

FORMATION OF COPPER OXIDE (Cu₂O) CRYSTALS ON THE SURFACE OF NATURAL FIBROUS FABRICS AND STUDY OF THEIR PHYSICAL AND MECHANICAL PROPERTIES

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Abstract

Structural color-forming materials are expected to replace pigments and dyes as a new type of color materials with good light fastness and bright color. In this study, copper oxide (Cu₂O) microspheres of different sizes were synthesized by a two-step spin-coating method, and the microspheres were firmly attached to fabrics using a polyvinyl alcohol (PVA) binder. Two different types of fabrics (cotton, silk) were evaluated to study their physical properties and color fastness. As a result, the tensile strength and tearing strength of Cu₂O structural fabrics slightly decreased, while the tearing strength of silk fiber fabrics increased, except for cotton fiber fabrics. Meanwhile, all structurally colored fabrics showed excellent color fastness to cutting, abrasion, and washing. This study provides experimental data for developing the feasibility of using structural colors in various natural fabrics.

Keywords: Textile fabrics, structural color, photonic crystals, Cu₂O microspheres, mechanical properties.

Introduction

The phenomenon of color formation results from the interaction of natural light and molecular matter and is mainly divided into two categories: chemical pigment color and structural color [1]. The color of chemical pigments is created by the selective absorption of light by chromophores in substances such as plant dyes and pigments [2]. However, a number of problems arise when using chemical dyes in the context of promoting green and sustainable development. These problems include the use of various chemicals and additives in the dyeing process, the complexity and high energy intensity of the technological processes, and the formation of large quantities of environmentally harmful colored wastewater during the production process [3,4]. The treatment of this wastewater is costly and poses a risk of

environmental pollution. However, structural color consists of optical phenomena that result from the interaction between physical structures on the micro- and nanometer scale and natural light [5,6]. These mechanisms include interference, scattering, and diffraction. When viewed from different angles, different gloss and color effects can be observed, as can be seen in natural phenomena such as peacock feathers and butterfly wings, which are classic examples of structural color [7]. Compared with traditional pigment paints, structural paint not only has variable gloss depending on the viewing angle, but also has high gloss, high saturation, and environmental adaptability, and is characterized by color control properties [8]. Structural color is divided into five main types: photonic crystals, thin-film interference, multilayer thin-film interference, scattering, and diffraction [9]. In particular, photonic crystals are characterized by constant dielectric periodicity, which allows them to selectively transmit light of certain wavelengths and block others. Structural color is created by selectively reflecting and diffraction of visible light by photonic crystals [10].

In recent years, there has been growing interest in the application of photonic crystal structures in tissues. However, most of the existing studies have focused on the use of polymer and inorganic photonic crystal structures with a low refractive index ($n = 1.4\text{--}1.5$) on tissue substrates, and there has been insufficient research on the application of metal oxide photonic crystal dyes with a high refractive index on tissue substrates [11–13]. Currently, the assembly of microspheres into photonic crystal structures on tissue substrates is mainly carried out by self-assembly; however, this method reduces the bond strength between the microspheres and the substrate [14]. To increase the bond strength, binders are added to the system. However, the refractive indices of such adhesives are usually close to the refractive index of the microspheres, which, while increasing stability, reduces the quality of color effects. This problem significantly limits the potential applications of photonic crystal structures in tissue dyeing [15]. This study presents a technique for structural dyeing of fabric by creating a structural color layer of Cu_2O . This approach makes it possible to produce structural colors using fabrics. However, it should be noted that the colors obtained using this technique have a higher degree of angular dependence [16,17]. Huang et al. deposited Cu and CuO films on polymer and white nonwoven fabrics and studied the effect of spray flow on the coloration of the fabric structure [18]. The resulting fabrics were orange-red, dark red, and dark green. In addition, these fabrics have enhanced UV protection, hydrophobicity, antistatic properties, and varying degrees of permeability, which makes them suitable for use in smart clothing and high-tech textiles [19]. This method has several advantages, including ease of control; in particular, the spraying process does not use water resources and does not produce harmful substances for the environment [20]. Similarly, Han-Bo-Ran Lee et al. used comparable methods to deposit $\text{Al}_2\text{O}_3/\text{TiO}_2$ multilayer membranes onto conductive electronic fabrics and successfully produced conductive fabrics in seven different colors [21,22]. The fabrics obtained using this approach exhibited bright colors and mechanical strength. However, more research is needed on common materials such as cotton and wool.

By changing the size of the microspheres, fabrics of different colors can be obtained. However, the natural specific structure of silk fibers reduces the degree of ordering of the microspheres on the fiber surface, which leads to a decrease in the dyeing effect [23, 24]. Ge et al. used the

capillary structure of fabrics. First, transparent granular lightweight SiO_2 crystals were obtained on the surface of individually grouped fibers by spraying [25]. The process was relatively simple, which reduced the collection time and prevented the clogging of the fabric pores. Then, an adhesive layer was applied to the surface to enhance the protection and stability of the above structure. As a result, the creation of sufficiently strong, soft and breathable fabrics, as well as the inclusion of multi-colored fabrics in their composition, is a reasonable approach.

This study focuses on the synthesis of Cu_2O microspheres by varying the ratio of copper acetate and trisodium citrate. These microspheres were sprayed to create structurally dyed fabrics of different colors using polyvinyl alcohol (PVA) as a binder. The study also explores the potential of applying Cu_2O microspheres to various types of fabrics, evaluating their physical properties and color stability under various conditions. Ultimately, this study aims to establish an experimental basis for developing vivid structural colors in various textile materials.

2. Materials and Methods

2.1. Materials

Copper acetate monohydrate ($\text{C}_4\text{H}_6\text{CuO}_4 \cdot \text{H}_2\text{O}$), polyethylenepyrrolidone, ethylene glycol, sodium hydroxide and anhydrous ethanol are the products of Shanghai McLean Biochemical Technology waste recycling enterprise located in Shanghai, China. Sodium citrate dihydrate ($\text{C}_6\text{H}_5\text{Na}_3\text{O}_7 \cdot 2\text{H}_2\text{O}$), polyvinyl alcohol (low viscosity type 0588) and ascorbic acid are the products of Shanghai Aladdin Biochemical Technology waste recycling enterprise, also located in Shanghai, China. Deionized water (conductivity $18 \text{ M}\Omega \text{ cm}$) was prepared in the laboratory. Cotton fabrics were purchased from Shenzhen Orange Fabric Textile Waste Enterprise located in Shenzhen, China; Silk fabrics were purchased from Ningbo Quantai Electronic Commerce Company, located in Shenzhen, China. All chemicals and solvents were of analytical grade, and no additional purification was performed.

2.2. Preparation of Cu_2O Microspheres

In this experiment, Cu_2O microspheres with a size of $210 \pm 10 \text{ nm}$ were synthesized by a two-step liquid-phase reduction method as follows:

Step 1: Preparation of copper stock solution.

Initially, 3 g of polyvinylpyrrolidone (PVP) ($\text{Mw} = 55,000$), 0.98 g of sodium citrate dihydrate ($\text{C}_6\text{H}_5\text{Na}_3\text{O}_7 \cdot 2\text{H}_2\text{O}$), and 0.664 g of copper acetate monohydrate ($\text{C}_4\text{H}_6\text{CuO}_4 \cdot \text{H}_2\text{O}$) were placed in a 500 mL volumetric flask. Here, copper acetate is used as a Cu^{2+} ion source. Then, 60 mL of ethylene glycol ($\text{HOCH}_2\text{-CH}_2\text{OH}$) and 200 mL of deionized water were added to the flask, and the mixture was stirred with a magnetic stirrer for 3 hours and then subjected to ultrasonic vibration for 10 minutes. As a result, the solid particles were completely dissolved and a light blue transparent solution was formed [26].

Step two: synthesis of Cu_2O microspheres.

Since the reduction reaction developed for this experiment should be carried out under alkaline conditions, strong alkaline sodium hydroxide (NaOH) was chosen as the source of OH^- ions. Initially, 20 ml of 0.4 M sodium hydroxide solution were slowly added to the initial copper solution, and the solution turned dark blue. After 10 minutes of vigorous stirring, 15 ml of 0.2

M ascorbic acid ($C_6H_8O_6$) reducing agent were added twice, and stirring was stopped after 60 minutes. As a result, a mixture of Cu_2O microspheres was obtained. The reaction products were centrifuged at 8000 rpm for 10 minutes to separate them. The obtained products were washed with a mixture of deionized water and anhydrous ethanol (1:1 ratio) to remove impurities, and then dried to obtain Cu_2O microspheres. The preparation process is shown in Figure 1. By changing the molar ratio of Cit^{3-} and Cu^{2+} (0.9, 1.0, 1.1, and 1.2), monodisperse Cu_2O microspheres with particle sizes ranging from 190 nm to 275 nm can be prepared.

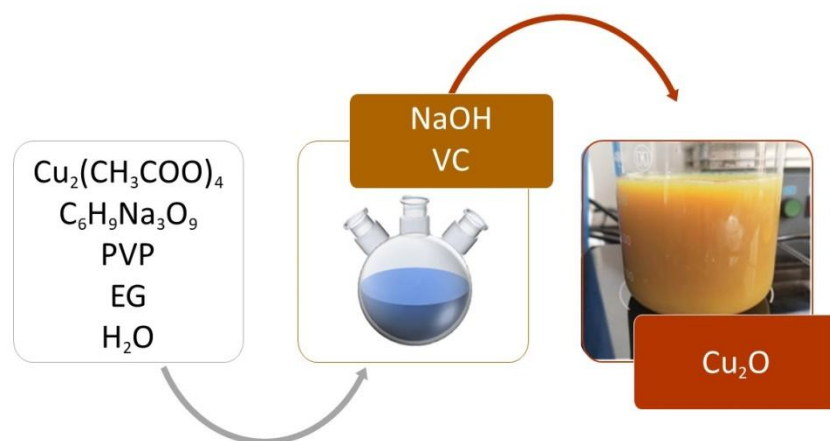


Figure 1. Preparation process of Cu_2O microspheres

2.3. Preparation of Cu_2O Structural Colored Fabrics

In this experiment, the structurally dyed fabrics were prepared in the following steps: first, the synthesized Cu_2O microspheres were completely dispersed in anhydrous ethanol to obtain a Cu_2O dispersion with a mass fraction of 2%. Second, polyvinyl alcohol (PVA) particles were dispersed in deionized water and subjected to ultrasonic vibration at 90 °C for 3 hours to prepare a PVA binder solution with a mass fraction of 2%. Then, the PVA adhesive material was uniformly sprayed on the fabric surface with a spray gun connected to an air compressor and dried at 60 °C for 5 minutes. This process achieves the effect of pre-treatment. Finally, the Cu_2O dispersion was sprayed on the pre-treated fabric with a spray gun. The compressor pressure was set at 0.5 psi and the spray nozzle diameter was set at 0.2 mm. The PVA- Cu_2O fabrics are dried at 50°C for 10 minutes to obtain structurally dyed fabrics. The preparation process is shown in Figure 2.

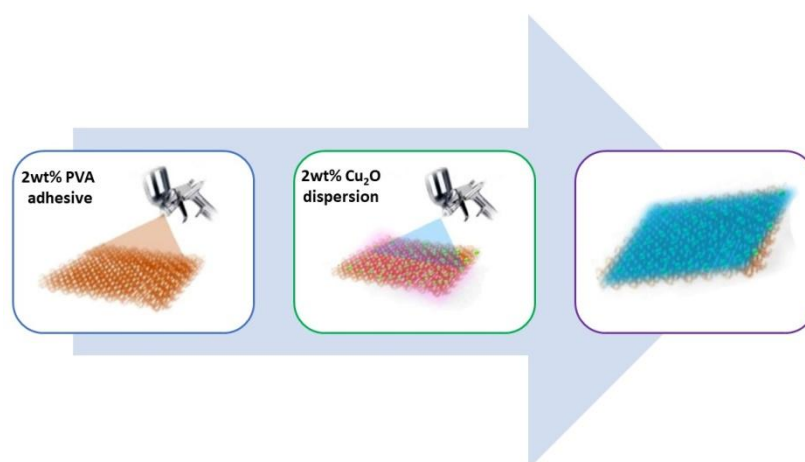


Figure 2. The process of making structural colored fabrics

2.4. Characterization methods

The surface morphology of Cu_2O microspheres was determined by scanning electron microscopy (FESEM, Gemini500, Carl Zeiss AG, Oberkochen, Germany). The elemental composition of the structurally dyed silk fabrics was determined by energy dispersive X-ray spectroscopy (EDS) using a scanning electron microscope to study the surface morphology (FESEM, Gemini 500, Carl Zeiss AG, Oberkochen, Germany). The crystalline phase structure of Cu_2O microspheres was analyzed by X-ray diffractometry (XRD, $k\text{-}\alpha$, Thermo Fisher, Thermo Fisher Scientific Inc, Waltham, MA, USA). Dry Cu_2O microspheres were ground and purified as samples, and a $\text{CuK}\alpha$ light source (wavelength 0.154178 nm) was used. The scanning speed is $10^\circ/\text{min}$, the angle range is from 10° to 80° , the current is 40 mA, and the voltage is 40 kV. The chemical structure of black cotton and silk fabrics before and after the application of the structural dye Cu_2O was investigated using a Fourier transform infrared spectroscopy (FTIR) analyzer (IRPrestige-21, Shimadzu, SHIMADZU Corporation, Kyoto, Japan). Optical images of the structurally dyed fabrics were obtained using a 3D video microscope (HIROX KH-7700, HIROX, Kyoto, Japan).

2.5. Mechanical properties and color fastness of structurally colored fabrics

The tensile properties of the specimens in the warp and weft directions were tested according to GB/T 3923.1-2013 Textiles - Tensile properties of fabrics - Part 1: Determination of maximum strength and maximum elongation using the tape method [27]. According to GB/T 3917.2-2009 Textiles - Tensile properties of fabrics - Part 2: Determination of tear strength of trouser-shaped specimens (single tear method) [28], the tear properties of the specimen were tested on the warp and weft. GB/T 19976-2005 Textiles - Determination of tear strength. The tear properties of the specimen were tested using the steel ball method [29]. Washing fastness: The fabric was placed in a 150 ml beaker, filled with water and stirred using a magnetic stirrer at 1200 rpm. Rubbing fastness: Rubbing fastness test according to national standard ISO 105-X12:2001 "Color fastness to rubbing for testing color fastness of textiles" [30]

3. Results and Discussion

3.1. SEM images of Cu₂O structural colored fabric

After Cu₂O was sprayed onto the fabric, the surface morphology of the fibers was observed using a scanning electron microscope (SEM) (Figure 3). Figures 3a, c shows the images of cotton, silk, and fibers before treatment, respectively. Figures 2b, d show the SEM images of cotton and silk fabric fibers after pretreatment and spraying with Cu₂O dispersion. They show that the Cu₂O microspheres are uniformly distributed on the surface of the fibers. The resulting structurally colored fibers were analyzed using energy dispersive spectroscopy (EDS) (Figure 4). Since the colored fibers were coated with gold during the analysis, the peaks of platinum element were recorded around 2 eV. To ensure the accuracy of the analysis, platinum element was ignored. As shown in Figure 4, the energy spectrum only contains copper (Cu), oxygen (O), and carbon (C) elements. The analysis of the energy spectrum of each element shows the presence of multiple peaks, the number of which depends on the number of electron shells of the element. The more electron shells an element has, the more peaks it has. Thus, the copper element has a spectrum of 2 peaks. As shown in Table 1 (element composition table), the percentage of Cu element is 46.47%, O element is 19.98%, C element is 33.55%, and their sum is 100.00%. Therefore, the EDS analysis results confirm that the Cu₂O microspheres have been successfully sprayed onto the fibers.

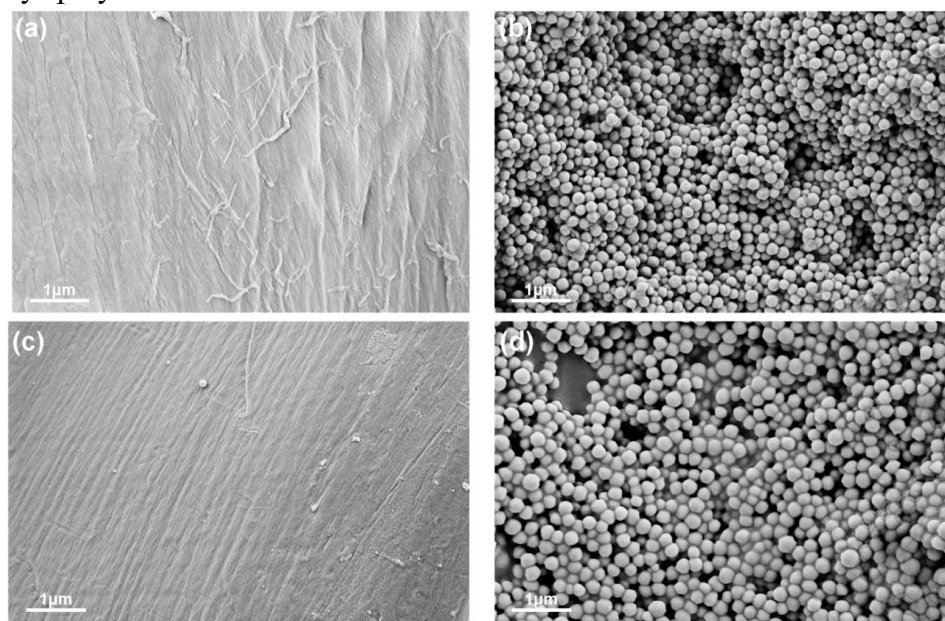


Figure 3. SEM images ($\times 15,000$) of different fibers before and after coating with Cu₂O microspheres: (a) raw cotton, (b) cotton after dyeing, (c) raw silk, (d) silk after dyeing

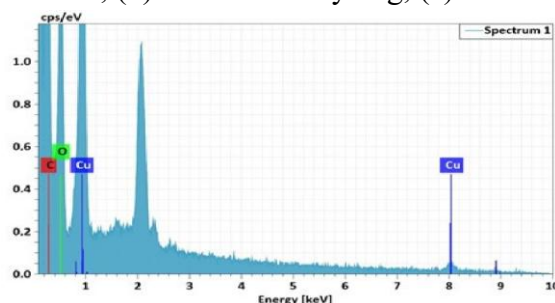


Figure 4. Energy-dispersive X-ray spectroscopy (EDS) elemental analysis.

Table 1. Table of chemical elements

Element	Atomic number	Element percentage (%)
Copper (Cu)	29	46.47
Oxygen (O)	8	19.98
Carbon (C)	6	33.55
Total		100

3.2. FTIR spectrum of structurally colored fabrics

Figure 5 shows the Fourier transform infrared (FTIR) spectra of copper(I) oxide (Cu_2O) powder, cotton and silk fabrics before and after spraying with Cu_2O microspheres. As can be seen from Figure 5, there is a peak at 630 cm^{-1} for the Cu_2O product, which corresponds to the characteristic peak of Cu-O stretching vibration in Cu_2O . This indicates that the preparation of Cu_2O is successful. It can be seen from Figures 4a-b that, compared with the untreated fabrics, a new peak appeared in the range of $3268\text{ cm}^{-1} \sim 3412\text{ cm}^{-1}$ in the fabrics treated with PVA and Cu_2O layers. This peak corresponds to the characteristic peak of -OH bond. These results indicate that PVA is successfully applied to cotton and silk fabrics. In addition, a characteristic peak characteristic of Cu_2O appeared at 630 cm^{-1} in the cotton/PVA/ Cu_2O and silk/PVA/ Cu_2O curves, confirming that the Cu_2O microspheres were successfully sprayed onto the fabrics.

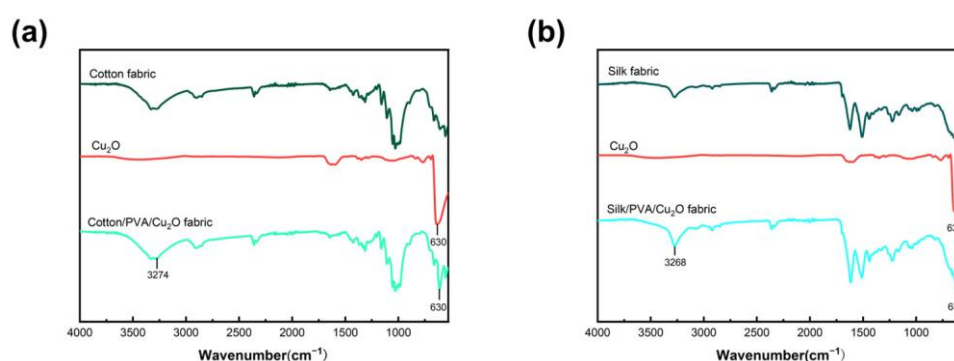


Figure 5. Comparison of FTIR spectra before and after spraying Cu_2O microspheres on different fabrics: (a) cotton fabric, (b) silk fabric

3.4. Color Properties of Cu_2O Structurally Colored Fabrics.

By changing the ratio of citrate and Cu^{2+} during the synthesis of Cu_2O , it is possible to obtain dyed fabrics with a structure of Cu_2O microspheres of green, yellow, orange or red colors. The results of forming structural colors by spraying Cu_2O structural color microspheres of different sizes onto the surface of textile fabrics are shown in Figure 6. As can be seen from Figure 6, the created structural color patterns have good integrity and structural color effect.

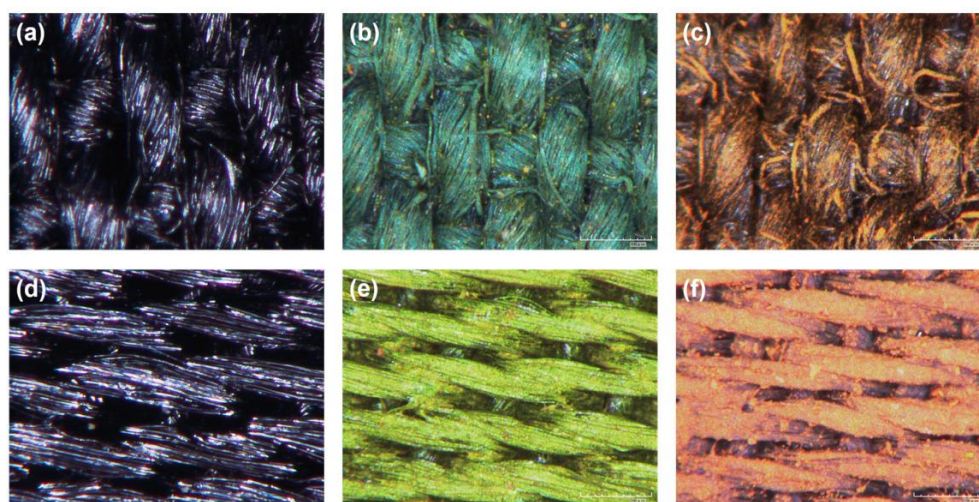


Figure 6. Photographs of structurally dyed fabrics: (a) raw cotton, (b) cotton after sputtering with 190 nm Cu_2O microspheres, (c) cotton after sputtering with 245 nm Cu_2O microspheres, (d) raw silk, (e) silk after sputtering with 190 nm Cu_2O microspheres, (f) silk after sputtering with 245 nm Cu_2O microspheres

3.4. Color Properties of Cu_2O Structurally Colored Fabrics

The reflectivity of the obtained structural colored fabrics was tested, the results are presented in Figure 7. Figure 7 shows that by changing the ratio of citrate and Cu^{2+} during the synthesis of Cu_2O , it is possible to obtain Cu_2O microspheres of 4 different sizes. The reflectance of the structural color patterns obtained by self-organization of microspheres of different sizes is high and the reflectance peak is sharp, which is explained by the good structural color effect. However, due to the strong absorption of Cu_2O microspheres in the blue-violet range of visible light (400-500 nm), the fabrics appear mainly yellow-orange, which makes it difficult to obtain blue color. However, in this work, it was possible to create orange fabrics using the unique yellow-orange color of Cu_2O microspheres.

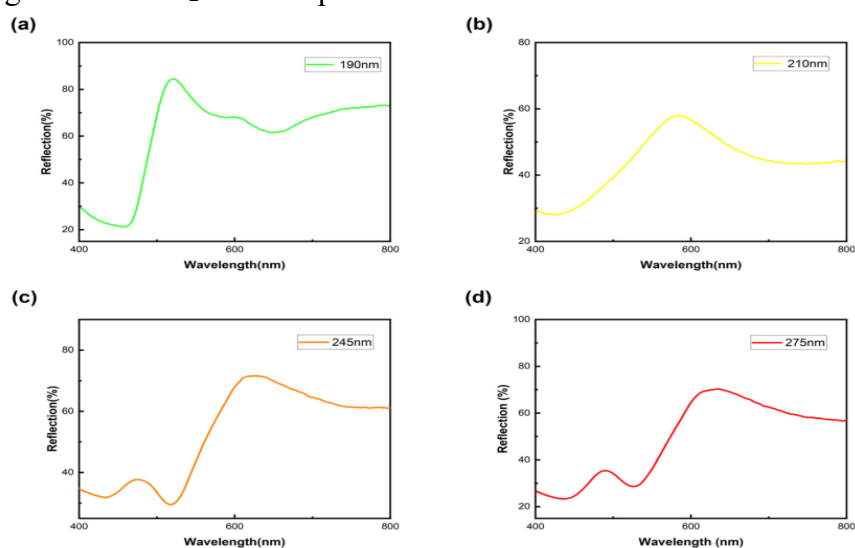


Figure 7. Reflectance curves of structurally stained fabrics: (a) 190 nm, (b) 210 nm, (c) 245 nm, (d) 275 nm.

3.5. Mechanical Properties of Structural Colored Fabrics

In order to compare the mechanical properties of cotton and silk fabrics with finished structurally dyed cotton and silk fabrics, tensile, tear and crack tests were conducted. The tensile test was carried out in accordance with GB/T 3923.1-2013 "Textiles - Tensile properties of fabrics - Part 1: Determination of maximum strength and elongation by tape method", and the tensile properties of specimens were tested in the warp and weft directions. The tear test was carried out in accordance with GB/T 3917.2-2009 "Textiles - Tensile properties of fabrics - Part 2: Determination of tear strength of trousers-shaped specimens (single tear method)", and the tear strength of specimens was tested in the warp and weft directions [31]. The tensile strength of the samples was tested in accordance with GB/T 19976-2005 "Textiles - Determination of tensile strength - Steel ball method".

Figure 8 compares the tensile strength of untreated and dyed fabrics; The tensile strength of colored cotton and silk fabrics slightly decreased after the tensile test. Figure 9 compares the tensile strength of untreated and dyed fabrics; The tensile strength of the composite-colored cotton fabric and silk fabric also slightly decreased after the tensile test. The decrease in tensile and tensile strength of composite-colored fabrics is due to the fact that a PVA (polyvinyl alcohol) binder is sprayed onto their surface during the manufacturing process. This substance forms a hard layer on the surface of the fabric, which has a certain effect on the deformation of the fabric when stretched and torn.

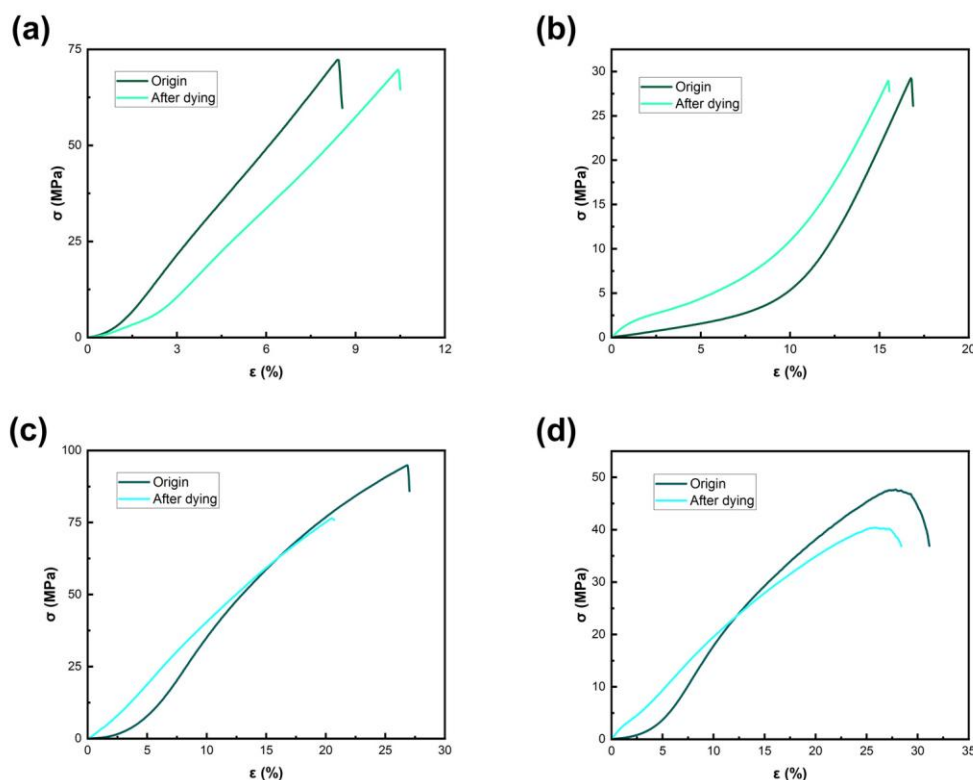


Figure 8. Comparison of fabric elongation properties: (a) cotton fabric (warp), (b) cotton fabric (weft), (c) silk fabric (warp), (d) silk fabric (weft)

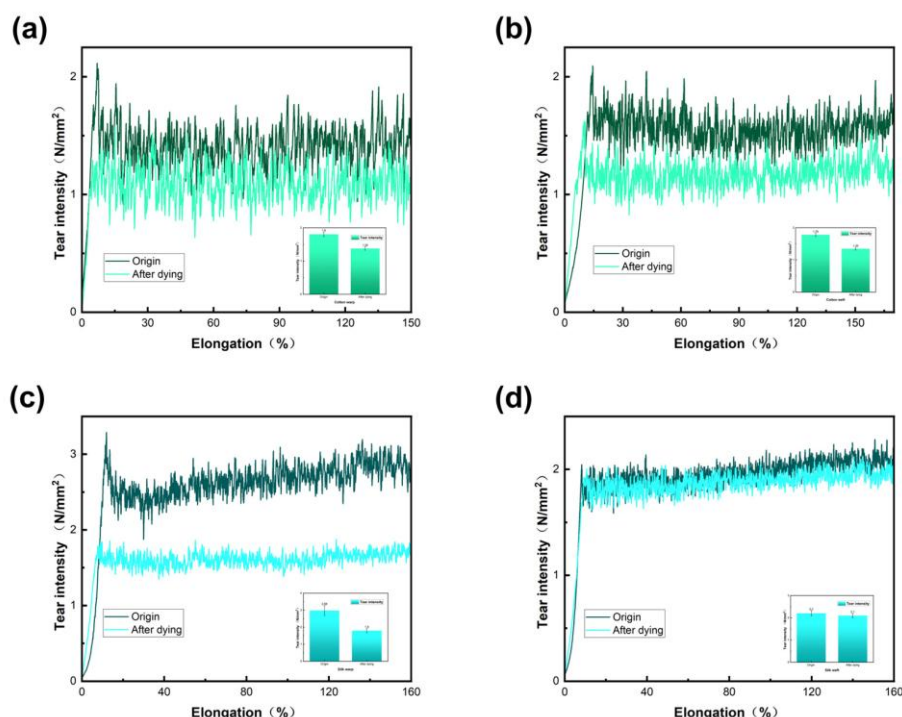


Figure 9. Comparison of tear properties of fabrics: (a) cotton warp, (b) cotton weft, (c) silk warp, (d) silk weft

Figure 10 shows the comparative analysis of the tensile strength of raw and dyed fabrics. After the high tensile strength test, the composite-colored cotton fabric decreased, but the high tensile strength of silk fabric increased to some extent. This is because in the high tensile strength test, the external force acting on the fabric is perpendicular (vertical) to the plane of the fabric. Due to the weave and arrangement of the fibers, the fabric has the ability to effectively distribute and resist external loads under high tensile strength. The decrease in the strength of cotton fabrics is due to their relatively loose weave structure and weak inter-fiber bonds. Although PVA (polyvinyl alcohol) glue strengthens the inter-fiber bonds to some extent, this reinforcement is not enough to compensate for the decrease in tensile strength due to the weak inter-fiber bonds. In contrast, silk fabrics exhibit high tensile strength when using PVA glue due to their high strength, abrasion resistance, dense weave structure and adhesive reinforcement.

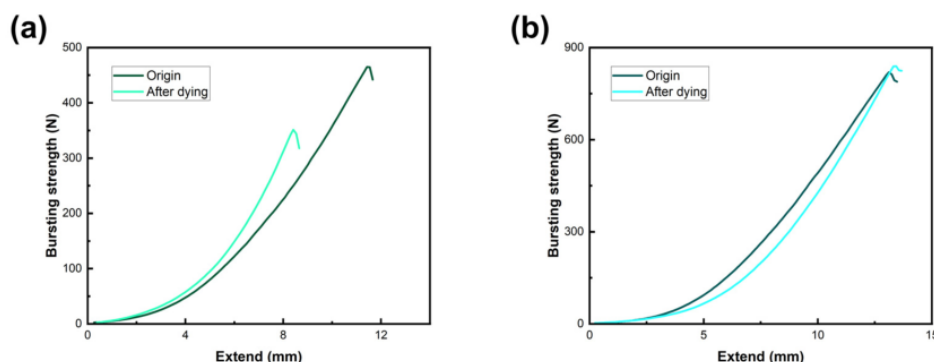


Figure 10. Comparison of the compression rupture properties of fabrics: (a) cotton, (b) silk

3.6. Cu₂O Structural Color Fabric Color Fastness

To test the color fastness of the synthesized structural colored fabrics, we used cotton fabric to evaluate the color fastness after cutting, rubbing and washing. Initially, the color fastness test after cutting was carried out according to GB/T 3917.2-2009. This standard specifies a method for determining the tearing strength of trouser-shaped test specimens (single tear method). The fabric was subjected to a cutting process, and the position of Cu₂O microspheres on the fabric surface near the cutting site was examined using scanning electron microscopy (SEM), as shown in Figure 11. The SEM images in Figure 11 show the state of the structural colored cotton fabrics after cutting. It can be seen that the Cu₂O microspheres are uniformly distributed in the area near the cutting site of the fabric. This observation indicates that the cutting process has little effect on the color fastness of the fabric.

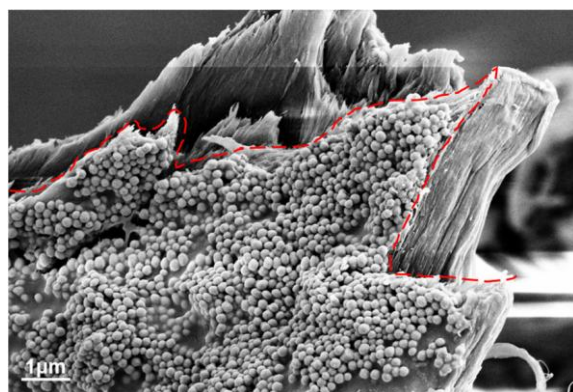


Figure 11. SEM image of a structurally colored cotton fabric after cutting ($\times 10,000$).

The abrasion resistance test was carried out in accordance with the European Union standard ISO 105-X12:2001 “Abrasion resistance in color fastness tests for fabrics”. The abrasion resistance of the fabric samples was tested using an abrasion resistance tester. As shown in Figure 12, after five abrasion tests, no obvious scratches or damage were found on the surface of the fabric sample, and the color remained unchanged. According to the test results, the abrasion resistance of the sample was rated at level 5 (the highest), indicating that the fabric fully meets the standard requirements for daily use.

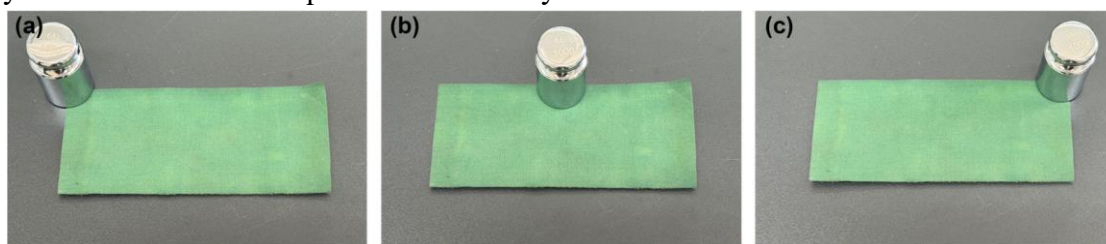


Figure 12. Comparison after friction resistance tests: (a) finished fabric, (b) fabric during friction, (c) fabric after friction.

As shown in Figure 13, the fabric samples with structural coloring on the surface did not lose their color even after 20 quick washes. This test shows that fabrics with composite colors have excellent washing fastness.

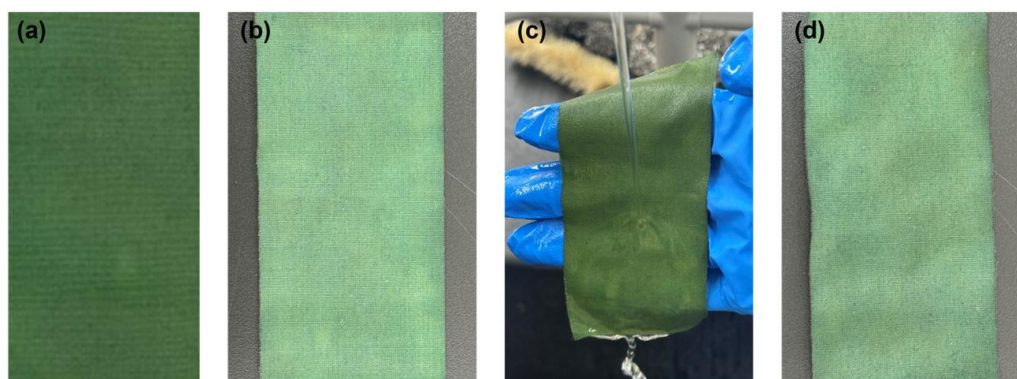


Figure 13. Comparison after washing fastness tests: (a) finished fabric, (b) fabric after 10 washing fastness tests, (c) fabric in washing fastness test (in process), (d) fabric after 20 washing fastness tests.

Conclusion

In this study, fabrics were prepared by spraying Cu_2O microspheres on two types of cotton and silk fabrics using PVA binder. It was found that structurally colored fabrics with green, yellow, brown or red Cu_2O microspheres could be produced by adjusting the ratio of citrate to Cu^{2+} during the Cu_2O synthesis process. Tensile test, tear test and elongation test were conducted to compare the changes in physical properties of cotton and silk fabrics. As a result of the abrasion and washing fastness tests, it was found that the structurally colored fabrics with Cu_2O microspheres had excellent cut, wash and abrasion resistance. As a result, this study provides important information for the development of structural color solutions for various fabrics.

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