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Using supercritical carbon dioxide as solvent to replace water in polyethylene terephthalate (PET) fabric dyeing procedures

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ABSTRACT

Dyeing fabrics in supercritical carbon dioxide (SCD) instead of water can save energy, reduce water use and prevent pollution. The special pilot plant was designed to test dyeing procedures in supercritical carbon dioxide and the analyses of the results indicate major benefits as compared to water based procedures. The dyeing of polyethylene terephthalate (PET) fabric in supercritical carbon dioxide using special pilot plant was investigated. Disperse dye, C.I. (color index) Disperse Blue 79, was used in this study. After dyeing, rinsing in supercritical carbon dioxide, which removes the excess dyes, was also discussed. At the same dyeing conditions, *K*/*S* (color yield) of dyed fabric significantly increased with increasing the dye concentration from 1% o.w.f. (on weight of fabric) to 5% o.w.f. Dyeing temperature and pressure had a strong influence on the color yield. When the temperature rose above 110 °C, the increase in color yield was obvious. At 20 MPa, 120–130 °C, dyeing reached equilibrium after 60 min. The excess dye of the dyed PET fabric was small. The suitable condition in supercritical carbon dioxide for removal of excess dye from the dyed fabric was 70 °C, 20 MPa. The PET fabric dyed in supercritical carbon dioxide had good fastness and physical properties.

1. Introduction

Clean water is a limited resource. Conventional dyeing of textile fabrics is generally performed in water based media (Ali et al., 2009; Hou et al., 2009; Xie et al., 2008). However, this process has intrinsic environmental problems including water pollution due to the inevitable use of an excess amount of water and the discharge of various chemical additives (Xie et al., 2009; Vankar et al., 2007; Bechtold and Turcanu, 2007). Moreover, a subsequent drying process with high energy consumption is necessary. Conventional dyeing process of polyethylene terephthalate (PET) fiber discharges much wastewater that is contaminated by various kinds of dispersing agents, surfactants and unused dye (Xie and Hou, 2008; Hicks and Dietmar, 2007). With all these additives the wastewater cannot be treated with conventional biological treatment. The wastewater treatment increases the cost of the dyeing process and these costs can be expected to rise when legislation would become more stringent (Lu et al., 2009; Ibrahim et al., 2008). In order to reduce or even eliminate this disadvantage, a new dyeing procedure was developed where carbon dioxide in supercritical conditions was used instead of water (Hou and Dai, 2005; Bach et al., 2002; Fernandez Cid et al., 2004; Lewis, 1999).

Even though several substances are useful as supercritical fluids, Carbon dioxide has been the most widely used because it is inexpensive, essentially nontoxic, nonflammable, recyclable, abundant, and chemically inert under most conditions (Bach et al., 2002; Lewis, 1999). Carbon dioxide has easily accessible critical conditions (31 °C, 7.4 MPa). Above the critical point of carbon dioxide, it retains the free mobility of the gaseous state, but with the increasing pressure its density will increase towards that of a liquid. Solvating power is proportional to density, whilst viscosity remains comparable with that of a normal gas, so the “fluid” has remarkable penetration properties (Lewis, 1999). It is very important in dyeing. The carbon dioxide gas used comes from industrial combustion and fermentation processes, ammonia synthesis and mineral springs, therefore, by using carbon dioxide in dyeing processes no additional carbon dioxide is produced. Supercritical carbon dioxide (SCD) dyeing technology is an innovative method to reduce waste discharge and energy consumption for the textile industry. This technology was applied firstly to dye synthesis fibers by using carbon dioxide, instead of water together with dispersing agent and surfactants, as a carrier of dispersion dyestuffs. Due to the SCD dyeing process without using water and other liquid solvents, the following drying step is not required. Both carbon dioxide and residual dye can be reused. This new clean dyeing technique appears to be economically attractive for the textile industry (Lin et al., 2001).

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Researches on dyeing of synthetic fibers in supercritical fluid have been reported (De Giorgi et al., 2000; Kauhara et al., 2001; Wen and Dai, 2007), and the effect of SCD dyeing conditions on the morphology of fibers has been investigated (Drews and Jordan, 1994; Hou et al., 2004). The solubility of disperse dyes in SCD and the influence of supercritical carbon dioxide on crystal growth and morphological changes of dye powder have also been reported (Bao and Dai, 2005; Shinoda and Tamura, 2003; Lin et al., 2001; Hou and Dai, 2009; Bach et al., 2001).

In this work, the special pilot equipment for SCD dyeing was designed and made. The dyeing of PET fabric in SCD was investigated. Because of C.I. Disperse Blue 79 being a commercially important disperse dye capable of dissolving in SCD over a wide range temperature and pressure (Bach et al., 2001), it was used to dye PET fabric. The dyeing properties of PET fabric with C.I. Disperse Blue 79 in SCD were investigated. After dyeing, rinsing in SCD to remove the excess dyes was also discussed.

2. Serial dyeing experiment

2.1. Materials

PET fabric (60 g/m^2, plain weave, weft and warp 100 den) by scouring and heat-setting was obtained from Wujiang Textile Factory (Suzhou, China). C.I. Disperse Blue 79 was obtained as powder from Hangzhou Jihua Chemical Company (Hangzhou, China) and used without further purification. The dye powder did not contain any additives, such as dispersing agents and surfactants, and it was press cake. The molecular structure of the dye is shown in Scheme 1. The purity of carbon dioxide was 99.99%.

2.2. Dyeing

To optimize the dyeing process in SCD, PET fabric (*ca.* 16 g) was dyed with the calculated quantity of dye at different temperatures (80, 100, 120 or 130 °C) and pressures (14, 17, 20 or 23 MPa). The dyeing times were 5, 10, 15, 20, 30, 40, 60 or 90 min.

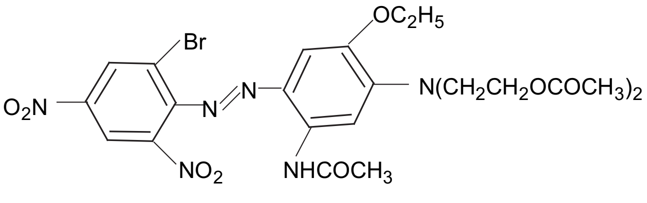
2.3. Removal of excess dye

Reduction clearing for the dyed PET fabric was carried out with sodium hydrosulphite (85%), 2 g/L, sodium hydroxide (30%), 3.5 g/L for 15 min at 70 °C, ratio being 1:80 (meaning 1 g dyed fabric to 80 g solution).

The dyed PET fabric was rinsed in SCD at different temperatures (70 or 80 °C) and constant pressure (20 MPa).

2.4. Analysis of the results

The color yield (K/S) of the dyed PET fabric was determined by a Datacolor SP600$^+$ spectrophotometer (Datacolor, USA). The tristimulus values of the dyed samples were measured in the visible spectrum region 360–700 nm, and the reflectance at the wavelength of maximum absorption ($\lambda_{\max}$) was used to calculate the color yield of dyed fabrics by the Kubelka–Munk equation (Eqn (1)).



Scheme 1. Chemical structure of C.I. disperse blue 79.

$$K/S = \frac{(1-R)^2}{2R} \qquad (1)$$

where K is the absorption coefficient of the substrate, S is the scattering coefficient of the substrate and R is the reflectance of the dyed fabric at $\lambda_{\max}$.

The fastness to washing and rubbing were determined according to ISO 105-C03: 1989 by Roaches Washtec-P and ISO 105X-12: 1993 by AATCC Standard Instrument Atlas CM-5, respectively.

The breaking and tearing strengths were measured according to ISO 13934.1—1994 by Hounsfield Test Equipment H5KS and ISO 13937.1: 1995 by Elmendorf Tearing Tester, respectively.

3. Result and discussion

3.1. Pilot plant design

In order to dye fabric levelly, the dynamic pilot plant for dyeing in SCD was designed. The principle of SCD dyeing pilot plant is shown in Fig. 1. Compressing process: Liquid carbon dioxide was introduced into the whole system by means of an injection pump, and the system was heated at the same time (shown in Fig. 1a). Dyeing circulation process: When the system reached the desired temperature and pressure, the medium was passed into the dyeing autoclave with a pump and circulated for a specified time (the same as the dyeing procedure, shown in Fig. 1b). Unloading pressure process: After certain circulating time, the dyeing and dye autoclaves were cooled as rapidly as possible. The pressure was gradually reduced to atmospheric pressure via a separating vessel (Fig. 1c). Removal of excess dye: The pressure of the dyeing autoclave with the dyed fabric was reduced to certain pressure via a separating vessel and carbon dioxide went into the tank. Then, the liquid carbon dioxide was introduced into the dyeing autoclave by the injection pump. This circulating process was carried out for certain time at suitable pressure and temperature (Fig. 1d).

The SCD dyeing pilot plant is consisted of five main sections: the pressure generation unit; the dyeing unit; recycling unit; control system and cosolvent adding system. There are a stainless steel dyeing autoclave (2.0 L) with a perforated stainless steel core, a dye autoclave (0.5 L) with two sintered metal plates, an injection pump, a circulation pump, and a separator (0.5 L). The schematic diagram of SCD dyeing pilot plant is shown in Fig. 2.

PET fabric was suspended on a stainless steel core, placed in the dyeing autoclave and the calculated quantity of dye was added to the dye autoclave. After the pilot plant was sealed, liquid carbon dioxide was introduced into the whole system by means of an injection pump (valves 11–14 being kept closed), and the system was heated at the same time. When the system reached the required temperature and pressure, valves 8 and 9 were opened, valve 10 was closed, and the circulation pump began to operate. The supercritical fluid with the dissolving dye was pumped through the fabric for the appropriate time. Then, the pathway to the dye autoclave (valves 8 and 9) was closed, valve 10 was opened, and the system was cooled as soon as possible. When the temperature dropped below the glass-transition temperature of PET fiber, valves 11–13 were opened to depressurize the system gradually. The unused dye was trapped in the separator and therefore ready for second use. After the dyeing procedure was completed, the dyed samples were removed.

In order to remove the excess dye from the dyed fabric with SCD, the liquid carbon dioxide was introduced into the dyeing autoclave with the dyed fabric by the injection pump (only valve 15 being