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SYNTHESIS AND STUDY OF BINARY Ag-Cu NANOCOMPOSITES

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ABSTRACT

In the last decade, increasing attention has been paid to polymer-based nanocomposites incorporating noble metal nanoparticles, due to their broad application potential. Especially valuable are systems that combine two types of metal nanoparticles, such as silver and copper, which show enhanced performance in catalysis, biomedical engineering, and in the development of advanced chemo- and biosensors. These materials are also important for detecting environmental pollutants and diagnosing diseases. In this study, a binary Ag/Cu nanocomposite was synthesized by the simultaneous chemical reduction of silver and copper salts using hydrazine hydrate, with polyvinylpyrrolidone (PVP) acting as a stabilizing agent. The process was enhanced by ultrasonic treatment to ensure uniform particle formation. The resulting material was thoroughly characterized using scanning electron microscopy (SEM), X-ray diffraction (XRD), and energy-dispersive X-ray spectroscopy (EDS). Characterization confirmed the formation of crystalline silver and copper nanoparticles along with inclusions of copper(I) oxide (Cu_2O). SEM revealed spherical particles predominantly 20–30 nm in size, while elemental analysis showed silver and copper contents of 22.41% and 25.62% (atomic), respectively. The presence of PVP and ultrasonic energy contributed to a narrow size distribution and reduced aggregation, improving stability. The synthesized nanocomposite exhibits properties that make it suitable for use in electrochemical devices, pharmaceutical applications, and ecological monitoring systems. This work contributes to Sustainable Development Goal (Industry, Innovation, and Infrastructure**)** by supporting the creation of advanced nanomaterials for future technological solutions.

Keywords: Nanocomposite Materials, Silver and Copper Nanoparticles, Joint Reduction, Scanning Electron Spectroscopy, X-Ray Diffraction, Energy Dispersion Analysis.

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INTRODUCTION

In today's technological landscape, materials known as nanocomposites are gaining widespread application across numerous industrial sectors, primarily owing to the exceptional physicochemical characteristics imparted by the nanoscale dimensions and extensive surface area of the embedded particles. These engineered systems have become integral to the advancement of cutting-edge solutions in fields such as microelectronics, renewable energy, medical diagnostics and treatment, as well as ecological sustainability.[1-3] Nanocomposite materials have properties that cannot be achieved using traditional macroscopic components. The high chemical activity of nanoparticles, their ability to form new phases, as well as their unique optical, electrical, and magnetic characteristics, make them indispensable in the creation of high-tech products.[4-6] For example, increasing the strength and heat resistance of polymers through the introduction of nanocomponents allows them to be used in conditions of extreme temperatures and mechanical loads.[7,8] An analysis of the literature sources shows that a significant part of modern research in the field of nanotechnology is focused on the synthesis and study of the characteristics of individual metal nanoparticles and their oxides.[9-14] This is due to their widespread demand, for example, as catalysts, components of solar panels, sensors, and water purification systems.[15-19] However, agglomeration of individual nanoparticles, leading to a decrease in their activity, remains an important problem. The use of polymer or organic stabilizers partially solves this problem by increasing

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the stability and reducing the dispersion of nanoparticles.[20-23] An innovative approach to the synthesis of nanocomposites is the inclusion of two or more types of nanoparticles of various metals in their composition.[24-26] However, this very promising area in the creation of new nanocomposites has not yet been sufficiently studied. But it is precisely such composites that have the potential to create new materials with combined or synergistic properties, which makes them extremely in demand in science and industry.[27] For example, the combination of silver and copper nanoparticles makes it possible to create materials with antibacterial properties[28,29] that can be used in biomedical coatings[30-32] or water purification devices.[33-35] To date, many methods of obtaining nanocomposites are known, including chemical reduction of inert matrices such as carbon materials, silica gel, polymers, and other substrates.[36-38] The methods of nanoparticle synthesis are diverse: from physical evaporation and chemical deposition to hydrothermal and sol-gel processes [39-43]. Each method has its advantages and limitations, including cost, process complexity, and end product characteristics. Particularly noteworthy are nanocomposite systems incorporating stabilized nanoparticles of silver and copper, which exhibit a remarkable combination of antimicrobial efficacy, catalytic functionality, and sensitivity in detection applications, making them highly valuable across various technological and biomedical domains.[44,45] Such materials, as already mentioned, can be used in medicine, water purification, the creation of chemosensors and biosensors for the diagnosis of diseases, and environmental monitoring of environmental pollution. The prospects for further research include studying the effects of various stabilizers, combined synthesis methods, and testing nanomaterials under real-world operating conditions. The development of nanocomposite synthesis technologies with improved characteristics can significantly affect a wide range of industries, including energy, healthcare, and environmental protection, making this field of research one of the most promising and relevant in the modern world. The aim of this study is to obtain an Ag-Cu nanocomposite by chemical reduction of ions of these metals in the presence of a polymer stabilizer, which uses polyvinylpyrrolidone.

EXPERIMENTAL

Materials and Reagents

The following chemicals were used during the work:

- Silver nitrate salt ($AgNO_3$) is pure for analysis.
- Copper Sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$) — also rated as "pure for analysis".
- Hydrazine hydrate ($N_2H_4 \cdot H_2O$) is a commercial 35% solution manufactured by Khimtekh (Russia).
- An aqueous solution of ammonia (25%).

Bidistilled water with a specific electrical conductivity not exceeding 0.5×10^{-4} cm/m was used for all operations. The water was obtained in a glass bidistillator, which ensured its high purity.

Polyvinylpyrrolidone (C_6H_9NO) was used as a stabilizer (produced by Sigma-Aldrich (CAS 9003 39-8). This polymer has proven to be an effective agent that prevents the agglomeration of nanoparticles during synthesis and ensures their uniform distribution in the matrix.

Joint Synthesis of Metallic Silver and Copper Nanoparticles

The process of obtaining silver and copper nanoparticles was carried out by the method of joint reduction of their ions. A 100 ml flask was used in the experiments. The procedure included the following steps:

1. 25 ml of solutions of 0.02 N. $AgNO_3$ and 0.02 N. $CuSO_4$ were added to the flask.
2. A solution of hydrazine hydrate ($N_2H_4 \cdot H_2O$) was introduced drop by drop into the resulting mixture, which served as a reducing agent.
3. To enhance the reducing properties of hydrazine, the mixture was adjusted to pH 11 by adding an ammonia solution.

The prepared solution was exposed to ultrasonic irradiation at 40 kHz for a duration of 15 minutes, with the temperature maintained at 60 °C. For this purpose, a 1.8-liter DSA 50-SK ultrasonic bath was used. Chemical reduction reactions are described by the following equations:

$$4AgNO_3 + N_2H_4 + 4NH_4OH \rightarrow Ag + N_2 + 4NH_4NO_3 + 4H_2O \quad (1)$$

$$2CuSO_4 + N_2H_4 + 4NH_4OH \rightarrow 2Cu + N_2 + 2(NH_4)_2SO_4 + 4H_2O \quad (2)$$

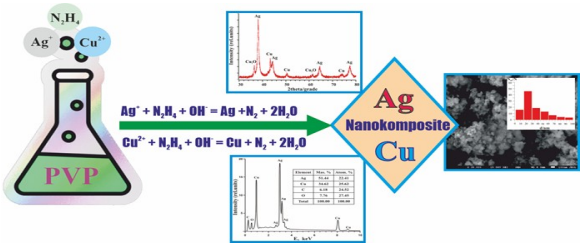
After completion of the reaction, the reduction products were separated by centrifugation at 6000 rpm. The resulting precipitate was dried in a vacuum dryer at 60 °C to remove moisture residues and stabilize the nanoparticles.

Instruments for the Determination of Nanocomposite Characteristics

To study the characteristics of the synthetic nanocomposite, the following analytical equipment was used:

- A scanning electron microscope (JEOL JSM-7800F) to analyze the morphology and size of nanoparticles. The high resolution of the microscope made it possible to visualize nanoparticles and study their distribution in the matrix.
- X-ray diffractometer (XRD-7000, Shimadzu) — for structural analysis. Technical specifications: anode Cu Kα; λ 1.544Å. This method made it possible to determine the crystal structure, phase composition, and sizes of crystallites.
- Elemental analysis was performed using a Rigaku X-ray Fluorescence Spectrometer (Japan). These analysis methods provided a comprehensive assessment of the synthesized nanocomposites, including their morphological and structural characteristics.

The general scheme of obtaining and studying a binary Ag-Cu nanocomposite is presented in a graphical abstract.



Scheme-1: The General Scheme of Obtaining and Studying a Binary Ag-Cu Nanocomposite

RESULTS AND DISCUSSION

Microstructural Analysis

A scanning electron microscope (SEM) image illustrating the morphology of the synthesized nanocomposite is presented in Fig.-1.

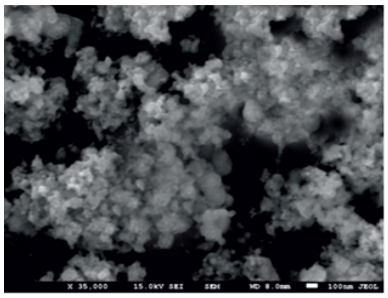


Fig.-1: SEM Image of the Synthesized Ag-Cu Nanocomposite

As can be seen from the image, the nanoparticles stabilized with polyvinylpyrrolidone (PVP) possess a predominantly spherical shape. The particle size varies between 20-30 nm, which is confirmed by the data of the histogram of the size distribution of the nanoparticle (Fig.-2).

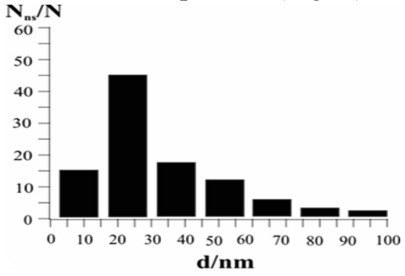


Fig.-2: Size Distribution of Nanoparticles in a Binary Ag - Cu Nanocomposite Synthesized in the Presence of Polyvinylpyrrolidone and an Ultrasound Bath

The analysis of micrographs showed that the use of PVP contributes to the uniform distribution of particles, preventing their agglomeration, which is important for further practical application of the nanocomposite.

Diffraction Studies

Figure-3 shows a diffraction pattern of a synthesized binary Ag-Cu nanocomposite. Analysis confirmed the presence of peaks corresponding to the diffraction angles characteristic of silver and copper (FCC), as well as additional peaks that indicate the formation of copper oxide (Cu_2O).

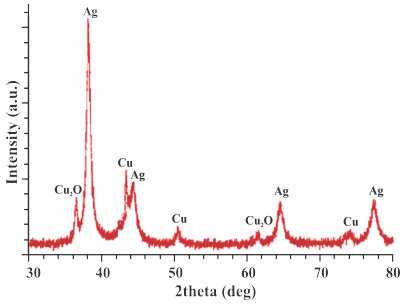


Fig.-3: Diffraction Pattern of a Binary Nanocomposite Ag-Cu Synthesized in the Presence of Polyvinylpyrrolidone

The appearance of peaks at angles 2θ = 36.48º and 2θ = 61.48º of associated with the presence of Cu_2O, which is formed due to the partial recovery of ions Cu^{2+} to Cu^{+}. These data are consistent with the literature data.[46-48] It should be noted that PVP functional groups containing nitrogen and oxygen atoms actively interact with metal ions, forming coordination bonds. PVP macromolecules are adsorbed on the surface of nanoparticles, preventing their further agglomeration and stabilizing the formed particles. Moreover, polyvinylpyrrolidone (PVP) is capable of functioning as a mild reducing agent, actively contributing to the synthesis of metal nanoparticles and their corresponding oxides. Table-1 shows calculations of the crystal lattice parameters of synthesized silver and copper nanoparticles based on the analysis of the diffraction pattern shown in Fig.-3.

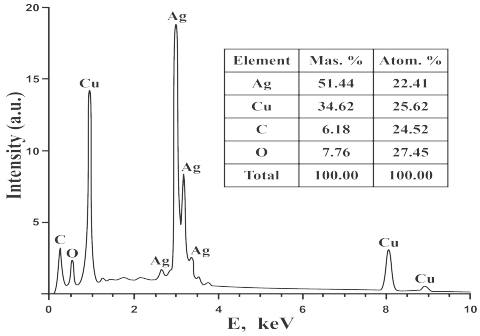
Table-1: Results of Processing Diffraction Pattern of Synthesized Nanoparticles

No.	Experimental data			Phase composition					
	2θ	d (Å)	I (a.u.)	Ag		Cu		Cu_2O	
				hkl	a (nm)	hkl	a (nm)	hkl	a (nm)
PVP									
1.	36.48	2.4752	24					111	0.428
2.	38.18	2.3626	100	111	0.409				
3.	43.42	2.0952	36			111	0.363		
4.	44.46	2.0928	31	200	0.410				
5.	50.18	1.8086	13			200	0.364		
6.	61.48	1.5208	10					220	0.428
7.	64.62	1.4478	23	220	0.408				
8.	74.24	1.2804	11			220	0.362		
9.	77,66	1,2327	24	311	0,408				

To identify the elemental composition of the binary nanocomposite, X-ray fluorescence (XRF) analysis was performed. The elemental distribution within the synthesized Ag-Cu nanocomposite was further examined using energy-dispersive X-ray spectroscopy (EDS), and the corresponding spectrum is presented in Fig.-4.

Analysis showed that the atomic content of silver (Ag) in the nanocomposite is 22.41%, and copper (Cu) is 25.62%. The obtained distribution data clearly indicate that the simultaneous chemical reduction process carried out in the presence of PVP leads to the formation of a nanocomposite material in which both metallic components are evenly dispersed throughout the matrix. The role of the stabilizer, Polyvinylpyrrolidone, plays a key role in the synthesis process. Its macromolecules with hydrophobic and hydrophilic parts are adsorbed on the surface of nanoparticles, creating spatial constraints for their further

growth and agglomeration. This helps to obtain nanoparticles with a given size and shape. In addition, PVP participates in the formation of primary metal clusters, which is important for the stability and functionality of the final nanocomposite.



Element	Mas. %	Atom. %
Ag	51.44	22.41
Cu	34.62	25.62
C	6.18	24.52
O	7.76	27.45
Total	100.00	100.00

Fig.-4: Energy Dispersion Spectrum and Elemental Composition of Ag-Cu Nanocomposite Synthesized in the Presence of PVP

CONCLUSION

In the course of the experimental studies, a binary nanocomposite containing nanoscale particles of silver and copper was successfully synthesized. The use of polyvinylpyrrolidone (PVP) as a stabilizer has demonstrated its important role in preventing particle aggregation and ensuring their uniform distribution. Due to the adsorption of PVP macromolecules on the surface of nanoparticles, it was possible to achieve not only spatial stabilization but also the participation of PVP in the formation of primary metallic nanoclusters. In addition, the use of ultrasonic treatment contributed to the uniform distribution of the stabilizer and accelerated recovery processes. The findings obtained from X-ray diffraction (XRD) analysis in combination with energy-dispersive spectroscopy (EDS) verified the incorporation of copper(I) oxide (Cu_2O) phases within the nanocomposite structure, alongside the anticipated silver and copper nanoparticles. The analysis of the elemental composition showed that the atomic content of silver is 22.41%, and copper is 25.62%. The data obtained indicate a uniform distribution of metals in the nanocomposite. The calculations of the crystal lattice parameters for the synthesized nanoparticles showed their correspondence to the values for bulk metals of silver and copper. This confirms the high degree of crystallinity and quality of the obtained material. Scanning electron microscopy revealed that the nanoparticles have a spherical shape, with predominant sizes in the 20-30 nm range. These characteristics provide the potential for using the nanocomposite in various practical applications. The resulting nanocomposite has prospects for creating electrochemical sensors designed for voltammetric measurements of the content of sulfur-containing compounds. Such sensors can be used in the analysis of environmental objects, food, and pharmaceuticals, as well as in assessing the quality of motor fuels for internal combustion engines. Thus, the results of the study confirm the possibility of effective synthesis of nanocomposites with specified properties, which opens up broad prospects for their use in science and technology.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

The present work is aligned with *SDG-9: Industry, Innovation and Infrastructure*. All the authors contributed significantly to this manuscript, participated in reviewing/editing, and approved the final draft for publication. The research profile of the authors can be verified from their ORCID IDs, given below:

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