INVESTIGATION OF ANTIMICROBIAL PROPERTIES OF TEXTILE MATERIALS AFTER WASHING

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Abstract: *This work is devoted to the study of antimicrobial properties of cellulose-containing textile materials treated with new safe biocidal products of thiosulfonate structure. A resource-saving method of providing antimicrobial properties to cellulose-containing textile materials is presented. High antimicrobial activity of biocidal products after washing was established. The duration of action and expediency of their use in the textile industry are proved. It is shown that after 10 washes the treated tissues lose only 14-15% of antimicrobial properties.*

Keywords: *textile materials, biocidal treatment, washing, microorganisms.*

1 INTRODUCTION

It is known that one of the most common types of destruction of textile materials under the influence of the environment is their microbiological damage, which occurs due to the development of three main types of microorganisms: bacteria, actinomycetes and fungi. It is also known that on the surface of any textile fiber can be found microflora, which at high relative humidity and optimum temperature for its development is able to eventually absorb the fibers as a nutrient substrate and leads to their destruction [1-4]. Textile materials based on natural fibers, such as cotton, linen, and others, which are utilized by the saprophytic microflora in the nutrient cycle, are most prone to microbiological damage. And today the problem of microbiological stability of textile materials used in different climatic zones, especially with high humidity, in particular in marine areas, remains unsolved [5-7]. In the textile industry there is a continuous state of exploration for more advanced and environmentally friendly technologies, nanomaterials for antimicrobial treatment of cellulose-containing textile materials for various purposes. Although modern biocidal substances inhibit the growth of most microorganisms, they are not effective enough, and some of them are toxic and dangerous to humans and the environment [8]. Therefore, the issue of development of ecological textile materials with long-acting antimicrobial properties remains relevant and open.

Analysis of literature studies has shown that today there are several thousand names of biocidal substances on the world market. The main participants in the global market for biocides are BASF SE (Germany), AkzoNobel N.V. (Netherlands), Ashland Inc. (USA), Champion Technologies (USA), Cortec Corporation (USA), Clariant AG (Switzerland), LANXESS AG (Germany), Lonza Group Ltd (Switzerland), Nalco Holding Company (USA), Thor Group Limited (UK) and Troy Corporation (USA) [9].

The world leader is the Swiss company Sanitized AG, which has been producing biocidal substances for textiles, leather and plastics for universal and special purposes for 70 years. The most well-known and effective developments for the textile industry of this company are Sanitized T25-25 series of biocides - based on silver, which reduces the development and reproduction of micro-organisms and suppresses odors, and Sanitized T 96-21 - bacteriostatic preparation based on triclosan [10, 11]. The antibacterial agent triclosan is the basis of many biocidal substances that act on gram-positive and gram-negative flora. But in recent years, the possibility of widespread use of triclosan as an antimicrobial agent is the subject of debate due to the ability of the preparation in chemical transformations to emit dioxin, and today the regulations include non-recommended antimicrobial substances.

One of the most effective for bio-sustainable processing of textile materials is also a foreign preparation LSL in the form of an emulsion. It is resistant to high temperatures and gives tissues a significant bioprotective effect. However, the disadvantage of this preparation is a marked decrease in the stability of cotton fabrics after prolonged exposure to light weather.

Increased antimicrobial activity has a textile material made of chemically refined linen fiber and contains the antiseptic drug iodpirone, which retains its softness and is widely used in the manufacture of special gaskets, bedding, and clothing. It retains antimicrobial properties to a number of micro-organisms, after five washes it contains 42% of the preparation, and after ten - 20% of its initial content. It is used as an antiseptic. Non-woven fabrics containing catamine AB + potassium iodide (alkyldimethylbenzylammonium chloride, cationic surfactant antiseptic) with high antimicrobial activity and resistance to wet treatments have been developed.

Kathon MW (USA) based on diazolines is considered to be a universal highly effective antimicrobial agent, which has been widely used in various industries. Methods for imparting antimicrobial properties to textile materials by introducing nitrofuran preparations into spinning solutions with their subsequent fixation in the fine structure of fibers during molding have also been developed. A significant place among them is occupied by guanidine compounds as physiologically active substances. Their bactericidal action is determined by their ability to bind to bacterial membranes, penetrate the cell nucleus and produce cellular enzymes [12]. But today it is classified as a toxic biocide.

Quaternary aluminium base salts (QAS) play a significant role as antimicrobial treatment. However, the number of critical publications on their use has increased. The authors note that some species of microorganisms have a natural resistance to QAS, others quickly acquire them, creating a biofilm that neutralizes active substances. In addition, there is a lack of activity of QAS against picornaviruses, pseudomonads, and mucoid strains of staphylococci [13].

California scientists have studied the biocides of 3-methylol-2,2,5,5-tetramethylimidazolidinone and 1,3-dimethylol-5,5-dimethylhydantoin and their compositions in different ratios for chemical modifications of textile cellulose. It has been found that the use of such compositions for cellulose treatment improves the strength and stability of biocidal functions of tissues treated with these drugs, in addition, these fabrics can withstand repeated washing and long shelf life [14].

Another achievement in this direction are cyclodextrins [3, 15], which are widely used for tissue treatment, because they due to their unique chemical structure show good absorption capacity, namely form complexes with various antimicrobial and other biologically active substances.

Nanotechnology also plays an important role in the development of antibacterial textiles. As a promising tool for creating highly stable, effective and environmentally friendly antibacterial textile coatings, sonochemistry was first used by applying inorganic nanoparticles (CuO, ZnO, MgO) to tissues without damaging the structure of textile materials [16-21]. The bactericidal properties of such textile coatings have been preserved after repeated washing, making them an alternative to known bactericidal preparations such as triclosan, various quaternary ammonium salts and other toxic compounds.

In particular, the work of textile materials with antimicrobial properties is devoted to many works of scientists who had found that the protection of natural textiles from biodegradation is possible due to the action of biocidal substances, and some of them even improve performance [22-31]. But, analyzing in general the properties of some biocidal products, we conclude that all of them have significant disadvantages: low weather resistance; short duration of action, high cost and insufficient resistance of the antimicrobial effect to washing. Another problem is the reduction of wear resistance during operation, in particular washing, during which the products are exposed to physico-chemical and mechanical factors. Therefore, we have a goal to obtain environmentally friendly textile materials with high long-acting antimicrobial properties, while maintaining quality characteristics.

2 METHODOLOGIES

The analysis of the Ukrainian market showed that the vast majority of fabrics for the production of overalls are cellulose-containing fabrics (cotton + polyester) - 68%, then for experimental studies selected 3 variants of textile materials (Table 1): option 1 - "Toctals Fabrics"); Option 2 - OJSC Ternopil Association "Texterno" (Ukraine); Option 3 - PJSC "Cherkasy Silk Factory".

Table 1 Characteristics of objects

Number option	Fiber content	Surface density [g/m^2]	Porosity [%]	Type of weave	Brands of dyes
1	100% cotton	245	41.2	twill	Straight orange Indosol
2	50% cotton 50% polyester	245	37.5	twill	Dispersive «Foron» RD-SN
3	35% cotton 65% polyester	220	30.6	twill	Optical bleach CBS-X (OBA 351)

As noted above, today the search for low-toxic biocidal products, which would not only solve the problem of protecting textiles and products from microbiological destruction, but also would improve their quality characteristics, is relevant worldwide. One of the new promising environmental developments in this direction today can be considered biocidal products of thiosulfonate structure, synthesized at the Department of Technology of Biologically Active Compounds, Pharmacy and Biotechnology of the National University "Lviv Polytechnic".

To protect cellulose-containing textile materials and clothing products from the negative effects of fiber-destroying and pathogenic microorganisms, we selected new biocidal preparations having the thiosulfonate structure: Ethylthiosulfanilate (ETS), Allylthiosulfanilate (ATS), Methylthio-sulfanilate (MTS), which exhibit a wide spectrum of antimicrobial activity and are non-toxic, and can be used for antimicrobial protection in various industries [32-35].

These biocidal products are effectively used as biocides to protect paints and varnishes; additives for protection against bio-damage of lubricating and cooling liquids; biocidal component of anti-corrosion composition for pipelines of circulating water supply systems, oil products, building materials and structures; algaecides for protection of surfaces, packaging materials, for sterilization of culture fluid in biotechnological productions, etc. [36].

Thiosulfonate biocides have not yet been tested in light industry, and we decided for the first time to experimentally investigate the antimicrobial properties of these drugs for textiles. This is dictated not only by the broad spectrum of action of thiosulfonate compounds, but also by the attempt to solve the problem of finding low-toxic and ecological biocides [34, 37]. These preparations, in our opinion, may be ideal for the terminology "environmental biocides", as they are also an active substance for the treatment of various skin mycoses and onychomycosis of the nails, competitive with nizoral and clotrimazole.

Antimicrobial treatment of cotton-polyester cloths was carried out at the Analytical and Research Testing Laboratory "Textile-TEST", Kyiv (Kyiv National University of Technology and Design). Samples of tissues were treated by water alcoholic solution (60/40) of ETS, MTS and ATS preparations in padding dyer at room temperature 18-20*°C* and relative humidity of the air 63-65%. Subsequently, these test specimens were pressed using padding dyer to a residual moisture content of 6-8% and dried at 70, 60 and 50°C. The concentration of ETS, MTS and ATS in water alcoholic solutions was 0.5%. Before all determinations of textile quality, fabric samples were being dried for 5-7 min [38].

The test samples were washed in a Bosch washing machine, bath module - 1:30 at a temperature of 40°C, rinsing - 3 cycles at a temperature of 21±3°C, dehydration - centrifuge according to the washing machine program, dried at room temperature. This mode of washing causes minimal damage to the fabric structure, but according to the developers it can be washed at temperatures up to 90° C and antimicrobial properties are not lost. Washing was carried out according to regulated standard methods [39]. In order to conduct rational experiments, research on the use of synthetic detergents was conducted not in the laboratory, but in real production conditions.

Experimental tests were conducted in the port of Odessa in the working conditions of 9 dockers-mechanics for one year. Workers' overalls were made from 100% cotton treated with ETS and MTS biocides, as they proved to be more effective. The workers worked in one shift under the same conditions. The monthly change of dockers was 15 days at night and during the day. Clothes were washed twice a month in a Bosh washing machine at a temperature of 60°C and synthetic detergent "Losk", squeezed at 800 rpm.

The guarantee error of the coefficient of variation (mc) was in the range of 1.5-2.5%. To determine the antimicrobial activity of textile materials we used certain strains of microorganisms that have a destructive effect on cellulose fibers. Studies of bacterial resistance and fungal resistance of tissue samples were performed according to standard methods. Sterile meat-peptone agar (MPA) for bacteria and wort agar (WA) for fungi were used for the experiment. The following types of microorganisms were used in the tests: *Escherichia coli, Staphylococcus aureus, Mycobacterium luteum, Candida tenuis* and *Aspergillus niger*. To do this, sterile agar medium cooled to 40-45°C was poured into Petri dishes, in which a suspension of microorganisms had been previously inoculated (microbial load: bacteria 109 CFU/ml; fungal spores 107 CFU/ml). The prepared samples were immersed in agar medium, cups with experimental and control samples were incubated in a thermostat for 24-48 hours at a temperature of 37°C for germination of bacteria and 48-72 hours at a temperature of 28-30°C for fungi.

3 RESULTS AND DISSCUSION

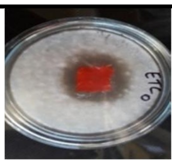
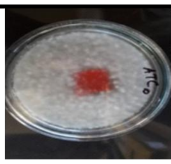
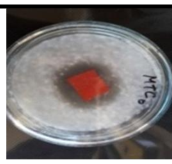
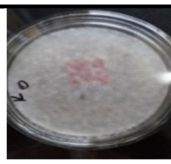
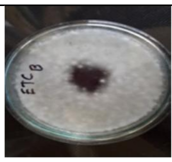
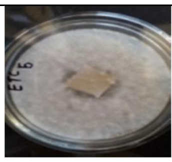
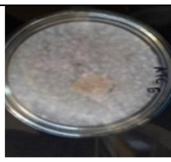
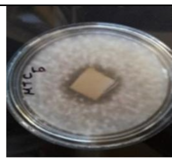
It is known that during washing the antimicrobial properties are lost and the duration of action of the preparations is reduced. Therefore, the aim of our work was to study the resistance of textile materials with biocidal treatment to repeated washing. Antimicrobial activity of textile materials treated with biocidal products ETS, MTS and ATS are shown in Table 2.

Table 2 Antimicrobial activity of cellulose-containing fabrics

Sample number	Fibrous composition	Type of processing	Zones of growth retardation of microorganisms* [mm]				
			Aspergillus niger	***Candida tenuis***	***Mycobacterium luteum***	***Escherichia coli***	***Staphylococcus aureus***
1	100% cotton	without processing	0	0	0	0	0
		ETS	40.0	69.0	49.0	28.0	34.7
			10.0	24.5	14.5	4.00	7.4
		MTS	44.0	56.0	67.5	35.0	32.0
			12.0	18.0	23.8	7.5	6.0
		ATS	20.0	50.0	43.0	25.0	33.7
			0	15.0	11.5	2.5	6.9
2	50% cotton 50% polyester	without processing	0	0	0	0	0
		ETS	27.0	65.0	39.0	0	32.6
			3.5	22.5	9.5		6.3
		MTS	22.0	58.4	43.0	0	31.7
			1.0	19.2	11.5		5.9
		ATS	0	49.0	43.7	0	31.4
			0	14.5	11.9		5.7
3	35% cotton 65% polyester	without processing	0	0	0	0	0
		ETS	29.0	67.5	39.0	0	27.0
			4.5	23.8	9.5		3.5
		MTS	38.0		45.0	0	26.0
			9.0		12.5		3.0
		ATS	0	50.0	40.0	0	28.7
			0	15.0	10.0		4.4

*includes the sizes of a fabric sample (20 mm) in the denominator - the zone of growth retardation of the sample.

As a result of the tests, it was proved that ETS, MTS and ATS selectively inhibit the activity of microorganisms (photos are presented in Figure 1). It was found that the delay of the growth zone of the selected test cultures of microorganisms depends not only on the preparation and its concentration, but also on the physiological group and type of microorganisms and fibrous tissue composition (corresponding ratio of cotton and polyester fibers). For example, the sample № 3 has a content of cotton fibers 35% and polyester 65%. For example, if the growth inhibition of *Mycobacterium luteum* and *Staphylococcus aureus* is affected by all selected preparations - ETS, MTS and ATS, but ETS inhibits only the growth of *Escherichia coli*. Gram-negative *E. coli* is the least sensitive to the action of three biocidal preparations, possibly due to the special structure of the cell wall. As for *Candida tenuis* - the zone of growth retardation is the largest - 67 mm in pure cotton fabric treated with ETS, but the same effect can be seen on samples treated with MTS and ATS. As for the fungus *Mucor*, this genus of fungi does not cause real decomposition of cellulose, but rather has the character of surface growth, but also reduces the physico-mechanical and physico-chemical properties of the fibers.

Sample number	***Aspergillus niger***			
	ETS	**ATS**	**MTS**	**Control**
1				
2				
3				

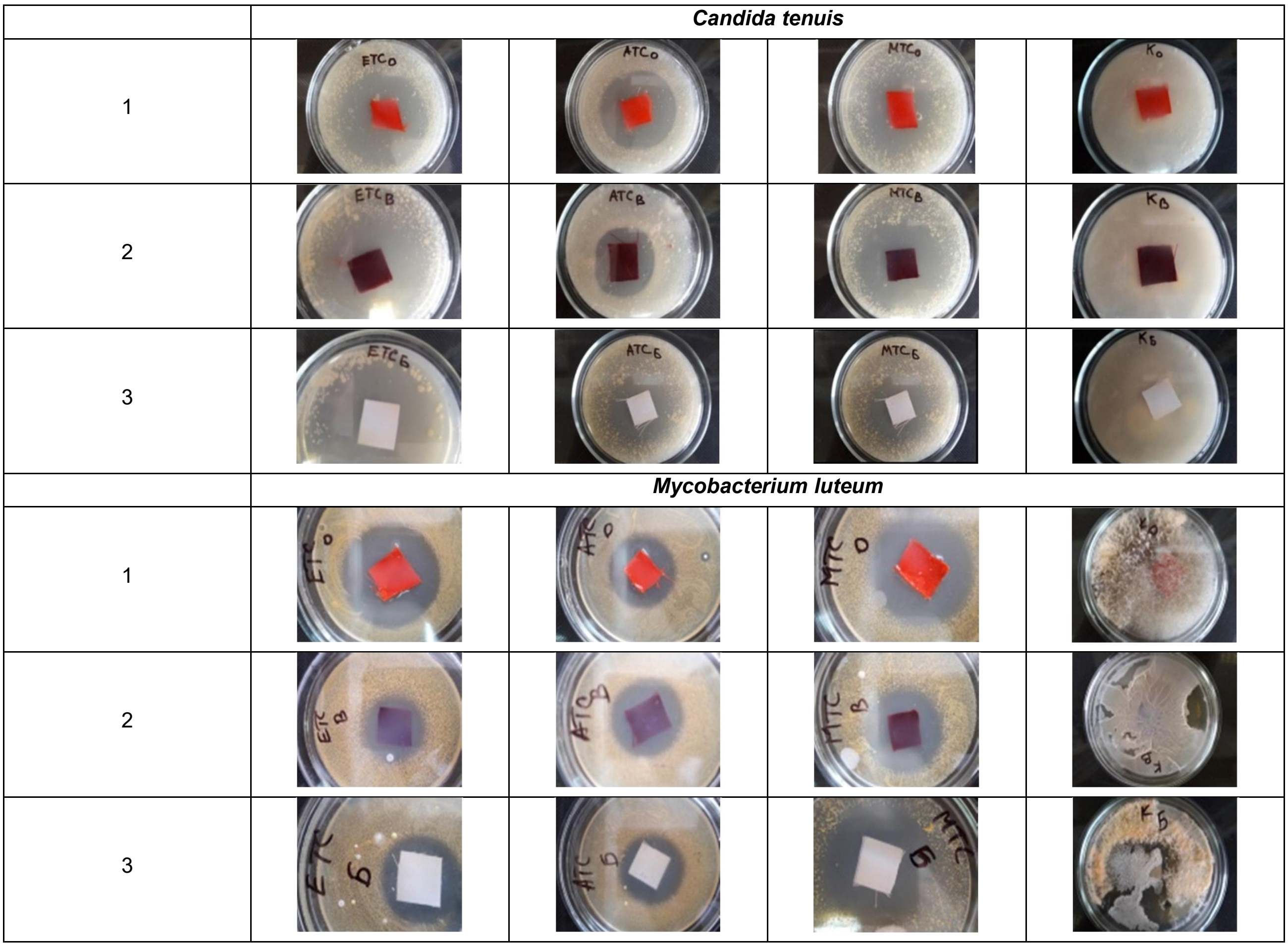


Figure 1 Photographs of experiments towards antimicrobial activity

The results of research again confirm that the most aggressive microorganism among fungi is *Aspergillus niger*, which causes a real breakdown of cellulose. The growth of colonies of fungi *Aspergilllus niger* is inhibited only by ETS and MTS. The best effect of biostability is achieved on pure cotton fabric. Antimicrobial activity was established experimentally after 1, 3 and 10 washes in water. The results of the resistance of biocidal products after repeated washing are clearly presented in Figures 2-4

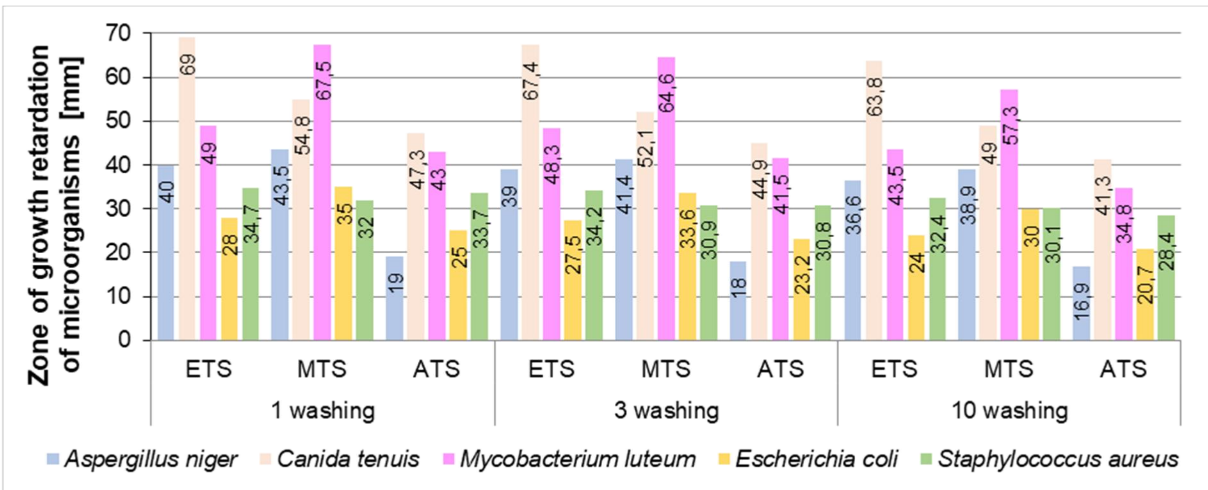


Figure 2 Resistance to antimicrobial activity var. 1 (**100% cotton**) after 10 washes

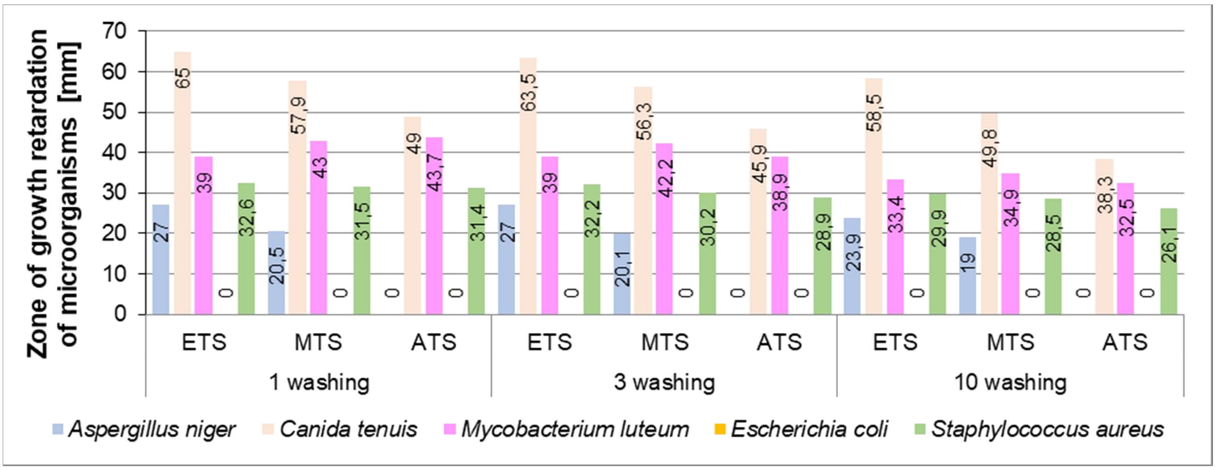


Figure 3 Resistance to antimicrobial activity var.2 (**50% cotton/50% polyester**) after 10 times washing

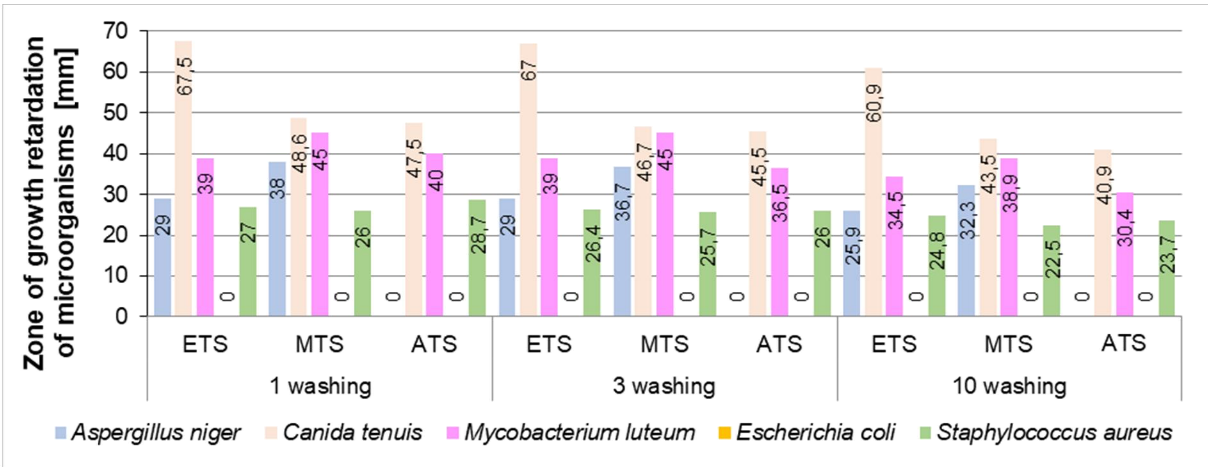


Figure 4 Stability of antimicrobial activity var.3 (**35% cotton/65% polyester**) after 10 times washing

The test results showed that the most effective of the three biocides after wet treatments in water was ETS, as the zone of growth retardation of microorganisms after 10 washes on all 3 variants of the studied samples decreased from 8.5 to 14% depending on tissue composition and microorganism. While in MTS this figure ranged from 11 to 15%, and the least effective in comparison was the biocidal product ATC, where the antimicrobial activity of the biocide after 10 washes decreased from 15 to 25%. But, in general, it can be stated that all biocidal products have high antimicrobial properties and can be effectively used in the textile industry. But in order to obtain a longer antimicrobial effect, ETS and MTS are recommended to use. Studies have also been conducted to determine the effect of biocidal treatment on the light fastness of textile materials with biocidal treatment. The results of studies [40] have shown that significant advantage of ETS, MTS and ATS preparations is the ability to inhibit the process of discoloration of dyeing on clothing textile materials, as well as in the process of light aging. It was also found that the preparations ETS, MTS and ATS, in addition to protection against light aging of paints, significantly inhibit light aging and the fibrous basis of the studied samples. It has been established that in the process of tissue irradiation, ETS, MTS and ATS preparations absorb part of the solar energy incident on the fabric and thus inhibit the process of discoloration of the dye present on it. This is due to the keratolytic properties of biocidal products. After washing, the migration of paint is not established. This is due to the keratolytic action of biocidal products, which are water-insoluble because the protective layers are lipophilic. In general, it can be argued that all biocidal products have a high enough resistance to wet treatments. After all, during washing in experimental variants of fabrics, the antimicrobial activity of ethyl biocide is lost by a maximum of 14% and methyl 15%, due to keratolytic properties and insolubility of these biocidal products. In order to determine the wear resistance of textile materials and products during operation, we conducted real tests in production conditions (Table 3).

Table 3 Antimicrobial activity of textile materials treated with biocidal products ETS and MTS in real operating conditions

Variants	Before 1 wash			After 1 year, after 24 washes		
	Aspergillus niger	***Candida tenuis***	***Mycobacterium luteum***	***Aspergillus niger***	***Candida tenuis***	***Mycobacterium luteum***
Without processing	0	0	0	0	0	0
ETS	40.0	69.0	49.0	8.6	9.4	7.3
MTS	44.0	56.0	67.5	5.3	5.7	6.0

These indicators once again confirm the feasibility of using these biocidal products and the effectiveness of impregnation of textile materials for the producing of overalls, because during operation in conditions of high humidity antimicrobial activity persists after repeated washes during the year. Due to the keratolytic action of biocidal products do not wash off quickly. Moreover, we aim for promising research on the creation of synthetic detergents with biocidal substances, taking into account the water-insoluble properties of biocidal products

4 CONCLUSION

1. Possibility and expediency of use in domestic textile production of thiosulfonate preparations ETS, MTS and ATS for antimicrobial treatment of cotton-polyester fabrics for their effective protection against destruction by cellulose-destroying and pathogenic microorganisms is studied. These biocidal products can be considered non-toxic and environmentally friendly.
2. The developed resource-saving method of providing antimicrobial properties to textile materials and products has shown an effective long-term effect in repeated washing, where the antimicrobial properties of cellulose-containing fabrics remain high enough after wet treatments.
3. The stability of adsorption of biocides on the surface of textile materials provides a long delay in the growth of microorganisms after 10 washes and is lost by 14% for ETS and 15% for MTS. Tests in real operating conditions have shown high activity of biocidal products after repeated washing (24 times a year) and long-term action and durability of textile products with antimicrobial properties.

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