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Functionalization of silk fabric using hyperbranched polymer coated attapulgite nanoparticles for prospective UV-resistance and antibacterial applications

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Abstract

Herein, the Attapulgite nanoparticles (ATP NPs) coated silk fabric was prepared by impregnation method using hyperbranched polymer as addition agent. The ATP NPs and prepared silk fabrics were characterized by means of scanning electron microscope (SEM), ultraviolet-visible (UV–Vis) spectroscopy, Fourier transform infrared spectrophotometer (FTIR), X-ray diffraction (XRD), Energy Dispersive Spectrometer (EDS). The results of SEM, EDS, FTIR and XRD confirmed that ATP NPs were successfully coated on the surface of silk fabric. Not only did the treated silk fabrics possess excellent antibacterial property and antibacterial resistance, but also exhibited outstanding anti-ultraviolet performance, which can meet the requirements of multifunctional products.

Introduction

Silk fabrics, an important kind of natural protein textiles, has been widely used in traditional luxurious clothing and decorations with its excellent appearance, affinity to skin, biodegradation and regeneration, etc [1–3]. However, due to the special character of protein, proteins provide a more comfortable microbial environment for bacteria and fungus more easily than other natural and synthetic fabrics. Moreover, they are especially prone to turn yellow and wrinkle after ultraviolet irradiation [4, 5]. To overcome the intrinsic deficiencies of silk fabrics, many functional materials such as silver, ZnO, TiO2, MgO and natural clay minerals have been introduced into the multi-functional modification of silk fabrics to modify them [6–9]. For instance, Zhang et al fabricated TiO2 nanoparticles (NPs) for the finishing of silk fabrics with HBP-NH3 as reducing agent. The functional silk fabrics possessed excellent anti-UV property [10]. Wong et al utilized chitosan via a 13.56 MHz RF plasma to treat silk fabrics for durable antibacterial property [11]. After 5 washing cycles, the antibacterial rate of finished silk fabrics decreased from 100% to 88.78% to E. coli, from 100% to 90.26% to S. aureus. Among these research, the usage of chemical reductants and large physical equipment may lead to high-cost inputs and complicated finishing process. Moreover, the finished silk fabrics only improved one deficiency of silk, but did not possess or study the multifunctional properties. In addition, natural clay minerals (including kaolinite, bentonite, montmorillonite, illite and attapulgite) have attracted extensive attention on the functional modification of textiles due to their abundant, cheap, non-toxic and special performance [12–14].

Attapulgite (ATP) is a typical kind of natural clay minerals which can dissolve easily in aqueous and react with different oxides or metals for its particular morphology, unique three dimensional structures and large surface area with reactive –OH groups [15, 16]. Due to these excellent characters, more and more attention has been paid to its performance and application. Zhang et al fabricated a novel and efficient ATP-BiOBr-TiO2 composite, which showed exceptional visible-light photocatalytic activity in degradation of 20 mg l−1 methyl orange [17]. Pan et al prepared the low-cost chitosan/ATP composite by self-assembly to remove uranium contaminant from aqueous solution owing to its wide availability, efficient sorption ability and good bio-acceptability [18]. Zang et al synthesized PPy/Ag/ATP nanocomposite with 109–109 surface resistivity via UV
induced dispersion polymerization, serving as multifunctional filler and biodegradable composite materials [13]. Although applied in various fields of nanocomposite materials, wastewater treatment and pigments, application of ATP in fabrics, especially silk fabrics, has been rarely reported, especially to silk fabrics till now. In these papers, there is a common point that ultrasonic vibration is more effective and feasible method to fabricate ATP NPs, thus leading to the dispersion of NPs [19–21].

In our previous study, an amino-terminated hyperbranched polymer (HBP) with good water solubility and weight-average (Mw) molecular weight about 3700 was synthesized. HBP was used as dispersing agent to prepare ZnO NPs colloid solution and also served as a binder to impart the ZnO nanoparticles on the bamboo fabric for generating antimicrobial and anti-ultraviolet activities [22]. The objective of the current work was to prepare the highly dispersed ATP NPs in HBP aqueous solution under ultrasonic vibration, and then get assembled on the silk fabrics through a simple impregnation method. By means of SEM, FTIR, XRD and EDS, the morphology and microstructure of ATP NPs and the finished silk fabrics were characterized, with the properties of the treated silk fabrics being tested by transmittance analyzer and antibacterial experiment. The mechanism of antibacterial property and antibacterial durability was also discussed accordingly.

Experimental

Materials

Puriﬁed ATP was purchased from Jiangsu Jiuchuan nano materials Co. Ltd (China). Bombyx mori silk fabrics (plain woven, 90 g m⁻², 0.35 mm thickness) were obtained from the local market of China. HBP was prepared according to the method described in our paper [22]. S. aureus (ATCC 6538) and E. coli (ATCC 8099) were obtained from Department of Chemical Engineering, Yancheng Institute of Industry Technology (China). All samples were prepared with deionized water.

Preparation of ATP NPs solution

Firstly, HBP (0.2 g) was dissolved in 100 ml deionized water to form dispersed solution. And then three different amount of ATP clay were added into the HBP solution to obtain the ATP colloidal solution with concentrations of 5 g l⁻¹, 10 g l⁻¹, and 20 g l⁻¹, respectively. Finally, three different concentration of ATP solution were dealt with ultrasonic oscillations operating at 20 kHz with power output of 400 W (Sonicator Company, Kunshan, China) at 80 °C for 60 min, so as to obtain nano ATP colloidal solution.

Preparation of ATP finished silk fabrics

Silk fabrics were immersed in the ATP colloid solution (with liquor-to-fabric ratio of 50:1) with constant stirring for 30 min at 80 °C. Then all the samples picked up from the solution were washed with tap water for several times to remove unﬁxed materials. The resulting silk fabrics were air-dried at ambient temperature to produce the ATP finished silk fabrics.

Characterization of ATP NPs and ATP finished silk fabrics

The morphology of silk fabrics treated with ATP NPs and ATP was observed by Model S-4700 scanning electron microscope (SEM) operated by an accelerating voltage of 15 kV. The UV absorption properties of ATP NPs were determined by ultraviolet-visible (UV–vis) spectroscopy (UV–3010 Hitachi, Tokyo, Japan). The structure of ATP NPs and ATP treated silk fabrics was characterized by Nicolet Nexus (Ramesy, MN) 470 Fourier transform infrared spectrophotometer (FTIR), with a resolution of 4 cm⁻¹ and D/MAX3C x-ray detector at voltage of 40 kV, current of 30 mA and scan rate of 2° min⁻¹. The distribution of elements on the finished silk fabrics was investigated by Energy Dispersive Spectrometer (EDS).

Property test of ATP finished silk fabrics

According to the standards of antibacterial knitwear (FZ/T 73023–2006, China), the antibacterial activity and the durability of ATP treated silk fabrics were tested against E. coli and S. aureus by shaking ﬂask. The standard is suitable for the natural, chemical and blended ﬁbers after antibacterial ﬁnished. The sample weighing 0.75 g ± 0.05 g was cut into small pieces of 0.5 cm × 0.5 cm and sterilized under the condition of 10⁵ kPa and 121 °C in the autoclaves sterilizer for 15 min. And they were dipped into a ﬂask containing 70 ml of phosphate buffered saline (PBS; pH ≈ 7.2) and 5 ml of bacterial culture which had a cell concentration of 3 × 10³–4 × 10⁵ CFU ml⁻¹. The ﬂask was placed on a rotary shaker at 24 °C ± 1 °C for 18 h. 1 ml solution was taken out from each ﬂask and diluted to 10, 100, 1000 ml and then distributed evenly onto an agar plate, respectively. All plates were incubated at 37 °C ± 1 °C for 24–48 h, with the colonies being counted then. The percentage reduction was determined as follows:
Reduction in CFU % CA

Where C and A refer to the bacterial colonies, counted post treatment with the unfinished silk fabric and ATP-finished silk fabric samples, respectively.

The finished silk fabrics were washed according to the protocol, laid in the appendix C of antibacterial standard of knitted fabrics (FZ/T 73023-2006, China). Ultraviolet protection factor (UPF) and transmittance curve of silk fabrics were measured by a UV-1000F Labsphere Transmittance Analyzer (USA) according to EN:13758-2001.

Results and discussion

Characterization of ATP NPs in HBP solution

With larger surface energy, ATP NPs are easily agglomerated in aqueous solution. To obtain the high dispersion of ATP NPs solution for the treatment of silk fabric, ATP NPs was added in the HBP solution under ultrasonic conditions. Due to the three-dimensional structure and amino group of HBP, it can be applied as the templates for the control of ATP NPs. Due to the presence of hydroxyl groups and the special construction of HBP, the ATP NPs were easily dispersed in HBP aqueous solution via ultrasonic wave. To further study the UV protection mechanism, the ATP NPs were measured by means of UV–vis absorption spectra.

The UV–vis spectrum of HBP and HBP capped ATP NPs was shown in figure 1. As the special inner structure of HBP, ATP NPs can be capped by HBP to enhance the solubility properties. The HBP capped ATP NPs exhibited a milky white and high dispersion in HBP solution. As HBP was abundant in imine groups and terminal primary amino groups, with absorption peaks at 298 nm (figure 1(a)). The high surface area, the charge on the lattice, and the inverted structure given the ATP nanoparticles UV absorption capacity. Compared with HBP solution, even at the concentration of 1 g l−1, the HBP-capped ATP NPs (figure 1(b)) absorbed more ultraviolet light in the region of 200 nm to 400 nm, demonstrating that the HBP-capped ATP NPs possessed good anti-UV properties.

Mechanism and microstructure of ATP NPs finished silk fabrics

To confirm the zeta potential values of HBP-ATP NPs and silk fabric, the sample was tested under different pH conditions, as shown in figure 2. The isoelectric points of silk fabric and HBP-ATP NPs were about 4.1 and 10.2, respectively. Silk fabric was negatively charged due to the different amount of NH2 and COOH [23]. The positive charge of HBP-ATP NPs may be probably because of the presence of abundant imino groups and terminal primary amino groups in the HBP, which gained H+ ions in the liquid phase and then turned into cationic groups at pHs lower than 10.2. As the HBP-ATP solution had a pH value of 9.1, in HBP aqueous solution ATP NPs showed positive charge, while the silk fabric exhibited negative charge.

The plausible interaction of the ATP NPs with silk fiber is shown in scheme 1. Silk fabric was immersed in the HBP-ATP NPs solution (ATP content range from 5 to 20 g l−1). Since ATP NPs had a large number of amino groups on its surface, they can easily be combined with silk fibers through intermolecular hydrogen bonds between amino end groups and carboxyl groups on silk fiber. Electrostatic bonding interactions between the

\[
\text{Reduction in CFU(\%)} = \frac{C - A}{C} \times 100
\]
negatively charged hydroxyl groups on silk fabric and positively charged amino end groups contributed to the enhancement of the stability and adhesion of HBP capped ATP NPs on the surface of silk fabric.

The silk fabrics were characterized by SEM, EDS, XRD and FTIR. Figure 3 showed the morphology of ATP NPs, silk fabric before and after ATP NPs treatment. The ATP NPs (figure 3(a)) exhibited a rice shape with diameter about 50–80 nm and the average length was about 1.5 μm. There was an obvious change between the finished and unfinished silk fabrics. Compared with the unfinished silk fabric with clean and smooth surface in figure 3(b), the ATP finished sample of figure 3(c) became rough, and a large number of ATP NPs were distributed uniformly on the surface of the finished silk fabrics. The result showed that the ATP NPs could be coated on the surface of silk fabrics. Even after washing for 20 times, the ATP NPs can also be founded on the surface of silk fabric (figure 3(d)), indicating that the ATP NPs and silk fibers had good bonding strength. Due to the high surface activity of ATP NPs, it tended to agglomerate in the solution, with protonated HBP being adsorbed on the surface of ATP NPs, playing a protective and dispersive role. The electrostatic adsorption between the negative charged silk fibroin and the positive charged HBP-ATP NPs was the reason for the NPs assembled on the fiber and with excellent fastness.

In order to further verify the distribution of ATP NPs on the surface of the silk fabrics, Energy Dispersive Spectrometer (EDS) was used to analyze the elemental composition. ATP is a hydrated magnesium aluminum silicate non-metallic mineral Si₈O₂₀Mg₅(Al(OH)₂(H₂O)₄·4H₂O with its ideal chemical structure put forward by Liu in 2012 [16]. The resulting EDS spectrum in figure 4 showed strong carbon, oxygen, and copper peaks as expected. Carbon and oxygen arise from the silk fabrics. As the main elements of ATP, the three peaks of Mg, Al and Si in the spectrum indicated the existence of ATP NPs on the surface of silk fabrics. Even after washing for 20 times, the ATP NPs can also be founded on the surface of silk fabric (figure 3(d)), indicating that the ATP NPs and silk fibers had good bonding strength. Due to the high surface activity of ATP NPs, it tended to agglomerate in the solution, with protonated HBP being adsorbed on the surface of ATP NPs, playing a protective and dispersive role. The electrostatic adsorption between the negative charged silk fibroin and the positive charged HBP-ATP NPs was the reason for the NPs assembled on the fiber and with excellent fastness.

Figure 5 showed XRD patterns of ATP NPs, silk fabrics before and after ATP treatment. ATP NPs had four-plane characteristic peaks (figure 5(a)) at 2θ = 8.36°, 13.74°, 19.84°, and 26.64°. The four peaks corresponded to the main diffraction of (110), (200), (040), and (400) respectively, which belonged to clay and reflected the crystal structure of ATP NPs. The silk fiber demonstrated the diffraction peaks at 2θ values of 20.08 and 24.39° (figure 5(b)) owning to the diffraction peak of α-helix and β-fold of silk fiber. Compared with the unfinished silk fabric, the ATP finished silk fabrics (figures 5(c)–(e)) had an obvious absorption peak at 2θ = 8.36° which was attributed to the basal space of ATP framework (figure 5(a)). Moreover, there were two weak absorption peak at
$2\theta = 19.84^\circ$ and $26.7^\circ$ corresponding to Si-O-Si crystalline layer (040) and quartz impurities. SEM, EDS and XRD all proved that ATP NPs deposited on the silk fabrics effectively [24, 25].

Figure 6 showed FTIR spectra of silk fabrics before and after ATP NPs treatment. It could reflect the microstructure changes of silk fabrics and the effect of ATP to the finished fabrics. The major difference between the unfinished and finished silk fabrics was that a new absorption peak at 788 cm$^{-1}$ and 1054 cm$^{-1}$ appeared in the spectrum of finished silk fabrics, which were respectively ascribed to the Si-O asymmetrical and symmetrical characteristic peaks, respectively. The result showed that ATP NPs could be treated successfully on silk fabrics. Furthermore, as the concentration of ATP increased, the characteristic absorption peak of silk fibroin at around 1630 cm$^{-1}$ (amide I), 1518 cm$^{-1}$ (amide II), 1228 cm$^{-1}$ (amide III) and the hydroxyl group absorption peak reinforced [26]. The results indicated that the main composition of silk remained after ATP treatment. In addition, asymmetric stretching of –NH bonds changed from 2932 cm$^{-1}$ to 2924 cm$^{-1}$, presenting a small redshift. All these above indicated that the hydroxyl group on the surface of ATP could have a chemical bond.
reaction with amino and carboxyl groups on the surface of the silk fabrics, which was conducive to the functional stability of the silk fabric treated by ATP after repeated washing.

**Antibacterial property of ATP NPs finished silk fabrics**

*S. aureus* and *E. coli*, the representative of Gram positive cocci and Gram negative bacillus, were selected to study the antimicrobial activity of ATP finished silk fabrics. Table 1 showed the bacteria acount of *S. aureus* and *E. coli* colonies of the silk fabrics after different concentrations of ATP treatment. The treated fabric possessed excellent anti-bacterial activity. It has reported that the great bacteria inhibition ability can be attributed to the broken bonds on the surface of nano-size ATP particles, which could present biochemical reactions with the bacteria [16, 27]. Furthermore, the strong absorbability of the ATP NPs reduced the nutrition of the bacteria, which also resulted in an inhibition of the bacteria. Moreover, with the increase of the concentration of ATP, the effect of the anti-bacterial activity was more obvious. When the concentration of ATP colloidal solutions increased from 5 g l⁻¹ to 20 g l⁻¹, the bacterial reduction rates increased from 78.4% to 98.2% to *S. aureus*, and 80.3% to 95.4% to *E. coli*.

Antibacterial durability was another important factor to reflect the antibacterial textile against bacteria after repeated laundering. The silk fabric samples treated by the concentration of 10 g l⁻¹ ATP colloidal solution were laundered 0, 5, 10, 20 times with detergents. The results were given in table 2. After 5 times of washing, the ratio of antibacterial activity decreased from 93.09% to 91.1% to *S. aureus*, and 88.18% to 84.46% to *E. coli*. With the

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**Figure 5.** X-ray diffraction of (a) ATP (b) silk fabric, (b)–(e) treated sample by 5 g l⁻¹, 10 g l⁻¹, 20 g l⁻¹ ATP colloidal.

**Figure 6.** FTIR spectra of silk fabrics: (a) unfinished sample, (b)–(d) treated sample by 5 g l⁻¹, 10 g l⁻¹, 20 g l⁻¹ ATP colloidal.
further increase of the laundering cycles, the antimicrobial ratio changed a little, which still maintained 84.14% to \textit{S. aureus} and 80.8% to \textit{E. coli} after 20 laundering cycles. The excellent laundering durability of ATP treated silk fabric could be attributed to the electrostatic bonding interactions between the negatively charged hydroxyl groups on silk fabric and positively charged ATP NPs. In addition, there were also hydrogen bonds between amino end groups and carboxyl groups on silk fiber. All the factors could enhance the bonding strength between ATP NPs and silk fabrics.

\textbf{Ultraviolet resistance of ATP NPs finished silk fabrics}

As ATP NPs has a high absorption of UV–vis spectrum, the ultraviolet protection factor (UPF), transmittance of ultraviolet radiation a (UVA, 315–400 nm) and ultraviolet radiation b (UVB 280–315 nm) were tested to explore the UV protective ability of ATP NPs finished silk fabrics in figure 7 and table 3.

In comparison with the unfinished silk fabric, the UVA and UVB values of the fabrics treated with ATP NPs decreased significantly with the increase of ATP concentration, while the UPF values increased significantly. When the concentration of ATP in the solution was 10 g l$^{-1}$, UPF value of the finished fabrics was 80.2, with its

\begin{table}[h]
\centering
\caption{The antibacterial properties of ATP finished silk fabrics.}
\begin{tabular}{|c|c|c|c|c|}
\hline
\textbf{Samples} & \textbf{Bacterial colonies[CFU/mL]} & \textbf{Antibacterial activity [%]} \\
& \textit{S. aureus} & \textit{E. coli} & \textit{S. aureus} & \textit{E. coli} \\
\hline
Unfinished silk & $2.68 \times 10^{6}$ & $1.21 \times 10^{6}$ & — & — \\
5 g l$^{-1}$ ATP & $5.47 \times 10^{5}$ & $2.38 \times 10^{5}$ & 78.4 & 80.3 \\
10 g l$^{-1}$ ATP & $1.85 \times 10^{5}$ & $1.43 \times 10^{5}$ & 93.09 & 88.18 \\
20 g l$^{-1}$ ATP & $4.75 \times 10^{4}$ & $5.56 \times 10^{4}$ & 98.2 & 95.4 \\
\hline
\end{tabular}
\end{table}

\begin{table}[h]
\centering
\caption{The antibacterial properties of ATP finished silk fabrics after repeated washing.}
\begin{tabular}{|c|c|c|c|c|}
\hline
\textbf{Times} & \textbf{Bacterial colonies[CFU/mL]} & \textbf{Antibacterial activity [%]} \\
& \textit{S. aureus} & \textit{E. coli} & \textit{S. aureus} & \textit{E. coli} \\
\hline
0 & $1.85 \times 10^{5}$ & $1.43 \times 10^{5}$ & 93.09 & 88.18 \\
5 & $2.37 \times 10^{5}$ & $1.88 \times 10^{5}$ & 91.1 & 84.46 \\
10 & $3.01 \times 10^{5}$ & $2.13 \times 10^{5}$ & 88.76 & 82.39 \\
20 & $4.25 \times 10^{5}$ & $2.32 \times 10^{5}$ & 84.14 & 80.8 \\
\hline
\end{tabular}
\end{table}
anti-UV protection class reaching excellent protection, which could fully satisfy the performance requirement of anti-ultraviolet products [28]. Moreover, the size of ATP NPs in the solution was less than 100 nm, reaching the nanoscale. Therefore, when the concentration of ATP in the solution was less than 10 g l\(^{-1}\), ATP NPs finished silk fabrics exhibited outstanding anti-ultraviolet performance and the ATP particles were nanoscale.

### Conclusion

ATP clay was successfully prepared by adding it to HBP solution, and ATP NPs colloid was adopted in the treatment of silk fabric, and the multifunctional fabric was obtained. The morphology and microstructure of ATP NPs and ATP finished silk fabrics were measured by various methods. The results indicated that the surface of the ATP finished silk fabrics became rough and covered a large amount of uniformly distributed ATP NPs. The bacterial reduction rates of ATP finished fabrics were 93.09% to \(S.\) aureus, and 88.18% to \(E.\) coli. After laundering 20 cycles, it still maintained about 80% to \(S.\) aureus and \(E.\) coli. Not only did the multifunctional silk fabrics have excellent antibacterial property and antibacterial durability, but also had outstanding anti-ultraviolet performance.

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**Table 3.** The ultraviolet resistance of ATP finished silk fabrics.

| Samples            | UPF  | UVA (%) | UVB (%) |
|--------------------|------|---------|---------|
| unfinished silk    | 6.7  | 19.94   | 9.81    |
| 5 g l\(^{-1}\) ATP | 35.2 | 6.69    | 1.39    |
| 10 g l\(^{-1}\) ATP| 80.2 | 3.47    | 0.65    |
| 20 g l\(^{-1}\) ATP| 125.1| 1.16    | 0.60    |
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