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# ABSTRACT

of the dissertation for the degree of Doctor of Phylosophy

# OBTAINING AND STUDYING NANOCOMPOSITES ON THE BASIS OF POLYMER MATRIXES

Speciality: 2304.01 – Macromolecular Chemistry

Field of science: Chemistry

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The dissertation work has been performed in the laboratory of "Nanostructured metal-polymer catalysts" of Institute of Catalysis and Inorganic Chemistry named after acad. M.Nagiyev of Azerbaijan National Academy of Sciences.

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## GENERAL CHARACTERIZATION OF THE WORK

**The relevance of the theme and degree of development.** It is known that synthesis of metalnanoparticles in the presence of matrixes, their stabilization and investigation of their properties is promising area in chemistry, as well as in medicine and physics today. It is obvious that the nano-scale state of the metal differently from its molecular structure due to its unique properties provides new opportunities in various application fields of the industry. From the point of nature of nanoparticles, Au, Ag, Pt, Pd among metal atoms, nano metal oxides, such as magnetite $Fe_3O_4$, CoO, NiO, CuO, $Cu_2O$, ZnO and etc. among metal oxides are especially different from others.

The main problem after synthesis of both metal and their nano metal oxides is their instability in sizes for a long time. Thus, this provides stability of their unique and specific superior properties. The studies on this direction show that polymer macromolecules not only stabilize this type nanodispers systems, but also provide the formation (and synthesis) of nanoparticles. Furthermore, the polymer structures are irreplaceable matrixes in arrangement of form and dimensions of the increasing particles. Polyelectrolytes, especially –COOH, $-NH_2$, -OH, and –CHO functional groups-containing natural and synthetic polymers take an important place in a set of these polymers. So that, such functional groups provide stability of initial size of metal nanoparticles surrounding them and preventing their aggregation.

Recently, the charging of drug preparations on combinations obtaining with nanogel and their metallic nanoparticles caused to come to light a new area of biotechnology to get more synergetic effect. In this case, gels observed in the form of supramolecule and the presence of metal nanoparticles cause the formation of the perfect and complete, antibacterial, biological superiorities. In particular, the coordination compound of nanogels is formed with antibacterial $Ag^o$ nanoparticles and magnetic nano-$Fe_3O_4$, after which the drug is immobilized. The resulting nanogel-nanometal-

drug triple combination has many positive effects in comparison to its predecessors in terms of influencing factor, long-term controlled release, and antibacterial properties.

In addition, the application of polymer composites containing various metal nanoparticles and nano-metal oxides in various fields of electronics and physics has led to the almost complete molecular distortion of these fields. Thus, metal nanoparticle-dispersed polymer systems are widely used in nonlinear optical materials, low-temperature solar collectors, data storage materials, nanophotonics, etc. The uniqueness of $Ag^o$ nanoparticles and magnetic liquids also increases their usability.

Considering the above-mentioned issues, the dissertation is relevant in terms of researching new types of nanomaterials based on poly-N-vinylpyrrolidone (PVPr), polyethylene glycol (PEG), polyacryl acid (PAAc), polyacrylamide (PAA) as well as $Ag^o$ and $Fe_3O_4$ nanoparticle-containing colloidal systems in the presence of natural polysaccharides such as chitosan (Cht), gum arabic (GA), arabinogalactan (AG). It is also of great interest to study the temperature and voltage dependence of certain physical properties, including volt-ampere characteristics, electrical resistance of nanoscale $Ag^o$ and $Fe_3O_4$ materials obtained in the presence of these natural and synthetic polymers. In addition, the immobilization of doxorubicin and trypsin as a model drug to natural polymers containing these $Fe_3O_4$ nanoparticles, as well as their PVPr grafted copolymers is also actual issue in terms of their use as a carrier matrix. Furthermore, the impregnation of the antibacterial drug levofloxacin on the complexes of natural polymers and complexes with $Ag^o$ nanoparticles of their composites with PVPr, and the testing antimicrobial activity of these nanobiocomposites, the bacteriological activities in the presence of *Staphylococcus aureus*, *Esherichia coli* and *Pseudomonas aeruginos* bacteria are particularly important.

Work has been accomplished according to the research work plan (State registration № **0115Az2096**) of Institute of Catalysis and Inorganic Chemistry named after acad. M.F.Nagiyev of ANAS.

**The aim and tasks of the research**. Synthesis of pH-

sensitive "smart" nanomaterials using additional reductants in the synthetic and natural polymer medium and including polymer materials to the system and the immobilization of doxorubicin and trypsin to these nanocomposites, the determination of their biological properties and electrical conductivity are the main goals of the study. The following issues have been solved in order to achieve this goal:

- Synthesis of the $Ag^{o}$ nanoparticles from poly-N-vinylpyrrolidone, polyethylene glycol, chitosan, arabinogalactan and gum arabic using both stabilizers and various reductants under special conditions and studies their structures;
- Synthesis of $Fe_3O_4$ nanoparticles in poly-N-vinylpyrrolidone, polyethylene glycol, gum arabic and chitosan medium and identification of the structure of the obtained gel-nanoparticle complexes by spectroscopic methods;
- Study of the swelling properties of synthesized natural and synthetic-based magnetite $Fe_3O_4$ nanoparticle gel complexes and immobilization of biologically active compounds, such as trypsin and doxorubicin in them;
- Study of volt-ampere properties, voltage and temperature dependence of resistance of composites with magnetite $Fe_3O_4$ and $Ag^{o}$ nanoparticle obtained in the presence of natural and synthetic polymers;
- Study of antibacterial properties of natural polymers containing silver nanoparticles and the homogeneous mixtures of their complexes with poly-N-vinylpyrrolidone and levofloxacin in some microbial media on special meshes.

**The methods of the research.** Modern methods of physico-chemical analysis (UV, FTIR, SEM, XRD, TQA, DTA, etc.) were used in the research to identify and characterize the initial and synthesized products.

**The main statements of the dissertation.** The main results of the dissertation research, the main provisions presented for defence as a result of comparing them with existing theories and investigations so far are as follows:

- Synthesis of $Ag^{o}$ and $Fe_3O_4$ nanoparticles in poly-N-

vinylpyrrolidone and polyethylene glycol media and their characterizing by physical research methods;

- Synthesis, stability, structural analysis and research of $Ag^o$ and $Fe_3O_4$ nanoparticles stabilizing in chitosan, gum arabic and arabinogalactan medium;

- The nature of the chemical interaction of $Ag^o$, $Fe_3O_4$ nanoparticle containing-biocomplexes on the basis of above-mentioned polymer composites with trypsin and doxorubicin;

- Study of biological activity, antibacteriality of synthesized $Ag^o$ and $Fe_3O_4$ nanoparticle gel materials and lysis zones of levofloxacin impregnated-surgical meshes in special microbial medium;

- Investigation of various parametric functions of physical properties, such as volt-amperes and electrical resistances of synthesized $Ag^o$ nanoparticle natural and synthetic polymer nanocomposites.

**Scientific innovation of the study:**

"Smart" hydrogels based on poly-N-vinylpyrrolidone, polyethylene glycol, chitosan, as well as gum arabic and arabinogalactan have been synthesized, and using them as stabilizers and reducing agents, $Ag^o$ and $Fe_3O_4$ nanoparticle containing-nanocomposites have been obtained; their physical parameters have been studied. The immobilization of trypsin and doxorubicin to the obtained metal nanoparticle biocomplexes has been also investigated. Finally, nano $Ag^o$ gel-levofloxacin solution has been impregnated on the surfaces by slight heating to give additional antibacterial properties to the meshes used in abdominal hernias and biotested *in vitro*.

**The theoretical and practical significance of the research.** $Ag^o$ and $Fe_3O_4$ nanoparticle gel complexes have been synthesized in the medium of poly-N-vinylpyrrolidone, polyethylene glycol, chitosan, gum arabic, arabinogalactan based-"smart" polymer matrixes and immobilization method of trypsin, doxorubicin and levofloxacin, which are biologically active in given value of pH has been developed. Such nanocomposites, which provide long-term controlled release from the gel under the influence of irritants of the

medium and have additional biological properties due to the basic matrix and metal nanoparticles, can be used as an effective matrix for the transport of drugs in medicine. Also, complexes of natural and synthetic polymer-based materials with $Ag^o$ and $Fe_3O_4$ nanoparticles are important not only in solving problems such as deficiency of these trace elements in the body, but also as a carrier in the targeted delivery of antibiotics and some enzymes in different medium.

In addition, the values of the physical properties, such as volt-amperes and specific resistance of $Ag^o$ and $Fe_3O_4$ nanoparticle-containing polymer composites allows them to be used in electronics as a base material in the manufacture of wires with specific conductivity and components of optical devices.

**Approbation and application**. The results of the dissertation have been presented at the following national and international scientific conferences: Proceedings of the scientific conference dedicated to the $105^{th}$ anniversary of Academician M.F Nagiyev, (Baku 2013), "All-Russian conference with international participation of young researchers in chemistry", Mendeleev-2014, Saint-Petersburg 2014), Proceedings of II International Scientific Conference of Young Researchers, Dedicated to the 91st anniversary of the H.Aliyev (Baku, 2014), XXVI International Chugayev Conference on Coordination Chemistry (Kazan, 2014).

The topic and practical significance and results of the dissertation work have been awarded the "M. Nagiyev Prize" presented by Institute of Catalysis and Inorganic Chemistry named after acad. M.Nagiyev of Azerbaijan National Academy of Sciences (2015). In addition, in order to summarize certain parts of the research, the research work has been submitted to the Science Development Fuundation under the President of the Republic of Azerbaijan and grant project programs of the Presidium of ANAS within the project program and successfully financed by the following projects:

2014 - "Application of metal nanoparticle size and zeta potential in the medium of polymers in Zetasizer Nano ZS90 device". Science Development Foundation under the President of

the Republic of Azerbaijan, Project Performer.

2018-2019 - "Synthesis and application of polymer-based nanogels in the treatment of cancer" project, ANAS 7 / 3-2018, as Performer;

2018-2019 - "Synthesis and research of hydrophobic and biocidal nitrogen and oxygen containing-polymers for immobilization of medicinal products", Science Development Foundation under the President of the Republic of Azerbaijan, as Performer;

**The title of the research organization where the dissertation work has been performed.** Dissertation work has been accomplished in the laboratory of "Nanostructured metal polymer catalysts" of Institute of Catalysis and Inorganic Chemistry named after acad. M.Nagiyev of ANAS. In addition, the antibacterial properties of the samples, biomedical tests, some structural spectroscopic analysis and physical parameters, such as volt-amperes have been tested at the Azerbaijan Medical University and the Institute of Physics of ANAS.

**Published scientific works on the dissertation**. Materials related to the content of the dissertation have been published in 24 scientific works. 14 of them are thesis of reports, and 10 are scientific articles published in foreign and national scientific journals.

**Volume and structure of the dissertation work**. The dissertation work consists of an introduction (15650 characters), 3 chapters (Chapter I - 48275, Chapter II - 28123, Chapter III - 114944 characters) with additions containing general results and test acts. The volume of the dissertation consists of 169 computer pages. There are 60 figures, 12 tables and a reference list of 176 titles.

## THE MAIN CONTENT OF THE WORK

**The Introduction** of the dissertation substantiates the actuality of the research theme, scientific novelty and practical significance of the obtained results.

**The first chapter** deals with the synthesis of natural and synthetic-based composites, especially synthesis of metal nanoparticle containing-composites based on poly-N-vinylpyrrolidone, polyethylene glycol and polysaccharides – chitosan, gum arabic, arabinogalactan, as well as their role together with metal nanoparticle containing-gels in the transport of drugs; literature review, comparative analysis and systematization of the last 10-15 years on immobilization of biological active compounds to this gel- and metal-gel polymers and their study, application in electronics and optoelectronics.

**The second chapter** is devoted to the techniques of the experiments. This includes research methods, initial compounds, their preparation and determination of physical-chemical, biological properties of the obtained products and application of physicochemical analysis methods, study of physical parameters. Methods for the determination of lysis zones of polymer-levofloxacin nanobiocomposites impregnated with $Ag^o$ nanoparticles are also given in this chapter.

**The third chapter** consists of the synthesis of $Ag^o$ and $Fe_3O_4$ nanoparticle composites based on natural and synthetic polymers, the confirmation of their structure by physical research methods and the results obtained from the study of immobilization of selected drugs to them and their transport. This chapter also explains in detail the study of antibacterial properties of new synthesized gel samples, combinations of gel complexes with $Ag^o$, $Fe_3O_4$ nanoparticles with levofloxacin, the possibility of using them as an effective matrix for the transport of enzymes and antibiotics using trypsin and doxorubicin as a model drug. In addition, some electrical and magnetic properties of $Ag^o$ and $Fe_3O_4$ nanoparticles containing-composites, volt-ampere characteristics have been studied depending on the nature of the polymer and nanoparticles,

and it also includes the results obtained from the study of voltage and temperature dependence of the selected resistance.

Throughout the research, the obtaining and study of $Ag^o$-based nanocomposites stabilized by one of the polysaccharide – GA are given in detail in the dissertation.

The synthesis of $Ag^o$ nanobiocomposite has been carried out in accordance with the known technique. At the same time, the reduction of $Ag^+$ ions in the presence of GA has been studied with 1% $NaBH_4$ and it has been determined that the reduction of $Ag^+$ ions in the GA medium (T = 363 K) is easier than $H_2O_2$ and the yield of the reaction product is 91%. It has been found that every 10 mg of the composite obtained by the precipitation of the reduction product with $NaBH_4$ in acetone contains 2.84 mg of $Ag^o$ (28%).

GA and $Ag^o$-containing nanocomposites has been comparatively studied on the X-ray diffractometer and X-ray analysis of the crushed composite has been performed on the Advance D8 (Bruker) X-ray diffractometer to determine the metallic phase after reduction of $NaBH_4$ and $Ag^+$ ions in the GA medium (Figure 1).

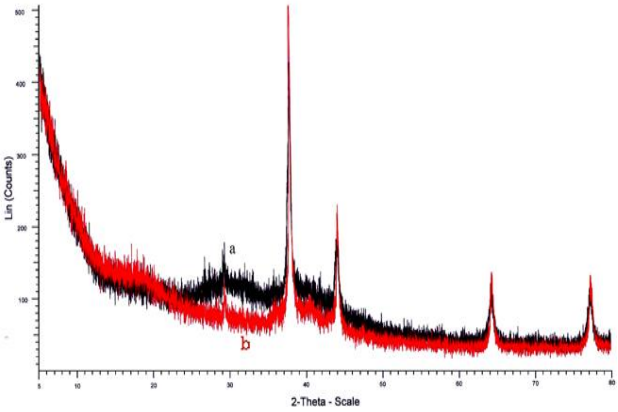



**Figure 1.** Diffractogram of GA-based composite containing $Ag^o$ nanoparticles: a - precipitated in acetone, b - precipitated in ethanol.

It has been found that the size of $Ag^o$ nanoparticles in the composite is 32-46 nm, and the lattice parameter of the separated

atomic phase is $a$=0.4080 nm (the standard lattice parameter for Ag is 0.40862 nm), the atomic radius is 0.14447 nm. It has been also established that the nanoscale $Ag^o$ nanoparticle core is surrounded by GA shell and is spherical in shape.

It has been shown that the reduction of silver ions to atomic silver occurs in the GA medium:

$$AgNO_3 + NH_4OH \rightarrow AgOH\downarrow + NH_4NO_3$$
$$2AgOH \rightarrow Ag_2O + H_2O$$
$$Ag_2O + H_2O_2 \rightarrow 2Ag + H_2O + O_2\uparrow$$

IR spectroscopic study of GA and synthesized nanobiocomposite of absorption bands 3400, 1620, 1460, 1380, 1060 $cm^{-1}$ showed that silver nanoparticles do not form an individual absorption band and the polysaccharide retains its original physical and biological properties. UV spectroscopy of the composite solution containing $Ag^o$ nanoparticles showed that the metallic phase exhibits conductivity due to the excitation of electrons belonging to the $Ag^o$ atoms. Using this property of the system, it is also possible to prepare high-performance nonlinear optical materials from an aqueous solution of metal composite.

$Ag^o$ nanobiocomposite obtained and stabilized in the presence of GA has 2 types of properties in terms of its composition. The polysaccharide part provides its immunomodulatory, gastroprotective properties, and $Ag^o$ nanoparticles provide antimicrobial and bactericidal properties. In this regard, the synthesized nanobiocomposite can be used in medicine for the above purposes.

The biocompatibility, biodegradability, non-toxicity, bioactivity and multifunctionality of Cht have justified its use as a natural biopolymer cationite for many years.

Metal ions form pseudo-complexes coordinating around the $–NH_2$ and –OH groups due to weak interaction forces in the $Ag^+$ ions-distributed Cht solution.

$$Cht + AgNO_3 \rightarrow Cht/Ag^+ NO_3^-$$

With the addition of $NaBH_4$ as a reducing agent, the silver ions are reduced under normal conditions, and this process continues for some time. The reduction equation of $Ag^+$ ions can be expressed as follows:

$$Cht\ /Ag^+\ NO_3^-\ +BH_4^-+3H_2O \rightarrow Cht/Ag^0+3.5H_2+B(OH)_3$$

The reduction process occurs with a gradual change in the color of the solution, which can be clearly seen in the following figure:

**Figure 2**. Color of Cht–Ag$^o$ suspension depending on the reaction time

As is seen from the Figure 2, during the reduction process, the color of the system changes from yellow to dark brown. This indicates the formation of a colloidal solution in which Ag$^o$ nanoparticles are formed. If we look at the spectrum of the Cht/Ag$^o$ colloid, we can observe a peak with a strong plasma resonance centered on the surface at 410 nm. According to the references, a colloidal solution containing 12±2 nm-sized Ag$^o$ nanoparticles forms a plasma resonance peak around 400 nm. If the peak is intense between 405-418 nm, the size of the formed Ag$^o$ nanoparticles varies within 9-30 nm.

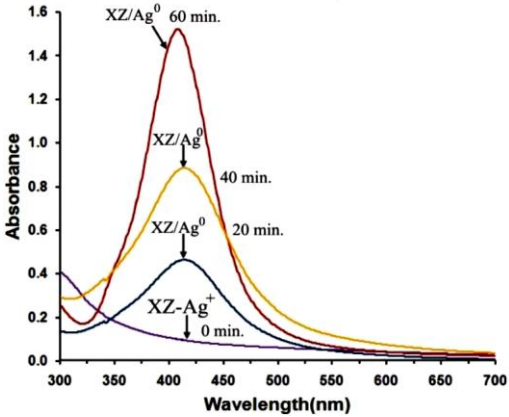



**Figure 3**. Dependence of absorption of Ag$^o$ nanoparticle solution formed in Cht medium on reduction time

It has been determined that if the reduction period lasts more than 1 hour the intensity of the peak does not change. Plasma resonance of Ag$^o$ nanoparticles in the range of 350-450 nm indicates that the silver nanoparticles are distributed in a narrow fraction in suspension. After 353-363 K, the formation of Ag$^o$ nanoparticles is accelerated when the reduction process is continued with a further increase in temperature.

X-ray phase study of the composite containing stabilized Ag$^o$ nanoparticles in Cht polymer medium has been carried out and the obtained roentgenogram has been given as follows:

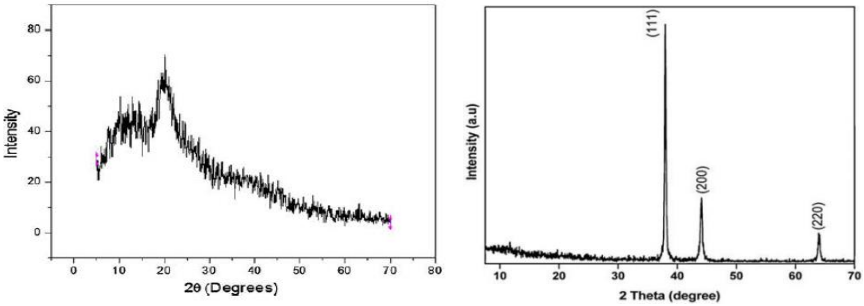



**Figure 4.** XRD spectra of Cht (left) and Cht-Ag$^o$ (right) composite

No characteristic peak of the crystalline phase is observed in the XRD spectrum of Cht. However, in the spectrum of the silver nanoparticles containing-composite, three strong intensities are formed at 2θ, which is proper to Ag$^o$ atoms. These are the criteria of Bragg reflection with fields of (111), (200) and (220) with values of 37.860, 43.680 and 64.120 special to Ag$^o$ atoms in 2θ. According to the results of X-ray phase analysis, Ag$^o$ nanoparticles form a volume-centered cube-shaped crystal structure in the chitosan structure. The size of Ag$^o$ nanoparticles formed in the volume of polysaccharides is determined by the Debye-Scherrer equation:

$$n=K\lambda/\beta cos\theta$$

here, K is the Scherrer constant (0.9 ÷ 1), λ is the wavelength (1.5418 A), β is 1/2 of the peak width, and θ is the Bragg angle. It has been found that the average size of nanoparticles is 14÷25nm, which depends on the reduction time and temperature. SEM micrographs of the chitosan-Ag$^o$ composite have been also recorded during the study. It has been established that Ag$^o$ nanoparticles on the composite surface are mostly structured in the form of oval granules. SEM micro-images of Ag nanoparticles are shown in Figure 5.

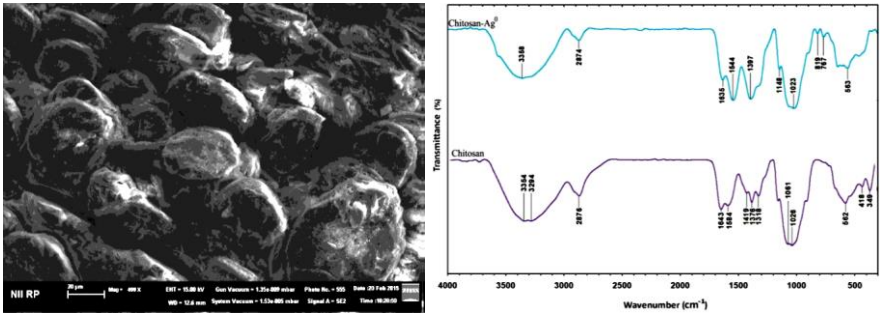



**Figure 5**. Surface micro-image of Cht-silver nanocomposite and IR spectra of pure Cht and Cht-Ag$^o$ composite

It is clearly seen that the size of Ag$^o$ nanoparticles is 12 and 23 nm and these non-spherical particles are located close to each other. During the process, Cht participates in the reduction of silver

ions together with sodium borohydride, and also prevents the aggregation of nanoparticles with each other. SEM micro-images also prove that the reduced silver atoms are nanoscale.

It is likely that the Cht macromolecule or functional groups – $NH_2$ and –OH have surrounded $Ag^o$ nanoparticles. The spectra of Cht, Cht-$Ag^o$ composite have been recorded to confirm the stabilization of nanoparticles in this form (Figure 5).

The spectrum of Cht shows peaks specific to the vibrations of the bonds for the O-H and N-H groups, respectively, in the region of 3354 and 3294 $cm^{-1}$. The adsorption band at 2876 $cm^{-1}$ is unique to the chemical shift of the aliphatic C-H bond, 1643 and 1584 $cm^{-1}$ – the N-H bond, and the intensities of 1419, 1376 and 1318 $cm^{-1}$ are particular to the aliphatic C-H bonds, and the 1061 and 1026 $cm^{-1}$ adsorption band is unique to the C-O adsorption band. In the spectrum with $Ag^o$ nanoparticles, adsorption bands 1643 and 1584 $cm^{-1}$ have been subjected to chemical shift and observed in the region of 1635 and 1544 $cm^{-1}$, respectively. A high-intensity peak is observed at 1544 $cm^{-1}$. The peak that undergoes changes in the region of 1397 $cm^{-1}$ characterizes Cht -$Ag^o$ complex, which indicates the interaction between silver nanoparticles and Cht. The stability or interaction of the system occurs due to the electrostatic forces and Van der Waals effects between functional groups in the Cht macromolecule that have electron density and somewhat positively charged metal nanoparticles (Figure 6).

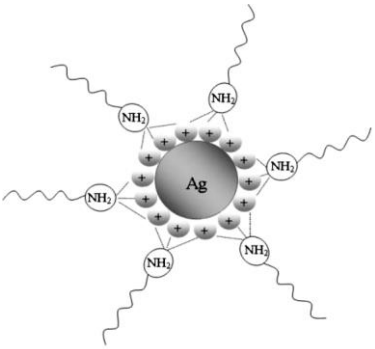



**Figure 6.** Structural description of the silver nanoparticle system stabilized with Cht

The study also determined the size of the reduced silver nanoparticles by replacing the stabilizing matrix with a synthetic polymer. For this purpose, a water-soluble polymer PEG and GA complex have been used.

When PEG and GA macromolecules are dissolved in an aqueous medium, the functional groups undergo conformational convergence with each other forming a homogeneous system. $Ag^+$ ions are surrounded by –OH and –COOH groups and form coordination.

$$PEG/GA + AgNO_3 \rightarrow PEG/GA/Ag^+ NO_3^-$$

When a reducing reagent is added to the system, the silver ions undergo a chemical conversion as follows:

$$PEG/GA/Ag^+ NO_3^- + NaBH_4 + H_2O \rightarrow PEG/GA/Ag^o + H_2 + B_2H_6 + NaNO_3$$
$$PEG/GA/ Ag^+ NO_3^- + HCOOH \rightarrow PEG/GA/Ag^o + CO_2 + H_2O + HNO_3$$

In both cases, depending on the temperature and reaction time, the color of the solution changes first to yellow, then to dark brown and black that indicates the reduction process of silver ions. After processing of the obtained nanocomposite with diethyl ether or ethyl alcohol and then after drying, the UV-Vis spectrum of its deionized-aqueous solution has been recorded at different temperatures (Figure 7).

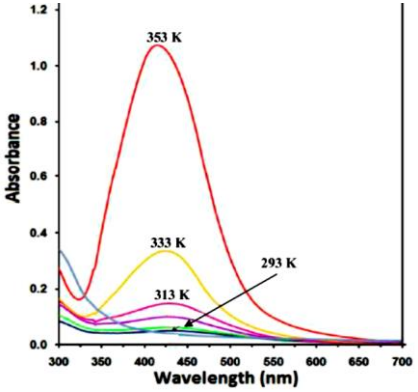



**Figure 7**. UV-Vis molecular electron spectra of the PEG-GA-$Ag^o$ colloidal system at different temperatures

As shown in Figure 7, typical plasma resonance at 410-425 nm is specific to $Ag^o$ nanoparticles. According to the experiments, it is possible to obtain 20 nm particles in 353 K in 2 hours under different reaction conditions. The particles keep their sizes constant for a long time in the above-mentioned PEG-GA system.

It has been found that changing the number of mole of PEG and AG affects the size and stability of the formed nanoparticles. Thus, if we increase the amount of GA in the composition, the size of $Ag^o$ nanoparticles will change in the order of 12-17 nm, and vice versa, if we increase the amount of PEG, the particle size varies around 34 nm. This is due to the share of functional groups that stabilize these nanoparticles in this process, which is plenty in the GA macromolecule. Figure 8 describes the XRD spectra of the system at different temperatures.

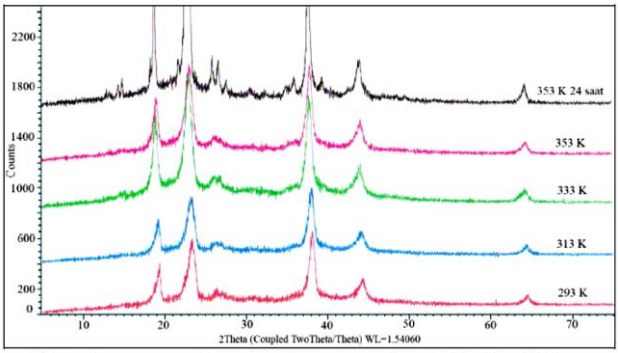



**Figure 8.** XRD spectra of PEG/GA/$Ag^o$ composite at different temperatures

As it is seen from Figure 8, the intensities of the peaks specific to silver atoms increases and the peak area expands with increase in temperature. Crystal fields of (111), (200), (220), (311) specific to the $Ag^o$ nanoparticles have been determined in the 37.910, 43.710, 64.060 degree values of 2θ and in 76.980 of Bragg reflection. These figures show that the obtained silver nanoparticles are in the form of the centralized cube.

IR spectroscopy method has been used to determine the chemical interaction between Ag nanoparticles and PEG/GA

structure (Figure 9). As is seen, intensive adsorption bands are observed in the region of 1053, 1080 and 1656 $cm^{-1}$. The peak of 1656 $cm^{-1}$ is specific to > C = O bond of carboxyl in GA. 1080, 1385 $cm^{-1}$ adsorption bands show wide bands of C-O bonds in the C-O and C-O-C groups in the polysaccharide macromolecule. The specific tensile vibration band of the aliphatic C–H bond is observed in the region of 1436 and 1335 $cm^{-1}$.

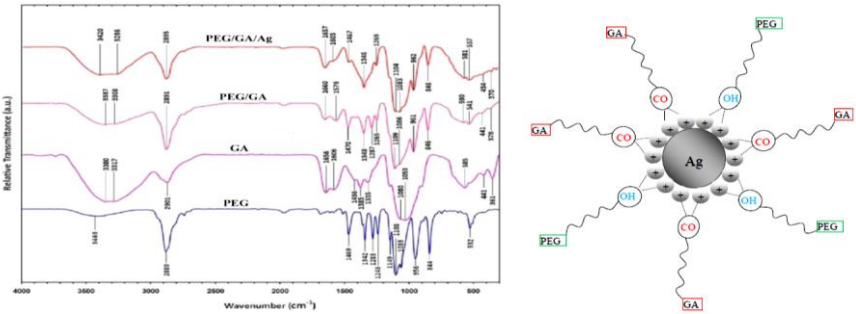



**Figure 9.** IR spectrum of PEG, PEG/GA and PEG/GA/Ag$^o$ composite, and the proposed composite construction of Ag$^o$ nanoparticle system stabilized in PEG/GA medium

Thus, the surface of the nanoparticles is positively charged and the hydroxyl groups in the PEG cover the Ag nanoparticles. This confirms that the stabilization of Ag$^o$ nanoparticles occurs due to Van der Waals forces between positively charged metal atoms and oxygen atoms in the hydroxyl group. Taking into account the above-mentioned, the stabilization of Ag$^o$ nanoparticles with regard to chemical structure can be schematically described.

In the next study, the effect of temperature on the morphology of spherical $Fe_3O_4$ magnetite nanoparticles stabilized by the PEG macromolecule has been studied. The synthesis of $Fe_3O_4$ magnetite nanoparticles functioned with PEG has been carried out by co-precipitation using very small amounts of organic matter to obtain a highly monodisperse form of nanoparticles. A simple following scheme for the magnetite $Fe_3O_4$ nanoparticles obtained after synthesis at different temperatures surrounding by PEG macromolecule is shown (Figure 10).

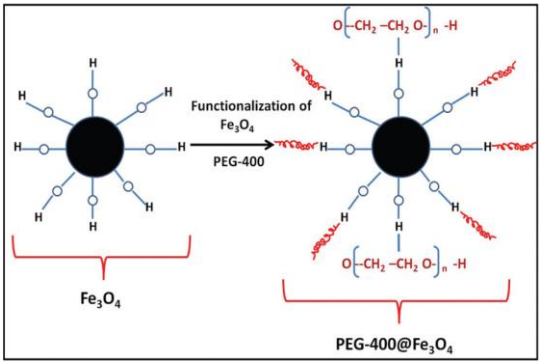



**Figure 10.** Schematic description of magnetite $Fe_3O_4$ nanoparticles surrounded by PEG-400

The synthesis reaction of magnetite $Fe_3O_4$ nanoparticles takes place according to the following chemical reactions:

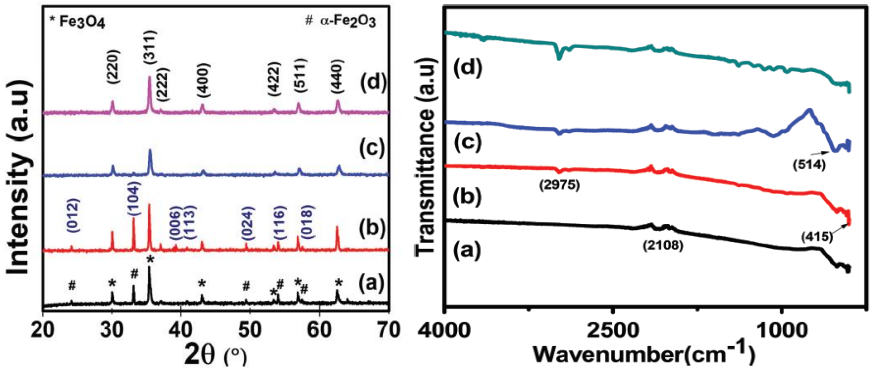
$$Fe^{3+} + 3OH^- \rightarrow Fe(OH)_3$$

$$Fe(OH)_3 \rightarrow FeOOH + H_2O$$

$$Fe^{2+}\ 2OH^- \rightarrow Fe(OH)_2$$

$$2FeOOH + Fe(OH)_2 \rightarrow Fe_3O_4 + 2H_2O$$

Diffraction peaks specific to magnetite oxide are observed in the XRD spectrum of PEG-magnetite $Fe_3O_4$ nanoparticle composites obtained from the synthesis reaction at different temperatures (Figure 11, a).




a) b)

**Figure 11.** XRD and FTIR spectra of PEG-$Fe_3O_4$ nanocomposites at different temperatures

According to the XRD analysis of magnetic nanoparticles, the main peaks 30.1 (220), 35.6 (311), 39.42 (400), 57.32 (511), and 62.74 (440) for nanocomposites obtained at 20ºC belong to $Fe_3O_4$ nanocrystals.

**Table 1.**

Temperature dependence of the size of magnetite $Fe_3O_4$ nanoparticles surrounded by PEG

| Reaction temperature, ºC | Size of crystallyne phase, nm | Average size of particles according to SEM, nm | Parameters of the lattice, (a) Å |
|---|---|---|---|
| 20 | 37.24 | 38.52 | 8.384 |
| 30 | 39.43 | 43.65 | 8.388 |
| 40 | 41.26 | 47.71 | 3.392 |
| 50 | 50.28 | 56.23 | 3.393 |

As seen from the Table 1, the increase in temperature during the synthesis of nanoparticles leads to a slight increase in the size of the magnetite $Fe_3O_4$ nanoparticles. With temperature increasing, relaxation with increasing the flexibility of the macromolecule chain occurs that doesn't lead to ideal surrounding by the polymer. The internal energy of the particles also increases, which leads to their partial aggregation.

FTIR spectra of all magnetite $Fe_3O_4$ nanoparticle samples obtained at different temperatures have been studied (Figure 11, b). The peaks special to the O-H groups observed in the region of 3514-3334 $cm^{-1}$ show that the magnetite $Fe_3O_4$ nanoparticles are surrounded by the polymer macromolecule. The intensive peak in the region of 667-420 $cm^{-1}$ is unique to the Fe-O bond. Most importantly, the peak in the region of 1124-1093 $cm^{-1}$ corresponds to the Fe-O-C bond, which indicates the relationship between PEG and $Fe_3O_4$ nanoparticles.

Typical SEM imagines of composites with $Fe_3O_4$ nanoparticles surrounded by synthesized chitosan and surface morphology of these compositions have been also studied (Figure 12). The size of the Cht surrounded-particles is larger than that of pure oxide that is related to covering of particles with polymer macromolecule.

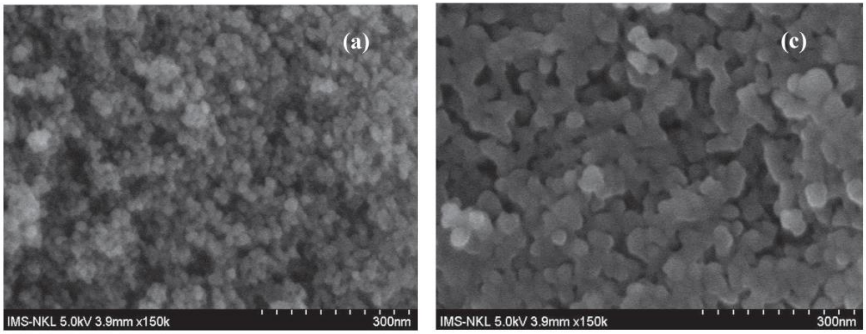



**Figure 12.** SEM micrographs of pure $Fe_3O_4$ and magnetite nanoparticles surrounded by Cht

In next studies, the biological activity of trypsin and doxorubicin antibiotics has been studied immobilizing them to $Fe_3O_4$ nanoparticle systems obtained in the presence of GA, AG fraction precipitating in alcohol, Cht, their graft copolymers with VPr in homogeneous systems.

The size of $Fe_3O_4$ nanoparticles structured in gel samples and degree of swelling of these gel systems in different medium, amount of immobilized trypsin and doxorubicin, degree of immobilization, and other parameters have been studied. Taking into account the release process of immobilizing trypsin and doxorubicin in gel samples, the swelling kinetics of magnetite-gel systems initially synthesized at different values of acidity of the medium have been investigated. The results are shown on the Figure 13:

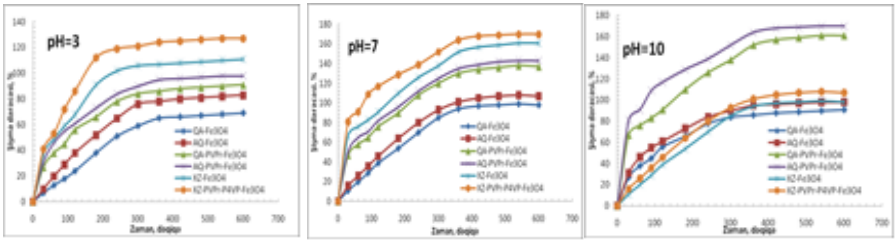



**Figure 13.** Kinetics of swelling prosess of nano $Fe_3O_4$-hydrogel composite at different pH

As it is seen from Figure 13, nano $Fe_3O_4$-hydrogel samples are sensitive to pH and conduct themselves differently depending on the nature of the functional groups in the basic matrix. Initially, it has been found that in all gels, swelling increases sharply in the first 60 minutes, and in the next periods, this increase occurs gradually and slightly until achieving swelling balance. In chitosan-based matrixes, the swelling degree of nanogel composite at pH=7 is higher than in other medium. The protonation of $–NH_2$ groups at pH=3 makes diffusion difficult of water molecules, whereas in the alkaline medium, this is related to the collapsing of the chitosan chain, as well as the repulsion of PVPr and P4VP macromolecules grafted to structure due to inability of the basic functional groups to polarize. The situation is different in other polysaccharide-based gel samples. GA and AG show a relatively high degree of swelling in alkaline medium because of the predominance of acidic functional groups in their structure, in contrast to these $Fe_3O_4$ nanogel composites. This causes rapid diffusion of water molecules to volume as a result of the easy attraction of –COOH groups in their structure with opposite-charged ions in the medium at pH=10. The results give us opportunity to use AG and GA based-$Fe_3O_4$ nanogel composites in long-term transport of drugs with biological activity at pH>7, and to use chitosan-matrix magnetite nanogel in transportation of drugs with biological activity at pH<7.

Immobilization of trypsin to magnetite $Fe_3O_4$ nanoparticle hydrogels has been carried out on the basis of appropriate techniques in the dissertation. The results have been compared with a sample of PVPr without magnetite iron oxide particles. The amount of trypsin immobilized to the composite without PVPr and polyacrylic acid (PAAc)-based magnetite $Fe_3O_4$ nanoparticles is 0.24 and 0.38 mg/g, respectively. In other words, as the chemical functionality of the polymer increases, the amount of immobilized trypsin increases. This is due to the presence of –COOH groups, which can easily chemically interact with the enzyme in PAAc.

Magnetite $Fe_3O_4$ nanoparticles increase the amount, activity and durability of the enzyme immobilized in natural and synthetic based gels and their magnetite $Fe_3O_4$ nanoparticle metal gel

complexes compared to non-magnetite $Fe_3O_4$ nanoparticles gel, which is a manifestation of the enzyme's constant contact with metal ions. On the other hand, metal oxide nanoparticles are in form of a stable complex with the matrix, so that the enzyme is bound to the polymer structure by the metal nanoparticle.

Studies of the release of immobilized trypsin from polymer gel and magnetite $Fe_3O_4$ nanoparticle composites under static condition within pH-3÷11 show that the enzyme is released into the medium within 24 hours at different, but close values of pH, depending on the nature of the polymer and the degree of ionization of its functional groups. Release from magnetite $Fe_3O_4$ nanoparticle composites in the completely different medium is characterized by the high degree of release. Release in gels obtained on the basis of free polymers is observed in neutral or close to neutral medium, whereas in acidic medium, contrarily, release occurs at the higher value in magnetite $Fe_3O_4$ nanoparticle composites.

As a next step in the study, the loading, transport, and release of antibiotics have been investigated using doxorubicin as a model drug for the synthesized magnetite $Fe_3O_4$ nanoparticle gel matrixes (Table 2).

**Table 2.**

Immobilization parameters of doxorubicin to gel and magnetite $Fe_3O_4$ nanoparticle complexes containing natural and synthetic polymers, V=20 ml, T=20 ºC, t=24 hours, $m_{gel}$=50 mg, $C_{0dox}$. = 500 mg/L.

| **Gel carriers** | **Equilibrium concentration, *mg/L*** | **Degree of immobilization *%*** | **Capacity of the gel, *mg/g*** |
|---|---|---|---|
| PVPr 10 kDa-10% MBAA | 452,4 | 9,52 | 19,04 |
| PAT 40 kDA-10% MBAA | 465,8 | 6,84 | 13,68 |
| PVPr 10 kDa-$Fe_3O_4$ | 367,3 | 26,54 | 53.08 |
| PAT 40 kDa-$Fe_3O_4$ | 342,6 | 31.48 | 62.96 |
| Cht - $Fe_3O_4$ | 304,8 | 39.04 | 78.08 |
| AG-PVPr (5%)-$Fe_3O_4$ | 310,7 | 37.86 | 75.72 |
| GA-PVPr (5%)-$Fe_3O_4$ | 309,2 | 38.16 | 76.32 |
| Cht - PVPr-P4VP-$Fe_3O_4$ | 293,4 | 41.32 | 82.64 |

In natural polymers and magnetite $Fe_3O_4$ nanoparticle complexes, the increase in the capacity of the carrier to the antibiotics is 4-5 times higher. In addition, the capacity to doxorubicin is many times higher than that of trypsin compared to the enzyme. This can be explained by the chemical composition of the doxorubicin molecule and the fact that the reactive functional groups are more active in space; the functional groups including in career composition can easily interact with small molecule of doxorubicin. This interaction, which leads to immobilization, has been characterized by physical research methods, and the existence of electrostatic interactions along with hydrogen bonds has been proven.

In this study, the time dependence of release process from gel of doxorubicin immobilized to chitosan-surrounded magnetite $Fe_3O_4$ nanoparticle matrix has been studied at different values of pH. The antibiotic has been released from the polymer at 37ºC. The time dependence of Dox release from chitosan-based nano-iron oxide is shown in Figure 14. As is seen, the amount of antibiotic released into the solution depends on the pH of the medium. The release of the antibiotic into the solution increases from an acidic to an alkaline medium. Thus, the release is higher at pH = 8.

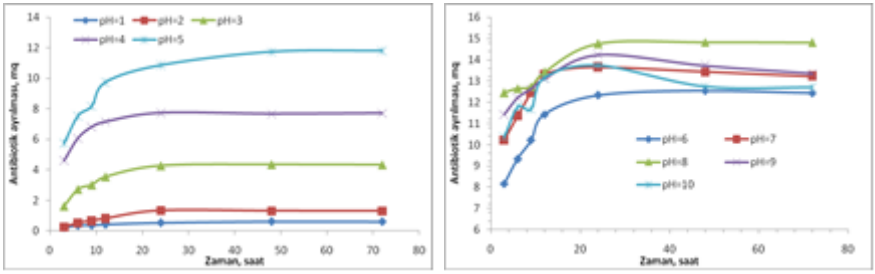



**Figure 14**. Time dependence of the release of doxorubicin-fixed gel containing magnetite $Fe_3O_4$ nanoparticles functioned with Cht in the range of pH=1÷10. T=37ºC, m=1g, V=15 ml

It has been determined that the kinetics of the release of Dox from Cht-nano $Fe_3O_4$ composite to the aqueous solution at 37ºC is two-phase. During the first 4 hours at the beginning of the experiment, the release of the Dox increases up to about 30-40 times at pH=8. However, after 12

hours although the release of the antibiotic increases, the release of Dox stabilizes after 24 hours as a result of the equilibrium process, and the release to the solution occurs in small portions within 48-50 hours. This allows the transport of Dox antibiotic from a Cht-nano $Fe_3O_4$ composite grafted with glutaraldehyde using a specific medium depending on the severity of the disease.

Experiments on the antibacterial activity of synthesized magnetite $Fe_3O_4$ nanoparticles-free composites, as well as composites containing $Ag^o$ and $Fe_3O_4$ nanoparticles have been carried out. *Staphylococcus aureus* as a member of the Gram-positive bacteria from the disease-causing type of purulent-inflament processes, Gram-negative bacteria *Escherichia coli* (intestinal spores), *Pseudomonas aeruginosa* as pigment-forming Gram-negative bacteria (blue-green purulent spores) have been taken as culture test according to the generally accepted rule to determine these properties. The most active bactericidal agents were samples containing $Ag^o$ and $Fe_3O_4$ nanoparticles, which had the most active lethal effect on Gram-negative (*Ps.aeruginosa* and *E.coli*) and Gram-positive (*St.aureus*) bacteria, i.e. sterile zones around the mesh was in the range of 35-40 mm. Samples can be considered as superior components in the preparation of materials with bactericidal effect.

One of the necessary steps in the problem of obtaining new types of piezoelectrics based on hybrids of polymer matrix nano- and micro-scale phase composites is the preparation of nanostructured polymer solution, its deposition on the piezoelectric substrate. Electrical gas discharge plasma is a convenient method in terms of eliminating the effect of immobilization in polymer composites.

The next part of the study is based on the study of a number of physical properties of $Ag^0$ nanoparticles obtained in PVPr and GA medium. N,N'- methylene-bis-acrylamide (MBAA) has been used as a crosslinking agent in the 3; 5; 7; 10 and 15% mass ratios of PVPr and GA, and silver nanoparticles have been obtained in the polymer medium. The study of the physical properties of the composites obtained in the polymer medium is main purpose here.

Three different samples have been prepared and observed to study their properties (Figure 15).

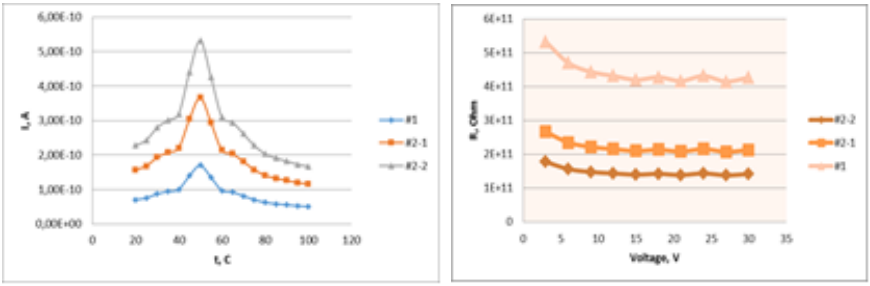



**Figure 15.** Dependence of thermally stimulated polarization and resistance of samples on applied voltage

As it is seen, in the initial phase of the temperature, the value of the current density increases and reaches its maximum value at 50°C. This condition of maximum value complies with all 3 types of samples. At temperatures above 50°C, the value of current density increases sharply. Maximum current density was lowest in sample # 1 and highest in sample # 2-2 (Figure 15). As seen from the graph, the resistance of all 3 samples decreases exponentially at small values of voltage drop. However, starting from about 15V, the electrical resistance of all 3 samples is no longer dependent on next voltage changes and remains stable.

The temperature dependence on the electrical resistance of the samples has been also studied (Figure 16).

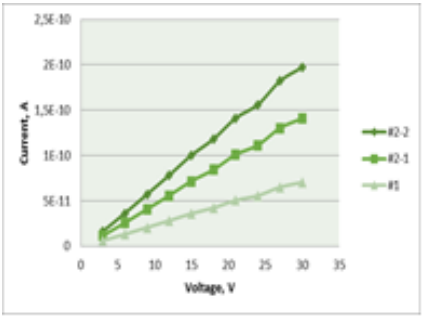



**Figure 16.** Temperature dependence of the resistance and volt-ampere characteristics of the samples

As seen from the graphs, sample # 1 has undergone the most changes. The resistance of the samples at $50^0$C has reached its minimum value. It is very interesting that the resistance of all 3 samples is different depending on the temperature. The resistance decreases to $50^0$C, after this point resistance of all samples increases sharply. This can also be confirmed by the phase transition in the samples.

Depending on the voltage, the current density in the applied samples also continues to increase mainly linearly.

## RESULTS

1. Memory-tuned polymer carriers formed in the presence of magnetite $Fe_3O_4$ nanoparticle composites have been synthesized. It has been determined that the release of doxorubicin immobilized from the memory-tuned $Fe_3O_4$ nanoparticle gel complex to solution in the first 3-5 hours occurs with a small amount and a slight jump. However, in the untuned gel samples, this release is observed with an increase of 7-12% and a higher jump release, respectively. Release of doxorubicin from tuned gel samples also occurs in smaller portions over a longer period of time.
2. Silver nanoparticles have been synthesized and their properties have been studied in a gum arabic medium, which is a natural polysaccharide. It has been found that the reduction of silver ions in the presence of sodium borohydride occurs faster than hydrogen peroxide. The obtained nanocomposites have been characterized by radiographic analysis and showed that the size of nanoparticles is 32 nm. It has been established that there is no chemical structural change in the gum arabic macromolecule after the reduction process.
3. Nanoparticles of $Ag^o$ and $Fe_3O_4$ have been obtained in the presence of gum arabic, arabinogalactan, chitosan, polyethylene glycol. The inclusion of a magnetite nanoparticle in the gel matrix increases its capacity due to

physiologically active substances (trypsin and doxorubicin). This increases the possibility of using these metal nanoparticle components in the transport systems of biologically active compounds. The higher gel capacity and immobilization rate compared to $Ag^o$ nanoparticles is due to the presence of magnetite nanoparticles not in the zero oxidation state, but in the form of mixed oxides of II and III valence, the valence potentials of which allow to form coordination.

4. "Smart" hydrogels based on poly-N-vinylpyrrolidone, polyethylene glycol, polyacrylamide, chitosan as well as gum arabic and arabinogalactan have been synthesized and $Ag^o$ and $Fe_3O_4$ nanoparticle composites have been obtained using them as stabilizers and reducing agents. The sorption ability of the obtained hydrogels to enzymes and antibiotics has been studied and it has been determined that they have an effective sorption capacity.
5. Silver nanoparticles have been synthesized in the co-polymer medium of poly-N-vinylpyrrolidone with polyacrylamide and a bio-nanocomplex of this nanogel complex with doxorubicin has been obtained. It has been determined that the durability of drug-immobilized silver nanoparticle nanogels depends on the particle size and mass ratio of the polymers, as well as the synthesis conditions. Thus, at pH = 5-8, the PVPr-grafted-PAA/$Ag^o$/doxorubicin system remains stable for a long time, and at pH=1-3, doxorubicin releases to the medium due to the weakening of the interaction between $Ag^o$/doxorubicin and PVPr-grafted-PAA matrix as a result of protonation of carbonyl in PVPr, amino groups in PAA.
6. Natural and synthetic polymers, as well as their $Ag^o$ and magnetite $Fe_3O_4$ nanoparticle polymer-colloidal systems and free levofloxacin have been impregnated on synthetic meshes with a size of 1 $cm^2$, mass of 30-40 mg and culture tests accomplished. It has been determined that most active bactericidal agents are samples containing $Ag^o$ and $Fe_3O_4$ nanoparticles. Thus, these samples had the most active lethal

effect against Gram-negative (*Ps.aeruginosa* and *E.coli*) and Gram-positive (*St.aureus*) bacteria, lysis zones were 35-40 mm in diameter.

7. Physical properties of new materials with high piezoelectric properties based on $Ag^{o}$ nanocomposites, stabilized in gum arabic and polyethylene glycol medium, were determined. It has been shown that the size of the silver nanoparticles stabilized in polyethylene glycol and gum arabic media varies in the range of 7-9 nm, and the current density flowing through the nanoparticles is about 5 times higher than others under appropriate conditions and its voltage dependence at 0-50 V is linear.

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