



## Original articles

Original article

<https://doi.org/10.17308/kcmf.2021.23/3302>

## Luminescent properties of colloidal mixtures of $Zn_{0.5}Cd_{0.5}S$ quantum dots and gold nanoparticles

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### Abstract

The aim of the study is to establish spectral-luminescent interaction effects in mixtures of colloidal  $Zn_{0.5}Cd_{0.5}S$  quantum dots passivated with 2-mercaptopropionic acid and Au and Au/SiO<sub>2</sub> nanoparticles. The studied samples of  $Zn_{0.5}Cd_{0.5}S$  quantum dots, Au and Au/SiO<sub>2</sub> nanoparticles and their mixtures were obtained by methods of colloidal synthesis and were characterised using transmission electron microscopy. The absorption, luminescence and time-resolved luminescence spectroscopy were used as the main investigation methods. The measurements were carried out at temperatures of 77 K and 300 K. The spectral-luminescent properties of “free”  $Zn_{0.5}Cd_{0.5}S$  quantum dots and those interacting with Au and Au/SiO<sub>2</sub> nanoparticles were compared. It was found that the luminescence properties of  $Zn_{0.5}Cd_{0.5}S$  quantum dots can be controlled under conditions of changing plasmon–exciton coupling achieved during the formation of a dielectric SiO<sub>2</sub> shell on the surface of Au nanoparticles as well as a result of a polymer introduced into the colloidal mixture.

**Keywords:**  $Zn_{0.5}Cd_{0.5}S$  quantum dots, gold nanoparticles, core/shell, silicon dioxide (SiO<sub>2</sub>), extinction spectrum, plasmon–exciton interaction

**Acknowledgements:** the study was carried out within the framework of the Grant of the President of the Russian Federation to Support Leading Scientific Schools of the Russian Federation, project NSH-2613.2020.2. The results of transmission electron microscopy using a Libra 120 microscope were obtained with the help of the equipment of the Center for Collective Use of Voronezh State University.

**For citation:** Ovchinnikov O. V., Smirnov M. S., Grevtseva I. G., Derepko V. N., Chevychelova T. A., Leonova L. Yu., Perepelitsa A. S., Kondratenko T. S. Luminescent properties of colloidal mixtures of  $Zn_{0.5}Cd_{0.5}S$  quantum dots and gold nanoparticles. *Kondensirovannye sredy i mezhfaznye granitsy = Condensed Matter and Interphases*. 2021;23(1): 49–55. <https://doi.org/10.17308/kcmf.2021.23/3302>

Овчинников О. В., Смирнов М. С., Гревцева И. Г., Дерепко В. Н., Чевычелова Т. А., Леонова Л. Ю., Перепелица А. С., Кондратенко Т. С. Люминесцентные свойства коллоидных смесей квантовых точек  $Zn_{0.5}Cd_{0.5}S$  с наночастицами золота. *Конденсированные среды и межфазные границы*. 2021;23(1): 40–55. <https://doi.org/10.17308/kcmf.2021.23/3302>

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## 1. Introduction

Much attention has been recently paid to attempts to create hybrid plasmon-exciton nanostructures based on metal (plasmonic) nanoparticles (NPs), semiconductor quantum dots (QDs), and/or dye molecules [1-10]. For such hybrid systems, QDs and dye luminescence spectra are heavily affected by the presence of metal nanoparticles (nanoresonators) which have modes with frequencies similar to the frequency of the luminescence maximum. Spatial distribution of the mixture components is also essential for the resulting spectral pattern. The variation of these parameters allows adjusting the modes of plasmon-exciton coupling (weak, intermediate, and strong), which opens the possibility to control the parameters of the spectral-luminescence properties of the emitter [10–12].

Some sources describe research aimed at identifying conditions for the formation of plasmon-exciton nanostructures providing resonance effects in the modes of weak (Purcell effect), intermediate (Fano effect), and strong (Rabi splitting) plasmon-exciton interaction [1, 2, 6–13]. Moreover, the researchers have detected plasmon-induced fluorescence amplification/quenching [9], the plasmon-enhanced Förster energy transfer [9], and induced exciton-plasmon-photon conversion [8]. However, the available results of the studies of plasmon-exciton interaction do not allow finding a solution to the fundamental problem of predicting the final luminescent properties of hybrid nanostructures.

It is important that there has not been developed yet a unified approach to creating hybrid nanostructures to adjust the modes of plasmon-exciton coupling from weak to intermediate and strong. Experimental data obtained by different research teams are contradictory and vary mainly between fluorescence amplification and quenching. Moreover, they do not provide major parameters and physically important experimental characteristics to explain the processes of plasmon-exciton interactions. The absence of detailed experimental data and their weak correlation with the results of theoretical calculations are due to the complicated nature

of plasmon-exciton interactions. Also, the researchers have not developed the problem of the formation of QD luminescence quenching centres when QDs interact with plasmonic NPs, as well as their role in shaping the final “hybrid” luminescent properties of plasmon-exciton nanostructures. Therefore, the development of techniques to control luminescent properties of hybrid nanostructures based on plasmonic NPs and QDs and/or dye molecules is a pressing problem.

This paper presents experimental data which show the possibility to control the luminescent properties of  $\text{Zn}_{0.5}\text{Cd}_{0.5}\text{S}$  QDs in the near field of spherical gold (Au) NPs. The research involved creating special conditions which made it possible to change a plasmon-exciton coupling by forming Au/SiO<sub>2</sub> core/shell NPs and to further separate the mixture components by introducing a polymer, which allowed changing the distance between them.

## 2. Experimental

Colloidal  $\text{Zn}_{0.5}\text{Cd}_{0.5}\text{S}$  QDs passivated with 2-mercaptopropionic acid (2-MPA) were synthesized using aqueous synthesis techniques [16, 17]. This approach involves mixing aqueous solutions of precursors for  $\text{CdBr}_2$  (224 mg, 50 ml), and  $\text{Zn}(\text{ClO}_4)_2$  (242 mg, 10 ml), followed by the introduction of 2-MPA (230  $\mu\text{l}$ ) into the reaction mixture and adjusting the pH level to 7 with 1 M of NaOH solution. Then,  $\text{Na}_2\text{S}$  aqueous solution (30 mg, 10 ml) was added to the colloidal solution.

The synthesis of spherical Au NPs was carried out by the Turkevich method [14]. 1.4 ml of 1%  $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7$  solution was added to the boiling 0.01%  $\text{HAuCl}_4$  solution (200 ml). The resulting mixture was boiled for 30 minutes with constant stirring. The SiO<sub>2</sub> shell on the surface of Au NPs (Au/SiO<sub>2</sub> core/shell NPs) was formed by Au NPs surface functionalisation by a monolayer of (3-mercaptopropyl)trimethoxysilane (3-MPTMS) with a subsequent formation of dense SiO<sub>2</sub> layers by sodium metasilicate ( $\text{Na}_2\text{O}(\text{SiO}_2)$ ). For this, 0.4 ml of 0.035% hydrolysed solution of 3-MPTMS was mixed with 30 ml of solution of colloidal Au NPs. Then the  $\text{Na}_2\text{SiO}_3$  aqueous solution (96 mg, 10 ml) was added to the reaction mixture. The flask with the reaction mixture was placed in a

water bath at 60 °C and was held there for 6 hours with continuous stirring.

Hybrid structures were formed by mixing colloidal solutions of  $Zn_{0.5}Cd_{0.5}S$  QDs with Au NPs, Au NPs in the presence of 4 % polymer solution of poly-(diallyldimethylammonium chloride) (PolyDADMAC), and Au/SiO<sub>2</sub> core/shell NPs in an approximate molar ratio  $[v(QDs)]:[v(NPs)]$  of  $\sim 10^4$  mole fraction (m.f.).

The size and morphology of  $Zn_{0.5}Cd_{0.5}S$  QDs, Au NPs, and Au/SiO<sub>2</sub> NPs were determined using a Libra 120 transmission electron microscope (TEM) (Carl Zeiss, Germany). Absorption properties were studied using a USB2000+ spectrometer (Ocean Optics, USA) with a USB-DT light source (Ocean Optics, USA). The luminescence spectra and the luminescence decay kinetics of  $Zn_{0.5}Cd_{0.5}S$  QDs were studied by a USB2000+ and a TimeHarp 260 time-correlated single-photon counting board (PicoQuant, Germany) with a PMC-100-20 module (Becker&Hickl, Germany) with the time resolution of 0.2 ns. A HPL-H77GV1BT-V1BT-V1 diode module with the wavelength of 380 nm was used to stimulate the luminescence.

### 3. Results and discussion

#### 3.1. Structural data

The analysis of TEM images showed that the approach to the synthesis of  $Zn_{0.5}Cd_{0.5}S$  QDs ensures the formation of individual nanocrystals with an average size of  $4.0 \pm 0.5$  nm (Fig. 1a). The existing dispersion in size in  $Zn_{0.5}Cd_{0.5}S$  QDs ensembles  $\sim 35\%$  is due to the chosen approach to colloidal synthesis in aqueous solution.

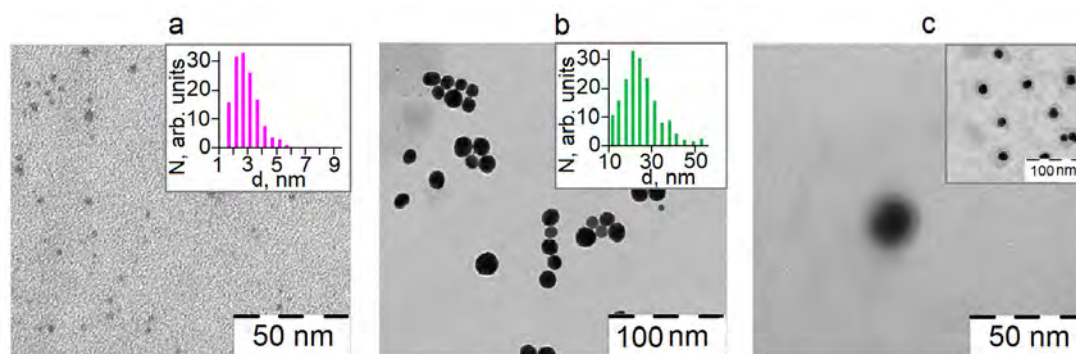
Fig. 1b and c show TEM images of Au NPs and Au/SiO<sub>2</sub> core/shell NPs. It is shown that the

Turkevich method results in the formation of Au NPs with a spherical shape. The average diameter of spherical Au NPs in the ensemble is  $20 \pm 3$  nm with a size distribution within 30% (Fig. 1b). The analysis of TEM images of Au/SiO<sub>2</sub> core/shell NPs (Fig. 1c) showed the formation of a SiO<sub>2</sub> shell on the surface of Au NPs with a thickness of  $10 \pm 03$  nm. There was practically no coagulation of Au/SiO<sub>2</sub> core/shell NPs.

#### 3.2. Spectral-luminescent properties of mixtures of colloidal $Zn_{0.5}Cd_{0.5}S$ QDs and Au, Au/SiO<sub>2</sub> nanoparticles

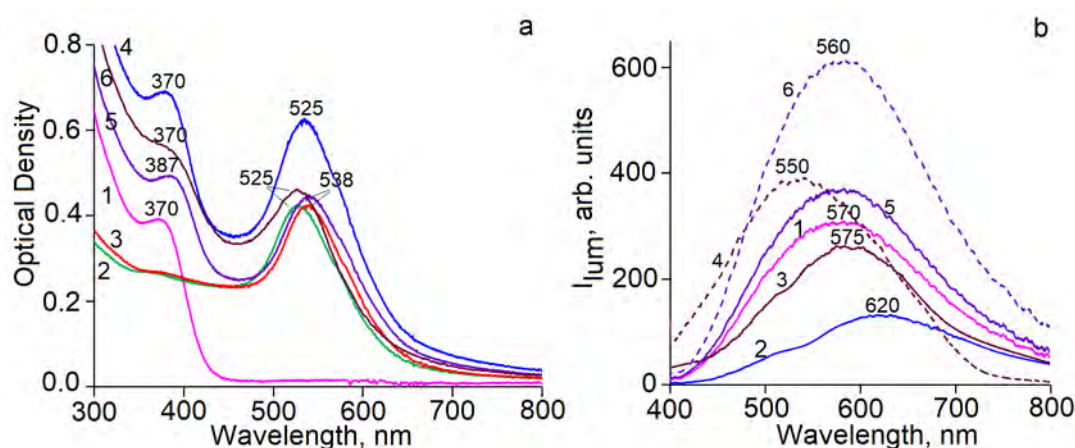
A characteristic feature for the exciton transition [18] in the optical absorption of  $Zn_{0.5}Cd_{0.5}S$  QDs is in the region of 370 nm (Fig. 2a, curve 1). The maximum of the light extinction spectrum for Au NPs is in the range of 525 nm (Fig. 2a, curve 2). Adding 4% PolyDADMAC polymer solution to Au NPs does not influence the position of the light extinction maximum in the region of 525 nm. The formation of a SiO<sub>2</sub> shell with a thickness of 10 nm on the surface of Au NPs leads to a long-wave spectral shift of the maximum of the light extinction spectrum of Au NPs from 525 to 538 nm as a result of changes in the total permittivity of the core/shell system (Fig. 2a, curve 3).

For mixtures of  $Zn_{0.5}Cd_{0.5}S$  QDs and Au NPs, the resulting light extinction spectrum is not a simple sum of the spectra of the mixture's components. The shift of the maximums of the attenuation bands of the mixture components and an increase in optical density throughout the extinction spectrum indicates a strong plasmon-exciton interaction between the components



**Fig. 1.** TEM images and size distribution's histogram of  $Zn_{0.5}Cd_{0.5}S$  QDs - (a); Au NPs - (b); Au/SiO<sub>2</sub> core/shell NPs with a shell thickness of 10 nm - (c)





**Fig. 2.** (a) Optical absorption spectra of Zn<sub>0.5</sub>Cd<sub>0.5</sub>S QDs (1), extinction spectra of Au NPs, Au NPs (PolyDADMAC) (2), Au/SiO<sub>2</sub> core/shell NPs (3), extinction spectra of mixtures of Zn<sub>0.5</sub>Cd<sub>0.5</sub>S QDs and Au NPs (4), Zn<sub>0.5</sub>Cd<sub>0.5</sub>S QDs and Au NPs (PolyDADMAC) (5), Zn<sub>0.5</sub>Cd<sub>0.5</sub>S QDs and Au/SiO<sub>2</sub> core/shell NPs (6). (b) Luminescence spectra of Zn<sub>0.5</sub>Cd<sub>0.5</sub>S QDs (1), mixtures of Zn<sub>0.5</sub>Cd<sub>0.5</sub>S QDs and Au NPs at  $T = 300$  K and  $T = 77$  K (2), Zn<sub>0.5</sub>Cd<sub>0.5</sub>S QDs and Au NPs (PolyDADMAC) at  $T = 300$  K (3) and at  $T = 77$  K (4), Zn<sub>0.5</sub>Cd<sub>0.5</sub>S QDs and Au/SiO<sub>2</sub> core/shell NPs at  $T = 300$  K (5) and at  $T = 77$  K (6)

of the mixture (Fig. 2a, curve 4). In case of the mixtures of Zn<sub>0.5</sub>Cd<sub>0.5</sub>S QDs and Au NPs in the presence of a PolyDADMAC polymer, as well as core/shell Au/SiO<sub>2</sub> NPs, in the resulting extinction spectra there was an increase in the optical density in the region of exciton transition of Zn<sub>0.5</sub>Cd<sub>0.5</sub>S QDs (Fig. 2a, curves 5 and 6) which can be explained not only by a contribution of Au NPs to the overall spectral contour of the light extinction, but a weak interaction between the components of the mixture.

The control of the size and the morphology of mixture components using the applied synthesis methods provided a considerable overlap between Au NPs light extinction peak (525 nm) and Au/SiO<sub>2</sub> core/shell NPs (538 nm) and Zn<sub>0.5</sub>Cd<sub>0.5</sub>S QDs luminescence spectrum (570 nm), which is crucial for the appearance of effects of plasmon-exciton interaction in the luminescent properties of the emitter.

For mixtures of Zn<sub>0.5</sub>Cd<sub>0.5</sub>S QDs and Au NPs, there was luminescence quenching accompanied by a transformation of the spectral contour of QD emission band resulting in a dip in the region of 525 nm (Fig. 2b, curve 2). At the same time, the luminescence lifetime was reduced from 21 ns to 4 ns. Lowering the temperature of the samples to 77 K did not qualitatively change the luminescent properties of the mixtures of Zn<sub>0.5</sub>Cd<sub>0.5</sub>S QDs and Au NPs. Such behaviour

of luminescent properties indicates a complex character of plasmon-exciton interaction in the studied mixtures due to several simultaneous effects, such as the Fano effect [1, 5, 10] and nonradiative recombination [15, 19] caused by changes in the immediate environment of Zn<sub>0.5</sub>Cd<sub>0.5</sub>S QDs. Increasing the distance between Zn<sub>0.5</sub>Cd<sub>0.5</sub>S QDs and Au NPs by a PolyDADMAC polymer led to a decreased quenching of QDs luminescence intensity, also accompanied by a transformation of the spectral contour (Fig. 2b, curve 3). What is more, the luminescence lifetime did not change. Lowering the temperature to 77 K contributed to a hypsochromic shift in Zn<sub>0.5</sub>Cd<sub>0.5</sub>S QDs luminescence band to the region of 550 nm and 1.5 times increase in its intensity (Fig. 2b, curve 4) accompanied by an increase in the luminescence lifetime from 23 to 25 ns. The control of the distance between the mixture components by means of a SiO<sub>2</sub> shell with a thickness of 10 nm on Au NPs surface led to a slight enhancement of QDs luminescent properties at a temperature of 300 K (Fig. 2b, curve 5). At a temperature of 77 K, Zn<sub>0.5</sub>Cd<sub>0.5</sub>S QDs luminescence intensity grew 8 times, whereas the luminescence lifetime rose from 23 to 40 ns (Fig. 2b, curve 6). Such behaviour of luminescent properties can be a manifestation of the Purcell effect complicated by the influence of the effects of exciton-phonon interaction, concentration

quenching, and possible exchange of electronic excitations which always occur in hybrid systems, including those involving a SiO<sub>2</sub> shell [1, 4, 11, 12, 20, 21].

Thus, the spatial distribution of plasmonic Au NPs and Zn<sub>0.5</sub>Cd<sub>0.5</sub>S QDs under strong spectral resonance enables controlling luminescent properties of Zn<sub>0.5</sub>Cd<sub>0.5</sub>S QDs. Obviously, this is due to switching between the modes of the plasmon-exciton coupling. Resonance effects in their pure form are complicated by multiple interfering factors, such as the electron-phonon interaction and the resonant nonradiative energy transfer between the components of the mixture.

### 3. Conclusions

The study demonstrated new experimental effects of interaction between Zn<sub>0.5</sub>Cd<sub>0.5</sub>S QDs and Au NPs resulting from changes in the plasmon-exciton coupling between the mixture components due to the formation of SiO<sub>2</sub> shells with a thickness of 10 nm on Au NPs surface and the introduction of a polymer in the colloidal mixture. It was shown that the formation of mixtures of Zn<sub>0.5</sub>Cd<sub>0.5</sub>S QDs and plasmonic Au NPs is accompanied by the intensity quenching, a transformation of the spectral contour of QD luminescence, and a decrease in the luminescence lifetime. Increasing the distance between the mixture components by introducing a PolyDADMAC polymer and the formation of SiO<sub>2</sub> shell with a thickness of 10 nm on Au NPs surface block the process of luminescence quenching at a temperature of 300 K and lead to an increase in the luminescence intensity at 77 K accompanied by an increase in the luminescence lifetime. The obtained data demonstrate the possibility of controlling Zn<sub>0.5</sub>Cd<sub>0.5</sub>S QDs luminescent properties under conditions of changing modes of plasmon-exciton interaction between the mixtures components. They also clearly indicate a complex character of the exciton-plasmon interaction in the studied mixtures due to several simultaneous effects, such as the Purcell effect, the Fano effect, nonradiative energy transfer from QDs to plasmonic particles, and resonance detuning.

### Conflict of interests

The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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All authors read and approved the final manuscript.

*Received 25 December 2020; Approved after reviewing 15 February 2021; Accepted 15 March 2021; Published online 25 March 2021.*

*Translated by Irina Charychanskaya*

*Edited and proofread by Simon Cox*