

## WATER/OIL REPELLENT PROPERTY OF POLYESTER FABRICS AFTER SUPERCRITICAL CARBON DIOXIDE FINISHING

by

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*The strong permeability and driving force of supercritical carbon dioxide renders it an ideal medium for fabrics finishing. This paper is to use supercritical carbon dioxide medium with a solution of organic fluorine to fabricate water/oil repellent polyester fabrics. A series of characterization methods including Fourier transform infrared spectrometry, energy dispersive spectrometry, and scanning electron microscopy were carried out to evaluate the fabrics finishing. Fourier transform infrared spectrometry showed that the transmittance peak appeared at 1202.4 and 1147.4  $\text{cm}^{-1}$ , indicating the presence of  $-\text{CF}_2-$  group on the surface of polyester fabrics. The results of energy dispersive spectrometer and scanning electron microscopy showed that the fluorine was evenly distributed on the fibers surface. In addition, a series of physical properties were detected, including contact angel, air permeability, breaking strength, and wearing resistance. The average water and hexadecane contact angles were  $147.58^\circ$  and  $143.78^\circ$ , respectively. Compared with the initial fabrics, the treated one has little change in air permeability, while its strength increased greatly. The treated fabrics gained good water/oil repellent properties while keeping good air permeability and improving mechanical property.*

Key words: *supercritical carbon dioxide, water/oil repellent finishing, polyester fabrics*

### Introduction

With the rapid development of the modern technology, the demand for polyester materials with the water/oil repellent function is increasing greatly in industrial and household applications. It is necessary to reduce the surface energy significantly to obtain stable unwettability of polyester materials [1, 2]. Therefore, a new coating with low surface energy should be formed on the surface of materials by applying a hydrophobic substance in the traditional procedure of textile finishing. However, the finishing procedure produces a large amount of wastewater, which is extremely difficult to be treated.

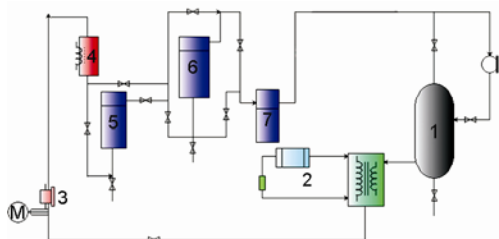
Supercritical fluid is a green solution for textile finishing, compared with the conventional methods. Carbon dioxide ( $\text{CO}_2$ ) is the most commonly used medium in supercritical fluid because of low critical pressure 7.38 MPa and temperature  $31.1^\circ\text{C}$ . Moreover,  $\text{CO}_2$  is inexpensive, non-toxic, non-flammable, environmentally friendly and chemically inert under most of conditions [3-5]. Meanwhile, supercritical  $\text{CO}_2$  ( $\text{SC-CO}_2$ ) also displays some special properties,

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for example, high diffusivity like gas, low viscosity and density like liquid, and low surface tension (close to zero). Therefore, all the properties give it a high solvency power tunable by adjusting pressure [6, 7]. At present, SC-CO<sub>2</sub> has been used as an environmentally friendly solvent instead of water and organic solvents for many industrial applications. However, it is seldom reported that SC-CO<sub>2</sub> has been applied in fabrics' finishing. Herein, the aim of this work is to study the effectiveness of finishing of polyester fabrics in SC-CO<sub>2</sub>.

## Experiments

All experiments were performed on a self-developed batch-type supercritical apparatus. The scheme of the apparatus is depicted in fig. 1. Solution of organic fluorine was packed into a cylinder and placed into an ingredient kettle (5). Carbon dioxide in a tank (1) was cooled



**Figure 1. Schematic drawing of the supercritical apparatus**

and changed into liquid by a refrigerator (2). It was pressurized to above the critical pressure 7.38 MPa, using a high-pressure pump (3), and was heated to above the critical temperature 31.1 °C, with a heat exchanger (4). The solution of organic fluorine were then dissolved in the SC-CO<sub>2</sub> fluid and flowed through a reaction kettle (6). The finishing experiments were conducted at temperatures and pressures ranging from 50 °C to 110 °C and 10 MPa to 26 MPa, respectively. After a requested finishing period, the CO<sub>2</sub> fluid and the dissolved solution in the system were separated with a separator (7). The gas was then recycled at pressures and temperatures ranging from 3 MPa to 4 MPa and from 25 °C to 40 °C, respectively.

## Results and discussion

### *Water/oil repellent properties of polyester fabrics*

Measurement of the wetting contact angles was usually used as a fast method to determine the water/oil repellent properties of polyester fabrics [8, 9]. Under the different conditions, water and hexadecane contact angles of polyester fabrics were determined with the treatment of organic fluorine solution in SC-CO<sub>2</sub>. The average contact angles of water and hexadecane for the treated fabrics were estimated to be 147.58° and 143.78°, respectively. The contact angles of initial polyester fabrics were under 50°. Therefore, the increasing data may demonstrate that the fluoride coating was probably formed on the polyester fabrics surface. It can be assumed that the formed fluoropolymer layer on the fibers may significantly reduce the surface energy of the polyester fabrics.

### *Characterization of finished polyester fabrics*

The special peaks of fluoride are at the range of 750~1400 cm<sup>-1</sup> in Fourier transform infrared (FT-IR) spectra, in order to prove the formation of fluoropolymer layer on the surface of polyester fabrics, FT-IR spectra of initial and treated polyester fabrics were observed. As shown in fig. 2, the transmittance peak in curve b at 3336.7 cm<sup>-1</sup> indicated the presence of -OH, which was attributed to the agent solution. Meanwhile, the appearance of transmittance peaks at 1147.4 cm<sup>-1</sup> and 1202.4 cm<sup>-1</sup> confirmed the presence of the -CF<sub>2</sub>- group on the surface of the treated fabrics.

In order to investigate the advantages of SC-CO<sub>2</sub> to the agent distribution, the fabrics were also treated in water medium. Qualitative information for the distribution of the fluorine molecules on the polyester fabrics were provided by energy dispersive spectrometry (EDS) and scanning electron microscopy (SEM) analysis. The EDS micrograph of polyester fibers treated in SC-CO<sub>2</sub> medium is shown in fig. 3. It was shown that fluorine distributed uniformly on the surface of fibers, fig. 3(a) and the content of it could reach 4%, fig. 3(b). The results manifested that an ultrathin and continuous fluoropolymer layer was formed, which was consistent with the results of FT-IR.

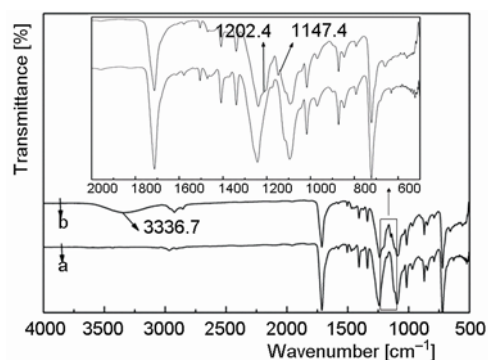
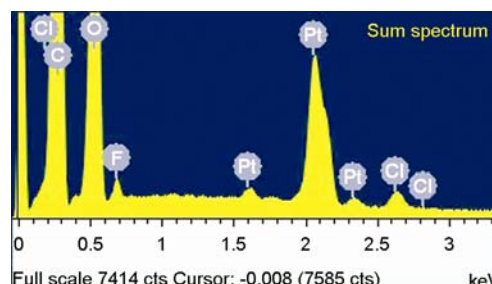
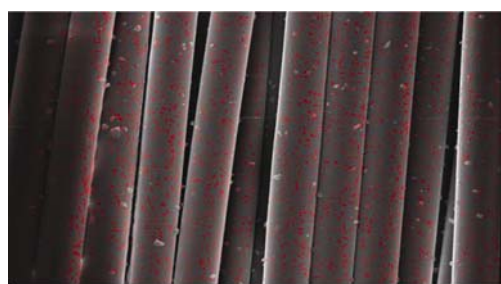


Figure 2. FT-IR spectra of initial (a), and treated (b) polyester fabrics



(a)

(b)

Figure 3. EDS mapping of fluorine (the site of red dot was fluorine in a)

The SEM micrographs are shown in fig. 4, the gaps among the polyester fibers were

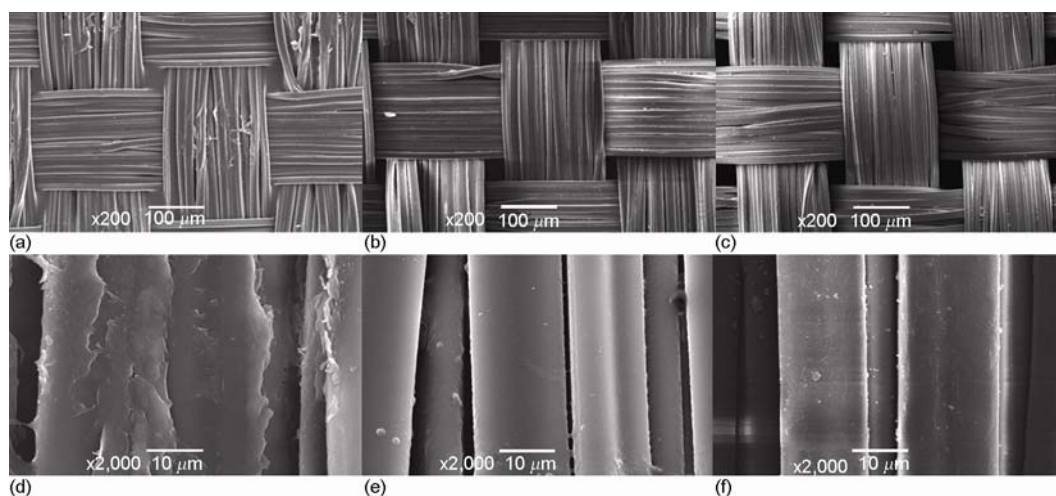


Figure 4. SEM micrographs of polyester fabrics under magnification ( $\times 200$  and  $\times 2000$ ) (a) and (d) treated in water medium, (b) and (e) treated in SC-CO<sub>2</sub> medium, and (c) and (f) initial

covered by the coating after the treatment in water, fig. 4(a), whereas the sample treated in SC-CO<sub>2</sub> medium, fig. 4(b) had almost no changes in comparison with the initial one, fig. 4(c). It can be seen from fig. 4(d) that coarse fluoropolymer coatings accumulated on the fiber and covered the holes between the fibers when the polyester fabrics were finished in water. However, a smooth layer was coated on the polyester fibers while the pores between the fibers were not covered by the layer in SC-CO<sub>2</sub> finishing process, fig. 4(e). Therefore, it exhibited that the finishing effect of polyester fabrics in SC-CO<sub>2</sub> was better than it in water. It could further prove that fluorine distributed uniformly on the surface of fibers when compared figs. 3(a) and 4(f). The reason could be the penetration and swelling effect of SC-CO<sub>2</sub> can promote the impregnation of fluorine-contained finishing agent without auxiliaries.

Meanwhile, it also can be obtained that the air permeability of treated fabrics has no changes in comparison with the initial ones from the SEM micrographs. The average air permeability of fabrics treated in SC-CO<sub>2</sub> and the initial ones was calculated to be 298.42 mm/s and 302.91 mm/s, respectively. The data declared that the air permeability of fabrics after water/oil repellent finishing had a slight change, so fabrics still remained good air permeability in SC-CO<sub>2</sub> finishing process.

### Physical properties of polyester fabrics

The results of the water and hexadecane contact angles of polyester fabrics indicated that the fluoropolymer layer formed on the fiber may significantly reduce the surface energy of polyester fabrics. Correspondingly, the mechanical properties of fabrics may be affected by the layer. Firstly, the wear resistance of the finished fabrics was also estimated by the water contact angles. It was found that the water contact angles were averagely decreased about 15° for all treatments after rubbing the fabrics for ten times. Therefore, the results indicated that the wear resistance of the treated fabrics in SC-CO<sub>2</sub> was better than it treated in traditional procedure.

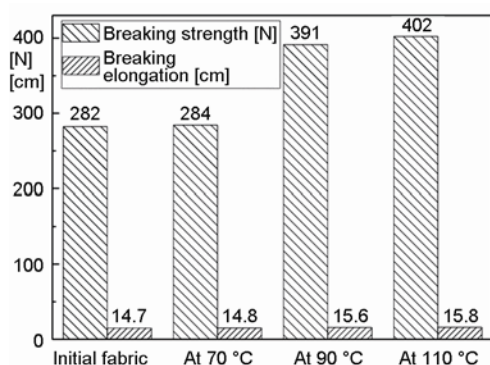


Figure 5. Tensile properties of polyester fabrics

increased to 110 °C. The possible reason may be that the strong permeability and driving force of SC-CO<sub>2</sub> improved the crystallinity of polyester fabrics at higher temperature.

### Conclusions

In this paper, a feasible way to produce water/oil repellent polyester fabrics was provided with organic fluorine in SC-CO<sub>2</sub>. The water and hexadecane contact angles of polyester fabrics demonstrated that the fluoride coating significantly increased the wetting contact

The tensile properties (breaking strength and breaking elongation) of the finished polyester fabrics under the condition of different temperatures were tested and shown in fig. 5. When the test condition was conducted at the pressure of 20 MPa, treated temperature of 70 °C, and the treated time of 20 min, there was no obvious difference between the initial fabrics and the finished ones in SC-CO<sub>2</sub>. If the temperature was increased from 70 °C to 90 °C, the breaking strength and the breaking elongation of the fabrics were improved from 282 to 391 N, and 14.8 to 15.6 cm, respectively. The tensile properties of the fabrics changed slightly when the temperature was

angles. FT-IR and EDS micrographs of the polyester fabrics clearly proved that a uniform fluoropolymer film was coated on the surface of polyester fiber. Meanwhile, the SEM results showed that the improvement of water/oil repellent effect on polyester fabrics was obtained in SC-CO<sub>2</sub> in comparison with the fabrics treated in water. The data of the fabrics' air permeability manifested that a slight change occurred while the good air permeability still remained after water/oil repellent finishing in SC-CO<sub>2</sub>. The breaking strength and elongation of the polyester fabrics increased greatly and exhibited best at 110 °C.

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