Research article

SURFACE and INTERFACE ANALYSIS

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An attempt of improving polyester inkjet printing performance by surface modification using β-cyclodextrin

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To improve the sharpness and the color yield of polyester fabrics with water-based pigment inkjet printing, surface modification was proceeded using β-cyclodextrin and citric acid. Fabrics were modified in the solution of β-cyclodextrin with the concentration 100g/L and citric acid 100g/L. The line width and image area of printed patterns, which could evaluate the printing performances, on the modified polyester fabric were decreased by 77% and 62% in comparison with that of the control, respectively. The color yield characterized by the *K/S* value was enhanced by 47%. Scanning electron microscope and atomic force microscope images confirmed that the capillary effect was decreased and the surface roughness was increased after the surface modification. The microscope images of the printed patterns clearly showed that the sharpness and the color yield were improved. Thereby, β-cyclodextrin modification offered a potential way to polyester pretreatment for pigment inkjet printing. Copyright © 2012 John Wiley & Sons, Ltd.

Keywords: polyester; surface modification; β-cyclodextrin; inkjet printing; image quality

Introduction

Inkjet printing was a flexible technology. Compared to the traditional screen printing, it provided many advantages, such as perfect adaptation to the special customized order of small batches and multi-species, less waste and less cost.[1–4] Water-based pigment inks took a place in the inkjet printing inks due to its complete chromatogram and excellent light fastness.[5–7] The adhesive force between the pigment and the fiber was weak. The fixation, which carried out through the crosslinking of adhesives was in the subsequent processing after printing. Then, ink droplets sprayed onto the fabrics easily penetrated to the surroundings, resulting in motion-blur patterns and the poor color yield.[8,9] Due to the specific purity and conductivity requirements for inkjet printing, none of the printing chemicals could be directly incorporated into the ink formulation. Pretreatment provided an alternative method to reduce the penetrations.[10] Many researches had reported the pretreatments of fabrics to be printed prior to the stage of inkjet printing.[11,12] The penetrations of ink droplets on hydrophobic fibers such as the polyester fiber were especially serious for the smooth morphology, hydrophobic property and few polar groups. Plasma technology was a useful tool to modify the polyester fabric.[13,14] Krump *et al.* proposed a method for the morphological changes and the chemical reactivity alteration of polyester fibers by using atmospheric plasma and microwave-plasma technologies.[15] Raffaele-Addamo *et al*. made an investigation in the air radiofrequency plasma treatment. The results showed that the dyeing property of the modified fabric was correlated to the topographical characteristics and the chemical surface composition.[16] The acrylic resin had also been applied by Park *et al.* to carry out the polyester pretreatment. The results showed that the printing quality was significantly improved.[17]

Cyclodextrins were known to form inclusion complexes in aqueous solution with a large number of organic molecules with hydrophobic character. And they had been used in wide areas for the non-toxicity and biodegradability. The applications of cyclodextrins and their derivatives in the textile domain appeared in the early 80s.[18] The research progress had extended to several textile processings such as spinning,[19] pretreatment,[20,21] dyeing,[22] printing[23] and finishing.[24–26] Cotton fabrics were modified with the β-cyclodextrin derivatives, and the possibility of entrapping sandalwood oil as an aroma-finishing agent was reported. No loss of tensile strength of the treated fabric was found.[27] Scalia *et al.* also investigated the incorporation of the sunscreen agent into cyclodextrin, which was covalently bonded to Tencel fabric.[28] The applications of β-cyclodextrin immobilized synthetic fabrics in adsorbing heavy metal ions (Pb^{2+}, Cd^{2+} and Ni^{2+}) from water had also been studied.[29,30] Blanchemain *et al.* investigated the coatings of cyclodextrins onto woven polyester vascular prothesis using citric acid as the crosslinking agent for the controlled release of ciprofloxacin. It would benefit the prevention of post surgery complications.[31] However, investigations on the applications of cyclodextrins in the fabric pretreatment for inkjet printing had not been reported.

In our previous work, cationic pretreatment was applied to the polyester fabric for inkjet printing. The higher sharpness and the deeper color yield were obtained comparing with that of the control.[32] This work aimed at investigating the use of β-cyclodextrin in polyester modification for ink-jet printing. Citric acid and sodium dihydrogen hypophosphite were used as the

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Key Laboratory of Eco-Textile, Ministry of Education, School of Textiles and Clothing, Jiangnan University, 1800 Lihu Road, Wuxi, 214122, People's Republic of China

crosslinking agent and the catalyst, respectively. The effects of the modification on the sharpness and the color yield for water-based pigment inkjet printing were investigated. In order to obtain the high sharpness and the deep color yield, β-cyclodextrin concentration and [β-cyclodextrin]/[citric acid] mass ratio were studied.

Experimental

Materials

100% polyester plain weave fabric (50D × 50D, 61.4g/m^2) was commercially available. β-cyclodextrin (β-CD) (Fig. 1) was supplied by Sinopharm Chemical Reagent Co., Ltd. Citric acid (CTR) was obtained from China Medical Group Shanghai Chemical Reagent Co. Sodium hypophosphite (SHP) was provided by Jiangsu Yonghua Fine Chemistry Co., Ltd. Pigment-based inks were prepared in the laboratory.

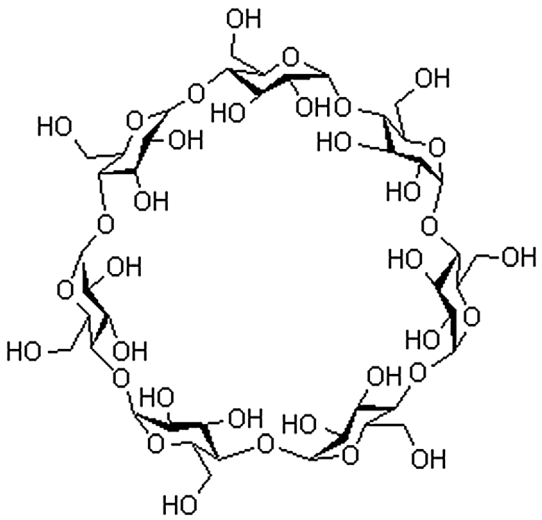

Figure 1. Chemical structure of β-cyclodexrtrin.

Polyester fabrics modification

β-CD, CTR and SHP were dissolved in water at 80°C under continuous stirring until adequate dissolution. Polyester fabric was twice dipped into the adequately stirred aqueous solution containing β-CD, CTR, SHP and padded with a P-130 padder produced by Taiwan Rapid Co. Ltd. The concentration of β-CD was varied from 40g/L to 100g/L. The concentration of SHP was 30g/L. The [β-CD]/[CTR] mass ratio was from 3:2 to 2:3. At this point, the β-cyclodextrin concentration remained constant 100g/L. This treatment gave a wet pick up of about 70%. Then, the fabrics were dried at 90°C for 2 min and cured at 160°C for 5 min. Finally, fabrics were washed several times with warm water to remove the residual β-CD and CTR left on the surfaces. The modified polyester fabrics in the fabric resistance, surface morphology and printed patterns on polyester fabrics parts were treated with 100g/L β-CD, 100g/L CTR and 30g/L SHP.

Inkjet printing

Aqueous pigment inks were prepared by adding ultra-fine pigment, the mixture of PEG_{1000} and PEG_{2000} as a binder and deionized water. The detailed formulation was in the literature [35]. The colors of pigment inks were composed of carbon black, magenta (PR.122), yellow (PY.14), cyan (PB.15:3), light cyan and light magenta. Inkjet printing was carried out on an Epson R-230 inkjet printer and variable 720 dpi in a low speed.

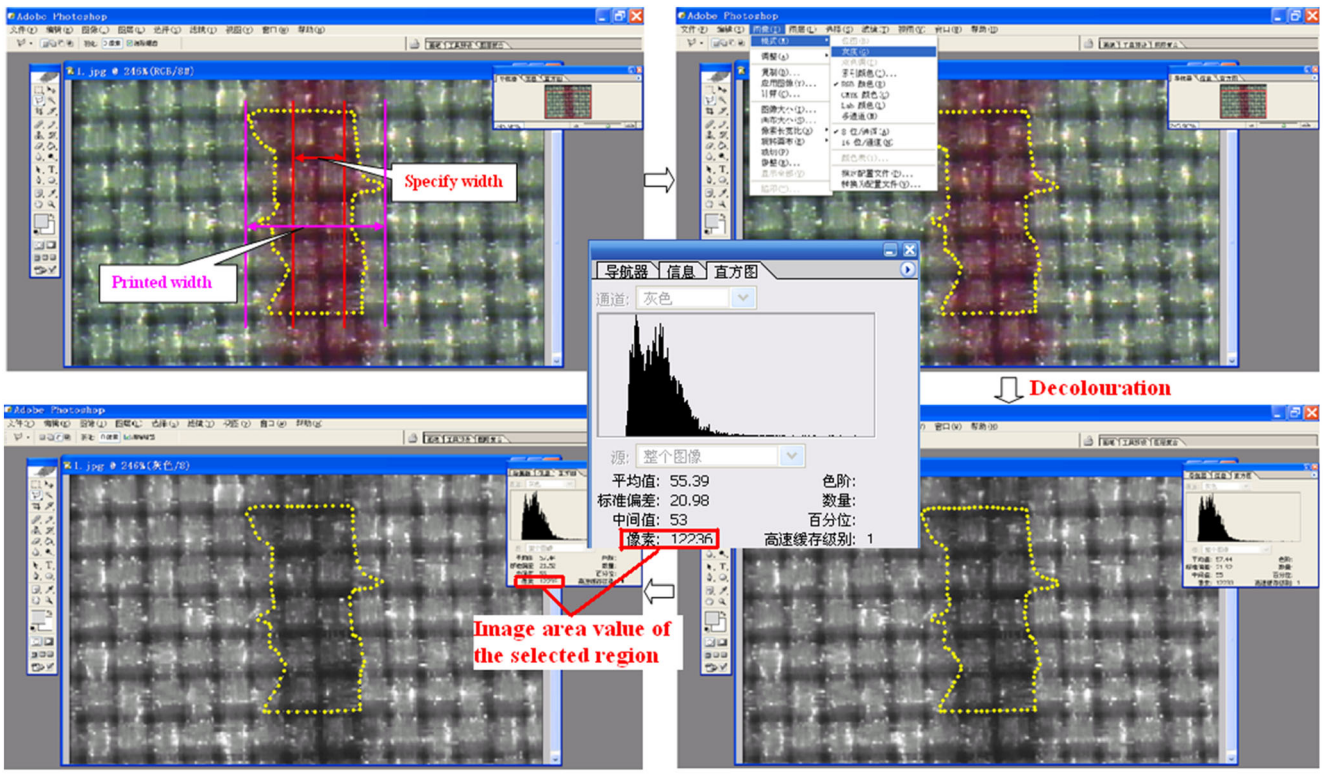

Figure 2. Characteration process of the image area taken by photoshop software.

Printed samples were dried at 70°C for 5 min, and fixed at 140°C for 5 min to promote the crosslinking of the binders.

Sharpness

Printed samples were photographed by DZ3-Ultra High Magnification Zoom Microscope supplied by Union Optical Co., Ltd. and Pinnalce Studio Version 8.0 software with a magnification of 75. Printed lines widths were specified as 0.5×10^3μm, 1.0×10^3μm and 1.5×10^3μm. Practical printed widths including the penetration areas were collected by Imager-Proplus Express software. The regions of the photographed lines in 5 cm including the penetration areas were delineated and decolorized to evaluate the image area via photoshop software and measured in pixels. A spot in the display screen meant one pixel. The higher the pixel value was, the poorer the sharpness was. The process was showed in Fig. 2. Three different regions were measured, and the mean value was calculated.

Table 1. Effect of the β-CD concentration on the *K/S* value and chromatometric values(L^*, a^*, b^*)

Values	Concentration of β-cyclodextrin(g/L)				
	0	40	60	80	100
K/S	3.59	4.63	4.75	4.88	5.31
L^*	51.28	49.20	49.01	48.78	48.30
a^*	38.48	40.52	40.98	41.46	42.32
b^*	−14.18	−13.55	−13.37	−13.05	−13.06

Color yields

Color yields were determined using X-Rite 8400 spectrophotometer obtained from American X-Rite Co. Ltd. Samples printed with magenta color patches were measured in the system of CIE Lab, D65 lamp-house and the 10° visual angle. The color yields denoted by the *K/S* values were determined with the Kubelka-Munk equation. Five different regions were measured to ensure the accurate results.

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

Where, K is the absorption coefficient; S is the scattering coefficient and R is the fractional reflectance (value from 0 to 1) of the colored fabric at the wavelength of maximum reflectance. The *K/S* value is proportional to the color yield.[34]

Drop shape analysis

Deionized water was used as the liquid probe to analyse the wettability of the fabric. With the fabric getting wet, the drop shapes of the water on the fabric surface continuously changed. The shapes were collected with DSA-100 drop shape analyzer from Krüss CO.LTD Germany.

Fabric resistance

Resistances of unmodified and modified polyester fabric samples were assessed by ASTM D 257–07 Standard Test Methods for DC Resistance or Conductance of Insulating Materials using LFY-406 fabric surface resistance tester (Textile Research Institute of Shandong Province) at ambient conditions. The higher fabric resistance is, the worse electrical conductivity will be, and vice versa.

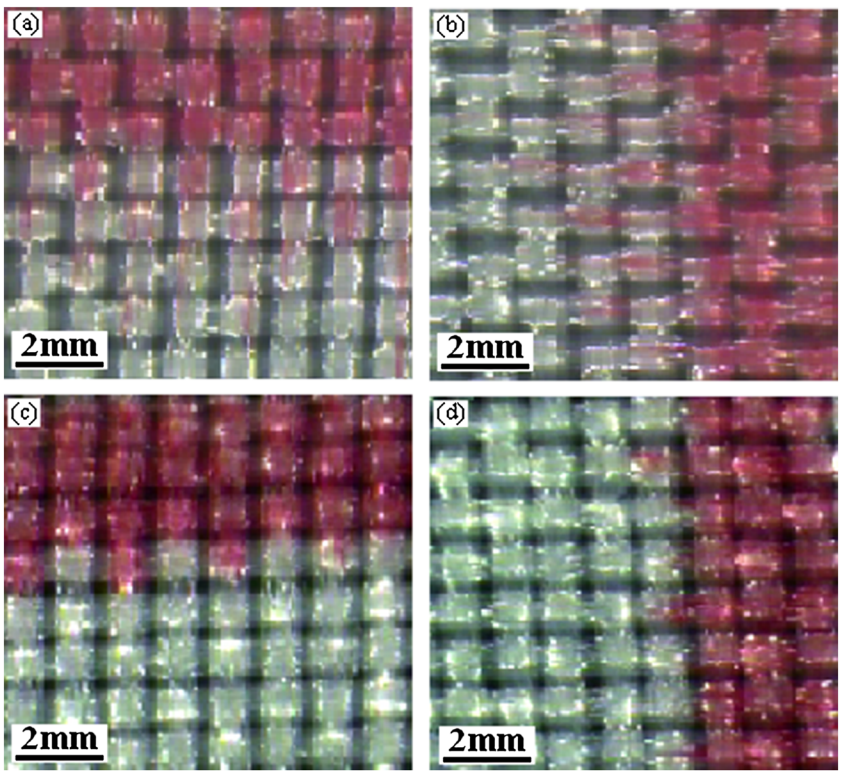

Figure 3. Images of pigment printed color lumps on polyester which were taken by DZ3-Ultra High Magnification Zoom Microscope:(a)warp unmodified; (b)weft unmodified; (c)warp 100g/L β-CD modified; (d) weft 100g/L β-CD modified.

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Scanning electron microscopy

The fabric surface morphology was observed using SEM (FEI Quanta 200), which was the best known instrument for surface analysis. Polyester fabrics were inspected at 1000 magnification to detect the surface morphological changes. Before the test, samples should be coated with gold. Images were obtained at ambient conditions.

Atomic force microscope

The morphological and topographical changes of polyester fabrics were also investigated by CSMP 4000 AFM (Beijing, China), employing a microfabricated V-shaped silicon cantilever with silicon conical tip. AFM images were collected in air. The roughness average, the root mean square and the mean height of the polyester fabric were collected by the Imager Statistical Analysis Software.

Results and discussion

β-CD concentration

The polyesterification occurred between β-CD and CTR.[30,33] The formed polymers (polyCTR-CD) physically adhered or were even entangled into the fibrous network. β-CD concentration in the aqueous solution directly affected the amount of polyCTR-CD, which was related to the penetration of the ink droplets and the color yields of printed patterns.

Table 1 presented the β-CD concentration dependence on the *K/S*, L^*, a^* and b^* values of the printed color patches. The *K/S*, a^* and b^* values progressively increased and the L^* value decreased with the β-CD concentration. The values increased while β-CD was added and still increased in a lesser extent up to 100g/L. It was deduced that the polyCTR-CD played an important role on the increase of the *K/S* value. PolyCTR-CD adhered or reduced the capillary effect and increased the roughness. Hence, few ink droplets penetrated to the surroundings. It was also proved by the bleeding performance in Fig. 3. As was shown in Fig. 3 (a) and (b), the bleeding phenomenon of the polyester fabric without modification was serious along the warp and weft yarns. The anti-bleeding performance of the modified fabric was significantly improved in Fig. 3(c) and (d). The amount of pigment particles per unit area in printed areas increased, and the *K/S* value enhanced. In addition, the specular reflection was also transformed to the diffuse reflection for the increased surface roughness. More light was absorbed for the multiple absorption and reflection than that of the untreated fabric.

[β-CD]/[CTR] mass ratio

The mechanism of the polyesterification between β-CD and CTR was shown in Fig. 4.[29] Each β-CD molecule with seven hydroxyl groups reacted with the carboxyl groups in the CTR molecules. The [β-CD]/[CTR] mass ratio was related to the reaction and had an important effect on the penetration of the ink droplets.

The effect of the [β-CD]/[CTR] mass ratio on the width was studied, and results were displayed in Fig. 5. In spite of the specified line width of $0.5 \times 10^3 \mu m$, $1.0 \times 10^3 \mu m$ and $1.5 \times 10^3 \mu m$, all of the printed widths increased with the variation of the mass ratio. The increase in the range 2:1 to 1:1 was slighter than that in the range 1:1 to 1:2. The increase in the line width meant that the sharpness was reduced. The polyesterification between β-CD and CTR occurred when the fabric was dried and cured. However, the reaction probably stopped at the intramolecular evaporation of some CTR molecules (Fig. 4). The cyclic esters were formed and the hydrophilicity of the fabric was reduced, which resulted in the penetration of the ink droplets. In the range 2:1 to 1:1, most of the CTR molecules formed polyCTR-CD with the β-CD

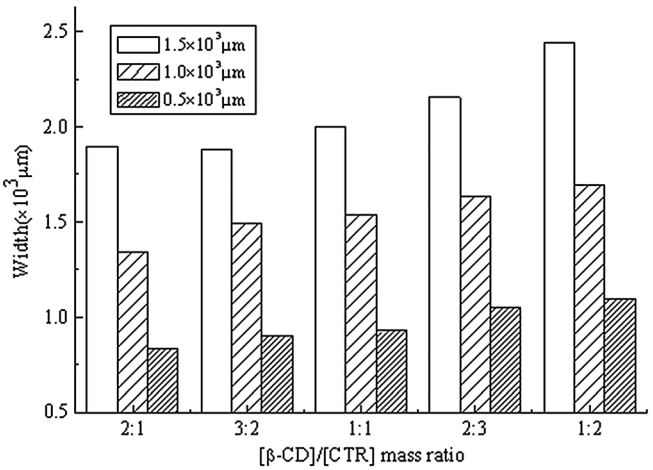

Figure 5. Effect of [β-CD]/[CTR] mass ratio on the printed width.

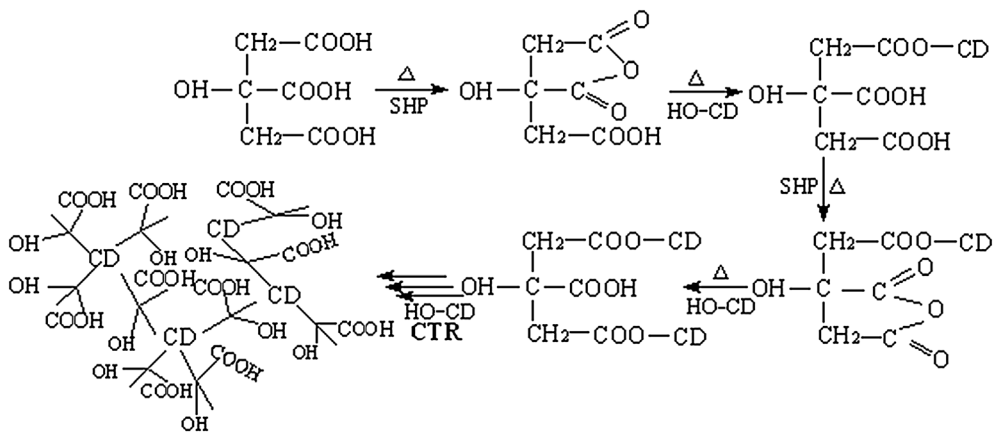

Figure 4. Reaction mechanism between β-CD and CTR.

molecules, accompanied by a small part of them generating cyclic esters through the intramolecular evaporations. Increasing the dosage of CTR enhanced the intramolecular evaporations. Hence, the width was increased. In the range 1:1 to 1:2, CTR dosage was excessive. Some CTR molecules only underwent the intramolecular evaporations reaction. The hydrophilicity of the fabric was decreased. The drop shapes of deionized water on the modified fabric in Fig. 6 made a description. The droplet on the modified polyester fabric with [β-CD]/[CTR] mass ratio 2:1 was absorbed after 15s. Nevertheless, the modified polyester fabric with [β-CD]/[CTR] mass ratio 1:2 obtained obvious decrease in hydrophilicity. It was also proved by the contact angles of the polyester fabrics. The contact angles of the fabrics treated with [β-CD]/[CTR] mass ratio 2:1 and 1:2 were 46° and 78°, respectively.

The image area, which considered the whole bleeding regions on the edge of the pattern, illustrated the sharpness more completely than the line width.

As shown in Fig. 7, the image area improved with the [β-CD]/[CTR] mass ratio similar to the line width. The increase of the image area in the mass ratio range 2:1 to 1:1 was slighter than that in the range 1:1 to 1:2. Most of the CTR molecules formed polyCTR-CD with the β-CD molecules, accompanying by a small part of them forming cyclic esters through intramolecular evaporations in the range 2:1 to 1:1. When the [β-CD]/[CTR] mass ratio was varied to 1:2, excessive CTR led to the sharp decrease in the hydrophilicity of the fabric. The ink droplets easily penetrated to the surroundings. It was necessary to pay attention to a threshold value of the [β-CD]/[CTR] mass ratio in order to observe the printing sharpness.

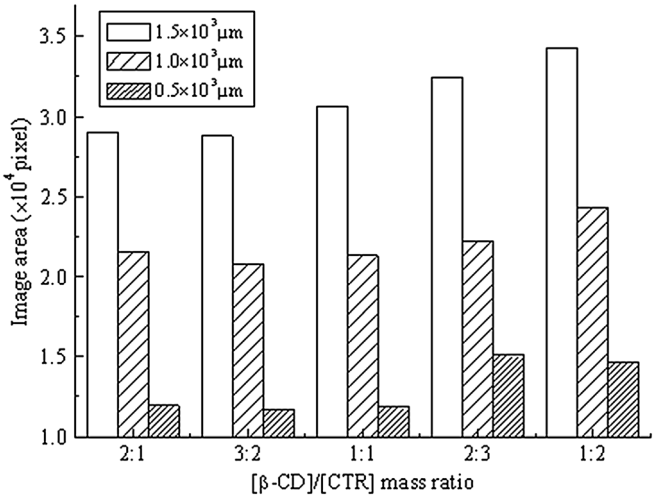

Figure 7. Effect of [β-CD]/[CTR] mass ratio on the image area.

Fabric resistance

The resistances of polyester fabrics with and without modification were given in Fig. 8. The fabric resistance value of the modified sample reduced from $3.92 \times 10^9\Omega$ of the control to $1.37 \times 10^6\Omega$. The result indicated that the modified sample achieved a higher electrical conductivity and water uptake capacity. It was attributed to the hydroxyl and carboxyl groups. The modified fabric was able to absorb more water and moisture from air. The presence of more moisture in modified polyester fabric increased their electrical conductivity and improved their antistatic properties.

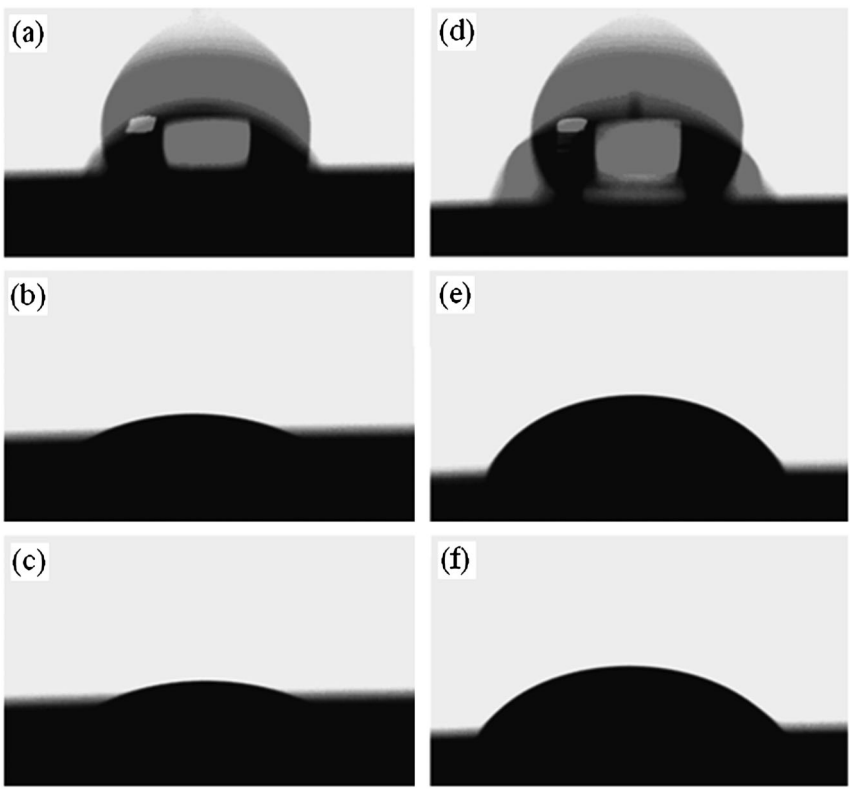

Figure 6. View of drop shapes: (a)0s (b)10s (c)15s after being dropped on modified polyester, [β-CD]/[CTR] mass ratio was 2:1, (d)0s (e)10s (f)15s after being dropped on the modified polyester, [β-CD]/[CTR] mass ratio was 1:2.

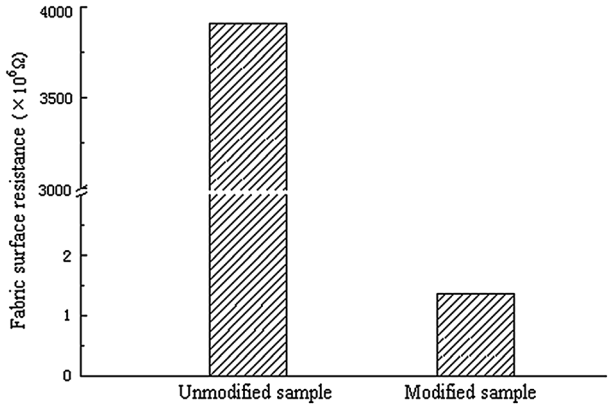

Figure 8. Polyester fabric resistances without and with modification.

Surface morphology

SEM images showed the changes in the surface morphology of the polyester fabric. In Fig. 9 (a), the polyester fabric without modification had smooth surface. The ability of holding on the ink droplets was poor. Ink droplets would penetrate to the surroundings and resulted in motion-blur patterns. However, Fig. 9 (b) treated with 100g/L β-CD, 100g/L CTR and 30g/L SHP showed that polyCTR-CD was adhered to the fabric surface and arranged disorderly. The roughness of the fabric surface increased. The glaring reflected light on the control was not seen from the modified polyester surface for the transition from the specular reflection to the diffuse reflection. More light was adsorbed, the color yield was deeper. In Fig. 9 (b), polyCTR-CD adhered to the gap areas between the yarns. The capillary effect was decreased. Ink droplets could not penetrate to the surroundings through the pore path. Thus, the sharpness of the pattern printed on the fabric was higher, and the color yield was increased.

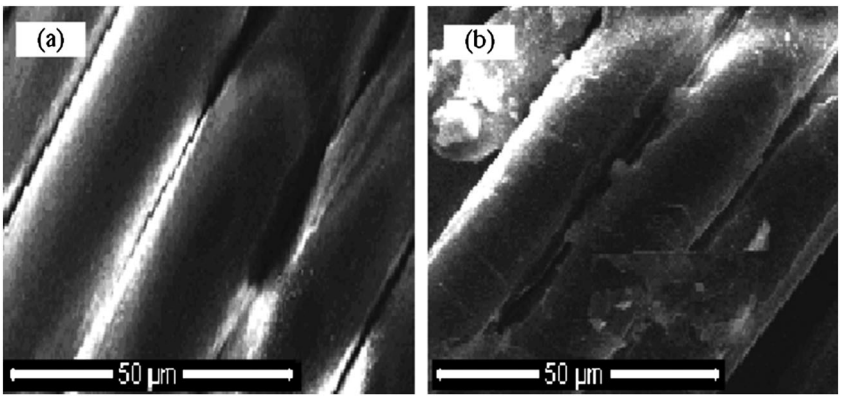

Figure 9. SEM images of the polyester fabrics without (a) and with (b) modification.

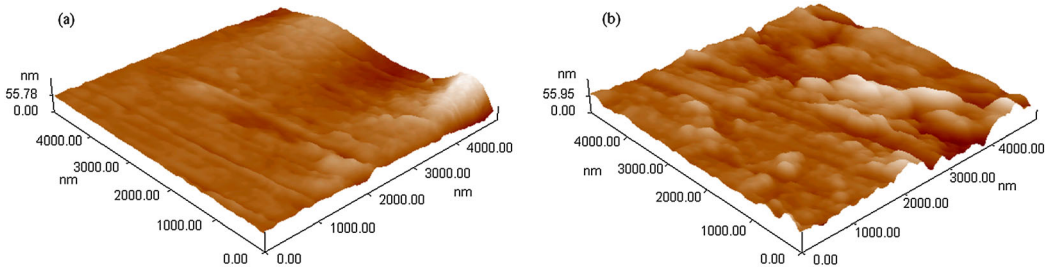

Figure 10. AFM images of the polyester fabrics without (a) and with (b) modification.

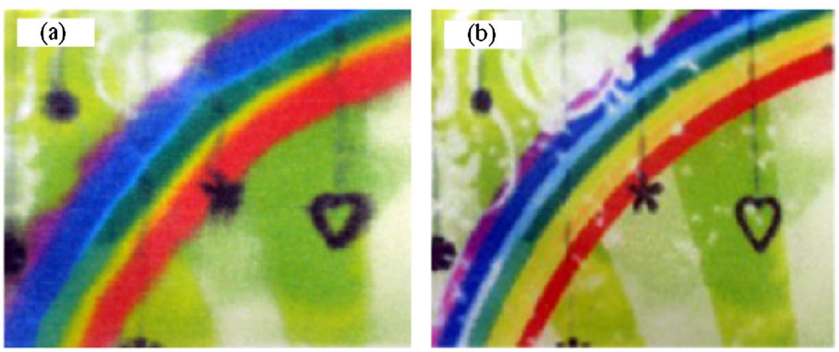

Figure 11. The inkjet printed patterns on the polyester fabrics without (a) and with (b) modification.

In addition, the morphology study of the β-CD modified polyester surface was also approached by AFM test with a higher magnification which can be used to clearly evaluate the roughness of the modified polyester surface. Fig. 10(a) showed the relatively smooth surface of the unmodified polyester ascribed to the processing of polyester fibers. By the Imager Statistical Analysis Software, the roughness average, the root mean square and the mean height of the polyester fabric without treatment were 4.49, 5.82 and 45.1nm, respectively. Fig. 10(b) revealed the surface morphology of the modified polyester fiber. The clear changes in the morphology of the modified polyester surface were noticed. The roughness average, the root mean square and the mean height of the modified polyester were 9.37, 12.60 and 64.3nm, respectively. All data were increased in comparison with that of the sample untreated. The enhancement of the roughness (from 4.49 to 9.37) was consistent with the changes of the SEM images. It allowed a good explanation for the increased *K/S* values in Table 1. The protruding portions on the surface were the disorderly arranged aggregates of polyCTR-CD. Some segments of the polyCTR-CD penetrated into the fiber matrix due to roll pressing and curing. The uneven distribution of polyCTR-CD aggregates caused the increasing of the surface roughness.

Printed patterns on polyester fabrics

The polyester fabrics with and without modification were printed with the same rainbow pattern of seven different colors to investigate the effect of the modification in Fig. 11.

In Fig. 11(a), the sharpness and the color yield of the untreated fabric were very poor. For example, the cyan of the rainbow could even not be easily identified between the blue color and the green color. The red pigment particles penetrated to the other areas and the orange pattern was obscured. The lightness of the yellow color was seriously damaged, and the width of the yellow region was declined. The violet pattern on the marginal area was partly covered up. The shapes of the black patterns could not be recognized. On the contrast, the β-CD modified fabric in Fig. 11(b) had excellent edge. The color yield was also deeper than that of the untreated polyester fabric. The rainbow with seven different colors of red, orange, yellow, green, cyan, blue and violet was clearly presented on the fabric surface. The shapes of the black patterns contained round, quincunx, heart-shaped and so on.

Conclusion

This investigation presented the possibility of surface modification on polyester to obtain the high sharpness and the deep color yield for inkjet printing using β-cyclodextrin and citric acid. The enhancement of 47% in the *K/S* value and the reductions of 77% and 62% in the line width and image area of the patterns were achieved once the sample was dipped into the solution containing 100g/L β-cyclodextrin, 100g/L citric acid and 30g/L SHP, respectively. SEM and AFM analysis confirmed that polyCTR-CD was absorbed and indicated that the capillary effect was decreased and the roughness was increased. The inkjet printing pattern on the modified polyester fabric was clearer in comparison with that on the untreated sample. β-cyclodextrin modified fabric exhibited the high sharpness and the deep color yield.

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