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Ultrasound for low temperature dyeing of wool with acid dye

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ABSTRACT

The possibility of reducing the temperature of conventional wool dyeing with an acid levelling dye using ultrasound was studied in order to reach exhaustion values comparable to those obtained with the standard procedure at 98 °C, obtaining dyed samples of good quality. The aim was to develop a laboratory method that could be transferred at industrial level, reducing both the energy consumption and fiber damage caused by the prolonged exposure to high temperature without the use of polluting auxiliary agents.

Dyeings of wool fabrics were carried out in the temperature range between 60 °C and 80 °C using either mechanical or ultrasound agitation of the bath and coupling the two methods to compare the results. For each dyeing, the exhaustion curves of the dye bath were determined and the better results of dyeing kinetics were obtained with ultrasound coupled with mechanical stirring. Hence the corresponding half dyeing times, absorption rate constants according to Cegarra–Puente modified equation and ultrasonic efficiency were calculated in comparison with mechanical stirring alone. In the presence of ultrasound the absorption rate constants increased by at least 50%, at each temperature, confirming the synergic effect of sonication on the dyeing kinetics. Moreover the apparent activation energies were also evaluated and the positive effect of ultrasound was ascribed to the pre-exponential factor of the Arrhenius equation. It was also shown that the effect of ultrasound at 60 °C was just on the dye bath, practically unaffecting the wool fiber surface, as confirmed by the results of SEM analysis.

Finally, fastness tests to rubbing and domestic laundering yielded good values for samples dyed in ultrasound assisted process even at the lower temperature. These results suggest the possibility, thanks to the use of ultrasound, to obtain a well equalized dyeing on wool working yet at 60 °C, a temperature process strongly lower than 98 °C, currently used in industry, which damages the mechanical properties of the fibers.

1. Introduction

In literature it has been reported that ultrasonic energy can be successfully applied to the textile wet processes, for example laundering [1], desizing, scouring, bleaching, mercerization of cotton fabric [2], enzymatic treatment [2,3], dyeing [4], leather processing [5], together with decoloration/mineralization of textile dyes in waste water [6]. Acoustic cavitations and the related effects, such as the formation of microjets are the physical mechanisms behind the ultrasonic textile wet processes [7].

Dyeing is a solid/liquid phase process which proceeds through the migration of the dye molecules from the bath to the solid surface of the fiber. Once the dye molecules get into the fiber, a slow process, which is diffusion controlled, starts to take place. The basic idea in ultrasound-assisted dyeing processes was that ultrasound can enhance the mass transfer by reducing the stagnant cores in the yarns. Improvements observed are generally attributed to cavitations phenomena and to other consequent physical effects such as dye dispersion (breaking up of aggregates with high relative molecular mass), degassing (expulsion of dissolved or entrapped air from fiber capillaries), strong agitation of the liquid (thickness reduction of fiber-liquid boundary layer), swelling (enhancement of dye diffusion rate inside the fiber). The acceleration in dyeing rates observed by many authors might be the cumulative effect of the above.

The behavior of ultrasonic waves in dye baths containing different fabrics was also studied. Waves transmission, reflectance and adsorption by fabrics were measured and it was found that textile fabrics transmit very little high intensity ultrasound, reflecting most of the sound energy. Even very light fabrics allow less than 4% of the high intensity ultrasonic energy to pass through. The ultrasonic energy transmitted through the fabric was slightly greater at 25 kHz than at 40 kHz or with fabrics not too hairy. It means that fabrics absorb negligible ultrasonic vibrational energy since most of the waves are reflected or transmitted [8] and the effects on their morphology are limited.

Moreover, approaches to the transfer of the process at industrial level were reported and design requirements for industrial size

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ultrasound bath for textile treatments were determined. Finite element analysis (FEA) was applied to investigate spacing and alignment of the ultrasound source transducers to reach effective and homogenous acoustic pressure distribution in the bath. Effects of sound pressure level, bath temperature, bath volume, textile material type and hydrophilicity degree of fabric were taken into account [9].

For what concern the environmental impact after dyeing, some authors monitored the selected pollution parameters in dye bath effluents, i.e. pH, total organic carbon (TOC), chemical oxygen demand (COD), and biochemical oxygen demand (BOD_5). The obtained results indicated higher exhaustion level of dyestuffs during ultrasound assistance in comparison to conventional dyeing, enabling a reduction in dyeing time and energy consumption. Moreover, the selected pollution parameters were diminished in all the dye-baths effluents after ultrasonic dyeing, with environmental protection improvement [10].

The effect of ultrasonic energy on dyeing processes was already widely investigated on cotton [11], cationized cotton [12], silk [13], acrylic [14], nylon [15] and polyester fibers [16–18] with good effects in all cases. However the wool structure is more complex than that of such fibers, since it can be represented as a set of compact protein units, cuticular and cortical cells, surrounded by a keratin cell membrane, held together by intercellular cement. Cuticle constitutes a real barrier to the transfer of dye molecules from aqueous solution causing a significant deviation from the theoretic model of dye diffusion (Fick's law) [19].

A good wool dyeing process must provide a satisfactory exhaustion of dye bath and an adequate penetration of dye into the fiber, with the practical advantages of good wet fastness and uniform coloration. The conventional methods for wool dyeing are based on long times at temperature of the bath close to the boiling point, in order to ensure good results of dye penetration and levelling. These conditions can damage the fibers, with bad effects on the characteristics of the finished material. The extent of the damage that can be caused to dyed wool depends on pH and time–temperature profile of the dyeing cycle. When the wool is maintained at temperatures near 100 °C in acid ambient for long times, the structure of the fiber is gradually damaged by hydrolysis of peptide bonds. Such damage can be minimized by reducing the operation time or, better yet, by reducing the dyeing temperature.

Therefore many low temperature wool dyeing processes were proposed by introducing specific auxiliary agents [20,21] and enzyme or plasma pre-treatments [22–24]. Recently, ultrasound assisted wool dyeing was studied with the aim to reduce temperature or dyeing time with respect to the conventional dyeing technique. Kamel et al. [25] found a significant improvement of kinetics in wool dyeing with natural dye. McNeil and McCall [26] reported that ultrasound show potential to reduce the chemical and energy requirements in wool dyeing with reactive and acid milling dyes, but not with acid levelling dyes. Furthermore, Battù et al. [27] observed that in wool dyeing at 85 °C with acid dyes ultrasound caused an enhancement of the dyebath exhaustion as high as about 25%, or vice versa a dyeing time nearly 20% shorter than conventional dyeing. Yükseloğlu and Bolat [28] confirmed these improvements using ultrasound in dyeing of wool fabrics at 80–90 °C with a pre-metallized dye.

In the present work the possibility of reducing the temperature of conventional wool dyeing with a typical acid levelling dye using ultrasound was further investigated, in order to reach exhaustion values comparable to those of the standard procedure, obtaining dyed samples of good quality. The aim was to develop a laboratory method that could be transferred at industrial level, reducing both the energy consumption and fiber damage caused by the prolonged exposure to high temperature without the use of polluting auxiliary agents which are commonly used to improve the mass transfer kinetics of dyes onto the fiber surface.

2. Experimental

2.1. Materials

Textile material was pure wool knitted fabric EMPA Mousseline, 200 g/m^2, yarn 2/48 Nm (2 fibers) 550 tors/mz, single fiber 360 tors/ms, previously washed for 10 min with 1 g/l solution of ECE surfactant and 10 ml/l NH_3 (33%), followed by rinsing first in lukewarm, then in cool water to completely eliminate foam which might affect the uniform migration of dye on the fabric.

The dye chosen was Telon Blue 100% (Acid Blue 80) by Dystar. It is a disulfonate acid dye that presents a maximum absorbance peak at 626 nm. Acetic acid of laboratory grade by Sigma–Aldrich was used for acidification.

2.2. Dyeing process

The first aim of this study was the determination of isothermal exhaustion curves of the dyebath, with mechanically or ultrasound alone agitation and coupling both, at different dyeing temperatures: 60, 70 and 80 °C. The experiments were performed without auxiliary chemicals.

An Elmasonic S60H (Elma GmbH & Co., Singen, Germany) ultrasonic and thermo-controlled bath was used. It can generate ultrasound at 37 kHz with effective ultrasound power of 150 W and heating power of 400 W.

The dyeings were made on 2.00 g wool samples introduced in a beaker containing the dyebath. Then the beaker was immersed in the water bath of the ultrasonic equipment. Mechanical agitation was provided by a magnetic stirrer turning at 110–120 rpm, plunged in the same bath. Tests without ultrasound were carried out with the sonication turned off.

A 1:50 material to liquor ratio was chosen, with 1% o.w.f. (over weight fiber) dye amount at pH adjusted to 4 by acetic acid addition. The dyeing temperature was maintained for 110 min, analyzing bath samples every 10 min to monitor dye exhaustion. The measurements were performed with an UV–VIS spectrophotometer UNICAM UV2 (ATI Unicam, Cambridge, UK) and evaluated by "Vision 32" software basing on Lambert–Beer law.

Finally, the dyed samples were squeezed, thoroughly rinsed with cold water, and dried at 100 °C.

2.3. Ultrasound effect evaluation

From exhaustion curves, besides the final bath exhaustion reached, times of half dyeing were also determined. It is the time required by a fabric to adsorb half the amount of dye adsorbed at equilibrium, so as it is small, the more dyeing process is fast.

Moreover, to evaluate the effect on wool dyeing produced by ultrasound, absorption rate constants and apparent activation energies were determined.

Adsorption rate constants were calculated from the exhaustion curves, by fitting the experimental values according to Cegarra–Puente modified kinetic equation [17]:

$$\ln\left[-\ln\left(1-\frac{E_t^2}{E_\infty^2}\right)\right] = a\ln t + a\ln K \tag{1}$$

where E_t is the dye concentration in the fiber at the time t, E_∞ the dye concentration at the equilibrium, K the absorption rate constant, and t is the dyeing time.

Arrhenius equation was used for apparent activation energy calculation:

$$K_T = K_0 e^{-\frac{E}{RT}} \tag{2}$$