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Preparation of Structural Colors on White Polyester Fabrics without Adding Any Black Additive

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Abstract: The colorful, unfading, and eco-friendly photonic crystal structural colors have attracted wide interest from textile researchers. However, to minimize the interference of incoherently scattered light, the preparation of the structural color fabric is still based on the black fabric, which seriously restricts the practical application of the structural colors in textile dyeing and finishing. This paper presented a straightforward strategy to construct structural colors on the fabrics by assembling polysulfide microspheres (PSMs) with self-absorbing light properties. The monodisperse PSMs were successfully synthesized through a polycondensation process between Na_2S_2 and 1,2,3-trichloropropane, whose particle size can be controlled by adjusting the volume ratio of ethanol-water. Four different structural colors were successfully obtained on white polyester fabric by varying the diameter of the PSMs arranged on the fabric surface in a face-centered cubic structure. Notably, the designed structural color fabrics had a low angle dependence, which was more meaningful for the textile color measuring and matching system.

Keywords: Structural color, Photonic crystals, White polyester fabric, Low angle dependence, Polysulfide microspheres

Introduction

Photonic crystals (PCs) are composed of materials with different dielectric constants, which have a photonic band gap (PBG) analogous to the electronic band gap of a semiconductor [1-4]. When PBG falls into the visible light range between 380 nm and 780 nm, the visible light at a specific wavelength is prohibited from propagation, and a structural color can be generated through the Bragg scattering [5,6]. Unlike traditional chemical dyes or pigments, the structural colors are produced by the physical interaction of unique microstructure and light, which have the advantages of high saturation, high brightness, and not fading. As a result, it has received considerable attention from researchers in the textile industry [7-9].

Consequently, constructing photonic crystals on fabric by self-assembling colloidal microspheres is considered as a promising method to fabricate structural color fabrics, such as gravity deposition [6,10], vertical deposition [11], electrostatic self-assembly [12,13], and spraying [14]. However, the interference of background light and incoherently scattered light from the white substrate material results in inconspicuous and dull structural colors, so how to effectively eliminate the incoherent scattering light is also a subject to explore.

Black materials have good light absorption properties, which can be introduced into photonic crystals to enhance the structural color saturation [15]. For instance, some researchers have obtained structural colors on black fabrics of different materials (e.g. cotton [16,17], polyester [18], silk [19], etc.). Unfortunately, black fabrics are usually obtained by dyeing processes requiring chemical dyes or pigments, which seriously limit the practical application of structural colors in textile dyeing and finishing. Other researchers have proposed a method doping black materials (e.g., carbon black [20], graphene [21], cuttlefish ink [15], etc.) into photonic crystals to produce vivid structural colors on white substrates, which makes it possible to obtain structural colors in one step on the white fabrics. However, the introduction of black materials could destroy the order degree of photonic crystals, and obtain disordered photonic crystals, leading to the reduction of structural color saturation. Therefore, constructing ordered photonic crystals with brilliant structural colors directly on white fabrics remains a great challenge.

Due to the large molar refractive index of sulfur atoms, polymer microspheres with a high refractive index and good light absorption can be synthesized by introducing sulfur atoms into the molecular chain through rational design, which is an ideal material for constructing photonic crystals [22,23]. There have been numerous studies on the construction of photonic crystals using inorganic or organic polymer microspheres as basic units. Among them, polymer microspheres synthesized by the polycondensation reaction, which refers to the reaction in which one or more monomers are condensed with each other to produce a polymer, have become a new material for preparing structural color [24,25]. Herein, without the introduction of black materials, we can obtain low angle dependence structural colors by self-assembling polysulfide microspheres with self-absorbing properties on the white polyester fabric. This simple strategy to obtain structural colors directly on white fabrics

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Experimental

Reagents and Materials

1,2,3-trichloropropane (TCP) was purchased from Macklin. Pluronic-F127 was purchased from Sigma Aldrich. Absolute ethanol, sublimed sulfur, and sodium hydroxide were purchased from Tianjin Beilian Fine Chemical Co., Ltd. Deionized water was used in all experiments. White plain polyester fabrics (81.6 g/m^2) were purchased from a local fabric market.

Synthesis of Monodisperse PSMs

Batches of monodisperse PSMs were prepared by the method proposed in a previous report [26]. First of all, 10 g NaOH was dissolved in 250 m*l* deionized water. When the mixture was heated to 95 °C, 8 g sublimed sulfur was added to the mixture, which was vigorously stirred for 1 h, then reddish-brown Na_2S_2 precursor solutions were obtained. Secondly, 25 m*l* of the Na_2S_2 solution was mixed with 125 m*l* of the mixed solution containing deionized water and absolute ethanol in a three-necked. Then, 0.25 g Pluronic-F 127 was added into the mixed solution when the temperature was heated to 70 °C. Next, 0.625 g TCP was added into the mixture with vigorous stirring, and PSMs suspension was obtained after the reaction system was maintained at 70 °C for 6 h. Finally, PSMs were obtained by centrifugation and redispersion three times with deionized water, followed by drying in an oven at 70 °C for 12 h. The size of the PSMs was highly dependent on the different volume ratios of water and absolute ethanol (see Table 1).

Fabrication of Photonic Crystals on White Polyester Fabrics by Vertical Deposition

In this study, PSMs were used to build photonic crystals by a simple vertical deposition. Firstly, PSMs were diluted to 1.0 wt % suspension in deionized water by ultrasonic dispersion, and a piece of clean white polyester fabric was vertically placed in a beaker that was filled with PSMs suspension. Secondly, the beaker with white polyester fabrics was located in an oven at a constant temperature of 60 °C. Finally, with water evaporation, the PSMs were gradually assembled on the fabric driven by capillary force, and the structural color fabric was obtained. The structural color fabrics prepared by self-assembly of PSMs 1-4 as basic units were denoted as Fabrics 1-4.

Characterization

The particle size, particle dispersion index (PDI), and zeta potential of PSMs were determined by a Malvern laser particle sizes analyzer (Nano-ZS90, Malvern, England). Fourier transform infrared (FTIR) spectra of PSMs were measured by Fourier transform infrared spectroscopy (Bruker, Equinox 55, Germany). The morphologies of white polyester fabrics, PSMs, and photonic crystals on polyester fabrics were observed by field emission scanning electron microscopy (FESEM, SU 8010, Japan). The $L^*a^*b^*$ values and reflectance spectra of structural colors on the white polyester fabric were measured by a spectrophotometer (Ultra Scan PRO, Hunter Lab, America) with colorimetric illuminant of D65 and standard colorimetric observer of 10 °. The structural colors of photonic crystals on white polyester fabrics were observed by a digital camera (DSC-HX30, Sony, Japan) and a 3D video microscope (KH-7700, HIROX, Japan).

Results and Discussion

Synthesis of Monodisperse PSMs

In this study, PSMs were synthesized via a condensation crosslinking reaction between Na_2S_2 and TCP in an alcohol-water medium, and the reaction route was presented in Figure 1. At the beginning of the reaction, Na_2S_2 and TCP underwent substitution reactions to generate a soluble oligomer, then the oligomer gradually condensed and crosslinked to form a primary micronucleus. As the reaction continues, the primary micronucleus gradually grew larger and eventually formed the PSMs.

FTIR was implemented to clarify the chemical composition of PSMs, which confirmed that sodium disulfide and TCP underwent substitution reactions. Figure 2 showed the FTIR spectra of the PSMs and TCP. The FTIR spectrum of TCP showed some characteristic peaks: The asymmetric stretching vibration and asymmetric bending vibration of -CH_2 appear at 2994 cm^{-1} and 1467 cm^{-1}, respectively. The peak at 753 cm^{-1} represents C-Cl stretching vibration. After the condensation reaction with Na_2S_2, the C-Cl stretching vibration peak basically disappeared, the stretching vibration of C-S appeared at 1143 cm^{-1}, and the absorption peaks at 3445 and 1660 cm^{-1} were due to the anti-symmetric

Table 1. Different parameters of PSMs spheres synthesized

Sample	Na_2S_2 (m*l*)	Water (m*l*)	Ethanol (m*l*)	Pluronic-F 127 (g)	TCP (g)	Time (h)	Temperature (°C)
PSMs 1	25	95	30	0.25	0.625	6	70
PSMs 2	25	90	35	0.25	0.625	6	70
PSMs 3	25	85	40	0.25	0.625	6	70
PSMs 4	25	80	45	0.25	0.625	6	70

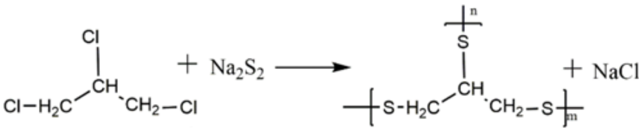

Figure 1. Main synthetic routes of PSMs.

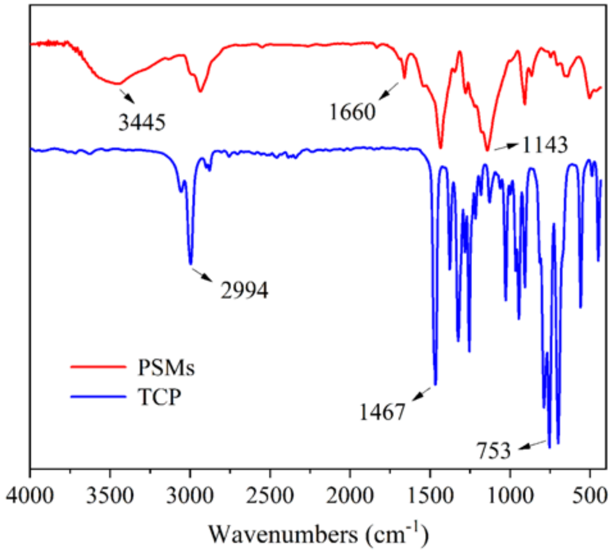

Figure 2. FT-IR spectra of the PSMs and TCP.

stretching vibration and bending vibration of -OH [27]. This proved that PSMs were successfully synthesized.

Under the same reaction conditions, the PSMs with average particle sizes of 221 nm, 238 nm, 253 nm, and 270 nm were obtained by changing the volume ratio of absolute ethanol to water (as shown in Table 1). The morphology and monodispersity of colloidal microspheres were essential factors in determining the quality of photonic crystals. The FESEM images of PSMs with a diameter of 260 nm were presented in Figure 3(a). It can be observed that the PSMs were spherical in shape, uniform in size, and separated from each other.

Table 2. Average diameter, PDI, and zeta potential values of PSMs

Sample	Diameter (nm)	PDI	Zeta potential (mV)
PSMs 1	221	0.056	-28
PSMs 2	238	0.049	-27.6
PSMs 3	253	0.075	-27.4
PSMs 4	270	0.064	-27.8

To understand the monodispersity of PSMs, particle size distribution and PDI of PSMs were tested. As shown in Figure 3(b) and Table 2, it can be seen that the particle size distribution curves of PSMs were narrow and sharp, and the PDI of PSMs 1-4 was all less than 0.08, which proved that PSMs had excellent monodispersity [28]. In addition, zeta potential is also a critical factor affecting the self-assembly of colloidal microspheres into ordered photonic crystals. It can be seen from Table 2 that PSMs have a large number of negative charges that can provide sufficient electrostatic repulsion, which is very important for the stability of PSMs suspensions. Therefore, PSMs are suitable for assembly into photonic crystals.

Fabrication of PSMs Photonic Crystals on White Polyester Fabrics by Vertical Deposition

Vertical deposition techniques with simple operation and high-efficiency advantages have been widely used in the preparation of photonic crystals [29]. In addition to hard and dense materials, textiles with many pores and undulations are also often used as a substrate. From the FESEM image of polyester fabric in Figure 4(a), the regular arrangement of one warp yarn floating across another two weft yarns was clearly observed, suggesting that the white polyester fabric had a typical 2/1 twill weave strucure. Moreover, it can also be seen that the polyester fiber had a smooth and flat surface. However, after vertical deposition self-assembly of PSMs,

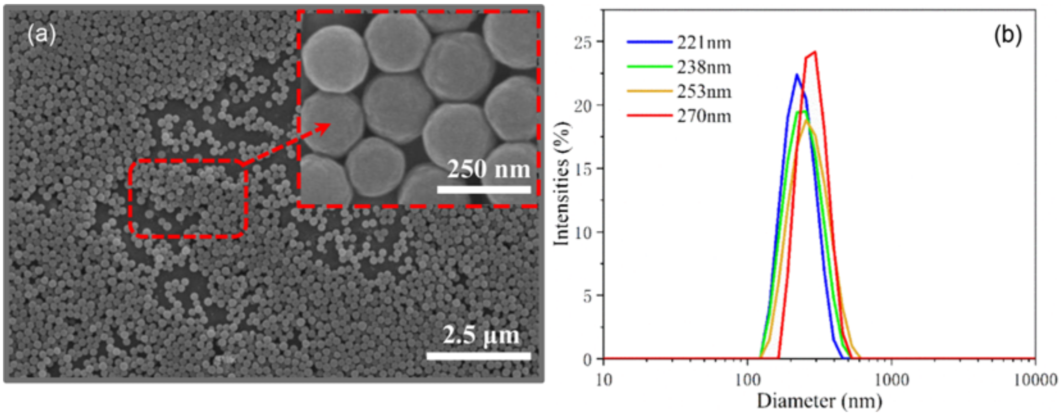

Figure 3. (a) FESEM images of PSMs with a diameter of 260 nm, inset: amplified FESEM image (b) particle size distribution diagrams of PSMs.

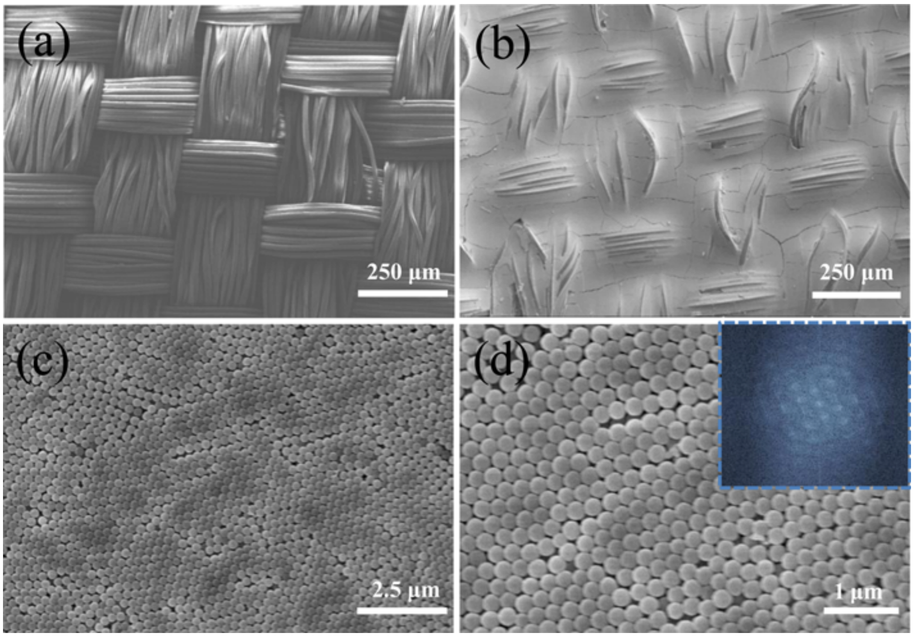

Figure 4. (a) FESEM images of the white twill polyester fabric and (b) FESEM image of the coated fabric after vertical deposition. (c, d) FESEM image of the resultant PSMs photonic crystals on fabric and an enlarged area of view from (c). Inset: the 2D-FFT image of the corresponding FESEM image.

the gaps between the adjacent yarns were filled by PSMs aggregates. Most of the yarns were covered by a film with cracks, and a small portion of the yarn outline can still be observed as shown in the FESEM image of Figure 4(b), indicating that the structural color coating was relatively thin.

To observe the arrangement state of the PSMs in the photonic crystals, Figure 4(c) showed a local position in Figure 4(b) by adjusting the magnification of the FESEM. The results demonstrated that the film with cracks was composed of numerous PSMs, and the PSMs were built into an ordered arrangement. Upon further enlargement to obtain Figure 4(d), it can be seen that each PSMs was surrounded by six PSMs, which implied that the PSMs were assembled into ordered face-centered cubic (FCC) structure by vertical deposition [30]. In addition, the degree of order of the colloidal microspheres in photonic crystals can also be characterized by two-dimensional fast Fourier transform (2D-FFT) analysis, which was calculated by Matlab software [31]. As can be seen from the inset of Figure 4(d), the corresponding 2D-FFT pattern with hexagonal symmetry could be seen, indicating that PSMs self-assembled by vertical deposition tended to arrange in a close-packed and formed a long-ordered FCC structure.

Optical Properties of the PSMs Photonic Crystals on White Polyester Fabrics

To visually display the structural colors of PSMs photonic crystals on the white polyester fabric, the prepared structural color fabrics were measured by using a digital camera, 3D video microscope, and spectrophotometer, respectively. Figure 5 showed the digital images of prepared structural color fabrics with photonic crystals assembled by PSMs with diameters of 221, 238, 253, and 270 nm, respectively. As can be seen from Figure 5, PSMs photonic crystals self-assembled by four different particle sizes showed different structural colors on the surface of polyester fabrics, respectively, in blue, green, yellow, and red. It was worth noticed that structural colors were obtained on the white polyester fabric without adding any black substance. This is due to the self-absorbing property of PSMs leading to the effective absorption of incoherently scattered light in photonic crystals.

Reflection spectrum was an essential means to objectively characterize the hue of structural color fabrics, and the hue of structural color fabrics can be judged according to the peak wavelength. Figure 6 showed the reflection spectra of structural colors on the fabrics. It can be clearly seen that the reflection spectra of Fabrics 1-4 showed reflection peaks at 465, 510, 590, and 650 nm, respectively, indicating that the structural color fabrics with photonic crystals assembled from different PSMs were blue, green, yellow, and red, respectively.

Bragg's law was a momentous theory to explain the phenomenon of structural color. When the reflection peak was located in the visible region, the vivid structural color could be observed by the naked eye. The peak wavelength of the photonic crystal structural color can be expressed using

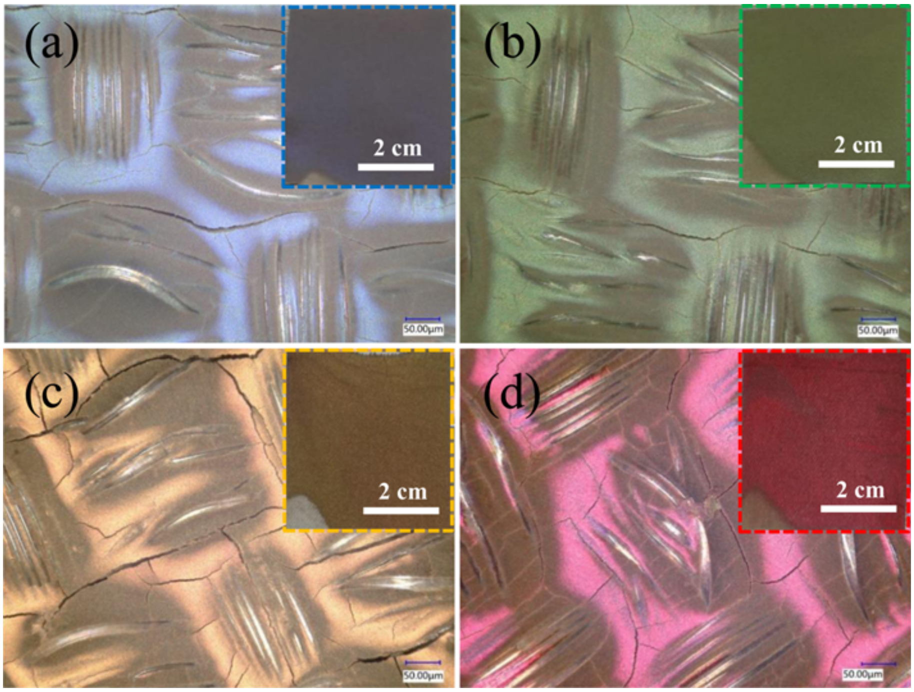

Figure 5. (a-d) Different structural colors on white polyester fabrics taken by a digital camera (inset) and 3D video microscope. The diameters of PSMs were 221, 238, 253, and 270 nm, respectively.

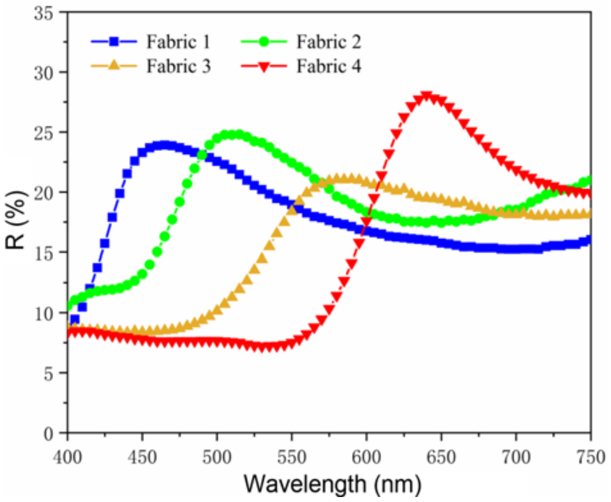

Figure 6. Reflection spectra of Fabrics 1-4.

the following equation (1) [32]:

$$\lambda_{\max} = \frac{2d_{hkl}}{m}(n_{avg}^2 - \sin^2\theta)^{1/2} \qquad (1)$$

where m is the order of Bragg diffraction, λ_{max} is the peak wavelength of the reflectance spectrum, n_{avg} is the effective refractive index of the system, d_{hkl} is the spacing distance between the diffracting planes, and θ is the angle between the incident light and the surface normal of the sample (same as the viewing angle).

Based on equation (1), the optical photographs, and reflectance spectra of the structural color fabrics, the intrinsic relationship between the hue of structural color fabric and PSMs photonic crystal was investigated. When the diameter of PSMs was 221, 238, 253, and 270 nm, the blue, green, yellow, and red structural color fabrics were obtained, and the peak wavelengths of the corresponding structural color in the reflection spectrum were 465, 510, 590, and 650 nm, respectively. The above results indicated that structural color fabrics of different colors could be prepared by adjusting the particle size of the PSMs, and the hue and peak wavelength of the structural color fitted well, which was consistent with Bragg's law. Specifically, the increase in the diameter of colloidal microspheres causes an increase in the peak wavelength of the structural color reflection spectrum, resulting in a red-shift of the hue of the structural color. Therefore, this will provide theoretical guidance for the design of structural colors.

To more accurately describe the color of the structural color fabric, referring to the 1976 norm of the Commission International de I'Eclairage (CIE) $L^*a^*b^*$ color scale, the $L^*a^*b^*$ values of the structural color fabric were tested by a spectrophotometer. L^* attributes the lightness. The larger the value, the brighter the color is. a^* axis points the red (positive) to green (negative), while b^* axis points the yellow (positive) to blue (negative).

The $L^*a^*b^*$ values of white polyester fabric and Fabrics 1-

Table 3. CIE $L^*a^*b^*$ values of white polyester fabric and Fabrics 1-4

Sample	PSMs/nm	L^*	a^*	Δa^*	b^*	Δb^*
White polyester fabric	-	97.14	2.15	-	-8.77	-
Fabric 1	221	54.24	4.01	-6.42	-1.53	9.76
Fabric 2	238	47.09	-2.41	8.05	8.23	16.22
Fabric 3	253	45.32	5.64	17.3	24.45	-14.41
Fabric 4	270	49.49	22.94		10.04	

4 were presented in Table 3. The *Δ* represented the difference between the parameters of the two samples. Combined with the physical meaning of a^* and b^*, the parameter Δa^* meant the difference between the structural color in red and green [33]. The Δa^* value of Fabrics 1-2 was negative, which indicated that the structural color of Fabrics 2 became greener, and the Δa^* values of Fabrics 2-4 were all positive, which suggested that the structural color of fabrics 2-4 became redder. Similarly, the parameter Δb^* represented the difference between the structural color in yellow and blue. Positive Δb^* values for Fabrics 1-3 indicated that the structural color of Fabrics 1-3 became yellower, and negative Δb^* value for Fabrics 3-4 suggested that the structural color of Fabric 4 became bluer. Meanwhile, the CIE $L^*a^*b^*$ values of Fabrics 1-4 were significantly different, which indicated that the colors of the samples were located in various positions in the color space, which represented that they have different hues, respectively. A regular phenomenon that can be observed was that the structural color of Fabrics 1-4 became red-shifted with PSMs diameter, which again clarified the intrinsic link between particle size and structural color.

Angle dependence refers to the phenomenon that the color of an object changes with the viewing angle, namely the iridescence, which is a vital feature of order photonic crystals. In this study, ordered FCC structures were constructed on white polyester fabric by vertical deposition self-assembly. However, the prepared structural color fabric had a low angle dependence and was an exciting phenomenon. Take the red fabric as an example, photographs of different viewing angles of the Fabric 4 were shown in Figure 7. Typically, the viewing angle perpendicular to the direction of the sample is defined as 0 °, and the viewing angle parallel to the direction of the sample is defined as 90 °. As shown in equation (1), when other parameters remain constant, the peak wavelength gradually becomes smaller as the viewing angle increases. That was, the photonic crystal structural color gradually blue-shifts as the viewing angle increases.

The PSMs photonic crystal structural color showed a weak trend from red to orange as the viewing angle changed from 0 ° to 60 °. It was worth emphasizing that, unlike conventional photonic crystals with an ordered FCC structure consisting of P(St-MAA) [30], SiO_2 [34], and PS [35], the photonic crystals assembled orderly by PSMs displayed low angle dependence structural colors. This phenomenon was mainly attributed to the properties of PSMs itself. The inset of Figure 3(a) showed that PSMs did not possess a smooth surface morphology like SiO_2 or PS microspheres, which confirmed that PSMs had a rough surface. It was the rough surface that scattered the incident light to a certain extent, which led to the low angle dependence of the PSMs photonic crystal structural color [26,36].

Conclusion

In this paper, a low angle dependence structural color could be prepared by assembling monodisperse PSMs on the surface of fabrics by a simple vertical deposition self-assembly method. Four different particle sizes of PSMs were prepared by varying the ethanol/water volume ratio during the reaction, and the PSMs were arranged in an ordered FCC structure on the surface of the white polyester fabrics. Subsequently, structural colors are obtained directly on the white fabric due to the self-absorbing light properties of

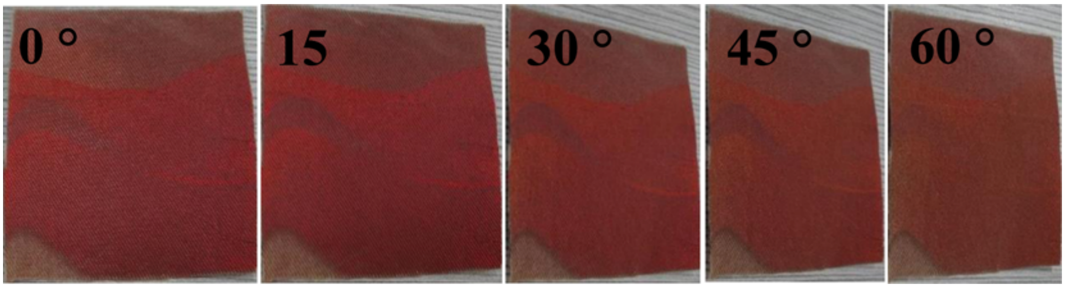

Figure 7. Digital photographs of Fabric 4 at different viewing angles.

PSMs. Varying the PSMs particle size was a crucial way to obtain different color structural color fabrics, and as the PSMs particle size increased, the color of structural color fabrics was red-shift. More importantly, the rough surface of the microspheres led to a low angle dependence of the PSMs photonic crystal structural color, opening a new chapter for the application of these convenient structural color construction materials in the textile field. But, the stability of photonic crystals needs to be considered and improved if the prepared structural color fabrics are to be used in practice, for example, introducing waterborne polyurethane [7], polyacrylate [37], and polyvinyl alcohol [19], etc.

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