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SHAROF RASHIDOV NOMIDAGI SAMARQAND DAVLAT UNIVERSITETI

САМАРКАНДСКИЙ ГОСУДАРСТВЕННЫЙ УНИВЕРСИТЕТ ИМЕНИ ШАРОФА РАШИДОВА

SAMARKAND STATE UNIVERSITY NAMED AFTER SHAROF RASHIDOV

ILMIY AXBOROTNOMA

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OBTAINING SEMICONDUCTOR CdS, CdSe AND CdSe/ZnS CORE/SHELL NANOPARTICLES AND THEIR COMPOSITES WITH POLYMERS

A.F. Ishankulov[1], N.R. Tursunova[1], D.R. Ergasheva[1], K.F. Khalilov[1], Yu.G. Galyametdinov[2], N.K.Mukhamadiev[1]

[1] Samarkand state university named after Sharof Rashidov,
[2]Kazan National Research Technological University
e–mail: Ishankulov-alisher@mail.ru

Abstract. In this work, high-performance CdS, CdSe and CdSe/ZnS nanocrystals with good luminescence were colloidally synthesized at high temperature and incorporated into micrometer-sized polymethylmethacrylate (PMMA) spheres formed by suspension polymerization reaction. CdS, CdSe and CdSe/ZnS quantum dots obtained by the colloid method were stabilized with oleic acid and their optical properties were studied. It was observed that the intensity peaks of quantum dots did not change after modification to PMMA polymer.

Keywords: Quantum dots, colloid synthesis, cadmium selenide, zinc sulfide, polymethylmethacrylate, “core/shell”, nanoparticles, luminescence, precursor.

CdS, CdSe VA CdSe/ZnS NANOZARRALARINI YARIMOQCHILARNING POLİMERLAR BILAN KOMPOZITLARINI TAYYORLASH.

Annotatsiya. Ushbu ishda yaxshi lyuminestsentsiyaga ega yuqori unumli CdSe/ZnS nanokristallari yuqori haroratda kolloid usulda sintez qilingan va suspenziya polimerizatsiya reaktsiyasi orqali hosil bo'lgan mikrometr o'lchamdagi polimetilmetakrilat (PMMA) sharchalariga qo'shilgan. Kolloid usulda olingan CdS, CdSe va CdSe/ZnS kvant nuqtalari olein kislota bilan barqarorlashtirildi va ularning optik xossalari o‘rganildi. PMMA polimeriga modifikatsiyalangandan keyin kvant nuqtalarning intensivlik cho’qilari o’zgarmaganligi kuzatildi.

Kalit so’zlar: Kvant nuqtalari, kolloid sintezi, kadmiy selenid, rux sulfid, polimetilmetakrilat, “yadro/qobiq”, nanozarrachalar, lyuminestsentsiya, prekursor.

ПОЛУЧЕНИЕ ПОЛУПРОВОДНИКОВЫХ НАНОЧАСТИЦ CdS, CdSe И CdSe/ZnS Ядро/ОБОЛОЧКА И ИХ КОМПОЗИТОВ С ПОЛИМЕРАМИ

Аннотация. В этой работе высокоэффективные нанокристаллы CdS, CdSe и CdSe/ZnS с хорошей люминесценцией были коллоидно синтезированы при высокой температуре и включены в полиметилметакрилатные (ПММА) сферы микрометрового размера, образованные реакцией суспензионной полимеризации. Квантовые точки CdS, CdSe и CdSe/ZnS, полученные коллоидным методом, стабилизировали олеиновой кислотой и исследовали их оптические свойства. Было замечено, что пики интенсивности квантовых точек не изменились после модификации полимером ПММА.

Ключевые слова: Квантовые точки, коллоидный синтез, селенид кадмия, сульфид цинка, полиметилметакрилат, «ядро/оболочка», наночастицы, люминесценция, прекурсор**.**

Introduction. In recent years, much attention has been paid to the study of composite organic-inorganic materials. Quantum dots (QDs) are nanosized semiconductor nanoparticles with fluorescent properties whose optical properties change depending on their chemical composition, size and shape. The incorporation of quantum dots into monodisperse polymer microspheres can produce materials

with reproducible properties, which can be used in applications such as optical coding, biological analysis, optical data storage, and sensing [1-3].

Nowadays, along with the growing demand of mankind for new nanotechnologies, chemical nanotechnologies are also developing rapidly [4]. Therefore, in the field of nanochemistry, the scientific literature pays great attention to the development of nanotechnologies and their application in various fields of human activity [5]. In the last decade, the synthesis of nanocrystals of metals and semiconductors, which defines the main direction of research in the development of chemical nanotechnologies, has become particularly important [6].

Among the well studied nanoparticles are cadmium chalcogenides, which have good luminescent properties over a wide range of electromagnetic spectrum. The development of a hybrid "core/shell" type quantum dots can lead to a further increase in the luminescence intensity. In particular, quantum dots consisting of elements group II and VI have a wide band gap and have high photoluminescence efficiency at room temperature [7].

QDs are nanoparticles composed of atoms of semiconductor materials whose electronic properties occupy a range of properties of hybrid structures and discrete molecules. These features are related to the onset of the quantum effect level [8]. The electron transfer band of quantum dots depends on their size, chemical composition, and nature. One way to influence the properties of QDs is to add additional atomic ions to their system. The literature analyzes that the conversion of semiconductor cations into atoms of 3D-transition metals or rare earth elements leads to the formation of magnetic properties in them, and on this basis significantly expands the field of application of a new type of composition quantum dots.

The synthesis of quantum dots with a core/shell structure is formed by the growth of monolayers by adding precursors to the reaction medium. The thickness of the shell is one of the important parameters determining the properties of the crystal, which allows one to determine the yield, stability, and other properties of quantum dots. The nature and composition of a semiconductor as an nucleus play an important role in the synthesis of quantum dots. By changing the composition of the semiconductor, effective luminescence can be achieved in the desired range. Among the most studied nanoparticles among QDs are CdSe nanocrystals with good luminescence properties [9].

Numerous studies on the changing of hydrophobic ligand with hydrophilic have been reported in the paper [10,11]. According to them, the transfer of the obtained quantum dots to the aqueous medium is easier in hydrophilic stabilizers.

The size of nanoparticles also largely depends on the synthesis time. In the colloidal method of synthesis, the stage of nucleation and rapid growth of nanoparticles is followed by the process of Oswald maturation, the end result of which is an increase in the polydispersity of QDs. Therefore, an important task in the synthesis of QDs is to control the stage of maturation of nanoparticles, which will make it possible to obtain monodisperse particles with certain sizes necessary for their practical application.

To this end, the task was set to develop a method for the conversion of hydrophobic ligand to hydrophilic in the process of synthesis of hybrid quantum dots. Through this process, nanoparticles with multifunctional properties can also be obtained.

Material and methods. Cadmium oxide (99%, pure for analysis), selenium (Se), sulfur (S), zinc oxide (ZnO, 99%, pure for analysis), oleic acid (OA, 98%, pure), 1-octadecene (ODE), 90%, pure for analysis), toluene (99%, chemically pure), ethanol (96%), acetone (99%, chemically pure), oleylamine (ODA, 96%).

Methods for obtaining quantum dots in a colloidal environment allow nanoparticles to form in a highly quantum efficient state over a short-range distribution range. In this case, a stabilizer is selected in organic solvents, such as amines, fatty acids, thiols, which ensure the distribution of

quantum dots in different media, depending on the conditions of synthesis, which in turn provides a monodisperse-sized distribution of quantum dots [11].

For the synthesis of PMMA, 40 ml of a 2% aqueous solution of polyvinylpyrrolidone K25 was poured into a 100 ml three-pronged round bottom flask and dissolved in 100 mg of sodium persulfate as an initiator. A mixture of 4 ml of methyl methacrylate, 0.2 ml of acrylic acid, 0.2 ml of divinylbenzene was injected into an aqueous solution using a high mechanical stirrer. Next, the mixture was heated to 65°C under an argon atmosphere using a glycerol bath on a magnetic stirrer. Polymerization was carried out at 1200 rpm for 5 hours. The resulting polymer suspension was diluted 2 more times with distilled water, separated into 3 fractions by centrifugation and washed with distilled water. The first fraction was obtained at a speed of 2000 rpm, the second at a speed of 6000 rpm, and the third at a speed of more than 6000 rpm.

The synthesis of CdSe/ZnS QDs was carried out with partial modification based on the method described by the authors [12,13]. The synthesis temperature was changed to 260°C. The synthesis was carried out in an argon atmosphere in a colloidal manner. A 1:1 mixture of ethanol and acetone was used to purify the reactants. The mixture was separated in a centrifuge at 10000 rpm for 10 minutes (the cleaning process was repeated 3 times). During high-temperature synthesis, oleic acid (OA) was used as an organic solvent octadecen and stabilizer to obtain CdSe/ZnS nanocrystals. To study the optical properties of the obtained nanocrystals, they were dissolved in an organic solvent.

Results and discussion. The obtained nanoparticles have a spectral range of 500-600 nm in the luminescence spectrum. The spectrum shows that the luminescence range is narrow and symmetrical. This suggests that nanocrystals have very few surface defects and are characteristic of colloidal synthesis. The selected synthesis method made it possible to obtain monodispersed quantum dots. The maximum photoluminescence intensity of quantum dots is 555 nm (Figure 2a). The quantum yield of the synthesized hybrid CdSe/ZnS quantum dots was determined by the coumarin method, based on a solution of rhodamine 6G (96%) in ethanol [14]. The quantum yield of hybrid CdSe/ZnS nanocrystals was 19%.

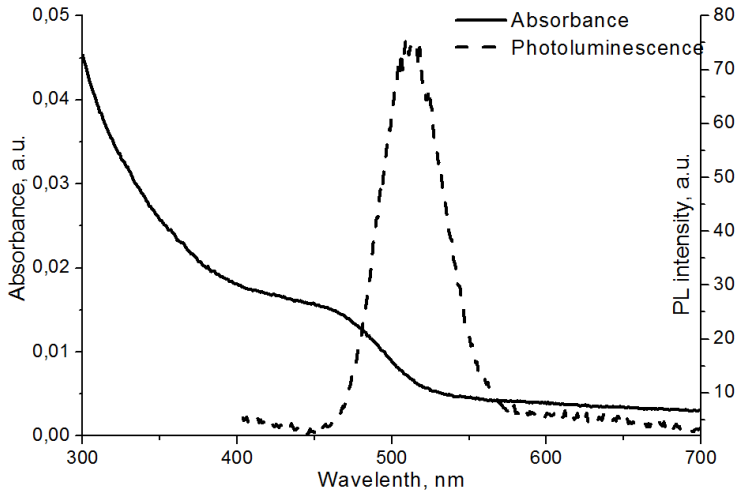


Figure 1. Absorbance and photoluminescence (λ_{ex} = 350 nm) spectra of CdSe QDs.

The photoluminescence and absorbance spectra of the QDs was recorded in n-hexane (Fig. 2). The maximum luminescence intensity was at 513 nm at wavelength excitation 350 nm.

As can be seen from the absorption spectra of the quantum dots CdSe/ZnS (Figure 2b), an exciton peak of 537 nm is observed in the field of view. The field of view of CdSe quantum dots is 500-600 nm. corresponds to the wavelength.

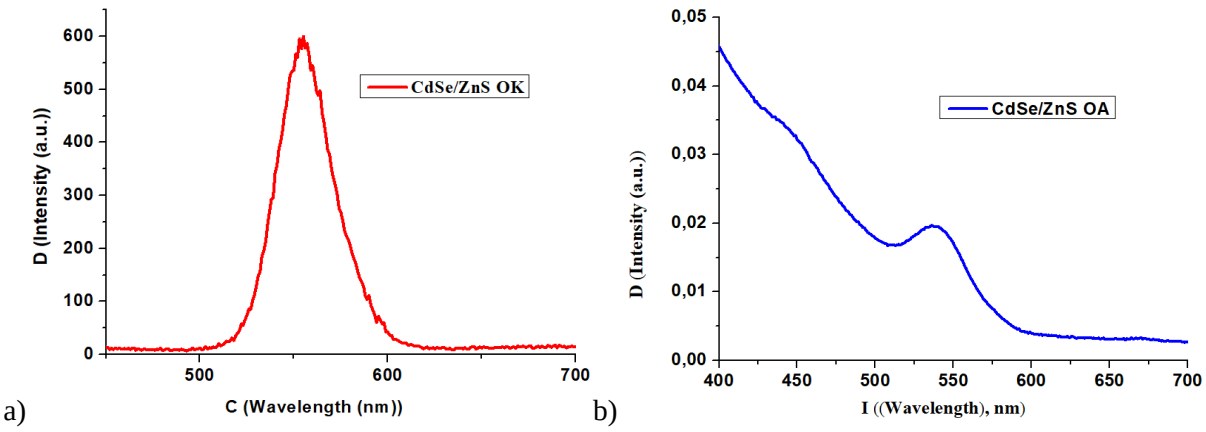


Figure 2. Luminescence (a) and absorption (b) spectra of CdSe/ZnS QDs

The synthesis of CdSe QDs was performed with partial modifications based on the method described by the authors [6,7].

The method recommended by the authors [8,9] was used to grow the ZnS shell around the CdSe nucleus.

To prepare a solution of zinc oleate (0.1M), a solution prepared from a mixture of 1.41 ml of oleic acid (OA) of 0.0405 g ZnO and 3.6 ml of octadecene was prepared by heating at 260°C. A precursor of a sulfur (0.1M) solution was obtained by dissolving 0.016 g of sulfur in 5 ml of octadecene at 200°C.

A 20 ml solution was prepared by adding 5 ml of octadecene to a solution of CdSe ($6.6 \cdot 10^{-6}$ mol/l) QD in toluene and heated to 200°C until the toluene in the mixture evaporated. Then, 2.2 ml of oleilamine and 4.7 ml of zinc oleate solution were added and mixed for 15 min. A total of 4.7 ml of sulfur precursor solution was added to the reaction mixture every 15 minutes. The mixture is stirred in a magnetic stirrer for 30 min at 200°C so that the components in the mixture are fully reactive. The purification process is performed in the same order as for CdSe quantum dots.

Synthesis was performed on the surface of CdSe nanocrystals based on changes in the addition time of sulfur precursors (for 3, 7, 10, 15 minutes) during ZnS shell growth.

Oleic acid was used as a stabilizer in the synthesis of CdSe QDs and was synthesized at high temperatures in an octadecene medium.

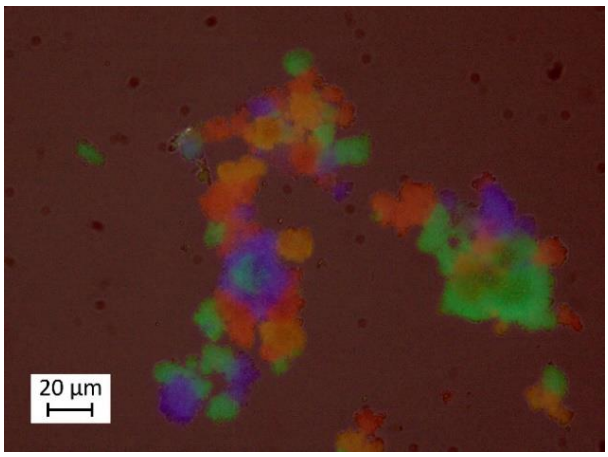


Figure 3. Optical microscope image of CdSe/ZnS and CdSe/CdS/ZnS QDs irradiated with laser light

The obtained hybrid CdSe/ZnS (size 2.8 nm) and CdSe/CdS/ZnS (size 4.7 nm) quantum dots and PMMA were mixed in a ratio of 1:1, 3.6 ml of propyl alcohol was added to it and stirred for 3 hours on a magnetic stirrer. mixed up. A 1:1 mixture of hexane and propanol was used to purify the unreacted starting materials. It was then dissolved in 5 ml of distilled water.

Conclusions. Oleic acid-stabilized hybrid CdSe/ZnS nanocrystals were synthesized in a colloidal manner. The selected synthesis method made it possible to obtain monodispersed quantum dots. The absorption and luminescence spectra of the hybrid CdSe/ZnS quantum dot have moved to a region with shorter wavelengths.

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