Effect of organosilicon resin containing disulfide bonds on elastic properties of cotton fabric

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Abstract Cotton fabrics are popular for their excellent properties, but their property of creasing easily largely limits the wide application of cotton fabrics. In this paper, an organosilicon resin emulsion containing disulfide bonds was prepared with epoxy-terminated silicone oil and 4,4-diaminodiphenyl disulfide (AFD) as raw materials, and it was used as a finishing agent for the elastic finishing of cotton fabrics. The effects of raw material ratio on the elastic properties of organosilicon polymer film and the effects of emulsifier dosage and emulsification time of emulsion polymerization on the properties of organosilicon resin emulsion were studied. In addition, the effects of organosilicon finishing agent dosage, curing temperature, and curing time on the elastic properties of cotton fabric were also studied. The research results show that the average particle size of the prepared organosilicon resin emulsion is about 250 nm, the particle size distribution is concentrated and the emulsion is stable. The delayed elastic recovery angle of the cotton fabric treated with an organosilicon finishing agent can reach 185°, showing excellent high delayed elasticity. At the same time, it was found that the finished cotton fabric exhibited an excellent and special temperature-responsive shape memory function based on the existence of disulfide bonds in the organosilicon polymer network structure.

Keywords Organosilicon resin · Disulfide bonds · Cotton · Shape memory · High delayed elasticity

Introduction

Cotton fiber has accompanied human development for thousands of years. Because of its excellent wearing properties such as breathability, moisture absorption, and softness, cotton fabric is deeply favored by consumers (Han 2018). However, the unique structure of cotton fiber makes it easy to wrinkle during use (Luo et al. 2021), thus affecting the beauty and smoothness of the fabric, which greatly limits the wide application of cotton fabric. With the development of technology and the changes of consumers’ aesthetic requirements, in recent years, the demand for improving the high delayed elasticity properties and shape memory properties of cotton fabrics
The disulfide bond is a relatively stable reversible covalent bond, but under certain conditions, the chemical bond connecting two sulfur atoms can break and recombine. The concept of dynamic disulfide bond was first proposed in glass-like polymer theory (Montarnal et al. 2011). This dynamic covalent bond structure will undergo a reversible response under external stimuli such as moderate temperature (60–90 °C) and mechanical force, resulting in the rearrangement of the cross-linked network, so that the material exhibits repairable properties similar to thermoplastic materials, shape change performance and machinability (Zhang 2016). A highly stretchable self-healing elastomer has been prepared by simultaneously combining aromatic disulfide and imine bonds into a polydimethylsiloxane network (Lv et al. 2018).

To make the creases of the fabric under the action of external force recover slowly over time, and improve the wearing performance of the cotton fabric, in this study, we use epoxy-terminated silicone oil and 4,4-diaminodiphenyl disulfide (AFD) as raw materials to synthesize an organosilicon resin finishing agent containing disulfide bond by emulsion polymerization. In addition, the properties of organosilicon resin films and their effect on the elastic properties of cotton fabric were also studied.

Material and methods

Reagents and instruments

Reagents

Epoxy-terminated silicone oil and 4,4-diaminodiphenyl disulfide (AFD) were provided by Anhui Keguang New Materials Co., Ltd.; Ethyl acetate is provided by Sinopharm group; The positive/non-ionic compound emulsifier was made by our laboratory; The cotton fabric (fabric parameters: CM100/2*160*90*57/58) is provided by Zhejiang Meixinda Textile printing and dyeing Technology Co., Ltd.
**Instruments**

Fourier transform infrared spectrometer Nicolet IS10 (Thermo Fisher Technology Co., Ltd.), Dynamic Thermomechanical Analyzer DMA Q800 (TA Instruments Co., Ltd.), Homogenizer ATS (Antos Nano Technology Co., Ltd.), Nanoparticle size analyzer Zetasizer Nano ZS (Malvern Instrument Co., Ltd.), Scanning electron microscope TM-1000 (Hita-Chi Co., Ltd.), Fabric heat setting machine UPEI (Taiwan (Xiamen) Rabbi Co., Ltd.), Automatic laser fabric wrinkle elasticity tester YG541 (Ningbo Textile Instrument Factory), Electronic balance AL104 (Mettler Toledo Instruments Co., Ltd.), Constant temperature heated magnetic stirrer DF-101S (Gongyi Yuhua Instrument Co., Ltd.), Electric heating constant temperature blast drying oven DHG-9070A (Sapeen Scientific Instruments Co., Ltd.).

Preparing organosilicon polymer film containing disulfide bonds

Organosilicon macromolecular polymers containing disulfide bonds were prepared by ring-opening reaction of 4, 4-diaminodiphenyl disulfide (AFD) with epoxy-terminated silicone oil. As shown in Fig. 1, when the molar ratio of the reactants is 1:1, the linear macromolecular product (Fig. 1a) is mainly formed. When the ratio of AFD is reduced and the epoxy-terminated silicone oil is excessive, another hydrogen on the amino group in AFD also participates in the reaction, and then the network macromolecular polymer (Fig. 1b) is obtained.

A certain amount of AFD was weighed, a small amount of ethyl acetate was added to dissolve it completely, and an appropriate amount of epoxy-terminated silicone oil was added dropwise to mix the reactants evenly, and then heated to 80 °C for 0.5 h in a water bath. The organosilicon prepolymer was then poured into the preheated PTFE template and slowly heated to 120 °C for 2 h, then heated to 150 °C for 1 h. Finally, it was naturally cooled to room temperature and demolded to obtain an organosilicon polymer film containing dynamic disulfide bonds.

Preparation of organosilicon resin emulsion containing disulfide bonds

As shown in Fig. 2, according to the proportion, a certain amount of AFD was weighed and dissolved in a small amount of ethyl acetate, and an appropriate amount of epoxy-terminated silicone oil was added dropwise, mixed evenly, and placed in a water bath at 80 °C for 0.5 h. After the reaction, the organosilicon prepolymer is obtained. A certain amount of positive/non-ionic compound emulsifier was added into the prepolymer, which was firstly stirred at low speed and then homogenized at high speed for 0.5 h. Then the emulsion was placed in a water bath at 80 °C and stirred at low speed for 6 h to obtain organosilicon resin emulsion containing disulfide bonds.

Application of organosilicon resin emulsion

The cotton woven fabric was immersed in the organosilicon resin emulsion finishing agent previously prepared, the fabric was soaked and rolled twice (wet pick up of 80%). The finishing process was completed by drying and then curing on the heat-setting machine.

![Fig. 1 Reaction equation of organosilicon polymer](image-url)
Examination

**Infrared spectral**

The prepared organosilicon polymer film was analyzed by the infrared spectrometer with Nicolet IS10 infrared spectrometer. In the spectral range of 500–4000 cm\(^{-1}\), the infrared spectrum of the organosilicon polymer film was obtained by scanning 32 times with a resolution of 5 cm\(^{-1}\).

**Mechanical properties**

The mechanical properties of the organosilicon polymer film were tested by DMA Q800 dynamic thermomechanical analyzer. At 25 °C, the sample was stretched at 30%/min to 120% of the deformation, and the stress–strain and stress-recovery properties of the elastic film were tested by tension film clamp.

**Emulsion performance**

The organosilicon resin emulsion was tested by Nano ZS nano-particle size and potential analyzer. The sample was diluted 1000 times to a nearly transparent and colorless solution, and the particle size and Zeta potential of the emulsion were tested.

**Scanning electron microscopy**

The surface morphology of the comparison sample (unfinished cotton) and finished cotton samples were analyzed by TM-1000 scanning electron microscope. After the standard sample preparation was completed, gold spraying was carried out under the conditions of vacuum degree of 9–11 (10\(^{-1}\) mmHg) and current of 9–13 mA. The test was carried out after spraying gold for 90 s.

**Wrinkle recovery angle of cotton fabric**

According to “Textiles—Determination of the recovery from creasing of a folded specimen of fabric by measuring the angle of recovery” (ISO:2313-1 2021), the recovery angle of cotton samples was tested by YG541 fabric wrinkle elasticity tester. Cotton samples were folded end to end and loaded for 5 min. The crease recovery angle of a folded sample at 15 s ± 1 s after the load was removed, expressed as rapid elasticity recovery angle; or the crease recovery angle.
of a folded sample at 5 min ± 5 s after the load was removed, expressed as delayed elasticity recovery angle.

**Temperature-responsive shape memory**

The cotton fabric was cut into a size of 40 mm × 20 mm, and the two ends of the sample were clamped with clips to keep it in U shape, and then placed in an oven at 85 °C. After 10 min, the sample was taken out and cooled to room temperature. The clip was removed, and the U-shaped opening length $L_0$ of the comparison sample and the U-shaped opening length $L$ of the finished cotton sample were tested. By comparing the opening lengths of the samples, the shape memory degree of the finished sample in response to temperature is reflected.

**Results and discussion**

**Preparation of organosilicon polymer containing disulfide bonds**

According to the reaction equation in Fig. 1, a certain amount of AFD and epoxy-terminated silicone oil were weighed in a molar ratio of 1:0.8, and placed in a water bath at 80 °C for 0.5 h to synthesize the organosilicon prepolymer. Then it was transferred into a polytetrafluoroethylene template for further polymerization at a high temperature to obtain the organosilicon polymer film. The infrared spectra of the reaction product organosilicon polymer film and the reaction raw materials are shown in Fig. 3.

As shown in Fig. 3, in the FTIR spectrum of the epoxy-terminated silicone oil, the stretching vibration peak of the epoxy group (C–O–C) is at 912 cm$^{-1}$ (Wei et al. 2022). In the FTIR spectrum of AFD, at 3300–3500 cm$^{-1}$, there are typical double absorption peaks of stretching vibration of the amino group (–NH$_2$) (Rao et al. 2020). They all disappeared in the FTIR of the reaction product.

In the FTIR spectrum of the reaction product, the characteristic absorption peak of the hydroxyl group (–OH) formed by the ring-opening reaction of the epoxy group appeared at 3361 cm$^{-1}$ (Watanabe et al. 2016). At 1593 cm$^{-1}$ and 520 cm$^{-1}$, the skeleton vibration absorption peak of the benzene ring and the absorption vibration peak of the disulfide bond (S–S) (Tiansheng et al. 2002) appeared similar to those in AFD, respectively. At 1042 cm$^{-1}$, there is the antisymmetric stretching vibration peak of the silicon-oxygen bond (Si–O–Si) (Rao et al. 2020) similar to that in the organosilicon polymer chain. The results of FTIR spectral analysis show that the chemical reaction between epoxy-terminated silicone oil and AFD occurs as shown in Fig. 1, which indicates organosilicon polymer containing disulfide bonds was synthesized.
Effect of raw material ratio on the resilience of organosilicon polymer film

According to the reaction equation shown in Fig. 1, the organosilicon polymer film was prepared with the raw materials molar ratios of epoxy-terminated silicone oil and AFD as 1:1, 1:0.9, and 1:0.8, respectively. The elastic properties of the organosilicon polymer film were tested with the dynamic thermomechanical analyzer DMA Q800. The organosilicon polymer film was stretched to 120% deformation at a deformation rate of 30%/min. After the external force was removed, the organosilicon polymer film rebounded quickly and then underwent a slow recovery process until the deformation no longer changed. The recovery of film at 0–15 S after removing the external force was defined as the rapid elasticity recovery stage, the recovery of film at 15 S–15 min was defined as the delayed elasticity recovery stage, and the remaining unrecovered deformation was defined as the permanent deformation. The recovery of the organosilicon polymer film after being stretched by the external force is shown in Fig. 4.

It can be seen from Fig. 4 that the deformation rate of rapid elasticity recovery of organosilicon polymer film prepared by different raw materials is very high, indicating that the synthesized organosilicon resin has high elasticity. As the proportion of AFD in the raw material decreases, the deformation rate of delayed elasticity recovery of the organosilicon polymer film gradually increases, and the residual permanent deformation of the polymer film decreases. This is because in the reaction system with the raw material molar ratio of 1:1.0, more linear macromolecules (Fig. 1a) are synthesized by the reaction between the epoxy-terminated silicone oil and AFD. The intermolecular force of this linear macromolecule is weak. When subjected to external force, the straight chains of macromolecules easily slide relative to each other, and the center of gravity of macromolecules moves, so the deformation is difficult to recover.

When the ratio of AFD decreases, the reaction synthesizes more network macromolecules (Fig. 1b). In the synthetic organosilicon polymer, the molecular weight of the polymer increases, and the cross-link density between the macromolecular chains also increases. When subjected to external force, the polymer macromolecular segments slip with each other. However, due to the large molecular weight of macromolecules and the cross-linking of disulfide bonds, the center of gravity of macromolecules is not easy to move. When the external force is removed, the macromolecular segments in the organosilicon polymer film are affected by the interaction force between the molecular chains of the network structure, and gradually return to the original state of the segments. Therefore, the more complex the macromolecular chain network cross-linked structure of the organosilicon polymer film is, the better the delayed elasticity recovery performance of the polymer film and the less the permanent deformation will be. When the raw material molar ratio is 1:0.8, the proportion of permanent deformation is only 0.41%.

Effect of the amount of emulsifier on the performance of organosilicon resin emulsion

According to the preparation process in Fig. 2, the organosilicon resin emulsion with a solid content of 30% was prepared with the raw material molar ratio of 1:0.8. The ratio of emulsifier to raw materials respectively was 6.67%, 13.33%, 20%, 26.67%, 33.33%, and the emulsifying time was 30 min. The performance of the prepared organosilicon resin emulsion is shown in Fig. 5.

It can be seen from Fig. 5, that when the dosage of the emulsifier is 6.67%, the particle size and polydispersity index (PDI) value of organosilicon resin emulsion are large, and the prepared emulsion is unstable.
and prone to delamination. With the increase of the amount of emulsifier, the particle size of the emulsion showed a decreasing trend. When the amount of emulsifier was 26.67%, the particle size and PDI value of the emulsion reached the lowest value, and the emulsion showed high stability. When the amount of emulsifier continues to increase, the particle size and PDI value of emulsion tend to increase. It shows that in this emulsion system, increasing the amount of emulsifier can increase the adsorption of surfactant on the interface of organosilicon resin particles, reduce the interfacial tension of emulsion particles, and reduce the interfacial energy of the emulsion system. Thus, the particle size of the emulsion particles is reduced and the stability of the emulsion is improved. However, excessive use of emulsifiers may be unfavorable for the close arrangement of surfactants on the surface of emulsion particles. The particle size distribution of the emulsion particles is uneven, and the PDI value increases, thereby affecting the stability of the emulsion.

By detecting the Zeta potential of the emulsion particles, it is found that the emulsion particles have a positive charge due to the use of cationic surfactant. This increases the charge repulsion between the emulsion particles, improves the stability of the emulsion, and is also beneficial to the adsorption of the emulsion particles on the cotton fiber surface. With the increase of the amount of emulsifier, the Zeta potential of the emulsion particles increases, and the stability of the emulsion is also improved. However, when the amount of emulsifier is 33.3%, the Zeta potential decreases, which needs to be studied in the future.

Effect of emulsifying time on the performance of organosilicon resin emulsion

According to the preparation process in Fig. 2, the organosilicon resin emulsion with a solid content of 30% was prepared with the raw material molar ratio of 1:0.8. The proportion of the emulsifier is 26.67%, and the high-speed homogeneous emulsifying time is 10, 20, 30, 40, and 50 min respectively. The performance of the prepared organosilicon resin emulsion is shown in Fig. 6.

As shown in Fig. 6, with the extension of homogeneous emulsifying time, the particle size and PDI value of the emulsion decreased greatly. When homogeneously emulsified for 20 min, the particle size and PDI value of the emulsion tended to be stable. It can be seen that prolonging the emulsifying time can increase the mechanical energy provided to the emulsion system, making the emulsion particles smaller and more uniform in size. After 20 min of emulsification, the emulsion particles can reach the size of 200–300 nm. And the particle size of the emulsion no longer decreases when the emulsification time continues to be extended. Therefore, the excessive extension of the emulsification time has no visible significance in improving the performance of the emulsion.

The Zeta potential of the emulsion gradually increased to a stable value with the extension of emulsifying time. When the emulsion was emulsified for 20 min, the Zeta potential of the emulsion reached +46.6 mV. Continuing to prolong the time, the Zeta potential of the emulsion no longer increased.
Effect of organosilicon finishing agent concentration on the elastic properties of cotton fabric

The application process is as described in “Application of organosilicon resin emulsion” section. The cotton fabric was immersed in different concentrations of organosilicon resin emulsion finishing agents. After the cotton fabric was padded and dried, it was cured at 160 °C for 6 min. Then according to ISO 2313-1:2021, measure the rapid and delayed elasticity recovery angle of the finished cotton fabric. The results are shown in Table 1.

As can be seen from Table 1, compared with the comparison sample, the crease recovery angle of cotton fabric after being finished with organosilicon resin emulsion showed a trend of first increasing and then slightly decreasing. When the concentration of the finishing agent is 30 g/L, the increase ratio of the rapid and delayed elasticity recovery angle of finished cotton fabric reaches the maximum. Continue to increase the concentration of the organosilicon resin finishing agent, and the wrinkle recovery angle of cotton fabric tends to decrease. We believe that the organosilicon resin emulsion finishing agent forms a layer of elastic polymer film on the surface of cotton fiber under the condition of high-temperature curing, which improves the elasticity of cotton fibers. Thus, the elasticity of the cotton fabric is improved, and the recovery angle of rapid and delayed elasticity is also improved. However, when the concentration of the finishing agent is too high, a thicker polymer film is deposited on the fiber surface. At the same time, more “fiber cross-linking points” bonded by the polymer film will be formed in the tissue structure of finished cotton fabrics. In this case, the increase in fiber diameter changes the elasticity of the cotton fiber. Moreover, the “fiber cross-linking points” formed by the deposition of excessive finishing agents weaken the removability of the fiber under external force. This reduces the elastic recovery of the cotton fabric after the external force is removed. Therefore, the crease response angle of cotton fabric is reduced and the elasticity is decreased. To sum up, when the concentration of the finishing agent is 30 g/L, the cotton fabrics can obtain better rapid and delayed elasticity performance.

Effect of curing temperature on the elastic properties of cotton fabric

The cotton fabric was immersed in the organosilicon resin emulsion finishing agent with a concentration of 30 g/L. After the cotton fabric was padded and dried, it was cured at 130, 140, 150, 160, 170, and 180 °C for 6 min, respectively. Then the rapid and delayed elasticity recovery angles of the finished cotton fabric were measured. The results are shown in Table 2.

It can be seen from Table 2, that with the increase of the curing temperature (from 130 to 150 °C), the rapid and delayed elasticity recovery angles of cotton fabric increase. When the curing temperature reaches 150 °C, the wrinkle recovery angle of cotton fabric reaches the maximum value, and the increase ratio of the recovery angle reaches the highest value. Continue to increase the curing temperature, the fabric recovery angle shows a decreasing trend. We believe that there are still underreacted epoxy and

| Table 1 | Effect of finishing agent concentration on elastic properties of cotton fabric |
|---------|-------------------------------|-----------------|-------------------|
| Finishing agent concentration (g/L) | Recovery angle (°) | Recovery angle increase ratio (%) |
|         | Rapid elasticity | Delayed elasticity | Rapid elasticity | Delayed elasticity |
| Comparison sample | 92 | 127 | – | – |
| 10 | 115 | 162 | 25.0 | 27.6 |
| 20 | 141 | 185 | 53.3 | 45.7 |
| 30 | 148 | 188 | 60.9 | 48.0 |
| 40 | 139 | 180 | 51.1 | 41.7 |
| 50 | 138 | 175 | 50.0 | 37.8 |
secondary amine groups in the organosilicon resin emulsion finishing agent. The high temperature during curing provides the conditions for its full reaction, which increases the molecular weight of the organosilicon polymer. A larger network polymer structure is formed, and a polymer film with greater elasticity is obtained, so the elasticity of the cotton fabric is improved. However, when the curing temperature is too high (from 160 to 180 °C), the disulfide bonds in the polymer structure may be oxidized, resulting in a decrease in the number of cross-linking points constructed by the disulfide bonds. The cross-linked network structure of the resin is destroyed, and the molecular weight of the silicone resin finishing agent is reduced. As a result, the elasticity and strength of the organosilicon polymer film are reduced, and the resilience performance of the cotton fabric is reduced accordingly. In summary, the elastic finishing of cotton fabric with the organosilicon resin emulsion finishing agent containing disulfide bonds can obtain good resilience performance at a curing temperature of 150 °C.

**Effect of curing time on the elastic properties of cotton fabric**

The cotton fabric was immersed in the organosilicon resin emulsion finishing agents containing disulfide bonds with a concentration of 30 g/L. After the cotton fabric was padded and dried, it was cured at 150 °C for 2, 4, 6, 8, and 10 min respectively. Then the rapid and delayed elasticity recovery angles of the finished cotton fabric were measured. The results are shown in Table 3.

As shown in Table 3, under the conditions of finishing agent concentration of 30 g/L and curing temperature of 150 °C, prolonging the curing time, the wrinkle recovery angle of cotton fabric can only increase slightly. When the curing time was 4 min, the rapid and delayed elasticity recovery angle of

| Curing time (min) | Recovery angle (%) | Recover angle increase ratio (%) |
|------------------|--------------------|----------------------------------|
|                  | Rapid elasticity   | Delayed elasticity              |
| Comparison sample | 92                 | 127                             |
| 2                | 139                | 178                             |
| 4                | 147                | 185                             |
| 6                | 148                | 185                             |
| 8                | 148                | 186                             |
| 10               | 148                | 186                             |

Table 2 Effect of curing temperature on the elastic properties of cotton fabric

| Curing temperature (°C) | Recovery angle (%) | Recover angle increase ratio (%) |
|------------------------|--------------------|----------------------------------|
|                        | Rapid elasticity   | Delayed elasticity              |
| Comparison sample      | 92                 | 127                             |
| 130                    | 105                | 143                             |
| 140                    | 132                | 175                             |
| 150                    | 145                | 185                             |
| 160                    | 143                | 182                             |
| 170                    | 140                | 180                             |
| 180                    | 141                | 179                             |

Table 3 Effect of curing time on the elastic properties of cotton fabric
cotton fabric reached the highest value. Continue to extend the curing time, the crease recovery angle of the fabric remains unchanged. It can be considered that after curing at 150 °C for 4 min, the residual reactive groups in the organosilicon resin emulsion finishing agent on the fabric surface have undergone a sufficient ring-opening reaction. A larger network macromolecular structure is formed, the organosilicon polymer film with good elasticity is obtained, and the wrinkle recovery angle of the fabric reaches the maximum value. Continue to prolong the curing time, the macromolecular structure of the organosilicon polymer film on the fiber surface does not change significantly since no or slight changes in recovery angle and recovery angle increase ratio of rapid and delayed elasticity are noticed. Thus, the curing time of 4 min was chosen as a suitable finishing time for the experiment.

Temperature-responsive shape memory properties of finished cotton fabric

According to the finishing process conditions obtained above, the padding process is used to finish the cotton fabric with organosilicon resin emulsion. The two ends of the samples were clamped with clips to keep them in U shape, and then placed in an oven at 85 °C. After 10 min, the samples were taken out and cooled to room temperature. The clips were removed, and the U-shaped opening length $L_0$ of the comparison sample and the U-shaped opening length $L$ of the finished cotton sample were compared. The testing process and the mechanism of dynamic reorganization of disulfide bonds are shown in Fig. 7.

As shown in Fig. 7, the comparison sample has a larger opening length $L_0$ after heat treatment at 85 °C, and the opening length $L$ of the finished cloth sample is significantly smaller than the opening length $L_0$ of the comparison sample (Fig. 7a). It shows that the comparison sample has good elasticity, and has a good property of returning to its original shape even if it is treated at 85 °C for 10 min. A layer of the elastic organosilicon resin film is deposited on the surface of the cloth sample finished with organosilicon resin emulsion, in which the macromolecular network structure contains a certain number of disulfide bonds (Fig. 7b).

At room temperature, when the finished cotton fabric is deformed by an external force, the organosilicon resin film on the fiber surface will generate a certain internal stress due to the deformation. The organosilicon macromolecular chain will produce a certain slip to reduce the internal stress in the film. However, due to the constraints of the network structure, the slippage of the macromolecular chain is relatively limited, and the internal stress in the film is difficult to be fully eliminated. Therefore, after the external force is removed, the fabric will have greater deformation recovery under the influence of the internal stress of the film.

However, when the deformed fabric is heated at 85 °C, the disulfide bonds in the film will be broken to generate sulfhydryl groups (Fig. 7c, d). The cross-linking density of the macromolecular network structure is reduced, and the chain segment slips and adjusts to a new position due to the influence of internal stress. During the 10 min heat treatment, the structure of the organosilicon macromolecular chains is constantly adjusted under the internal stress. The internal stress of the organosilicon polymer film generated by the fabric deformation is continuously reduced, and the breaking of the disulfide bonds also accelerates the adjustment process of the macromolecular segments until the internal stress of the polymer film tends to zero. When the fabric is cooled to room temperature, the new shape of the fabric is fixed in a new position through the reorganization of the disulfide bonds in the organosilicon polymer film. Therefore, the opening length ($L$) of the fabric sample finished with organosilicon resin emulsion is lower than that ($L_0$) of the comparison sample. At the same time, the elasticity of the finished fabric sample did not change at room temperature. Since the organosilicon resin emulsion contains the disulfide bond structure, the finished cotton fabric has the shape memory property caused by the temperature change, which is called "temperature-responsive shape memory" functional finishing in this paper.

Surface morphology of finished cotton fabric

Scanning electron microscopy was used to analyze the cotton sample finished with the organosilicon resin emulsion. The surface morphology of the cotton sample is shown in Fig. 8.

It can be seen from Fig. 8, the fiber surface of the comparison sample is smooth and clean, with small cracks caused by processing, however, the fibers of
Fig. 7 Temperature-responsive shape memory properties of finished cotton fabric. (a, b) Test process and schematic diagram; (c, d) Mechanism of dynamic recombination of disulfide bonds

Fig. 8 SEM image of cotton fabric: a Comparison sample, b Finished cotton sample
the finished cotton samples with organosilicon resin emulsion are coated with a layer of the polymer film. Due to the non-uniformity of the evaporation rate of the finishing agent during the drying process, the polymer film forms certain wrinkles. The presence of such polymer film increases the elasticity of the fibers and yarns, thereby improving the elastic properties of cotton fabrics.

Conclusion

In conclusion, we have successfully prepared organosilicon resin emulsion containing dynamic disulfide bonds by emulsion polymerization to improve the high delayed elasticity of cotton fabrics and to give them shape memory functions. When the ratio of 4, 4-diaminodiphenyl disulfide in the raw material is smaller than the epoxy-terminated silicone oil, the network structure of the organosilicon elastic film prepared is more complex. The more complex the network structure of the elastic film is, the better the elastic resilience will be, and the permanent deformation tends to zero. According to this experimental method, a cationic organosilicon resin emulsion with an average particle size of about 250 nm and a concentrated particle size distribution was prepared. The surface of cotton fabric finished with organosilicon resin emulsion is covered with a layer of polymer film. When the cotton fabric is deformed by an external force at room temperature, the slippage of the macromolecular segment of the network structure in the polymer film is limited. Therefore, after the external force is removed, the internal stress that is difficult to eliminate will promote the recovery of the deformation gradually. The cotton fabric finished with cationic organosilicon resin emulsion has a delayed elasticity recovery angle of 185°, which is 45.7% higher than that of the comparison fabric sample, showing excellent delayed elasticity performance. At the same time, when the finished cotton fabric is deformed by an external force at a high temperature, the disulfide bonds in the polymer film are broken, and the internal stress promotes the slippage of the chain segments until the internal stress tends to zero. After the fabric is cooled, the disulfide bonds in the polymer film are reorganized at new positions, and the fabric shape is fixed, exhibiting excellent and special temperature-responsive shape memory properties, which undoubtedly broadens its application field.

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Declarations

Conflict of interest The authors declare that they have no conflict of interest.

Human and animal rights This article does not contain any studies with human participants or animals performed by any of the authors. Informed consent was obtained from all individual participants included in the study.

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