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SYNTHESIS OF $SIO_2SNO_XCUO_Y$-BASED HYBRID NANOCOMPOSITE MATERIALS BY SOL–GEL TECHNOLOGY AND IN-DEPTH ANALYSIS OF THEIR PHYSICOCHEMICAL PROPERTIES

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Abstract: In this study, the aim was to synthesize $SiO_2SnO_xCuO_y$-based hybrid nanocomposite materials using sol–gel technology and to investigate, on a scientific basis, the main parameters influencing this process, such as the kinetics of hydrolysis and condensation, the nature of the solvent medium, pH indicators, and the initial molar ratios of metal oxide precursors. The course of the hydrolysis process in ethanol, isopropyl and isobutyl alcohol media based on tetraethoxysilane (TEOS), the formation of the gel phase, and the features of incorporation of SnO and CuO nanoparticles into the SiO_2 matrix were comparatively analyzed. The obtained results comprehensively revealed the factors affecting the structural and physicochemical stability of nanocomposite materials and substantiated the optimal synthesis conditions for creating metal-oxide-based hybrid systems. This work provides a scientific and technological basis for the application of metal-oxide nanocomposites in optical, catalytic and surface-related technologies.

Keywords: sol–gel method, tetraethoxysilane, $SiO_2SnO_xCuO_y$ nanocomposite, hydrolysis kinetics, metal oxide nanoparticles, ethanol, isopropyl alcohol, isobutyl alcohol, pH, hydrolysis–condensation, hybrid materials.

Introduction. Nanostructured composite materials, especially silicon dioxide matrices enriched with metal oxides, have become one of the most promising areas in recent decades for the development of nanocatalysts, optoelectronic devices, highly sensitive sensor elements and surface-active materials with high stability [1; 2; 3; 4]. SiO_2-based hybrid nanocomposite materials are distinguished by a large specific surface area, good chemical and thermal stability, controllable structure and the ability to uniformly distribute metal nanoparticles within the matrix [5]. In particular, the incorporation of SnO and CuO oxides into the SiO_2 matrix endows such composites with a number of functional properties such as photoactivity, catalytic selectivity and high activity in oxidation–reduction processes [6; 7; 8].

The sol–gel process is one of the most convenient and controllable methods for the synthesis of nanocomposite materials, ensuring molecular-level mixing of

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components and enabling control over the formation of porous structures [9; 10]. As a result of the hydrolysis and condensation of TEOS in an acidic medium, a sol (colloidal system) is formed, followed by the formation of a gel phase. In this process, parameters such as pH, solvent polarity, water content and catalyst concentration have a significant influence on the structure and mechanical strength of the gel [11].

In the course of this study, the effects of ethanol, isopropyl alcohol and isobutyl alcohol media on the hydrolysis–condensation process were comparatively investigated. These media differ in terms of TEOS hydrolysis rate, the degree of porosity of the resulting gel and the characteristics of nanoparticle incorporation into the matrix [12; 13; 14]. Uniform incorporation of metal oxide nanoparticles into SiO_2-based gels is a key factor determining the physicochemical properties of the final composite, because the distribution of SnO_x and CuO_y particles within the matrix governs parameters from catalytic activity to mechanical strength [15; 16].

The main scientific novelty of this study lies in the detailed comparison of the TEOS hydrolysis process in three different organic solvent media, the determination of precise limits for pH control, the scientific justification of the effect of increasing water content on the course of the process, and the evaluation of the efficiency of metal oxide nanoparticle incorporation into the SiO_2 matrix. In addition, all stages of the synthesis process were systematically improved in order to establish optimal technological parameters.

Main part. Sol–gel technology is one of the most widely used methods for the preparation of metal oxide nanocomposite materials. Compared to conventional high-temperature synthesis methods, it offers advantages such as lower energy consumption, molecular-level mixing of the constituent components and the ability to control the porosity of the gel structure [9; 10]. In the synthesis of $SiO_2SnO_xCuO_y$ composite systems, TEOS was chosen as the main precursor, since tetraethoxysilane allows the formation of a highly uniform structure through hydrolysis and condensation reactions [11].

During the incorporation of metal oxide nanoparticles into the SiO_2 matrix, the chemical nature of SnO and CuO compounds, their solubility in organic solvent media, their hydrolysis rates and interactions with TEOS hydrolysis products play an important role [12; 13]. SnO nanoparticles are known for forming stable bonds within the gel structure, whereas CuO nanoparticles form highly active centers, thereby enhancing the catalytic or photoactive properties of the composite, as reported in the scientific literature [6; 14].

In this research, the effect of three different solvents—ethanol, isopropyl alcohol and isobutyl alcohol—on the structure formation process was studied in depth. These solvents differ in polarity, molecular size and hydrophilic–hydrophobic balance, which directly affect the kinetics of TEOS hydrolysis [9]. Ethanol, due to its relatively high polarity, solvates water molecules effectively and accelerates the hydrolysis process. The polarity of isopropyl alcohol is lower than that of ethanol, but it supports the formation of a stable sol phase. Isobutyl alcohol

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has the lowest polarity and sharply increases the porosity of the gel, but reduces the hydrolysis rate.

According to recent reports in the scientific literature, even small changes in pH during TEOS-based sol–gel processes can significantly affect gel structure, particle size, degree of condensation and textural parameters (such as BET surface area) [10; 11]. For this reason, strict control of pH at around 2 was maintained in the experiments. If pH is too low, the gel condenses rapidly and the structure becomes non-uniform; if pH is too high, the hydrolysis process slows down and gelation is delayed [9].

Furthermore, the efficiency of metal oxide nanoparticle incorporation into the SiO2 matrix largely depends on the degree of gel porosity, the stability of the sol phase and the uniformity of hydrolysis–condensation reactions [13; 15]. During the introduction of SnO_x and CuO_y nanoparticles into the TEOS hydrolysis products, particular attention was paid to preserving their colloidal stability and preventing aggregation, for which continuous stirring of the reaction mixture was maintained under controlled conditions.

Methodology. The study was carried out on the basis of a three-stage experimental methodology. In the first stage, the TEOS hydrolysis process was compared in three different solvent media. For this purpose, TEOS:C2H5OH: H2O, TEOS: i-C3H7OH: H2O and TEOS: i-C4H9OH: H2O systems were prepared with the same initial molar ratio of 1:4:4. In each system, the water content was subsequently increased stepwise and the amount of solvent was reduced. The aim was to evaluate the effect of water on the hydrolysis process [9].

To activate the hydrolysis process, 0.1 M HCl was added dropwise as a catalyst to all reaction mixtures. Maintaining pH at around 2 ensured that the hydrolysis and condensation processes proceeded in a controlled and uniform manner. pH measurements were performed in real time using a laboratory pH meter. The mixtures were stirred at moderate speed on a magnetic stirrer for 4 hours. The appearance of turbidity was recorded as an indication of the onset of hydrolysis.

In the second stage, the prepared sol solutions were dried at 70°C for 12 hours. During drying, the degree of gel condensation increased, hydroxyl groups formed bonds with each other and a stable solid phase was formed. The resulting gel structures were aged under cooler conditions for 7 days, which ensured full development of the network structure [10].

The third stage consisted of preparing microscope slides and applying a gel layer onto them. The microscope glass slides were kept in 30% HNO_3 solution for 24 hours to activate –OH groups on the surface. This provided better adhesion of the gel to the glass surface. The slides were washed with alcohol and dried; then one-third of the surface was coated with the prepared sol–gel solution and dried for 24 hours. This method ensured a uniform spreading of the gel layer [11].

The incorporation of metal oxide nanoparticles was carried out at the intermediate stage of TEOS hydrolysis, which helped their stable distribution within the sol structure. The concentrations of SnO and CuO precursors were used at previously determined optimal ratios, since an excessive amount of metal oxides could disrupt the gel structure or intensify aggregation processes [12].

All prepared samples were evaluated by visual inspection, hardness assessment, uniformity of gel structure, mass loss during drying and resistance of the gel to cracking.

Analysis. In the synthesis of the obtained nanocomposite systems, the effects of the three organic solvents on the TEOS hydrolysis process were analyzed. It was found that hydrolysis in ethanol medium proceeded significantly faster than in isopropyl and isobutyl alcohol media. The high polarity of ethanol ensures good mixing with water, as a result of which H_2O molecules necessary for the hydrolysis reaction form a stable solvated environment around TEOS. This accelerates the kinetics of the hydrolysis–condensation process and promotes the formation of a stable gel. The literature also reports a higher hydrolysis rate of TEOS in ethanol medium [9; 10; 11].

The slowing down of the hydrolysis process in isopropyl alcohol medium is mainly explained by steric hindrance and partial hydrophobic character of the solvent. The relatively larger molecular size of isopropyl alcohol molecules partially interferes with the accumulation of water molecules around TEOS. As a result, the condensation process proceeds more slowly, although the structural stability of the gel is relatively high. This observation is also confirmed in the scientific literature, where it is reported that in isopropyl alcohol medium the porosity is sufficient, but achieving uniformity of the gel is more difficult.

Since isobutyl alcohol has the lowest polarity, it was observed that the TEOS hydrolysis process in this medium proceeded much more slowly. Water molecules in this medium do not become sufficiently solvated, and intermediate products formed during hydrolysis may remain partially uncondensed. However, the gel formed in isobutyl alcohol medium had a high porosity, which increases the potential of such materials to be used as adsorbents or catalyst supports.

An increase in the water content accelerated the hydrolysis process in all three solvent systems, as H_2O is the main hydrolyzing agent of TEOS. However, an excessive increase in water content led to non-uniformity of the gel structure and, in some cases, accelerated glass-like densification. The optimal water content was determined to be a 5–6 mol ratio, which is consistent with the results of previous studies.

The degree of incorporation of SnO and CuO nanoparticles into the gel structure was analyzed, and it was found that the best results were obtained when they were added at the intermediate stage of TEOS hydrolysis. In this case, metal oxide nanoparticles are incorporated at the molecular level into the sol structure and, during gel condensation, become stably located between branched Si–O–Si bonds. This increases the mechanical strength and stability of the composite material.

Analysis of the mass loss of the gel layers during drying, the gelation rate and surface stability showed that the composites synthesized in ethanol medium

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were the most stable and uniform. In contrast, in isobutyl alcohol medium, the surface porosity was high, but the structural stability of the gel was relatively lower.

Results. The following key scientific results were obtained during the study: Firstly, the TEOS hydrolysis process in ethanol, isopropyl and isobutyl alcohol media was comparatively analyzed, and ethanol was identified as the most suitable solvent. Its high polarity ensured that TEOS hydrolysis proceeded at an optimal rate and facilitated the formation of a uniform gel structure.

Secondly, an increase in the molar ratio of water accelerated the hydrolysis process, but when the water content exceeded 6 mol, non-uniform gel structures and disruption of the gelation process were observed. This confirms the sensitivity of the hydrolysis process to the amount of water.

Thirdly, it was determined that the optimal stage for incorporating SnO_x and CuO_y nanoparticles into the sol structure is the partial hydrolysis stage of TEOS. At this stage, nanoparticles bind stably to the SiO2 matrix and ensure the structural stability of the final composite.

Fourthly, the highest porosity gel structures were obtained in isobutyl alcohol medium. It was scientifically substantiated that such structures could exhibit high efficiency in adsorption and catalytic processes.

Fifthly, all obtained nanocomposite materials showed specific stability characteristics during drying and aging, indicating that they can be used in the future to develop highly efficient nanocatalysts, sensor elements and photoactive materials.

Conclusion. The influence of solvent nature, hydrolysis–condensation conditions, the stage of metal oxide nanoparticle incorporation and pH on the sol–gel synthesis of $SiO_2SnO_xCuO_y$ hybrid nanocomposite materials has been thoroughly studied. The hydrolysis of TEOS in ethanol medium was found to be the most suitable option, leading to the formation of a uniform and mechanically stable gel structure. Increasing the amount of water enhanced the activity of the hydrolysis reaction but, when excessive, caused disruption of the gel structure. The incorporation of metal oxide nanoparticles at the proper stage ensured their uniform distribution within the network structure of the SiO_2 matrix. The results of this study provide an important scientific and technological basis for the application of metal-oxide-based nanocomposites in optical, catalytic, adsorption and sensor technologies.

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