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ELECTROSYNTHESIS OF COLLOID SOLUTIONS OF CdTe AND ZnSe NANOPARTICLES AND THEIR OPTICAL PROPERTIES

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Abstract: *By the method of electrosynthesis with the simultaneous extraction in xylene obtained CdTe and ZnSe nanoparticles in the size of 2-3 nm by using of electrolytes based on (NH₄OH, CdSO₄ and Na₂TeO₃) and (H₂SO₄, H₂SeO₃, ZnSO₄). The mechanism formation of nanoparticles consists of the electrochemical stage formation of telluride or selenide ions and of CdTe and ZnSe chemical stage formation determined by cyclic voltammetry. Spectral and photoluminescent studies have shown that of the obtained nanoparticles have an intensive absorption band of 350 to 450 nm with the presence of several double exciton band on the optical spectrum and the bright blue photoluminescence, the spectrum of which also has consist with two bands that's corresponds to exciton and defective photoluminescence. From the obtained colloidal solutions, an optical nanocomposite was synthesized on the basis of a liquid crystalline matrix of cadmium caprylate (CdC₈) with 0.5 mol% of CdTe and ZnSe nanoparticles.*

Keywords: *electrosynthesis, cadmium telluride, zinc selenide, nanoparticles, liquid crystal nanocomposite.*

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Introduction

Promising materials among semiconductor quantum dots are CdTe and ZnSe nanocrystals or nanoparticles, since these materials have the highest photoluminescence efficiency in the visible region of the spectrum. The band gap of ZnSe crystals is 2.7 eV [1,2] and CdTe is 1.44 eV [3]. The use of ZnSe / CdTe nanostructures in optoelectronic systems is more efficient than that of ZnSe or CdTe alone, since ZnSe is capable of efficiently absorbing light quanta with energy of more than 2.7 eV [4]. For nanoparticles of these materials, the absorption threshold shifts to a higher-energy part of the spectrum. For example, small CdTe nanoparticles have intense blue photoluminescence with quantum yields of ~ 70–80% [5]. In combination with a GaN laser, optoelectronic devices can be developed on their basis, since the kinetics of luminescence relaxation of such nanoparticles is in the nanosecond range [5, 6]. The band gap of CdTe crystals is 1.44 eV [7] but on the band gap significantly affecting size of CdTe nanocrystals

[8]. Sapra S. [9] revealed that on the surface of nanocrystals a system of intrinsic point defects and new recombination levels that determined luminescence, was realized. In this case, the surface of small objects begins to play the same role as the volume of the crystal. The size of CdTe nanoparticles in comparison with CdS and CdSe more significantly to influence of their optical properties. For CdTe with a nanoparticle size of 2 nm, the band gap can reach 2.8–3 eV [9]. Most of the methods for producing CdSe, ZnSe, and CdTe nanoparticles are environmentally harmful [10, 11] and technologically complex [12]. One of the simple methods obtain CdTe and ZnSe is the method of electrochemical synthesis [13-15]. The development of methods for synthesis of colloidal solutions of CdTe and ZnSe nanoparticles without anticoagulant additives and impurities of precursor agent the important task for their further use in the formation of nanocomposite optical materials [16].

Experimental part

CdTe nanoparticles in the form of colloidal solutions were obtained by electrolysis from a solution based on 25% NH_4OH , 0.05 mol / l CdSO_4 , and 0.01 mol / l Na_2TeO_3 . This electrolyte was electrolyzed at a temperature of 50 °C and a current density of 250 mA / cm^2 by means of a titanium cathode and a platinum anode. ZnSe nanoparticles were synthesized in an electrolyte based on (0.1 mol / l H_2SeO_3 , 0.2 mol / l ZnSO_4 , 1 mol / l H_2SO_4 and 20% glycerol) in the galvanostatic mode through the use of a graphite anode and a titanium cathode. The electrosynthesis current was 150 mA/ cm^2 ; the temperature was 90 °C. For the extraction of ZnSe nanoparticles during electrosynthesis, the cell was filled with 1/4 volume by xylene. The electrolyte temperature was monitored by means of a thermostat U1. Voltammetric studies of processes for obtaining CdTe and ZnSe were carried out by means of P-8S potentiostat in a three-electrode mode with a silver chloride reference electrode and a platinum counter-electrode. Studies of chemical composition of the obtained colloidal solutions of nanoparticles extracted from xylene were carried out through the use of EDAX spectroscopy. The obtained colloidal solutions were evaporated on a titanium foil at a temperature of 150 °C until a film was formed on its surface which was then

analyzed by the EDAX method (EDAX Element PV6500/00 F). In a similar manner, CdTe and ZnSe powders were prepared for X-ray phase analysis (DRON-4) in this case, and colloidal solutions were evaporated to the powder state at 350 °C.

Electronic absorption spectra were recorded in the range of 250 - 1000 nm with a Perkin Elmer UV / VIS Lambda 35 spectrophotometer. Luminescence spectra were recorded in the range of 300 - 700 nm by means of a Perkin Elmer LS 55 spectrophotometer. To create glassy nanocomposites containing CdTe and ZnSe nanoparticles in the first stage, 1 ml of a colloidal solution $[\text{CdTe}] = 0.012 \text{ mol} \cdot \text{l}^{-1}$ was added to the cadmium caprylate matrix (0.5 grams of powder). The mixture was ground in an agate mortar, transferred to a test tube, and dried in a vacuum oven at a temperature of 100° C until the xylene was completely removed, and then sintered in an atmosphere of inert gas at a temperature of 160° C. After that, the melt was quickly cooled to a room temperature. A transparent glassy mesomorphic nanocomposite with a total content of such nanoparticles of ~ 0.5 mol% was obtained. To study optical properties of the obtained nanocomposites, the samples were placed between two quartz glasses.

Results and discussion

The concentration of TeO_3^{2-} ions dominating in the solutions of tellurous acid at pH = 10 was

established from the calculation of ion equilibrium diagrams (Fig. 1) [17].

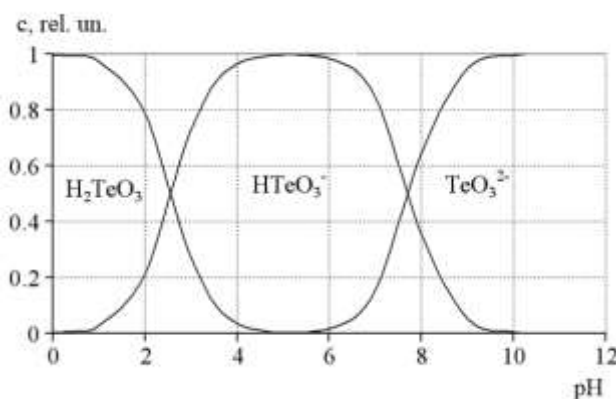


Fig. 1. pH diagram of ionic equilibrium of tellurous acid.

Fig. 2 shows the polarization curves in solutions with substances for CdTe synthesis (1) and

separately for Cd (2) and Te (3) synthesis. The current waves responsible for obtaining Cd and

Te are in the range of potential $-1.1 \div -1.3$ V vs. (C.S.E.) [18]. The wave in charge of the formation of the CdTe begins within the -1.4 V region [18].

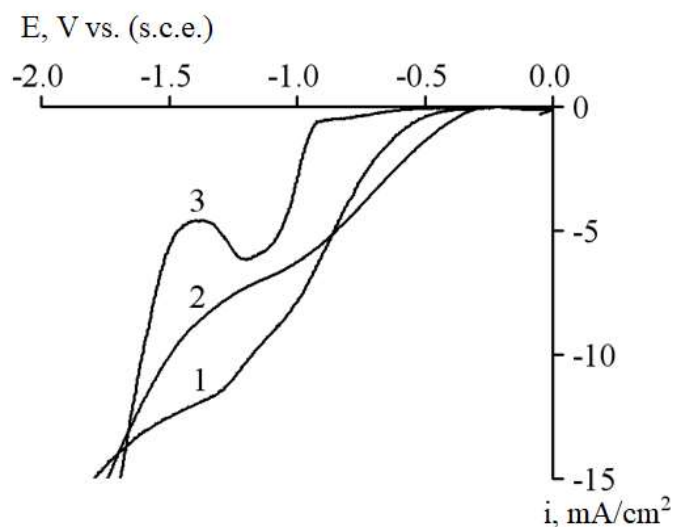


Fig. 2. Polarization curves in the solutions 25% NH_4OH , 0.05 mol / l CdSO_4 , 0.01 mol / l Na_2TeO_3 (1), 25% NH_4OH , 0.05 mol / l CdSO_4 (2), 25% NH_4OH , 0.01 mol / l Na_2TeO_3 (3)

To determine the staging's of CdTe deposition, we measured and analyzed the cyclic current – voltage dependences of the process of CdTe obtaining from an ammonia electrolyte (Fig. 3). Analysis of the maximum wave current density from the root of the sweep rate of potential showed that this dependence is linear and does not converge to 0 (Fig. 4) [19]. Thus, from our CVA studies (Fig. 2 - Fig. 4), with due regard

for the data of ion equilibrium diagrams (Fig. 1), we can conclude that the process of CdTe deposition in an ammonia electrolyte involves a diffusion and a chemical reaction [18, 19]. In this case, the formation mechanism of CdTe nanoparticles in an ammonia electrolyte is two-stage and can be described by reactions 1 and 2 [20].

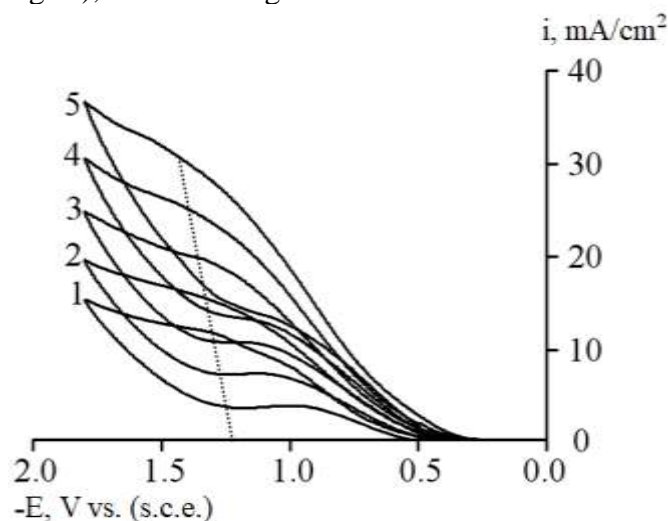


Fig. 3. Cyclic volt-ampere dependences of the CdTe electrosynthesis process from ammonia electrolyte based on tellurous acid and cadmium sulfate at the sweep rates of potential of 28 (1), 44 (2), 59 (3), 74 (4) and 92 mV / s).

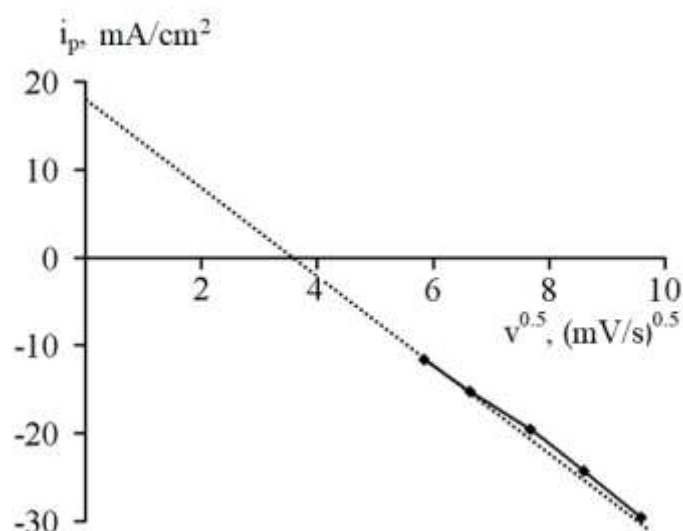
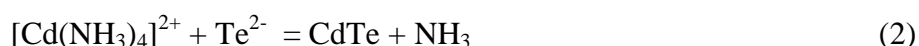


Fig. 4. Dependence of the intensity of current peak of cyclic current-voltage CdTe electrosynthesis process on the square root of sweep rate of potential.



Analysis of the obtained CdTe nanoparticles by EDAX spectroscopy (Fig. 5) showed that the nanoparticles obtained by electrosynthesis from an ammonia electrolyte based on tellurous acid and cadmium sulfate with their simultaneous extraction into xylene had an equal molar

composition of tellurium and cadmium. This indicates that the nanoparticles obtained by electrosynthesis and extracted into xylene form the stoichiometric CdTe compound rather than a solid solution of variable chemical composition containing of cadmium and tellurium.

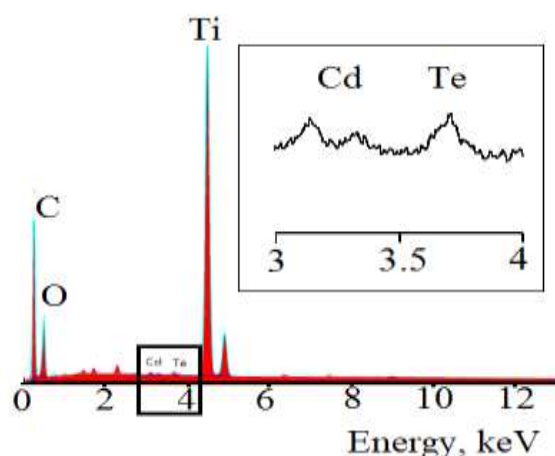


Fig. 5. EDAX spectrum CdTe film obtained by evaporation of colloidal solution of CdTe nanoparticles at 150° C on a titanium substrate.

It should be added that carbon residues organic substances adsorbed on the surface of CdTe nanoparticles became apparent due to the analysis of the diffractogram at 20 degree (Fig.

6) [21, 22]. Analysis of the main peaks of the diffractogram showed that the structure of the obtained nanoparticles mainly consists of wurzite — the hexagonal phase of CdTe [23,

24]. Although some peaks, as shown in Fig. 6, also apply to the cubic phase [24]. Thus, the structure of nanoparticles consists of the combination of two phases.

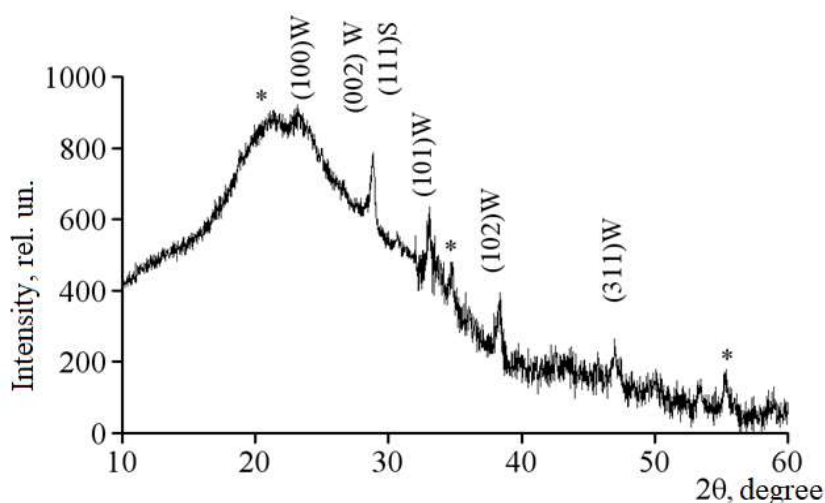


Fig. 6. XRD of the powder obtained by evaporation of a colloidal solution of CdTe nanoparticles in xylene at 350 ° C, where W is wurcrite (hexagonal structure of CdTe), S is sphalerite (cubic structure of CdTe) * - carbon

Investigation of the optical properties CdTe nanoparticles colloidal solutions obtained by electrosynthesis showed that it's has intense absorption in the UV and near visible spectral regions (Fig. 7). Figure 7 shows that over a wide absorption band a 5 exciton peaks of light absorption at 355, 360, 367, 380, and 385 nm can be distinguished. According to published data [9], these absorption bands correspond to nanoparticle sizes from 1 to 3 nm. For nanoparticles of metal chalcogenides, as shown

in [25], in addition to the main exciton absorption band, characteristic double repetition of the excitonic transition was also observed, that is, a lower-intensity absorption band was observed nearby. In our case, in Fig. 7, two double absorption bands can be observed at 355-360 nm and 380-385 nm respectively. The range of these absorption bands in a colloidal solution corresponds to CdTe nanoparticles with an average size of 2 nm [26, 27].

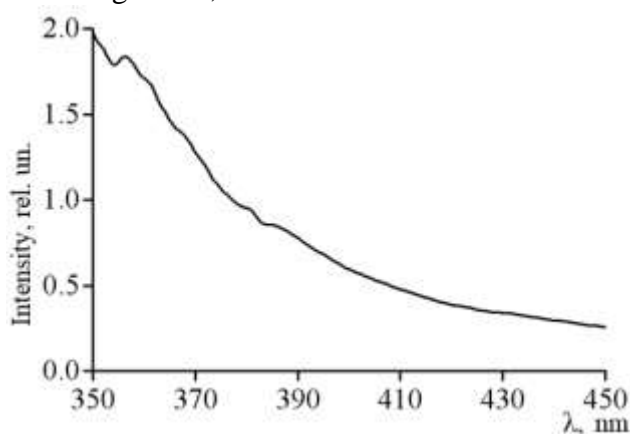


Fig. 7. The absorption spectrum of a colloidal solution of CdTe nanoparticles obtained by electrosynthesis with simultaneous extraction into xylene from solution 25% NH_4OH , 0.05 mol / l CdSO_4 , 0.01 mol / l Na_2TeO_3 at current density 250 mA/cm².

The obtained colloidal solutions have a bright blue photoluminescence (Fig. 8) characteristic of CdTe nanoparticles 2 nm in size [8, 10]. Fig. 8 shows that photoluminescence has two

emission bands that are offset 20 nm from each other. When the photoluminescence excitation wavelength is shifted to the UV region, only one of the luminescence bands at 410 nm increases in intensity, while the second at 430 nm remains almost unchanged. According to [28], just the first emission band can be attributed to the

excitonic photoluminescence while the second band corresponds to the luminescence of defects cubic and hexagonal structures, the difference energy levels between which is 0.044 eV (which corresponds to 55 nm) [27] for the CdTe nanoparticles.

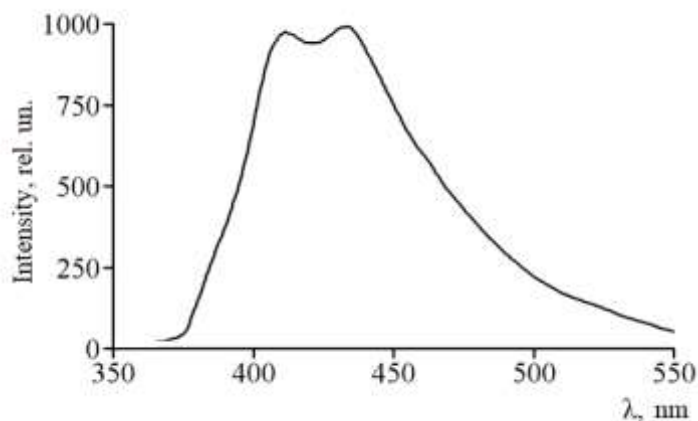


Fig. 8. Photoluminescence spectrum of colloidal solution of CdTe nanoparticle obtained by means of electrosynthesis with simultaneous extraction into xylene.

Analysis of the absorption spectrum of the colloidal solution (Fig. 7) by means of the known empirical dependences [26] allowed estimate the concentration of CdTe nanoparticles in xylene and calculate the concentration of CdTe nanoparticles in the obtained CdC₈ + CdTe nanocomposite which is ~ 0.5 ml. %. Fig. 9 shows the absorption spectrum of the CdC₈ + CdTe nanocomposite from which it can be seen that, in contrast to the

colloidal solution where the exciton absorption band is characterized by exciton repetitions in the form of second band, in the CdC₈ + CdTe nanocomposite have one exciton transition, which is sharper than in the colloidal solution. This is most likely due to the formation of intermediate associates of CdTe nanoparticles in a colloidal solution and their absence in the CdC₈ matrix, where their formation is hindered by higher viscosity and density than in xylene.

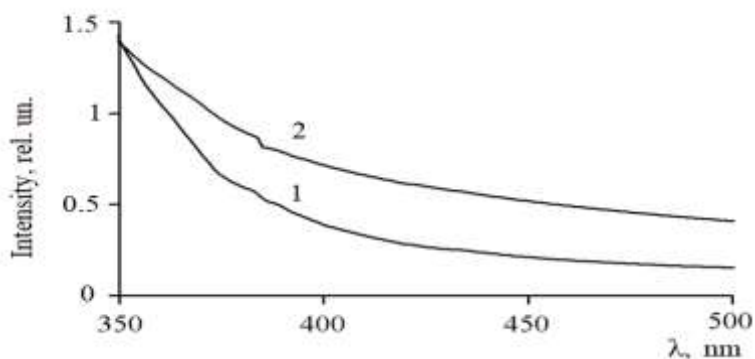


Fig. 9. Optical spectra of colloidal solution of CdTe nanoparticles (1) and nanocomposite CdC₈+CdTe (2) where the concentration of CdTe nanoparticles in the nanocomposite is 0.5 mol. %.

It was found that the photoluminescent properties of the CdC₈ + CdTe nanocomposite are similar to the properties of colloidal solutions (Fig. 10). In the nanocomposite, there

is also observed a mixture of the main exciton photoluminescence band in UV region with a decrease in the excitation wavelength and the

stability of the defect's photoluminescence band in the structure of CdTe nanoparticles.

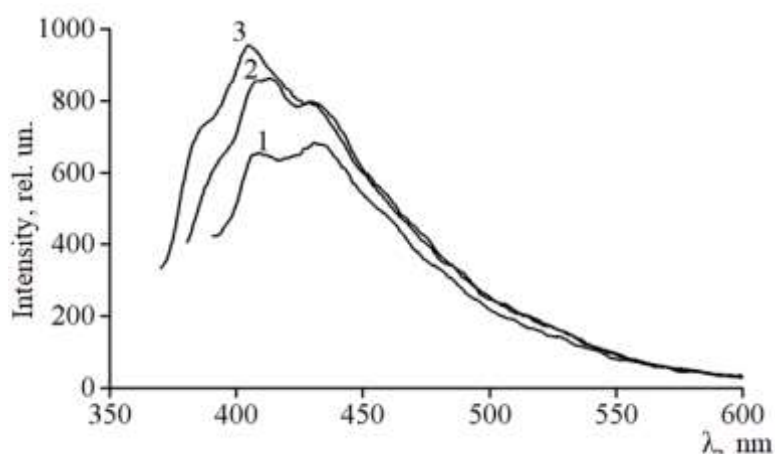
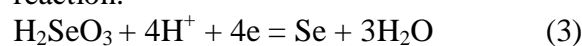


Fig. 10. Photoluminescence spectra of CdC₈ + CdTe nanocomposite with a concentration of CdTe nanoparticles in the nanocomposite of 0.5 mol. %, at different wavelengths of excitation: 380 (1), 370 (2) and 360 nm (3).

In fig. 11 presents the results of voltammetric investigations of the electrosynthesis of ZnSe nanoparticles. Curve 1 fig. 11 have only a wave of hydrogen evolution for a background electrolyte based on sulfuric acid. A curve 2 fig. 11 electrolyte based on selenic acid and background electrolyte have wave of selenium. The waves of Zn and Se formation differ by 100 mV (curves 3 and 2 in Fig. 11). The ZnSe

formation wave is shifted by 50 mV from the Zn formation wave and by 150 mV from the Se formation wave. From this we can conclude that the selenium phase is first formed by the reaction:



whither the zinc ions diffuse to form the compound ZnSe by the reaction as follows:

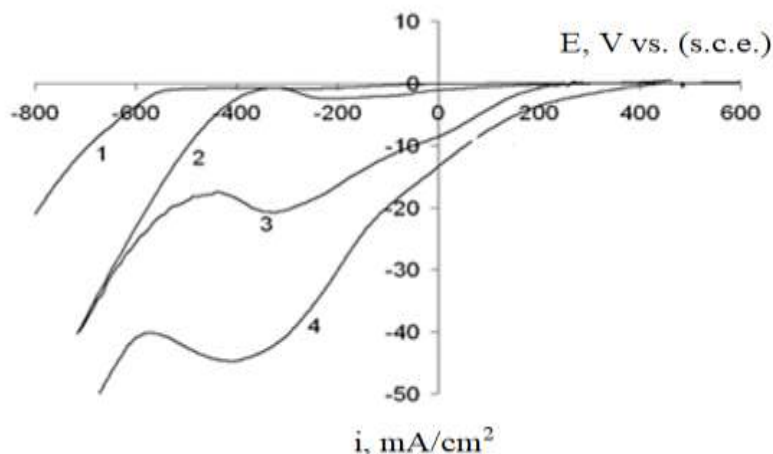
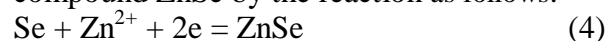


Fig. 11. Polarization curves solutions of substances included in ZnSe deposition electrolyte

XRD analysis of ZnSe nanoparticles powder (Fig. 12) obtained by electrosynthesis established that these nanoparticles have peaks

of crystalline modification of the wurcrite type [29].

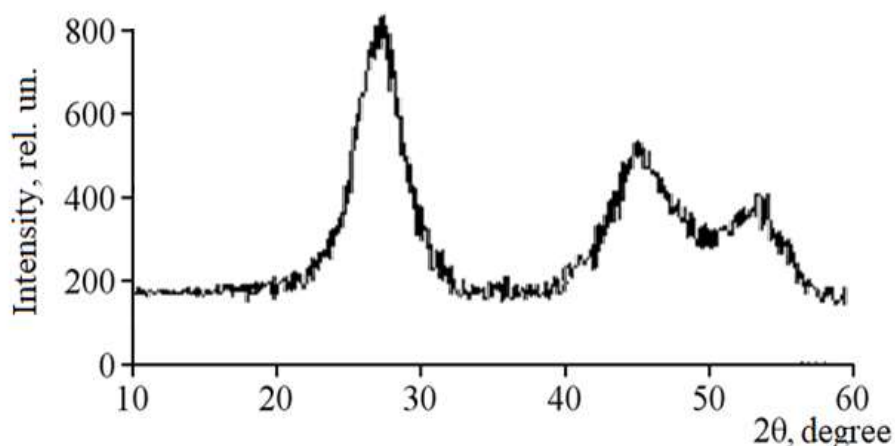


Fig. 12. XRD of ZnSe nanoparticles powder after heat treatment at 400°C

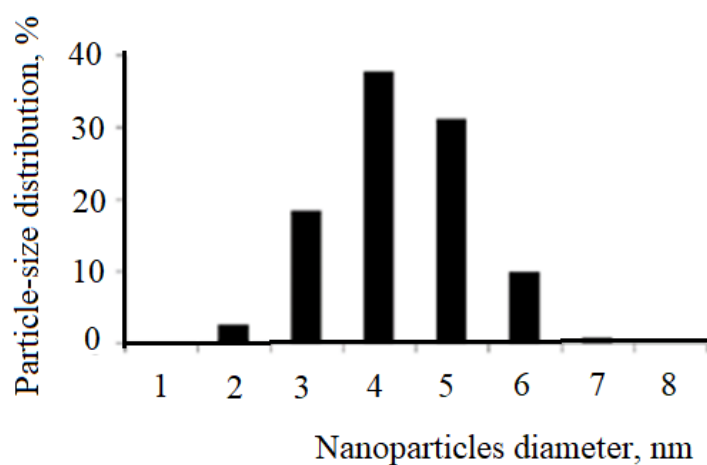


Fig. 13. Diagram of size distribution of ZnSe nanoparticles in colloidal solution (in xylene)

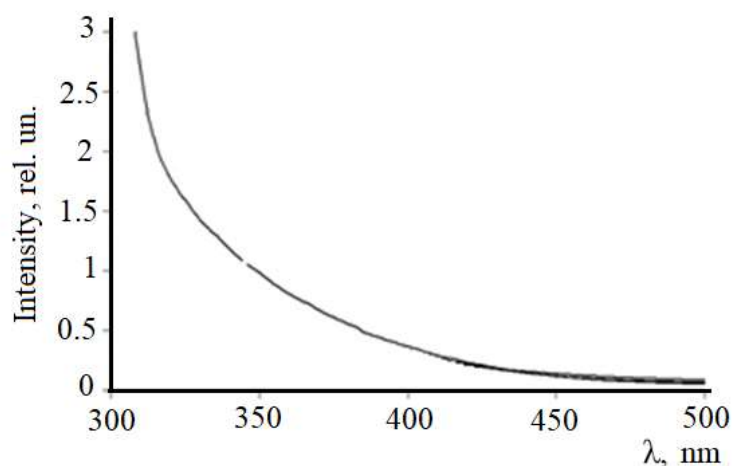


Fig. 14. Optical spectrum of 0.5 mol.% ZnSe nanoparticles in a matrix based on cadmium octanoate CdC₈.

Using the z-scanning technique according to the calculations of light scattering [30] by a colloidal solution of ZnSe nanoparticles in xylene, it was found that

mainly in the solution ~ 90% of nanoparticles of small size 3-6 nm (Fig. 13). Investigation of the optical properties of the CdC₈ + ZnSe nanocomposite in Fig. 14 showed a wide

absorption band from 300 to 400 nm in the UV region of the spectrum. The analysis of the photoluminescent properties of the CdC₈ + ZnSe nanocomposite showed the broadening of the photoluminescence band (Fig. 15) of characteristic mainly to small nanoparticles (3-6

nm, $\lambda_{\max} = 480-520$ nm). Also, on the photoluminescence spectra there is a radiation arm in the range $\lambda_{\max} = 540-600$ nm, which corresponds to the size of larger nanoparticles (8-10 nm) [29].

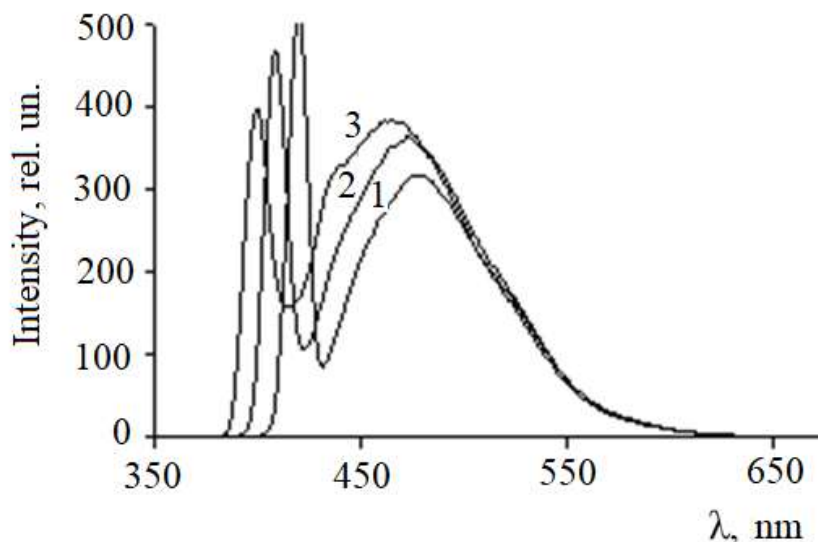


Fig. 15. Photoluminescence spectra of the solution of ZnSe nanoparticles in xylene at excitation wavelengths 1–400, 2–410, 3–420 nm.

Conclusion

Colloidal solutions of CdTe nanoparticles with a size of 2-3 nm of mixed hexagonal and cubic structure and ZnSe nanoparticles of hexagonal structure were obtained by electrosynthesis during simultaneous extraction into xylene. It was found that nanoparticles have an intense absorption of 350 to 450 nm and a bright blue

photoluminescence of 400 to 500 nm. Using the obtained colloidal solutions, optical nanocomposites based on the liquid crystal matrix of cadmium caprylate with a 0.5 mol% of CdTe and ZnSe nanoparticles were synthesized. These nanocomposites can be promising for use in high-speed optical switches.

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ЭЛЕКТРОСИНТЕЗ КОЛЛОИДНЫХ РАСТВОРОВ НАНОЧАСТИЦ CdTe, ZnSe И ИХ ОПТИЧЕСКИЕ СВОЙСТВА

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Методом электросинтеза с одновременной экстракцией в ксилол получены наночастицы CdTe и ZnSe размером 2-3 нм с использованием электролитов на основе (NH₄OH, CdSO₄ и Na₂TeO₃) и (H₂SO₄, H₂SeO₃, ZnSO₄). Механизм образования наночастиц состоит из электрохимической стадии образования теллурида или селенида ионов и химической стадии образования CdTe и ZnSe. Спектральные и фотолюминесцентные исследования показали, что полученные наночастицы имеют интенсивную полосу поглощения от 350 до 450 нм с наличием нескольких двойных экситонных полос на оптическом спектре и ярко-синей фотолюминесценцией, спектр которой также состоит из двух полос, что соответствует экситонной фотолюминесценции, а также фотолюминесценции, вызванной дефектами кристаллической решетки. Из полученных коллоидных растворов синтезировали оптические нанокомпозиты на основе жидкокристаллической матрицы каприлата кадмия (CdC₈) с 0.5 мол. % наночастиц CdTe или ZnSe.

Ключевые слова: электросинтез, теллурид кадмия, селенид цинка, наночастицы, жидкокристаллический нанокомпозит.

CdTe, ZnSe NANOHİSSƏCİKLƏRİN KOLLOİD MƏHLULLARININ ELEKTROSİNTEZİ VƏ ONLARIN OPTİK XASSƏLƏRİ

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Elektrosintez metodla NH_4OH , CdSO_4 , Na_2TeO_3 və H_2SO_4 , H_2SeO_3 , ZnSO_4 elektrolitlərdən istifadə edərək 2-3 nm ölçülü CdTe və ZnSe nanohissəcikləri alınmışdır. Nanohissəciklərin əmələgəlmə mexanizmi elektrokimyəvi (tellurid və yaxud selenid ionların əmələ gəlməsi) və kimyəvi (CdTe, ZnSe əmələgəlməsi) mərhələlərdən ibarətdir. Alınan kolloid məhlullardan mayekristallik kadmium kaprilat (CdC_8) matrisi və 0.5 mol. % CdTe (və yaxud ZnSe) əsasında optik nanokompozitlər sintez olunmuşdur.

Açar sözlər: *elektrosintez, kadmium tellurid, nanohissəciklər, optik nanokompozitlər*