

# Dyeing of Modal fiber in Supercritical Carbon Dioxide Using Disperse Dye C.I.(Color Index) Disperse Yellow 54

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**Abstract:** In this work, Modal fibers were dyed in supercritical carbon dioxide using C.I. Disperse Yellow 54, and dyeing process was investigated and optimized. The key parameters including dye dosage, temperature, pressure and time of dyeing process were discussed both theoretically and experimentally. The degree of crystallinity and strength of the fibers were affected by fiber modification and dyeing. The final results obtained from this experiment indicated that only 20min was required to reach the equilibrium condition in supercritical carbon dioxide, and the dye yield increased with the increase in temperature. The dye-uptake increased along with the increase of pressure, however when the pressure was extended up to 18Mpa, the dye-uptake decreased slowly. The optimized conditions include winding of fibers around dyeing axis with the combination of different CO<sub>2</sub> inlets, dyeing temperature at 100°C, pressure at 18MPa and time 20min.

**Keywords:** Modal fibers, supercritical CO<sub>2</sub>, disperse dye, dyeing technique.

## 1. Introduction

Wastewater generated from dyeing process is one of the main causes of pollution in textile industry. According to the published data and statistics analyzed so far, water and electric power consumption during preparation of cotton fabrics are 72-10<sup>3</sup>m<sup>3</sup>/t and 590-1100kWh/t [1], respectively. In recent years, more and more attention has been paid to the environmental pollution issues caused by dyeing wastewater. Dyeing with supercritical fluids has many advantages, such as avoiding chemical agents, lesser dyeing time, recycle dyes and solvents, and currently some developed countries have been in the process of experimenting and prototyping at pilot scale [2-4]. CO<sub>2</sub> was used in this experiment, because it has not only low critical temperature and pressure, but also it is available at low cost, in high purity, exhibiting low viscosity and favorable diffusivity. Moreover, CO<sub>2</sub> derived from fermentation, burning and ammonia synthesis processes can be used to decrease the greenhouse effect and change waste into valuable substances.

Modal fibers with partial characteristics of Tencel belong to regenerated cellulose fiber, which has better performance than cotton and viscose [5-9]. And it has lot of advantages such as high tensile strength especially in wet condition, easily dryable and good hygroscopic properties, smooth handle feel and good dimension stability, suitable to be blended with other materials and good dyeing properties, environmental

friendly at all stages of the production process and easily biodegradable in nature [10-12].

This paper reports the dyeing properties of modal fiber with C.I. Disperse Yellow 54 in supercritical CO<sub>2</sub>. The dyeing process and its key parameters were investigated. Further, the effects of fiber modification and dyeing on fiber strength were also studied.

## 2. Experimental

### 2.1 Apparatus

The Experimental and Laboratory setups of Low-temperature Plasma and Supercritical CO<sub>2</sub> (SC-CO<sub>2</sub>) dyeing were used to modify and dye the modal fiber. Infrared spectra were obtained using a Fourier Transform Infrared Spectrometer (FT-IR, PerkinElmer, spectrum100) to identify the functional groups and chemical bonds of the modified fibers. The structural analysis of the fiber was carried out using a X-ray Diffractometer (XRD, Bruker D8 Discover, Germany). The aberration and strength were detected by TC-P2 Color-difference Meter (Joint-stock Corporation) and TG605 Electronic Fiber Strength Tester (Laizhou Electronic Company).

### 2.2 Materials

Modal fiber of 1.67dtex×38mm was provided by Hailong Co. Ltd. Shangdong. C.I. Disperse Yellow 54

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was obtained from Dalian Chemical Co. Ltd., as shown in Figure 1. Sodium hydroxide and acetone (Tianjin Chemical Reagent Development Centre Co. Ltd.), benzoyl chloride (Shenyang Xinxi Reagent Company) and CO<sub>2</sub> (Purity, 98%) were used. All reagents above were used without further purification.

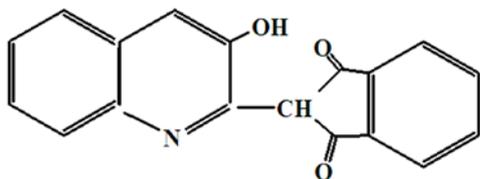


Figure 1 Molecular structure of Disperse Yellow 54.

### 2.3 Low-temperature plasma modification

A glow discharge generator was used for the treatment of the modal fiber. The discharge power, system pressure and anode-cathode distance were 60W, 0.07Pa and 6mm, respectively, and the duration of the treatment was 10 min. After modification by low-temperature plasma, fiber surface and fiber surface area became rougher and larger respectively, which would benefit further modification and dyeing in supercritical CO<sub>2</sub>.

### 2.4 Benzoyl chloride modification

The modal fiber modified by low-temperature plasma was further chemically modified in order to enhance its hydrophobicity after grafted benzoyl group for improving the interaction between dye and fiber.

For benzoyl chloride modification, the modal fiber was first immersed in the sodium hydroxide solution (20wt.%) for an hour, then it was washed several times with distilled water until pH was neutral and dried at 60°C for 5h. Then, the fiber was impregnated in benzoyl chloride solution for 10min at room temperature, further, it was washed with sodium hydroxide solution (5wt.%) several times and washed with distilled water until its pH was 7. Finally the fiber was dried at 60°C for 10h.

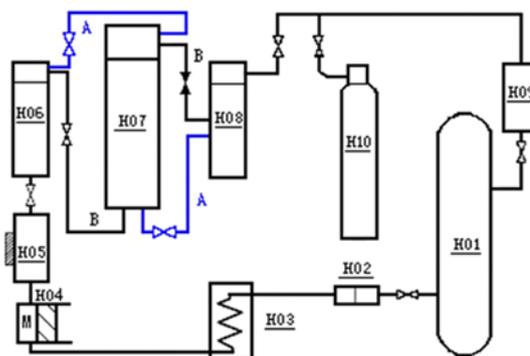
### 2.5 Dyeing in supercritical CO<sub>2</sub>

Modal fiber (5g) packaged by gauge was twisted into a beam dyeing, and C.I. Disperse Yellow 54 was loaded into dyeing vessel. Then CO<sub>2</sub> was injected into dyestuff and dyeing vessel, respectively, and the vessels were pressured and heated upto the needed pressure and

temperature. When the dyeing process was over, both CO<sub>2</sub> and the excess dyestuff were recycled.

This dynamic dyeing process is available by a combination of inside-outside dyeing process and outside-inside dyeing process. Inside-outside dyeing process refers to dye solution entering from the inlet port underneath the dyeing vessel to the outlet port of its upper part, while the outside-inside dyeing process can be defined as the dye solution entering from the upper inlet port to the underneath outlet port.

The combination of inside-outside dyeing and outside-inside dyeing processes can be realized by controlling the specific valves in the same dyeing process (Figure 2, A indicates inside-outside dyeing and B shows outside-inside dyeing process.). Temperature, pressure and time are controlled by a computer during the process, and experimental data was collected in real time.



H01-CO<sub>2</sub> Tank H02-Filter H03-Refrigerant container  
H04-High-pressure pump H05-Preheater  
H06-Dyeing vessel H07-Dyeing vessel  
H08-Extraction vessel H09-Flowmeter H10-CO<sub>2</sub> cylinder

Figure 2 Schematic diagram of SC-CO<sub>2</sub> dyeing.

### 2.6 Dyeing conditions

Dyeing in supercritical CO<sub>2</sub> was conducted by the three factor-levels orthogonal tests, as listed in Table 1.

Table 1 Factors and Levels--L<sub>9</sub> (3<sup>7</sup>)

	A Status	B Pressure	C Temperature	D Time	E Flow mode
1	Winding	16	90	10	A
2	Scattering	18	100	20	B
3		20	110	30	A+B
R		3.864	7.71	0.65	1.00
S		65.92	91.90	0.68	2.68