WATER IN SUPERCRITICAL CARBON DIOXIDE DYEING

by

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This paper investigates the effect of water serving as entrainer on the dyeing of wool fabrics in supercritical carbon dioxide. Compared with previous supercritical dyeing methods, addition of water makes the dyeing process more effective under low temperature and low pressure. During dyeing process, dyestuff can be uniformly distributed on fabrics's surface due to water interaction, as a result coloration is enhanced while color difference is decreased.

Key words: *entrainer, wool fabrics, supercritical carbon dioxide, reactive dyes*

Introduction

Supercritical carbon dioxide (SC-CO_2) dyeing has high diffusivity and low viscosity that makes the dyeing process faster than water dyeing, meanwhile SC-CO_2 exhibit economical and ecological advantage [1-6]. The study of wools dyeing focused on the relation between dyes and the amino group of wools. In conventional water dyeing process, chemical bond was generated when using lanasol dyestuff as reactive dyestuff [7-9]. However, the common reactive dye failed to be dissolved in the SC-CO_2. In order to solve this problem, researchers considered introducing entrainers and thus entrainers were introduced to enhance the polarity of SC-CO_2 and dyes, including methanol, ethanol and water, and so on. Among these entrainers, water is the cheapest and most easily obtained. M. van der Kraana *et al.* [10] claimed that the addition of water enhanced colouration when dyeing natural fibres with reactive disperse dyes in the SC-CO_2. The aim of this study was to investigate the effect of water as entrainer on the coloration in wools dyeing.

Experimental

Materials

The lanasol red G shown in fig. 1 was provided by Ruyi Woolen Textile Co., Ltd (Jining, China). The wool fabrics were from Fuxin Wool Textile Group Co., Ltd (China). The carbon dioxide (99.97%) was purchased from Zhonghao Guangming Research & Design Institute of Chemical Industry Corporation (China). Demineralised water was used in pretreatment and as entrainer.

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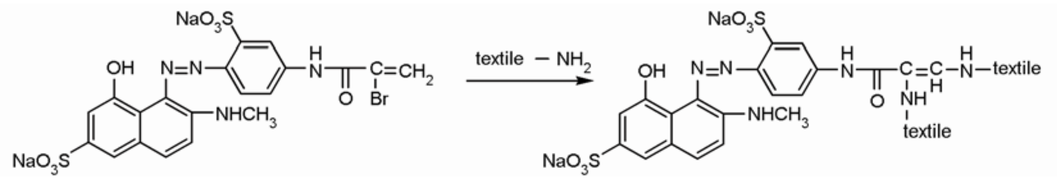

Figure 1. Reaction of reactive dyes with textile amino groups

Equipment and procedure

The high-pressure apparatus used in this study was shown in fig. 2. The dyestuff (0.2 ± 0.01 g) was dissolved in different amounts of entrainer. The wool fabrics (10 ± 0.2 g) were folded in the dyeing vessel so that the mixture of SC-CO_2 and entrainer was forced to flow through the layers of textile. In the experiments of SC-CO_2, the wool was prior placed in the vessel and wetted with different amounts of water. The temperature of dyeing process was determined by setting the temperature of an electric heater. The system was pressurized with a pump. The CO_2 was pumped with flow rate of 20 ± 0.2 g per minute, and the entrainer was pumped with flow rate of 0.4 ± 0.02 g per minute. When the temperature and pressure reached the setting values, the circulation pump was started. The amount of entrainer added into the dyeing system varied in each experiment.

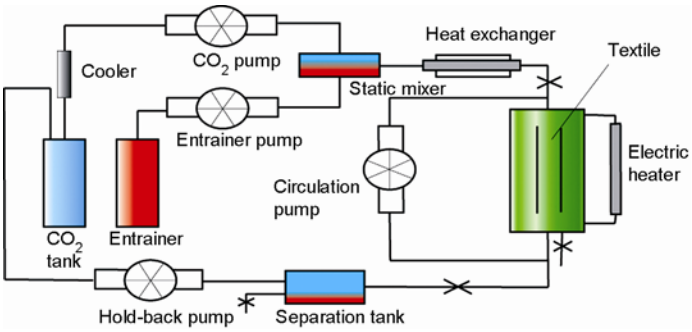

Figure 2. High-pressure dyeing apparatus

Colour analysis

The dyed pieces of wool were analyzed by measuring the reflectance curve between 350 and 750 nm with Color iQC and Color iMatch. The minimum of the reflectance curve ($R_{\min}$) was used to determine the ratio of light absorption (K) and scatter (S) *via* the Kubelka-Munk function:

$$\frac{K}{S}=\frac{(1-R_{\min})^2}{2R} \tag{1}$$

A point is selected on the center of fabric as the contrast and ten points on the fabric are randomly selected, and then the value of ΔE_n is measured *via* the equation:

$$\Delta E_n=\sqrt{(\Delta L_n-\Delta L_0)^2+(\Delta a_n-\Delta a_0)^2+(\Delta b_n-\Delta b_0)^2} \tag{2}$$

$$\Delta E=\frac{1}{n}\sum_{i=1}^{n}\Delta E_i \tag{3}$$

where ΔE, ΔL, Δa, and Δb stand for value of color difference, lightness value, reddish or greenish value, and partial-ellow or partial-blue color value, respectively. The subscript (0) stands for the value of the center point of fabric and the subscript (n) stands for the value of randomly selected points from one to ten.

Results and discussion

Effect of adding entrainer on the solubility of dyes

In the SC-CO_2 dyeing process, the dye powder was firstly dissolved in the SC-CO_2 and then transported towards the textile. The solubility of the dye impacts the transport of dye towards the textile [11]. The distribution of dyestuff on the surface of fibers whether using water as entrainer or not was tested respectively. As shown in fig. 3(a), massive dyestuff exhibit on the surface of the fiber without entrainer which reflected the dyestuff did not well distribute in the SC-CO_2. Figure 3(b) showed on the surface of the fiber there was no massive dyestuff which reflected the dyestuff well-distribute in the SC-CO_2 and the dye permeated into the fiber with entrainer. Therefore, the solubility of dyes on the surface of wool fibers was obviously improved when adding entrainer from fig. 3.

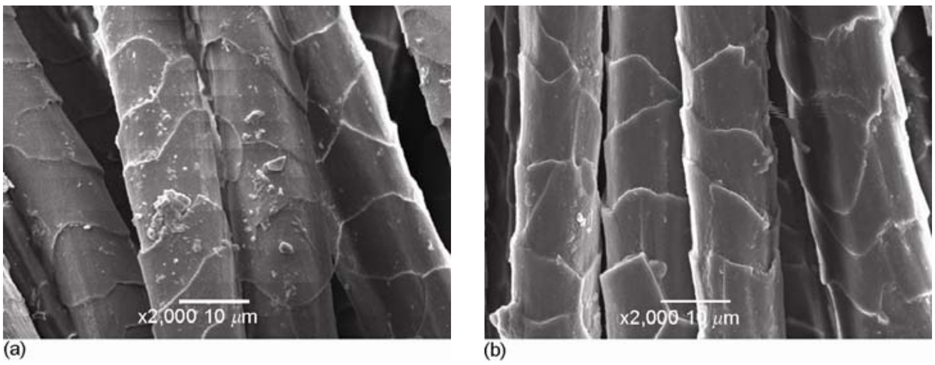

Figure 3. The scanning electron microscopy (SEM) images of the wool samples dyed without entrainer (a), and with entrainer, (b) in SC-CO_2 fluid

Effect of entrainer on the value of K/S and color difference

The influence of water amount was investigated in a series of experiments. Prior to the experiments, textiles were soaked in demineralised water of 5 mL. The amounts of entrainer were 10 mL, 20 mL, 30 mL, 40 mL, and 50 mL, respectively. The influence of the various amount of entrainer on the colouration (K/S) and color difference (ΔE) of textiles dyed in SC-CO_2 for 60 min at the pressure of 20 Mpa and temperature of 90 °C was shown in fig. 4.

The results exhibited that higher colouration (K/S) and lower color difference (ΔE) were obtained when water was added. The experiments gave maximum colouration (K/S) was 9.57 when the amount of entrainer was 50 mL. The value of K/S increased significantly when the amount of water changed from 30 mL to 40 mL. The value of K/S increased by 23% representing better coloration while the color difference (ΔE) decreased by 65% which indicated better levelness.

Therefore, it can be stated from the above results that water as entrainer plays a positive role in the reaction between the dye and the fiber. Because entrainer was a polar solvent, it can enhance the binding force between the dye and the fiber. It also increased the solubility of reactive dyes in the SC-CO_2 with adding the amount of entrainer.

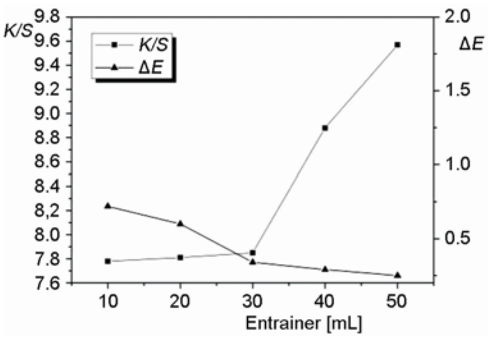

Figure 4. Influence of the amount of entrainer on the colouration (*K/S*) and color difference (Δ*E*) of textiles dyed in SC-CO_2

Conclusion

Reactive of lanasol dyes are suitable for dyeing wool textiles in SC-CO_2. According to the results observed by SEM, it was shown that the solubility of dyes on the surface of wool fibers was obviously improved with adding entrainer. Meanwhile, the analysis results of *K/S* and color difference of wool fibers showed that the colouration increased with the amounts of entrainer in the SC-CO_2 and the textiles. Experiments showed that maximum colourations of wool are obtained when both the SC-CO_2 and the textiles are saturated with entrainer. The positive effect of water on the dyeing process is caused by the enhancement of solubility of reactive dyes in the SC-CO_2. For this phenomenon, it is concluded that entrainer should be added in supercritical dyeing of wool with lanasol dyes. However, to make this conclusion certain, more researches on solubility and reaction kinetics should be done in the future.

Acknowledgments

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