

# CO<sub>2</sub> Utilization for the dyeing of yak hair: Fracture behaviour in supercritical state



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## ABSTRACT

In this paper, using carbon dioxide as a dyeing medium, some basic color and mechanical behaviour data for yak hairs were firstly obtained with different parameters. The effects of system temperature, pressure, time and depressurization rate on yak hairs were investigated in terms of the color strength, the short fiber content, the breaking strength and the elongation at break. The results indicated that the short fiber content of yak hairs was increased with the raising of system temperature, pressure, time as well as depressurization rate, while the breaking strength and the elongation at break of yak hairs were decreased with the increase of system temperature, time as well as depressurization rate. Moreover, supercritical carbon dioxide for the fracture mechanism of yak hairs was proposed based on the Bernoulli's principle and Boyle's Law.

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## 1. Introduction

Carbon dioxide can be tuned into supercritical state when its temperature and pressure reach more than 31.10 °C and 7.38 MPa, respectively. In the supercritical state, the viscosity and diffusivity of carbon dioxide are near that of a gas, while its density is like that of a liquid, thus resulting in the adjustability for the solvent strength by changes of pressure and/or temperature [1]. Simultaneously, the swelling of supercritical carbon dioxide towards fibers, as well as the dissolving power to dyes, make carbon dioxide available for the textile dyeing. Since carbon dioxide was firstly introduced into textile dyeing field to replace water by Professor Schollmeyer at Deutsches Textilforschungszentrum Nord-West e. V. (DTNW) in Germany in the late 1980s, numerous research activities have been conducted around the world [2–4]. To date, the coloration with supercritical carbon dioxide for synthetic fibers such as polyethylene terephthalate [5,6], polypropylene [7,8], poly-*m*-phenyleneisophthalamide [9,10], and polyacrylonitrile [11] has obtained the favorable effect. Some of researches have even met the requirements of commercial dyeing production [12]. Furthermore, supercritical carbon dioxide dyeing exploration on wool and other natural fibers has also been documented [13,14].

When dyeing in supercritical carbon dioxide, disperse dyes are selected as the main dyes since carbon dioxide can only dissolve

non-polar and/or low-polar organic solid substances due to its low dielectric constant. The commercial dyeing production of polyester (polyethylene terephthalate or PET) has been firstly achieved in supercritical carbon dioxide because of the non-polarity of PET [12]. In the supercritical dyeing process, the disperse dye molecules diffuse into the fiber matrix where they are physically bonded [14]. For natural fibers, the affinity between fiber macromolecules and non-polar dye molecules cannot be formed due to the intrinsic characteristic of polar, which greatly increases the dyeing difficulty in supercritical carbon dioxide. Gebert et al. pretreated wool and cotton with 10% aqueous solution of Glezin CD, and confirmed the dyeing feasibility of natural fibers with disperse dyes in supercritical carbon dioxide [15]. Schmidt et al. described the dyeing of natural and man-made fibers with C.I. Disperse Yellow 23 modified with 2-bromoacrylic acid and 1,3,5-trichloro-2,4,6-triazine as reactive groups in supercritical carbon dioxide, and proved that it is possible to dye natural fibers in supercritical carbon dioxide without pre-treatment of the fiber [13]. The dyeing results showed that the wash, rub and light fastness of all dyes were rated at between 4 and 5.

Yak hair, a rare specialty animal fiber, is mainly produced in China with 400,000 t per year. The structure of yak hair is similar to wool, consisting of two main morphological components: the cuticle and the cortex. Simultaneously, yak hair has a small amount of medulla, thereby presenting better warmth retention property than wool and the hand feeling as soft as cashmere. Moreover, yak hair is completely odorless, non-allergenic and nonirritating as it contains no animal oils or residues [16]. Thus, textiles made from

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yak hairs have been one of the most luxurious products, and well received by the people's favorite. Currently, yak hair dyeing products are increasingly in demand, and their vivid colors are not only useful for a beautiful appearance but also alter to accommodate changes in fashion, style, and personal preference [17]. Nevertheless, the increasing environmental and health hazards in the aqueous dyeing process throughout the world have become a major concern.

To achieve the ecofriendly production, in our previous study, dyeing of yak hairs was investigated using supercritical carbon dioxide as a dyeing medium, and the dyeing feasibility of yak hairs was proved. However, most of raw yak hairs are brown or black, limiting their further application because of the natural colors. Hence, decolorizing process is required to obtain the white yak hairs which satisfy the dyeing production, but fiber damage occurs due to the existing weak structures after decolorizing [18]. In supercritical carbon dioxide, high temperature and pressure condition can aggravate the mechanical damage of wool [19]. According to the authors' best knowledge, there are few reports concerning the systematic study of the fracture behaviour of yak hairs in supercritical carbon dioxide, which is always an obstruction to the following spinning and weaving production. Therefore, as a part of the approach to tackle the problem of the strength loss, fundamental studies about the effect of carbon dioxide on the fracture behaviour of yak hairs in supercritical dyeing process are crucial important to improve the dyeing properties of yak hairs.

The objective of this work is to dye yak hairs in supercritical carbon dioxide, and the effects of system temperature, pressure, time and depressurization rate of carbon dioxide on yak hairs were investigated firstly in terms of the color strength (K/S value), the short fiber content, the breaking strength and the elongation at break. Furthermore, supercritical carbon dioxide for the fracture mechanism of yak hairs was also proposed. The data obtained can provide basic information for further research and practical application of carbon dioxide in the ecofriendly dyeing for yak hairs.

## 2. Experimental

### 2.1. Materials and chemicals

Decolorized yak hairs (fineness 18 μm, short fiber content 25%, breaking strength 4.87 cN, elongation at break 36.50%) were kindly supplied by Qinghai Xuezhoushanrong Co., Ltd. (China). Reactive Disperse Blue R without any addition of surfactants and salts was supplied by Shanghai Huiyi Industrial Development Co., Ltd. (China) and used without further purification, as shown in Fig. 1. Carbon dioxide gas (99.9 vol%) was obtained from China Haohua (Dalian) Research & Design Institute of Chemical Industry Co., Ltd.

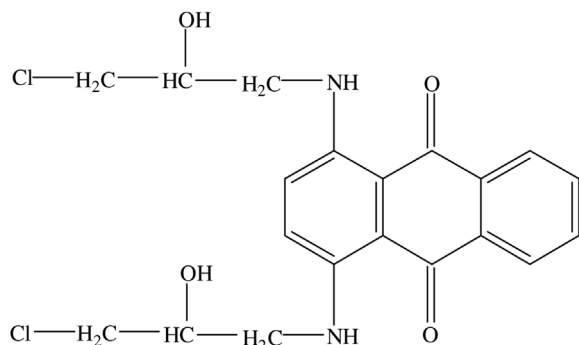


Fig. 1. Chemical structure of the dye used in supercritical carbon dioxide dyeing.

### 2.2. Apparatus and procedure

#### 2.2.1. Supercritical carbon dioxide dyeing

The supercritical carbon dioxide dyeing for yak hairs was carried out in a self-built batch apparatus, as depicted in Fig. 2. Before dyeing, carbon dioxide in a carbon dioxide cylinder (1) was filtrated with a filter (2), and cooled to liquid by using a refrigerator (3). The liquid carbon dioxide was then injected into the dyeing system, pressurized with a high-pressure pump (4) to above the critical pressure and heated with a heat exchanger (5) to above the critical temperature. In this process, carbon dioxide was tuned into supercritical state. During dyeing, the supercritical carbon dioxide fluid was firstly introduced into a dyeing vessel (6) where the solid dyes were dissolved, and then flowed into a dyeing vessel (7) to dye yak hairs. The dissolution of the dyes and the dyeing for yak hairs were conducted continuously with the action of a circulating pump (8). After the dyeing process was finished, the depressurization rates of the supercritical carbon dioxide could be regulated by using a micrometering valve mounted between dyeing vessel and cooler. The flow rate of supercritical fluid was recorded with a flowmeter. The supercritical carbon dioxide dissolving with dyes was cooled by employing a cooler (9), and the dye residues were precipitated out gradually in separators (10,11). The carbon dioxide was gasified at pressures and temperatures ranging from 3 MPa to 4 MPa and from 25 °C to 40 °C, filtrated with a filter (12), and reflowed into the carbon dioxide cylinder for next experimental use. The dyed yak hairs were removed and used for further measurement.

#### 2.2.2. Color measurement

The color strength (K/S value) of the dyed yak hairs was measured by the light reflectance technique using the Kubelka-Munk equation, as shown in Eq. (1) [20]. K/S values were measured with a Color-Eye 7000A spectrophotometer (X-rite, America) using D65 light source and the 10° observation angle.

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

where K is the absorbance coefficient of the fiber to be tested; S is the scattering coefficient of the fiber to be tested; R is the reflectance of the fiber at each wavelength.

#### 2.2.3. Short fiber content and mechanical properties measurement

Short fiber content refers to the ratio of fiber numbers whose length are shorter than a certain limit ( $x_s$ ) and the total number of fibers ( $x_n$ ). Short fiber content of the yak hairs, as an important

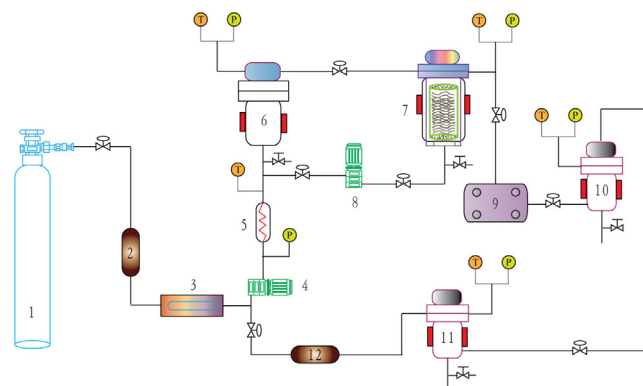


Fig. 2. Schematic diagram of the supercritical carbon dioxide dyeing apparatus equipped with (1) Carbon dioxide cylinder; (2) filter I; (3) Refrigerator; (4) High-pressure pump; (5) Heat exchanger; (6) Dye vessel; (7) Dyeing vessel; (8) Circulating pump; (9) Cooler; (10) Separator I; (11) Separator II; (12) Filter II.

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