

CO₂ utilization for the waterless dyeing: Characterization and properties of Disperse Red 167 in supercritical fluid

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ABSTRACT

The physico-chemical behavior of disperse dye is the crucial point and basis for utilization CO₂ in supercritical state as an environmentally friendly waterless dyeing medium, which has triggered widespread concern. In the present work, the physico-chemical behavior of Disperse Red 167 was investigated in supercritical CO₂, for the first time. Scanning electron microscopy results show that the melting of Disperse Red 167 was observed with CO₂ temperature increasing from 120 °C to 160 °C. Fourier transform infrared spectrometry spectra reveal that the slight shifts and differences in the intensities of the characteristic bands of Disperse Red 167 was presented before and after supercritical CO₂ treatment. XRD and TG analysis indicate that crystal form of Disperse Red 167 transformed from β-type to α-type due to the dissolution and melting recrystallization, and the thermal decomposition temperatures were shifted to a lower temperature when the CO₂ temperature was higher than 120 °C. Moreover, dyeing experiments with the recycled Disperse Red 167 samples present similar colorimetric values and colorfastness properties to original dye. Therefore, all the investigations further offer the evidence that the melting recrystallization of disperse dyes occurred under higher temperature condition in supercritical CO₂, which confirms the recycling performance of disperse dye, and also provides the feasibility for thermosol dyeing in supercritical CO₂.

1. Introduction

In order to both decrease the duration of the dyeing period and the emission of dyestuff materials to water, the textile industry is continuously searching for the alternative ecological dyeing techniques [1]. One of the most remarkable techniques is waterless dyeing using supercritical fluids instead of water as the dyeing medium, which was firstly introduced into textile field by Pro. E. Schollmeyer in 1988 [2,3]. CO₂ collected from industrial combustion and fermentation processes, ammonia synthesis and mineral springs, is the most widely used solvent in supercritical fluids because of its low cost, wide availability, and environmentally friendly and chemically benign nature [4]. In supercritical state, the viscosity and diffusivity of CO₂ are like that of a gas, while its density is near that of a liquid, with a solvent strength that can be easily tuned by the variation of temperature and / or pressure [5]. Moreover, the non-polar characteristic of CO₂ makes this fluid fit for the coloration of synthetic fibers and natural fibers since supercritical CO₂ presents the dissolving power towards dyes. After the dyeing process, a simple depressurization and / or cooling step allows the supercritical CO₂ to be gasified into CO₂ gas and the excess dyes to be separated out

for reuse. The rinsing and dry steps are also eliminated, which further reduce the CO₂ emission and energy consumption [6]. Simultaneously, there is no irritating odor generated due to the sealed dyeing condition. Thus, supercritical CO₂ dyeing is becoming an interesting alternative to the traditional aqueous dyeing process due to its notable environmental benefits.

Theoretically, disperse dyes are the main applicable dyes in supercritical CO₂ dyeing process since their low polarity molecular structures can guarantee an adequate dissolution in supercritical CO₂. Subsequently, the dissolved disperse dye molecules are absorbed onto fiber surface and then diffused into the polymer matrix, where the color fixation is achieved by the physical and / or chemical bonds between the polymer macromolecule and the dye molecules [7]. On the contrary to the aqueous dyeing process, disperse dyes without any auxiliaries, namely crude disperse dyes, are used in supercritical CO₂ dyeing procedure to alleviate the crystal growth, transformation and suspension of dye molecules, thereby producing evenly distributed and bold colors [8].

At present, researches on dyeing of polyester fibers with trichromatic disperse dyes in supercritical CO₂ has been reported intensively

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from laboratory scale to industrial scale [9–11]. J. Long investigated the leveling properties of polyester fabrics in supercritical CO₂, and found that the leveling properties were improved with a temperature from 90 °C to 110 °C, a pressure from 10 MPa to 20 MPa, a dyeing time from 1 h to 2 h, as well as a time ratio of fluid circulation to static dyeing from 0.017 to 0.100 [12]. L. Zheng conducted polyester bobbins dyeing experiments with Disperse Blue 79, Disperse Red 60 and Disperse Yellow 119 by employing an industrial scale supercritical CO₂ apparatus, and demonstrated that satisfactory and commercialization dyeing with the uniform and bright color were obtained in supercritical CO₂ [13].

The effect of supercritical CO₂ on the chemical, supramolecular and morphologic structure of fibers has also been investigated [14], and the results revealed that CO₂ in supercritical state does penetrate into the fiber and promote the migration of oligomer from the polymer to the surface, resulting in the change of the crystal index [15–17]. In addition, the solubility data for numerous disperse dyes in supercritical CO₂ has been measured as it has an obvious influence on the compatibility, color matching and technological feasibility of different dyes during supercritical dyeing [18–20]. However, up to now no information has been available for the influence of supercritical CO₂ on physicochemical and application characteristics of disperse dyes, although they are crucial basis for fibers' supercritical CO₂ dyeing. Especially, as the growing dyeing demand for high performance fibers, such as aramid, ultra-high molecular weight polyethylene and polyphenylene sulfide [21], increasingly higher temperature is adopted in supercritical CO₂ process to swell polymers, which will lead to the change of crystal and other dyeing properties, and may eventually affect recycling of dyes after supercritical CO₂ dyeing.

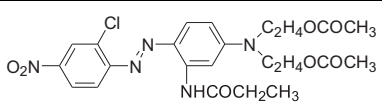
C.I. Disperse Red 167, one of the dark trichromatic disperse dyes for automotive and textile, is a commercially important disperse dye capable of dissolving in supercritical CO₂, and used in the dyeing of synthetic fibers [22]. In the present work, Disperse Red 167 was exposed to supercritical CO₂. The effect of CO₂ temperature on the surface morphology, chemical and crystal structures, thermal property as well as optical absorption property were assessed by Scanning electron microscopy (SEM), Fourier transform infrared spectrometry (FT-IR), X-ray diffraction (XRD), Thermogravimetric analysis (TG), and Ultraviolet-visible absorption spectroscopy (Uv-Vis). Furthermore, the treated Disperse Red 167 were also reused in the dyeing of polyester fabrics to confirm their dyeing properties in supercritical CO₂.

2. Experimental

2.1. Materials and chemicals

Disperse Red 167 without any addition of surfactants and salts was supplied by Zhejiang Longsheng Group Co., Ltd. (China) and used without further purification. Scoured polyester fabrics were supplied by Liaoning Chaoyi Industry & Trade Group (China). CO₂ gas (99.90 vol %) obtained from China Haohua (Dalian) Research & Design Institute of Chemical Industry Co., Ltd. was used for supercritical fluid dyeing. Disperse Red 167 used in supercritical CO₂ dyeing were listed in Table 1.

Table 1
Chemical structure of Disperse Red 167.

Dye	Color index	Formula	Molecular weight (g/mol)	Melting point (°C)	Purity	Chemical structure
Disperse Red 167	11338	C ₂₃ H ₂₆ ClN ₃ O ₇	519.93	126	> 98%	

2.2. CO₂ treatment

Before treatment, Disperse Red 167 was packed into a dye cylinder, and placed into a dye vessel. After the dye vessel was sealed, CO₂ stored in a gas cylinder was firstly filtered with a purifier, and then liquefied by employing a refrigerator. The CO₂ liquid was pressurized to above critical pressure (7.38 MPa) using a high-pressure pump and was heated to above critical temperature (31.10 °C) with a heat exchanger. In supercritical state, CO₂ fluid was injected to the dye vessel in which Disperse Red 167 would be treated. When the request treatment temperature and pressure were attained, the magnetic pump was started to form a typical supercritical CO₂ cycle. As described in our previous work [21], the treatment experiments were conducted for 70 min at a system pressure of 30 MPa with different temperatures (100 °C, 120 °C, 140 °C, and 160 °C). After the treatment, the CO₂ fluid was separated adequately in a separator under the low temperatures of 25 °C to 40 °C. The precipitated dyes were deposited at the bottom of the separator while the depressurized CO₂ gas was cooled and recycled into the gas cylinder. The treated Disperse Red 167 was removed and used for further analysis. The schematic diagram of the supercritical CO₂ apparatus was displayed in Fig. 1.

2.3. Characterization

2.3.1. Scanning electron microscopy (SEM) analysis

The surface morphological investigations of Disperse Red 167 before and after supercritical CO₂ treatment were carried out on a JSM-7800F Field Emission Scanning Electron Microscope (JEOL, Japan). The Disperse Red 167 samples were firstly fixed on the aluminum plate via a conducting adhesive and degassed sputter coated with the gold for 80 s to provide the conductivity for the impinging electrons. Then, the dye samples were investigated at 1000 and 5000 magnification to observe their surface morphological changes.

2.3.2. Melting behavior analysis

The melting of Disperse Red 167 before and after supercritical CO₂ treatment was recorded on a phase equilibria apparatus (Waters, USA) equipped with a monochromatic light source and a CCD video camera at a system pressure of 30 MPa with temperatures from 100 °C to 140 °C.

2.3.3. Fourier transform infrared spectrometry (FT-IR) analysis

Disperse Red 167 before and after supercritical CO₂ treatment were recorded by employing a Nicolet 8700 FT-IR spectrophotometer (Thermo Fisher Scientific, USA) with a traditional KBr pellet sampling method. During test, the transmittance of the infrared in individual powder samples of Disperse Red 167 was scanned 30 times at room temperature with a resolution of 4 cm⁻¹ in the range of 400–4000 cm⁻¹.

2.3.4. X-ray diffraction (XRD) analysis

The crystalline states of Disperse Red 167 samples before and after supercritical CO₂ treatment were conducted by employing an D/max-3B XRD instrument (Rigaku, Japan) with a CuKα radiation from 10° to 70° at room temperature. The test condition was that wavelength

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