

# Synergistic Effect of Silk Fibroin/Chlorogenic Acid/Citric Acid on Anti-ultraviolet and Antibacterial Properties of Cotton Fabric

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## Abstract

In order to obtain a durable anti-ultraviolet and antibacterial cotton fabric, silk fibroin, honeysuckle extract (chlorogenic acid) and citric acid were used to prepare a compound environment-friendly finishing agent, and then the cotton fabric was modified in this paper. Micro-morphology and properties were compared between cotton fabrics finished with a composite solution of silk fibroin/chlorogenic acid/citric acid and those finished first with silk fibroin then chlorogenic acid. Results showed that amidation and esterification crosslinking reactions occurred between the compounds and cotton fibers. Cotton fabric treated with the composite solution had higher UPF value (>90 after 30 launderings) and antibacterial rates (>95% for *Staphylococcus aureus* and *Escherichia coli*). There existed strong covalent bonds and good synergistic effects among silk fibroin, citric acid and chlorogenic acid, which could endow the cotton fabric with more durable anti-ultraviolet and antibacterial properties.

## Keywords

Silk fibroin, Chlorogenic acid, Citric acid, Cotton, Synergistic effect, Antibacterial, Anti-ultraviolet.

## 1. Introduction

In daily life, people will be exposed to a large number of harmful microorganisms, which will multiply in suitable environmental conditions and spread diseases through contact, affecting people's health. Secondly, ultraviolet radiation is a kind of light wave in sunlight which is harmful to human body. Excessive exposure to ultraviolet radiation will cause erythema and black spots on the skin, aging the skin, and even skin cancer. With the improvement of people's awareness of health and hygiene, antibacterial and anti-ultraviolet textiles are becoming more and more popular. Cotton fabrics are soft, comfortable to wear, moisture absorption and breathable, which are deeply loved by people, but there are also some defects, such as poor elasticity, easy to shrink, poor shape retention of clothing, poor anti-ultraviolet performance and so on. Therefore, it is necessary to improve the properties of cotton fabrics by using suitable finishing agents [1-3].

Silk fibroin is a natural protein material composed of a variety of amino acids, of which leucine can accelerate cell metabolism, serine and threonine can delay skin aging, and tryptophan and tyrosine can absorb ultraviolet rays.

Hence, silk fibroin serves as a prime ingredient in the production of diverse high-quality clothing fabrics and composite materials enriched with silk fibroin. This approach enables cotton, hemp, and chemical fibers to absorb natural protein amino acids, thereby conferring upon them remarkable attributes like skin-friendliness, high hygroscopicity, antistatic properties, elasticity, bulkiness, wrinkle resistance, and so forth. Consequently, these fibers acquire the surface properties of silk fibroin and emulate its silky luster. Various excellent functions of the silk fibroin are transferred to the edible fiber to protect the human skin, which is in line with the consumption concept of green environmental protection of people [4-9].

Chlorogenic acids are esters formed by the condensation of the hydroxyl group of quinic acid and the carboxyl group of trans-phenylacrylic acid, which have a virus-inhibiting effect, tumor proliferation, anti-inflammation and bacteriostasis, intestinal tract regulation, reproduction promotion, blood circulation promotion and blood pressure reduction, biological activity of prevent osteoporosis and so on. Chlorogenic acid is the main chemical component of honeysuckle, which can protect collagen from free radicals such

as active oxygen, effectively prevent ultraviolet damage to human skin, and endow fabrics with better performance. In recent years, the researches on chlorogenic acid have developed rapidly at home and abroad. How to extract the effective components of chlorogenic acid efficiently, how to cooperate with other chemical components to give full play to their biological activities, and how to use green plant extracts instead of chemical and physical processing products to finish fabrics will become the focus of attention in the textile field [10-12].

Some scholars have applied silk fibroin and plant extracts to the modification of fabrics respectively, but the binding fastness and washability of fabrics are both poor, some researchers have used plant extracts and other chemicals together in the finishing of fabrics, which improve the binding fastness, but cause some pollution to the environment [13-15]. In this paper, natural extracts with good human body affinity were used, and chemical substances were used as little as possible in the whole finishing process to reduce environmental pollution, moreover, silk fibroin and plant extracts were used simultaneously for functional modification of cotton fabrics, which have not been used by other researchers so far. In order to explore the synergistic

reaction effects among silk fibroin, honeysuckle extract (chlorogenic acid) and citric acid, two methods were used to treat cotton fabrics, one was to use silk fibroin/chlorogenic acid/citric acid composite solution to treat cotton fabrics, the other was to use silk fibroin/citric acid to treat cotton fabrics firstly, and then use chlorogenic acid to treat fabrics secondly. By comparing the structure and properties of cotton fabric after two different treatment methods, the reaction mechanism of silk fibroin/chlorogenic acid/citric acid complex solution with cotton fiber was studied, and the synergistic effect of silk fibroin/chlorogenic acid/citric acid complex solution on the anti-ultraviolet and antibacterial properties of cotton fabric was discussed. Our aim is to find an environmentally friendly and synergistic treatment process to make the fabric have natural and long-lasting functions, and provide a theoretical basis for the preparation of functional cotton products.

## 2. Experimental

### 2.1. Materials

Cotton fabric and Bombyx mori silk were purchased from Suzhou Youguantai Textile Co. Ltd. Dried Honeysuckle was provided by Yancheng Chunyu chemical Co. Ltd; All chemicals (CaCl<sub>2</sub>, HCl, C<sub>2</sub>H<sub>5</sub>OH, Na<sub>2</sub>CO<sub>3</sub>, citric acid, sodium hypophosphite) used for the following investigations were of analytical grade. Deionized water was used throughout the experiment.

### 2.2. Preparation of Silk Fibroin Solution

In order to remove the sericin protein in silk, 5g/L Na<sub>2</sub>CO<sub>3</sub> solution was prepared, the bath ratio was 1:50, and the silk was degummed twice in Na<sub>2</sub>CO<sub>3</sub> solution at 100°C for 60min each time. The calcium chloride-ethanol-water solution was prepared according to the molar ratio of 1:2:8. The degummed silk fabric was dissolved in the above solution at 80°C for 2h, and the silk fibroin solution was dialyzed in flowing pure water for 3 days with a dialysis bag. Finally, 3g/L HCl was

added to the silk fibroin solution, and the solution was degraded at 80°C for 2h to obtain silk fibroin solution [14-15].

### 2.3. Extraction of Chlorogenic Acid from Honeysuckle

The preparation method of the honeysuckle extract is the ethanol extraction method, and the main component extracted is chlorogenic acid. The main method was to prepare ethanol aqueous solution (the volume fraction of ethanol was 75%), put the dried honeysuckle into the ethanol aqueous solution, the solid-liquid ratio was 1:25 (g: mL), the extraction temperature was 90°C, the extraction time was 2h, and then filtered and dried to obtain chlorogenic acid [11]. The extraction rate of chlorogenic acid was 88.2%.

### 2.4. Treatment Process of Cotton Fabric Finished with Silk Fibroin/Chlorogenic Acid/Citric Acid Composite Solution

The silk fibroin, the chlorogenic acid and the citric acid were prepared into a composite finishing solution, wherein the mass ratio concentration of the silk fibroin was 8%, the concentration of the chlorogenic acid was 6%, the concentration of the citric acid was 8%, 1% of sodium hypophosphite was also added as a catalyst, and the bath ratio was 1:50. The cotton fabric was immersed in the finishing solution at 60°C for 45min, and then pre-baked at 80°C for 5min, baked at 160°C for 3min, and finally washed and dried.

### 2.5. Treatment Process of Cotton Fabric Finished first with Silk Fibroin and then with Chlorogenic Acid

Silk fibroin and citric acid were mixed into a finishing solution, in which the mass concentration of silk fibroin was 8%, the concentration of citric acid was 8%, 1% of sodium hypophosphite was

added as a catalyst, and the bath ratio was 1:50. The cotton fabric was immersed in the finishing solution at 60°C for 45min, then pre-baked at 80°C for 5min, baked at 160°C for 3min, and finally washed and dried. Then the chlorogenic acid solution was prepared, in which the mass ratio concentration of the chlorogenic acid was 6%, and the concentration of the citric acid was 8%, the silk fibroin/citric acid modified cotton fabric was subjected to secondary treatment with the chlorogenic acid solution, the treatment temperature was 60°C, the treatment time was 45min, then pre-baked at 80°C for 5min, baked at 160°C for 3min, and finally washed, dried and tested.

### 2.6. Scanning Electron Microscopic (SEM) Analysis

The surface morphology of cotton fabrics was observed by the Quanta 200 scanning electron microscope. The cotton fiber was gold sputtered and given the electronic conductivity under a vacuum prior to the observation. The measurements were performed at 20°C and the relative humidity of 65%.

### 2.7. Fourier Transform Infrared (FT-IR) Spectroscopy

Nicolet 6700 FT-IR spectrophotometer was used to observe the infrared spectra of cotton fabrics with the traditional transmission technique of KBr pellets. The measurements were performed at 20°C and a relative humidity of 65%, and the scanning wavenumber range was 400-4000cm<sup>-1</sup>.

### 2.8. UV Resistance and Washing Durability Test of Cotton Fabrics

GB/T 18830-2002 《Evaluation of Textiles Anti-ultraviolet Properties》 was referenced to evaluate the UV resistance of cotton fabrics before and after treatment. YG(B)912E anti-ultraviolet tester was used to test the ultraviolet transmittance of cotton fabrics, and the

ultraviolet protection factor (UPF) was used to evaluate the anti-ultraviolet effect of fabrics. The higher the UPF value, the better the UV resistance of the fabric. The washing durability of the treated cotton fabric against repeated launderings was evaluated according to AATCC 124-2010 «Smoothness appearance of fabrics after repeated home laundering».

## 2.9. Antibacterial Properties Test of Cotton Fabrics

GB/T 20944.3-2008 «Evaluation of Antibacterial Properties of Textiles-Part 3:Oscillation Method» was referenced to evaluate the antibacterial properties of cotton fabrics before and after treatment. The oscillating flask method was adopted and the antibacterial rate was calculated. *Staphylococcus aureus* (S. aureus/ATCC 6538) and *Escherichia coli* (coli/ATCC 25922), the gram-positive and gram-negative bacteria commonly found on the human body, were chosen as the tested bacteria.

## 3. Results and Discussion

Three kinds of cotton fabrics were prepared, including the untreated cotton fabric (short for No.1 cotton fabric in the following paper), the cotton fabric finished by silk fibroin first and then by chlorogenic acid (short for No.2 cotton fabric), and the cotton fabric finished by silk fibroin/chlorogenic acid/citric acid composite solution (short for No.3 cotton fabric), then the micro-morphology, antibacterial and anti-ultraviolet properties of three kinds of cotton fabrics were researched and compared, and the synergistic effect of silk fibroin, citric acid and chlorogenic acid on the properties of cotton fabrics was explored.

### 3.1. FTIR Spectroscopy of Cotton Fabrics

Figure 1 was an infrared spectrogram of three cotton fabrics. By comparing the three curves, it was observed that the O-H bending vibration peak of the adsorbed

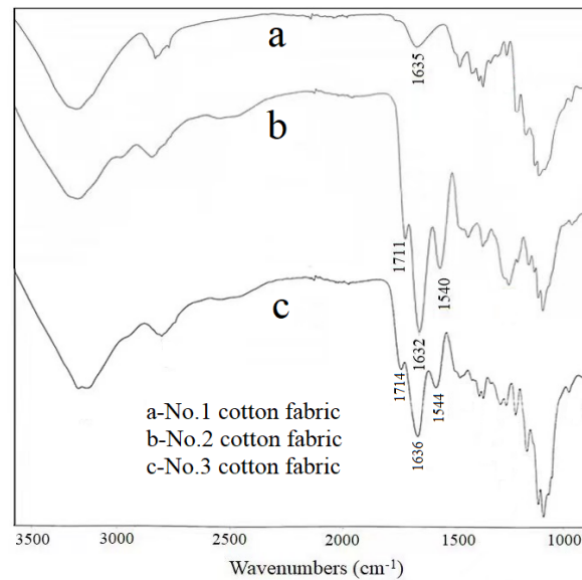


Fig. 1. Infrared spectrum of cotton fabrics before and after treatment

water treated by both methods at  $1635\text{ cm}^{-1}$  showed significant enhancement compared to the untreated fabric, indicating a substantial improvement in the affinity of the treated cotton fabrics towards water molecules. Obvious stretching vibration absorption peaks of non-cyclic ester C=O appeared at  $1711\text{ cm}^{-1}$  and  $1714\text{ cm}^{-1}$  in B and C curves respectively, and new characteristic peaks—stretching vibration absorption peaks of amide C-N appear at  $1540\text{ cm}^{-1}$  and  $1544\text{ cm}^{-1}$  respectively [14-16]. To sum up, the amidation and esterification crosslinking reactions occurred in the two finishing methods of cotton fabric: The amino group in the silk fibroin can have an amidation reaction with the carboxyl group in the citric acid to generate an amide bond, the carboxyl group in the silk fibroin can have an esterification reaction with the hydroxyl group in the cotton cellulose molecule to generate an ester bond, the carboxyl group in the chlorogenic acid can have an esterification reaction with the hydroxyl group in the cellulose molecule. So after high temperature baking, the silk fibroin, the chlorogenic acid and the citric acid have undergone amidation and esterification crosslinking reactions with the cotton cellulose macromolecules, thus

forming a firm covalent bond. In addition, there are a lot of amide bonds in silk fibroin molecules, so the characteristic peaks of amide bonds on B and C curves may also be related to the grafting of silk fibroin onto cotton fibers. The structural formulas of the cotton cellulose, the silk fibroin, the chlorogenic acid and the citric acid are as follows:

Figure 2 was the longitudinal scanning electron micrograph of three cotton fibers. Comparing the three figures, it could be seen that the surface of cotton fiber before treatment was relatively smooth, while the surfaces of cotton fibers treated by the two methods were relatively rough. This was mainly due to the formation of chemical bonds among silk fibroin, chlorogenic acid, citric acid and cotton fibers, in the process of washing, part of the weak structure was washed away, resulting in the stripping of cotton fibers, and leaving a notch on the surface of fibers [15]. Comparing figure b and figure c, it could be found that the surface of the fiber in figure c was smoother, while the etching of the fiber in figure b was more serious, mainly because No.2 cotton fabric had been soaked twice and baked twice at high temperature, and the peeling effect of the fiber was more serious than that in figure c. In addition, when silk fibroin/chlorogenic acid/citric acid composite solution was used to finish cotton fabric,

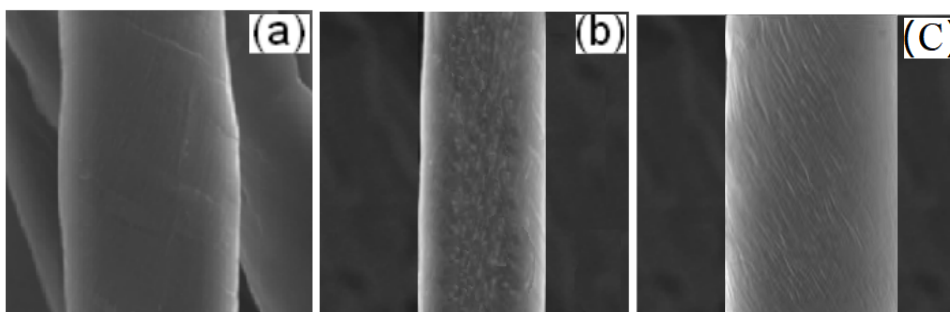
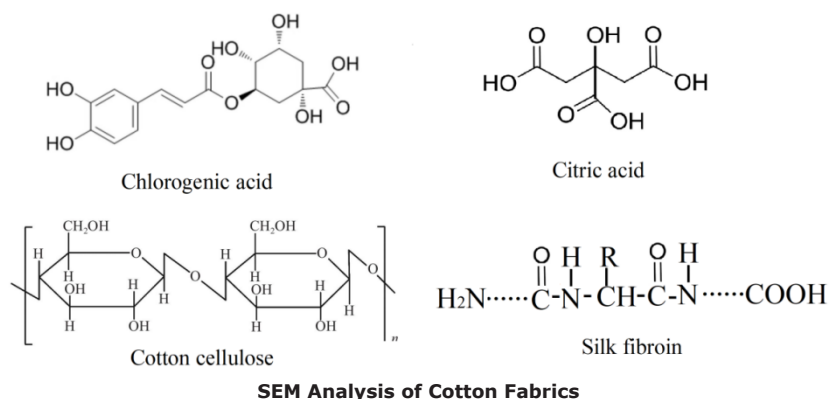


Fig. 2. SEM photos of cotton fabrics (a) No.1 cotton fabric; (b) No.2 cotton fabric; (c) No.3 cotton fabric ( $\times 3000$ )

there existed the synergistic effect among them, and chlorogenic acid could interact with the amino acid residues of silk fibroin by hydrogen bonding and Van der Waals force, the synergistic effect and the interaction might be more conducive to the formation of silk fibroin film on the fiber surface, thus making the No.3 fiber surface smoother.

### 3.2. UV Resistance and Washing Durability of Cotton Fabrics

The UPF value of No.1, No.2 and No.3 cotton fabrics were tested and compared, as well as the UPF value of No.2 and No.3 cotton fabrics after 5, 10, 20 and 30 repeated launderings, respectively.

Figure 3 showed that the UPF values of the cotton fabric finished by the two methods were both significantly improved. Silk fibroin is rich in protein, among the 18 amino acids, leucine can accelerate cell metabolism, serine and threonine can delay skin aging, and tryptophan and tyrosine can absorb ultraviolet rays. Therefore, the UPF

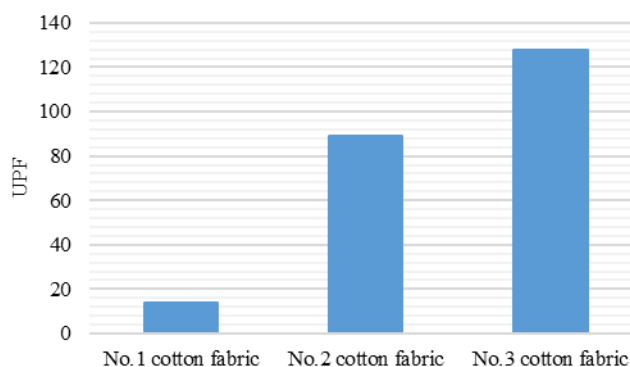


Fig. 3. UPF value of No.1, No.2 and No.3 cotton fabrics

value of silk fibroin can be significantly improved by finishing cotton fabrics with silk fibroin. The main ingredient of honeysuckle extract is chlorogenic acid, a natural polyphenol substance, which can protect collagen from free radicals such as active oxygen and effectively prevent ultraviolet damage to human skin. Therefore, the UPF value of cotton fabric finished by silk fibroin and honeysuckle extract was significantly improved. According to the whiteness test data, the whiteness value of No.2 and No.3 cotton fabrics did not change much, indicating that the treated cotton fabric did not have obvious yellowing.

Figure 3 showed that the UPF value of No.3 cotton fabric was much larger than that of No.2 cotton fabric, indicating that there might exist a synergistic effect among silk fibroin, citric acid and chlorogenic acid, which was more conducive to improving the anti-ultraviolet performance of the fabric. Figure 4 showed that the UPF value of No.2 cotton fabric tended to decrease with the increase of washing times, but after 30 times of washing, the UPF value was still greater than 60. From figure 5, we could see that the UPF value of No.3 cotton fabric was still larger than 90 after 30 times of washing, that was, it still had

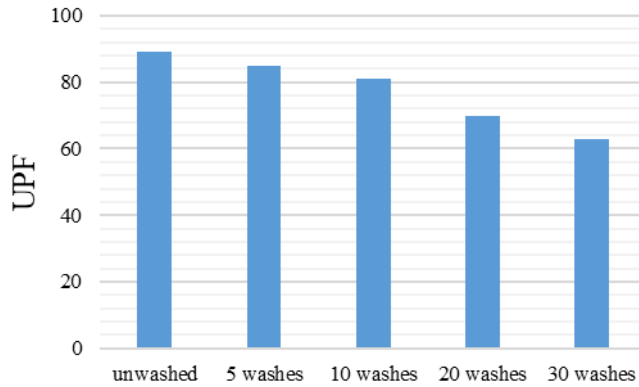


Fig. 4. UPF value of No.2 cotton fabric after washes

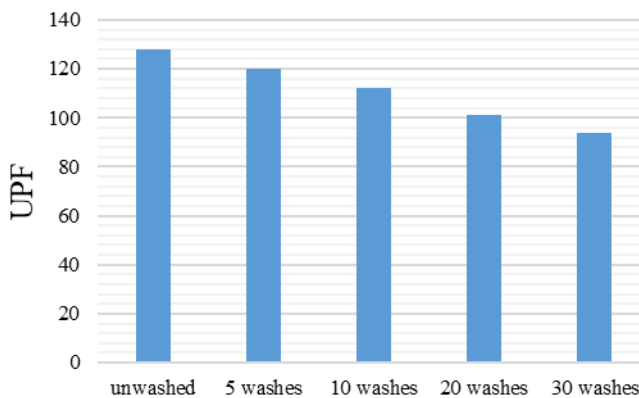


Fig. 5. UPF value of No.3 cotton fabric after washes

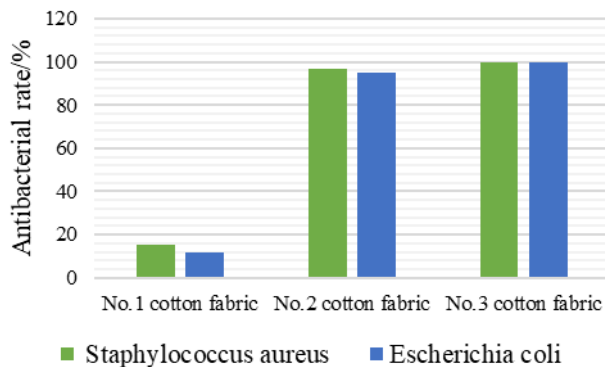


Fig. 6. Antibacterial rate of cotton fabrics to *Staphylococcus aureus* and *Escherichia coli*

good anti-ultraviolet performance, which fully met the performance requirements of anti-ultraviolet textiles. The results showed that the washing durability of cotton fabrics treated by silk fibroin/chlorogenic acid/citric acid composite solution was better, and the covalent bonds among them were stronger, which might also be related to the synergistic effect of silk fibroin, chlorogenic acid and citric acid.

### 3.3. Antibacterial and Washing Durability of Cotton Fabrics

The antibacterial rates of No.1, No.2 and No.3 cotton fabrics to *Staphylococcus aureus* and *Escherichia coli* were tested and compared, and the antibacterial rates of No.2 cotton fabric and No.3 cotton fabric were tested and compared after washing for 5 times, 10 times, 20 times

and 30 times, respectively. The results were shown in figure 6, 7 and 8.

Figure 6 showed that the antibacterial rate of the untreated No.1 cotton fabric was very low, while the antibacterial rates of the No.2 and No.3 cotton fabrics to *Staphylococcus aureus* and *Escherichia coli* were both significantly improved, and the antibacterial rates of No.3 cotton fabric were both 100%. It is mainly because that the main component of honeysuckle is chlorogenic acid, which has significant antibacterial effect. It has strong inhibitory effect on both Gram-positive and Gram-negative bacteria, which may be due to the non-competitive binding of chlorogenic acid to arylamine acetyltransferase in bacteria, thus inhibiting the proliferation of bacteria. In addition, chlorogenic acid can also destroy the structure of bacterial cell wall and cell membrane in a short time, increase the permeability of cells, resulting in the leakage of cell electrolytes, enzymes, DNA and RNA, thus affecting the stability of cell structure and causing the gradual death of cells [15].

Figure 7 and Figure 8 showed the antibacterial rates of No.2 and No.3 cotton fabrics after 30 times of washing. They showed that the antibacterial rates tended to decrease with the increase of washing times, the antibacterial rate of No.3 cotton fabric was still greater than 95% after 30 washes, and its antibacterial property was much better than that of No.2 cotton fabric, indicating that the firm covalent bonds were formed among silk fibroin, chlorogenic acid, citric acid and cotton fibers during the composite finishing, so the washing resistance of No.3 cotton fabric was much better than that of No.2 cotton fabric. It was further proved that there was a good synergistic effect among silk fibroin, chlorogenic acid and citric acid, which made the cotton fabric have more durable antibacterial property.

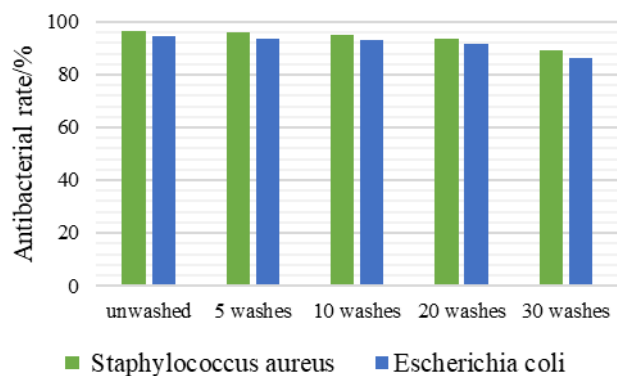


Fig. 7. Antibacterial rate of No.2 cotton fabric after washes

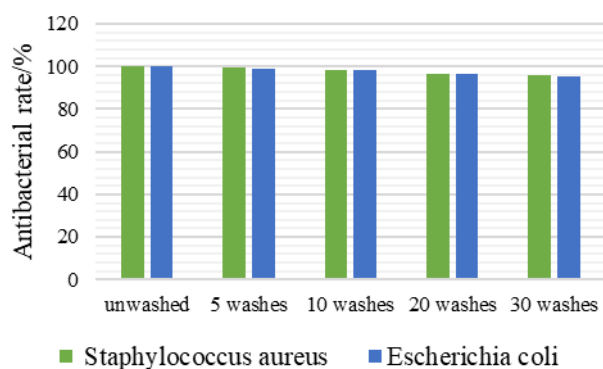


Fig. 8. Antibacterial rate of No.3 cotton fabric after washes

### 3.4. Analysis of the Synergistic Effect of Silk Fibroin, Chlorogenic Acid, and Citric Acid on the Properties of Cotton Fabric

Silk fibroin, chlorogenic acid, citric acid and cotton fiber can undergo esterification and amidation crosslinking reactions. In addition, compounds in chlorogenic acid can interact with amino acid residues of silk fibroin by hydrogen bonding or Van der Waals force. These interactions may change the conformation or stability of silk fibroin and increase its reactivity, thereby synergistically affecting and altering the function and properties of the cotton fabric. Citric acid can enhance the adhesion and stability of silk fibroin and chlorogenic acid on cotton fabrics, prevent their shedding and loss in the use process, and thus improve the stability of

cotton fabrics. At the same time, citric acid has the ability to dissolve and chelate metal ions, and the honeysuckle extract may contain trace metal ions, so citric acid can complex with the metal ions in the honeysuckle extract to form a stable complex, reduce the content of metal ions on the surface of cotton fabrics, reduce the occurrence of photooxidation reaction, and thus improve the anti-ultraviolet performance of fabrics. In addition, silk fibroin molecules have excellent strength and toughness, which can increase the tensile and wear resistance of cotton fabrics and make them have longer service life. The active ingredients in the honeysuckle extract can interact with the surface of the cotton fiber to form a protective film, so that the durability of the cotton fabric is improved. The application of citric acid enhances the softness and glossiness of cotton fabric,

resulting in a more comfortable texture. Furthermore, the silk fibroin/chlorogenic acid/citric acid composite finishing cotton fabric exhibits superior long-lasting antibacterial properties and ultraviolet resistance due to the synergistic effect among these components [7,11,14-15].

## 4. Conclusion

Silk fibroin, chlorogenic acid and citric acid were used to prepare a durable anti-ultraviolet and antibacterial compound finishing agent for cotton fabric. When silk fibroin/chlorogenic acid/citric acid composite solution was used to finish cotton fabric, there existed the synergistic effect and the interaction among them, which might be more conducive to the formation of silk fibroin film on the fiber surface, so the surface of cotton fiber was much smoother. The UPF value of cotton fabric finished with silk fibroin/chlorogenic acid/citric acid composite solution reached 128, and the antibacterial rate to *Staphylococcus aureus* and *Escherichia coli* reached 100%. After 30 times of washing, the UPF value of the fabric was still above 90, and the antibacterial rate was still more than 95%. Compared to using silk fibroin followed by chlorogenic acid for finishing treatment, employing a composite solution consisting of silk fibroin/chlorogenic acid/citric acid provided superior washing durability due to stronger covalent bonds formed among these components. Thanks to their remarkable synergistic effect, combining silk fibroin, chlorogenic acid, and citric acid yielded cotton fabrics with exceptional long-lasting antibacterial properties and ultraviolet resistance. In future research endeavors, this combination can also be applied in modifying chemical fibers to enhance their wearability.

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