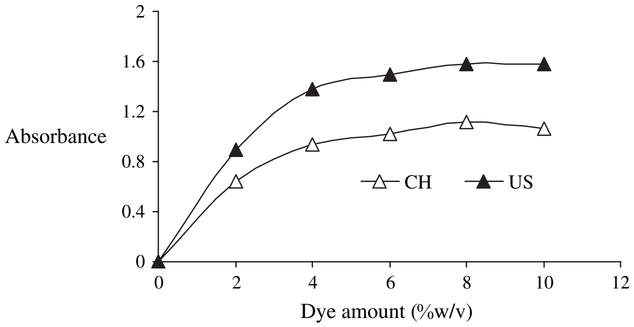

Fig. 2. Plots of dye extraction versus dye amount using conventional and ultrasonic heating conditions. Extraction conditions: for US, 500 W, 1 h, at 80 °C; for CH, 1 h, at 100 °C.

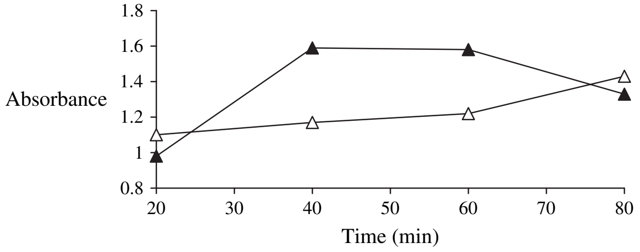

Fig. 3. Effect of time on the extent of dye extraction using conventional and ultrasonic conditions. Extraction conditions: for US, 500 W, at 80 °C, using 8% w/v dye amount; for CH, at 100 °C, using 8% w/v dye amount. For legend, see Fig. 2.

temperature to conclude effective extraction at the boil. Therefore, it was desirable to find out whether applying power ultrasonic will provide additional energy and thus lower the temperature needed for dye extraction. Expectedly, the extractability was found to increase as the temperature increases up to 70 °C, thereafter a plateau started to appear up to 80 °C as shown in Fig. 5. This behavior reflects the additional enhancing effect due to power ultrasonic that provides easy, efficient route for dye extraction without the need to boil the water as heating may lower the brightness of the extracted dye. Similar results of ultrasonic dye extraction using other natural dyes have been reported [23].

3.2. Dyeing

3.2.1. Effect of dye bath pH

Fig. 6 shows that the pH values of the dye bath have considerable effect on the dyeability of wool fabrics with lac dye under both US and CH conditions. It is clear that US has improved the dyeability of the fabrics at pH 1.5 and 2.5 values but started to decline sharply as the pH increases with rather lower dyeability under US condition. The effect of dye bath pH can be attributed to the correlation between dye structure and wool fibres. Since the lac dye used is a water soluble dye containing carboxyl groups, it would interact ionically with the protonated terminal amino groups of wool fibres at acidic pH via ion exchange reaction. The weak carboxylate anion of the dye that may be present at low pH (1.5–2.5 value) replaces that of acid due to its higher affinity. The anion of the dye has a complex character, and when it is bound on fibre, further kinds of interactions take place together with ionic forces. This ionic attraction would increase the dyeability of the fibre as clearly observed in Fig. 6. At pH > 2.5, however, the ionic interaction between the carboxylate anion of the dye and wool fibres decreases due to decreasing number of protonated terminal amino groups of wool fibres and thus lowering its dyeability. It is to be mentioned that the lower dyeability in the case of US than in the case of CH at pH > 2.5 may be attributed to the enhanced desorption of the dye as its ionic bond is getting decreased.

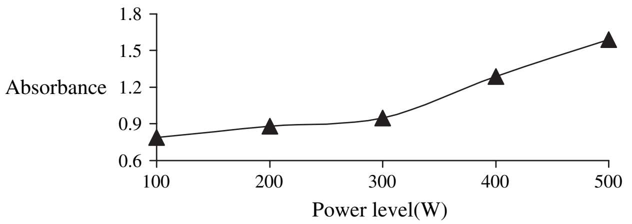

Fig. 4. Effect of ultrasonic power level on the extent of dye extraction. Extraction conditions: 8% w/v dye amount, at 80 °C, 40 min. For legend, see Fig. 2.

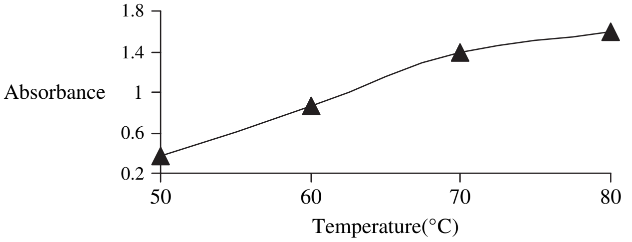

Fig. 5. Effect of temperature on the extent of dye extraction using ultrasonic heating. Extraction conditions: 500 W, 8% w/v dye amount, 40 min. For legend, see Fig. 2.

3.2.2. Effect of salt addition

It is known that salt addition in wool dyeing is necessary in the case of using high affinity acid or reactive dyes as it retards the dye migration and thus obtaining better level dyeings. Fig. 7 shows the effect of salt concentration on the colour strength obtained for the dyed fabrics under both US and CH. It is clearly indicated that in the absence of salt, the colour strength in the case of US is higher than that obtained under CH. As the salt concentration increases, however, the colour strength decreases in both CH and US with pronounced decrease in the latter case than the former. This result emphasizes the effect of power ultrasonic, which helps better dyeing in the absence or presence of salt.

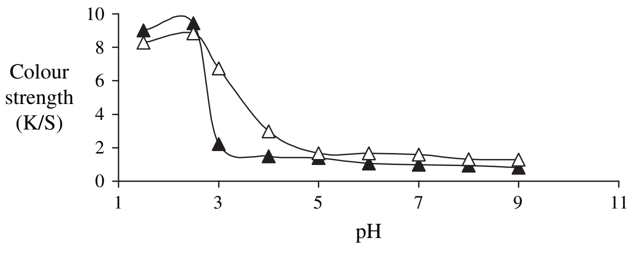

Fig. 6. Effect of dye bath pH on the colour strength of dyed wool fabrics. Dyeing conditions: 500 W, LR 50:1, 50 ml aqueous dye extract (8% w/v), 10 g L^{-1} sodium chloride, 1 h, at 80 °C. For legend, see Fig. 2.

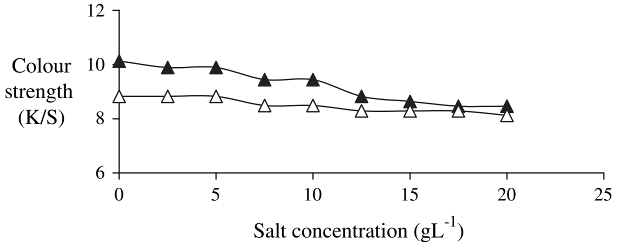

Fig. 7. Effect of salt addition to the dye bath on the colour strength of dyed wool fabrics. Dyeing conditions: 500 W, LR 50:1, 50 ml aqueous dye extract (8% w/v), pH 2.5, 1 h, at 80 °C. For legend, see Fig. 2.

However, the retarding effect was also enhanced in comparison with the case of CH condition.

3.2.3. *Effect of temperature*

The effect of temperature on the dyeability of wool fabrics with lac dye was conducted under US and CH conditions at different temperatures (30–80 °C). As shown in Fig. 8, it is clear that the colour strength increases with the dyeing temperature up to 60 °C in both cases of US and CH with pronounced increase in the latter case than the former. However, as the temperature goes up from 70 to 80 °C, the values of K/S in US were higher than those in CH. On the other hand, at 60 °C or above a plateau value of K/S was observed in CH, the case which was absent in US. Generally, the increase in dye-uptake can be explained by fibre swelling and hence, enhanced dye diffusion. Also, the ultrasonic power provides other additional factor of de-aggregation of dye molecules and thus leading to further enhancement of dye diffusion and better dyeability than CH. Thus, the anomalous behavior of the temperature in the range 30–60 °C may be attributed to propensity of lac dye for surface coloration and dye aggregation, which reveals higher K/S in this temperature range than in the case of US in which the de-aggregation effect is rather effective. At higher temperatures (70–80 °C), however, the effect of power ultrasonic is clear in further dye penetration and thus higher K/S values were obtained relative to the case of CH.

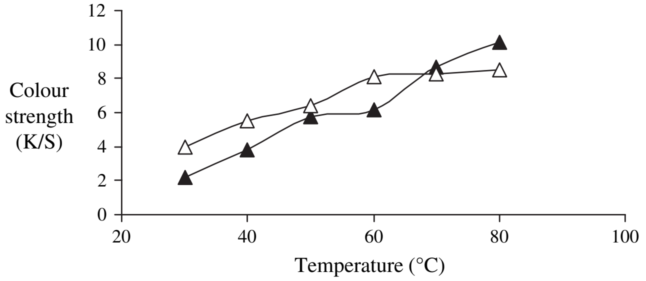

Fig. 8. Effect of dyeing temperature on the colour strength of dyed wool fabrics. Dyeing conditions: 500 W, LR 50:1, 50 ml aqueous dye extract (8% w/v), pH 2.5, 1 h. For legend, see Fig. 2.

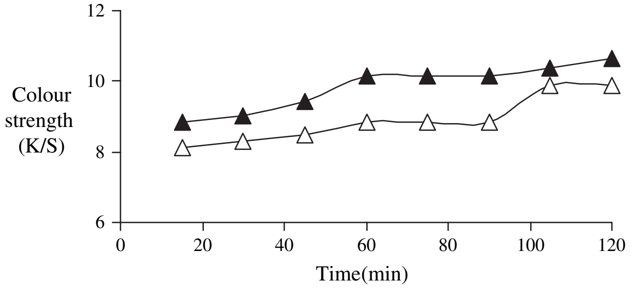

Fig. 9. Effect of dyeing time on the colour strength of dyed wool fabrics. Dyeing conditions: 500 W, LR 50:1, 50 ml aqueous dye extract (8% w/v), pH 2.5, at 80 °C. For legend, see Fig. 2.

3.2.4. *Effect of dyeing time*

The effect of dyeing time was conducted at high concentration of the dye, i.e. 8 g/100 ml water. As shown in Fig. 9, the colour strength obtained was increased as the time increases in both US and CH with much higher colour strength at all points in the US case. A plateau is attained after 60 min and then started to rise up after 90 min of dyeing in both cases. The enhancement effect due to ultrasonic power is rationalized above and the higher dye-uptake observed after 90 min, which is rather clear in CH, may be due to the dye aggregation on the surface of the fibre as a result of the long dyeing time and the remained high concentration of the dye in the dye bath.

3.2.5. *Effect of ultrasonic power*

The effect of ultrasonic power on the dyeability of wool fabrics with lac dye was conducted at different power levels (100–500 W). As shown in Fig. 10, the colour strength of dyed fabrics seemed to be directly proportional with the power supplied. This behavior emphasizes again the assisting effect of power ultrasonic on the dyeability of wool fabrics with lac dye.

This assistance can be explained as has been suggested before [15,24] to dispersion (breaking up of micelles and high molecular weight aggregates into

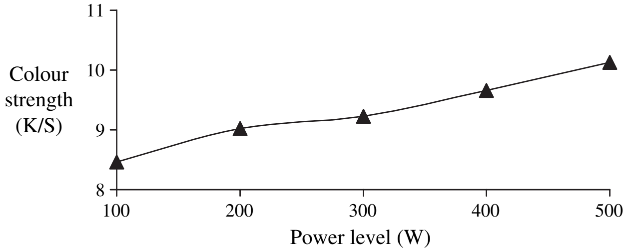

Fig. 10. Effect of power level on the colour strength of dyed wool fabrics. Dyeing conditions: LR 50:1, 50 ml aqueous dye extract (8% w/v), pH 2.5, at 80 °C, 1 h. For legend, see Fig. 2.

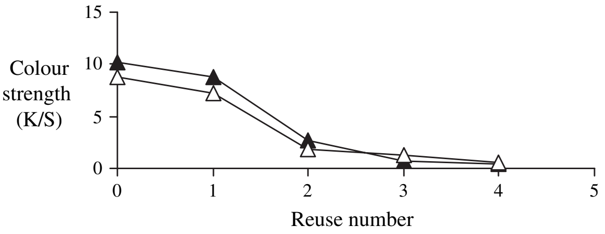

Fig. 11. Effect of reuse number on the colour strength of dyed wool fabrics. Dyeing conditions: 500 W, LR 50:1, pre-exhausted dye bath of 8% w/v (corresponds to zero reuse number in the figure), pH 2.5, at 80 °C, 1 h. For legend, see Fig. 2.

uniform dispersions in the 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).

3.2.6. *Effect of reuse number of the dye bath*

As dyeing has been conducted in concentrated solution of lac dye, it was necessary to check the possibility of reusing the dye bath again and see whether US method would be rather effective in this case. Fig. 11 shows the effect of reuse number on the colour strength obtained. It is clear that US reuse is more effective than in the case of CH and as expected repeating the reuse always leads to different coloured samples from one batch to another until complete exhaustion of the dye bath.

Fastness properties of the US and CH dyed fabrics are shown in Table 2. The results indicate fair to good fastness properties of the dyed samples using both ultrasonic and conventional techniques.

3.3. *Kinetics of dyeing*

It is known that the rate of any process means a change in one of the starting materials that takes place in the process or the product that obtained per unit time. Applying this definition in the dyeing process can be regarded as the change in the dye-uptake per unit time.

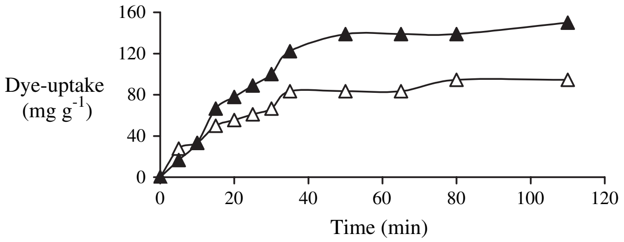

Fig. 12. Ultrasonic and conventional dyeing rates of wool fabrics. Dyeing conditions: for conditions see experimental part and for legend, see Fig. 2.

Time–dye-uptake isotherms of wool fabrics ultrasonically and conventionally dyed with lac dye are shown in Fig. 12. The figure shows that the dye-uptake values of ultrasonically dyed samples are generally better than those prepared by a conventional heating method. Note that the behavior of the dyeing isotherms in this figure indicates that US uptake is lower for the first 10 min. Thereafter, the isotherms of both methods started to be differentiated from each other to show better dye-uptake for lac dye under ultrasonic condition in comparison with conventional heating.

Generally, the dyeing process is a solid/liquid phase process, which proceeds by the movement of the dye molecules from liquid phase to the solid surface of the fibre by virtue of their affinity, and then diffusion takes place inside the fibre. Therefore, the first process would be a fast adsorption controlled process and, once the dye molecules get into the fibre, the second slow process, which is diffusion controlled, starts to take place. Accordingly, the influence of ultrasonic power on the rate of dyeing would become clear in the diffusion process to reveal higher dye-uptake in short time for ultrasonically dyed samples in comparison with conventionally dyed ones. This enhancement effect of ultrasonic power may be due to the cavitation effect. Similar indication for the enhancement effect of ultrasonic on the diffusion rate of nylon dyeing has been recently reported [16]. At the beginning of dyeing, however, there is evidence that US dye-uptake is lower than CH dye-uptake. This may be due to the simultaneous process of de-aggregation of dye molecules, re-orientation of fibre and degassing of the dyeing system under ultrasonic condition. Therefore, one would expect that the fast adsorption process will be favorable under CH and,

Table 2
Fastness properties of dyed wool fabrics under conventional heating and ultrasonic conditions

Dyeing methods	Wash–perspiration–rubbing–light[a]											
	A	C	W	A_a	C_a	W_a	A_b	C_b	W_b	Rd	Rw	LF
CH	3–4	3–4	3–4	4	3–4	4	3–4	3–4	3–4	2–3	1–2	5–6
US	3–4	3–4	3	3–4	3	3–4	3–4	3	3	2–3	1–2	6

[a] A = change in colour, C = staining on cotton, W = staining on wool, Rd = dry rubbing, Rw = wet rubbing, LF = light fastness, subscript a = acidic, subscript b = alkaline.

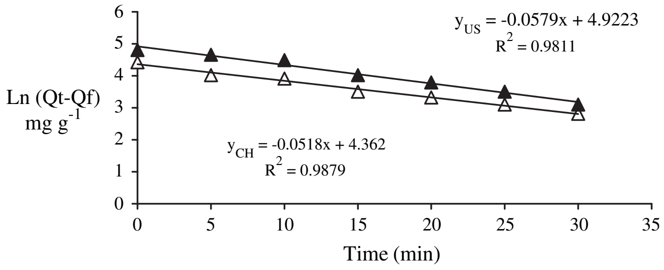

Fig. 13. Plots of $\ln(Q_t - Q_f)$ versus time of dyeing with lac dye under conventional and ultrasonic conditions. For legend, see Fig. 2.

soon after covering the surface of the fabric with dye molecules, the dye-uptake decreases with the dyeing time relative to the case of US condition.

The data in Fig. 12 can be analysed by using the derivable general form of the first order rate Eq. (2): [25,26]

$$\frac{A_t - A_f}{A_0 - A_f} = e^{-kt} \tag{2}$$

where A_t is the absorbance at time t, A_0 is the initial absorbance, A_f is the final absorbance, t is the reaction time and k is the reaction rate. Since the absorbance of solution is directly related to the concentration by Lambert–Beer law, Eq. (2) can be rewritten in terms of dye-uptake to give Eq. (3):

$$\frac{Q_t - Q_f}{Q_0 - Q_f} = e^{-kt} \tag{3}$$

where Q_t is the dye-uptake at time t, Q_0 is the dye-uptake at zero time, and Q_f is the final dye-uptake, t is the dyeing time and k is the dyeing rate. Taking the logarithm of Eq. (3) would lead to Eq. (4) and since Q_f is known, $Q_t - Q_f$ can be calculated.

$$\ln|Q_t - Q_f| = \ln|Q_0 - Q_f| - kt \tag{4}$$

A plot of $\ln|Q_t - Q_f|$ versus time is expected to be linear with a slope of $-k$ and an intercept of $\ln|Q_0 - Q_f|$ if the reaction is first order. Fig. 13 shows the plot of $\ln|Q_t - Q_f|$ as a function of time for dyeing of wool fabrics with lac dye using both ultrasonic and conventional heating methods. As can be seen in this figure, the linear fitting of Eq. (4) holds indeed and the values of dyeing rate constants could be obtained as listed in Table 3.

The time of half dyeing $t_{1/2}$, which is the time required for the fabric to take up half of the amount of dye taken at equilibrium, is estimated either from each isotherm directly (Fig. 12) and/or from the following equation:

$$t_{1/2} = \ln 2/k \tag{5}$$

The values of half dyeing $t_{1/2}$ are given in Table 3. The rate constant of dyeing wool fabrics with lac dye is clearly increased with ultrasonic treatment in comparison with conventional heating. Also, the values of $t_{1/2}$ of dyeing are clearly short for those samples dyed with ultrasonic in comparison with those dyed conventionally.

3.4. Standard affinity

The data for dyeing equilibria are generally reported as the standard affinity of dyeing, $-\Delta\mu$ [27]. It has been reported that the dyeing of wool fibres using natural dyes follows the same mechanism as that of disperse dyes, i.e. via partition mechanism [7]. Therefore, the standard affinity can be calculated using Eq. (6):

$$-\Delta\mu = RT \ln\frac{[C]_f}{[C]_s} \tag{6}$$

where R is the gas constant, T is the absolute temperature (K), $[C]_f$ and $[C]_s$ are dye concentrations in the fibre and the dye bath, respectively. From Table 3, it can be seen that the standard affinity of wool fibre in the case of US is higher than in the case of CH. This is another evidence for the enhancement effect of ultrasonic power, increasing the affinity of lac dye toward wool fibres in comparison with conventional heating.

3.5. Ultrasonic efficiency

Ultrasonic efficiency ($\Delta k\%$) in accelerating the dyeing rate was examined by introducing the following equation:

$$\Delta k\% = \frac{(k_{US} - k_{CH})}{k_{CH}} 100 \tag{7}$$

where, k_{US} and k_{CH} are the rate constants of dyeing with ultrasonic and conventional heating, respectively. As shown in Table 3, the value of ultrasonic efficiency is positive indicating a favorable effect of ultrasonic power on the dyeing process [16].

Table 3
Dyeing rate constant k, efficiency of ultrasonic Δk, times of half dyeing $t_{1/2}$, standard affinity $-\Delta\mu$ and amount of final dye uptake by wool fabrics using lac dye

$k \times 100$ (min^{-1})		Δk (%)	$-\Delta\mu$ (kJ/mol)		$t_{1/2}$ (min)		Q_f (mg/g)	
US	CH		US	CH	US	CH	US	CH
5.79	5.18	11.78	11.68	9.39	11.97	13.38	122.22	83.33

4. Conclusion

Ultrasonics proved effectiveness in the dye extraction and dye-uptake of wool fibres with lac dye, the enhanced effect was about 41% and 47% more than conventional heating, respectively. This technique in addition to its advantage of saving the processing time and energy offers better environmental impact as it helps much dye-uptake and also efficient dye bath reuse.

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