Obtaining chromogenic structured yarns from a mixture of carbon and polyester fibers

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Abstract. In this article, considering the problem of the difficulty of dyeing carbon fiber, it is proposed to use structural dyeing to achieve the dyeing of carbon fiber/polyester blended yarn. Using poly (styrene - methacrylic acid) colloidal microspheres as structural units, a photonic crystal color-forming structure was obtained using a carbon fiber/polyester blend yarn coating method. Polydimethylsiloxane (PDMS) encapsulation technology was used to analyze the effects of coating fluid mass fraction and self-assembly temperature on the structural color, to study the assembly process of colloidal microspheres into photonic crystals on the surface of the yarn, and to improve structural colored yarns. Photonic crystal structural color is the visual result of light diffraction by the crystal, providing high saturation, high brightness, and radiant effect. In this paper, the construction of photonic crystal coloring structures on the surface of carbon fiber and polyester substrates is proposed, and an efficient method of constructing photonic crystals and an effective method of increasing the stability of photonic crystals is studied. The research results provide strategic support for the color and functionality of carbon and polyester fibers.

1 Introduction

Carbon fiber has excellent physical and chemical properties such as corrosion resistance, high-temperature resistance, high strength, lightweight, high modulus, etc., and is widely used in aerospace, infrastructure, military equipment, sports, and other fields [1]. However, the graphene-like structure on the surface of carbon fiber makes its surface chemically inert and difficult to color with conventional chemical dyes. With the improvement of people's living standards, the same color carbon fiber cannot meet the needs of consumers. Currently, most of the colored carbon fibers on the market are obtained by modifying the surface of the carbon fiber and then dyeing it with chemical dyes. Although this method can obtain colored carbon fibers, it requires the prior modification of the carbon fiber matrix, which damages the excellent properties of carbon fiber to a certain extent and complicates...
the preparation process. It is necessary to search for new technologies suitable for dyeing carbon fibers.

According to the production mechanism and color source, it can be divided into two categories: chemical chromogenesis and structural chromogenesis. Unlike chemical coloring, which uses dyes as the primary coloring agents to produce colors, structural coloring does not need to rely on the effects of chemical dyes. It is based on the diffraction, interference, dispersion of light into current colors, and other effects of the specific microstructure of the object. [2]. In nature, the colors of butterfly wings, clam shells, peacock feathers, etc. are typical structural colors. In general, structural colors have special visual effects such as high saturation, high brightness, and radiant effects [3]. It is an ecological and clean coloring method, and it is an important complementary method to the current traditional paint pigment coloring. A photonic crystal [4] is a type of dielectric structure with photonic band gaps periodically located in air gaps. Due to its bandgap properties, the diffraction of light at the crystal surface can be used to create a variety of structural color effects. Currently, colored structural textiles made using photonic crystal color-generating structures in textiles have attracted the attention of researchers [5].

Research on the construction of photonic crystal color-generating structures in carbon fiber products is widespread. They assembled polystyrene (PS) microspheres on the surface of carbon fibers by electrophoretic coating to obtain structured colored carbon fibers [6]. A nanoparticle coating method was used to attach a layer of titanium dioxide (TiO₂) film to the surface of carbon fiber fabrics and successfully prepared carbon fiber fabrics with bright structural colors [7]. These studies have contributed to the research progress of creating photonic crystal structure colors in carbon fiber products and provided new strategies for the eco-coloring of carbon fibers [8]. Most researchers have focused on the color creation of photonic crystal structures of carbon fibers or carbon fiber-like fabrics, and there are several studies on the construction of photonic crystal color structures using carbon fiber yarns as a substrate [9]. First, because pure carbon fiber yarn is difficult and expensive to make, it is hardly available on the market; secondly, when carbon fiber and other fibers become a mixed yarn after mixing, carding, braiding, twisting, and other processes, its surface porosity is much larger than that of carbon fiber and carbon fiber fabric, which will undoubtedly produce regular photonic crystal color increases the difficulty of building a structure [10, 17, 18]. As the secondary structure that makes up the fabric, yarn largely determines the performance of the fabric. If we first get the structural color carbon fiber yarn and then weave the structural color carbon fiber yarn through the dyed weaving process, we can not only directly obtain the structural color carbon fiber fabric, but also, we can enrich the carbon fiber through different colored yarns. The color of the fabric is improved, thereby improving the quality of the carbon fiber fabric.

In this paper, poly (styrene-methacrylic acid) (P(St-MAA)) colloidal microspheres are used as the core structure to construct photonic crystals, and deep-coating them to obtain carbon fiber/polyester blended yarns.

Chromogenic structure of P(St-MAA) photonic crystal. The effect of coating fluid mass fraction and self-assembly temperature on structural chromogenic yarns was studied, the process of microsphere assembly on the yarn surface was observed [11] and the iridescence effect of structured chromogenic yarns was quantitatively analyzed. Structural-colored fabrics are woven from a large number of structurally colored yarns [12], which provides a new idea for the preparation of colored carbon fiber products.

2 Experimental part

Materials: styrene (St), methacrylic acid (MAA), ammonium persulfate (APS), analytical grade, Shanghai McLean Biochemical Technology Co., Ltd.; nitrogen (purity 99.99%),
Hangzhou Hangyang Co., Ltd.; polydimethylethylene-based siloxane (PDMS) and related curing agent, Aladdin Reagent (Shanghai) Co., Ltd.; deionized water (conductivity 18 mkm/cm), for laboratory use; carbon fiber/polyester blend yarn (black 40 tex), Yixing Dongfeng Textile Co., Ltd.

**Laboratory equipment:** DHJ type electrothermal constant temperature (hot air flow) drying oven (Shanghai Jinghong Experimental Equipment Co., Ltd.); RE-2000B rotary evaporator (Shanghai Yarong Biochemical Equipment Factory); ALTRA55 surface emission scanning electron microscope (Zeiss, Germany) KH-700 three-dimensional video microscope (Japan Haoshi Co., Ltd.); Lambda 900 UV-visible spectrophotometer (Perkin Elmer, USA); MA98 multi-angle spectrophotometer (X-Rite, USA).

**P(St-MAA) colloidal microsphere coating liquid preparation of P(St-MAA) colloidal microspheres**

It was prepared by non-alkaline emulsion polymerization. Fig. 1 Scheme of preparation of P(St-MAA) colloidal microsphere emulsion. After mixing 120 g of deionized water, 1.4 g of MAA, and 14 g of St, it was poured into a three-necked flask equipped with a mechanical stirrer (stirring speed of 320 r/min), a reflux condenser, and a nitrogen inlet tube. Then it is placed in a water bath when the temperature of the water bath reaches 75 °C, an aqueous solution of APS (0.1 g of APS dissolved in 20 g of deionized water) is added, and after 8 hours of reaction, it is cooled at room temperature. The entire polymerization process is always carried out in a nitrogen atmosphere. P(St-MAA) colloidal microspheres with different particle sizes are prepared by changing the monomer dosage, mixing speed, polymerization temperature, initiator dosage, and other parameters.

![Fig. 1. Schematic of preparation of colloidal microsphere and structural colored carbon/polyester blended yarn.](image)

The prepared P(St-MAA) colloidal microsphere emulsion is poured into a one-neck flask, then it is placed in an oil bath at a temperature of 70 °C, and the P(St-MAA) colloidal microsphere emulsion is evaporated under negative pressure using a rotary evaporator, will be held. In the process of increasing the solid content, the rotation speed is set to 20 r/min, and after the mixture is evaporated for a certain time, P (St-MAA) colloidal microsphere collection fluids with different solid content are obtained, carbon fiber/photons with a crystal chromogenic structure are produced in polyester mixed yarns. The drying method was used to determine the solid content of P(St-MAA) colloidal microsphere coating fluid, and the solid content was used as a measure of its concentration.

**Preparation of P(St-MAA) photonic crystal chromogenic structure on yarn**

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Figure 1 is a scheme for the preparation of structural colored carbon fiber/polyester blended yarn. It can be seen that black carbon fiber/polyester blended yarn is the main material and P(St-MAA) is the main structural building unit of colloidal microspheres, centrifugal force method (pulling method: parallel pulling, pulling speed 2cm/s P(St-MAA) colloidal crystal color structure is built on black carbon fiber/polyester blended yarn. In the preparation process, the carbon fiber/polyester blended yarn is first immersed in the collection liquid for 5 seconds, and slowly pulled out and placed. The drying is done in a high-temperature oven and the water vapor after spinning, a carbon fiber/polyester blend yarn with a structural color effect is obtained.

**PDMS encapsulation of structured chromogenic yarns**

To improve the color fastness of the structured colored carbon fiber/polyester blended yarn, PDMS was used to encapsulate the structured colored carbon fiber/polyester blended yarn. First, mix PDMS and a suitable curing agent in a mass ratio of 8:1, then use a sponge to take 4 grams of the mixture and apply it uniformly to the structural color carbon fiber/polyester mixed yarn, finally, it is placed in a glass container and vacuum sealed oven dried at 70 °C for 3 hours, PDMS can be used to encapsulate structured colored carbon fiber/polyester blended yarn.

**Making fabric from structured colored yarn**

Three 5m-long PDMS-encapsulated structured colored carbon fiber/polyester blended yarns were used to obtain the knitted product, and then the yarns were knitted with a crochet needle to obtain the knitted fabric.

**Testing and characterization**

**Morphological observation of carbon fiber/polyester blend yarn**

Surface emission scanning electron microscopy (FESEM) was used to observe the yarn morphology before and after self-assembly of P(St-MAA) colloidal microspheres to form a photonic crystalline structure.

**Characterization of structural color effects of yarn and fabric**

A three-dimensional video microscope is used to observe the brightness and uniformity of the color of the photonic crystal structure on the surface of yarns and fabrics. A UV-visible spectrophotometer was used to examine structural color reflectance. Test conditions: 10 fields of view, D65 light source, wavelength scan range 400-800 nm. A multi-angle spectrophotometer was used to observe the iridescence phenomenon of the structural color of the yarn surface under different light sources and observation angles.

**Colorfastness test of structural colored yarns**

To test the color fastness of the structural color on the surface of the structural colored carbon fiber/polyester blended yarns, the structural colored carbon fiber/polyester blended yarns were placed in a rotor cup filled with water, and then stirred with a magnetic stirrer for 15 minutes in a simulated washing machine condition during the experiment. (rotational speed: 150 rpm) was placed. In the abrasion resistance test, the yarn surface finger wipe test was used. During the experiment, a structured colored carbon fiber/polyester blend yarn was repeatedly rubbed 50 times with the thumb and index finger. The friction pressure of the finger is 100 N, and the area is 1 cm². A three-dimensional video microscope was used to observe the color difference of the samples before and after the washing and rubbing experiments.

**3 Results and discussion**

**The influence of the mass fraction of the collection fluid on the structural color effect**

Different mass fractions of P(St-MAA) colloidal microsphere assembly solutions contain different amounts of colloidal microspheres, which significantly affect the
regularity of the photonic crystal and the color of the corresponding structure [13]. Figure 2 shows a three-dimensional video microscope image of a carbon fiber/polyester blended yarn and a reflective contour map before and after self-assembly with different mass fractions. It can be seen that before self-assembly, the yarn is black and does not show structural color, as the mass fraction of colloidal microsphere assembly solution increases from 20% to 70%, the color of the structure color on the yarn is brighter, and its corresponding reflectivity is 70% of the mass fraction of the collection fluid, the structural color of the yarn is the brightest and the reflectivity is also the highest. However, as the collection liquid mass fraction (80%) continued to increase, the yarn flowed slightly, the structural color effect deteriorated, and the corresponding reflection peak decreased. In conclusion, when the mass fraction of P(St-MAA) colloidal microsphere assembly solution is 70%, a brighter structural color can be obtained in the carbon fiber/polyester blend yarn.

![Figure 2](image)

**Fig. 2.** Three-dimensional video microscope images and carbon fiber/polyester blended yarns before and after self-assembly with different mass fractions. (a) original sample; (b) 20%; (c) 30%; (d) 40%; (e) 50%; (f) 60%; (g) 70%; (h) 80%.

Figure 3 shows electron microscope photographs of carbon fiber/polyester blended yarns after self-assembly with different mass fractions of assembly solutions. It can be seen that when the mass fraction of the collection liquid is 30%, the P(St-MAA) colloidal microspheres are dispersed and deposited on the surface of the yarn. When the mass fraction of the collection liquid on the surface of the yarn is 70%, the P (St-MAA) colloidal microspheres are evenly distributed and regularly located on the surface of the yarn, when the mass fraction of the collection liquid is 80%, the P (St-MAA) colloidal microspheres partially irregularly located on the surface of the yarn. These phenomena show that when the mass fraction (70%) of P(St-MAA) colloidal microsphere assembly solution, P(St-MAA) colloidal microspheres can form a regular and ordered photonic crystal structure on the surface of the yarn.

![Figure 3](image)

**Fig. 3.** Electron microscope photographs of carbon fiber/polyester blended yarn after self-assembly with different mass fractions of colloidal microsphere solutions.
The above results show that the effect of the structural color is directly related to the order of the photonic crystal formed by P(St-MAA) colloidal microspheres. If the mass fraction is too low, the number of P(St-MAA) colloidal microspheres in the collection liquid is too small, which is not enough to arrange the photonic crystals evenly on the yarn surface, resulting in poor structural color effects, and mass fraction if it is moderate, the number of microspheres in the collection liquid is enough to form regular and orderly photonic crystals on the surface of the yarn, and the color of the structure of the yarn is bright, and if the mass fraction is very high, the photonic crystals created by the colloidal microspheres in the collection liquid will cover the whole yarn evenly can cover, but due to the excessive number of colloidal microspheres, they collide with each other and make it impossible for some colloidal microspheres to self-assemble effectively. This affects the structural color effect.

In conclusion, when the mass fraction (70%) of the P(St-MAA) colloidal microsphere assembly solution is moderate, the P(St-MAA) colloidal microspheres can form regular and ordered photonic crystals on the yarn surface. This creates a bright structural color.

**Effect of self-assembly temperature on the structural color effect of yarn**

At different temperatures, P(St-MAA) colloidal microspheres have different states of assembly motion, which greatly affects the construction of regular and ordered photonic crystals and corresponding structural colors [14]. Figures 4 and 5 showed the three-dimensional video microscope images and the reflection peak images of the carbon fiber/polyester blended yarn at different self-assembly temperatures, respectively [15].

As can be seen from Figures 4 and 5, when the self-assembly temperature is 30 °C, the color of the yarn structure is uneven, the color is poor, and the corresponding reflection peak is also low; when the self-assembly temperature is 40, 60, 100, 130 °C, the structural color of the yarn is bright and uniform. The color of the structure does not change much between different self-assembly temperatures, and the peak reflectance remains at about 21%. However, when the self-assembly temperature continues to increase, i.e. at 150 °C, the color of the structure changes and deteriorates, and the peak value of the corresponding reflection also decreases.

![Fig. 4. Three-dimensional video and microscope photos of self-assembly of mixed yarns at different temperatures.](image-url)
In conclusion, if the self-assembly temperature is in a certain range (40-130 °C), bright structural colors can be obtained in the yarn. In consideration of low carbon and environmental protection, the preferred temperature is 40 °C. The phenomenon of the color difference in the yarn structure at very low self-assembly temperature is that when the temperature is too low, the free energy of the colloidal microspheres is low, and the capillary force is not enough to move the colloidal microspheres. When collecting and organizing colloidal microspheres, if the temperature is too high, the free energy of colloidal microspheres will be too high and they will have strong Brownian motion. When the solvent in the assembly solution is completely evaporated, some of the microspheres of the colloids are not arranged regularly according to the three-dimensional ordered structure.

**Analysis of the process of assembly of colloidal microspheres on a yarn**

Figure 6 shows the three-dimensional video microscope images of the structurally colored carbon fiber/polyester blend yarn at different times at an assembly temperature of 40 °C. It can be seen that the white milk-like collection liquid is first adsorbed on the surface of the yarn, and then as the solvent in the collection liquid continues to evaporate, the microspheres gradually accumulate on the surface of the yarn, forming photonic crystals, and the yarn structural color becomes more and more vivid and bright.

Figure 7 shows electron micrographs of P(St-MAA) colloidal microspheres on the yarn surface at different stages. Among them, Fig. 7(a) shows that P(St-MAA) colloidal microspheres begin to adhere to the surface of the yarn and that P(St-MAA) serves as the basis of the photonic crystal in Fig. 7(b). P(St-MAA) colloidal microspheres begin to adhere to the yarn surface. The colloidal microspheres begin to accumulate on the surface,
and the photonic crystal structure gradually becomes regular and ordered. Figure 7(c) shows the complete stage. The P(St-MAA) photonic crystal is formed on the surface of the carbon fiber yarn as a solid photonic layer. Colloidal microspheres prove that in the formation of photonic crystals on the yarn, they go through the stages of preparation, assembly on the surface of the yarn, and completion.

![Preparation stage](image1)
![Accumulation stage](image2)
![Complete stage](image3)

Fig. 7. Scanning electron microscope images of P(St-MAA) colloidal microspheres on the yarn surface at different stages.

It can be seen that P(St-MAA) colloidal microspheres in the collection fluid surround the yarn when the collection fluid first contacts the yarn. Gradually, due to the adsorption effect of the yarn, a part of the nearby colloidal microspheres is adsorbed to the core and shell of the yarn, and the assembly process (preparation stage) begins. If the wire in the preparatory stage at room temperature is placed parallel to the furnace, the colloidal microspheres in the collection liquid will begin to collect in the direction of the core and sheath, due to the effect of temperature solvent evaporation and the colloidal microspheres’ gravity. During the process, the collection layer at the initial stage is used as a base to start the collection, and some colloidal microspheres begin to accumulate into blocks and appear as liquid photonic crystal fragments. When the solvent completely evaporates, the movement of the colloidal microspheres stops, and the initial liquid photonic crystal fragments are regularly located on the surface of the yarn [16]. Finally, a solid photonic crystal coating and structural color yarn are formed on the yarn surface.

Analysis of the phenomenon of iridescence of structured colored yarns

The main feature of the structural color of photonic crystals, which differs from chemical colors, is the change of color with an angle, that is, the phenomenon of iridescence. This phenomenon can be explained by the Bragg diffraction equation:

$$\lambda_{\text{max}} = \frac{2d_{\text{hkl}}}{m} \sqrt{n_{\text{eff}}^2 - \sin^2 \theta}$$

Among them: $\lambda_{\text{max}}$ is the forbidden wavelength of the photon, nm; $m$ - diffraction order; $d_{\text{hkl}}$ is the distance between planes of a specific crystal plane, nm; $n_{\text{eff}}$ - effective refractive index, %; $\theta$ is the angle of the incident light, (°).

$m$, $d_{\text{hkl}}$, and $n_{\text{eff}}$ are constant values, and when the angle of incidence of light $\theta$ changes, $\lambda_{\text{max}}$ also changes. At this time, the color of the structural color is observed to change accordingly.

Analysis of color fastness of structured colored yarns

During the experiment, after making the structurally colored carbon fiber/polyester blend yarn, it was found that light scratches can cause the destruction of the photonic crystal coating on the surface of the yarn and seriously affect the color of the structurally colored yarn. PDMS was used in this paper to improve the color fastness of structured colored yarns. Figure 8 shows the reflectance curves of conventional transparent glass plates and structured chromogenic yarns before and after PDMS encapsulation. As can be seen from Figure 8(a), in the visible wavelength range, the reflectance curves of the conventional transparent glass coating and PDMS-coated transparent glass plates are almost
parallel, and the difference between the two at any wavelength always remained around 2%: figure 8(b), structural staining before and after PDMS encapsulation. The maximum reflectivity of the yarn varies by 2%. Based on the above information, it can be concluded that there is no significant change in the reflectance curve before and after PDMS encapsulation, whether it is a traditional transparent glass coating or a structured chromogenic yarn, which means that the brightness of the structured chromogenic yarn PDMS has little effect on the structural color.

Fig. 8. Reflectance curves of conventional transparent glass coatings and structured chromogenic yarns before and after PDMS encapsulation.

Figure 9 shows the washability test results of the construction-colored carbon fiber/polyester blended yarn. Figure 9(a) shows that after long-term water washing of the structural color yarn without coating, the photonic crystal on the surface almost completely falls off and the bright structure color is completely lost. Figure 9 (b), the structural color intensity of the PDMS-coated structural color yarn remains almost unchanged after the water washing test.

Fig. 9. Washing resistance test results of structurally colored carbon fiber/polyester blended yarn before and after PDMS encapsulation.

Figure 10 shows the results of the abrasion resistance test of a yarn made of a structurally colored carbon fiber/polyester blend. After repeated finger rubbing, the photonic crystal coating of the non-encapsulated structural color yarn was destroyed, leaving fine photonic crystal fragments. The structural color of the yarn is completely lost.
on the surface. The PDMS-coated structural colored yarn can retain the original structural color even after passing the abrasion test.

Fig. 10. Abrasion resistance test results of structural colored carbon fiber/polyester blended yarn before and after PDMS encapsulation.

Figure 11 shows the knitting process based on structured colored carbon fiber/polyester blend yarn. We can see colored fabric where the yarns are intertwined and woven from different colored yarns to make a fabric of the same color.

Fig. 11. Knitting process based on structured colored carbon fiber/polyester blended yarn.
4 Conclusion

This paper used a deep-coating method to fabricate a poly(styrene-methacrylic acid) (P(St-MAA)) photonic crystal structure on a carbon fiber/polyester blended yarn, and the mass fraction of the assembly liquid and the self-assembly The effect of temperature on the color of the structure was studied. The effect of P(St-MAA) colloidal microspheres on yarn was summarized, the iridescence effect of structural colored yarn was quantitatively analyzed, and the color fastness of structural colored yarn was improved by encapsulation technology, and the main conclusions are as follows.

- Regular photonic crystals with bright structural colors on the surface of the carbon fiber/polyester blended yarn when the mass fraction of P(St-MAA) colloidal microsphere assembly solution is 70% and the self-assembly temperature is 40 °C can be built.
- The process of self-assembly of P(St-MAA) colloidal microspheres on the yarn surface to form photonic crystals can be generalized in a simple preparation step, assembly, and finishing steps, and the corresponding structural color changes after the sequence is formed.
- The color of structural colored carbon fiber/polyester blended yarn varies at different viewing angles, showing a noticeable iridescent effect.
- Polydimethylsiloxane (PDMS) coated structural color carbon fiber/polyester blended yarn can maintain the original structural color effect even after washing and rubbing tests and has excellent color fastness.

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References


