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STUDY OF THE OBTAINING OF SILVER NANOPARTICLES ON THE POLYMER SURFACE USING PHOTOCHEMICAL ACTIVATION

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Abstract. The article is devoted to the study of the actual problem of obtaining nanoparticles of antimicrobial drugs and materials. The paper discusses the future of the method of synthesis of silver nanoparticles of various sizes, shapes and compositions. Nanobacteric properties and experimental application of nanotubes in polymer production are shown. It is known that the antibacterial properties of silver nanoparticles depend on its competent structure and morphology. Silver nanoparticles have been thoroughly investigated due to their unique characteristics, including optical, protective, antibacterial and electrical properties. In the era of the antibiotic crisis, with the growth of antimicrobial resistance and a reduction in the number of newly developed drugs, silver nanoparticles are potential candidates due to their significant antimicrobial activity, limited development of resistance and extensive synergistic effect in combination with other drugs. The effect of silver nanoparticles depends on the delivery system, the combination of compounds and their own properties, such as shape and size, which are largely influenced by the synthesis process. Recovery using chemicals or light, irradiation with gamma rays, laser, electron beams or microwave, as well as biological synthesis or a combination of these methods are notable examples of methods for the synthesis of silver nanoparticles. In this paper, updated methods for the synthesis of silver nanoparticles are considered, as well as their advantages and disadvantages. The factors influencing the synthesis process are discussed further.

Keywords: nanomaterials, photochemical activation, silver nanoparticles, protective properties, polymer materials.

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ФОТОХИМИЯЛЫҚ АКТИВТЕНДІРУ АРҚЫЛЫ ПОЛИМЕР БЕТІНЕН КҮМІС НАНОБӨЛШЕКТЕРДІ АЛУДЫ ЗЕРТТЕУ

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Аннотация. Мақала микробқа қарсы препараттар мен материалдардың нанобөлшектерін алудың өзекті мәселесін зерттеуге арналған. Мақалада әртүрлі өлшемдегі, пішіндегі және құрамдағы күміс нанобөлшектерін синтездеу әдісінің болашағы талқыланады. Полимер өндірісінде нанотүтікшелердің нанобактериялық қасиеттері мен тәжірибелік қолданылуы көрсетілген. Күміс нанобөлшектерінің бактерияға қарсы қасиеттері оның сауатты құрылымы мен морфологиясына байланысты екені белгілі. Күміс нанобөлшектері оптикалық, қорғаныш, бактерияға қарсы және электрлік қасиеттерін қоса алғанда, бірегей сипаттамаларына байланысты мұқият зерттелді. Антибиотикалық дағдарыс дәуірінде микробқа қарсы төзімділіктің жоғарылауымен және жаңадан жасалған препараттар санының азаюымен күміс нанобөлшектері микробқа қарсы белсенділігінің, төзімділіктің шектеулі дамуының және басқа препараттармен бірге кең синергетикалық әсерінің арқасында әлеуетті үміткерлер болып табылады. Күміс нанобөлшектерінің әсері жеткізу жүйесіне, қосылыстардың комбинациясына және синтез процесі айтарлықтай әсер ететін пішіні мен өлшемі сияқты өзіндік қасиеттеріне байланысты. Химиялық заттарды немесе жарықты қолдану арқылы тотықсыздандыру, гамма-сәулелермен, лазермен, электронды сәулелермен немесе микротолқынды пешпен сәулелену, сондай-ақ биологиялық синтез немесе осы әдістердің комбинациясы күміс нанобөлшектерін синтездеу әдістерінің көрнекті мысалдары болып табылады. Бұл жұмыста күміс нанобөлшектерін синтездеудің жаңартылған әдістері, сондай-ақ олардың артықшылықтары мен кемшіліктері қарастырылған. Синтез процесіне әсер ететін факторлар әрі қарай талқыланады.

Түйін сөздер: наноматериалдар, фотохимиялық активация, күміс нанобөлшектері, қорғаныш қасиеттері, полимерлік материалдар.

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ИССЛЕДОВАНИЕ ПОЛУЧЕНИЯ НАНОЧАСТИЦ СЕРЕБРА НА ПОВЕРХНОСТИ ПОЛИМЕРА С ПОМОЩЬЮ ФОТОХИМИЧЕСКОЙ АКТИВАЦИИ

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Аннотация. Статья посвящена исследованию актуальной проблемы получения наночастиц антимикробных препаратов и материалов. В статье рассматривается и дискутируется будущее метода синтеза наночастиц серебра различных размеров, форм и составов. Показаны нанобактериальные свойства и экспериментальное применение нанотрубок в производстве полимеров. Известно, что антибактериальные свойства наночастиц серебра зависят от их правильной структуры и морфологии. Наночастицы серебра были тщательно исследованы благодаря их уникальным характеристикам, включая оптические, защитные, антибактериальные и электрические свойства. В эпоху антибиотического кризиса, с ростом устойчивости к противомикробным препаратам и сокращением числа новых разрабатываемых лекарственных средств, наночастицы серебра являются потенциальными кандидатами благодаря их значительной антимикробной активности, ограниченному развитию устойчивости и обширному синергетическому эффекту в сочетании с другими лекарственными препаратами.

Действие наночастиц серебра зависит от системы доставки, комбинации соединений и их собственных свойств, таких как форма и размер, на которые в значительной степени влияет процесс синтеза. Восстановление с использованием химических веществ или света, облучение гамма-лучами, лазером, электронными пучками или микроволновой печью, а также биологический синтез или комбинация этих методов являются примечательными примерами методов синтеза наночастиц серебра. В данной работе рассматриваются обновленные методы синтеза наночастиц серебра, а также их преимущества и недостатки. Факторы, влияющие на процесс синтеза, обсуждаются далее.

Ключевые слова: наноматериалы, фотохимическая активация, наночастицы серебра, защитные свойства, полимерные материалы.

Introduction. In recent years, nanoscience and the production of nanoscale materials and products has been one of the main directions of the development of modern science and technology. This direction in the field of materials science and technology is actively developing, capturing more and more new areas of science and industrial production (Pul, 2005). Modern advances in nanotechnology open up new opportunities for the development of fundamentally new technological processes for the production of nanoscale antimicrobials and materials. An important feature of metallic nanomaterials, which plays a key role in their use in medicine, cosmetics, food and light industry, is their low toxicity.

The increased interest in nanotechnology among researchers in recent years is not surprising, since this field has advanced to an unprecedented level. In general, nanotechnology is a multifaceted field that can be applied to electronics (Natsuki, 2015) sensors, optics (Doan, et al., 2019), mechanics, catalysis, chemistry, cosmetics, pharmaceuticals, medicines and biomedical sciences, food technology and the environment (Doan, et al., 2023). Differences in chemical composition, morphology, size, or controlled dispersion contribute to changes in the characteristics of nanoparticles (NP). This change may be the result of the synthesis process, which is also influenced by many factors. Today, the production of nanoparticles requires that the resulting particles are nanoscale, and their synthesis is simple, inexpensive (Lu, et al., 2019), environmentally friendly (Rajapaksha, et al., 2023) and adapted to specific applications (Vu, et al., 2021).

The bactericidal properties of metallic silver and its compounds have been known since time immemorial. In small concentrations, it is safe for human cells, but harmful to most bacteria and viruses, therefore it has become widely used for disinfection of water and food in everyday life and in the fight against infections in the treatment of people. To date, the unique antimicrobial (Erlykh, 2008) and antiviral properties of silver (Krutikov, et al., 2008) compounds have been comprehensively studied (Kiseleva, et al., 2011). The bactericidal properties of metallic silver are associated with its slow oxidation and release of Ag^+ ions into the environment, therefore, the use of nanosilver preparations as a special class of biocidal agents seems promising. Nanoparticles have high antibacterial efficiency due to their developed surface, which ensures maximum contact with the environment.

The number of publications, growing every year, devoted to the study of antiviral and antibacterial activity of silver nanoparticles, proves the presence of increased researchers are interested in this problem from both fundamental and applied points of view. There is no longer any doubt that silver NPS have high activity against all biological objects, starting from viral particles and ending with the human body. At the same time, there are practically no works aimed at quantifying the biological effect of silver NPS on humans and animals. Unfortunately, it is also indisputable that under certain conditions many LPS (including silver, although perhaps to a lesser extent) are able to have an extremely negative impact on living systems and even cause severe and irreversible changes in the body. In recent years, a new field of medicine has begun to emerge – nanotoxicology, whose efforts are aimed at developing integrated approaches to the study of the biological and toxicological activity of NPS, depending on their composition, size, shape and functional cover of the surface.

The literature presents a number of methods for obtaining metal nanoparticles in non-metallic materials, which can be used to obtain silver nanoparticles in polymeric materials. The methods known from the literature for obtaining metal nanoparticles in polymeric materials are conventionally divided into physical and chemical.

Among the physical methods for obtaining silver nanoparticles, the method of magnetron sputtering metal particles (including silver) in a vacuum chamber and then applying them to the surface of the material is known (Patent RF, 2003). This method is based on the use of an abnormal combustion discharge in an inert gas, in which the positively charged ions formed in the discharge bombard the surface of the cathode in the erosion zone, releasing metal particles from it, which then deposit in the form of a thin layer on the surface of the material to be processed. Here, the high kinetic energy of the particles emerging from the cathode surface provides a good level of adhesion of the obtained nanoparticles to the base.

The physical group also includes the method of obtaining medical dressings, that is, a method in which silver nanoparticles are physically dispersed between fabric fibers as a result of a chemical reaction. To obtain antibacterial fibrous material, silver reduction from an aqueous solution of silver nitrate is used. Then, first (Lansdown, 2002) reducing or reducing agents are attached to the fibrous material, and then silver is applied to their surface (Vishnyakov, et al., 2008). The process consists of processing fibrous materials to carry out the process.

Chemical methods are also common in the production of silver nanoparticles in polymers. Here, chemical reducing agents (most often dimethylborane (Lansdown, 2002), sulfuric acid hydrazine (Heggers, 2005), sodium hypophosphite, salts of glucose, citric or tartaric acid are used to produce silver nanoparticles. Chemical methods include photochemical processes that occur under the influence of electromagnetic waves of light. Light radiation is the largest source of renewable and clean energy, it is non-toxic and does not pollute the environment, and does not leave residues in chemical processes. Both sunlight and artificial light can be used in photochemical processes. Silver, which has a relatively high reduction potential (+0.799 V), can be reduced by photons of light. This can be used to produce silver nanoparticles.

In the article, it is proposed to use physico-chemical processes occurring in thin layers of electrolyte solutions under the influence of sunlight to obtain surface particles of silver. For the photochemical recovery of silver, it is necessary to create a photosensitive layer of semiconductor silver halide on the surface of the polymer. Using a suspension of silver halides for this purpose or directly obtaining them as a result of exchange reactions on the surface of the polymer ensures proper adhesion of particles to the surface of the polymer.

Materials and basic methods. Plates made of low-pressure polyethylene (grade PND 273-83 «PolymerInvest» Russia) and polypropylene grade RR H030 were used for the study. Light-colored polymer samples were selected to provide visual monitoring of the course of chemical and photochemical processes.

Preliminary preparation of the plates: The surface was treated with P-2000 sandpaper, roughened in a solution of $K_2Cr_2O_7$ – 6,5%, H_2SO_4 -93.5% for 5-10 minutes at room temperature, and degreased with a solution of Na_3PO_4 - 20g/l, Na_2CO_3 - 20g/l.

Photochemical studies were carried out in a scientific laboratory room. Solar radiation were used as a source of electromagnetic waves. The radiation flux density was determined by the SM 206-SOLAR solar radiation meter and was 800-1100 W/m^2. In order to form a sorption layer on the polymer surface, the polymer sample was moistened by immersing it in appropriate salt solutions for several minutes. In this case, the following water-soluble salts were used for photochemical reactions: $CuCl_2$, $AgNO_3$ (chemical purity 99.9% SIBPROEKT Russia).

The structure and composition of the films were studied using an ISM-6490-LV scanning electron microscope.

The phase composition of conductive films formed during photochemical processes was determined using an EDX-7000 instrument, Shimadzu Corporation, Japan.

Results and Discussion. As a result of preliminary preparation (mechanical and etching), a rough layer is formed on the surface of polymers containing hydroxyl and carbonyl groups belonging to aldehydes, ketones and acids, as well as double bonds.

For activation, the samples are immersed in 100 g/l of $CuCl_2$ solution. The obtained samples were kept for 40 minutes at a solar radiation intensity of 1200–1400 W/m² in an area with unobstructed access to sunlight. Figure 1 shows the dynamics of the effect of sunlight flux on the reduction of copper chloride. From the dynamics of change, there is an increase in the reduction process from 10 to 40 minutes or reaches a climax. Then the density of the sunlight flux required for the reduction of copper chloride on the polymer surface is 1200–1400 W/m².

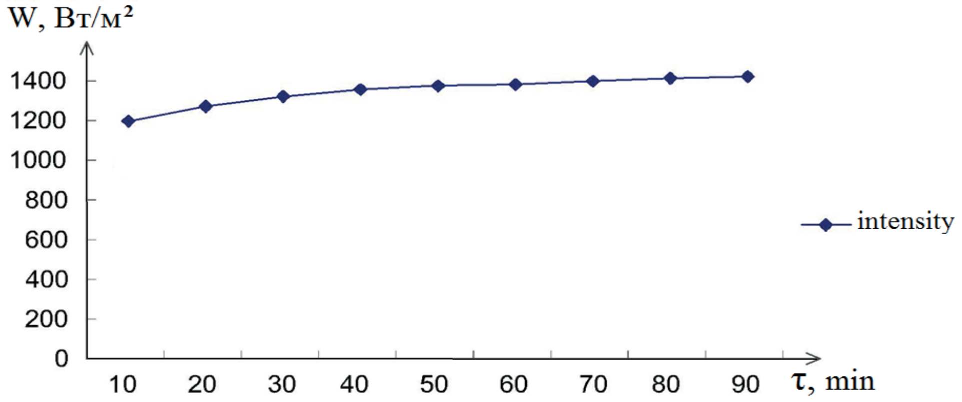


Figure 1 - Dynamics of changes in the flux of sunlight during the reduction of copper chloride.

The samples, as blackened as possible, are washed in distilled water and dried. A monovalent copper chloride with semiconductor properties is formed on the surface of the sample. After the formation of the semiconductor layer, under the influence of photons of sunlight, the photons release additional energy, and the excited electrons acquire the necessary oxidizing ability to restore monovalent copper. When copper dichloride is introduced into the sorption layer, its interaction with the aldehyde group of the polymer R_nCHO becomes possible by reaction

$$R_nCHO + 2CuCl_2 + H_2O = R_nCOOH + 2CuCl + 2HCl \qquad (1)$$

At the same time, a black film was formed on the surface of the sample, characteristic of finely dispersed metal particles obtained by chemical reduction. After washing the sample, active centers consisting of copper monochloride remain on the surface. This indicates that the transformation of $CuCl_2 \rightarrow CuCl$ occurs as a result of these processes. Since the aldehyde group of the polymer R_nCHO is a solid-phase associated part of the polymer, the resulting CuCl forms a strong bond with the polymer surface. Monovalent copper chloride has semiconductor properties and promotes further photochemical reduction of silver.

$$CuCl + e = Cu + Cl^- \qquad E = 0,137 \qquad (2)$$

Upon activation of the surfaces of polymer materials, the reducing ability of which is absent (polyethylene) or very low, the samples after wetting in $CuCl_2$ solution and drying in the sun were additionally treated with a 40% solution of ascorbic acid.

As a result of all these processes, the surface layer of the polymer will contain copper monochloride well bonded to the base. Subsequent impregnation of polymers with a solution of $AgNO_3$ 20 g/L leads to an exchange reaction involving copper chloride and the formation of a photosensitive layer of silver chloride, on which a reduction reaction takes place under the influence of a photon of sunlight.

$$2AgNO_3 + Cu = 2Ag + Cu(NO_3)_2 \quad (3)$$
$$2AgNO_3 + CuCl_2 = 2AgCl + Cu(NO_3)_2 \quad (4)$$
$$AgCl \xrightarrow{\ominus} Ag + Cl^- \quad (5)$$

and under the action of the vacancy, the oxidation reaction

$$CuCl + Cl^- \xrightarrow{\ominus} CuCl_2 \quad (6)$$

hence the total reaction will have the form

$$AgCl + CuCl \rightarrow Ag + CuCl_2 \quad (7)$$

$$2AgCl + C_6H_8O_6 \xrightarrow{h\nu} 2Ag + C_6H_6O_6 + 2HCl \quad (8)$$

After washing, almost only a layer of elemental silver particles remains on the polymer surface, which will be a catalyst for the subsequent chemical deposition of metals.

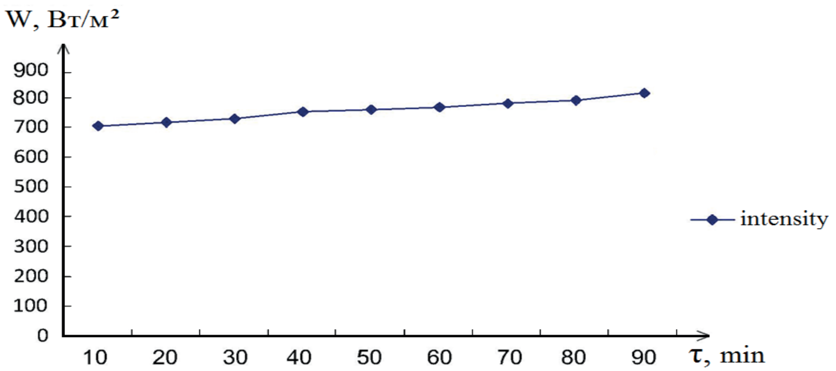
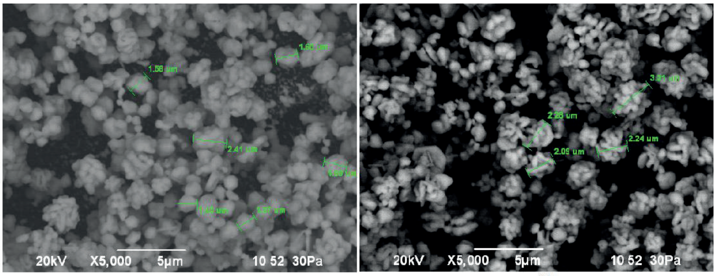


Figure 2 - Dynamics of changes in the flux of sunlight during the reduction of silver nitrate.

In Figure 2, a curve was constructed describing the effect of sunlight on the photochemical reduction of the polymer surface with silver nitrate. The dynamics of the change shows an increase in the reduction process from 10 to 40 minutes or a peak. Accordingly, the solar radiation flux density required for the reduction of silver nitrate on the polymer surface is 700–800 W/m². Silver nanoparticles formed on the polymer surface can be seen in SEM images (Fig. 3).



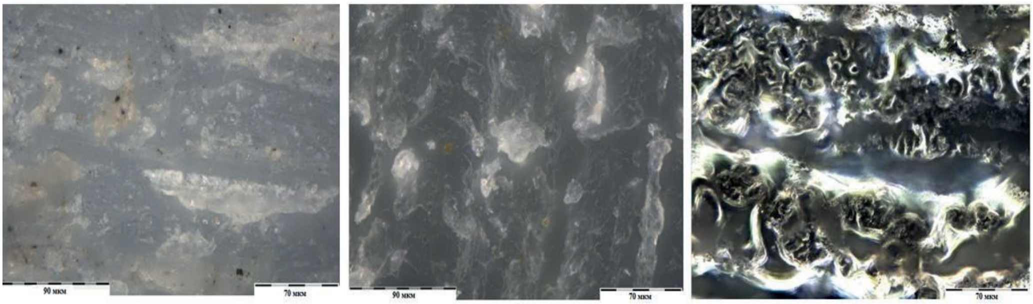
a b

Figure 3 - SEM image of a silver film on polymer materials: a – polyethylene, b – polypropylene

To study the physico-chemical processes, polymer samples were moistened with a solution of $CuCl_2$ - 100 g/L. The amount of solution retained by a 2 ×5 cm sample was 0.06 ml. The copper content in this layer of the solution is about 25 × 10^{-3}g. The experiments were carried out at a solar radiation flux density of 1200-1400 W/m^2 and an air temperature of 25°C.

The last impregnation of the polymer with a solution of silver nitrate leads to the course of the exchange reaction involving chloride of copper and the formation of a photosensitive layer chloride of silver on which, under the action of a solar photon.

In this case, the silver particles are evenly distributed over the surface of the polymer. Some inhomogeneity in particle size seems to be due to the difference in the energy characteristics of the polymer surface. The film has an adhesion to the surface of the polymer and is well resistant to numerous creases and washings. So, after washing 5 times in a solution containing a synthetic detergent powder, the silver content did not decrease.



a b c

Figure 4 - Processes in the formation of silver-containing films on the surface of polymers: a –surface of the plate before activation; b - the surface of the plate after activation; c - the surface of the plate containing silver, which has electronic conductivity

Experiments made it possible to establish that silver films with electrical conductivity are formed only when the content of silver nitrate in the sorption layer on the polymer surface is 20 g/L and exposed to sunlight. By increasing the share of the second processes, it is possible to achieve the production of films with sufficient electrical conductivity.

The resistance of the surface film at a distance of 1 cm was 0,07 × $10^{-2}\Omega\cdot cm^2$ (Abzhalov, et al., 2022).

The formation of a thin silver film is also confirmed by the data of X-ray phase analysis of the sample surface shown in Figure 5, where, in addition to the peaks of the elements that make up the initial polymer, peaks of metallic silver appear.

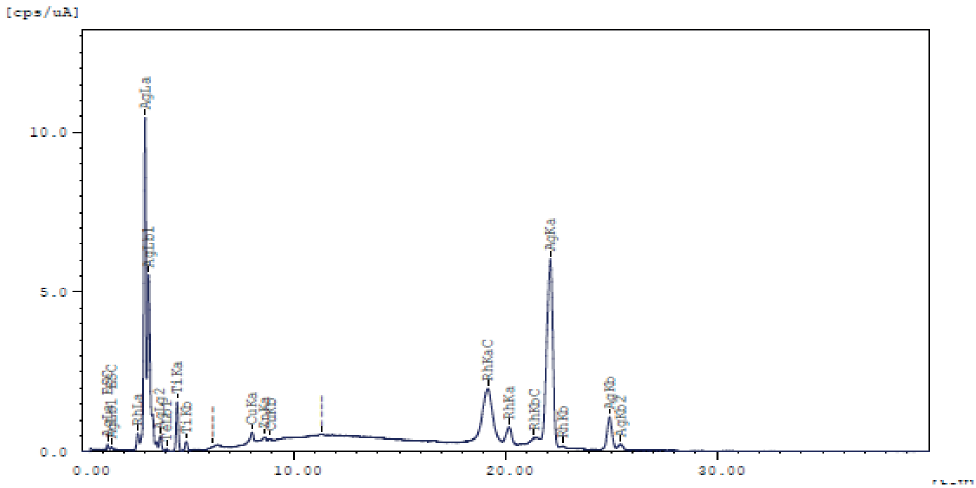


Figure 5 X-ray phase analysis of the sample surface.

The methods of mathematical processing are considered in order to determine the reliability of the results of experimental work carried out in laboratory conditions. The equation for recording the degree of blackening of samples based on a linear mathematical function is shown:

$$z=17,7314+0,4802x+0,2011y \quad (9)$$

Table 1 - dependence of the degree of blackening of a sample treated with copper (II) chloride on changes in temperature and time

Temperature, ^{0}C	Time, min	Degree of blackening, %	Temperature, ^{0}C	Time, min	Degree of blackening, %
25	40	54,3	35	40	61,5
25	50	58,1	35	50	62,3
25	60	61,0	35	60	65,2
30	40	60,1	40	40	64,4
30	50	59,2	40	50	66,5
30	60	63,1	40	60	68,3

The data in Table 1 show that as the temperature increases and the time in the process increases, the degree of blackening of samples treated with copper (II) chloride increases. A mathematically processed version of the degree of blackening of samples treated with copper (II) chloride according to these data is shown in Figure 6.

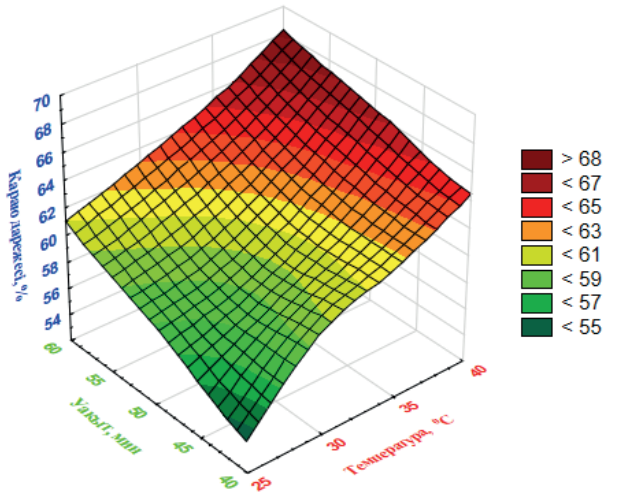


Figure 6 - Dependence of the degree of blackening of the sample treated with copper (II) chloride on temperature and time changes

According to the data in Figure 6, an increase in the degree of blackening under the influence of temperature and time during the blackening process of samples treated with copper (II) chloride is characterized by a change in the quadratic appearance of the plane from green to saturated red.

Conclusion. Studies have shown that this is the result of photochemical and chemically active processes occurring in the sorption layer on the surface of the polymer under the influence of sunlight, copper and silver films were obtained. So, as a result of the interaction of the polymer with copper (II) chloride, monovalent copper chloride is initially formed. This copper compound is a semiconductor and subsequently undergoes a photochemical reaction with ascorbic acid under the influence of photons of sunlight, which further acts as a catalyst for chemical silvering. That is, the use of thin sorption films of a silver nitrate solution as a reaction medium allows the process to be carried out under the influence of electromagnetic waves of light rays. This, firstly, facilitates the formation of crystallization centers due to the reduction of silver ions by photons. Secondly, such radiation activates ascorbic acid molecules, which facilitates the reduction of silver ions in the space between the crystallization centers. In this case, the necessary conditions are created for obtaining silver nanoparticles. Such modified polymeric materials can be used to impart antibacterial and antiviral properties to household and medical materials, as well as to protect against electromagnetic radiation.

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