Bactericidal Textile Productions from Cellulose with Silver Nanoparticles

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Abstract: Stabilized silver nanoparticles, in solutions sodium-carboxymethyl cellulose (Na-CMC), were synthesized and their structure and physico-chemical properties were studied. The forms and sizes of silver nanoparticles were studied by atomic force microscopy and transmission electron microscopy methods, which silver nanoparticles are loaded in solutions CMC and grafted cotton fabrics. Investigations show that spherical silver nanoparticles with size 5-35 nm and with content of 0.0085% mass. in cotton fabrics has a high bactericidal activity. Stabilization of silver nanoparticles helps to save the bactericidal and bacteriostatic activity during washing the cotton fabrics and textiles on their basis.

Key words: Silver, nanoparticles, Na-carboxymethylcellulose, cotton fabrics.

1. Introduction

At the present time huge practical interest to make cellulose fibers, textile materials and products based on has bactericidal activity against pathogenic microbes and bacteria [1]. For making such materials as a polymeric agent could be used natural cellulosic fibers, textile materials and products based on.

Bactericidal and bacteriostatic activity to the cellulosic materials and products may be formed by incorporating silver ions in structure with after their reduction until nanoparticles. Interaction with polymer matrixes is occurred by the formation of coordination bonds [2].

At the present time determines that silver and its compounds are able to inhibit the growth and kill more than 650 kinds of bacteria, viruses and fungi, at the same time keep up microelements an essential part all of living organisms [3, 4].

Silver nanoparticles have an extremely large specific surface area, increasing the surface area bring to increases of contact with bacteria, viruses and fungi. Significantly increase is occurred bactericidal activity, even in decreasing the concentration of silver at hundreds times [5]. However, decreasing in size of silver until nanoparticles, substantially increase the speed their agglomeration by increasing the surface energy.

Thus, the most interesting scientific and practical aspect of stabilized silver nanoparticles is, obtaining bactericidal and bacteriostatic textile materials and products from natural polymers [6].

The aim of this work is making bactericidal, antifungal fibers and textile materials and products based on, contained stabilized silver nanoparticles and the study their structures and properties.

2. Experiments

Cellulosic fibers, cotton fabrics (c/f), textile fabric and purified Na-carboxymethylcelluloses (Na-CMC) with degree of substitutions (DS) = 0.65-0.85 and degree of polymerizations (DP) = 200-600 were used as a polymer matrix.
An aqueous solution of silver nitrate (AgNO₃) was used for the formation of silver nanoparticles in solutions Na-CMC. As strain pathogen microorganisms of humans and animals were used *Staphylococcus epidermidis, Candida albicans*.

For the formation stabilized silver nanoparticles 0.1-0.4% aqueous solutions of purified samples of Na-CMC with various DS and DP was used after removal the gel fraction by centrifugation on laboratory centrifuge at 4,000 rev/min for 10 min.

For this purpose calculated quantity of 0.1-0.001 mol/L aqueous solutions of AgNO₃ was added drop by drop to Na-CMC solution liberated from gel fraction with intensive stirring, until obtain homogeneous of solution Ag⁺CMC⁻.

Photochemical reduction of silver ions in the structure of Ag⁺CMC⁻ until nanoparticles was performed at 25 °C, by irradiating high pressure mercury lamp DRSh-250. In order to obtain dispersed silver nanoparticles UZDN-1, Y-4, 2 type disperser was used.

By means of work up treating cellulosic fibers, cotton fabrics and textile products with solutions contained the stabilized silver nanoparticles and after their subsequent heat treatment obtained crosslinked water-insoluble compositions of cellulosic fibers and cotton fabrics with antibacterial and antifungal properties.

The size and shape of silver nanoparticles in the Na-CMC solution was controlled by spectrophotometer method in UV-spectroscopy, spectrophotometer model Specord M210 in the wavelength range from 200 to 900 nm. The size, shape and size distribution of the silver nanoparticles in the cotton fabrics were determined by electron microscopy, type transmission electron microscope TEM-100 (Ukraine) and atomic, force microscope AFM, 5500 (Austria). The variation coefficients were determined by the calculation corresponding micrographs on MathCad program.

Content of silver nanoparticles in cotton fabric were determined by atomic absorption spectroscopy model of spectrophotometer PERKIN-ELMER 3030 B (USA) with a flame analyzer (acetylene-air).

Bactericidal activity of cotton fabrics, contained silver ions and nanoparticles were studied by microbiological method [7] at the pathogenic bacterium *Staphylococcus epidermidis* and fungus *Candida albicans*.

### 3. Results and Discussion

At the first step of investigation solutions of Na-CMC with different DS and DP were obtained to form the stabilized silver nanoparticles. The formation of silver nanoparticles in the structure of samples Na-CMC were carried out at 0.2%-0.4% concentrate solutions to provide them free access to both interfiber and intermolecular spaces of cellulose textiles after their impregnation.

Formation of nanoparticles from silver ions in the structure of dilute solutions of Na-CMC with different DS, (0.65-0.85) and DP, (200-600) were carried out by photochemical reduction of silver ions [8, 9].

It has been established that adding silver ions in Na-CMC solutions causes to increase the viscosity of system due to decreasing the solubility of Ag⁺CMC⁻ and appearance of coordination bonds between molecules with formation of polycomplexes. Increasing the relative viscosity of Na-CMC contained silver cations dependence on decreasing the solubility due to formation of intermolecular coordination bonds between the silver ions and carboxylate anions in macromolecules of Na-CMC [10].

Synthesized silver nanoparticles, by photochemical method, in Na-CMC solution provided high stability and did not come to agglomeration during keeping at long times, to compare with nanoparticles reduced from silver ions in aqueous solutions by chemical agents.

After UV-irradiation solutions of Na-CMC contained silver ions formed enough stable colloidal systems of nanosilver pale-yellow color, contained at
the optical spectra maximum at \( \lambda_{\text{max}} = 416 \) nm, which characterizes nanoparticles of silver with sizes 5-25 nm (Fig. 1, curve 3) [11]. It can be seen in the spectra of initial solutions of Na-CMC and Ag\(^{+}\)CMC\(^{-}\) at the 250-900 nm optical region was no changes observed (Fig. 1, curves 1, 2.).

With increasing the time of photolysis the color of solutions is changed from pale yellow to brown. According to literature such changes are probably dependent on with an increasing the number and size of formed silver nanoparticles.

For the confirm, this assumption absorption spectra’s were taken at the different times of irradiation of systems Ag\(^{+}\)CMC\(^{-}\) at concentration of Na-CMC - 0.2% and \(1 \times 10^{-2}\) mol/L silver nitrate. Fig. 2 shows the UV-spectrum solutions of Na-CMC contained silver nanoparticles which obtained at different times of photo irradiations.

It can be seen in Fig. 2, after 5 min photo irradiation in the spectrum observed the shoulder at region \( \lambda_{\text{max}} = 270 \) nm, which could be attributed to stable polyanions charged silver clusters, approximately Ag\(_{8}\)\(^{2+}\) [12, 13], (Fig. 2, curve 2).

After 15, 20 min photolysis at the spectrum observed increase the intensity of the absorption band at \( \lambda_{\text{max}} = 270 \) nm, which depends on with the formation of large stabilized clusters of silver with sizes 2-8 nm [13] (Fig. 2, curves 2, 3).

Further irradiation brings to the appearance of new absorption peaks with maximums at regions \( \lambda_{\text{max}} = 290 \) nm and \( \lambda_{\text{max}} = 420 \) nm which belongs to larger clusters and nanoparticles of silver with sizes 5-35 nm [11].

Homogeneity formed nanoparticles in different sizes can be obtained because of the macromolecules of Na-CMC covers the silver nanoparticles create charged shell around them to prevent nanoparticles’ aggregation. Commonly, when polymer concentration is high, it tends to stabilize nanoparticles.

Increasing the local concentration of Na-CMC on the surfaces of silver nanoparticles created on the one hand, supports electrostatic and steric stabilization, and on the other hand - creates an conditions in which don’t completely exclude interaction generated radicals by UV-photolysis of Na-CMC. To confirm the stability of formed silver nanoparticles in polymer matrix the UV-spectra of Na-CMC solutions containing stabilized silver nanoparticles has been taken which (Fig. 3), keeping for the different periods of time.

It can be seen in Fig. 3, form and content of silver nanoparticles in Na-CMC solutions, at during keeping times at home conditions absorption peak practically unchanged. Based on the experimental results it could be concluded that, with depending on the molecular weight, degree of substitution and the ratio components of Na-CMC and AgNO\(_3\) able to control the size and shape of silver nanoparticles which will
forms in solutions of Na-CMC by photochemical reduction.

Analyzing spectroscopic data nanocomposites based on silver and Na-CMC, were suggested that the negative ions of carboxymethyl group, is “catcher” for the positive charged of silver ions.

Well known that on basis of the photographic process, halogens of silver are formation of colloidal silver particles with prolonged irradiation of photons above the band gap.

This electron-stimulated process of formation of colloidal particles had been explained on the basis of Mott-Gurney theory [15], the essence which as follows. During the photochemical reduction the optically generated electron migrates and cached in electron catcher at the interfaces and near the surface (Scheme 1).

Cations of silver attracts to carboxymethyl anion of CMC to form an ionic bonds. Reduction reaction begins with connected silver cations, at “nanoreactors” with formation clusters, which turn into nanoparticles, due to recovery of cations near nanoreactors.

This is the first step in the sequence which the electrons and interstitial atoms catching, forming clusters and silver nanoparticles, which make on basis latent image.

For the purpose to determine the form and size of silver nanoparticles in the structure of Na-CMC, carry out investigations samples by atom - force microscope. Received data are presented in Fig. 4.

You can see from AFM images, that at low concentration of silver ions formed spherical polydisperse nanoparticles with sizes 2-8 nm. With increasing ions of silver concentrations after photolysis forms monodisperse nanoparticles of silver with sizes 5-35 nm.

This can be explained by the fact that at the same time silver cations in compositions’ CMC connected and disconnected with carboxylic anions (nanoreactor) [15] of CMC are reduced by increasing the time of photolysis.

These processes occur with different speed, which probably bring to an increase in the polydispersity of formed nanoparticles.

Thus, the size and shape of silver nanoparticles formed by the photochemical reduction of silver cations, at systems Ag⁺ CMC⁻, depend on DS and the concentration of solution of Na-CMC, and concentration of Ag⁺ and times of photochemical irradiation.

Also was found that by keeping the concentration of carboxylate anions in the solution of Na-CMC with increasing content of silver cations in system by the same time occur increase the size of silver nanoparticles and their contents.

There is also observed changing the form of silver nanoparticles formed from spherical to rod like structure [13].

Based on the results of studies selected next conditions

![Scheme 1 Assumption scheme of formation nanoparticles of silver in structure Na-CMC.](image-url)
Fig. 4  AFM microphotographs of solutions Na-CMC, contained silver nanoparticles (a), (b) and size distribution of nanoparticles (c), (d). Concentration of [Na-CMC] = 0.2%; [AgNO₃] = 1 × 10⁻² mol/L. Time of UV-irradiation (a) 20 min and (b) 30 min.

for the formation of homogeneous in size of silver nanoparticles. Time of UV-irradiation 30 minutes, the contents of Na-CMC in solution 0.2%, content of AgNO₃ in solution 0.016 wt.%. In selected conditions forms spherical stable silver nanoparticles with sizes 5-35 nm.

At the present time, the joint efforts of chemists, biologists and physicians are attention on the problem of giving polymeric materials additional therapeutic and prophylactic properties by forming the silver nanoparticles into structure of drugs [6].

We have obtained biodegradable hydrogels, films and cotton fabrics and productions with a high bactericidal activity on the base of Na-CMC and cellulose contained silver nanoparticles [16].

For the aim of obtaining the bactericidal cotton fibbers, materials and fabrics they have been grafted with Ag₀/CMC solutions.

Therefore at grafting low concentration of Na-CMC solution and the sizes of nanoparticles and ions of silver they able to inter interfiber and intermolecular free spaces of materials and fabrics obtained from cellulose.

Obtained wet cotton fabrics and products subjected to additional UV-irradiation, where occurred restoration of the unreacted silver ions into structure of Na-CMC matrixes.

We have investigated the samples with transmission electron microscope. The result data are shown in Fig. 5. You can see from Fig. 5, the size of spherical silver nanoparticles are at range 2-30 nm, their content in cotton fabrics was 0.0086 wt. %.
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Supposed scheme of formation of silver nanoparticles in the structure of cotton fabric, complex fibers and elemental of cellulose fiber are shown in Fig. 6.

When cotton fabrics, complex of fibers and elemental fibers of cellulose (Fig. 6) grafts with the mixture solutions of \( \text{Ag}^0 \) CMC and CMC-\( \text{Ag}^+ \) they inter between free spaces of fibers (1) and between free spaces of complexes of fibers including in the structure of fabric (2).

It was found that the macromolecule of Na-CMC, contained nanoparticles and ions of silver able to inter between free spaces fibrils elemental of fiber (3) cellulose, which are a “trap” for the nanoparticles and ions of silver.

After repeated photoirradiation of wet fiber the silver ions which situated on between strings, between fibers and between fibrils space reduction until

Fig. 5 TEM images of (a) initial cotton fiber and (b) silver nanoparticles on the surface cotton fiber.

Fig. 6 Supposed scheme of formation and fixing of silver nanoparticles in the structure of (a) cotton fibers; (b) complex fibers and (c) elemental of cellulose fibers.
nanoparticles in the structure of Na-CMC which, after drying changes into an insoluble state by formation of intermolecular hydrogen and covalent bonds between the carboxyl and hydroxyl groups of the macromolecules of Na-CMC and cellulose. This explained by the silver nanoparticles stability in the structure of fibers and fabrics based on with repeated washings.

Bactericidal activity of obtained samples of cotton fabrics grafted with solution of CMC, contained ions and nanoparticles of silver at test cultures of \textit{Staphylococcus epidermidis} and \textit{Candida albicans} was studied at the laboratory of the Institute of Microbiology, Uzbek Academy of Sciences.

For the determine the antimicrobial activity of the samples, were added to test-tubes containing thioglycolic environments (for \textit{Staphylococcus epidermidis}) and Saburo (for \textit{Candida albicans}) at the following systems: (1) Control cotton fabrics physiological solution; (2) Cotton fabric + Ag\textsuperscript{+}, C\textsubscript{Ag}\textsuperscript{+} = 0.0086 mass.%; (3) Cotton fabric + Ag\textsuperscript{0}, C\textsubscript{Ag}\textsuperscript{0} = 0.0086 mass.%; (4) Cotton fabric + Ag\textsuperscript{0}, C\textsubscript{Ag}\textsuperscript{0} = 0.086 mass. %.

As a control in the same medium was added physiological solution of 10% NaCl with compared to the volume of the medium.

Within six hours test culture was added in the final concentration of 150 cells/ml in each test-tube.

Samples were incubated at 34 °C during 48 hours (for \textit{Staphylococcus epidermidis}) and 72 h (for \textit{Candida albicans}).

The result of microbiological investigations showed that all samples have a degree of antimicrobial activity against pathogenic microorganisms of human.

Results of investigations samples of fabric are shown in Table 1.

You can see from the presented data in the Table 1, all the samples except the control and the sample number No. 2 (cotton fabric + Ag\textsuperscript{+}, C\textsubscript{Ag}\textsuperscript{+} = 0.0086 mass. %); completely inhibit the growth of fungus \textit{Candida albicans}.

Low activity of cotton fabric, contained silver ions connected with CMC, can be explained apparently so that the contact of silver ions bounded to the surface of these strains to make a coordination bond with functional groups where is on the surface of the strains. This brings to fast inactivation of the silver ions [17].

Samples No. 2, No. 3 and No. 4 inhibited the growth of bacteria \textit{Staphylococcus epidermidis} by 72%, 94.6% and 88%, respectively. Also you can see from the table the sample No. 3 has a high bactericidal activity against \textit{Staphylococcus epidermidis} is 94.6%. It is probably depends on the smallest sizes of silver nanoparticles which [18], their content in the cotton fabric is 0.0086 mass.%.

| №  | Samples                  | Content of silver nanoparticles in the cotton fabric, mass. % | Strains                        |
|----|--------------------------|---------------------------------------------------------------|---------------------------------|
|    |                          |                                                               | \textit{Staphylococcus epidermidis} | \textit{Candida albicans}       |
| 1  | Control                  | -                                                             | 150 KOE*/mL                      | 150 KOE*/mL                     |
| 2  | Cotton fabric + Ag\textsuperscript{+} | 0.0086                                                  | 24 KOE/mL                       | 15 KOE/mL                       |
| 3  | Cotton fabric + Ag\textsuperscript{0} | 0.0086                                                  | 6 KOE/mL                        | Not exist                       |
| 4  | Cotton fabric + Ag\textsuperscript{0} | 0.086                                                   | 9 KOE/mL                        | Not                             |

Table 1  Antimicrobial activity of cotton fabrics, contained silver ions and nanoparticles against strains of \textit{Staphylococcus epidermidis} and \textit{Candida albicans}.
Table 2  Comparative results of the bactericidal activity of cotton fabrics grafted with solutions of Na-CMC contained silver nanoparticles with different shapes and sizes.

| №  | Samples cotton fabric treated with a solution of CMC × Ag⁰ at different concentrations of silver ions | The size of the silver nanoparticles in the solutions Na-CMC (nm) | Strains                  |
|----|--------------------------------------------------------------------------------------------------|---------------------------------------------------------------|---------------------------|
| 1  | Control                                                                                         | -                                                             | Staphylococcus epidermidis | Candida albicans          |
| 2  | C₄Ag = 0.000003 mol                                                                           | 2-8 nm                                                        | 5 × 10¹² KOE/mL           | 1 × 10⁷ KOE/mL            |
| 3  | C₄Ag₀ = 0.00003 mol                                                                            | 5-35 nm (spherical)                                          | Not exist                 | Not exist                 |
| 4  | C₄Ag = 0.0003 mol                                                                             | l₁ = 130-420 nm, l₂ = 15-40 nm                              | 1 × 10¹⁰ KOE/mL           | Not exist (whiskers)      |

Also you can see from the table, the most active against the strain of *Staphylococcus epidermidis* and bacterium *Candida albicans* is cotton fabrics grafted with solution of Na-CMC, contained in the structure spherical nanoparticles of silver sizes 5-35 nm, it could be explained by the content of silver nanoparticles and their surface area [19-20].

Cotton fabrics grafted with solution of Na-CMC, contained whiskers nanoparticles of silver with sizes in length 130-420 nm and in width 15-40 nm, less bactericidal active compared with cotton fabric grafted with solution containing silver nanoparticles of spherical structures with the size 5-35 nm.

But, they are more active compared with cotton fabric grafted with solution contained silver nanoparticles with sizes 2-8 nm, it could be explained by high content of silver nanoparticles in the cotton fabric.

The comparatively high bactericidal activity of silver nanoparticles (Table 2) with the silver ions (Table 1) probably due to the fact that:

- Silver nanoparticles can not make chemical bond with the functional groups [21] the strains of surface bacterium *Staphylococcus epidermidis* and fungus *Candida albicans*, and probably seems able to penetrate through wall of cell and into the nucleus of cells and inhibit their growth and activity;
- Lowering the size of silver nanoparticles increased the total area of the surface and accelerating their ability to penetrate the cell nucleus of the above strains.

Comparatively bactericidal activity of cotton fabrics after repeatedly washing is presented in Table 3. It can be seen in Table 3, after washing five times (cotton fabric + Ag⁰) due to washing of silver nanoparticles from the surfaces of the fiber polymeric matrix that their content decreases from 0.0086 to 0.0023 wt.%

Bactericidal activity of the samples (cotton fabric + Ag⁰) kept after 2 times washing against strains *Staphylococcus epidermidis* and 4 times washing for strain *Candida albicans*.

For the washing cotton fabrics contained silver nanoparticles more than five times by decreasing the content of silver nanoparticles as they exhibit bacteriostatic properties against these strains.

4. Conclusions

It was found that the grafted cotton fabrics and products based on stabilized silver nanoparticles
contribute to provide bactericidal and bacteriostatic properties. Bactericidal activity of fabrics depends on the content, size and form of the silver nanoparticles in the structure of fabrics.

Stabilization of silver nanoparticles in the structure of the polymeric matrix brings to keep their bactericidal and bacteriostatic activity during washing up the cotton fabrics and products. Based on the results of investigation it was found the optimum conditions for obtain bactericidal and bacteriostatic cotton fabrics and textiles.

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