Preparation of Acrylic Yarns with Durable Structural Colors Based on Stable Photonic Crystals

Wanbin Ma, Hao Liu, Wenyu He, Yunxiao Zhang, Yucheng Li, Yang Zhao, Chengcai Li, Lan Zhou, Jianzhong Shao, and Guojin Liu*

ABSTRACT: Structural coloration of photonic crystals (PCs) is considered an ecological and environmental way to achieve colorful textiles. However, constructing PCs with obvious structural colors on traditional flexible yarns is still a great challenge. As a secondary structure that forms textiles, compared with fibers and fabrics, the yarns are rougher, hindering the construction of regular PCs. In this work, the flexible acrylic yarns with vivid structural colors, named PC-based structural color yarns, were prepared by constructing regular PCs via assembling poly(styrene-butyl acrylate-methacrylate) (P(St-BA-MAA)) colloidal microspheres on yarns. Specifically, the properties of P(St-BA-MAA) colloidal microspheres were investigated. The PCs with better structural stability and obvious structural colors were prepared by presetting the acrylic adhesive layer on yarns. Moreover, the color durability and color regulation methods of prepared PC-based structural color yarns were evaluated and discussed. The results showed that the P(St-BA-MAA) colloidal microspheres exhibited even particle sizes, excellent monodispersity, and a typical hard core–soft shell structure. And the glass-transition temperature ($T_g$) of the microspheres was tested to be about 65.6 °C. The cationic acrylate regarded as a pretreatment agent could not only improve the combination between the PC layers and the yarns by acting as a “bridge” but also enhance the structural color effect by smoothing the yarn surface. The results showed that when the mass fraction of cationic acrylate was 3 wt %, the microspheres were beneficial to access regular PCs with obvious structural colors. The PCs with bright structural colors could be constructed on black acrylic yarns, and the colors of yarns were still bright after rubbing and washing tests, indicating that the prepared PC-based structural color yarns have good color fastness. Moreover, the color hue of PC-based structural color yarns could be regulated by adjusting the particle sizes and viewing angles. This study provides strategic support for the structural coloration of flexible materials.

1. INTRODUCTION

Textiles are a necessity of human life, which have functions such as covering the body, insulation, decoration, and other functions. Color is an important indicator to improve the quality of textiles. Textiles are often colored with chemical colorants including dyes or pigments in the traditional textile industry. Although chemical colorants have been used for many years, they still have a number of drawbacks including the fact that they are not recyclable, pollute the environment, and prone to color fading. With the development of industrial civilization, a number of new coloring technologies without dyes or pigments used to achieve the coloration of textiles are constantly emerging. Nowadays, the structural coloration of textiles, which is achieved by constructing a special organizational structure that can display scattering, interference, and diffraction, has attracted close attention from researchers. Photonic crystals (PCs), a kind of artificial crystal structure formed by two materials with different refractive indices in a cyclical arrangement, can produce gorgeous structural colors with the Bragg diffraction of the light. In recent years, the research of structural colors by constructing photonic crystals on textile substrates has become a hot topic. For example, Liu et al. used a vertical deposition method to assemble poly(styrene-methyl acrylic acid) (P(St-MAA)) microspheres with different particle sizes on polyester fabrics; the resulting fabrics could present a well-defined texture, flexible hand feel, and dual-sided color effect. Diao et al. built photonic crystals with polystyrene (PSt) microspheres on silk fabrics, and the resulting silk fabrics showed lifelike and dynamic structural colors. In addition to the fabrics, there are

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also many reports from home and abroad on the construction of PC structures on fibers directly. Liu et al.22 used silica \( \text{SiO}_2 \) microspheres as the units to fabricate PCs on nylon (PA) fibers via a thermal auxiliary evaporation method. The PA fibers presented dazzling structural colors, and the particle size of \( \text{SiO}_2 \) microspheres and structural colors were different. The above studies have promoted the development of textiles with structural colors.

As we all know, there are three types of textile substrates: fibers, yarns, and fabrics. Although many of the photonic crystals have been successfully fabricated on fiber and fabrics, there are a few studies on constructing photonic crystals on the yarns. Generally, the traditional flexible yarns are a kind of textiles obtained by one or more fibers in different spinning methods. As a secondary structure that forms textiles, the performance of the yarns is completely different from the fibers and fabrics.53 Usually, the yarns have fluffy, and the surface often has obvious hair feathers. In this case, compared to fiber and fabric, the surface of the yarns presents the greatest roughness. As we all know, it is better to construct PCs on dense and smooth solid substrate surfaces. Thus, the fuzziness and roughness significantly increase the difficulty of constructing a regular photonic crystal on flexible yarns.27–29 Based on the rich experience of our research team on the structural coloration of textiles, it is believed that if we can construct PCs with bright structural colors on the flexible yarns, it will build a complete structural coloration system of textiles and promote the development of structural coloration. Therefore, it is essential to investigate a method that can construct photonic crystals with obvious structural colors on the flexible yarns.

Studies on structural coloration of textiles based on PCs have been carried out for more than 10 years. In addition to the preparation methods, more and more researchers have devoted their energy to studying the stability of photonic crystals. Researchers have found that although the photonic crystals on the textile substrates can show bright structural colors, when experiencing folding, bending, friction, and washing, the photonic crystals on the substrates are easily falling off, and the corresponding structural colors will also disappear. It is foreseeable that the structural colors of photonic crystals on the yarn will also encounter similar problems. To improve the stability of the photonic crystals, that is, the durability of the structural colors, the researchers carried out a series of studies.27,28 As for the photonic crystals on textile substrates, it is not difficult to understand that the stability of the photonic crystals will be related to the substrates and the crystal structure itself. However, many current studies only focus on the substrates or the photonic crystal itself singly; studies about how to improve the stability of photonic crystals by modulating substrates and the crystal structure simultaneously have been hardly reported. Based on the previous research experience, we conduct research from these two aspects to prepare high-stability photonic crystals on the yarns.

In this work, a simple and convenient strategy was reported to construct PC-based structural color yarns with strong color durability by assembling poly(styrene-butyl) acrylate-methacrylate) (P(St-BA-MAA)) colloidal microspheres on yarns. Specifically, the properties of P(St-BA-MAA) colloidal microspheres were studied. The effects of the mass fraction of cationic acrylate used to prepare the yarns in advance on the resulting structural color effect were thoroughly investigated. In addition, the color durability and color regulation method of prepared PC-based structural color yarns were evaluated and analyzed. It is believed that this study will provide some reference and guidance for the construction of PCs with brilliant structural colors on flexible materials.

2. EXPERIMENTAL SECTION

2.1. Materials. Styrene (St, A.R. grade, Aladdin Reagent (Shanghai) Co., Ltd., China), butyl acrylate (BA; A.R. grade, Aladdin Reagent (Shanghai)), methyl acrylic acid (MAA, A.R. grade, Aladdin Reagent (Shanghai) Co., Ltd., China), sodium dodecyl sulfate (SDS, A.R. grade, Aladdin Reagent (Shanghai) Co., Ltd., China), ammonium persulfate (APS, A.R. grade, Aladdin Reagent (Shanghai) Co., Ltd., China), and ammonia (NH\textsubscript{3}·H\textsubscript{2}O; A.R. grade, Aladdin Reagent (Shanghai) Co., Ltd., China) were used without further purification. Black acrylic yarns (430.6 tex) were bought from local markets. The solid content of cationic acrylate is 45% (Shanghai Xunda New Material Technology Co., Ltd.). Deionized water (>18 MΩ·cm, Millipore Milli-Q) was used for the entire experiment.

2.2. Preparation of the P(St-BA-MAA) Colloidal Microspheres. P(St-BA-MAA) colloidal microspheres with uniform particle size and good monodispersity were prepared by a semi-intermittent emulsification polymerization method. First, 110 g of St, 3.5 g of MAA, 0.5 g of SDS, 49 g of BA, and 45 g of H\textsubscript{2}O were mixed in a three-neck flask, and the mixture was emulsified at 1000 r/min for 40 min. Then, 75 g of deionized water was added to a three-neck flask equipped with a reflux condenser, a mechanical stirrer, and a nitrogen inlet. When the temperature was heated to 80 °C, 0.3 g of NH\textsubscript{3}·H\textsubscript{2}O was added to the flask to ensure that the reaction was in an alkaline environment. Then, 6.5 g of prepared pre-emulsion and 0.5 g of APS dissolved in 1.8 g of H\textsubscript{2}O were added to the reactor together and stirred vigorously. The seed emulsion polymerization was kept at 85 °C for 20 min. Next, the remaining pre-emulsion of 0.8 g of NH\textsubscript{3}·H\textsubscript{2}O dissolved in 7.5 g of H\textsubscript{2}O and 0.3 g of APS dissolved in 7.5 g H\textsubscript{2}O was added to the reactor simultaneously and the reaction mixture was further kept at 85 °C for 3 h. Then, another 0.3 g of APS dissolved in 7.5 g of H\textsubscript{2}O was added to the reactor for 2 h to ensure the completion of monomer conversion. The entire reaction was carried out under a nitrogen atmosphere with mechanical stirring at about 230 r/min. Finally, when the temperature was cooled to room temperature, the resulting colloidal suspension of P(St-BA-MAA) microspheres was filtered through a sieve (200 mesh) to remove any large agglomerates and then stored in polyethylene terephthalate plastic (PET) bottles. Different particle sizes of P(St-BA-MAA) colloidal microspheres can be prepared by varying the polymerization parameters, such as monomer dosage, stirring speed, and polymerization temperature.

2.3. Preparation of Photonic Crystal-Based Structural Color Yarns. P(St-BA-MAA) PCs with structural colors on black yarns were prepared with the colloidal emulsion of P(St-BA-MAA) colloidal microspheres via a dip-coating method. First, the yarns were neatly arranged and oscillated in ultrapure water for 30 min, removing impurities on their surfaces and preventing them from tangling, and then these yarns were dried in an oven at 80 °C for 20 min. Then, the dried yarns were immersed in the cationic acrylate solution (mass fraction is 3%) for 10 min; the excessive cationic acrylate solution was swept away, utilizing a high-density sponge with a thickness of 3 cm, and then the yarns were dried in an oven at 80 °C for 10 min. Finally, the colloidal dispersion of P(St-BA-MAA)
microspheres was diluted with deionized water to a specific concentration; the dried and cooled yarns were immersed in 20 mL of the microsphere dispersion for 10 min, and the superfluous emulsion was slowly swept away with a high-density sponge along the direction from the bottom to the top of the hairiness. The ordered P(St-BA-MAA) PCs were obtained on the yarns after evaporating the water in the colloidal dispersion at an appropriate temperature. The experimental schematic diagram of P(St-BA-MAA) colloidal microspheres assembled on the yarns by a dip-coating method is shown in Figure 1.

2.4. Characterization. 2.4.1. P(St-BA-MAA) Microspheres and Cationic Acrylate. The particle sizes and monodispersity of P(St-BA-MAA) microspheres were determined by a Malvern laser particle size analyzer (Nano-S, Malvern, England). It is worth noting that the P(St-BA-MAA) colloidal suspension was approximately 1000-fold-diluted with Milli-Q water prior to measurements. The morphology of P(St-BA-MAA) microspheres was observed by field emission scanning electron microscopy (FESEM, ALTRA55, Germany) and transmission electron microscopy (TEM, JEM2100, Japan). All FESEM images were collected under an electron gun with an accelerating voltage of 3 kV. The glass-transition temperature of P(St-BA-MAA) microspheres was measured by a DSC8000 differential scanning calorimeter (Perkins Elmer Instruments) at a temperature of 0−200 °C and a heating rate of 5 °C/min for the specimen. The adhesive strength of the adhesive was evaluated by testing the intensity between the two fabrics with cationic acrylate.

2.4.2. Photonic Crystals and Structural Colors. The arrangement of the P(St-BA-MAA) PCs on black yarns was measured by a field emission scanning electron microscopy (FESEM, ALTRA55, Germany). The photographs of PC-based structural color yarns and their woven fabrics were taken by a cell phone (Honor20, Huawei, China) and a 3D video microscope (KH-7700, HIROX, Japan). The iridescent phenomenon of PC-based structural color yarns was observed by a multiangle spectrophotometer (MA98, X-Rite) with a colorimetric illuminant of D65 and a colorimetric preferred observer of 10°. The MA98 multiangle spectrophotometer can supply the images, L*a*b* values, and reflectance spectra of the samples beneath a variety of viewing instructions.

The endurance of structural colors is connected to the stability of PCs. The durability of PCs with structural colors on flexible yarn substrates was determined using a rubbing test and a washing test. The rubbing fastness of PCs with structural colors was tested using a counterbalance with a mass of 200 g and an area of 6.28 cm². The countertop presses the substrate onto the acrylic material and drags the yarn parallel underneath the countertop. The whole yarn was dragged over the countertop, and then a complete round was finished. Ten rounds had been repeated to take a look at the rubbing fastness. For the washing test, the pattern was once washed with water for 5 min. The stability of crystals was once evaluated with the aid of evaluating the coloration adjustments earlier than and after rubbing and washing. The photographs of the stability testing of PC-based structural color yarns were taken by a cell phone (Honor20, Huawei, China) and a 3D video microscope (KH-7700, HIROX, Japan).

3. RESULTS AND DISCUSSION

3.1. Properties of P(St-BA-MAA) Colloidal Microspheres. The uniformity of the sizes and shapes of colloidal microspheres plays a crucial role in the optical properties of PCs. P(St-BA-MAA) colloidal microspheres’ particle size distribution and morphologies are shown in Figure 2a−c. The prepared microspheres with diverse hydrated particle sizes have narrow and sharply distributed peaks, suggesting that these P(St-BA-MAA) microspheres have a uniform particle size and outstanding monodispersity, which is conducive to the preparation of highly ordered PC structures, as shown in Figure 2a. The surface morphologies of P(St-BA-MAA) colloidal microspheres are given in Figure 2b by FESEM, revealing that these microspheres are of good sphericity, which provide the ability to fabricate ordered PCs. Figure 2c shows the TEM images of P(St-BA-MAA) colloidal microspheres with a diameter of 255 nm, indicating that the P(St-BA-MAA) nano-microspheres have a representative core−shell structure.29,30 According to the hydrophilicity of the comonomer, the hydrophobic PSt domain is mainly located on the core of
prepared microspheres, and a thin shell rich in PMAA and PBA covers the PSt core. The P(St-BA-MAA) is considered a kind of colloidal microspheres with a hard core and soft shell, where the hard core (PSt) is expected to maintain the skeleton of prepared PCs, while the soft shells rich in PBA are expected to enhance the connection between adjacent microspheres of prepared PCs. Moreover, the soft shells are rich in PMAA, where the carboxyl groups (−COOH) are expected to form a hydrogen bond to further strengthen the connection of adjacent microspheres.

As we all know, the glass-transition temperature (T_g) is the temperature corresponding to the transformation from the glassy state to the high elastic state; the lower the glass-transition temperature of the materials, the better the adhesion. Figure 2d shows the differential scanning calorimetry (DSC) thermograms of P(St-BA-MAA) colloidal microspheres, from which it can be seen that their glass-transition temperature (T_g) is around 65.6 °C, indicating that when the assembly temperature is higher than 65.6 °C, the P(St-BA-MAA) colloidal microspheres will become softer. In this case, the adjacent microspheres can squeeze, thereby forming a dense crystal structure. However, the assembly temperature should not be too higher than T_g of colloidal microspheres. Otherwise, the microspheres will be severely degraded, so as to stick together, and the PCs cannot be formed.

3.2. Fabrication of Photonic Crystals with High Stability on Acrylic Yarns. Before the self-assembly of P(St-BA-MAA) colloidal microspheres, the cationic acrylate acting as an adhesive was used for preprocessing the original yarns. Then, the P(St-BA-MAA) colloidal microspheres were assembled on the acrylate layer to fabricate the PC structure. Based on this design, the adhesives act as a “bridge” to achieve
a firm combination between the PC layers and the yarns. To evaluate the adhesive strength of the cationic acrylate, an adhesive test was performed, as shown in Figure 3. According to the phenomenon in Figure 3, it can be considered that cationic acrylate presented a strong adhesive, which is expected to closely combine the crystal layers and yarns.

It is well known that a smooth substrate surface is more favorable for the self-assembly of the colloidal microspheres. However, as for textile yarns, their surface is usually rough and
presents obvious hair feathers. Therefore, it is not difficult to understand that the rough yarn surface is not conducive to constructing regular photonic crystals. Fortunately, in addition to enhancing the connection between the yarn and the crystal structure, we found that the cationic acrylate can also smoothen the yarn surface. Figure S1a,b shows the microscopy images of the original yarns and the yarns treated with cationic acrylate, respectively. After the treatment with cationic acrylate, the yarn surface was obviously smooth, and the hair feathers were not prominent. Furthermore, as shown in the AFM images of Figure S1c,d, it can be seen that the roughness ($R_a$) of the yarn surface was significantly reduced after the treatment with cationic acrylate. Based on the above results, the treatment of cationic acrylate would be beneficial to enhance the smoothness of yarns, which was expected to construct regular photonic crystals with bright structural colors.

The effects of the mass fraction of cationic acrylate used to prepare the yarns in advance on the resulting structural color effect and corresponding photonic crystal arrangement were investigated, as shown in Figure 4. The structural colors on the yarns are dull when the mass fraction of cationic acrylate is between 0 and 1 wt %, and even the center and edge of the yarns have uneven hues (Figure 4a,b). When the mass fraction increases to 3 wt %, the structural colors are vivid and bright (Figure 4c). However, as the mass fraction continues to increase, the structural colors become dull (Figure 4d). Thus, the optimum mass fraction of adhesive is around 3 wt %. From the FESEM images of PCs on acrylic yarns using adhesive with different mass fractions, it can be seen that the P(St-BA-MAA) colloidal microspheres directly self-assembled on yarns without preprocessing by cationic acrylate, the microspheres are easily dislodged, so the number of microspheres attached to the yarn substrate is small. It is obvious that the yarn...
substrate is exposed and the PC layer on the surface is uneven (Figure 4a). With the increase of adhesive concentration, the quantity of microspheres fixed on the yarn surface increased significantly but still did not completely cover the yarn surface (Figure 4b). For the mass fraction of 3 wt %, the P(St-BA-MAA) microspheres in resulting PCs are dense and ordered (Figure 4c). However, when the mass fraction of adhesive is 5 wt %, the excessive adhesive makes the P(St-BA-MAA) microspheres aggregate irregularly and the periodic structure of the crystals is disrupted (Figure 4d). Based on the results of the PC arrangement and structural color effect, the ideal mass fraction of adhesive is about 3 wt %.

Furthermore, according to the results of Figure 4, when the mass fraction of cationic acrylate is 0 wt %, the yarns do not preprocess by cationic acrylate before self-assembly, the structural colors on the yarns are dim and the corresponding crystals are scattered. In contrast, after the pretreatment of cationic acrylate with an appropriate amount (3 wt %), the P(St-BA-MAA) microspheres aggregate regularly and the periodic structure of the crystals is disrupted (Figure 4d). Based on the results of the pretreatment, the yarns can show bright structural colors and the crystal arrangement is regular. Why is there such a phenomenon? The possible reason is that the cationic acrylate forms a film on the yarn surface, which ameliorates the roughness of the yarn, makes the yarn smooth, and is conducive to forming regular PCs. According to many studies, the smoothness of the surface of substrates is expected to improve the regularity of PCs and the corresponding structural color effect. A schematic diagram is used to illustrate the influence of using adhesive treatment. As shown in Figure 5, the cationic acrylate solution served as an aqueous adhesive to attach a bonding layer to the yarns, building a "bridge" between the PC and the yarns and also smoothing the surface of the yarn, thus improving the stability of PCs on the yarn’s body to get a more vivid structural color.

3.3. Evaluation of Color Durability of Photonic Crystal-Based Structural Color Yarns. For textiles, rubbing and washing fastness are very important. Actually, a lot of related work has been performed to study the rubbing and washing fastness of PC-based structural color yarns. However, we have to point out that those PC-based structural color yarns with PCs are very different from the conventional dyed yarns; the conventional testing methods for rubbing and washing fastness were set according to the performance of the dyes, and the conventional testing usually has recognized standards and comparison samples; in other words, we cannot use conventional testing methods to characterize the washing and rubbing fastness of those yarns with structural colors.

In our present study, after a lot of experiments, we begin to establish a method to characterize the rubbing and washing fastness of the PC-based structural color yarns. Figure 6 illustrates the results of the PC-based structural color yarns undergoing rubbing and washing tests. It can be seen that after 10 times of rubbing test and 10 min of washing test, the structure color is still relatively bright and the structural colors are still brighter, illustrating that the prepared PC-based structural color yarns have good color fastness. Furthermore, an additional device with agitation to simulate a washing machine was used to test the washing fastness of the prepared PC-based structured color yarns. The yarn was first soaked in the apparatus for 20 min and then washed under agitation at 300 r/min for 25 min for one cycle, and the washing test was performed for five cycles. Figure S2 shows the structural color change of the assembled acrylic yarn before and after the washing process. From the digital photos and three-dimensional video microscope images, it can be observed that after washing the assembled acrylic yarn maintained a bright and uniform structural color almost the same as the original samples, which implied that the prepared photonic crystals on acrylic yarn substrates had good washing color fastness in our study. To further confirm the friction fastness, we used fingers to fold and bend the photonic crystal-structured color yarn 15 times. Figure S3 shows the photographs and three-dimensional video microscope images of the PC-based structural color yarn before and after bending and folding with fingers. It can be seen that there is no significant difference before and after folding and bending, which further demonstrates that the structural color of the acrylic yarn exhibits good stability during folding and bending. This has laid a good foundation for the application of PC-based structural color yarns.

3.4. Color Regulation Method of Photonic Crystal-Based Structural Color Yarns. The PC-based structural color yarns were woven into colored fabrics using a crochet hook, as shown in Figure 7. It is observed that the structural colors constructed by the dip-coating method on the three most common types of flexible yarn substrates are all excellent. Furthermore, when the particle sizes of P(St-BA-MAA) colloidal microspheres are different, the resulting yarns show different structural colors. In this case, more structural colors

![Figure 7](https://example.com/figure7.png)
can be obtained by regulating the particle sizes of the colloidal microspheres. Besides modulating the particle sizes of colloidal microspheres, altering the viewing angle is considered to be another approach to modify the structural color of PCs on yarns. From Figure 7a, variable structural colors of the yarns are observed when the shooting angle is 15, 45, and 90°, and the colors change from blue-cyan to emerald green. This is just the typical rainbow phenomenon of structure colors produced by PCs.

Moreover, the iridescence effect of structural colors was specifically characterized by an MA98 multiangle spectrophotometer. When the incident light source is 45 and 15°, Figure 8a illustrates a variety of distinct structural colors of P(St-BA-MAA) PCs on the yarns at different viewing angles. Figure 8a illustrates that for the 45° light source, the greatest reflectance peak changes toward the long-wave direction as the observation angle increases from −15 to 45° and somewhat toward the short-wave direction as the angle increases from 75 to 110°. When the viewing angle changes from −15 to 15° for the 15° light source, the greatest reflectance peak shifts in the long-wave direction. Therefore, the mentioned phenomenon is in accordance with the consequences shown in Figure 8a. This means that the band gap of the manufactured PC can be changed by adjusting the viewing angles.

4. CONCLUSIONS

In this work, the PCs with distinct structural colors assembled by P(St-BA-MAA) colloidal microspheres were successfully manufactured on acrylic yarns by the dip-coating method. The monodispersed P(St-BA-MAA) colloidal microspheres have an obvious core–shell structure, and the glass-transition temperature (T_g) was around 65.6 °C. The cationic acrylate acts as a pretreatment agent to process the yarns before assembling. It was considered that cationic acrylate could not only improve the combination between the PC layers and the yarns but also enhance the structural color effect. The results revealed that when the mass fraction of cationic acrylate was 3 wt %, the microspheres were beneficial to access regular PCs with obvious structural colors. Moreover, the prepared PC-based structural color yarns had good color fastness, and the structural colors of yarns were still bright after multiple rubbing or washing. PCs with structural colors can be successfully built on black acrylic yarns. Furthermore, the hue of structural colors varied by adjusting the particle sizes of microspheres and viewing angles. This technology is a progressive and innovative method used to fabricate PC-based structural color yarns and is considered to have broad prospects in textile dyeing fields.

**Figure 8.** Iridescent effect images (a) and reflective cloud map (b) of P(St-BA-MAA) PC-based structural color yarns measured by a multiangle spectrophotometer.

**ASSOCIATED CONTENT**

**Supporting Information**
The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsomega.2c03672.

Effect of the yarn surface before and after treatment; and the washing and rubbing fastness of the photonic crystal-based structural color yarns (PDF)

**AUTHOR INFORMATION**

**Corresponding Author**
Guojin Liu — Zhejiang Provincial Key Laboratory of Fiber Materials and Manufacturing Technology, Zhejiang Sci-Tech University, Hangzhou 310018, People’s Republic of China; Key Laboratory of Advanced Textile Materials and Manufacturing Technology, Ministry of Education, Zhejiang Sci-Tech University Hangzhou, Zhejiang 310018, People’s Republic of China; Zhejiang Provincial Innovation Center of Advanced Textile Technology, Shaoxing 312000, China; orcid.org/0000-0003-3003-2858; Email: guojin900618@163.com; Fax: 86-571-86843251

**Authors**
Wanbin Ma — Zhejiang Provincial Key Laboratory of Fiber Materials and Manufacturing Technology, Zhejiang Sci-Tech University, Hangzhou 310018, People’s Republic of China
Hao Liu — Zhejiang Provincial Key Laboratory of Fiber Materials and Manufacturing Technology, Zhejiang Sci-Tech University, Hangzhou 310018, People’s Republic of China
Wenyu He — Zhejiang Provincial Key Laboratory of Fiber Materials and Manufacturing Technology, Zhejiang Sci-Tech University, Hangzhou 310018, People’s Republic of China
Yunxiao Zhang — Zhejiang Provincial Key Laboratory of Fiber Materials and Manufacturing Technology, Zhejiang Sci-Tech University, Hangzhou 310018, People’s Republic of China
Yucheng Li — Zhejiang Provincial Key Laboratory of Fiber Materials and Manufacturing Technology, Zhejiang Sci-Tech University, Hangzhou 310018, People’s Republic of China
Yang Zhao — Zhejiang Provincial Key Laboratory of Fiber Materials and Manufacturing Technology, Zhejiang Sci-Tech University, Hangzhou 310018, People’s Republic of China
Chengai Li — Zhejiang Provincial Key Laboratory of Fiber Materials and Manufacturing Technology, Zhejiang Sci-Tech University, Hangzhou 310018, People’s Republic of China
Lan Zhou — Key Laboratory of Advanced Textile Materials and Manufacturing Technology, Ministry of Education, Zhejiang Sci-Tech University Hangzhou, Zhejiang 310018, People’s Republic of China
Jianzhong Shao — Key Laboratory of Advanced Textile Materials and Manufacturing Technology, Ministry of Education, Zhejiang Sci-Tech University Hangzhou, Zhejiang 310018, People’s Republic of China

Complete contact information is available at: https://pubs.acs.org/10.1021/acsomega.2c03672

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