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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

**SYNTHESIS AND STUDY OF METAL NANOCOMPOSITES
BASED ON POLYMER MATRICES**

Specialty: 2304.01 – “Macromolecular Chemistry”

Field of science: Chemistry

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The work was performed at PLE “Institute of Chemistry” of the Ministry of Science and Education of the Republic of Azerbaijan in the Laboratory of Nanostructured Metal-Polymer Catalysts.

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GENERAL CHARACTERIZATION OF THE WORK

Relevance of the topic and the degree of its development. It is well known that the synthesis and stabilization of metal nanoparticles in polymer matrices, as well as the study of their properties, are promising directions not only in modern chemistry but also in medicine and physics. The nanoscale state of metals, owing to their unique properties, creates new opportunities for their application in various industrial sectors.

After synthesizing nanoscale metals and metal oxides, the primary challenge is maintaining their particle size and superior properties over an extended period. Research in this field shows that polymer macromolecules not only stabilize these nanodispersed systems but also direct the formation of these nanoparticles¹.

Furthermore, polymers are considered irreplaceable matrices for controlling the shape and size of the resulting particles. Among these polymers, polyelectrolytes, particularly natural and synthetic polymers containing functional groups such as -COOH, -NH₂, -OH, and -CHO, occupy a special position. By encapsulating metal nanoparticles, these functional groups prevent their aggregation and maintain their initial nanoscale dimensions. At the same time, gel-forming nanobiomaterials, such as carriers, coatings, and ultrathin films based on polymer gels combined with metal nanoparticles, are being actively developed to enhance the therapeutic efficacy of pharmacologically active drugs, representing a highly relevant field of research².

Accordingly, composites containing Ag, Au, and Fe₃O₄ nanoparticles serve as highly effective antibacterial agents.

Furthermore, the study of certain physical properties of materials containing Ag⁰ and Fe₃O₄ nanoparticles synthesized in the presence of natural and synthetic polymers bearing the aforementioned functional

¹ Aguilar, N.M. Polymers as versatile players in the stabilization, capping, and design of inorganic nanostructures / N.M. Aguilar, J.M. Perez-Aguilar, V.J. González-Coronel [et al.] // ACS Omega -2021, Vol. 6, Iss. 51, -p. 35196-35203.

² Nicolae-Maranciuc, A., Chicea, D. Polymeric Systems as Hydrogels and Membranes Containing Silver Nanoparticles for Biomedical and Food Applications: Recent Approaches and Perspectives // Gels – 2025, Vol. 11, Iss. 9, p. 699. DOI: 10.3390/gels11090699.

groups, including their current-voltage characteristics and the temperature- and voltage-dependent resistance, is of considerable interest. In addition, the use of natural polymers containing Fe₃O₄ nanoparticles, as well as their graft copolymers with poly-N-vinylpyrrolidone, as a carrier matrix for immobilizing trypsin and doxorubicin as model drug substances, represents another relevant area of research. Moreover, incorporating the antibacterial drug levofloxacin into complexes of natural polymers and their composites with poly-N-vinylpyrrolidone containing Ag⁰ nanoparticles, and evaluating the antimicrobial activity of these nanobiocomposites against bacteria such as *Staphylococcus aureus*, *Escherichia coli*, and *Pseudomonas aeruginosa*, is particularly significant.

On the other hand, a serious problem in the modern oil and gas industry is water encroachment as service life increases. This creates additional economic and technical challenges and consequently increases the product's cost. Among the technologies used to ensure effective isolation of the bottomhole environment and the casing-tubing annulus, cement-based and gel-forming compositions are distinguished by their functional advantages and are widely used. For many years, cement-based consolidation systems have been associated with cracking from rock movement, rapid disintegration, and degradation during perforation³. From this perspective, developing cement-polymer composite formulations with enhanced elasticity that can be readily perforated, are resistant to thermal effects, and exhibit adhesion to rock surfaces is a relevant area of research. Additionally, formulating cement with polymer/nanoFe₃O₄ dispersed solutions increases its thermal stability, thereby extending its service life.

Given the above, the dissertation is relevant to the study of new types of medical and cement materials based on colloidal systems containing Ag⁰ and Fe₃O₄ nanoparticles, developed in the presence of poly-N-vinylpyrrolidone (PVP), polyethylene glycol (PEG), and polyacrylamide (PAA), as well as the natural polysaccharides chitosan (Cht), gum arabic (GA), and arabinogalactan (AG).

The work was carried out in accordance with the scientific research

³ Cheng, X. Effect of Hole on Oil Well Cement and Failure Mechanism: Application for Oil and Gas Wells / X. Cheng, C. Cao, L. Wu, W. Song // ACS Omega -2022, Vol. 7, Iss. 7, 5972-5981.

plan (State Registration No. **0115Az2096**) of the Institute of Catalysis and Inorganic Chemistry named after Academician M.F. Naghiyev of ANAS (currently the Public Legal Entity "Institute of Chemistry" under the Ministry of Science and Education of the Republic of Azerbaijan).

Aim and objectives of the research. - The main aim of the research is to synthesize pH-sensitive “smart” materials containing silver and magnetite nanoparticles in synthetic and natural polymer matrices, using additional reducing agents and incorporating polymeric substances into the system as reducing agents, and to investigate the feasibility of using these nanocomposites as carriers and to determine their biological and electrical conductivity properties. Another important direction of the research is the preparation and investigation of polymer-containing compositions with magnetite nanoparticles to ensure elasticity, adhesion, and uniform heat distribution in cement mixtures after hardening.

To achieve the stated aim, the following objectives are outlined:

- ✓ Synthesis of Ag^0 nanoparticles in the presence of PVP, PEG, Cht, AG, and GA as stabilizing agents, and, under specific conditions, with various reducing agents, followed by structural investigations;
- ✓ Synthesis of Fe_3O_4 nanoparticles in PVP, PEG, GA, and Cht media and spectroscopic identification of the resulting gel-nanoparticle complexes;
- ✓ Investigation of the swelling properties of synthesized natural - and synthetic-based magnetite Fe_3O_4 nanoparticle-containing gel complexes and study of their carrier properties for the preparation of biologically active compounds by immobilizing trypsin and doxorubicin as model preparations.
- ✓ Investigation of the current-voltage characteristics and the dependence of electrical resistance on voltage and temperature in Fe_3O_4 and Ag^0 nanoparticle-containing composites prepared in the presence of natural and synthetic polymers;
- ✓ Investigation of the antibacterial activity of homogeneous mixtures of natural polymers containing silver nanoparticles and their complexes with PVP and levofloxacin in specific microbial media on specialized meshes;
- ✓ Preparation of cementitious compositions by mixing cement with three types of water: I - ordinary water, II - water + PAA, and III -

a PAA-containing liquid stabilized with Fe_3O_4 nanoparticles exhibiting magnetite properties, and determination of the setting time, density, compressive strength, and other physicochemical parameters of the samples;

- ✓ Investigation of the temperature dependence of the electrical conductivity, spreading diameter, and setting time of hardened cement paste prepared with PAA-nano- Fe_3O_4 mixtures at varying concentrations

Research Methods. Modern physicochemical analytical methods, including UV-Vis spectroscopy, Fourier-transform infrared (FTIR) spectroscopy, X-ray diffraction (XRD), thermogravimetric analysis (TGA), electrochemical impedance spectroscopy (EIS), and others, were used to identify and characterize the initial and synthesized products.

Main Provisions Submitted for Defense. Based on the dissertation's principal findings and their comparison with existing theories and prior studies, the main provisions for defense are as follows:

- Synthesis and characterization of Ag^0 and Fe_3O_4 nanoparticles in PVP and PEG media using physical research methods.
- Synthesis, stability, structural analysis, and investigation of Ag^0 and Fe_3O_4 nanoparticles stabilized by Cht, GA, and AG.
- Investigation of the nature of the chemical interaction between biocomplexes containing Ag^0 and Fe_3O_4 nanoparticles, based on the aforementioned polymer composites, and trypsin.
- Investigation of the biological activity and antibacterial properties of synthesized Ag^0 - and Fe_3O_4 -containing gel materials, as well as the lysis zones of surgical meshes impregnated with levofloxacin in specific microbial media.
- Investigation of the physical properties, including current–voltage characteristics and electrical resistance, of natural and synthetic polymer nanocomposites containing synthesized Ag^0 nanoparticles as a function of various parameters.
- Spectroscopic investigation of the structure and principal physicomachanical properties of hardened cement paste samples prepared using PVP, PAA, and their complexes containing magnetite nanoparticles.

Main Scientific Novelty of the Research.

- “Smart” hydrogels based on PVP, PEG, Cht, GA, and AG were synthesized. Composites containing Ag^0 and Fe_3O_4 nanoparticles were obtained using these hydrogels as stabilizing and reducing agents, and their physical properties were investigated.
- The carrier properties of the biocomplexes containing the obtained metal nanoparticles were investigated by immobilizing trypsin and doxorubicin.
- To impart additional antibacterial properties to medical meshes used in the treatment of abdominal hernias, a nano- Ag^0 gel-levoﬂoxacin solution was applied to their surfaces via mild heating, and the resulting materials were subjected to in vitro bioassays.
- In addition, cement slurries containing PAA, PVP, PAA/ Fe_3O_4 , and PVP/ Fe_3O_4 were developed as effective plugging materials with high adhesion to rock surfaces to isolate formation water in the bottomhole zone in the oil industry. The polymer and nanoparticle phases were immobilized within the cement matrix.
- The developed cement slurry can be used not only for the long-term, effective strengthening of bottomhole zones in the oil and gas industry but also for construction. It was established that hardened cement, owing to its elastic properties, is resistant to mechanical impacts and seismic activity, while the presence of magnetite nanoparticles enhances adhesion to metal equipment.

Theoretical and Practical Significance of the Research.

Gel complexes containing Ag^0 and Fe_3O_4 nanoparticles were synthesized in “smart” polymer matrices based on PVP, PEG, Cht, GA, and AG. An immobilization method was developed that enables these materials to exhibit carrier properties and antibacterial activity at specific pH values. Such nanocomposites, capable of providing prolonged, controlled release in response to environmental stimuli and possessing additional biological properties arising from both the matrix and the metal nanoparticles, can serve as effective matrices for the delivery of pharmaceutical agents. In addition, complexes of natural- and synthetic-polymer-based materials with Ag^0 and Fe_3O_4 nanoparticles may be important as carriers for the targeted delivery of antibiotics and certain enzymes across various environments, while also helping address

deficiencies of these trace elements in the body.

Furthermore, physical parameters such as current–voltage characteristics and electrical resistivity of polymer composites containing Ag^0 and Fe_3O_4 nanoparticles enable these materials to serve as substrates for the development of electrically conductive wires and components for optical devices in electronics.

In addition, colloidal solutions containing dispersed Fe_3O_4 nanoparticles can be obtained in PAA and PVP media. These solutions contain magnetite nanoparticles with sizes ranging from 40 to 100 nm and exhibit thermal stability of 80–140 °C. An increase in the elasticity, compressive strength, and resistance to fracture of cement masses prepared by mixing cement with the polymer-nano- Fe_3O_4 solution was observed. The presence of metal oxide nanoparticles in the composition also imparts thermal stability to the material, thereby increasing its service life. The results demonstrate the potential to expand research further and develop hardened cement paste with enhanced elasticity and compressive strength. This can be achieved by regulating the average molecular weight of the polymer, the size and amount of nanoparticles in the composition, or by using biopolymers and by-products of various industrial processes.

Building on the dissertation's scientific results and citations in published periodicals, the research can be further expanded, and the newly developed Ag^0 - and Fe_3O_4 -containing composite matrices could be used to deliver other physiologically active substances. Furthermore, combining natural and synthetic polymers that serve as primary matrices with other non-toxic, low-molecular-weight substances and metal nanoparticles may provide a basis for developing new carrier systems, including materials with controllable electrical properties, and for extending research into other fields.

Approbation and Application. The results of the dissertation research were presented at the following national and international scientific conferences: Mendeleev-2014: All-Russian Conference of Young Scientists in Chemistry with International Participation (St. Petersburg, 2014); XXVI International Chugaev Conference on Coordination Chemistry (Kazan, 2014); OrgChem-2016: Cluster Conference on Organic Chemistry (St. Petersburg, 2016); IV International Scientific Conference of Young Researchers, dedicated to

the 93rd Anniversary of the National Leader of Azerbaijan, Heydar Aliyev (Baku, 2016); Ankara IX International Congress on Scientific Research (Ankara, 2023); International Biotechnology & Nanotechnology Congress (Ardahan, 2024); Tokyo 8th International Innovative Studies & Contemporary Scientific Research Congress (Tokyo, 2024); XXVII All-Russian Conference of Young Chemists with International Participation (Nizhny Novgorod, 2024); "Modern Problems of Macromolecular Compounds Technology" International Scientific Conference (Baku, 2024); International Ceramics and Composite Materials Symposium (Isparta, 2024).

The scientific findings regarding the topic and practical significance of the dissertation were honored with the M. Naghiyev Award (2016), conferred by the Institute of Catalysis and Inorganic Chemistry named after Academician M. Naghiyev of the Azerbaijan National Academy of Sciences.

Name of the Organization Where the Dissertation Was Conducted. The dissertation research was carried out at the Laboratory of Nanostructured Metal-Polymer Catalysts of the “Institute of Chemistry” Public Legal Entity under the Ministry of Science and Education of the Republic of Azerbaijan. In addition, the antibacterial properties, structural and spectroscopic characteristics, and physical parameters, including the current–voltage characteristics of the samples, were analyzed at Azerbaijan Medical University, the “Institute of Physics” Public Legal Entity under the Ministry of Science and Education of the Republic of Azerbaijan, and the Joint Institute for Nuclear Research (Dubna, Russia).

Published Scientific Works Related to the Dissertation. Materials reflecting the dissertation's research have been published in 21 scientific works. These include 11 conference abstracts and 10 scientific articles in national and international peer-reviewed journals.

Volume and Structure of the Dissertation. The dissertation comprises an Introduction (19426 characters), four chapters (Chapter I - 47786 characters; Chapter II - 31031 characters; Chapter III - 81661 characters; Chapter IV - 43796 characters), General Conclusions (3262 characters), and appendices containing test evaluation reports. The dissertation totals consist of 226962 characters and 176 computer-

formatted pages, including 75 figures, 9 tables, and a list of 181 cited references.

MAIN CONTENT OF THE WORK

The Introduction substantiates the dissertation's relevance, outlines its aim and objectives, highlights its scientific novelty and practical significance, and presents the main provisions to be defended.

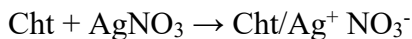
The first chapter provides a comprehensive review, systematization, and comparative analysis of literature from the past 10-15 years on the synthesis of natural- and synthetic-based composites containing metal nanoparticles, particularly those based on PVP, PEG, and polysaccharides such as Chitosan (ChT), GA, and AG. The chapter also examines the role of hydrogels and metal nanoparticle-containing gels in drug delivery, the immobilization of biologically active compounds in these gel and metal-gel systems, and their potential applications in electronics and optoelectronics.

The second chapter is devoted to experimental methodology. It describes the experimental procedures, starting materials and their preparation, methods for evaluating the physicochemical and biological properties of the synthesized products, and the application of physical analytical methods to investigate their parameters. This chapter also presents methods for determining the inhibition zones of polymer-levofloxacin nanobiocomposites impregnated with Ag^0 nanoparticles, as well as the preparation procedures for polymer-cement nanocomposites containing magnetite (Fe_3O_4) nanoparticles.

The third chapter presents and discusses the results obtained from the synthesis of Ag^0 - and Fe_3O_4 -containing composites based on natural and synthetic polymers, structural confirmation using physical analytical methods, and the investigation of the immobilization and delivery of selected pharmaceuticals. This chapter provides a detailed description of the antibacterial properties of newly synthesized gel systems and combinations of gel complexes containing Ag^0 nanoparticles with levofloxacin. It also examines the feasibility of using these materials as effective matrices for delivering enzymes and antibiotics, with trypsin and doxorubicin as model drugs. In addition, certain physical properties of Ag^0 -containing composites were investigated, including their current-

voltage characteristics and the dependence of electrical resistance on voltage and temperature as a function of the polymer and nanoparticle nature.

Owing to its biocompatibility, biodegradability, non-toxicity, bioactivity, and multifunctionality, Cht has long been established as a natural biopolymeric cation exchanger. In a Cht solution containing dispersed Ag^+ ions, the metal ions coordinate with the $-\text{NH}_2$ and $-\text{OH}$ groups, forming pseudocomplexes through weak interactions.⁴



During our studies, adding NaBH_4 as a reducing agent reduced silver ions under ambient conditions, and the reaction continued for a defined time interval. The reduction process was accompanied by a gradual color change in the solution, as illustrated in the figure below.



Figure 1. Color change of the Cht- Ag^0 suspension as a function of reaction time

As shown in Figure 1, during the reduction process, the system's color changes from yellow to dark brown, indicating the formation of a colloidal solution containing Ag^0 nanoparticles. The following equation represents the reduction of Ag^+ ions:



⁴El-Araby, A. Chitosan, chitosan derivatives, and chitosan-based nanocomposites: eco-friendly materials for advanced applications (a review) / A. El-Araby, W. Janati, R. Ullah [et al.] // Frontiers in Chemistry -2024, Vol.11, 1327426.

One of the physical characterization methods used to confirm the presence of nanoparticles is UV-Vis spectroscopy, which produces a characteristic, intense absorption peak at a specific wavelength for each colloidal solution containing metal nanoparticles (Figure 2).

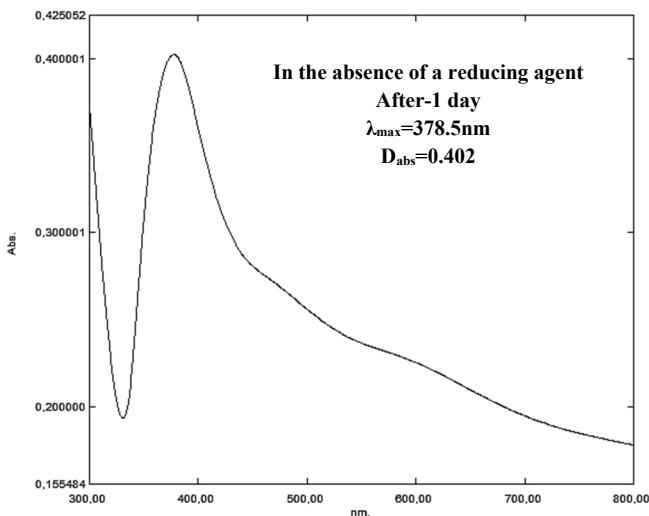


Figure 2. UV-Vis electronic absorption spectrum of Ag^0 nanoparticles formed in the Cht matrix without a reducing agent

As shown, the UV-Vis spectrum of the Cht/ Ag^0 colloid obtained after one day without a reducing agent shows a single characteristic absorption peak at 378.5 nm, attributed to silver atoms with a face-centered cubic (FCC) crystal lattice. This result indicates that the Ag^0 nanoparticles formed in the colloidal system are very small and exhibit a highly uniform, monodisperse distribution. Such peaks are characteristic of spherical nanoparticles with a low degree of aggregation, confirming that Cht functions both as a reducing agent and as an effective stabilizer.

Given that the surface plasmon resonance (SPR) band of Ag^0 nanoparticles typically occurs within the 350-450 nm wavelength range, the observed absorption peak indicates that the nanoparticles are distributed within a relatively narrow size fraction in the suspension.

Thus, the result supports this interpretation⁵. The multifunctional nature of Cht enables the reduction of a certain amount of Ag⁺ ions even in the absence of an external reducing agent. This process can be attributed to the stabilizing and weakly reducing properties of Cht.

It is well known that X-ray diffraction (XRD) is a principal method for determining crystalline phases. An X-ray phase analysis was performed on the composite containing Ag⁰ nanoparticles stabilized in the Cht polymer matrix. For comparative analysis, the X-ray diffraction pattern of the initial Cht polymer was first examined (Figure 3), and it was determined that the matrix is amorphous.

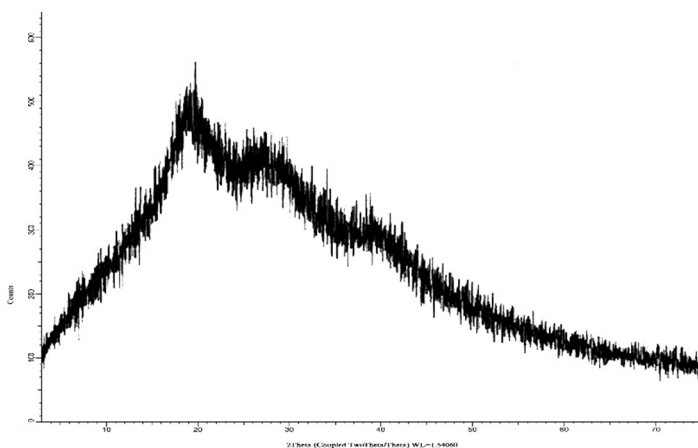


Figure 3. XRD pattern of pure Cht

As shown, no characteristic peak corresponding to a crystalline phase is observed in the X-ray diffraction pattern of pure Cht. The broad, low-intensity peak at approximately $2\theta \approx 19.8^\circ$ indicates the amorphous nature of Cht. This amorphous character can be attributed to Cht's non-crystalline structure and the irregular distribution of its hydrophobic and hydrophilic regions. The expected low degree of crystallinity confirms Cht's high functionality and its suitability as a matrix for nanocomposite

⁵ Kulikouskaya, V. Chitosan-capped silver nanoparticles: A comprehensive study of polymer molecular weight effect on the reaction kinetic, physicochemical properties, and synergetic antibacterial potential / V. Kulikouskaya, K. Hileuskaya, A. Kraskouski [et al.] // SPE Polymers -2022, Vol. 3, Iss. 2, 77-80.

synthesis. However, in the diffraction pattern of the composite containing silver nanoparticles (Figure 4), three intense diffraction peaks characteristic of Ag⁰ atoms are observed at specific 2θ values.

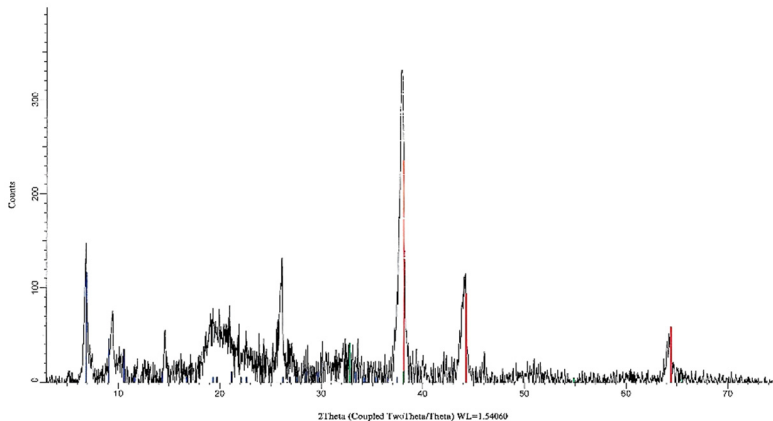


Figure 4. XRD pattern of Cht/Ag⁰ composite

These correspond to the (111), (200), and (220) planes, with characteristic Bragg reflection angles for Ag⁰ atoms at approximately 2θ ≈ 38°, 44°, and 64°, respectively⁶. Based on X-ray phase analysis, the Ag⁰ nanoparticles form a face-centered cubic (FCC) crystal structure within the Cht matrix. The size of the Ag⁰ nanoparticles in the polysaccharide matrix was determined using the Debye–Scherrer equation:

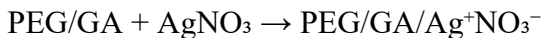
$$D = K\lambda / (\beta \cos\theta)$$

Where D is the average crystallite (nanoparticle) size, K is the Scherrer constant (0.9–1.0), λ is the X-ray wavelength (1.5418 Å), β is the full width at half maximum (FWHM) of the diffraction peak, and θ is the Bragg angle. The average nanoparticle size was determined to range from 14 to 25 nm, depending on the reduction

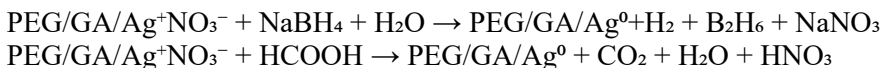
⁶ Ali, M.H. Analysis of crystallographic structures and properties of silver nanoparticles synthesized using PKL extract and nanoscale characterization techniques / M.H. Ali, M.A.K. Azad, K.A. Khan [et al.] // ACS Omega -2023, Vol. 8, Iss. 31, 28133-28142.

time and temperature.

During the research, the size range of the reduced silver nanoparticles was also determined by replacing the stabilizing matrix with a synthetic polymer. For this purpose, water-soluble PEG and GA were used as a combined polymer system. When PEG and GA macromolecules dissolve in an aqueous medium, they form a homogeneous system in which their functional groups undergo conformational interactions and approach one another. Ag^+ ions become coordinated by the -OH and -COOH groups:



Upon addition of a reducing agent to the system, the silver ions undergo chemical transformations described by the following reaction equations:



It is well known that molecular electronic spectroscopy (UV-Vis spectroscopy) is a principal method for confirming the presence of metal nanoparticles in solution and verifying their formation. Surface plasmon resonance (SPR) is specific to metal nanoparticles and is characterized by an intense absorption peak in the spectrum. After treatment of the obtained nanocomposite with diethyl ether or ethanol, the resulting solution in deionized water was analyzed by UV-Vis spectroscopy at different temperatures (Figure 5). The characteristic surface plasmon resonance band observed in the 409-415 nm range in the UV-Vis spectra of the synthesized Ag nanoparticle colloidal solutions showed no significant changes over 2 hours to 10 days, confirming the high colloidal stability of the nanoparticles. These results demonstrate that PEG and GA form an effective protective matrix that stabilizes Ag nanoparticles.

It is well known that the plasmonic properties of nanoparticles vary with their size, shape, dispersity, and interactions with the surrounding medium. One of the most widely used methods for

monitoring these changes is UV-Vis spectroscopy. This method enables monitoring the optical stability of metal nanoparticles in a colloidal medium and tracking changes over time.⁷

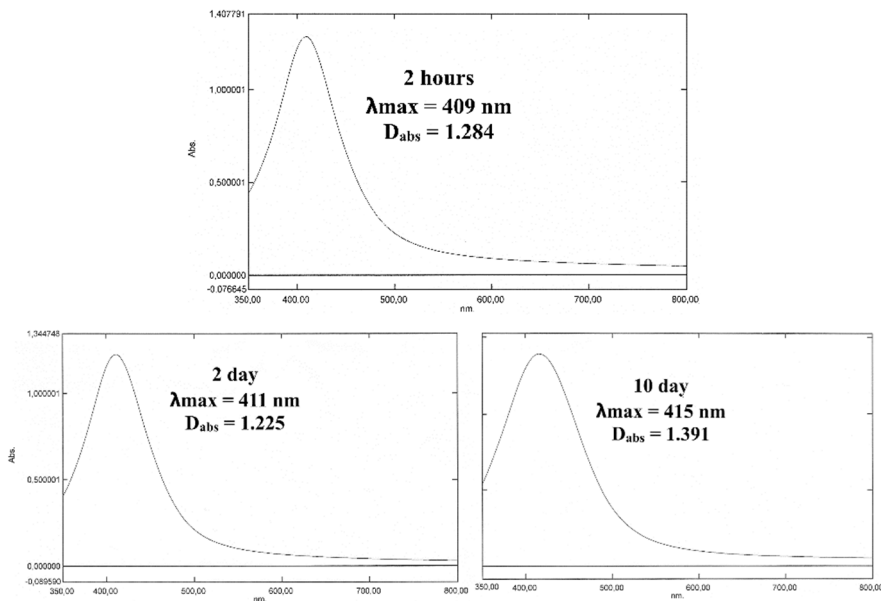


Figure 5. UV-Vis absorption spectra showing the dependence of absorbance in the PEG-GA-Ag⁰ colloidal system on reduction time

The UV-Vis spectroscopic analyses performed in this study yielded several important findings regarding the evaluation of the nanoparticles' optical properties and the monitoring of the colloidal system over time. In this regard, the PEG-GA-Ag⁰ composite system demonstrated both the effectiveness of the synthesis process and the material's initial stability. The changes observed over time in wavelength and absorbance values enabled a dynamic analysis of the stability of the colloidal medium, aggregation processes, and particle dispersity. This, in turn, enables evaluation of the system's overall behavior and identification of stability disturbances at an early stage.

⁷ Arif, M. UV-Vis spectroscopy in the characterization and applications of smart microgels and metal nanoparticle-decorated smart microgels: a critical review / M. Arif, H. Raza, T. Akhter // RSC Advances -2024, Vol. 14, 38120-38134.

In subsequent studies, the sizes of Fe_3O_4 nanoparticles formed within the gel samples and the degree of swelling of these gel systems in different media were investigated. The amounts of immobilized trypsin and doxorubicin, their immobilization efficiency, and other parameters were also determined. Considering the release process of trypsin and doxorubicin immobilized in the gel samples, the time dependence of the swelling behavior of the initially synthesized magnetite-gel systems was first investigated at different pH values of the medium. The results are presented in the figures below.

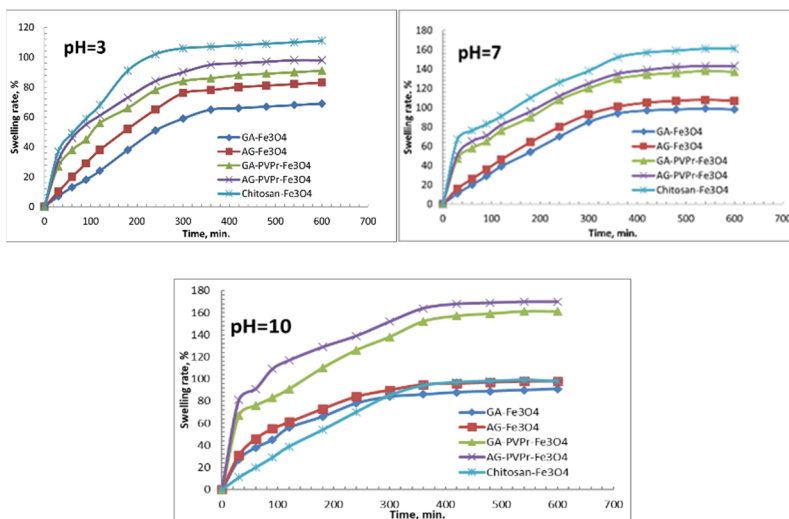


Figure 6. Time-dependent swelling of the nano- Fe_3O_4 hydrogel composite at different pH values

As shown, the nano- Fe_3O_4 hydrogel samples exhibit pH sensitivity that varies with the nature of the functional groups in the main matrix. It was established that, for all gels, the degree of swelling increases sharply during the initial 60 minutes, then gradually, with smaller increments, eventually reaching swelling equilibrium.

In the Cht-based matrix, the degree of swelling of the nanogel composite reaches its maximum at $\text{pH} = 7$, compared with all other samples and media. In contrast, at $\text{pH} = 10$ it decreases to its minimum. This can be attributed to the shrinking and collapse of the Cht chains at $\text{pH} = 10$, as well as the inability of the basic functional groups of the

grafted PVP macromolecules to become polarized in this medium, resulting in repulsion of the ions present. At pH = 3, although the degree of swelling is lower than that observed at pH = 7 due to the protonation of -NH₂ groups, it remains relatively high compared with the other samples.

In the unmodified GA and AG samples, the degrees of swelling at pH = 7 and pH = 10 remain relatively similar; no sharp increase is observed in the alkaline medium. In contrast, the PVP-grafted derivatives (GA-PVP and AG-PVP) reach their maximum swelling at pH = 10. This can be explained by the ionization of the -COOH groups in the alkaline medium, which causes electrostatic repulsion between the polymer chains and expansion of the network. At pH = 3, all GA/AG-based samples (both unmodified and PVP-grafted) exhibit the lowest degree of swelling.

The results demonstrate that magnetite nanogels based on Cht matrices can be used for the delivery of biologically active pharmaceutical substances at pH = 7, whereas Fe₃O₄ nanogel composites based on PVP-grafted GA/AG can be utilized for the delivery of biologically active preparations in media close to pH = 10 (slightly alkaline conditions).

In the next stage of the study, the loading, transport, and release capabilities of the synthesized gel matrices containing magnetite Fe₃O₄ nanoparticles were investigated using doxorubicin as a model drug (Table 1).

Table 1. Immobilization parameters for doxorubicin onto magnetite Fe₃O₄ nanoparticle-containing complexes of natural and synthetic polymers. (V = 20 mL, T = 20°C, t = 24 h, m(gel) = 50 mg, C₀(dox.) = 500 mg/L)

Gel carriers	Equilibrium concentration mg/L	Immobilization degree, %	Gel capacity, mg/g
PVPr 10 kDa-Fe ₃ O ₄	367,3	26,54	53.08
PAAc 40 kDa-Fe ₃ O ₄	342,6	31.48	62.96
Cht - Fe ₃ O ₄	304,8	39.04	78.08
AG-PVPr (5%)-Fe ₃ O ₄	310,7	37.86	75.72
GA-PVPr (5%)-Fe ₃ O ₄	309,2	38.16	76.32

As shown in the table, Fe_3O_4 nanoparticle-containing complexes based on natural polymers (Cht- Fe_3O_4 , GA-PVP- Fe_3O_4 , and AG-PVP- Fe_3O_4) exhibit a higher antibiotic carrier capacity than analogous complexes based on synthetic polymers (PVP- Fe_3O_4 and PAA- Fe_3O_4). This can be explained by the polyfunctionality of natural polymer macromolecules and the greater activity of hydroxyl, carboxyl, and charged groups that can participate in interactions in solution under the corresponding pH conditions. As a result, the carrier's functional groups can readily undergo electrostatic and chemical interactions with the small-molecule doxorubicin.

The next part of the study focused on investigating several physical properties of the Ag^0 nanoparticles obtained in the presence of GA and PEG media, particularly the dependence of the sample resistance on the applied voltage [2]. To investigate these properties, three samples were prepared, and measurements were performed at three temperatures (Figure 7).

#1GA+PEG +Ag (100-150 kgf/cm²) ($\tau = 25$ min)($t = 105-110^\circ\text{C}$) ($t=20^\circ\text{C}$)
 #2-1GA +PEG +Ag (100-150 kgf/cm²) ($\tau=25$ min)($t=110-120^\circ\text{C}$) ($t=40^\circ\text{C}$)
 #2-2GA +PEG +Ag (100-150 kgf/cm²) ($\tau=25$ min)($t=105-110^\circ\text{C}$) ($t=60^\circ\text{C}$)

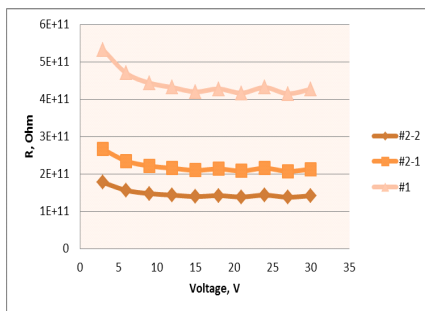


Figure 7. Dependence of the sample resistance on the applied voltage[2]

As shown in the graph, at low applied voltages, the electrical resistance of all three samples decreases exponentially. However, starting at approximately 15 V, the electrical resistance of all samples becomes independent of further changes in the applied voltage and remains stable.

During the study, the temperature dependence of electrical resistance and the current-voltage (I-V) characteristics of analogous polymer and silver nanoparticle-containing samples were also investigated. The corresponding dependence curves are shown in Figure 8.

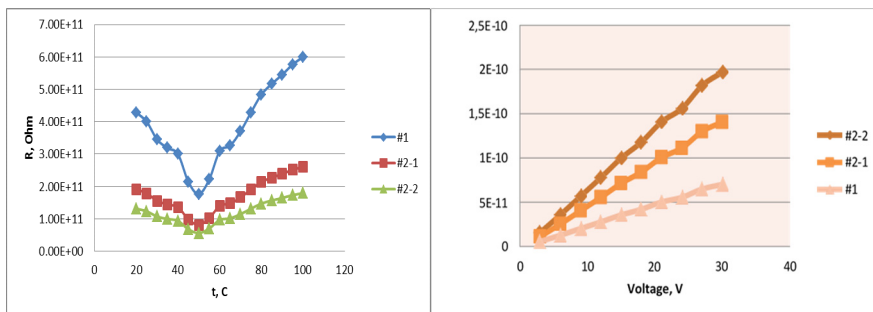


Figure 8. Dependence of the samples electrical resistance on temperature and their current-voltage characteristics[2]

As the graphs show, Sample #1 undergoes the most pronounced changes. At 50 °C, the resistance of the samples reaches its minimum value. The resistance of all three samples exhibits distinct temperature-dependent behavior. While the resistance decreases with increasing temperature up to 50 °C, it then increases sharply above this temperature. This behavior can be attributed to a phase transition occurring within the samples.

The current through the samples also continues to increase with the applied voltage, indicating a predominantly linear dependence.

Chapter four presents the results of studies conducted to enhance certain physico-mechanical properties of well-plugging cement by incorporating high-molecular-weight PVPr and PAA, as well as their magnetite nanoparticle-stabilized modifications, into polymer matrices and into mixtures prepared at various water–cement ratios. Furthermore, the results of FTIR, XRD, thermogravimetric analysis (TGA), and electrochemical impedance spectroscopy (EIS) of the hardened cement paste are discussed. In addition, a comparative study was carried out on the adsorption capacities of hardened concrete containing 1 wt% PVPr and 30–50 nm magnetite nanoparticles in formation water and Caspian Sea water.

It is well known that one of the key parameters when applying plugging cements for near-wellbore zone reinforcement is the fluidity of the freshly prepared cement slurry. The effect of PVPr concentration on the spreadability of the cement paste at various water–cement ratios is presented in Figure 9(a).

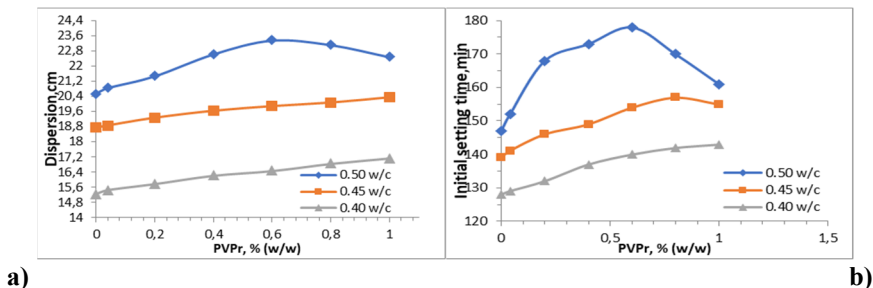


Figure 9. Dependence of the spread and initial setting time of freshly prepared cement paste on PVPr content at different water-to-cement ratios at $T = 24\text{ }^{\circ}\text{C}$ [3]

As shown, PVPr has a pronounced effect on the cement slurry's spreadability at a water–cement ratio of 0.5. As the concentration of PVPr in the fresh paste increases, the slump flow increases across all water–cement ratios. Above a PVPr dosage of 0.6%, a gradual decrease in spreadability is observed in all cases. As the water–cement ratio decreases, the thickening of the added PVPr solution is offset by the polymer's viscosity, which enhances overall flowability. At higher PVPr concentrations ($>5\%$), the cement paste's spreadability decreases relative to the control sample. This can be attributed to the polymer forming strong ionic interactions with cations in the cement matrix, thereby accelerating hydration, as well as to its active participation in the hydration process. This behavior is also clearly reflected in the investigation of the initial setting times of the cement slurry (Figure 9b). As shown, low PVPr concentrations retard the setting process. However, when the PVPr content exceeds 0.8%, the initial setting time decreases, approaching the value observed in the polymer-free reference system. Therefore, a PVPr concentration of 0.6% can be considered the optimal dosage for injection applications [3].

It is well established that X-ray diffraction (XRD) analysis plays a

crucial role in determining structural and crystalline phase changes in cement stone, as well as the interactions of incorporated additives with the cement matrix. To this end, the effects of incorporating PVPr and magnetite (Fe_3O_4) nanoparticles into cement stone on the formation of crystalline phases and structural characteristics were investigated using XRD. During the study, X-ray diffraction patterns of hydrated cement, PVPr-modified concrete stone, and PVPr/ Fe_3O_4 -modified concrete stone were obtained [8].

The XRD pattern of cement-based concrete shows characteristic diffraction peaks at 2θ values of 26.58° , 29.55° , 32.49° , 34.12° , 41.32° , 47.23° , 50.68° , and 51.87° , corresponding to dicalcium and tricalcium silicates and calcium aluminate silicates (Figure 10).

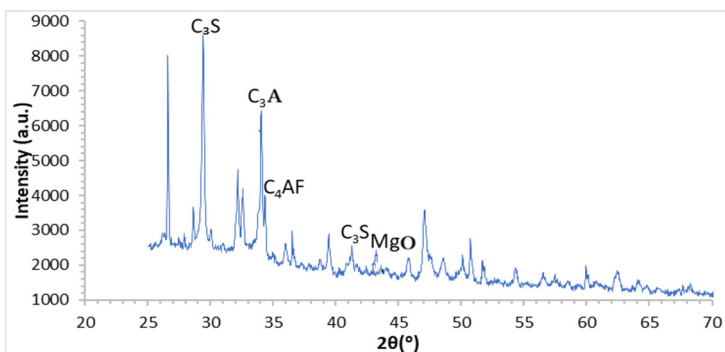


Figure 10. XRD diffractogram of the control concrete sample after 28 days of curing.[8]

To perform comparative analyses, various concrete samples were prepared. To elucidate the structural roles of both the polymer and magnetite nanoparticles, X-ray diffraction patterns of PVPr-modified concrete and PVPr/ Fe_3O_4 -modified concrete stones were also recorded (Figure 11).

It was established that increasing the magnetite nanoparticle content within the concrete matrix leads to clearer resolution of characteristic peaks and a slight increase in their intensity in the X-ray diffraction pattern. In addition, the more rigid crystalline structure of the magnetite nanoparticles further enhances the X-ray diffraction pattern of the concrete. It is known that iron atoms in magnetite nanoparticles are not present as free ions (charged species) but as discrete particles ranging

from 30 to 45 nm in size. The observation of characteristic magnetite nanoparticle peaks at their corresponding 2θ values confirms that the nanoparticles do not undergo size degradation within the concrete matrix. Furthermore, the incorporation of magnetite nanoparticles into the concrete pores, while retaining their physical and chemical state, contributes to the reinforcing properties of the concrete matrix [8].

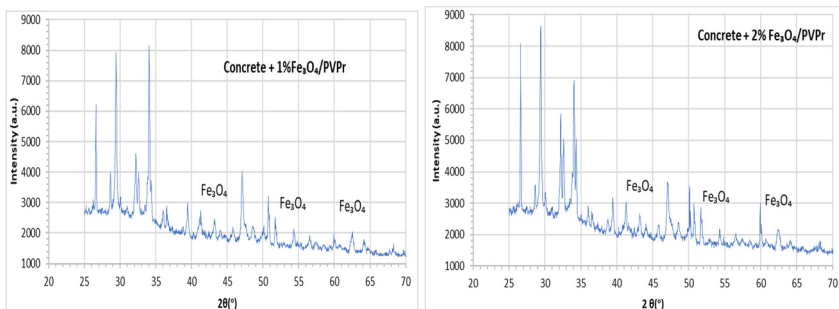


Figure 11. XRD diffractograms of concrete samples containing 1% and 2% Fe_3O_4 /PVPr after 28 days of curing[8]

During the study, certain adsorption and physical properties of concrete samples prepared by incorporating magnetite nanoparticles synthesized in a PAA medium into Portland cement were also investigated. After the hardened stones had fully cured for 28 days, mass changes in Caspian Sea water and formation water were evaluated and compared with those of the control concrete sample (Figure 12).

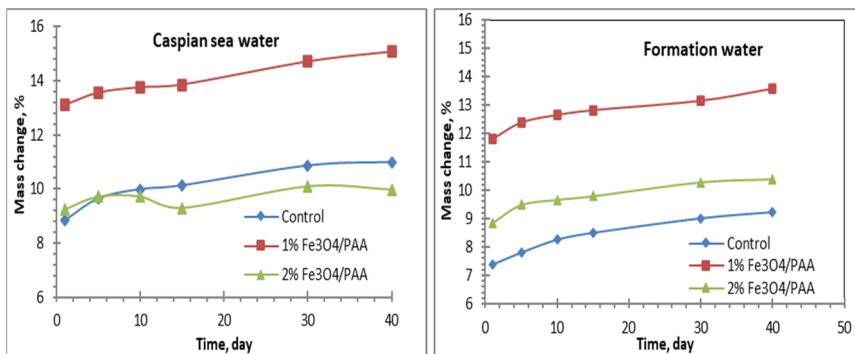


Figure 12. Time dependence of mass changes in cement stone treated with PAA/ Fe_3O_4 magnetite nanoparticles in Caspian Sea water and formation water[5]

It was established that the control concrete and the concrete containing 2 wt% PAA/Fe₃O₄ nanoparticles undergo adsorption to a similar extent during the first 10 days. In contrast, the concrete containing 1 wt% PAA/Fe₃O₄ nanoparticles exhibits a 4-4.5% greater mass increase, reaching an adsorptive mass increase of up to 15% over 40 days. In the concrete containing 2 wt% PAA/Fe₃O₄ nanoparticles, ion penetration is restricted, resulting in an even smaller mass variation than in the control concrete. Overall, the mass gain clearly stabilizes after approximately 40 days in all cases. The higher adsorption observed in the concrete sample containing 1 wt% PAA/Fe₃O₄ nanoparticles is presumably attributable to the presence of magnetite particles. The PAA macromolecules only affect the long-term stability of the magnetite nanoparticle size. At a concentration of 1 wt%, the surface distribution of PAA-stabilized magnetite nanoparticles promotes the formation of pores that facilitate the penetration of water molecules into the inner layers. As the concentration of magnetite nanoparticles increases, the concrete surface becomes more hydrophobic, thereby hindering the penetration of salt ions and water molecules. Since adsorption occurs predominantly at the surface, the iron oxide nanoparticles act as a protective barrier, limiting the ingress of hydrated ions into the concrete matrix.

Observations in formation water yielded results somewhat different from those in seawater. The control concrete sample exhibited a 9–9.2% increase in mass over 40 days in formation water. In the concrete modified with PAA-stabilized magnetite nanoparticles, absorption of formation water increased with higher nanoparticle content. However, in this case as well, the concrete containing 1 wt% Fe₃O₄/PAA exhibited the highest ion absorption. As the content of polymer-stabilized magnetite nanoparticles increased, ion penetration became more restricted, resulting in reduced water adsorption. This difference is naturally attributed to the distinct chemical compositions of the two aqueous media [5].

To limit undesirable chemical processes, various physicochemical methods and advanced instruments can be used to evaluate corrosion in reinforced concrete systems. Among these

techniques, electrochemical impedance spectroscopy (EIS) is particularly effective for characterizing electrochemical systems and determining the nature of electrolytic processes occurring within them. Furthermore, when organic or inorganic substances are incorporated into cement, EIS is an appropriate technique for evaluating the pozzolanic effect during hydration. While EIS studies of concrete/polymer or nanoparticle/concrete structures containing various polymers and metal oxide nanoparticles have been reported, studies on preparing polymer–nanoparticle–concrete systems from cement paste, along with comprehensive microstructural, mechanical, and EIS characterization, remain exceedingly rare.

Taking the above considerations into account, the present dissertation prepared and characterized concrete samples containing varying amounts of magnetite nanoparticles stabilized with low- to medium-molecular-weight hydrophilic anion-exchange PAA by electrochemical impedance spectroscopy (EIS) after 28 days of curing (Figures 13 and 14). The prepared samples were coded as follows, and their EIS spectra were subsequently analyzed [9]. **Control**-concrete containing neither polymer nor nanoparticles; **1A** - 0.025 wt% PAA; **3A** - 1 wt% PAA-Fe₃O₄; **4A** - 0.025 wt% PAA with 0.2 wt% each of FeCl₃ and FeCl₂; **5A** - 2 wt% PAA-Fe₃O₄; **12B** - concrete sample containing 0.1 wt% each of FeCl₂ and FeCl₃ salts.

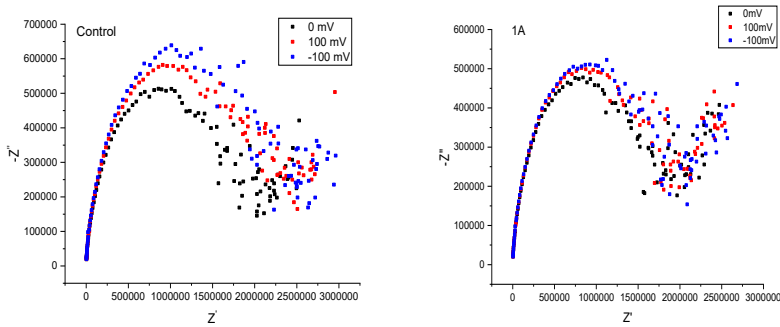


Figure 13. Electrical impedance spectra of the control concrete sample and of concrete containing 0.025 wt% PAA after 28 days of curing at a water-to-cement ratio of 0.5. Z' - $\text{Re}Z$ (Ω); Z'' - $(-\text{Im}Z)$ (Ω)[9]

As shown by the positions of the points in the figure, adding PAA to concrete leads to convergence of resistance values across various stress levels and results in an increase in Z'' beyond $2 \times 10^6 Z'$. The immobilization of polyelectrolyte PAA macromolecules within the cement matrix enables long-range particle connectivity and ensures electrical conductivity between them. Furthermore, the close alignment of the resistance points confirms that the calcium and aluminosilicates in the concrete are structured within the cavities of the PAA chains [9].

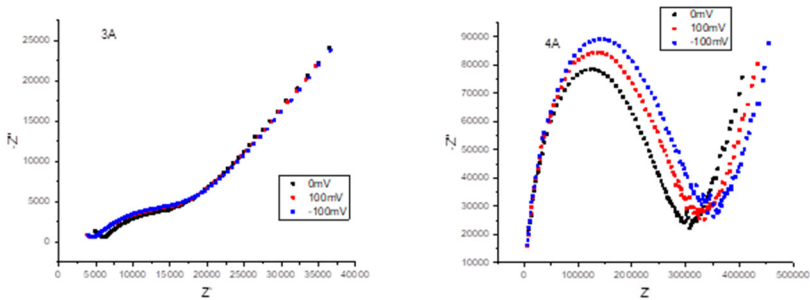


Figure 14. Electrical impedance spectra of concrete containing 1 wt.% PAA- Fe_3O_4 (3A) magnetite nanoparticles and of concrete containing 0.2 wt.% $\text{FeCl}_3/\text{FeCl}_2$ salts (4A) with 0.025 wt.% PAA. The water-to-cement ratio was 0.5, and the curing period was 28 days. Z' – $\text{Re}Z$, Ohm; Z'' – $(-\text{Im}Z)$, Ohm.[9]

The incorporation of a PAA-iron salt complex into the concrete composition affects both its mechanical properties and its electrical impedance spectra (EIS). The superior electrolyte properties of iron salts, compared with PAA and silicates, have a pronounced effect on the electrical resistance of the concrete (Figure 14, curve 4A).

CONCLUSIONS

1. Silver nanoparticles were synthesized in the presence of GA, a natural polysaccharide, and their properties were investigated. It was established that the reduction of silver ions proceeds more rapidly in the presence of sodium borohydride than with hydrogen peroxide. The obtained nanocomposites were characterized by X-ray diffraction (XRD), which indicated a nanoparticle size of approximately 32 nm. It was also established that no changes in the chemical structure of GA macromolecules occurred after the reduction process.[1]
2. Ag⁰ and Fe₃O₄ nanoparticles were obtained in the presence of GA, AG, Cht, and PEG, and their biological activities were investigated. Incorporating magnetite nanoparticles into the gel matrix was shown to increase the matrix's immobilization capacity for physiologically active substances. This enhances the potential of these metal nanoparticle-containing components for use in delivery systems for biologically active compounds. Compared with Ag⁰ nanoparticles, the gel containing magnetite nanoparticles exhibits higher capacity and degree of immobilization. This is attributed to magnetite not being in the zero oxidation state but being a mixed oxide containing Fe(II) and Fe(III), whose valence states enable coordination interactions.[2]
3. Natural and synthetic polymers (Cht, GA, AG, PVP, PEG, PAA), along with their Ag⁰ and Fe₃O₄ nanoparticle-containing polymer–colloidal systems and free levofloxacin, were impregnated into synthetic meshes measuring 1 cm² and weighing 30-40 mg, followed by biological testing with test cultures. It was established that the samples containing Ag⁰ and Fe₃O₄ nanoparticles exhibited the strongest bactericidal activity. These samples demonstrated the most pronounced bactericidal effect against Gram-negative (*P. aeruginosa* and *E. coli*) and Gram-positive (*S. aureus*) bacteria, with lysis zones ranging from 35 to 40 mm in diameter.[7]
4. The physical properties and feasibility of obtaining novel

materials with enhanced piezoelectric properties based on Ag0 nanocomposites stabilized in GA and PEG media were investigated. The sizes of silver nanoparticles stabilized in GA and PEG were found to range from 7 to 9 nm. Under comparable conditions, the electric current passing through the nanostructured samples was approximately five times higher than that of the other samples and showed a linear dependence over the voltage range of 0–50 V.[2]

5. Mass changes in concrete samples modified with PAA/Fe₃O₄ nanoparticles at concentrations of 1–2 wt.% relative to the cement mass were investigated in seawater and formation water. The results showed that, compared with concrete samples containing only PAA, the mass changes of PAA/Fe₃O₄ nanoparticle-containing samples in highly mineralized seawater strongly depended on the nanoparticle concentration. In addition, structural analysis of the samples was performed using Fourier-transform infrared (FTIR) spectroscopy and X-ray diffraction (XRD), enabling determination of the chemical states of the polymer and nanoparticles in the material. According to the results of electrochemical impedance spectroscopy (EIS), the resistance values of PAA/Fe₃O₄-modified concrete samples differed significantly from those of the control and PAA-containing concrete samples.[5]
6. The mechanical and physical properties of concrete prepared at water-to-cement ratios of 0.50, 0.45, and 0.40 and modified with high-molecular-weight PVP were investigated. At a water-to-cement ratio of 0.50, the water absorption of concrete containing 0.8 wt.% PVP increased from 15.65% to 20.71% after 14 days and from 16.74% to 21.67% after 28 days. The spread (slump flow) of the fresh cement paste increased from 20.54 cm to 23.12 cm. Meanwhile, the initial setting time increased from 147 to 170 min, and the final setting time from 238 to 261 min. Low-intensity additional peaks observed in the X-ray phase analysis (XRD) of concrete containing 1 wt.% PVP confirmed the polymer's immobilization within the cement matrix.[3]

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