Dyeing of Meta-aramid Fibers with Disperse Dyes in Supercritical Carbon Dioxide

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Abstract: Dyeing characteristics of meta-aramid fibers were investigated in supercritical carbon dioxide by employing three disperse dyes and a carrier. The effects of dyeing temperature, pressure, time, dye concentration, CO_2 flow, and carrier concentration on dyeing properties were investigated. The results showed that meta-aramid fiber could be dyed in supercritical carbon dioxide. Its color depth was improved with increasing dyeing temperature, pressure, time, dye concentration, CO_2 flow, and carrier concentration. Moreover, the color depth could be significantly improved by adding the carrier. The dyeing procedure of supercritical carbon dioxide fluid did not influence the chemical structure and antistatic properties of the meta-aramid fiber. The maximum decomposition temperature and breaking strength of the dyed meta-aramid fiber are slightly increased. The dyed meta-aramid fiber in supercritical carbon dioxide had good fastness, which was rated at 4-5.

Keywords: Meta-aramid, Supercritical carbon dioxide, Disperse dye, Dyeing, Fastness

Introduction

Polyisophthaloyl metaphenylene diamine (meta-aramid) is a linear macromolecule constituted with amide groups and meta-phenyl. This fiber, which is characterized by its excellent thermostability, flame retardance, electric insulativity, and radiation resistance, is extensively used in the industries of aerospace, national defense, electronics, communication, chemistry, environmental protection, and ocean development [1,2].

It is difficult for meta-aramid fiber to be colored to deep shade with aqueous medium because of its extremely high glass-transition temperature (T_{g} , 270 °C) and high crystallinity [3]. Moreover, color fastness to light of meta-aramid fiber is poor, thus causing severe color change under light source (vellow to bronze). Dope dyeing method was employed to solve these problems, while its disadvantages of dull color and inflexible production mode cannot meet the need of small batch dyeing. In order to improve the dyeability of meta-aramid fiber, significant studies have been carried out including liquid ammonia pretreatment [4] and UV/ozone Irradiation [5]. At present, high-temperature dyeing of aramid fiber with carrier is the most economic and feasible technique [6,7]. However, most carriers constantly release a strong irritant odor, which is difficult to remove from textiles. Meanwhile, dyeing procedure of aramid fiber discharges produces large amounts of wastewater containing various kinds of surfactants, unused dyes, and carriers [8]. Compared to common municipal sewage, the wastewater with these additives has high toxicity and poor biodegradability and cannot be treated with traditional biological methods.

Supercritical carbon dioxide fluid, as an environmentalfriendly medium, has been rapidly developed in recent years and has obtained numerous achievements in the field of extraction and organic synthesis. Using supercritical carbon dioxide as solvent to replace water presents the advantages of high uptake rate, short dyeing process, recycling of dyes and carbon dioxide, and zero waste water emission in textile dyeing procedures, which fully complies with the clean, green, and environmental manufacturing concept [9]. To date, coloration of synthetic fibers in supercritical carbon dioxide, such as polyethylene terephthalate (PET) [10,11], polylactide [12], polyamide 6 and 66 [13], has achieved commercial requirements. There are also considerable research works on dyeing of natural fibers in supercritical carbon dioxide, for instance, pretreated/modified cotton fibers and reactive disperse dyes for wool, silk and cotton fibers [13,14].

In the present work, the dyeing of meta-aramid fibers with C.I. Disperse Red 60, C.I. Disperse Yellow 114, and C.I. Disperse Blue 79 was investigated in supercritical carbon dioxide. The improvements of dyeing properties on metaaramid fibers were also investigated by employing an environmental and non-toxic carrier-CINDYE DNK in supercritical carbon dioxide under various conditions. Furthermore, the thermal, physical and antistatic properties of the metaaramid fabric were also measured.

Experimental

Materials

Meta-aramid fabrics (*m*-aramid fiber 93 %, *p*-aramid fiber 5 %, organic conductive fiber 2 %, 2/1 / twill weave, weft and warp 32s/2) were supplied by Dandong Unik Textile Co., Ltd. (China) in this study, and the fabric sample with a dimension of about 110×180 mm was used in beam dyeing procedure. C.I. Disperse Red 60 (>98 %), C.I. Disperse Yellow 114 (>98 %), and C.I. Disperse Blue 79 (>98 %) without any addition of surfactants and salts were supplied

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Table 1. Dyes used for supercritical dyeing

by Qinghai Xuezhousanrong Co., Ltd. (China) and used without further purification. CINDYE DNK (liquid) supplied by Ningbo Bozzetto Group (China) was used as carrier. Analytical reagent grade sodium hydroxide was used to rinse meta-aramid fabric. The purity of carbon dioxide was 99.99 %. The chemical structures of disperse dyes are listed in Table 1.

Pretreatment

About 4.21 g meta-aramid fabric was scoured in 50 ml deionized water containing 3 g/l soda ash and 2 g/l soap powder to remove spinning and knitting waxes and oils. The bath was raised to 60 °C for 15 min. The washing process was then carried out for 20 min. Following this, meta-aramid fabric was rinsed with cold water at 20 °C and dried at room temperature. Before dyeing procedure, meta-aramid fabric was dipped into the CINDYE DNK for 5-10 min, making its liquid rate 60 % to 100 %.

Dyeing Procedure

All experiments were performed on a self-developed batch-type supercritical dyeing apparatus. Schematic drawing of the apparatus was depicted in Figure 1. Water-scoured meta-aramid fabric was fixed, without tension, onto a porous dyeing beam and was placed into the dyeing kettle (7). The disperse dye with a ratio of 1.5 % to 5.5 % o.m.f. (on the mass of fabric), was packed into a dye cylinder and placed into the dye kettle (6). Carbon dioxide in the CO₂ tank (1) filtered with a purifier (2) was cooled and changed into liquid by a refrigerator (3). It was pressurized to above the critical pressure at 7.38 MPa using a high-pressure pump (4) and was heated to above the critical temperature at $31.1 \,^{\circ}$ C with a heat exchanger (5). The solid dyes were then dissolved in supercritical carbon dioxide fluid and flowed through the



Figure 1. Schematic drawing of the supercritical carbon dioxide dyeing apparatus; (1) CO_2 tank, (2) purifier, (3) refrigerator, (4) high-pressure pump, (5) heat exchanger, (6) dye kettle, (7) dyeing kettle, and (8) separator.

dyeing kettle in which meta-aramid fabric would be dyed. The dyeing experiments were conducted for 10 min to 90 min at dyeing temperatures, pressures, ranging from 80 °C to 160 °C, 18 MPa to 34 MPa, respectively.

After a requested dyeing period of 10 min to 90 min, the fabric was extracted with fresh carbon dioxide for 20 min to remove unfixed dyes from the fabric and the pipelines. During this process, temperature in the dyeing kettle was maintained at 70 °C under isobaric conditions at 16 MPa. Finally, the fluid and the dissolved dyes in the dyeing system were separated with a separator (8), and then recycled at pressures and temperatures ranging from 3 MPa to 4 MPa and from 25 °C to 40 °C, respectively. The meta-aramid fabric was removed and used for color measurement.

Colorimetric Measurements

The reflectance of the folded two-ply of the dyed sample was measured in the range of 380-720 nm by employing a Color-Eye 7000A spectrophotometer (X-rite, America). The color depth (K/S value) was calculated from the reflectance at the wavelength of maximum absorption for each dye by using the Kubelka-Munk equation [15]:

$$\frac{K}{S} = \frac{\left(1-R\right)^2}{2R} = aq \tag{1}$$

where K is the absorbance coefficient of the fabric to be tested; S the scattering coefficient of the fabric to be tested; R the reflectance of the fabric at each wavelength; a a constant and q is the adsorbed dye on the fabric. The K/S values from the fabric samples can be calculated for measurements made in a reflectance mode, and are directly correlated to the dye concentration on the dye substrate.

FT-IR Test of meta-Aramid Fiber

The chemical structure changes of meta-aramid fabrics before and after dyeing in supercritical carbon dioxide were characterized using an FT-IR spectrophotometer (Nicolet iS5, USA) by attenuated total reflection (ATR) method at room temperature. Thirty-two scans with 4 cm⁻¹ resolution were carried out for each case under the reflection mode.

XRD Test of meta-Aramid Fiber

The crystalline states of meta-aramid fabrics dyed in supercritical carbon dioxide with and without CINDYE DNK were determined via an XRD instrument (D/Max-3B, Rigaku, Japan). The test conditions were: wavelength 1.5406×10^{-10} m, tube voltage 40 kV, current 25 mA, sequential scanning counting mode and scanning speed 4 °/min. The standards employed were DS 1 °, SS 1 °, and RS 0.3 mm.

Thermal and Antistatic Properties

Thermal degradation behavior of meta-aramid fabrics before and after dyeing in supercritical carbon dioxide was conducted using a LINSEIS thermal analysis machine (STA PT 1600, Germany) at a heating rate of 10 °C/min. In addition, the antistatic properties of meta-aramid fabric were tested by an induction electrostatic tester (YG342D, China).

Breaking Strength

The breaking strength measurements were carried out on a universal material testing machine (gauge length, 100 mm; test speed, 100 mm/min; preliminary tension, 2 N; TH-8102S, TopHung, China), and the strength value (σ_b /MPa) was calculated according to equation (2):

$$\sigma_b = \frac{4F_p}{\pi D^2} \tag{2}$$

where F_p is the maximal tensile fracture force in kg; *D* is the mean diameter of meta-aramid fibers in centimeter. Each experiment is carried out five times, and each data entry was the average of five specimens.

Fastness Test

Color fastness to light, washing and rubbing of the dyed meta-aramid fabrics was estimated according to ISO 105-B02-2013 Color fastness to artificial light: Xenon arc fading lamp test, ISO 105-C10-2006 Color fastness to washing with soap or soap and soda, and ISO 105-X12-2001 Color fastness to rubbing.

Results and Discussion

Effect of Dyeing Temperature on *K/S* Value of meta-Aramid Fiber

The dyeing properties of meta-aramid fibers were affected by many factors, such as dyeing temperature, pressure, time, CO_2 flow, the substantivity of the dye, and other factors. In order to investigate the effect of dyeing temperature in supercritical carbon dioxide, meta-aramid fibers were dyed with C.I. Disperse Red 60, C.I. Disperse Yellow 114, and C.I. Disperse Blue 79 at temperatures ranging from 80 °C, to 160 °C, with a pressure 30 MPa, a dyeing time 70 min, a dye



Figure 2. Effect of dyeing temperature on the *K/S* value in supercritical carbon dioxide; (**■**) C.I. Disperse Blue 79, (**▲**) C.I. Disperse Red 60, (**●**) C.I. Disperse Yellow 114, (**□**) C.I. Disperse Blue 79+CINDYE DNK, (\triangle) C.I. Disperse Red 60+CINDYE DNK, (**○**) C.I. Disperse Yellow 114+CINDYE DNK.

concentration 4.5 %, a CO_2 flow 50 g/min, and a carrier concentration 80 %. As depicted in Figure 2, *K/S* values of the fibers were slightly increased as temperature from 80 °C to 120 °C, and then arrived at a maximum at 140 °C, which indicated that the color depth of meta-aramid fiber was not noticeably improved at various dyeing temperatures with the tested disperse dyes alone. Compared with the literature data from Kim *et al.* [8], the *K/S* value of the fabric significantly was increased by adding CINDYE DNK with the temperature from 80 °C to 160 °C, which indicated that dyeing of meta-aramid fibers with CINDYE DNK could obviously improve color depth in supercritical carbon dioxide.

For the dyeing of meta-aramid fibers, the addition of CINDYE DNK could also increase the K/S value at temperature 130 °C in water [16]. Theoretically, it is difficult for aramid fiber to be colored to deep shade due to its extremely high T_g and high crystallinity. However, supercritical carbon dioxide could swell and plasticize the aramid fibers to a certain degree, which led to a decrease in T_{g} . Moreover, CINDYE DNK could combine with fibers in the form of van der Waals force and hydrogen bonds, which could change fiber-to-fiber bonds into fiber-to-carrier bonds, diminishing the bonding force between aramid fibers and increasing occurrence probability of the holes and the diffusion rate of dyes. The carrier could also plasticize the fibers and improve the activity ability of fiber macromolecule chains, thereby causing the free volume of aramid fibers to increase. Thus, the dyeability of meta-aramid fibers in supercritical carbon dioxide was available.

Effect of Dyeing Pressure on the *K/S* Value of meta-Aramid Fiber

The effect of dyeing pressure on the color depth of meta-



Figure 3. Effect of dyeing pressure on the *K/S* value in supercritical carbon dioxide; (**•**) C.I. Disperse Blue 79, (**\Delta**) C.I. Disperse Red 60, (**•**) C.I. Disperse Yellow 114, (\Box) C.I. Disperse Blue 79+ CINDYE DNK, (\triangle) C.I. Disperse Red 60+CINDYE DNK, (\circ) C.I. Disperse Yellow 114+CINDYE DNK.

aramid fiber with C.I. Disperse Red 60, C.I. Disperse Yellow 114, and C.I. Disperse Blue 79 was observed from 18 MPa to 34 MPa with a temperature 140 °C, a dyeing time 70 min, a dye concentration 4.5 %, a CO_2 flow 50 g/min and a carrier concentration 80 % (Figure 3). It is clear from the results in Figure 3 that the color depth of the meta-aramid fibers gradually increased with increasing pressure. On the other hand, there were remarkable improvements on the *K/S* values of dyed samples in the presence of CINDYE DNK under identical conditions. In principle, the density of supercritical carbon dioxide was low at lower pressures, and the solubility of disperse dyes and the swelling of the fibers could be improved with increasing pressure [8,17], resulting in the rising of color depth of the fibers.

Effect of Dyeing Time on the *K/S* Value of meta-Aramid Fiber

The effect of dyeing time on the color depth of metaaramid fiber with C.I. Disperse Red 60, C.I. Disperse Yellow 114, and C.I. Disperse Blue 79 was observed from 10 min to 90 min with a temperature 140 °C, a pressure 30 MPa, a dye concentration 4.5 %, a CO₂ flow 50 g/min, and a carrier concentration 80 %. Liang *et al.* showed that the maximum color strength of meta-aramid fibers was obtained after dyeing of 60 min with CINDYE DNK in water medium [16]. As shown in Figure 4, the coloration of the dyed fibers steadily increased from 10 min to 90 min, while the *K/S* value increased enormously with time under the action of CINDYE DNK. This observation can be explained in terms of the uniform distribution of the dyes both on the fibers and in the carbon dioxide fluid because of the homogeneous mass transfer of the fluid within enough time.



Figure 4. Effect of dyeing time on the *K/S* value in supercritical carbon dioxide; (**n**) C.I. Disperse Blue 79, (**A**) C.I. Disperse Red 60, (**•**) C.I. Disperse Yellow 114, (\Box) C.I. Disperse Blue 79+ CINDYE DNK, (\triangle) C.I. Disperse Red 60+CINDYE DNK, (\circ) C.I. Disperse Yellow 114+CINDYE DNK.



Figure 5. Effect of dye concentration on the *K/S* value in supercritical carbon dioxide; (**a**) C.I. Disperse Blue 79, (**A**) C.I. Disperse Red 60, (**•**) C.I. Disperse Yellow 114, (**□**) C.I. Disperse Blue 79+CINDYE DNK, (\triangle) C.I. Disperse Red 60+CINDYE DNK, (**◦**) C.I. Disperse Yellow 114+CINDYE DNK.

Effect of Dye Concentration on the *K/S* Value of meta-Aramid Fibers

The effect of dye concentration on the color depth of meta-aramid fibers with different quantity of C.I. Disperse Red 60, C.I. Disperse Yellow 114, and C.I. Disperse Blue 79 (1.5 %, 2.5 %, 3.5 %, 4.5 %, or 5.5 % o.w.f.) was investigated with a temperature 140 °C, a pressure 30 MPa, a dyeing time 70 min, a CO₂ flow 50 g/min, and a carrier concentration 80 %. As shown in Figure 5, when the dye concentration was low, the *K/S* value of the dyed samples significantly increased with the dye concentration from 1.5 % o.w.f. to 4.5 % o.w.f., and a plateau was then reached at 5.5 % o.w.f.

It indicated that the dyes on fibers got saturation of adsorption with 4.5 % o.w.f. at this supercritical condition.

Effect of CO₂ Flow on the K/S Value of meta-Aramid Fiber

Figure 6 shows the effect of CO_2 flow on the color depth of meta-aramid fibers with C.I. Disperse Red 60, C.I. Disperse Yellow 114, and C.I. Disperse Blue 79 from 10 g/min to 50 g/min with a temperature 140 °C, a pressure 30 MPa, a dyeing time 70 min, a dye concentration 4.5 %, and a carrier concentration 80 %. It can be seen from Figure 6 that the *K/S* value of meta-aramid fibers gradually increased as the CO_2 flow increased. This may be caused by decreased metaaramid fiber T_g in supercritical carbon dioxide. Thus the dye molecule was able to penetrate more easily into the amorphous phase of the fibers owing to swelling of the fibers by



Figure 6. Effect of CO₂ flow on the K/S value in supercritical carbon dioxide; (**a**) C.I. Disperse Blue 79, (**A**) C.I. Disperse Red 60, (**•**) C.I. Disperse Yellow 114, (**□**) C.I. Disperse Blue 79+ CINDYE DNK, (\triangle) C.I. Disperse Red 60+CINDYE DNK, (\circ) C.I. Disperse Yellow 114+CINDYE DNK.



Figure 7. Effect of carrier concentration on the *K/S* value in supercritical carbon dioxide; (\Box) C.I. Disperse Blue 79, (\triangle) C.I. Disperse Red 60, (\circ) C.I. Disperse Yellow 114.

supercritical carbon dioxide, which improved the dyeability of the fibers [18].

Effect of Carrier Concentration on the K/S Value of meta-Aramid Fiber

The effect of carrier concentration on the color depth of meta-aramid fiber with C.I. Disperse Red 60, C.I. Disperse Yellow 114, and C.I. Disperse Blue 79 from 60 % to 100 % was investigated with a temperature 140 °C, a pressure 30 MPa, a dyeing time 70 min, a dye concentration 4.5 %, and a CO_2 flow 50 g/min. A moderate increase was found from 60 % to 100 % for all three dyes, as shown in Figure 7. Increased color depth mentioned above could indicate more efficient swelling effect with higher CINDYE DNK concentration facilitating dye molecules absorbed into the fibers.

Effect of Supercritical Carbon Dioxide and CINDYE DNK on Properties of meta-Aramid Fiber

To investigate the possible change in the chemical structure of meta-aramid fibers, FT-IR spectroscopy measurements were performed, and the spectra were given in Figure 8. It can be seen from Figure 8 that there is almost no change in



Figure 8. FT-IR spectra of meta-aramid fibers and CINDYE DNK.



Figure 9. XRD spectra of meta-aramid fibers.

the spectra of meta-aramid fibers before and after dyeing in supercritical carbon dioxide with and without CINDYE DNK. The meta-aramid fibers' characteristic absorption peaks, which are attributed to the C=O and -CN stretching vibration peak, appear in all spectra at 1645.4 cm⁻¹ and 1527.9 cm⁻¹. The absorption peaks at 1476.7 cm⁻¹ and 1603.6 cm⁻¹ are assigned to benzene. The spectrum of CINDYE DNK is similar to meta-aramid fibers, where the absorption peaks at 1900-1650 cm⁻¹ and 3500-3180 cm⁻¹ are assigned to -NHCO. The absorption peaks of benzene appear at 1600-1585 cm⁻¹ and 1500-1400 cm⁻¹. Therefore, as an aromatic amide compound, based on Like-Dissolves-Like Theory, it has a good expansion effect to aramid fibers in dyeing procedure.

The meta-aramid fibers with and without CINDYE DNK in supercritical carbon dioxide were selected to analyze the crystal structures of the fibers via XRD. Figure 9(a) shows that there is no distinct difference in the XRD spectra of meta-aramid fibers dyed in supercritical carbon dioxide, which indicated that supercritical carbon dioxide did not change the crystal structure of the fibers. Figure 9(b) presents the XRD spectra of the meta-aramid fibers with CINDYE



Figure 10. TG thermodiagrams of meta-aramid fibers.

DNK. It illustrated that the crystallinity of meta-aramid fibers treated with CINDYE DNK was slightly decreased, probably caused by the swelling effect of CINDYE DNK occurred on the chain of polymer molecules.

The thermal degradation behavior of the meta-aramid fibers determined by thermo-gravimetric analysis was shown in Figure 10. The maximum decomposition temperature of the control sample appeared at 422.11 °C, while those of meta-aramid fibers, which were dyed with and without CINDYE DNK, were increased by 9.21 and 3.46 °C, respectively. The thermal property of meta-aramid fibers was then improved after dyeing with CINDYE DNK in supercritical carbon dioxide. The mechanism behind such a thermal stability improvement probably lies in the neatened molecular chains of meta-aramid fiber polymer in the presence of CINDYE DNK after dyed in supercritical carbon dioxide [16].

Physical Properties of meta-Aramid Fibers

In order to investigate the mechanical properties of metaaramid fibers, the breaking strengths of dyed fibers in supercritical carbon dioxide were measured. The results are



Figure 11. Breaking strengths of dyed meta-aramid fibers in supercritical carbon dioxide.

Table 2. Antistatic properties of meta-aramid fibers (140 $^{\circ}$ C, 30 MPa, 70 min, 4.5 %, 50 g/l)

	Peak	Damping	Endpoint
Disperse dye	voltage	period	voltage
	(V)	(S)	(V)
Control sample	1314	4.37	656
Disperse Blue 79	1330	4.9	664
Disperse Red 60	1232	3.17	615
Disperse Yellow 114	1232	3.43	615
Disperse Blue 79+CINDYE DNK	1336	4.95	667
Disperse Red 60+CINDYE DNK	1306	4.26	648
Disperse Yellow 114+CINDYE DNK	1254	3.62	620

given in Figure 11. It can be seen that, compared with the control sample, the breaking strength of meta-aramid fibers dyed in supercritical carbon dioxide with CINDYE DNK was slightly increased. In principle, carbon dioxide can penetrate into hydrophobic fibers and act as a good solvent. It has little impact on crystallinity and melting temperature of various polymers [8]. Moreover, CINDYE DNK has a

good expansion effect to aramid fibers, which can significantly neaten the macromolecular structure of meta-aramid fibers, thus leading to the increase in meta-aramid fiber's breaking strength [16].

The effect of the supercritical carbon dioxide and CINDYE DNK on the antistatic properties of meta-aramid fibers was tested in the presence of organic conductive fibers. As shown in Table 2, the damping periods of the dyed samples were not significantly changed. Therefore, based on the above testing, it can be seen that there was little influence of supercritical carbon dioxide fluid and CINDYE DNK on the chemical structure and antistatic properties of meta-aramid fibers.

The color fastness of the dyed fibers to washing, rubbing, and light was shown in Table 3. The fastness to washing and rubbing is excellent, which was rated at 4-5. Thus, the excellent light color fastness can be invaluable advantages considering the very low light color fastness of meta-aramid fabrics dyed with water medium.

Conclusion

The dyeing characteristics of meta-aramid fibers were investigated in supercritical carbon dioxide under various conditions such as dyeing temperature, pressure, time, dye concentration, CO₂ flow, and carrier concentration. The results showed that, although the color depth was relatively low, meta-aramid fiber could be dyed in supercritical carbon dioxide, while significant improvements in color depth were achieved by employing CINDYE DNK. Meanwhile, the color depth was improved with increasing dyeing temperature, pressure, time, dye concentration, CO₂ flow, and carrier concentration; moreover, the fastness data showed that acceptable washing fastness (shade and stain), rubbing fastness (wet and dry), and light fastness were obtained at 4-5 for meta-aramid samples colored in supercritical carbon dioxide. More importantly, supercritical carbon dioxide fluid did not influence the chemical structure and antistatic properties of the meta-aramid fiber. The maximum decomposition temperature and breaking strength of the meta-aramid fiber were slightly increased.

Table 3. Colorfastness of the dyed meta-aramid fiber to washing, rubbing and light irradiation

Disperse dye	Washing fastness				Rubbing fastness		
	Shade	Stain			Dere	Wat	Light fastness
		Wool	Nylon	Cotton	Dry	wet	
Disperse Blue 79	4-5	4-5	4-5	4-5	4-5	4-5	4
Disperse Red 60	4-5	4-5	4-5	4-5	5	4-5	4
Disperse Yellow 114	4-5	4-5	4-5	4-5	4-5	4-5	4
Disperse Blue 79+CINDYE DNK	4-5	4-5	4-5	4-5	5	5	4-5
Disperse Red 60+CINDYE DNK	4-5	4-5	4-5	4-5	5	5	4-5
Disperse Yellow 114+CINDYE DNK	4-5	4-5	4-5	4-5	5	5	4

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Acknowledgements

This work was supported by National Special fund for cleaner production (Ministry of Finance [2011] 430), National Science and technology transformation project (Ministry of Finance [2011] 329), and Doctoral Initiating Project of Liaoning Province Foundation for Natural Sciences (20111069).

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