EFFECT OF PRESSURE OF SUPERCRITICAL CARBON DIOXIDE ON MORPHOLOGY OF WOOL FIBERS DURING DYEING PROCESS

by

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In this paper, the effect of pressure release rate on dyeing of wool fibers was studied in the supercritical carbon dioxide dyeing. Surface morphology, chemical composition and color difference at different pressure release rates were investigated by employing scanning electron microscope, color matching, and measuring instrument. Experiment data reveal that wool fibers are easy to be damaged with increasing pressure release rate. Fiber's surface brightness varies also with the pressure release rate. The phenomena are theoretically explained using Bernoulli's principle.

Key words: *wool fibers, supercritical carbon dioxide, color difference, pressure release rate, Bernoulli's principle*

Introduction

Dyeing of wool fibers in supercritical carbon dioxide is environmental friendly for cleaner production, which can avoid a large amount of effluent discharge and serious environment pollution as those in conventional chemical processes [1, 2]. As a dyeing medium, supercritical carbon dioxide has the characteristics of cleanness, environmental protection and sustainable utilization, thus having broad application prospects, and the supercritical carbon dioxide dyeing technology has also gained increasing attentions. It was reported that the high temperature procedure of this technology had certain impacts on wool fibers. For example, Schmidt *et al.* [3] examined and discussed the damage in different kinds of fibers in supercritical carbon dioxide under the temperatures of 100, 120, 140, and 160 °C, respectively. As a matter of fact, pressure procedure also plays important roles in the dyeing process of wool fibers in supercritical carbon dioxide. However, the effect of pressure release rates on wool fibers in the supercritical carbon dioxide had rarely been reported in open literature, therefore a theoretical insight into the supercritical carbon dioxide dyeing is much needed for industrialization production of wool fibers.

Experimental

Wool fibers were placed in the supercritical carbon dioxide dyeing device for staining. As shown in fig.1, dyeing process was carried out in the dyeing kettle (7). Carbon dioxide stored in the carbon dioxide tank (1) was filtered by a purifier (2), and then cooled into liquid by a precooler (3). As pressure promoted by a high-pressure pump (4) and temperature

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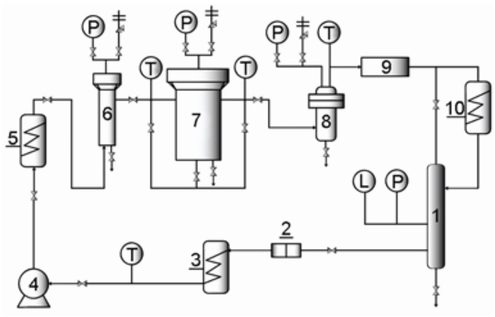

Figure 1.The flow chart of supercritical carbon dioxide dyeing system
(1) carbon dioxide tank, (2) purifier, (3) precooler, (4) high-pressure pump, (5) heat exchanger, (6) dye kettle, (7) dyeing kettle, (8) separator, (9) flowmeter, (10) refrigerator

elevated by a heat exchanger (5) in the treatment system were attained at a required condition, the dyeing process began. The pressure release rate was controlled by the flowmeter (9). After a required dyeing period, the wool fibers was removed. Finally, carbon dioxide would be changed into gas in separator (8) and cooled into liquid by a refrigerator (10).

Results and discussion

Wool is a kind of protein fiber, and is mainly composed of the cuticle, cortex and medulla layer. Supercritical carbon dioxide fluid may produce rapid air flow during pressurize and release pressure process which will affect scale layer of wool fibers [4]. The air flow may result in changes of surface morphology of fibers. The effect of release rates on the changes of surface morphology of wool fibers were explored in supercritical carbon dioxide fluid by the scanning electron microscope (SEM) (JSM-6460LV, JEOL, Japan). The release rates of pressure were 10 L/min, 40 L/min, and 70 L/min, respectively. As is shown in fig. 2(a), the morphological characteristics of unstained wool fibers showed their integrity and obviously visible height scales. The morphological scales of stained wool fibers with different pressure release rates were obviously damaged in figs. 2(b), (c), and (d), and their visible height and coverage density also appeared to decrease. Therefore, the surface of wool fibers was changed significantly under different pressure release rates of supercritical carbon dioxide fluid dyeing from fig. 2. The damage was aggravated with the pressure release rate increasing from 10 L/min to 70 L/min.

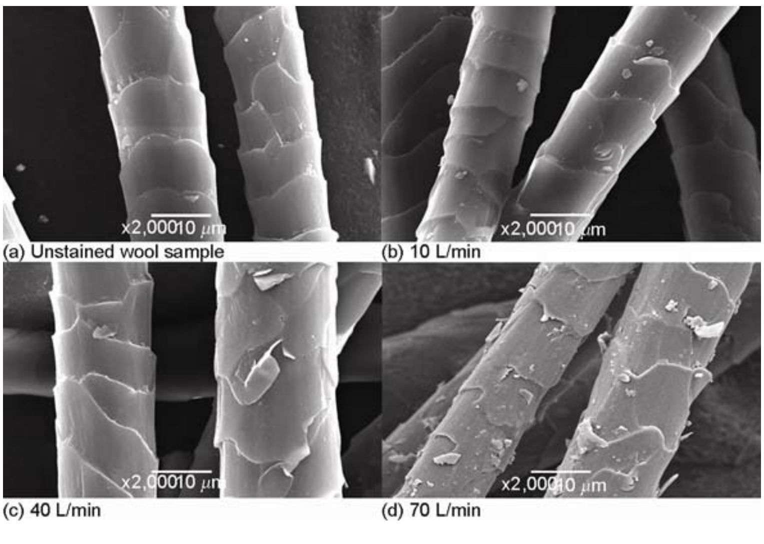

Figure 2. The SEM images of the wool samples before dyeing (a), and dyed at system pressure release rate of 10 L/min (b), 40 L/min (c), and 70 L/min (d) in supercritical carbon dioxide fluid (25 MPa, 70 min, 120 °C)

The color difference of wool fibers was measured by employing a color matching and measuring instrument (Datacolor SF600, America). The color difference value is usually calculated by the formula of CIE DE 2000 and expressed in ΔE:

$$\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{\frac{1}{2}} \tag{1}$$

where ΔE, ΔL^*, Δa^*, and Δb^* stand for value of color difference, lightness value, reddish or greenish value, and partial-yellow or partial-blue color value, respectively. The effect of pressure release rates on the color difference of wool fibers at different pressure release rates were shown in supercritical carbon dioxide in tab. 1. It can be seen from tab. 1 that the brightness value and partial-red value of wool fibers decreased with the pressure release rate increasing. The destruction of scales increased the diffuse reflectance, so the brightness value and partial-red value of wool fibers decreased.

Table 1. The color difference of wool fibers dyed with different pressure release rate in the supercritical carbon dioxide (25 MPa, 70 min, 120 °C)

Samples	ΔL*	Δa*	Δb*	ΔE*
The control sample	0	0.04 R	0.57 Y	0.57
Rate at 10 L/min	–35.70 D	44.36 R	–3.99 B	57.08
Rate at 40 L/min	–40.37 D	39.80 R	–4.53 B	56.87
Rate at 70 L/min	–49.75 D	36.33 R	–4.03 B	61.73

Wool fibers are a kind of hydrophilic fibers and they have a higher moisture regain in the air. According to the phase diagram of water, the water would form a liquid droplet which will attach scale layer in wool fiber when the dyeing conditions of pressure and temperature changed. When the wool fibers were dyed in supercritical carbon dioxide fluid, the airflow of the outside scale layer sped fast while the inside airflow sped slowly during the process of pressure release. To simplify the calculation model, the pressure releasing was regarded as the laminar flow and fibers were located in the bottom [5, 6]. According to the usual form of Bernoulli's equation [7-9]:

$$\frac{v^2}{2} + \frac{p}{\rho} + \mathrm{g}z = \text{constant} \tag{2}$$

where v, p, ρ, g, and z stand for velocity at a point, pressure, density, acceleration of gravity, and height above an arbitrary reference level, respectively. With the fluid speed increasing, the pressure on the interface which contact with the object and fluid will decrease. In contrast to the case, the pressure will increase. The power of the pressure would produce a great destructive force to wool scales because of their small area. As shown in fig. 3, when a drop of water is affected by a force pointing to the lateral direction of wool scale, it burst open due to the destructive force and then resulted in extensive damage to the scale layer. The pressure became smaller with increasing the fluid velocity. Consequently, the scale layer could be damaged more seriously if the force increased with the acceleration of pressure release rates.

Conclusions

The effect of dyeing pressure release rate on wool fibers in supercritical carbon dioxide were investigated and discussed. According to the results observed by SEM, it was shown that the pressure release rates had significant damage to scale layer of wool fibers. The color difference results indicated that the pressure release rate became faster, the brightness value became smaller. Meanwhile, based on the Bernoulli's principle, the reason of damaged

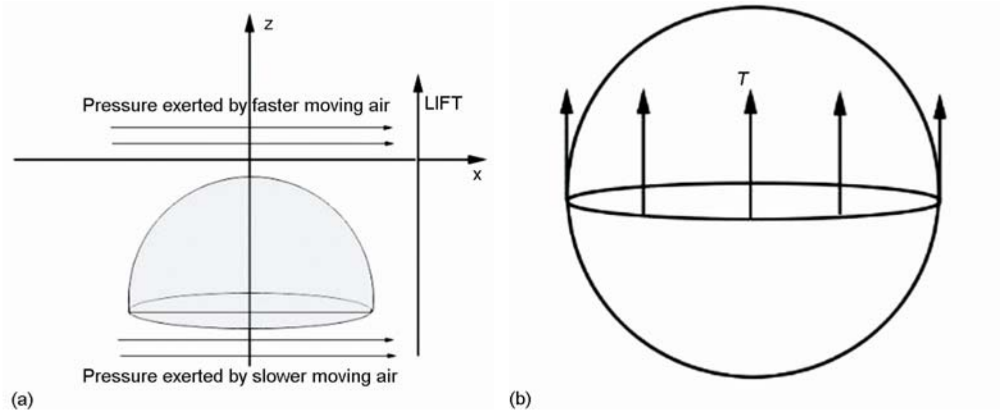

Figure 3. Force analysis of a drop of water in the internal of scale based on Bernoulli's principle; (a) Bernoulli's principle, (b) force analysis of a drop of water

fibers surface by the pressure release rate was analyzed; the relevant analysis and simulation work were done. The faster pressure release rate speed, the greater destructive force to the scale layer. Therefore, in order to avoid the deterioration of damage degree of scale layer structure of wool fibers, the pressure release rate can be controlled as slowly as possible.

Acknowledgments

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