Progress of silver-loaded cellulose composite antimicrobial materials

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Abstract. Silver-loaded cellulose composite antimicrobial materials have become a hot spot in the field of scientific research due to their excellent antimicrobial properties, good biocompatibility and environmental friendliness. The preparation methods include in situ chemical reduction, hydrothermal reduction, sol-gel and electrostatic self-assembly. These materials achieve antibacterial effects through mechanisms such as disrupting bacterial cell walls, inhibiting enzyme and protein activity, and disrupting bacterial DNA, and have broad application prospects in medical, packaging, and water treatment fields. This article provides a detailed summary of the research progress in the preparation methods, antibacterial mechanisms, and applications of silver loaded cellulose composite antibacterial materials, and looks forward to future research trends.

Keywords: cellulose, nanosilver, antimicrobial composites.

1. Introduction

With the development of science and technology and the improvement of living standards, people have higher and higher requirements for environmental hygiene and health, and at the same time, affected by various types of epidemics, the research and application of antimicrobial materials are getting more and more attention. In the field of healthcare, the application of antimicrobial materials can effectively prevent infection and improve the safety of medical devices. In daily life, antimicrobial materials are also widely used in food packaging, water treatment, textiles and other fields.

Silver-based antimicrobial agents are an important class of antimicrobial materials, which mainly inhibit or kill various microorganisms (such as bacteria, viruses, fungi, etc.) by releasing silver ions. Silver has broad-spectrum antimicrobial properties, and it is not easy to produce drug resistance, so silver antimicrobial agents have broad application prospects in many fields such as medical, textile, food packaging, etc. [1].

In recent years, the research of silver-based antimicrobial agents mainly focuses on the following aspects: a) Silver nanoparticles: nanosilver has attracted a lot of attention in the field of antimicrobial applications due to its unique physicochemical properties, such as large specific surface area, excellent electrical conductivity, thermal stability and oxidation resistance, as well as powerful antimicrobial effects [2]. Silver nanoparticles can directly damage the cell walls and cell membranes of bacteria, leading to the leakage of substances inside the cells and thus killing the bacteria. In addition, silver nanoparticles can also react with enzymes and proteins inside the cells to inhibit their activities, further enhancing their antibacterial effects [3-4]. b) silver ion-exchanged glass and ceramics: Silver ion-exchanged glass and ceramics with long-lasting antibacterial properties can be prepared by embedding silver ions into glass and ceramic materials. These materials can be used for the antimicrobial treatment of medical devices, kitchen utensils, sanitary ware and other products. c) silver antimicrobial polymer composites: silver nanoparticles are embedded into the polymer matrix, and silver antimicrobial polymer composites with long-lasting antimicrobial performance and good mechanical properties can be prepared. Such composites can not only realize the slow release of silver ions and improve the antibacterial effect, but also improve the antibacterial stability and durability of the material [5-6].

Cellulose is the most abundant organic polymer on earth, which has the properties of good biocompatibility, biodegradability, renewability and environmental protection. Silver-loaded cellulose composite antimicrobial materials precisely combine the advantages of nano-silver and cellulose, which have excellent antimicrobial properties, as well as good biocompatibility, biodegradability and environmental protection [7-9]. It has been found that the antimicrobial properties of silver-loaded cellulose composite antimicrobial materials mainly depend on the content and distribution of silver ions or silver nanoparticles [10]. As the content of silver ions or silver nanoparticles increases, the antimicrobial performance of silver-loaded cellulose composite antimicrobial materials will increase accordingly [11-12]. The distribution of silver ions or silver nanoparticles in cellulose also affects the antimicrobial performance of silver-loaded cellulose composite antimicrobial materials [13-14]. This thesis summarizes the preparation method, antimicrobial mechanism and application areas of silver-loaded cellulose composite antimicrobial materials.

2. Preparation of silver-loaded cellulose composite antimicrobial materials

Methods for preparing silver-carrying antimicrobial cellulose usually include reduction, sol-gel, and electrostatic self-assembly methods. In these methods, cellulose is used as a carrier and silver ions or silver nanoparticles are used as antimicrobial agents, and the silver ions or silver nanoparticles are immobilized on the surface or inside of the cellulose by chemical reaction or physical adsorption.

2.1. In situ chemical reduction

In situ chemical reduction is a common method for the preparation of silver-loaded cellulose composite antimicrobial materials. In this method, reduction occurs during the preparation process such as impregnation, precipitation, etc., so that the reduction product is attached to the cellulose network structure [15]. This method usually includes the following steps, cellulose first needs to be pretreated to increase its adsorption capacity for silver ions. Common pretreatment methods include acid-base treatment, oxidation treatment, etc. The purpose is to introduce active sites on the cellulose surface to facilitate the adsorption and reduction of silver ions [16]. This step requires control of conditions such as soaking time and temperature, which affect the surface properties of cellulose. Then the silver ion adsorption is carried out, i.e., the pretreated cellulose is immersed in a silver salt solution (e.g., silver nitrate or silver chloride solution) to adsorb silver ions on the cellulose surface [17]. The conditions of this step, such as solution concentration, immersion time, and temperature, affect the subsequent formation of silver nanoparticles. Next, chemical reduction is carried out, i.e., chemical reducing agents, such as sodium nitrite, citric acid, etc., are added to reduce the silver ions adsorbed on the surface of cellulose to silver atoms to form silver nanoparticles. In this step, the selection of appropriate reducing agents and reduction conditions (e.g., PH, temperature, etc.) is critical, and they will directly affect the size, shape and distribution of silver nanoparticles. Finally, the residual chemicals are removed by washing and drying steps to obtain the final silver-loaded cellulose composite antimicrobial material.

The in situ chemical reduction method can generate silver nanoparticles directly in the cellulose matrix, which is not easy to fall off and avoids the loss of silver nanoparticles during the transport process; the size and shape of the silver nanoparticles can be controlled by adjusting the reaction conditions, such as the temperature and the concentration, in order to optimize the properties of the composites. The advantages of the in situ chemical reduction method are simple operation and low equipment requirements, thus low cost; it is suitable for large-scale production; the size and distribution of silver nanoparticles can be controlled by adjusting the reaction conditions to optimize the properties of the composites. However, the reducing agents and solvents used in the in situ chemical reduction method may have certain impacts on the environment and human health, and the residual problem of the reagents in the fiber membrane needs to be solved; the size and distribution of the silver nanoparticles need to be finely controlled by adjusting the reaction conditions, or else they may affect the antimicrobial effect of the final materials, and smaller, uniformly distributed silver nanoparticles usually provide better antimicrobial properties [18].

Overall, the in situ chemical reduction method is a widely used method for the preparation of silver-loaded cellulose composite antimicrobial materials in practical production, and it is suitable for applications where large-scale production of silver-loaded cellulose composite antimicrobial materials is required, such as textile industry and medical device manufacturing. However, taking into account environmental considerations, this method needs to be optimized to reduce the use of chemicals and the emission of waste.

Currently, Xu Yongjian et al. used an in-situ reduction method to reduce silver ions on the surface of cellulose fibers to obtain cellulose-Ag composite antimicrobial materials by using bamboo pulp fibers as the matrix and cellulose as the reducing agent [19]. The reducibility of cellulose was improved by cellulase pretreatment, and more Ag^+ was reduced on the fiber surface. Feng et al. introduced carboxyl groups onto nanocellulose by the oxidation reaction of TEMPO (2,2,6,6-tetramethylpiperidin-1-yloxy), and the modified cellulose was immersed in a silver nitrate solution, using sodium citrate as a reducing agent, and the silver nanoparticle-anchored nanocellulose was obtained by an ion exchange reaction [20]. Yang Qian et al. used chemical reduction reaction to reduce silver nitrate solution using NCC solution to reduce Ag^+ to silver nanoparticles to obtain cellulose-silver nanoparticles [21]. Barud H S et al. immersed bacterial cellulose in $AgNO_3$ solution, added triethanolamine and mixed for 12h, washed with water for several times and then refluxed with 30% (wt/v) ethanol solution at 80°C and dried at 100°C [22]. This method using triethanolamine as a dispersant and reducing agent can make the spherical particles well dispersed in the bacterial cellulose body. Both scanning electron microscopy and X-ray diffraction observed 8 nm average diameter spherical silver nanoparticles adsorbed on bacterial cellulose fibrils. Dongping Sun et al. prepared Ag/BC composites by in situ reduction of silver nanoparticles (40-60 nm in diameter) in the spatial network structure of bacterial cellulose using a liquid phase reduction method [23].

2.2. Hydrothermal reduction

Hydrothermal reduction method is a method to reduce silver ions to silver nanoparticles in a high temperature and high-pressure water environment, and adsorb silver nanoparticles on cellulose materials by physical adsorption or chemical bonding [24]. The advantages of the hydrothermal reduction method are mild reaction conditions, enhanced reaction activity, simple reaction process, no need to add other reducing agents, silver nanoparticles can be generated directly on the surface of cellulose without the need for subsequent immobilization steps, and the prepared products have high crystallinity, high purity, narrower size distribution and good dispersion. However, the control of the reaction conditions is stricter and the operation is relatively complicated.

Yu et al. immobilized silver nanoparticles on fibers using hydrothermal reduction [25]; in the experiments, the ligand cellulose was used both as a reducing agent and a stabilizer, which made the process greener and more economical. Based on the nature of hydroxyl groups on nanocellulose with reducing ability to metal ions, Yang et al. proposed a green method to prepare silver nanoparticles [26]; in the experiments, the hydrothermal reduction method was used to prepare bacterial cellulose/silver nanoparticle composites using cellulose as both reducing agent and stabilizer, and a green process was adopted to prepare bacterial cellulose/silver nanoparticle composites without introducing other chemical reagents. Meanwhile, the method can also be extended to the synthesis of other cellulose composites, especially those materials that are in direct contact with the human body, such as medical devices, self-sterilizing textiles, water purification devices and food packaging. Yan Yan Dong Silver@cellulose composites were synthesized by hydrothermal method using microcrystalline cellulose solution, silver nitrate and aluminum chloride hexahydrate as raw materials [27]. Shuming Li Well-dispersed silver nanocomposites of cotton stalk synthesized cellulose were prepared using hydrothermal reaction with cotton stalk synthesized cellulose as the matrix phase, silver nitrate as the raw material and fructose or glucose as the reducing agent [28].

2.3. Sol-gel method

The sol-gel method first makes a sol by pre-mixing a silver salt (e.g., silver nitrate) and cellulose in a solvent (e.g., water, alcohol, etc.). Then a reducing agent (e.g. sodium nitrite, citric acid, etc.) is added to the sol to reduce the silver ions in it to silver nanoparticles. As the reaction continues, the sol gradually becomes thicker and eventually forms a gel. The silver nanoparticles in the gel are uniformly distributed in the cellulose matrix. The sol-gel method can be carried out at room temperature, and the preparation process is simple with low equipment requirements, making it suitable for large-scale production. However, drying and heat treatment may lead to changes in the properties of the cellulose matrix. Hao Chen Silver nanoparticles were obtained by reducing silver nitrate using the aldehyde group on DANC, and then crosslinked with the free amino group on CS by Schiff base reaction, an elastic brown gel can be obtained that is nanocellulose-silver-chitosan composite gel [29]. Different ratios of gel complexes can be prepared by changing the dosage ratio of nanocellulose and chitosan. Maria L C S et al. treated the BC films with $AgNO_3$ solution at room temperature, then with a solution containing 0.2 wt% of polyvinylpyrrolidone or hydrazine as a colloidal protectant for the gel, hydroxylamine, and vitamin C as a reductant [30]. Scanning electron microscope photographs showed that the silver nanoparticles with particle size 40 ~100 nm, adhered to the BC microfibers, and the X-ray diffraction results also showed that the silver cubic phase was deposited on the BC microfibers.

For silver loading of cellulose membrane materials, there are two routes. One is to prepare the regenerated cellulose membrane first and then load it with silver. Benavente et al. firstly separately prepared the regenerated cellulose membrane and the nanosilver sol, and after that the regenerated cellulose membrane was immersed in the nanosilver sol to obtain the regenerated cellulose membrane product loaded with nanosilver [31]. The disadvantage of this method is that the nanosilver particles cannot enter into the interior of the regenerated cellulose membrane material. The second method is to mix the nanosilver particles with the cellulose solution before casting the membrane, which solves the problem in the first method. For example, Chook et al. first prepared a nanosilver sol using a modified Torun reagent method, and then added this nanosilver sold to a cellulose-alkali/urea solution to obtain a regenerated cellulose membrane containing nanosilver of about 0.5 mm thickness, where the nanosilver could be mixed homogeneously with the cellulose in the cellulose solution, and therefore, the nanosilver could enter into the interior of the regenerated cellulose membrane in the process of casting the membrane, and the final product has a long time antimicrobial ability [32].

2.4. Electrostatic self-assembly method

Electrostatic self-assembly is a method of adsorbing silver nanoparticles onto cellulose materials using electrostatic interactions. It uses the negative charge of the hydroxyl group in the cellulose molecular chain to adsorb positively charged silver ions through electrostatic interactions, and then cellulose-based metal nanocomposites are prepared by in situ reduction. The electrostatic self-assembly method is a simple process, easy to operate and low cost; by regulating the charge of cellulose material and silver nanoparticles, the distribution of silver nanoparticles on the surface of cellulose material can be accurately controlled to regulate the properties of composites. However, the adsorption effect of silver nanoparticles in this method is easily affected by the surface charge and concentration of cellulose materials, and these parameters need to be precisely controlled to ensure the best antimicrobial performance. Martins et al. prepared nanocellulose/silver composites by electrostatic self-assembly, using polyelectrolyte as a macromolecular linker combining the cellulose nanoparticles and silver nanoparticles, and formed CNF/polyelectrolyte/Ag layer-by-layer composites [33]. Electrolyte/Ag layer-by-layer assembly, which endowed nanocellulose with antimicrobial properties.

3. Antimicrobial mechanism of silver-carrying cellulose composite antimicrobial materials

At present, silver-carrying cellulose composite antimicrobial materials have the following antibacterial mechanism: (1) The role of the cell wall: the bacterial cell membrane with a negative charge, silver ions Ag^+ can rely on the Coulomb gravitational force firmly adsorbed cell membrane, and the cell wall of peptidoglycan reaction occurs, resulting in bacterial intrinsic components are destroyed or production dysfunction. (2) Enzymes or proteins: Ag^+ can penetrate the bacteria into the cell, and bacteria and other groups in the body, such as sulfhydryl (-SH), so that the protein coagulation, destroying the activity of cell synthesis enzyme activity, protease inactivity, the loss of cell division and proliferation and death, preventing the normal metabolism of microorganisms. (3) Electric field adsorption: Ag^+ accumulates on the surface of bacterial cell membrane, affecting the membrane permeability of bacteria, destroying the microbial electron transport system and material transport system. (4) Role with bacterial DNA: Ag^+ can block ATP production, interfere with nucleic acid synthesis of microorganisms, and destroy DNA replication. It can also produce reactive oxygen radicals (ROS), which can combine with bacterial DNA and replace the hydrogen in the double helix structure of the DNA molecule, thus leading to the deformation of the structure of the bacterial DNA molecule and the loss of the ability to reproduce. (5) Catalytic effect: Ag^+ has a catalytic effect on the formation of free radicals, which can activate the surrounding oxygen and produce hydroxyl radicals (-OH) and reactive oxygen ions (O_2^-), which has a strong redox effect and can inhibit or kill bacteria. These mechanisms make silver antimicrobial agents in the antibacterial field has a pivotal position. (6) The role of multi-drug resistant bacteria: the antibacterial effect of silver ions is not easy for microorganisms to produce resistance, the inhibition of multi-drug resistant bacteria is more significant, and is expected to become an effective means of dealing with infections in the medical field.

4. Silver-loaded cellulose composite antimicrobial material applications

Silver-loaded cellulose composite antimicrobial materials have a wide range of applications in the fields of healthcare, packaging and water treatment [34]. At the same time, there is a trend to use them in a wider range of household products and construction materials. Antimicrobial textiles, for example, provide long-lasting antimicrobial protection. Outdoor and sporting goods can inhibit the growth of microorganisms caused by humidity, keeping them clean and comfortable. Door handles, sinks, and bathroom fittings reduce the growth of bacteria and microorganisms. In the construction field, silver-loaded cellulose composite antimicrobial materials can be used to manufacture antimicrobial coatings, antimicrobial flooring, and antimicrobial isolation materials in medical settings or laboratories, which help reduce bacteria and fungi on surfaces, block the spread of pathogens, and improve indoor air quality.

4.1. Medical field

Medical textiles: for the manufacture of antimicrobial textiles with antimicrobial materials in medical cellulose composites, such as surgical gowns, nurses' gowns and patients' gowns, in order to reduce the risk of hospital-acquired infections. Dressings and dressings: for the manufacture of dressings and dressings with antimicrobial properties to help prevent wound infections [35-36]. Operating room supplies: tools, equipment and surfaces in the operating room are coated with silver-loaded cellulose composite antimicrobial materials, effectively preventing the risk of cross-infection of surgical instruments [37]. Implantable Medical Devices: The use of silver-loaded cellulose composite antimicrobial materials for implanted medical devices, such as pacemakers and joint replacements, can reduce the probability of post-implantation infection.

The preparation of novel medical dressings from nanocellulose has received increasing attention. Wu et al. loaded silver nanoparticles onto nanocellulose using an in-situ reduction method, and the network structure of the cellulose prevented the shedding of the silver nanoparticles, thus minimizing the toxicity [38]. Despite the slow release of silver ions from the material, it still showed significant antimicrobial activity against Escherichia coli (E. coli), Staphylococcus aureus (S. aureus), and Pseudomonas aeruginosa. Yan et al. developed poly (lactic acid) fiber membrane ophthalmic bandages containing nanocellulose and silver nanoparticles in order to kill infective pathogens and to promote cellular proliferation, avoiding the need for long term medication and repeat medication administration [39]. Singla et al. prepared a nanosilver/nanocellulose bio composite film and ointment, which significantly improved skin tissue repair in vivo, and thus the complex can be used as an antimicrobial wound dressing for accelerated tissue repair and regeneration [40-41].

4.2. Packaging materials

Fresh food packaging: It is suitable for packaging meat, seafood and other fresh food, which can effectively extend the shelf life of food and reduce the possibility of food corruption and spoilage [42-43]. Takeaway and fast-food packaging: applying antimicrobial materials in takeaway and fast-food packaging can reduce the spread of microorganisms on hand-contact surfaces, improve the hygiene of the packaging and reduce the risk of contamination of takeaway food. Pharmaceutical packaging: The application of antimicrobial materials in pharmaceutical packaging can help prevent microbial contamination of pharmaceuticals and safeguard the quality and safety of pharmaceuticals [44].

Zhang et al. used cellulose as raw material and prepared cellulose antimicrobial cling film by one-step coagulation method using $AgNO_3$ and polyvinylpyrrolidone (PVP) [45]. Sarwar et al. prepared polyvinyl alcohol/nanocellulose/silver nanocomposite film by solution calendering method and evaluated the antimicrobial properties of composite film, which showed that composite film had no cytotoxic effect on HepG2 cells and the cell viability was over 90% [46]. The composite film is biodegradable and can be used in food packaging. Zhu et al. prepared silver nanoparticle-coated pulp fibers by in situ immobilization of silver nanoparticles using thiol-containing silylated pulp fibers followed by sandwich-like silver pulp/CNF composite paper using alternate filtration method [47]. This cellulose composite paper has the advantages of simple preparation, high separation efficiency, good durability and antimicrobial properties, and has potential applications in antimicrobial packaging. Adepu et al. produced nano-silver-bacterial cellulose anti-pollution materials, they found that the food can be preserved in them for 30 days without decay, while the general plastic bags can only be preserved for 15 days, so the silver-carrying bacterial cellulose nanoparticles can be used in the food packaging [48]. Therefore, silver-containing bacterial cellulose can be used in food packaging and health care.

4.3. Water treatment

Outdoor activities utensils: water bottles and cups are made of antimicrobial materials, which can effectively guarantee the hygiene of drinking water during field activities. Water filtration materials: Due to the antimicrobial properties of silver ions, silver-loaded cellulose composite antimicrobial materials can also be used in the manufacture of water filtration materials for improving water quality and preventing the propagation of microorganisms in water. Water treatment equipment and piping: The use of materials with antimicrobial properties in the equipment and piping of water treatment plants helps to reduce the growth of microorganisms and maintain the cleanliness of the entire water treatment system.

Zahid et al. developed an environmentally friendly method to deposit silver nanoparticles on CNC using biocompatible polyvinylpyrrolidone (PVP) as a reducing agent [49]. The method showed uniform in situ deposition of silver nanoparticles on CNC. The prepared nanocomplexes (CNC/PVP/Ag) had good hydrocolloid stability (8 months) and antimicrobial activity (minimum inhibitory concentrations of 12.5 μg/mL and 25 μg/mL for E. coli and S. aureus, respectively), and retained efficient bacterial filtration performance (E. coli viability reduced by 100 %) after two water filtration cycles.

5. Conclusion

The antimicrobial properties of silver-loaded cellulose composite antimicrobial materials are remarkable, including broad-spectrum antimicrobial resistance, long-lasting antimicrobial resistance, low antimicrobial adaptability, and disruption of biofilm. The present preparation methods of these materials include reduction, sol-gel and electrostatic self-assembly. The antimicrobial mechanism of these materials was that through the mechanisms of microbial membrane disruption, protein and enzyme inactivation, and DNA and RNA damage, the growth of microorganisms is effectively inhibited, and the hygiene level of products and the environment is improved. Silver-loaded cellulose composite antimicrobial materials have wide applications in the fields of medicine, packaging and water treatment.

Although important progress has been made in the preparation and application of silver-loaded cellulose composite antimicrobial materials, they still face a number of challenges, such as improving the silver-loading efficiency, optimizing the parameters of silver nanoparticles to enhance the antimicrobial properties, and improving biocompatibility. Future research needs to further address these issues in order to promote the wider application of these materials in medical, food, environmental protection and other fields.

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