The improvement of bactericidal properties and change of colour characteristics of knitted materials at using nanosilver and carboxymethyl starch

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ABSTRACT – REZUMAT

The improvement of bactericidal properties and change of colour characteristics of knitted materials at using nanosilver and carboxymethyl starch

The availability of bactericidal knitted cotton fabrics by processing a biodegradable bactericidal nano composition containing nanoparticles of silver and Na-carboxymethyl starch is studied in this work. The nanocomposite based on Na-carboxymethyl starch and silver nanoparticles were successfully fixed on the surface of knitted cotton fabrics through the formation of links between carboxymethyl groups of carboxymethyl starch and nanosilver, as well as air interlacing between nano composition and material. The analysis of the change in the colour of knitted cotton fabrics after processing them with a solution of the nano composition of silver and Na-carboxymethyl starch showed the stability of the colouristic indicators of the colour during antibacterial treatment.
Knitted cotton fabrics treated with the developed nano composition exhibit high antibacterial activity towards gram-positive fungal cultures of Bacillus subtilis, Staphylococcus aureus, and gram-negative Pseudomonas aeruginosa. Consistency of colour and the presence of bacteriostatic properties after repeated washing of knitted cotton fabrics confirms the stability of the antimicrobial properties of reusable fabrics.

Keywords: *nano composition, antibacterial activity, Suprem fabric, Interlock fabric, natural component*

Îmbunătățirea proprietăților bactericide și modificarea caracteristicilor de culoare ale tricoturilor prin utilizarea nano-argintului și a amidonului carboximetilic

În această lucrare este studiată posibilitatea obținerii tricoturilor bactericide din bumbac, prin procesarea unui nano-compozit bactericid biodegradabil care conține nanoparticule de argint și amidon Na-carboximetil. Nanocompozitul pe bază de amidon de Na-carboximetil și nanoparticule de argint a fost fixat cu succes pe suprafața tricoturilor din bumbac prin formarea de legături între grupările carboximetil ale amidonului carboximetil și nano-argint, precum și prin intercalarea aerului dintre nanocompozit și materialul textil. Analiza modificării culorii tricoturilor din bumbac după prelucrarea lor cu o soluție de nanocompozit de argint și amidon Na-carboximetil, a arătat stabilitatea indicatorilor coloristici în timpul tratamentului antibacterian.
Tricoturile din bumbac tratate cu nanocompozitul dezvoltat prezintă activitate antibacteriană ridicată față de culturile fungice gram-pozitive de Bacillus subtilis, Staphylococcus aureus și Pseudomonas aeruginosa gram-negative. Rezistența culorii și prezența proprietăților bacteriostatice după spălarea repetată a tricoturilor din bumbac confirmă stabilitatea proprietăților antimicrobiene ale materialelor textile reutilizabile.

Cuvinte-cheie*: nanocompozit, activitate antibacteriană, tricot Suprem, tricot Interlock, componentă naturală*

INTRODUCTION

Cotton fibres are the most widespread for the production of knitted materials with bactericidal properties [1]. As it is known, cotton is the most susceptible to microbial action [2]. In addition, natural fibres also have a varied retention period of microbes [3]. In this direction, the leading position is occupied by the antimicrobial processing of knitted fabrics [4, 5]. Antimicrobial materials made of cotton, linen, woollen, synthetic and silk fibres are widely used in the production of medical dressings [6, 7], napkins [8, 9], sanitary-hygienic products [10, 11], underclothing and bed linen [12, 13], hosiery [14, 15], as well as protective clothing at working with causative pathogens of dangerous infections [16].

The situation with the coronavirus COVID-19 showed the need to intensify work on the production of high-quality safe knitted fabrics with bactericidal properties [17–19]. Analysis of the latest advances in science in this direction shows that silver nanoparticles are often used for the antibacterial processing of knitted materials [20–22]. Today there are many methods of the synthesis of silver nanoparticles, but most of them are based on toxic reagents (derivatives of ammonia, glycerol, and others), used either to stabilize the resulting nanoparticles [23, 24] or to reduce silver ions [25, 26]. At the same time, it is necessary to exclude using toxic substances in the synthesis of bactericidal compounds applied for antibacterial

processing of knitted fabrics to use nanoparticles that ensure safety for the consumer [27–30].

Based on the foregoing, the purpose of the research was the formation of stabilized silver nanoparticles safe for humans in a solution of Na-carboxymethyl starch (NaCMS), the production of bactericidal knitted materials by processing the resulting nano composition, as well as the study of the effect of antimicrobial treatment on the bactericidal and physico-mechanical properties of the obtained materials.

The choice of NaCMC is due to the fact that it is widely used in the textile industry due to its biodegradability, has gel-forming, sorption and other biological properties, and is also a natural, environmentally friendly component that does not cause irritation to human skin [31].

EXPERIMENTAL PART

Materials

KnThe knittedotton fabric of the styles "Suprem" and "Interlock" (manufacturer of Uzbekistan) was chosen for research. "Suprem" is a bleached knitted fabric with, a surface density of 80 g/m^2. Suprem is considered one of the types of stockinette structure (figure 1, *a*), recognized as the thinnest among cotton jerseys (manufacturer Uzbekistan) [32]. "Interlock" knitted fabric is dyed brown with the reactive dye Orange 2R from "BEZEMA CHT Switzerland AG", surface density 120 g/m^2, dimensional stability, has a low level of stretching (manufacturer Uzbekistan). Interlock in weaving, the concept of the front and backside is absent (figure 1, *b*). It possesses good heat-shielding properties. Both canvases are 100% cotton. The choice of cotton knitted fabric for obtaining antibacterial materials with bactericidal properties based on nanosilver and carboxymethyl starch in our research was due to the fact that the knitted weave provides the greatest flexibility and softness of the fabric, and it has also high adsorption capacity, capillarity, air permeability, which contributes to the creation of the basis of products with antibacterial finishing for household and medical purposes.

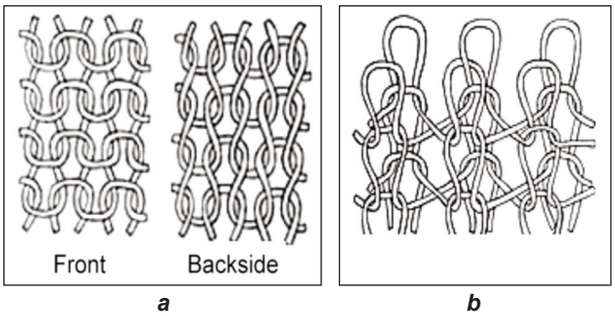


Fig. 1. Knitted weaving: *a* – supreme (stockinette stitch); *b* – interlock

Chemically pure silver nitrate (JSC "Reakhim", GOST 1277-75) was used to obtain the nanocomposite. All chemical substances were used as purchased, without any further purification or processing. NaCMS produced by LLP "Khlopkoprom-cellulose" (manufactured of Kazakhstan), the mass fraction of NaCMS of 99.6% (ST LLP 40936697-004-2015) was used as a reducing agent.

The reduction of silver ions and synthesis of silver nanoparticles

The method of reduction of silver ions using NaCMS and ultraviolet radiation was used in this work.

Photochemical reduction of silver ions in the Ag^+CMS^- structure to nanoparticles was carried out by irradiating them with an RDS-250-3 ultrahigh-pressure mercury-quartz ball lamp.

Ultrasonic dispersers of the UVRP-1, U-4.2 brands were used to recover dispersions of silver nanoparticles.

The synthesis of nanoparticles was carried out by reducing silver ions in an aqueous solution of NaCMS under the combined action of ultraviolet radiation with a wavelength of 280–400 nm and ultrasonic waves with a frequency of 1.7 MHz.

The reaction proceeds according to the following equation:

$$R-COO^- + Ag^+ \xrightarrow{hv} R-COO:Ag^0 \quad (1)$$

$$R-COO^- + Ag^0 + e \rightarrow R-COO^-:Ag^0 \quad (2)$$

$$R-COO^-:Ag^0 + Ag^+ \xrightarrow{hv} R-COO:Ag_2^0 \quad (3)$$

$$R-COO^-:Ag_2^0 \xrightarrow{hv} R-COO:Ag_n^0 \quad (4)$$

Due to the presence of negatively charged ions (COO–) in the carboxymethyl group, carboxymethyl starch interacts with silver cations, linking them into a strong complex (1), reducing them under the influence of UV directly in this complex (2), and stabilize sequentially formed during synthesis small charged clusters and silver nanoparticles (3) [33, 34]. Thus, the entire process of nanoparticle formation from the initial cation to the final state particle proceeds in direct contact with the polymer matrix [35].

Recovering of aqueous composite solution

50 ml of water was poured into the container, 0.5 g of NaCMS was added and it was stirred with a mechanical stirrer for 10 min, then 50 ml of 0.50% (1.0%) solution of silver nitrate was added into this solution and it was stirred until a homogeneous solution for 15–20 minutes to get an aqueous composite solution containing 0.500 mass % of NaCMS and 0.15 mass % (0.30 mass %) of silver nanoparticles. The resulting solution was subjected to ultrasonic dispersion and ultraviolet radiation for 15 minutes.

Processing of knitted fabrics

The processing of knitted fabrics in order to avoid deformation of their structure was carried out by the method of aerosol application of the solution. 100 ml of the obtained composite solution of silver nanoparticles (100% application) was sprayed onto 100 g of

knitted material by aerosol method, after which the material was subjected to ultraviolet radiation for 15 min. After radiation, the treated material was dried in a model 202-OE drying oven at 120°C to a residual moisture content of 6%.

Diagnostics and testing of knitted materials

The electron microscopy was carried out using a JEOL JSM–6490 LV scanning electron microscope with an accelerating voltage from 0.3 to 30 kV to evaluate the surface morphology of the fibres of knitted fabrics and the sizes of nanoparticles.

The study of the antibacterial activity of knitted fabrics treated with the nanocomposite composition $Ag^{+}CMS^{-}$ was carried out in accordance with the international standard ISO 20743:2007 "Textiles – Determination of the antibacterial activity of antibacterial finished products".

The change in the colour characteristics of knitted fabrics after antibacterial treatment with $Ag^{+}CMS^{-}$ nano composition was tested on the laboratory colourimeter according to the method [36] in standard radiation D_{65} [37]. Color characteristics were determined by the CIELAB formula, recommended by the Commission internationale de l'éclairage (CIE) [38].

The breaking load and elongation of the strip were determined on the tensile testing machine of the RT-250 type according to GOST 8847-85.

The resistance to abrasion of materials was determined on the IT-3M, TI-1 or TI-1M devices by the number of abrasion cycles before the destruction of the fabric according to GOST 12739-85. The method is based on determining the resistance of the knitted fabric to abrasion by the number of revolutions of the device heads until the destruction of the elementary sample.

The moisture content of knitted fabrics was determined according to GOST 8845-87. The principle of determining moisture content was that a certain amount of knitted fabric was dried to constant weight, and the amount of moisture in the studied knitted fabric was found from the difference between the initial weight and the weight of the dry residue.

The capillary test was determined according to GOST 3816-81 (ISO 811-81) "Textile fabrics. Methods of determination of hygroscopic and water-repellent properties". The essence of the method is to determine the height of the capillary rise of fluid in the fabric. Wet processing of knitted fabrics was carried out in accordance with ISO 6330:2012 "Textiles – Domestic washing and drying procedures for textile testing.

RESULTS AND DISCUSSION

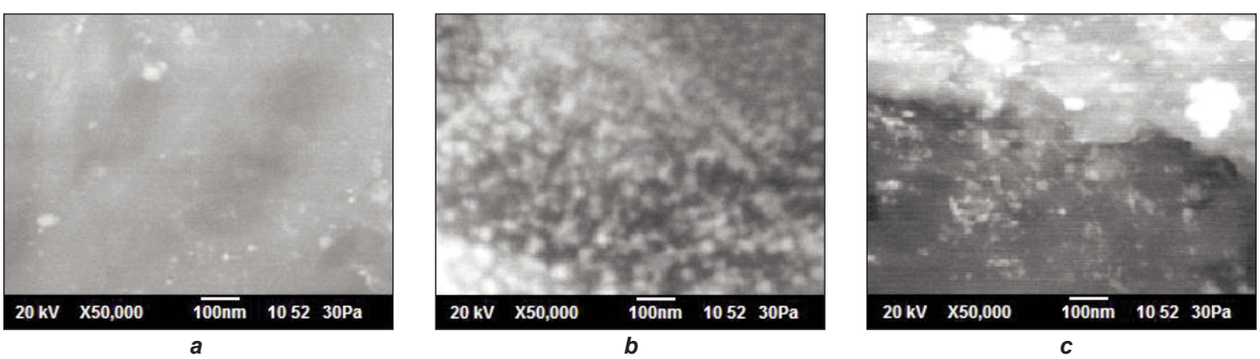
Surface morphology of dyed knitted fabrics treated with Ag carboxy-methyl-starch (AgCMS) nano composition

The surface morphology of the fibres of knitted fabrics modified with the AgCMS nano composition was determined in a scanning electron microscope by scanning electron microscope analysis of figure 2.

The scanning electron microscopy results show that the treatment of knitted fabrics with the nanocomposite composition AgCMS (figure 2, *a* and *b*) leads to a uniform distribution of almost spherical Ag agglomerates on the surface of the fabrics, mainly with sizes of about 10–40 nm. A large number of Ag nanoparticles are visible on the surface of the knitted fabrics, which indicates the successful fixation of AgCMS. Larger agglomerates with sizes from 10 to 120 nm are formed on the surface of knitted fabrics at treating with colloidal silver without the addition of carboxymethyl starch (figure 2, *c*). The absence of carboxymethyl starch led to the agglomeration of Ag particles and the formation of larger silver clusters. These results showed the role of carboxymethyl starch in the reduction of nanosilver and its ability to stabilize the AgCMS system.

The conditions for the implementation of the recovery method and the results of testing the antibacterial activity of knitted fabrics treated with the developed nanocomposite solution AgCMS are presented in table 1 and figure 3, respectively.

The presented results indicate (figure 3) that knitted fabrics treated with the developed nanocomposite solution $Ag^{+}CMS^{-}$ have high antimicrobial activity towards gram-positive fungal cultures of Bacillus subtilis (5.5–6.9), Staphylococcus aureus (5.8–7.0) and gram-negative Pseudomonas aeruginosa (5.6–.6). It can also be seen that with an increase in the silver content in the cloth twice, the antibacterial activity increases by only 0.7–1.2 units. Control samples treated with colloidal silver without reduction with



a *b* *c*

Fig. 2. Scanning electron microscopy of knitted fabric fibres modified with AgCMS nano composition; the processing of textile fabric: *a* – CMS – 0.5 wt.% and Ag – 0.15 wt. %; *b* – CMS – 0.5 wt. % and Ag – 0.30 wt. %; *c* – Ag – 0.15 wt. %

Table 1

CONDITIONS OF THE IMPLEMENTATION OF THE RECOVERY METHOD OF NANO COMPOSITE KNITTED MATERIALS

№ samples	Knitted materials	Concentration of NaCMS in solution (wt.%)	Concentration of silver salt in solution (wt.%)	Concentration of silver nanoparticles on the material (wt.%)
1	Suprem (white)	absent	0.25	0.15
2	Suprem (white)	0.500	0.25	0.15
3	Suprem (white)	0.500	0.50	0.30
4	Interlock (brown)	absent	0.25	0.15
5	Interlock (brown)	0.500	0.25	0.15
6	Interlock (brown)	0.500	0.50	0.30

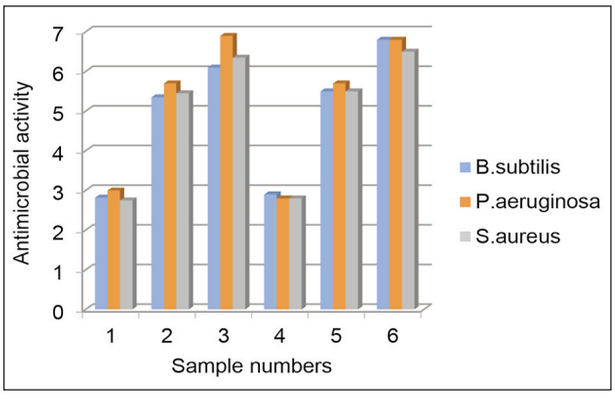


Fig. 3. The value of the antibacterial activity of knitted fabrics modified with a solution of $Ag^{+}CMS^{-}$: 1,2,3 – Suprem; 4,5,6 – interlock

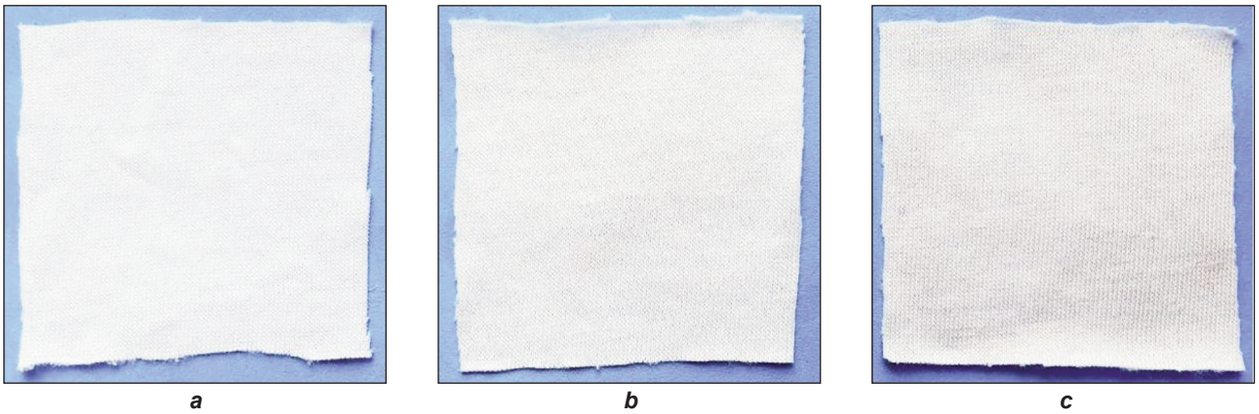
carboxymethyl starch have almost half the antibacterial effect in relation to all selected fungi. From the obtained data, it follows that the developed nanocomposite solution AgCMS, proposed for antibacterial processing of knitted materials, has a depressing effect in relation to the selected fungi and therefore, meet the requirements for cellulose materials directed to the manufacture of sanitary-hygienic and technical purpose.

The research of the effect of treatment with a solution of the AgCMS nanocomposite on the colcolouror characteristics of bleached and dyed knitted fabrics is shown in figures 4 and 5.

Figures 4, *b* and 5, *b* show that the colour changes slightly at using bleached and coloured knitwear with a solution containing 0.15% Ag. The material acquires a slightly greyish colour, that is, it darkens a little at processing bleached knitwear with a solution containing Ag – 0.30% (figure 4, *b*). The change can also be seen visually at processing coloured knitwear with a solution containing Ag – 0.30% (figure 4, *c*). This is due to the fact that silver particles introduced into the nano composition system can affect the structure and properties of light-gamma dyes. The colour characteristics of knitted fabrics were used adopted by the International Commission on Lighting in the CIE L *a* b* colour space to clarify the effect of the AgCMS nano composition.

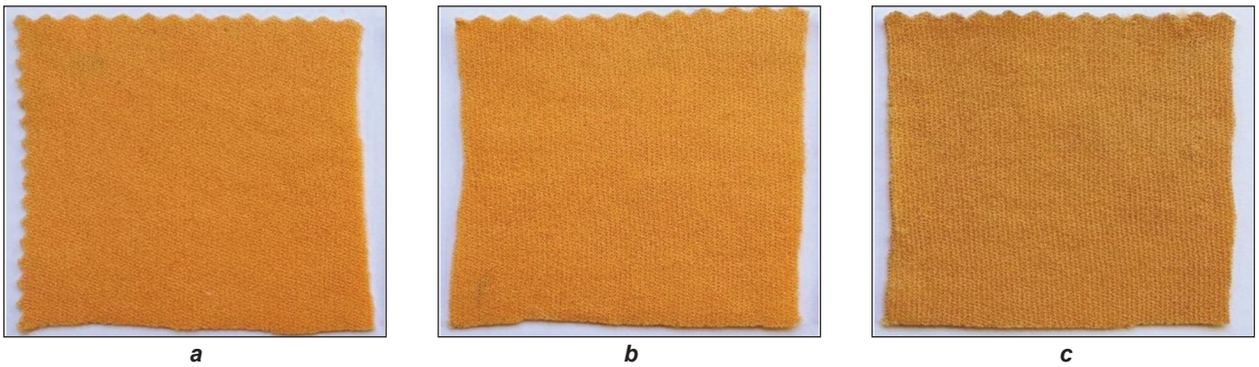
The results of colorimetric studies of changes in the colour characteristics of fabric after bactericidal finishing with nanoparticles of silver and carboxymethyl starch are shown in table 2.

According to the data in table 2, the original Suprem fabric was white with a high-value L* and insignificant a* and b* values. The values of lightness L* of the treated fabric decreased slightly from 95.51 to 94.01. After treatment with a nanocomposite of silver and CMS. The L* value also decreased slightly at processing a painted fabric. The discolouration of knitted



a *b* *c*

Fig. 4. Photographs of bleached knitwear "Suprem", before and after treatment with a nanocomposite solution AgCMS: *a* – original sample without treatment; *b* – treated with a solution containing CMS – 0.5 wt.% and Ag – 0.15 wt. %; *c* – treated with a solution containing CMS – 0.5 wt. % and Ag – 0.30 wt. %



a *b* *c*

Fig. 5. Photographs of knitted fabric "Interlock", dyed brown before and after processing with nanocomposite solutions AgCMS: *a* – original sample without processing; *b* – treated with a solution containing CMS – 0.5 wt. % and Ag – 0.15 wt. %; *c* – treated with a solution containing CMS – 0.5 wt. % and Ag – 0.30 wt. %

Table 2

COLOUR COORDINATES AND REFLECTION SAMPLES COEFFICIENT					
№ samples	**Name of the material**	**Samples**	**Brightness L***	**Coordinates**	
				a*	**b***
1	Suprem (white)	original	95.51	2.75	–9.37
2	Suprem (white)	0.5% CMS+0.15%Ag	95.25	2.06	–10.59
3	Suprem (white))	0.5% CMS +0.30%Ag	94.01	2.94	–10.96
4	Interlock (brown)	original	73.08	24.72	33.63
5	Interlock (brown)	0.5% CMS +0.15%Ag	72.41	24.27	33.13
6	Interlock (brown)	0.5% CMS +0.30%Ag	68.92	21.06	29.38

fabrics depends on the content of Ag nanoparticles. The colour characteristics of both the white and the dyed fabric did not change significantly with Ag content of 0.15% in the knitted fabric. The analysis of the change in the colour of cotton knitted fabrics carried out in this work after treatment with a solution of the nano composition of silver and AgCMS showed the stability of the colouristic indicators of colour during antibacterial treatment and proved the effectiveness of the proposed nano composition used in the bactericidal treatment of sanitary and hygienic and household products.

Further, the operational and hygienic properties of the initial and processed by the nanocomposite knitted fabrics were investigated. We have determined the following indicators: breaking force on the loop stitches, extensibility, stretching property in width and abrasion resistance from the performance properties that affect the service life of knitted products. The results of testing the operational characteristics of the initial and prototypes of knitted fabrics treated with the AgCMS nanocomposite solution are shown in table 3.

Table 3

CHARACTERISTICS OF KNITTED FABRICS TREATED WITH NANO COMPOSITE SOLUTION AgCMS AND WITHOUT TREATMENT (FABRICS TREATED WITH SOLUTION CONTAINING 0.5%CMS+0.15%Ag)					
Type of treatment	**Name of knitted fabric**	**Quality indicators**			
		Breaking load on the buttonhole posts (H)	**Extensibility (mm)**	**Stretchability in width (%)**	**Abrasion resistance (turns)**
AgCMS	Suprem	105	11 ±1	65	27
AgCMS	Interlock	156	6 ± 1	32	54
Without treatment	Suprem	100	12±1	66	24
Without treatment	Interlock	147	6±1	35	45
Requirements of GOST 28554-90		Not less than 80	-	I – from 0 till 40 II–from 41 till 100 III – above 100	common15–30 fast 31–60 extra fast 61 and more

Table 4

HYGIENIC CHARACTERISTICS OF KNITTED FABRICS TREATED WITH NANO COMPOSITE SOLUTION AgCMS (0.5%CMS+0.15%Ag) AND WITHOUT TREATMENT

Type of treatment	Name of knitted fabric	Quality indicators			
		Breaking load on the buttonhole posts (H)	Extensibility (mm)	Stretchability in width (%)	Abrasion resistance (turns)
AgCMS	Suprem	161	44	8,2	6.6
AgCMS	Interlock	163	48	9,0	7.0
Without treatment	Suprem	157	41	8,0	6.5
Without treatment	Interlock	158	47	8,8	7.0

According to table 3, samples of knitted fabric treated with nanocomposite solution of AgCMS, in comparison with samples without treatment, have greater strength and resistance to abrasion, and according to other indicators, at the level of standard requirements. It is possible that carboxymethyl starch, being a polymer component of nanocomposite solution sprayed onto a knitted fabric, forms polymer films on its surface, which increase tensile strength and abrasion resistance.

The test results of characteristics affecting the hygienic properties of knitted materials samples treated with the AgCMS nanocomposite solution are shown in table 4.

The results of table 4 show that samples of knitted fabric treated with nanocomposite solution of AgCMS, compared to samples without treatment, have higher capillarity, fluid loss and porosity. This confirms that the processing of knitted fabric with developed nanocomposite solution AgCMS has a beneficial effect on the hygienic properties of knitted fabric. The improvement in the hygienic properties of knitted fabrics can be explained by the fact that the carboxymethyl starch used in the composition of the nanocomposite solution is a hygroscopic polymer. At processing knitted fabric with the developed composition, reticulated polymer structures are formed on its surface, which lead to an increase in the hygienic characteristics of the materials under study.

The test results of the effect of the number of washings on the antibacterial activity of knitted fabrics treated with the nanocomposite solution AgCMS are shown in figure 6.

It was found that a sufficiently high antimicrobial activity retention, slightly decreasing only after the tenth wash in a washing solution as a result of the study of the resistance of knitted fabrics treated with

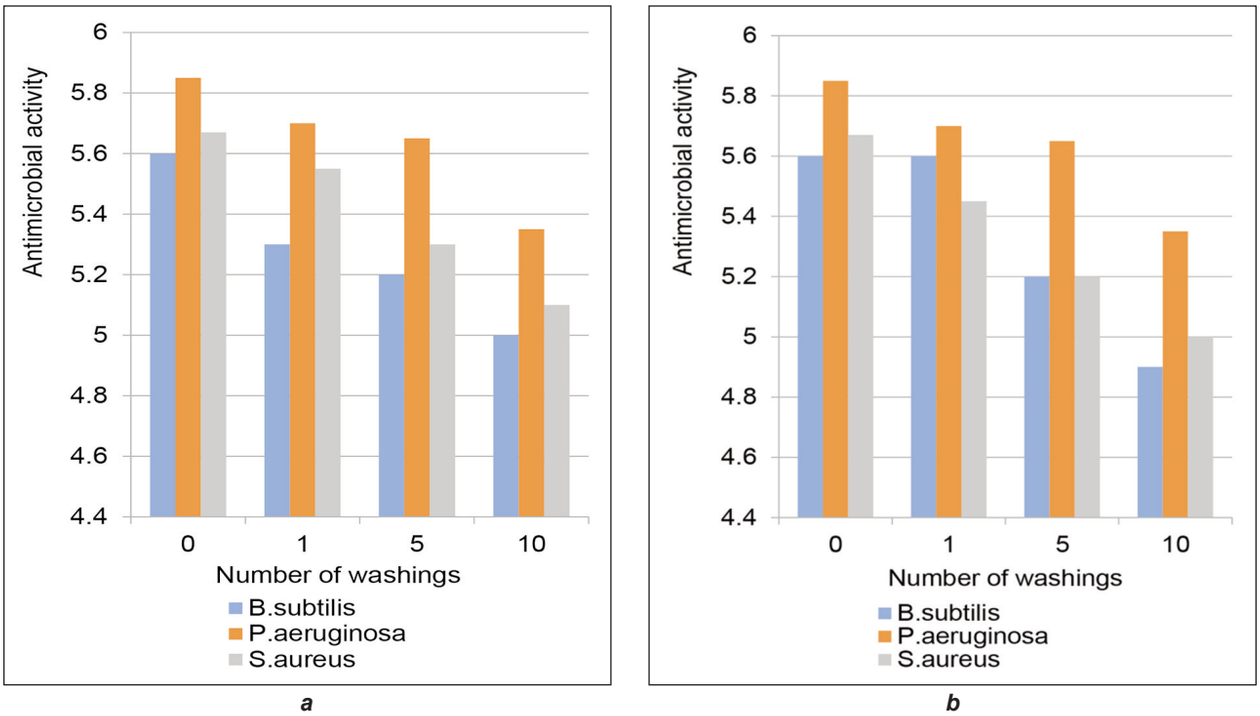


Fig. 6. The effect of the number of washings on the value of the antibacterial activity of knitted fabrics modified with solution of AgCMS: a – suprem; b – interlock

a nanocomposite solution of AgCMC to wet treatments (figure 6, *a* and *b*). The stability of the antimicrobial properties of reusable fabric was proved by the presence of bacteriostatic properties after multiple washes.

CONCLUSIONS

The studies have shown that the usage of NaCMS in the developed composition makes it possible to obtain stable systems of silver nanoparticles in the knitted fabric with a sufficient distribution of nanoparticles in size. The stability of nanoparticles is achieved due to the fact that NaCMS, binding with silver particles, creates a charged shell around them, preventing aggregation.

It has been shown that cellulose knitted fabrics treated with the developed nanocomposite solution of AgCMS have high antimicrobial activity towards gram-positive fungal cultures of *Bacillus subtilis*, *Staphylococcus aureus* and gram-negative *Pseudomonas aeruginosa*. The stability of colour indicators of dyeing during antibacterial treatment proved the effectiveness of the proposed nano composition. The discolouration of knitted fabrics depends on the content of Ag nanoparticles.

It was found that the treatment with the developed composition has a beneficial effect on the consumer and hygienic properties of the knitted material. The improvement in the hygienic properties of knitted fabrics can be explained by the fact that the carboxymethyl starch used in the composition of the nanocomposite solution is a hygroscopic polymer. At processing knitted fabric with the developed composition, reticulated polymer structures are formed on its surface, which lead to an increase in the hygienic characteristics of the materials under study.

The knitted fabrics exhibit excellent antibacterial properties and excellent washing resistance. After 10 wash cycles, the resulting fabrics still showed excellent bacterial resistance against fungal cultures of *Bacillus subtilis, Staphylococcus aureus* and *Pseudomonas aeruginosa*. The stability of the antimicrobial properties of reusable fabric was proved by the invariability of colour and the presence of bacteriostatic properties after repeated washing.

It should be noted that the composition formulation includes natural components, which makes it possible to recommend the developed composition based on silver nanoparticles for processing sanitary and hygienic products and knitted materials in order to impart bactericidal properties to them.

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