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# ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

# THE EFFECT OF TECHNOLOGICAL CONDITIONS ON THE PHYSICAL PROPERTIES OF Cd<sub>x</sub>Zn<sub>1-x</sub>S NANOPARTICLES

Speciality:	2222.01 – Physics and technology of
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#### **GENERAL CHARACTERISTICS OF THE WORK**

**Relevance of the topic and the degree of development.** Nanoparticles have unusual physicochemical properties compared to the bulk crystals of the group to which they belong. The production properties of nanomaterials can be controlled by directly controlling the formation reactions and interactions of nanoscale particles. Nano-sized materials have a wide range of applications due to their unique physical properties. Such materials are widely used in the development of new types of sensors, energy converters, the creation of new types of supercapacitors, etc.

Nanomaterials, especially, semiconductor nanoparticles due to the quantum effects, interesting optical, electrical, and other properties observed in them are considered promising materials for industrial application. Many transition metal chalcogenides (sulfides and selenides) are considered effective catalysts for the degradation of organic pollutants due to their bandgap energy and the spectrum coincides with the visible region of the solar spectrum. CdS and ZnS binary semiconductor compounds, with a bandgap value of 2,4 eV and 3,6 eV, respectively, are the most widely studied materials in terms of degradation of organic pollutants and application in photovoltaic devices. Subsequent studies have shown that the CdS compound becomes unstable as a result of radiation, damaging the photocatalyst and releasing toxic free cadmium ions into the environment, resulting in photocorrosion of the compound. Although ZnS is considered a useful material in corrosive environments, it has not been possible to use it as a catalyst in the visible light range due to its wide bandgap energy.

Thus, in order to overcome these shortcomings, it is necessary to synthesize binary sulfides in the form of hybrid materials or ternary semiconductor compounds. CdZnS nanomaterials are considered to be more efficient materials as broad-spectrum absorption and bandgap energy compounds than binary compounds.

Currently, binary and ternary semiconductor metal chalcogenides are synthesized in various methods in the form of CdS, ZnS, or  $Cd_xZn_{1-x}S$  compounds. These compounds are used in photonics and optoelectronics in the manufacture of photovoltaics, solar cells, electroluminescence devices for cathode ray tubes, emission displays, and photocatalysts as affordable materials.

Metal nanoparticles are unstable under normal conditions due to their high surface energy in the dispersed state. Different types of stabilizers (polar and non-polar) are used to prevent the agglomeration of such nanoparticles in colloidal systems. During the synthesis of samples, the concentration of initial reagents, temperature, pH value, etc. parameters should also be considered along with the reaction environment and technological conditions. Each of the selected parameters significantly affects both the structure and the physical and chemical properties of the formed nanoparticles.

**The object and subject of the research.** The  $Cd_xZn_{1-x}S$  nanoparticles obtained by sonochemical and SILAR methods using various stabilizers (PVA, 3MPA, and styrene) have been the object of this research. The main subject of this research is the study of the effects of technological conditions on the structure and physical properties of synthesized binary and ternary  $Cd_xZn_{1-x}S$  nanomaterials.

The aims and purpose of the research. The main purpose of the dissertation is the synthesis of  $Cd_xZn_{1-x}S$  nanoparticles in different technological conditions, and the study of their structure, optical and dielectric properties, surface morphology, and chemical composition, affecting crystallization environment parameters on the properties of formed nanoparticles.

The following *tasks* have been completed to achieve the set goal:

- the selection of initial reagents in the synthesis of binary and ternary semiconductor compounds and optimization of reaction parameters;
- sonochemical synthesis of nanoparticles Cd<sub>x</sub>Zn<sub>1-x</sub>S (x=0; 0,2; 0,4; 0,6; 1) by influence of inert gas;
- the synthesis of  $Cd_{0.4}Zn_{0.6}S$  nanoparticles using different stabilizers (PVA, 3MPA, and styrene) and a study of the effect of these stabilizers on the properties of the formed nanoparticles;
- synthesis of Cd<sub>x</sub>Zn<sub>1-x</sub>S (x=0; 0,01; 0,1; 0,2; 0,4) nanoparticles by co–sorption of cations by the SILAR method;
- the formation of  $Cd_{0.2}Zn_{0.8}S$  nanoparticles by the SILAR method at different reaction temperatures and the study of the role of the

formation temperature in the properties of the formed nanoparticles;

- study of the effect of thermal annealing on the structure and optical properties of samples obtained at room temperature.

**Research methods.** Structural properties for phase identification of samples X-ray diffraction (XRD) analysis; optical properties in the ultraviolet-visible (UV–Vis) region to record the absorption or transmittance spectra of the sample UV-Vis spectrophotometer (wavelength 190–1100 nm); electromagnetic in the infrared (IR) region IR spectroscopy (at frequencies 400–4000 cm<sup>-1</sup>) for the study of spectra of nanomaterials as a result of absorption or irradiation of rays; dielectric measurements for the study of temperature and frequency dependences of the dielectric constant (at frequencies  $10^2-10^7$  Hz); optical microscopy, which allows magnifications of the sample surface through visible light; scanning electron microscopy (SEM) to determine the shape and size of the formed particles; energy dispersive X-ray analysis (EDX) methods are used to determine the stoichiometric composition.

#### **Basic provisions for defense:**

1. Dependence of the stoichiometric composition of particles obtained during the formation of  $Cd_xZn_{1-x}S$  nanoparticles by the SILAR method on the type of used matrix and the formation temperature;

2. The effect of various stabilizers on the size and optical properties of obtained nanoparticles by sonochemical method;

3. The influence of reaction parameters (type and concentration of electrolyte solution) on the electronic spectra of nanoparticles;

4. The effect of different formation cycles on the distribution of ions along the layer thickness by the SILAR method;

5. The changes in the physical properties of  $Cd_xZn_{1-x}S$  nanoparticles depend on the technological conditions of SILAR and sonochemical synthesis methods.

#### Scientific innovations.

1. For the first time, different combinations of  $Cd_xZn_{1-x}S$  compounds have been synthesized in two different methods (sonochemical and SILAR) and their properties have been studied;

2. The investigated samples have been synthesized for the first time using various stabilizers (PVA, 3MPA, and styrene) and their effects on

the properties of the formed nanoparticles have been studied;

3.  $Cd_xZn_{1-x}S$  nanoparticles have been formed by the SILAR method at different reaction temperatures using a PVA polymer matrix, and the temperature dependence of the properties of the obtained materials has been studied;

4.  $Cd_{0.2}Zn_{0.8}S/PVA$  nanocomposites have been synthesized at different formation cycles by the SILAR method, and the volume distribution of cations has been studied.

The theoretical and practical significance of the research. The optimization of reaction parameters for the formation of  $Cd_xZn_{1-x}S$  nanomaterials by sonochemical and SILAR methods has been carried out. This approach can be used as a model for similar research. The fact that the obtained compounds have favorable structural and physical properties and that these properties can be controlled by various stabilizers makes them of practical importance.

**Approbation and application.** The main provisions of the dissertation and the results obtained have been discussed at a number of scientific conferences at the national and international levels and published in their materials:

-5<sup>th</sup> International Conference "Nanotechnologies" (Tbilisi, 19–22 November 2018);

 $-1^{st}$  International science and engineering conference (Baku, 29–30 November 2018);

-Second international scientific conference of young scientists and specialist Multidisciplinary approaches in solving modern problems of fundamental and applied sciences, ANAS-75, (Baku, 03-05 March 2020);

 $-2^{nd}$  International Conference on Light and Light–Based Technologies (Ankara, 26–28 May 2021).

Name of the organization where the dissertation work is executed. The dissertation work has been carried out in the Scientific Laboratory of Nanoresearch at Baku State University.

**Structure, volume and main content of dissertation work.** The dissertation covers a total of 161 pages, including 53 figures, 8 tables, introduction, 4 (four) chapters, results, list of 158 references used, abbreviations and a list of symbols. The volume of the dissertation (with the

exception of gaps and pictures in the text, tables, graphs, appendices and list of reference) -172923 characters (introduction -11122, Chapter I -51076, Chapter II -29913, Chapter III -59077, Chapter IV -19049, result -2686 characters).

#### CONTENT OF THE DISSERTATION WORK

In the introduction, the relevance of the topic is substantiated, information has been given about the goals and objectives of the work, the object and subject of research, research methods, scientific novelty, practical significance, and other important characteristics of the work.

**In the first chapter** a review of the literature on the structure, physical and chemical properties, and application field of CdS, ZnS, CdZnS nanoparticles synthesized by sonochemical and SILAR methods. In this chapter, the properties of the synthesized materials change as a result of the influence of various parameters, as well as the possibility of variation of their properties depending on these parameters, the reaction parameters optimized in terms of potential application are identified.

**The second chapter** is devoted to the synthesis methods of  $Cd_xZn_{1-x}S$  nanoparticles. The selected initial reagents, their properties, and parameters are noted here, and the technology used by two methods (SILAR and sonochemical methods) in the obtaining of samples is described in detail. The  $Cd_xZn_{1-x}S$  based nanoparticles have been synthesized in a PVA polymer matrix by the SILAR method using different initial reagents at different cycles. So that, two pairs of cation sources have been used in the reaction for the precipitation of nanoparticles: the first (I) cation sources – cadmium acetate dihydrate (Cd(CH<sub>3</sub>COO)<sub>2</sub>·2H<sub>2</sub>O) and zinc acetate dihydrate (Zn(CH<sub>3</sub>COO)<sub>2</sub>·2H<sub>2</sub>O), the second (II) cation sources–cadmium nitrate dihydrate (Cd(NO<sub>3</sub>)<sub>2</sub>·2H<sub>2</sub>O) and zinc sulfate heptahydrate (ZnSO<sub>4</sub>·7H<sub>2</sub>O). The samples have been synthesized at room temperature for 5 cycles. The sorption time has been chosen to be 40 minutes in all experiments.

The reaction has been carried out at 1, 2, and 5 growth cycles to determine the physical changes that occurred in the synthesized

samples, as well as the effect of the cycle number on the structure of the compounds. Furthermore, the reaction has been carried out at 3 temperatures  $-25^{\circ}$ C (room temperature), 40°C, and 65°C to determine the effect of the growth temperature on the physical properties of the obtained materials.

Besides studying the effect of temperature on the formation process, the effect of thermal annealing on the properties of samples synthesized at room temperature has been studied. For this purpose, the samples have been thermal annealed in a vacuum oven at 60°C and 120°C for 5 hours. The properties of the obtained samples have been investigated and compared with the properties of the initial samples.

Furthermore, the information about the sonochemical method and the formation process of nanoparticles obtained by this method utilizing various stabilizers is described. Thus,  $Cd_xZn_{1-x}S$  nanoparticles of different composition (x=0; 0,2; 0,4; 1) have been synthesized by sonochemical method. The aim was to study the effect of stabilizers on the properties of formed nanoparticles.

Thus, different compounds have been synthesized with the same stabilizers. At the same time, the same compositions have been synthesized with different stabilizers.  $Cd_xZn_{1-x}S$  nanoparticles have been synthesized in various combinations for different values of x.

To study the interaction of different stabilizers (PVA, 3MPA, and styrene) with nanoparticles,  $Cd_{0.4}Zn_{0.6}S$  has been synthesized with these stabilizers. The percentage of stabilizers in the solution has been selected as 2% and 7%. All reactions have been carried out at room temperature for 2 hours and the reaction medium has been continuously exposed to nitrogen gas to prevent oxidation.

The reaction in different ratios was as follows:

$$Cd^{2+}+Zn^{2+}+S^{2-}\leftrightarrow CdS/ZnS/Cd_{x}Zn_{1-x}S.$$
(1)

Detailed information about of XRD, UV–Vis spectrophotometer, IR spectroscopy, optical microscopy, SEM, EDX investigation methods is described at the end of the chapter.

In the third chapter structural and physical properties of the obtained samples have been devoted to research. Initially, the peaks  $2\theta=26^{\circ}$ ,  $37^{\circ}$ ,  $43^{\circ}$  observed from the XRD of the CdS/PVA thin film obtained by the SILAR method were attributed to the CdS formed in the hexagonal phase (100), (102) and (110), which correspond to Miller indices.

Figure 1.1 shows the XRD pattern of  $Cd_xZn_{1-x}S/PVA$  samples synthesized by the I cation source. The peaks observed at  $2\theta \le 22^{\circ}$  belong to the PVA polymer matrix. The peaks observed at  $2\theta = 26^{\circ}$  and  $43^{\circ}$  which confirms the formation of the  $Cd_xZn_{1-x}S$  crystal with a hexagonal structure. These peaks correspond to the (002), (110) Miller indices. As the concentration of  $Cd^{2+}$  ions in the solution increases, the diffraction peaks shift to smaller angles. This shift is explained by Bragg's law.



**Figure 1.** XRD patterns of  $Cd_xZn_{1-x}S/PVA$  nanocomposites synthesized using I and II cation sources.  $1 - Cd_xZn_{1-x}S/PVA$  (x=0,01(a), 0,1(b), 0,2(c), 0,5(d), 0,8(e)) and  $2 - Cd_xZn_{1-x}S/PVA$  (x=0,1(a), 0,2(b))

The main peaks in the XRD pattern of  $Cd_xZn_{1-x}S/PVA$  (x=0,1, 0,2) nanocomposites synthesized by the II cation source correspond to  $Cd_xZn_{1-x}S$  with a hexagonal structure (figure 1,2). Other peaks in the range of  $2\theta < 22^{\circ}$  are related to the PVA polymer matrix.

At the same time, the effect of the number of cycles on the stable reaction parameters on the physical properties of the formed nanomaterials has been studied. In this case, the weakly observed peak in the sample synthesized in 1 cycle has been attributed to the PVA polymer. Diffraction peaks for  $Cd_xZn_{1-x}S$  nanoparticles have been observed more intensively in the 2 cycled synthesized samples. Experiments have shown that as the number of cycles increases, the particles size and the degree of crystallization is decreased, half-width of the diffraction peaks are decreased.

The XRD pattern of  $Cd_xZn_{1-x}S/PVA$  compounds obtained by sonochemical methods is shown in Figure 2.



**Figure 2.** XRD patterns of sonochemically synthesized nanocomposites.  $1 - CdS / PVA (x=1), 2 - Cd_xZn_{1-x}S/PVA (a) x=0, b) x=0,2, c) x=0,4$ 

In figure 2.1. are shown the XRD patterns of the CdS/PVA nanocomposite  $(20^{\circ} \le 20 \le 60^{\circ})$ . Here, the peaks observed at  $20 = 25,87^{\circ}$ , 43,10°, 53,07° confirms the formation of CdS nanoparticles in the cubic phase. The observed peaks correspond to the Miller indices (111), (200), (311), respectively. Other observed peaks are attributed to the CdS combination formed in the hexagonal phase. The 3 main peaks in the XRD pattern of the ZnS nanoparticles (figure 2.2(a)) correspond to ZnS with a cubic phase formation of 27,89°, 48,18°, and 58,38°. These peaks correspond to the (111), (220) and (311) Miller indices, respectively. The main peaks  $2\theta=22,79^{\circ}$ ,  $46,81^{\circ}$ ,  $55,76^{\circ}$ , and  $2\theta=27,40^{\circ}, 45,82^{\circ}, 54,12^{\circ}$  in the XRD results of the Cd<sub>0.2</sub>Zn<sub>0.8</sub>S and Cd<sub>0.4</sub>Zn<sub>0.6</sub>S nanoparticles belong to the hexagonal phase of the  $Cd_xZn_{1-x}S$  crystal structure. The Miller indices of the observed peaks are (101), (103) and (004), respectively. Based on the results of XRD, it can be noted that as the ratio of  $Zn^{2+}/Cd^{2+}$  ions in  $Cd_xZn_{1-x}S$  nanoparticles increases, all peaks shift to smaller angles. In the solution,

when x=0,  $Cd_xZn_{1-x}S$  nanoparticles belong to ZnS nanoparticles formed in the cubic phase, at values of x equal to 0,2 ( $Cd_{0.2}Zn_{0.8}S$ ) and 0,4 ( $Cd_{0.4}Zn_{0.6}S$ ), it was determined that CdZnS nanoparticles formed in the hexagonal phase. The Debye–Sherrer formula has been used to determine the size of the formed nanoparticles. The nanoparticle size for CdS/PVA nanocomposite was calculated to be 3,8 nm, 3,3 nm for ZnS/PVA nanocomposite, 2,25 nm for Cd<sub>0.2</sub>Zn<sub>0.8</sub>S/PVA nanocomposite, and 2,04 nm for Cd<sub>0.4</sub>Zn<sub>0.6</sub>S/PVA nanocomposite.

The effect of the stabilizers used on the physical properties of these nanoparticles has been studied by synthesizing the  $Cd_{0.4}Zn_{0.6}S$  compound with various stabilizers. The Miller indices of these peaks observed in this case (101), (103), (201) correspond to the parameters of the crystal lattice in the hexagonal phase of  $Cd_xZn_{1-x}S$ . The particles size of these nanocomposites have been calculated and determined to be 7 nm for  $Cd_{0.4}Zn_{0.6}S/PVA$ , 9 nm for  $Cd_{0.4}Zn_{0.6}S/styrene$  and 5 nm for  $Cd_{0.4}Zn_{0.6}S/3MPA$ .

Thus, during the synthesis of nanoparticles using different stabilizers under the same technological conditions, they all formed the same structure. However, depending on the type of stabilizer, the particle size has changed. This is explained by the change in the free Gibbs energies of these particles as a result of the particle-stabilizer interaction, depending on the type of stabilizer. That is, stabilizers used to prevent the growth of nanoparticles have different interactions with the nanoparticles, depending on their physical parameters. This has led to differences in the sizes of the nanoparticles. At the same time, the energy states of the nanoparticles and stabilizers changed as a result of the surface tension force, which in turn led to a change in the size of the resulting nanoparticles.

This chapter also provides spectroscopic studies of samples. The properties of the  $Cd_xZn_{1-x}S/PVA$  nanocomposite have been studied based on absorbance and transmission spectra with a UV–Vis spectro-photometer. The values determined for the bandgap value of the samples obtained by the SILAR method are given in table 1. Here, the  $E_g$  value is shown for samples obtained using different cation sources, at different values of cation sources –  $Cd^{2+}/Zn^{2+}$ , and by changing the number of cycles.

The  $E_g$  values of the samples obtained by the sonochemical method are given in table 2.

<b>Table 1</b> . Bandgap values of $Cd_xZn_{1-x}S/PVA$ nanocomposites obtained by the SILA	٩R
nethod	

Samples	Number of cycles	Source of cation	Bandgap, Eg(eV)
CdS/PVA	5	Ι	2.45
Cd <sub>0.01</sub> Zn <sub>99.99</sub> S/ PVA	5	Ι	3.21
Cd <sub>0.1</sub> Zn <sub>0.9</sub> S/ PVA	5	Ι	3.15
Cd <sub>0.1</sub> Zn <sub>0.9</sub> S/ PVA	5	II	3.10
Cd <sub>0.2</sub> Zn <sub>0.8</sub> S/ PVA	5	Ι	3.05
Cd <sub>0.2</sub> Zn <sub>0.8</sub> S/PVA	5	II	3.14
Cd <sub>0.2</sub> Zn <sub>0.8</sub> S/ PVA	1	Ι	3.25
Cd <sub>0.2</sub> Zn <sub>0.8</sub> S/ PVA	2	Ι	3.13
Cd <sub>0.5</sub> Zn <sub>0.5</sub> S/ PVA	5	Ι	3.00
Cd <sub>0.8</sub> Zn <sub>0.2</sub> S/ PVA	5	Ι	2.80

Table 2. Eg values of Cd<sub>x</sub>Zn<sub>1-x</sub>S nanoparticles obtained by sonochemical method

Samples	<b>E</b> <sub>g</sub> , (eV)
CdS/PVA (2%)	2,25
ZnS/PVA (2%)	3,80
Cd <sub>0.2</sub> Zn <sub>0.8</sub> S/PVA (2%)	2,80
Cd <sub>0.4</sub> Zn <sub>0.6</sub> S/PVA (2%)	2,45
Cd <sub>0.4</sub> Zn <sub>0.6</sub> S/PVA (7%)	2,45
$Cd_{0.4}Zn_{0.6}S/styrene$ (7%)	2,55
Cd <sub>0.4</sub> Zn <sub>0.6</sub> S/3MPA (7%)	2,80

According to the results, it can be noted that the bandgap value varies depending on the type of used stabilizers. This change may be due to the following factors:

1. The XRD view shows that the largest particle size (9 nm) has been formed when using a styrene stabilizer. The size of the particles synthesized with PVA stabilizer has been 7 nm, and the smallest size (5 nm) existed when using mercaptopropionic acid. The difference obtained in the particles size are depend the type of used stabilizer, whether it is polar or non-polar, its molecular weight, and its spatial structure. It is assumed that, the interaction between polar molecules and formed nanoparticles is greater than the interaction between non-polar molecules and nanoparticles. Therefore, smaller particles have been obtained when using polar stabilizers. Thus, nanoparticles' fast interaction with broken bonds on the surface. The change in particle size depending on the type of stabilizing material also led to a change in the bandgap value. The nanoparticles synthesized with mercaptopropionic acid were the smallest particles, with a size of 5 nm and a maximum bandgap value (2,8 eV). The  $E_g$  value of nanoparticles synthesized with PVA and styrene stabilizers was 2,45 eV and 2,55 eV, respectively.

2. The second factor is the effect on the optical properties of nanoparticles of changes in the potential energy of their charge carriers and the change in their effective mass as a result of this change. In this case, the Schrödinger equation of the system can be written as follows<sup>1</sup>:

$$-\frac{\hbar^2}{2m^*} \left[ \frac{\partial^2}{\partial x^2} + \frac{\partial^2}{\partial y^2} + \frac{\partial^2}{\partial z^2} \right] \Psi(r) + V(r)\Psi(r) = E_r \Psi(r).$$
(2)

As can be seen from (2), the electronic spectrum of carriers depends on the parameter V (r). Thus, changes in the interaction of carriers with the environment also lead to changes in the electronic spectrum. This, in turn, contributes to the change in the bandgap value during the synthesis of nanoparticles with various stabilizers.

To study the effect of the medium on the properties of nanoparticles, CdS nanoparticles obtained by sonochemical methods have been placed in different concentrations of three different electrolyte solutions. Based on the absorption spectra, the bandgap value of these samples has been calculated and given in table 3.

Concentrations / Eg (eV)				
Electrolyte solutions	0,01 M	0,1 M	1 M	2 M
CdS+Cd(CH <sub>3</sub> COO) <sub>2</sub>	3,7	3,3	2,3	2,1
CdS+Cd(NO <sub>3</sub> ) <sub>2</sub>	2,95	2,8	2,75	2,7
CdS+NaCl	3,6	2,97	2,65	2,5

Table 3.  $E_{\rm g}$  value of CdS nanoparticles placed in electrolyte solutions of different concentrations

<sup>&</sup>lt;sup>1</sup>Paolo D.S., Looking at the Schrödinger Equation For Nanotechnology // International Journal of Engineering Science and Innovative Technology, 2013, v.2, p.418-410.

As shown in table 3, the bandgap of nanoparticles depends on the nature of the medium. From the results obtained, it became clear that the bandgap value decreased with the increasing concentration of electrolyte solutions. Thus, when CdS was added to a Cd(CH<sub>3</sub>CO<sub>2</sub>)<sub>2</sub> solution, the bandgap value decreased to some extent as the electrolyte solution density increased. However, in Cd(NO<sub>3</sub>)<sub>2</sub>and NaCl solutions, the bandgap value for CdS has been significantly reduced. This is explained by the difference in the solubility of CdS nanoparticles in electrolyte solutions depending on the anionic component of the electrolyte solution. It has been shown that changes in the medium can change the bandgap value from 2,25 eV to 3,6 eV. As the concentration increases, the ionic density of the particles near the surface changes, which leads to a change in the potential energy of the charge carriers in the nanoparticles. Changes in potential, in turn, have led to changes in the energy spectrum of carriers. As a result of the measurements, it has been found that in some cases the bandgap of the nanoparticle is 2 times smaller than that of the crystals (2,42 eV)<sup>2</sup>. As noted, changes in the nature of the energy spectrum have led to changes in the effective mass of carriers.

At the same time, optical images of cross-sections of  $Cd_{0.2}Zn_{0.8}S/PVA$  nanocomposites synthesized at different cycles have been shown in figure 3.



**Figure 3.** Optical microscopy images of  $Cd_{0.2}Zn_{0.8}S/PVA$  obtained using the I cation source in different cycles (a) -2 cycles and (b) -5 cycles.

<sup>&</sup>lt;sup>2</sup>Ankan B., Meher S. R., Deepak K.K., Electronic and band structure calculation of wurtzite CdS using GGA and GGA+U functionals // Condensed Matter: Materials Science, 2021, p.1-6.

Figure 3(a) shows optical images of  $Cd_{0.2}Zn_{0.8}S/PVA$  nanocomposites synthesized in 2 cycles and figure 3(b) in 5 cycles. Here, the optical density of the distribution of ions across the volume of the layer depends on the number of cycles. It has been found that the ions are more evenly and homogeneously distributed at 2 cycles along the thickness of the layer. As the number of cycles increased, the distribution of particles within the volume became heterogeneous. The reason for this heterogeneity is a combination of several facts:

1) Difference of thermodynamics situation of particles on the surface and volume of the layer: Here, there is only interaction between the nanoparticles formed along the volume within the layer. On the surface, nanoparticles interact with the environment in addition to themselves. For this reason, the difference in energy values on the surface and in volume means that the surface and volume particles have different Gibbs energy values. Therefore, the location of nanoparticles on the surface and in volume becomes heterogeneous over time.

2) Decreasing of the ability of ions to penetrate as the number of cycles increases: Because the PVA matrix is a porous structure, ions are sorbed at sorbtion centers (porous) during the SILAR synthesis method. In the early stages of the synthesis process, the centers on the surface and in the volume of the layer are captured by ions. The subsequent growth process occurs mainly in the area close to the surface. As the number of cycles increases, the centers on the surface are completely filled, and the surface is completely covered by these ions. Thus, in the following periods, the sorbed ions could not penetrate into the layer and accumulate on the surface. It has been considered that the heterogeneity of the volume distribution of ions could be due to the decrease in the ability of ions to penetrate into the layer with the increasing number of cycles.

3) As a result of interaction of nanoparticles with a double electric *layer*: Nanoparticles interact with the polymer matrix to form a double electric layer. Charged particles with and without a dipole moment in the medium interact with charged particles of the same name in volume. This interaction results in the migration of particles. Absorption in the sorption centers within the polymer matrix has a physical nature (Van der Waals forces). If the sorption is based on physical absorption,

the interaction energy of the particles inside the matrix is also not very large. In this case, the interaction with the environment can lead to the easy migration of these particles. The optical density of the surface of nanoparticles depends on the density of the solution, which leads to a change in the color of the surface of the polymer matrix on the surface.

From the SEM images of  $Cd_{0.2}Zn_{0.8}S/PVS$  nanocomposites, it has been determined that the particle size of the samples synthesized in 2 periods is in the range of 7,4-12,8 nm, and the particle size of the sample synthesized is in 5 periods is in the range of 8,89-22,7 nm. This is because as the number of cycles increases, the formed particles agglomerate to form larger particles.

The Cd<sub>0.2</sub>Zn<sub>0.8</sub>S/PVA nanocomposites obtained at 2 and 5 formation cycles have been investigated by EDX to determine the stoichiometric composition of the investigated samples (figure 4). Thus, the atomic ratios of Zn/Cd in the compounds obtained in 2 cycles were 1,3, and in 5 cycles this ratio was 2,3. However, at the beginning of the reaction, this ratio had to be equal to the theoretically calculated value (4). This is because the rate of particle formation is weak at room temperature and that the ratio depends on the number of cycles. It has been found that the particles haven't formed completely in samples with a small number of cycles. As the number of cycles increases, the amount of sorbed nanoparticles is has been increased, too. Based on



Figure 4. Elemental analysis of  $Cd_{0.2}Zn_{0.8}S/PVA$  nanocomposites: (a) – 2 cycles and (b) – 5 cycles.

the results of the element analysis, the peaks of the elements Cd, Zn, and S in the spectrum confirmed the formation of  $Cd_{0.2}Zn_{0.8}S/PVS$  nanocomposites. The peaks of the carbon and oxygen elements are attributed to the polymer matrix.

From the element analysis, the atomic percentages of cadmium, zinc, and sulfide ions are 3,7, 5,5, and 8,5, respectively. This result indicates that the atomic percentage of elements in the  $Cd_{0.4}Zn_{0.6}S/3MPA$  compound is approximately equal to the amount of reagents selected in the first stage of the reaction.

Dielectric properties of the samples obtained by the SILAR method have been measured in a variable electric field, and at different - 25°C, 60°C, and 100°C temperatures. From the frequency dependence curve of the dielectric constant at all temperatures, it has been determined that the value of the dielectric constant of the system decreased with the entry of  $Zn^{2+}$  ions into the CdS/PVA system. As a rule, the increase in dielectric constant in the PVA system with increasing temperature is due to the increased mobility of dipoles under the influence of temperature and their ability to easily self-align with the external field. At the same time, according to the Guy-Chapman model, an interfacial layer is formed between the surface of the nanoparticle and the polymer, which creates a double electric layer with a thickness of about 1 nm, which is closely related to inorganic and organic substances. The thickness of the double electric layer is directly proportional to the temperature. That is, as the temperature increases, the thickness between the double electric layers also increases. Since the value of the dielectric constant depends on the parameters of the environment, the thickness of the intermediate layer can also be affected.

For pure PVA, the value of the dielectric constant at 1 kHz at  $27^{\circ}$ C is shown as  $8,03^{3}$ . The changes in dielectric constant with the introduction of various metal complexes into the PVA system are explained by the anion and O – H groups effects of these particles on the polymer. The phenomenon of polarization in PVA systems based on metal particles can also be influenced by the ionic radii of the metal particles.

<sup>&</sup>lt;sup>3</sup>Prabha K. and Jayanna H. S. Studying the Frequency Dependence of Dielectric Properties of Gamma Irradiated PVA (1-x) PSx Polymer Blends // Open Journal of Polymer Chemistry, vol. 5, pp.47–54, 2015.

In the  $Cd_xZn_{1-x}S$  system under consideration, the ionic radius of  $Cd^{2+}$  ions is greater than the radius of  $Zn^{2+}$  ions. For  $Cd^{2+}$ , this value is approximately r=95 pm, and for Zn, it is r=75 pm. Due to the large ionic radius,  $Cd^{2+}$  ions can be oriented faster under the influence of an external electric field than  $Zn^{2+}$  ions. Due to this,  $Cd^{2+}$  ions also contribute to the orientation of the hydroxyl groups of the PVA polymer and their easy polarization.

Frequency dependence of dielectric constant of  $Cd_xZn_{1-x}S$ -based nanoparticles obtained by sonochemical methods at different temperatures (28°C, 40°C, 50°C, 60°C, 70°C, 80°C, 90°C, 100°C) have been investigated. As can be seen from the frequency dependence graph of the dielectric constant of CdS nanoparticles, the value of the dielectric constant decreases with increasing frequency for all samples.

In the fourth chapter the effect of temperature on the properties of synthesized  $Cd_xZn_{1-x}S$  compounds has been studied. For this purpose,  $Cd_{0.1}Zn_{0.9}S/PVA$ ,  $Cd_{0.2}Zn_{0.8}S/PVA$  and  $Cd_{0.8}Zn_{0.2}S/PVA$  samples obtained by the SILAR method at room temperature have been thermally annealed in a vacuum furnace at 60°C and 120°C for 5 hours.

The samples retained their structure when annealed to  $60^{\circ}$ C, and relative amorphization was observed at 120°C. As a result of the thermal annealing of samples, the diffraction angles in the Cd<sub>x</sub>Zn<sub>1-x</sub>S combination shifted to slightly smaller angles with an increasing value of x, and new peaks have been observed in the diffraction pattern. The sharp peak observed at 2  $\theta$ =18,5° is a Cd(OH)<sub>2</sub> peak and corresponds to the Miller index (001). The purely trigonal  $\beta$ –Zn(OH)<sub>2</sub> phase is also responsible for this peak. This is explained by the formation of the zinc hydroxide group along with the cadmium hydroxide group. The size of the nanoparticles was calculated based on the XRD results using the Debye–Scherrer formula and the bandgap values are given in table 4.

Table 4 revealed that the particle size of the samples obtained at room temperature differed slightly from the particle size of the thermal annealed of these samples at 60°C. Thus, the calculated particle size for the Cd<sub>0.1</sub>Zn<sub>0.9</sub>S/PVA compound at room temperature was 3,2 nm, and the particle size after 60°C was 3,7 nm. After 120°C thermal annealing of this sample, it increased by about 2 times and was at 7,8 nm. In the Cd<sub>0.2</sub>Zn<sub>0.8</sub>S/PVA compound, the size of the particles under

Sample	Temperature	R, nm	E <sub>g</sub> ,eV
Cd <sub>0.1</sub> Zn <sub>0.9</sub> S/PVA	synthesis at room temperature ( <i>sample A</i> )	3,2	3,15
Cd <sub>0.1</sub> Zn <sub>0.9</sub> S/PVA	termal annealing T=60°C	3,7	4,51
Cd <sub>0.1</sub> Zn <sub>0.9</sub> S/PVA	termal annealing T=120°C	7,8	3,33
Cd <sub>0.2</sub> Zn <sub>0.8</sub> S/PVA	synthesis at room temperature (sample B)	3,3	3,05
Cd <sub>0.2</sub> Zn <sub>0.8</sub> S/PVA	termal annealing T=60°C	3,3	3,15
Cd <sub>0.2</sub> Zn <sub>0.8</sub> S/PVA	termal annealing T=120°C	7,0	3,14
Cd <sub>0.2</sub> Zn <sub>0.8</sub> S/PVA	synthesis at T=40°C (sample D)	4,3	3,14
Cd <sub>0.2</sub> Zn <sub>0.8</sub> S/PVA	synthesis at T=65°C (sample E)	2,6	2,37
Cd <sub>0.8</sub> Zn <sub>0.2</sub> S/PVA	room temperature( <i>sample C</i> )	3,4	2,70
Cd <sub>0.8</sub> Zn <sub>0.2</sub> S/PVA	termal annealing T=60°C	3,5	2,77
Cd <sub>0.8</sub> Zn <sub>0.2</sub> S/PVA	termal annealing T=120°C	11,9	2,72

Table 4. The average size and band gap value of nanocomposites

normal conditions and after thermal annealing remained at 3,3 nm, which showed no change. After 120°C thermal annealing of this compound, the particle size was calculated to be 7 nm. At room temperature and after thermal annealing at 60°C, the particle size of the  $Cd_{0.8}Zn_{0.2}S/PVA$  compound was calculated to be 3,4 nm and 3,5 nm, respectively. Similarly, a significant change in particle size was observed after thermal annealing at 120°C and particle size was calculated as 11,9 nm.

In all samples, the reason for the increase in particle size at a temperature of 120°C was the increase in the mobility of particles under the influence of heat inside the polymer matrix and the aggregation of nanoparticles, ie the occurrence of agglomeration. Another reason was the softening of the polymer matrix at this temperature and the increase in the intermolecular distance.

These factors lead to an increase in the diffusion coefficient of nanoparticles in the polymer matrix, resulting in an increase in particle size. The reason for the sharp increase in particle size in samples with high concentrations of  $Cd^{2+}$  ions is the high ionic radius of  $Cd^{2+}$  (0,95Å) and the effect of these measurements on the crystal lattice parameters.

The XRD pattern of  $Cd_xZn_{1-x}S/PVA$  nanocomposites which obtained different synthesis temperatures is given in figure 5. The intensity of the diffraction peak of the  $Cd_xZn_{1-x}S/PVA$  nanocomposite obtained at room temperature is less than the intensity of the diffraction peaks of the samples obtained at high temperatures. The sample obtained at 40°C corresponds to the  $Cd_xZn_{1-x}S$  compound formed in the hexagonal phase, while maintaining its structure. The structure of the sample obtained at a temperature of 65°C also corresponds to the combination of  $Cd_xZn_{1-x}S$  formed in the hexagonal phase. Diffraction angles of  $2\theta$ =42,14° and 51,76° were observed, and these peaks correspond to the Miller indices (110) and (201). In addition, the new observed peak at  $2\theta$ =33,2° refers to CdS formed in the hexagonal phase and corresponds to the (200) Miller index. The peak observed at  $2\theta$ =38,4° belongs to ZnS, with the Miller index (102) corresponding to the cubic structure. It is clear from the XRD pattern that peaks pertaining to both ternary CdZnS and binary CdS and ZnS compounds were observed.



**Figure 5**. XRD pattern of  $Cd_{0.2}Zn_{0.8}S/PVA$  samples synthesized at different temperatures: a) room temperature, b) T=40°C, c) T=65°C.

The formation of both binary and ternary compounds during the synthesis of  $Cd_{0.2}Zn_{0.8}S/PVA$  nanocomposites at 65°C was also confirmed by the bandgap value. The formation of a ternary CdZnS nanocomposite from the diffraction pattern was observed with more intense peaks at 40°C. Here, the particle sizes were larger than the particle sizes formed at a subsequent increase in the synthesis temperature (65 °C). Differences in peaks at different temperature values during the

Sample	Temperature	R, nm	E <sub>g</sub> ,eV
Cd <sub>0.1</sub> Zn <sub>0.9</sub> S/PVA	synthesis at room temperature ( <i>sample A</i> )	3,2	3,15
Cd <sub>0.1</sub> Zn <sub>0.9</sub> S/PVA	termal annealing T=60°C	3,7	4,51
Cd <sub>0.1</sub> Zn <sub>0.9</sub> S/PVA	termal annealing T=120°C	7,8	3,33
Cd <sub>0.2</sub> Zn <sub>0.8</sub> S/PVA	synthesis at room temperature ( <i>sample B</i> )	3,3	3,05
Cd <sub>0.2</sub> Zn <sub>0.8</sub> S/PVA	termal annealing T=60°C	3,3	3,15
Cd <sub>0.2</sub> Zn <sub>0.8</sub> S/PVA	termal annealing T=120°C	7,0	3,14
Cd <sub>0.2</sub> Zn <sub>0.8</sub> S/PVA	synthesis at T=40°C (sample D)	4,3	3,14
Cd <sub>0.2</sub> Zn <sub>0.8</sub> S/PVA	synthesis at T=65°C ( <i>sample E</i> )	2,6	2,37
Cd <sub>0.8</sub> Zn <sub>0.2</sub> S/PVA	room temperature(sample C)	3,4	2,70
Cd <sub>0.8</sub> Zn <sub>0.2</sub> S/PVA	termal annealing T=60°C	3,5	2,77
Cd <sub>0.8</sub> Zn <sub>0.2</sub> S/PVA	termal annealing T=120°C	11,9	2,72

Table 4. The average size and band gap value of nanocomposites

formation process occurred when the density remained constant. As the synthesis temperature increases, the diffraction peaks shift to larger angles. At the same time, smaller particles formed with increasing temperature.

From the IR spectra of  $Cd_xZn_{1-x}S/PVA$  based nanoparticles obtained by the SILAR method at 25°C temperature, the peaks of 1682 cm<sup>-1</sup> and 1641 cm<sup>-1</sup>, which are clearly which visible in the spectrum facilitate the oscillation of carbonyl groups. In samples synthesized at a high temperature of 40°C, these peaks were shifted to the right and their intensity was reduced. Such changes are associated with the formation of a solid solution in the polymer matrix, the vibration of chemical bonds, the reduction of energy, and, consequently, a decrease in the frequency of oscillating motion. These peaks were not observed in the next increase in the synthesis temperature. This is explained by the visually observed destruction of the PVA matrix (softening) at high temperatures.

In contrast to the pure PVA spectrum, the 694  $cm^{-1}$ , 676  $cm^{-1}$ , and

692 cm<sup>-1</sup> peaks for each Cd<sub>x</sub>Zn<sub>1-x</sub>S nanoparticle placed on the nanocomposite were clearly visible. At a temperature of T=65°C, against the background of a peak of 692 cm<sup>-1</sup>, several peaks can be observed. This is explained by the formation of CdS and ZnS compounds in addition to the hybrid structure of Cd<sub>x</sub>Zn<sub>1-x</sub>S nanoparticles at high temperatures. Sliding in inorganic compounds is due to the stiffness of the nanoparticles and the elongation of the electron bonds present.

From the SEM images of compounds obtained at different synthesis temperatures (Cd<sub>0.2</sub>Zn<sub>0.8</sub>S/PVA), it was determined that the size of the particles obtained at room temperature is 53,5-72,8 nm, the size of the particles obtained at 65°C a temperature is in 4,17-5,04 nm diapason. That is, the reason for the smaller size of the particles is that the particles formed at the sorption centers do not agglomerate. Under the influence of temperature, the ability of charged ions to penetrate the volume of the layer increases and is formed in these centers. Here, the particles are evenly distributed, completely covering the polymer surface. At 65°C, the particles were tightly packed together. As a result of the effect of temperature, the rate of particle formation increased, where smaller particles were formed. Thus, the particles entered the polymer matrix.

Elemental analysis of  $Cd_{0.2}Zn_{0.8}S/PVS$  nanocomposite obtained at different synthesis temperatures determined that the  $Zn^{2+}/Cd^{2+}$  ratio in the sample obtained at room temperature was 1,4. However, according to theoretical calculations based on the amount of initial reagents selected in the reaction, this ratio should have been 4. This is due to the inability of particles to form completely due to the slow formation rate at room temperature. Elemental analysis of  $Cd_{0.2}Zn_{0.8}S/PVA$  sample synthesized at 65°C revealed that the ratio of Zn and Cd elements is 3,7. This ratio approximately coincided with the theoretically calculated ratio of the initially selected reagents. Observations of peaks for the elements Cd, Zn, and S in the spectrum of the element analysis confirm the formation of the compound  $Cd_{0.2}Zn_{0.8}S$ .

### **MAİN RESULTS**

**1.** It was determined that the stoichiometric composition of  $Cd_xZn_{1-x}S$  compounds formed by the SILAR method depends on the parameters of the crystallization medium and the type of sorption centers. The ratios of the concentrations of the cations sorbed at the absorption centers at different formation temperatures are different. This is explained by the fact that the adsorption energies of cations in these centers are different, and as a result, the ratio of the concentration of ions absorbed by the sorption centers varies with temperature.

2. Differences in the size and bandgap value of  $Cd_{0.4}Zn_{0.6}S$  nanoparticles were obtained with different stabilizers while keeping all technological parameters (pH, concentration, temperature, etc.) constant. This is explained by the variety of nanoparticle interaction energies depending on the type of stabilizers used. This is because the stabilizers are polar or non-polar, the values of dipole moments and dielectric constant are different.

**3.** It was found that the bandgap value of nanoparticles changed under the influence of the environment. This is due to the change in the potential energy of the charge carriers (electron-holes) in the particles as a result of their interaction with the environment. This, in turn, leads to a change in the electronic spectra of the charge carriers located inside the nanoparticles. It was found that when CdS nanoparticles were placed in a 0,01 M, 0,1 M, 1 M, 2 M concentrated NaCl electrolyte solution, the bandgap value varied from 3,6 eV to 2,5 eV, respectively.

**4.** The distribution of cations forming particles in the  $Cd_2Zn_{0.8}S/PVA$  compound obtained by the SILAR method at different times was studied along with the thickness of the layer. It is shown that the nature of the distribution has changed depending on the number of maturation periods. Thus, according to the results of elemental analysis, the distribution is homogeneous in two maturation periods and non-homogeneous in five maturation periods. In this case, the concentration of metal atoms on the surface was at its maximum. This is due to two factors: a) the effect of the field created by the double electric field during the maturation process on the redistribution of parti-

cles and b) the migration processes due to the different energy conditions on the surface and volume.

**5.** It is shown that the crystalline structure and optical properties of the synthesized nanoparticles depend on the method of synthesis and the temperature of formation. Thus, CdS, ZnS,  $Cd_xZn_{1-x}S$  phases were formed at 65°C during SILAR formation. Depending on the technological conditions, the bandgap energy of the obtained particles ( $Cd_{0.2}Zn_{0.8}S$ ) changed (T=25°C –  $E_{g(sonochemical)}$ =2,8 eV, T=25°C –  $E_{g(SILAR)}$ =3,05 eV, T=65°C –  $E_{g(SILAR)}$ =2,37 eV). This was due to changes in particle size and crystal structure.

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