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O. Krupko¹, L.P Shcherbak², Yu.B. Khalavka², V.G. Pylypko²

Effect of silver ions on the optical properties of colloidal solutions of CdS/L-Cysteine nanoparticles

¹Bukovinian State Medical University, Chernivtsi, Ukraine, krupko@ukr.net.

²Yuriy Fed'kovych Chernivtsi National University, Chernivtsi, Ukraine

The synthesis of CdS/Ag⁺ heterostructures was carried out based on colloidal solutions of CdS nanoparticles stabilized by the amino acid L-cysteine by the method of ion substitution and co-precipitation from aqueous solutions at room temperature and a hydrogen index value of 7.

The effect of argentum ions on the optical properties of colloidal solutions of cadmium sulfide nanoparticles in the conditions of an oxidizing environment was investigated.

Argentum ion concentrations were determined, the introduction of which leads to an increase in the photoluminescence of the system and the growth of nanoparticles compared to the original colloidal solution of CdS/L-Cysteine NPs.

Keywords: heterostructures, CdS nanoparticles, Ag⁺ ions, optical properties, photoluminescence.

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Introduction

The creation of core-shell heteronanostructures expands the possibilities of using semiconductor nanocrystals as new effective catalysts, and photocatalysts [1-4], as well as for the production of flat displays, diodes, and sensors [5, 6, 8, 9]. In order to modify the optical properties of CdS nanoparticles (NPs), in many works, the possibility of doping them with cations of d-elements Ag⁺, Cu²⁺, Pt²⁺ [3,7-14] and other different practical applications [1-4, 21-24] was investigated. CdS NPs stabilized by L-cysteine (L-Cys) are also widely used in analytical chemistry (in particular, as sensors of cations of d-elements - Zn²⁺, Ag⁺, Cu²⁺, Co²⁺, Ni²⁺, Hg²⁺, and s-elements Ca²⁺, Mg²⁺ [15-20], as photocatalysts [1, 4, 8, 9, 14, 21-23], as active elements of light-emitting diodes [24, 25], as photoelectrochemical and fluorescent nanosensors for detecting L-cysteine, L-morphine, D-methamphetamine [15, 26].

The alloying effect of ions of d-elements is detected when introduced into the reaction mixture at various stages of synthesis [7], in various reaction media [27-31], and obtained by various methods [4, 8-13, 15-26].

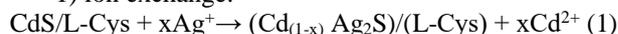
One of the methods of obtaining double sulfides is the replacement of metal cations in the nanosized sulfide of another metal. The possibility of such a synthesis is substantiated by the values of the solubility product of the initial sulfide and that obtained by ion substitution ($K_s(\text{CdS}) = 1 \cdot 10^{-27} > K_s(\text{Ag}_2\text{S}) = 1 \cdot 10^{-50}$).

The purpose of this work was to study the influence of CdS/L-Cys/Ag heterostructure synthesis methods and dopant concentration on the optical properties of colloidal solutions of CdS/L-Cys nanoparticles.

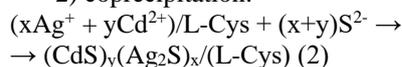
I. Experimental

The synthesis of CdS/(Ag⁺) heterostructures was carried out according to two schemes:

1) ion exchange:



2) coprecipitation:



Synthesis according to both schemes was carried out using high-quality starting reagents, namely: 0.5 M

solution of $\text{CdCl}_2 \cdot 2.5\text{H}_2\text{O}$ of the "high grade" grade, 0.05 M solution of "high grade" L-Cysteine; 0.5 M solution of $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$ (99% purity Aldrich), 0.1 M solution of NaOH, $1 \cdot 10^{-3}$ M solution of AgNO_3 "CP".

In the experiments, colloidal solutions of CdS/L-Cys NPs with a fixed ratio of all precursors were used, namely $[\text{Cd}^{2+}]:[\text{L-Cys}]:[\text{S}^{2-}] = 1.1 : 2.2 : 1$. After draining the initial solutions of CdCl_2 reagents and L-cysteine, the pH of the solution was adjusted to 7. The ratio between the content of $\text{Cd}^{2+}/\text{Ag}^+$ ions was 400:1, 80:1 and 40:1.

All syntheses were carried out at a temperature of 298 K using deionized distilled water and without prior deaeration of the solutions.

The study of the optical properties of the solutions was carried out at a temperature of 298 ± 5 K using MDR-4 and USB-650 spectrophotometers (Ocean Optics). The optical density of the solutions was measured in the range of 0.01–2 with increasing wavelength in the range of 350–1000 nm.

Photoluminescence (PL) was measured on a Perkin-Elmer LS55 luminescence spectrometer. All investigated solutions were excited by light with a wavelength of $\lambda = 360$ nm. The quantum yield of photoluminescence was evaluated using a standard solution of anthracene as a luminescence standard (the quantum yield of which was taken as 100%).

To study the structure of CdS NPs by TEM, a drop of

the analyzed solution was applied to a graphite substrate and dried under a vacuum for 12 hours. Transmission electron microscopes CM-12, EM-420, Tecnai F30, manufactured by FEI, were used for electron microscopy. The resolution of the method reached 0.5 - 0.3 nm depending on the magnification. The image was recorded with a CCD camera.

II. Results and Discussion

2.1. Study of the optical properties of the CdS/L-Cys/ Ag^+ colloidal solution obtained by the ion exchange method

To study the effect of impurity ions of Argentum on the spectral characteristics of NPs, a solution of CdS/L-Cys NPs synthesized with a slight excess of the concentration of cadmium ions to S^{2-} ions (25.6% Cd^{2+} : 23.2% S^{2-} or 1.1 : 1) and 51.2% L-Cys. This ratio was selected experimentally from previous studies (the selected colloidal solution is stable over time, characterized by monodisperse particles and a quantum yield of 15%).

The dependence of optical characteristics on the concentration of Ag^+ ions is given in the table. 1 and illustrated in Fig. 1.

In Fig. 1 shows the absorption (a) and luminescence

Table 1.

The dependence of the optical characteristics of solutions on the CdS/L-Cys/ Ag^+ system, from Ag^+ concentration

№	$[\text{Ag}^+]$, mol/l	$[\text{Cd}^{2+}]/[\text{Ag}^+]$	λ_{lim} nm	λ_{max} nm	$\lambda_{\text{max-lum}}$ nm	Stokeslan dslide, nm	D, nm
1	0	-	409	389	506	117	3.8
2	$1 \cdot 10^{-5}$	400.0	409	385	499	114	3.8
3	$5 \cdot 10^{-5}$	80.0	419	390	513	123	4.0
4	$1 \cdot 10^{-4}$	40.0	423	392	516	124	4.1
5	$1.6 \cdot 10^{-4}$	25.0	436	*	*	*	4.5
6	$2.8 \cdot 10^{-4}$	14.3	440	*	*	*	4.6
7	$4.5 \cdot 10^{-4}$	9.0	449	*	*	*	5.0
8	$5.5 \cdot 10^{-4}$	7.3	451	*	*	*	5.1
9	$6.5 \cdot 10^{-4}$	6.1	458	*	*	*	5.4

Note: * - measurements were not performed; λ_{lim} - optical absorption edge, λ_{max} - optical absorption maximum, $\lambda_{\text{max.lum}}$ - luminescence maximum, D - particle size.

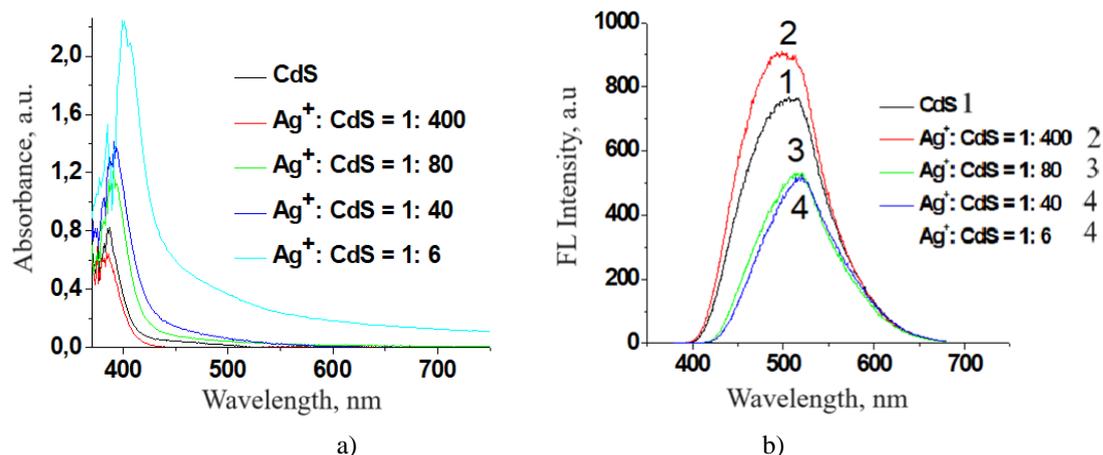


Fig. 1. Absorption (a) and luminescence (b) spectra for solutions obtained according to Scheme 1 with different concentrations of Ag^+ ions.

(b) spectra of the initial solution of CdS NPs and a series of solutions with different concentrations of silver ions obtained according to the scheme (1). The peak of the absorption spectrum of the original solution (375 nm) shifts to the long-wavelength region in proportion to the amount of the introduced impurity, while the spectral curve is noticeably broadened. Since in the investigated concentration interval of the alloying agent, when the impurity content increases, no reflexes characteristic of Ag₂S NPs occur in the region of 515-520 nm [32], the broadening of the spectrum should be associated with the localization of Argentum on the surface of CdS/L-Cys NPs.

If the concentration of Ag⁺ ions (1·10⁻⁵ mol/l), which is the minimum level studied, enhances photoluminescence, then a further increase in concentration results in an increase in the Stokes shift and causes photoluminescence quenching. This trend indicates an increase in the defectivity of nanocrystals.

A similar trend is also observed in works [27, 28], where it is shown that with a low impurity content in the Ag/CdS/L-Cys structure, the PL intensity increases, and exceeding a certain critical concentration of Argentum in the reaction mixture causes quenching of photoluminescence.

Taking into account the values of solubility products of Argentum sulfides (according to various sources $K_s(\text{Ag}_2\text{S}) = 8 \cdot 10^{-51} \div 1 \cdot 10^{-49}$) and Cadmium CdS ($K_s(\text{CdS}) = 1 \cdot 10^{-26} \div 1 \cdot 10^{-27}$), it can be expected that when Argentum ions are added to CdS NPs, ionic replacement of Cadmium by Argentum should occur with the formation of a more stable sulfide, since the solubility product of Ag₂S is much smaller than that of CdS. However, the appearance of both types of spectra (Fig. 1) does not change, new peaks do not appear, therefore the nucleus of the original particles does not change its composition.

The effect of Argentum ion concentration on the optical properties of the CdS colloidal solution is shown in Figure 2.

The nature of the concentration dependence of the absorption edge of the investigated CdS/L-Cys NPs on the concentration of the impurity ion of Argentum (Fig. 2) makes it possible to establish the critical concentration of Ag⁺ ions ($[\text{Ag}^+] = 1.5 \cdot 10^{-4}$ mol/l), which can be determined by the change in the absorption spectrum of the initial NP solutions and is a nomogram for determining higher values of the content of this impurity in the analyzed solution. A break in the dependence can be interpreted as evidence of a change in the mechanism of interaction of Ag⁺ with NPs. Perhaps, with a lower content, Argentum ions are introduced into the core of NPs in the position of Cadmium vacancies, while not significantly changing the band gap. Above the critical concentration of $[\text{Ag}^+]$, one can expect not only an increase in the size of NPs but also

a change in the structure of the core, if we take into account the results of [33], where it is shown that an increase in the size of CdS NPs above 5–6 nm causes their transformation from hexagonal into the cubic phase. In addition, since the shape of the peak on the absorption and PL spectra does not change and new naturally occurring sulfides are not formed, but only the growth of primary nuclei occurs, it cannot be excluded that the reason for the growth of NPs is their aggregation. The reason for this conclusion is the change in the color of the synthesized solutions from light lemon to dark brown during their storage. Also, with an increase in the content of Ag⁺ ions, the stability of colloidal solutions decreases and coagulation is observed.

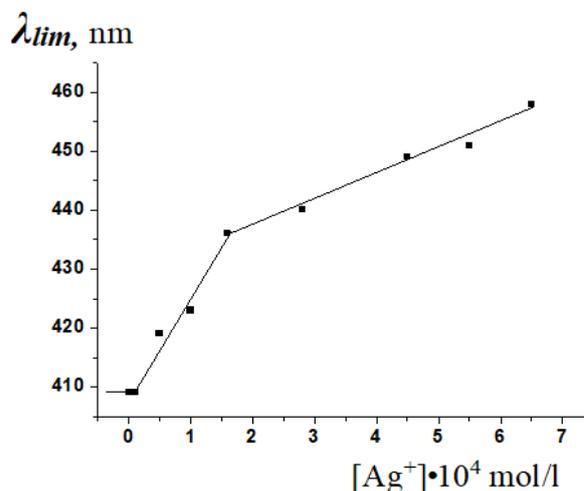


Fig. 2. Influence of the content of Argentum ions on the position of the absorption edge of the colloidal solution of CdS/L-Cys NPs.

2.2. Study of the optical properties of the Ag⁺/CdS/L-Cys/ colloidal solution obtained by coprecipitation

The results of a series of experiments on coprecipitation of cadmium and argentum cations by sulfide ions are summarized in table 2 and illustrated in Fig. 3. From the spectra of optical absorption (Fig. 3, a.), it follows that with the addition of Ag⁺ ions with a concentration of more than 1·10⁻⁵ mol/l, the edge of optical absorption shifts to the long-wavelength region. Since the content of Cadmium ions is significantly higher compared to the content of Ag⁺, both spectral curves reflect the formation of CdS/L-Cys NPs without evidence of Ag₂S formation.

The effect of $[\text{Ag}^+]$ on the position of the absorption edge of solutions (Fig. 4) shows that the order of introducing Ag⁺ ions at least up to $[\text{Ag}^+] = 6 \cdot 10^{-5}$ mol/l within the experimental error does not significantly affect

Table 2.

Dependence of optical characteristics in the Ag⁺/CdS/L-Cys system on $[\text{Ag}^+]$

№	$[\text{Ag}^+]$, mol/l	$[\text{Cd}^{2+}]/[\text{Ag}^+]$	λ _{lim} , nm	λ _{max} , nm	λ _{max.lum} , nm	Stokeslandslide, nm	D, nm
1	0	-	409	389	506	117	3.8
2	1·10 ⁻⁵	400	411	386	491	105	3.8
3	5·10 ⁻⁵	80	416	394	508	114	3.9
4	1·10 ⁻⁴	40	422	394	514	120	4.1

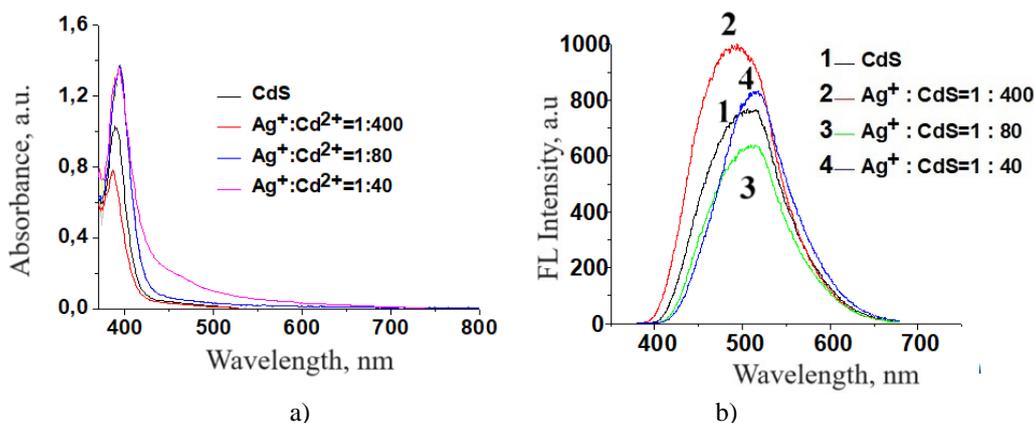


Fig. 3. Spectra of optical absorption (a) and luminescence (b) in the $(Ag^+ + Cd^{2+})S^{2-}$ -L-Cys system.

the position of the limiting wavelength of the optical absorption of the colloidal solution.

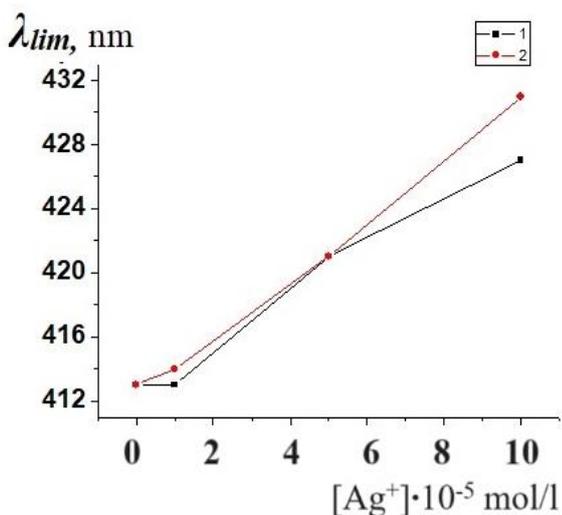


Fig. 4. The position of the limiting wavelength as a function of the concentration of Ag^+ ions introduced into the CdS/L-Cys+ Ag^+ (1) and $(Ag^+ + Cd^{2+})S^{2-}$ -L-Cys+ S^{2-} (2) system.

At the same time, the hypsochromic shift of λ_{lim} caused by the addition of a minimal amount ($1 \cdot 10^{-5} \text{ mol/l}$) of Argentum ions and the increase in PL intensity, as in the previous case (deposition according to

scheme 1), may indicate the introduction of an impurity into the semiconductor lattice. At the same time, the admixture acts as a polarizer to sulfide ions.

CdS quantum dots doped with Ag and stabilized by oleic acid obtained in [10] showed a similar red spectral shift in the absorption spectra when low concentrations of silver were added. The lifetime of excitons at minimum concentrations of silver increased and was suppressed when the concentration of the alloying agent increased. The emission intensity of CdS/Ag nanocomposites in work [11], compared to the original CdS solution, slightly decreases, and the width of the emission peak expands, which the authors explain by the capture of charge recombination after the introduction of Ag into the CdS lattice. The obtained photoluminescence results can be explained in a similar way, namely a slight increase in the band gap for sample 2 (Fig. 3, b). A further increase in the concentration of Argentum cations leads to an increase in the size of nanoparticles (Table 2) and a change in their optical characteristics relative to the initial solution of cadmium sulfide (sample 1) and the minimum concentration of the alloying agent (sample 2) (Fig. 3, a, b, samples 3, 4).

The increase in the concentration of the formed particles and their size is confirmed by TEM images of NPs from the investigated systems (Fig. 5).

Evaluation of the results of TEM images shows that the CdS and CdS/Ag nanoparticles formed in the work have a spherical shape, which is also confirmed by the data

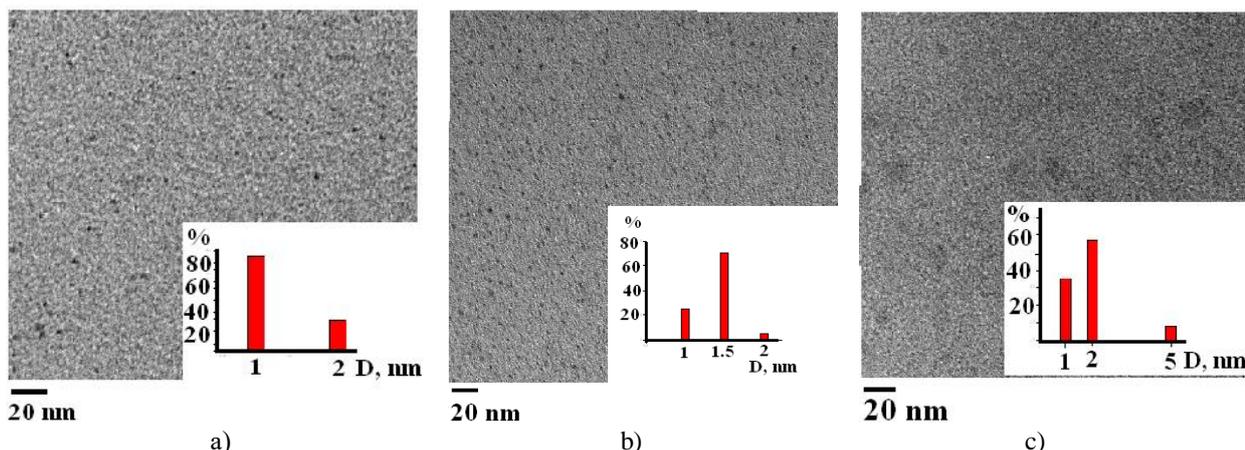


Fig. 5. TEM image of NPs from solutions obtained according to scheme 2: a) CdS, b) $[Cd^{2+}]/[Ag^+] = 80/1$, c) $[Cd^{2+}]/[Ag^+] = 40/1$; in the inset is a histogram of the distribution by size of NPs.

[9, 11, 23], despite the different conditions of their preparation. An increase in the concentration of Argentum ions leads to the aggregation of nanoparticles, with subsequent coagulation over time. A similar tendency to the process of aggregation of CdS nanoparticles doped with Ag was also observed in [17].

Conclusions

The effect of Ag⁺ ions on the optical properties of the CdS semiconductor was investigated. It was found that the introduction of Ag⁺ ions (concentration 1·10⁻⁵ mol/l) into a colloidal solution with nanosized CdS particles causes an increase in the quantum yield of luminescence compared to the original solution of CdS NPs. The improvement of the luminescence output is caused by the fact that the introduction of impurity ions with a +1 charge contributes to the formation of hybrid structures that participate in energy conversion in the excited state of the system. Absorption spectra are characterized by a shift of the absorption edge to the long-wavelength region. The absorption edge is not clear, which corresponds to the defectiveness of the obtained systems.

In all cases, the introduction of admixtures of Ag⁺ ions causes an increase in the number of formula units included in the composition of the agglomerate.

Based on the obtained results of optical studies of the CdS/L-Cys/Ag⁺ system, it is possible to conclude about the possibility of using the obtained colloidal CdS/L-Cys systems in analytical chemistry as sensors for the detection of Ag⁺ ions.

Krupko O. – PhD in Chemical Sciences, Associate Professor of the Department of Medical and Pharmaceutical Chemistry of Bukovinian State Medical University;

Shcherbak L.P. – Doctor of Chemical Sciences, Professor of the Department of General Chemistry and Chemical Materials Science;

Khalavka Yu.B. – Doctor of Chemical Sciences, Vice-Rector for Research Associate Professor, Yuriy Fedkovych Chernivtsi National University;

Pylypko V.G. – Doctor of Philosophy, Head of the laboratory of the Research Center «Functional Materials Technology», Yuriy Fedkovych Chernivtsi National University.

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О.В. Крупко¹, Л.П. Щербак², Ю.В. Халавка², В.Г. Пилипко²

Вплив іонів срібла на оптичні властивості колоїдних розчинів наночастинок CdS/L-Cysteine

¹*Буковинський державний медичний університет, Чернівці, Україна, krupko@ukr.net*

²*Чернівецький національний університет імені Юрія Федьковича, Чернівці, Україна*

Проведено синтез гетероструктур складу CdS/Ag⁺ на основі колоїдних розчинів наночастинок CdS стабілізованих амінокислотою L-цистеїн методом йонного заміщення та співосадження із водних розчинів за кімнатної температури та значення водневого показника 7.

Досліджено вплив іонів аргентуму на оптичні властивості колоїдних розчинів наночастинок кадмій сульфід у умовах окиснюваного середовища.

Визначено концентрації іонів Аргентуму, введення яких призводить до підвищення фотолюмінесценції системи та росту наночастинок у порівнянні із вихідним колоїдним розчином НЧ CdS/L-Цистеїн.

Ключові слова: гетероструктури, наночастинок CdS, йони Ag⁺, оптичні властивості, фотолюмінесценція.