

EFFECT OF SUPERCRITICAL CARBON DIOXIDE ON DYEABILITY AND PHYSICAL PROPERTIES OF ULTRA-HIGH-MOLECULAR-WEIGHT POLYETHYLENE FIBER

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Abstract:

Supercritical carbon dioxide dyeing, a new type of anhydrous dyeing method, has a lot of advantages, mainly conservation of energy, prevention of pollution, reusability of dye, and many more. This study presents a viable method for the dyeing of an ultra-high-molecular-weight polyethylene (UHMWPE) fabric by using supercritical carbon dioxide (scCO₂) as a medium. Five hydrozono propanenitrile dyes that are functional colorants having antibacterial activity were applied for the dyeing of the UHMWPE fabric in scCO₂ at a pressure of 20 MPa and at temperature of 120°C. The dyeability of UHMWPE fabric under scCO₂ was evaluated by color measurement, whereby the color strength K/S was calculated. As the treating time and concentration of dye increased, the dyeability of the UHMWPE fabric displayed the tendency to continually improve. As decaline was added into scCO₂ as the cosolvent, we obtained higher K/S. Furthermore, color fastness to rubbing and sublimation of the dyed UHMWPE fabric were determined according to Japanese Industrial Standards (JIS) L 0849 2 and JIS L 0854, and the trend showed that the increase in fastness corresponded to the increase in duration of the treatment. The influence of scCO₂ dyeing on the mechanical properties of UHMWPE was also examined. Consequently, it was found that dyeing in scCO₂ containing decaline reduced the crystallinity of the UHMWPE fabric and the breaking strength decreased. The antimicrobial property of UHMWPE dyed with N'-(2-chloro-4-methylphenyl)-2-oxo-2-(p-tolyl)acetohydrazonoyl cyanide was tested against three different microorganisms, and the results have been reported.

Keywords:

supercritical carbon dioxide dyeing, hydrozono propanenitrile dye, decaline, crystallinity, tensile strength, antibacterial activity

1. Introduction

Supercritical fluid dyeing (SFD) is an alternative process to dye hydrophobic synthetic fibers [1-4]. For SFD processes, carbon dioxide is commonly applied as a medium due to its beneficial characteristics, such as nontoxicity, low price, recyclable nature, and easy access to the critical conditions at $T_c = 304.34$ K and $P_c = 7.38$ MPa [5]. CO₂ – beyond its critical point – has low viscosity, high diffusivity [6], high density, high solubility, and so on. Recently, modern tools such as ultraviolet (UV) radiation, microwave radiation, and plasma treatment are being applied to improve the dyeability of fiber by etching the fiber surface before the dyeing process [7-11]. However, supercritical carbon dioxide (scCO₂) has inert behavior toward organic compounds, so it does not damage the materials. One of the most important reasons for using scCO₂ is that the dyeing process does not require the use of water, unlike the other conventional dyeing processes. Carbon dioxide in its supercritical state takes on a role similar to that of water (being a solvent) in an aqueous dye. This dyeing method has many benefits such as conservation of energy, prevention of pollution, reusability of dye, and so on [12-14]. Polyester (polyethylene terephthalate [PET]) [15-19] and nylon fibers [20, 21] were successfully dyed using the scCO₂ process. In previous works, we also reported on the SFD

of polyester and Nylon-6 fabrics with newly synthesized dyes with antibacterial activities [22, 23]. Polypropylene (PP) fiber, which is generally colored by adding pigment in a spinning dope, was also dyed under scCO₂ medium and is expected to be commercialized for sportswear [24].

Ultra-high-molecular-weight polyethylene (UHMWPE) fiber is also one of the materials that are hard to be dyed in aqueous media such as PP. Only carbon and hydrogen compose the polymer by simply repeating, so its chemical structure is quite elemental. The fiber has low density, high degree of crystallinity (DC), and has high draw ratio when produced by gel spinning. Therefore, it displays extreme physical properties such as high strength and high modulus [25, 26], which make UHMWPE fiber important for applications in the industrial field such as manufacture of protective equipment, rope, fishing nets, and so on. Despite having such beneficial properties, due to its high crystallinity, a dense molecular structure and lack of functional groups, it is difficult for the fiber to interact with many substances, including conventional dyestuff. Therefore, many researchers have been studying various methods to dye the UHMWPE fiber [27-29].

In this paper, the SFD technique has been employed to color UHMWPE fabric. The hydrozono propanenitrile dyes with

antimicrobial property were prepared and used for the SFD of UHMWPE. We investigated the influence of addition of decaline in scCO_2 as a cosolvent on the dyeability of UHMWPE by measuring the color uptake, expressed as the color strength K/S . Decaline and paraffin oil have been successfully used for gel spinning of UHMWPE [30-33]; so decaline is expected to adsorb onto UHMWPE, inducing fiber swelling and decreasing the crystallinity, which could promote dye diffusion in UHMWPE fiber during scCO_2 dyeing. The effects of treatment in scCO_2 containing decaline on the mechanical properties of UHMWPE were also examined. Furthermore, color fastness to rubbing and sublimation, as well as the antibacterial activities against three microbial cultures, was also tested and the results are discussed.

2. Experimental

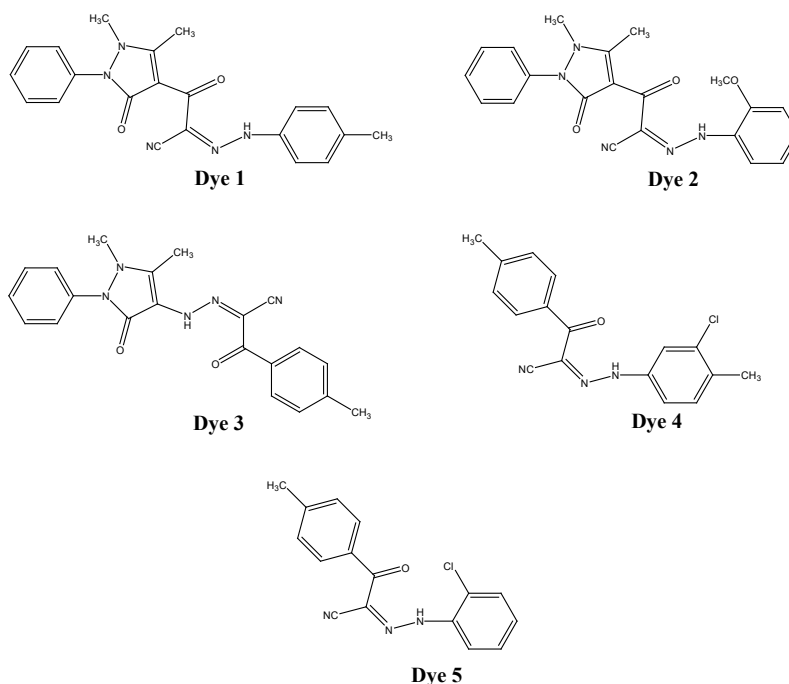
2.1. Materials

The UHMWPE plane woven fabric (160 g/m²) used in this study was IZANAS® (SK60 1320dtex), kindly supplied by Toyobo Co., Ltd., Otsu, Japan, and it was used for dyeing without any

pretreatment. All dyes used in this study were synthesized in our laboratory according to the method described in literature [34] and used for dyeing experiments. The dyes were prepared by mixing a solution of β -oxoalkanenitriles in ethanol; then, the mixture was solidified with sodium acetate solution to form the aryl diazonium salts. The solid products formed were collected by filtration and recrystallization. The International Union of Pure and Applied Chemistry (IUPAC) names and chemical structures are given in Figure 1. Decaline, with a purity of 95%, was purchased from Nacalai Tesque, Inc., Kyoto, Japan, and used without further purification.

2.2. Apparatus

Figure 2 shows a diagram of an apparatus for scCO_2 dyeing. The liquefied CO_2 departing from a cylinder moved inward to the cooling unit, an externally closed cooling water circulator (CF300, Yamato Scientific Co., Ltd.) and was infused into an intelligent pump (PU-2086, Jasco Corporation). High-pressure CO_2 ultimately flowed into a dyeing vessel (EV-2; Jasco Corporation). A dyeing vessel with volume of 50 mL, an oven with magnetic stirrer (SCF-Sro; Jasco Corporation) was used in the scCO_2 dyeing in batch method.



Code	Name (IUPAC)
Dye 1:	2-(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1H-pyrazol-4-yl)-2-oxo-N'-(p-tolyl) acetohydrazonylcyanide
Dye 2:	2-(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1H-pyrazol-4-yl)-N'-(2-methoxyphenyl)-2-oxoacetohydrazonoyl cyanide
Dye 3:	2-([1,1'-biphenyl]-4-yl)-N'-(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1H-pyrazol-4-yl)-2-oxoacetohydrazonoyl cyanide
Dye 4:	N'-(2-chloro-4-methylphenyl)-2-oxo-2-(p-tolyl) acetohydrazonoyl cyanide
Dye 5:	N'-(2-chlorophenyl)-2-oxo-2-(p-tolyl) acetohydrazonoyl cyanide

Figure 1. Chemical structure and IUPAC names of the dyes

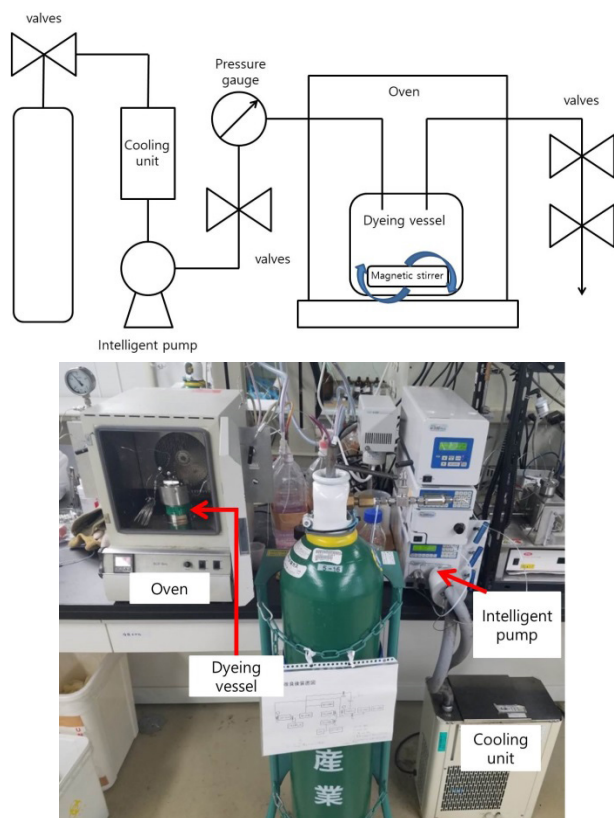


Figure 2. An apparatus for supercritical CO₂ dyeing

2.3. Procedures

The UHMWPE fabric (5 cm × 5 cm) was folded and put in a coil of stainless wire (SUS316) to prevent direct contact with dyestuff, and the coil was seated inside the vessel. The dye varying from 2% over mass of fabric (omf) to 6% omf was loaded in the bottom of the vessel, and 7 mol% of the cosolvent decaline was added in the bottom of the vessel. The amount of decaline was decided according to a previous study [34] and recalculated in mole percent (mol%), which is the ratio of moles of decaline to the total moles of CO₂ and decaline at 120°C and 20 MPa.

The vessel was then sealed and heated to 120°C. Then, CO₂ was pumped into the vessel and kept at 20 MPa with stirring. After a definite reaction time, the CO₂ was released by opening the valve slowly until the pressure of the dyeing vessel reached atmospheric pressure. The vessel was detached from the oven and cooled down at room temperature for 20 minutes. The dyed sample was taken out from the vessel and rinsed with 50 mL water for 1 minute or cold acetone for 10 seconds to remove unadsorbed dye on the fiber surface.

2.4. Measurements

Color uptake expressed as the color strength, *K/S*, of the dyed UHMWPE fabrics and was calculated using the Kubelka-Munk equation [Eq. (1)], with reflectance *R* measured using a spectrophotometer (CM-3600d; Konica Minolta, Inc.).

$$K/S = (1 - R)^2 / (2R) \quad (1)$$

where *K* is the absorption coefficient and *S* is the scattering coefficient.

Color fastness to rubbing and sublimation of the dyed UHMWPE fabric was determined according to Japanese Industrial Standards (JIS) L 0849 2 and JIS L 0854, corresponding to International Organization for Standardization (ISO) 105-X12 and ISO 105-A01, respectively. The rubbing test was carried out with dry samples only. Under the rubbing test, the fabric was placed on the finger of the crock meter and moved back and forth across the dyed UHMWPE fabrics at a constant speed 100 times. The specimen for the sublimation test was inserted between polyester fabrics, and it was subjected to 12.5 kPa and 120°C condition for 80 minutes. The results of these evaluations were given by five grades, from 1.0 for the lowest to 5.0 for the highest, and the results were determined by examination with the naked eye.

Differential scanning calorimetry (DSC) was performed on a TA Instruments DSC 2920 under N₂ flow at a heating rate of 10°C/min. The fibers were cut into 4–5 mm pieces, placed in an aluminum pan, and closed with an aluminum lid. The DSC measurement was carried out with an empty aluminum pan. The sample weight was approximately 4 mg. The DC was calculated from Eq. (2) using the heat of fusion obtained by the following relation:

$$\text{Degree of crystallinity, } \Delta C (\%) = \Delta H / \Delta H_m \quad (2)$$

where ΔH is the heat of fusion of the sample and ΔH_m is heat of fusion of a perfect crystal of PE, viz., 286.7 mJ/mg [35].

The UHMWPE woven fabric slipped and slid during tensile measurement even when adhesive agents and tapes were used. To decrease sliding of the specimen, the test was carried out for a multifilament specimen consisting of 10 monofilaments visually counted and extracted from each UHMWPE fabric at 25°C and at a cross-head speed of 10 mm/min using the testing machine AGS-J (Shimadzu Co.). For each fabric's sample, 20 multifilament specimens were tested, and the maximal breaking stress was recorded. The cross-sectional area of the multifilament sample was estimated from the diameter of the monofilament, measured using a scanning electron microscope. Then, the average of the maximal breaking strength and the modulus were calculated.

Assessment of antibacterial activity against Gram+ve bacteria (*Staphylococcus aureus* and *Bacillus subtilis*) and Gram-ve bacteria (*Escherichia coli*) was conducted according to quantitative assessment by percentage reduction test (American Association of Textile Chemists and Colorists [AATCC] 100-2004).

Specimens of UHMWPE dyed in supercritical medium were shaken in a known concentration of bacterial suspension and the reduction in bacterial activity in the standard time was measured. The efficiency of the antimicrobial treatment was determined by comparing the reduction in bacterial concentration of the treated sample with that of control sample, expressed as a percentage reduction in the standard time.

$$\text{Reduction } (\%) = [(A - B) / B] \times 100 \quad (3)$$

where *A* and *B* are the surviving cells (colony forming unit [cfu]/mL) in the flasks containing the control and test samples, respectively, after 24 hours of contact time.

3. Results and discussion

3.1. Dyeing conditions and color strength

Figure 3 shows the relations between the color strength *K/S* of the UHMWPE fabric dyed with 2% omf of dye in *scCO*₂ and the dyeing time at a temperature of 120°C and a pressure of 20 MPa [36].

Except for Dye 1, the dye uptake – expressed as color strength (*K/S*) – was enhanced with increase in the dyeing time up to 3 hours, though the *K/S* values for Dye 1, Dye 2, and Dye 3 were small. The results may be attributed to the low affinity of the abovementioned three dyes containing a polar pyrazole moiety and/or their larger molecules to the UHMWPE fiber, as compared with Dye 4 and Dye 5. Since Dye 4 provided the highest *K/S*, additional experiments were carried out with only Dye 4.

The effect of decaline on the dyeing of the UHMWPE fabric in *scCO*₂ was examined. Decaline is generally used for the spinning of UHMWPE [30, 31]. Therefore, we expected improvement of its dyeability by swelling the fibers. The results are given in Figure 4 [37, 38]. The aqueous dyeing was carried out with 6% omf Dye 4 at 120°C and at a liquor ratio of 1:20. The *K/S* of the samples dyed in *scCO*₂ was much higher than that for samples dyed in water, and it increased on adding decaline, especially for long dyeing durations. As expected, the fiber could gradually swell in the presence of decaline, which induces the rapid and easy diffusion of dye molecules and further dye adsorption on the fiber surface.

Figure 5 shows the influence of concentration of Dye 4 on the dye uptake, expressed as the color strength *K/S* of the UHMWPE fiber dyed in decaline/*scCO*₂ [39, 40]. For short dyeing, mainly, 1 hour, and at lower dye concentrations, mainly, 2% omf and 4% omf, there was no significant effect of dye concentration on *K/S*. On the other hand, the *K/S* increased with dyeing time up to 3 hours, and no saturation point was found in the range of experimental conditions. The results suggested that specific time periods and concentrations were required for the dye to be adsorbed onto the fiber and to start diffusing into it. Unusually, the difference in *K/S* between 4% omf and 6% omf is much larger than the difference between 4% omf and 2% omf at any dyeing time. This may be due to plasticization lowering the crystallinity caused by adsorption of larger amount of dyestuff. The details are discussed in Section 3.2, “Dyeing conditions and physical properties”. The dyeing results were quite different from those for the widely used synthetic fibers, e.g., PET and polyamide (Nylon) with conventional disperse dyes, where *K/S* is in the range of 12–21 for PET fibers and dyeing starts saturating in 1 or 2 hours [41–44]. This fact maybe due to the high crystallinity and low affinity of UHMWPE to the dye. Further experiments are needed to clarify the dyeing mechanism.

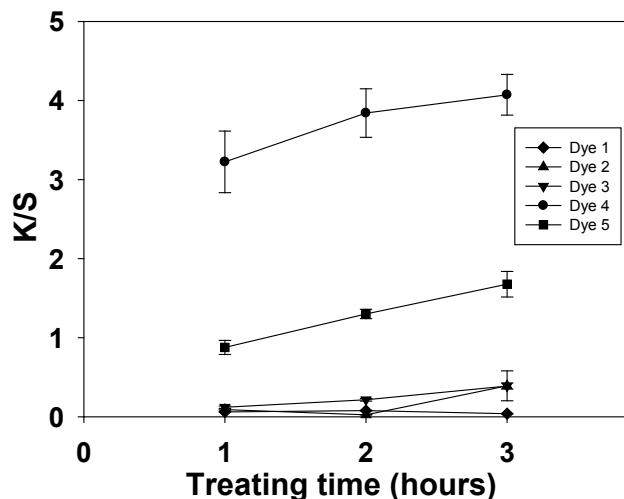


Figure 3. Effects of dye type on *K/S* of UHMWPE dyed with 2% omf in *scCO*₂

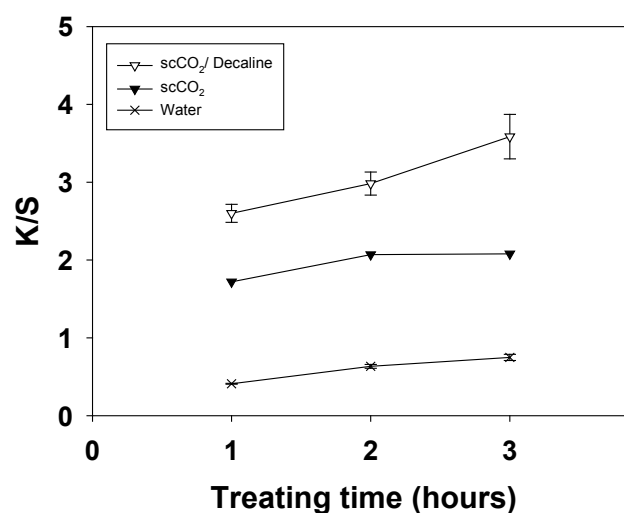


Figure 4. Effects of dyeing medium on *K/S* of UHMWPE dyed with 6% omf of Dye 4

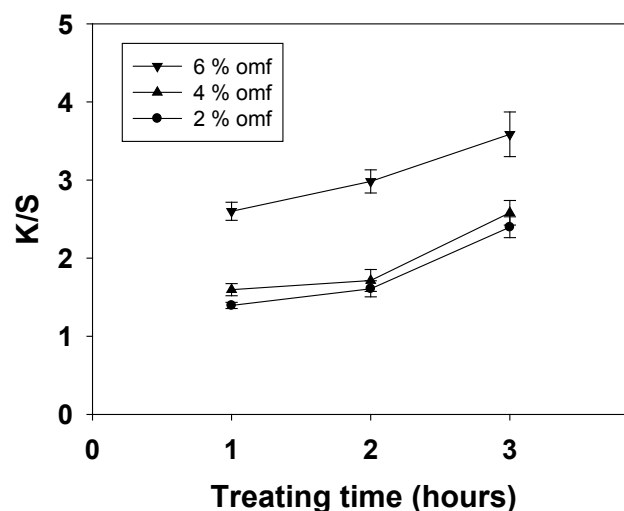


Figure 5. Effects of concentration of Dye 4 on *K/S* of UHMWPE sample dyed in *scCO*₂ containing decaline

3.2. Dyeing conditions and physical properties

The DC of UHMWPE was calculated from the heat of fusion estimated by DSC. The results are shown in Figure 6. The DC of the virgin sample was approximately 90% [39, 40]. The water treatment and scCO₂ treatment had no substantive effect on the DC, although the DC remarkably decreased by dyeing in scCO₂, especially on including decaline.

According to literature [45, 46], treatment of a polymer in scCO₂ after including cosolvent promotes the crystallization through adsorption, swelling, plasticizing, and reorientation. However, adding dye and decaline in scCO₂ reduced the crystallinity down to around 60%–70% in this study. It was assumed that the dye and decaline penetrate into the fiber in scCO₂, which destroys the crystalline regions of UHMWPE; then, the dye and decaline remain in the UHMWPE due to either high affinity with UHMWPE or low affinity with CO₂ after depressurization. This may disturb its reorientation, resulting in the decrease of the crystallinity in the scCO₂ process. Taking into account the result shown in Figure 5, the increase of *K/S* is strongly related to the reduction of crystallinity, which creates more amorphous adsorption sites for the dye.

Tensile test was performed to investigate the influence of treatment medium on the physical properties of UHMWPE fiber. The results are exhibited in Figure 7.

The maximal breaking strength of virgin UHMWPE fiber was 1.74 GPa, and it gradually decreased with the duration of the treatment, regardless of medium. Considering the decrease of DC shown in Figure 6, the reduction of breaking strength is verified by the lowering of crystallinity. On the other hand, the simple treatment with scCO₂ also scaled down the strength, although it did not affect the crystallinity (Figure 6). This result may be attributed to the phenomenon of a crystal-to-crystal transformation in the UHMWPE, from an orthorhombic unit cell to a more mobile hexagonal phase in scCO₂ [47]. It has been reported that the larger mobility of the hexagonal phases results in higher degree of freedom for rotations around the fiber axis. As a result, the scCO₂ treatment decreases the breaking strength of UHMWPE fiber without decreasing the fibrous crystallinity.

The modulus of UHMWPE fiber was calculated and plotted against the DC in Figure 8.

The modulus of the virgin UHMWPE fiber was 53 GPa and it was lowered by scCO₂ treatment, which is directly related to the decrement in breaking strength. In addition, the modulus was enhanced with increase in the crystallinity, as compared with the samples treated in the same medium, as shown by the data points within the circles in Figure 8. These results suggest that not only crystallinity but also other factors, e.g., crystal size, crystal structure, disorder of crystal, and orientation of molecules in the amorphous region, which are influenced by temperature, pressure, and solvent (i.e., medium of crystallization), determine the physical properties of UHMWPE. Further studies are required to explain the relations among those elements.

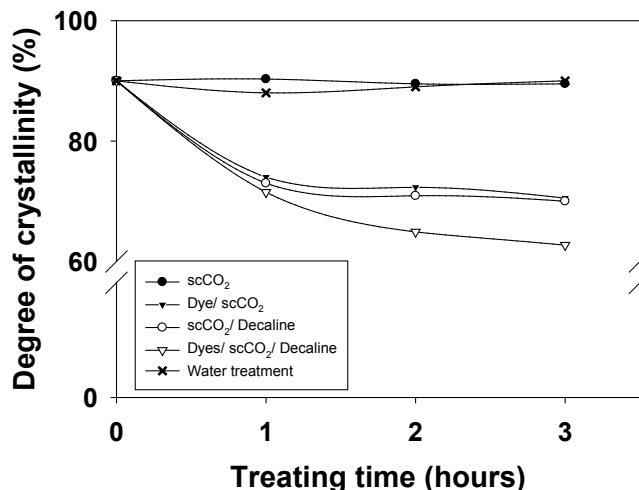


Figure 6. Effects of decaline on crystallinity of UHMWPE in scCO₂ dyeing with 6% omf of Dye 4

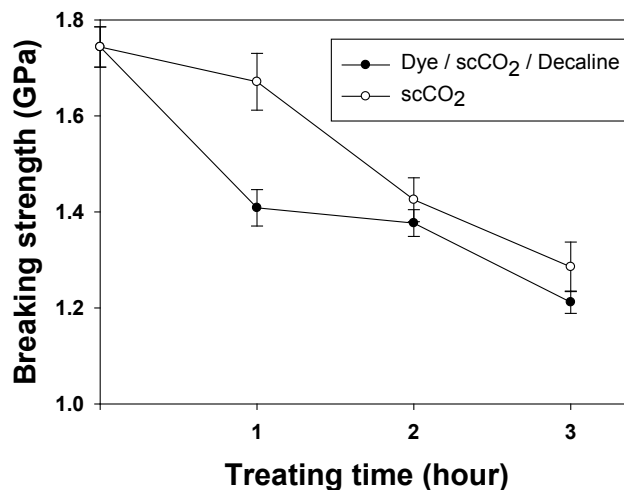


Figure 7. Effects of treatment medium on breaking strength of UHMWPE (the fabric was dyed with 6% omf of Dye 4)

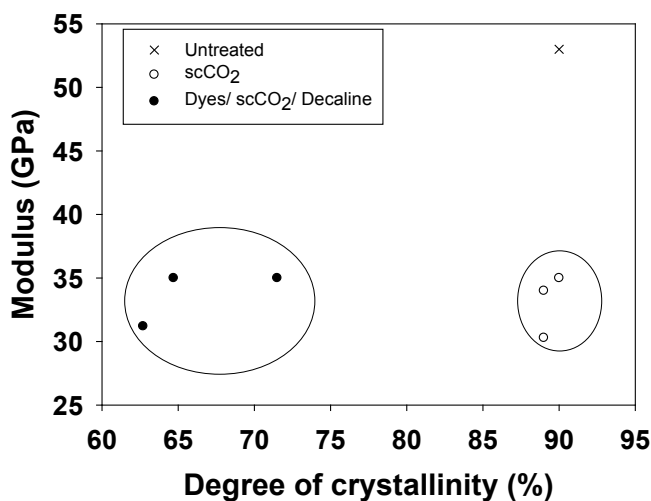


Figure 8. Effects of crystallinity on the modulus of UHMWPE (the fabric was dyed with 6% omf of Dye 4)

3.3. Color fastness

The color fastness of UHMWPE fabric dyed in scCO₂ to rubbing and sublimation is summarized in Table 1. Fastness of the fabric dyed for both 1 and 2 hours was excellent; however, the grade decreased to 3.0 for the 3-hour-dyed sample. This result is probably due to over dyeing, in which the dye molecules accumulate on the fiber surface. The accumulated dye was easily eliminated by rubbing and sublimation, resulting in the lower grade [48].

Table 1. Color fastness of UHMWPE fabric dyed with 6% of Dye 4 and decaline in scCO₂

Dyeing time (hours)	Fastness (grade)	
	Rubbing	Sublimation
1	4–5	4–5
2	4–5	4–5
3	3	3

3.4. Antibacterial property

In the test against *S. aureus*, *B. cereus*, and *E. coli* in AATCC Bacteriostasis Broth, the inoculated control and inoculated test fabrics were evaluated for percentage bacterial reduction by cell counting. The results of the percentage reduction test are shown in Table 2. The reduction percentages for *S. aureus* and *B. cereus* correspond to the bacterial numbers on the respective control tests, i.e., (1.18, 1.03, 1.09) × 10⁶/mL. The reduction percentage was found to be 73% for *S. aureus* and 47.2% for *B. cereus*, while no activity was found against *E. coli*.

The UHMWPE fabric dyed with Dye 4 in scCO₂ containing decaline showed certain good antibacterial activity due to the presence of the antipyrine moiety in the dye [17, 18], and the activity increased with increase in the dye concentration. However, the reduction of bacteria did not come close to 100%.

Table 2. Antibacterial activity of UHMWPE dyed with Dye 4 and decaline in scCO₂

Microorganism	Concentration (% omf)	Control after incubation period (24 hours)	Treated (cfu/mL)	Bacterial reduction (%)
<i>Staphylococcus aureus</i>	2	1.18 × 10 ⁶ 3320 × (37 ^T)	(0.13 × 10 ⁶)	37.4
	4		(0.57 × 10 ⁶)	12.2
	6		(0.02 × 10 ⁶)	73.0
<i>Bacillus cereus</i>	2	1.03 × 10 ⁶ 3320 × (90 ^T)	(0.23 × 10 ⁶)	26.4
	4		(0.07 × 10 ⁶)	47.2
	6		(0.07 × 10 ⁶)	46.5
<i>Escherichia coli</i>	2	1.09 × 10 ⁶ 3320 × (60 ^T)	(1.09 × 10 ⁶)	No activity
	4		(2.65 × 10 ⁶)	
	6		(39.8 × 10 ⁶)	

This imperfect antimicrobial activity may be attributed to the low dye uptake, as expressed by the *K/S* values, i.e., a small amount of Dye 4 was adsorbed on the UHMWPE fiber surface, as compared with adsorption on the PET and Nylon 6 surfaces reported in previous studies [17, 18].

4. Conclusions

The UHMWPE fiber was dyed with five hydrozono propanenitrile dyes that have antimicrobial function in scCO₂. The dye uptake – expressed as the color strength, *K/S* – was higher on the UHMWPE fabric dyed with relatively smaller molecule bearing double methyl groups, N'-(2-chloro-4-methylphenyl)-2-oxo-2-(p-tolyl) acetohydrazonoil cyanide (Dye 4), although the dyeability was significantly low, as compared with polyester and polyamide fibers. To improve the dyeability of UHMWPE fibers, decaline, which is generally used for gel spinning, was added in scCO₂ as a cosolvent. The dye uptake – expressed as the color strength, *K/S* – increased by approximately 48% in comparison with a simple scCO₂ dyeing for 1 hour, which was 5.2 times larger than the water-dyeing process. However, the process of UHMWPE in scCO₂ containing decaline and/or dye adsorption to the fiber reduced its crystallinity and mechanical strength. The color strength increased with an increase in concentration of Dye 4 and treating time up to 3 hours, suggesting slow dye diffusion into UHMWPE fiber. The color fastness to rubbing and sublimation of UHMWPE dyed at lower concentration of Dye 4 was 4–5 grade, which is acceptable for commercial products. The UHMWPE fiber dyed with Dye 4 exhibited a good antibacterial property against Gram+ve bacteria (*Staphylococcus aureus*) and the activity was insufficient.

Acknowledgments

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