

# One-step synthesis of sulfur quantum dots in the presence of primary, secondary, and tertiary amines

Dmitriy Shurpik , Inna Tanaeva , Olga Mostovaya , Ivan Stoikov\* 

A.M. Butlerov Chemical Institute, Kazan Federal University, Kazan 420008, Russia

\* Corresponding author: [ivan.stoikov@mail.ru](mailto:ivan.stoikov@mail.ru)



## Abstract

Quantum dots synthesized on the metal basis and the materials covering them can be immunogenic, which can lead to dangerous consequences for the research objects. It is possible to avoid these limitations by using metal-free quantum dots. Among the known non-metallic nanomaterials, sulfur quantum dots are of particular interest. Now the use of sulfur quantum dots as sensor systems is limited due to the absence of receptor groups in their structure. This problem can be solved by using compounds containing polyfunctional fragments as a passivation agent or base in the synthesis of quantum dots. In this study, we developed a method for obtaining sulfur quantum dots using primary and secondary amines. Using fluorescence spectroscopy, it was shown that the fluorescence intensity changes depending on the reaction time. Thus, in the case of a reaction time of 125 h, the maximum fluorescence values are observed for all the studied samples. The highest intensity among all the samples is characteristic of sulfur quantum dots based on pyrrolidine, which acts as a base. In this case, the lowest sizes of sulfur quantum dots were recorded (3 nm, polydispersity index = 0.28). The feasibility of using pyrrolidine as the main reagent for the synthesis of sulfur quantum dots has been revealed.

## Key findings

- A method for producing sulfur quantum dots using primary and secondary amines was developed.
- After 125 h of reaction progress, the maximum fluorescence intensity of sulfur quantum dots was observed.
- The lowest sizes (3 nm) were found for sulfur quantum dots.

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## 1. Introduction

Quantum dots are semiconductor nanocrystals with sizes close to the wavelength of an electron (1–10 nm) [1–4]. Inside a quantum dot, the potential energy of electrons is lower than that outside it, so their movement is limited in all three directions [1]. Techniques for the creation and application of quantum dots are widely used in modern laser technologies, pharmaceuticals, and medicine [1].

Currently, quantum dots are synthesized based on elements from groups II and VI (CdTe, CdS, CdSe, ZnS, ZnSe, etc.), less often III and V (InP, InAs) or IV and VI groups (PbS, PbSe, PbTe) of the periodic table [5]. The introduction of organic molecules such as trioctyl phosphine oxide (TOPO) or thioglycolic acid (TGA), capable of adsorbing on

the surface of quantum dots, helps to stabilize these nanoobjects and make them stable in various environments [5].

Despite the use of metal quantum dots in medicine and analysis of biological systems, these nanomaterials have a number of disadvantages that seriously limit their use both in vitro and in vivo [6–8].

Metal-based quantum dots and the materials they are coated with may be immunogenic, with potentially dangerous consequences for the organism [8]. Another factor that hinders their use is the core material – heavy metals with toxic properties. For example, under the influence of UV radiation, CdSe quantum dots become highly toxic to cells, since during photolysis the particles dissolve with the release of cadmium ions. Also, the desorption of cadmium

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ions is facilitated by the effect of an oxidizing environment on the core of the quantum dot. Toxicity also occurs when free radicals are generated by the surface of the nucleus [9]. It is possible to circumvent these limitations by using metal-free quantum dots. Among the known non-metallic nanomaterials, sulfur quantum dots are of particular interest due to their unique chemical properties and biological activity [10–12].

In 2018, Lihua Shen and his colleagues [10] proposed a new method for synthesizing sulfur quantum dots via the «assemble-fission» mechanism, which involved treating sulfur powder with alkali using polyethylene glycol-400 (PEG-400) as a passivation agent at 70 °C. The optical properties of the reaction mixture and the change in the size and shape of quantum dots were carefully studied for 125 h using transmission electron microscope images to establish the mechanism of their formation. To date, the use of sulfur quantum dots as sensor systems is limited due to the absence of receptor groups in their structure. This problem can be solved by using compounds containing polyfunctional fragments as a passivation agent or base in the synthesis of quantum dots.

Here we present a concept for the synthesis of sulfur quantum dots using a series of primary, secondary, and tertiary amines as a base.

## 2. Materials and methods

The absorption spectra were recorded on a Shimadzu UV-3600 spectrometer; the cell thickness was 1 cm, and the slit width was 1 nm. Deionized water with a resistivity of >18.0 MOhm-cm was used to prepare the solutions. The deionized water was obtained from a Millipore-Q purification system. The absorption spectra of all sulfur quantum dot samples were recorded by diluting the solutions 300 times at 293 K. Fluorescence spectra were recorded on a Fluorolog 3 fluorescence spectrometer (Horiba Jobin Yvon). The excitation wavelength  $\lambda_{\text{ex}} = 360$  nm, and the emission scanning range was 400–600 nm. The excitation and emission slits were 1.5 nm. Quartz cuvettes with an optical path length of 10 mm were used. Fluorescence spectra were automatically corrected by the Fluorescence program. Deionized water obtained from the Millipore-Q purification system was used as a solvent. The experiments were carried out at 293 K. The particle size distribution by intensity, volume, number, and the polydispersity index in the solution were determined by dynamic light scattering on a Zetasizer Nano ZS nanoparticle size analyzer (Malvern) at a temperature of 20 °C, the scattered light detection angle was 173° in polystyrene cuvettes, with a 4 mW He-Ne laser and a wavelength of 633 nm. The error in size determination was less than 2%.

Transmission electron microscopy (TEM) analysis of the S/PEG-400/pyrrolidine ( $1 \cdot 10^{-4}$  M) system in water was performed using an Exalens transmission electron microscope with an Oxford Instruments X-Maxn 80T EDS detector. For

sample preparation, 10  $\mu\text{l}$  of the suspension was placed on a 3 mm diameter Formvar<sup>TM</sup>/carbon coated copper grid, which was then dried at room temperature. After complete drying, the grid was mounted in the transmission electron microscope using a special microanalysis holder. The analysis was performed at an accelerating voltage of 80 kV in STEM mode using an Oxford Instruments X-Maxn 80T EDS detector.

Most of the reagents were purchased from Aldrich and used without further purification. The following reagents and solvents were used in the course of the work: sulfur, polyethylene glycol-400, hydrogen peroxide 30%, acetic acid 50%, dimethylamine, triethylamine, pyrrolidine, piperidine, hexane-1,6-diamine, ethylenediamine, diethylenetriamine, *N,N*-dimethylethylenediamine, *N,N*-diethylethylenediamine with 99.9% purity and distilled water, deionized water.

### 2.1. General method for the synthesis of sulfur quantum dots using amines as bases and PEG-400 as a passivation agent

A round-bottom flask equipped with a magnetic stirrer was charged with sulfur powder (0.70 g;  $2.19 \cdot 10^{-2}$  mol), PEG-400 (1.5 ml), dimethylamine (33%; 7.5 ml; 0.913 g/cm<sup>3</sup>, 0.15 mol)/ triethylamine (100%; 7.0 ml; 0.728 g/cm<sup>3</sup>; 0.05 mol)/ pyrrolidine (100%; 4.1 ml; 0.866 g/cm<sup>3</sup>; 0.05 mol)/ piperidine (100%; 4.9 ml; 0.862 g/cm<sup>3</sup>, 0.05 mol)/ hexane-1,6-diamine (5.8 g; 0.05 mol)/ ethylenediamine (100%; 3.3 ml; 0.899 g/cm<sup>3</sup>; 0.05 mol)/ diethylenetriamine (100%; 5.4 ml; 0.955 g/cm<sup>3</sup>; 0.05 mol)/ *N,N*-dimethylethylenediamine (100%; 5.5 ml; 0.807 g/cm<sup>3</sup>, 0.05 mol)/ *N,N*-diethylethylenediamine (100%; 7.0 ml; 0.827 g/cm<sup>3</sup>, 0.05 mol) and deionized water was added, bringing the volume of the solution to 26.5 ml. The reaction was carried out for 125 h at 70 °C. Then the reaction mixture was divided into two equal parts of 13.25 ml. Hydrogen peroxide (30%) was slowly added to one of them to etch polysulfide anions from the surface of quantum dots, and then the solution was brought to a neutral medium with acetic acid (50%).

## 3. Results and Discussion

Previously, Jothi Vinoth Kumar [13] and co-workers synthesized highly emissive blue sulfur quantum dots (SQDs) using elemental sulfur and ethylenediamine at 60 °C for 7 h by hydrothermal acceleration method. The synthesized SQDs were used in this study as fluorescent probes for the quantitative detection of Hg<sup>2+</sup> [13].

The successful application of ethylenediamine in the synthesis of sulfur quantum dots allowed to hypothesize that the use of primary, secondary, and tertiary amines as a base (Figure 1) ought to solve two problems simultaneously. Firstly, the size of the synthesized nanoparticles might be regulated by varying the nature of the organic base. Secondly, synthesis conditions will be developed for

obtaining quantum dots framed by polyfunctional compounds. In this regard, in order to replace sodium hydroxide traditionally used in the synthesis of sulfur quantum dots with organic bases, some aliphatic amines were selected: dimethylamine ( $pK_a = 11.094$ ), triethylamine ( $pK_a = 10.75$ ), pyrrolidine ( $pK_a = 11.31$ ), piperidine ( $pK_a = 11.31$ ), hexane-1,6-diamine ( $pK_a = 10.93$ ), ethylenediamine ( $pK_a = 9.98$ ), diethylenetriamine ( $pK_{a1} = 10.101$ ,  $pK_{a2} = 9.386$ ,  $pK_{a3} = 4.889$ ), *N,N*-dimethylethylenediamine ( $pK_{a1} = 9.53$ ,  $pK_{a2} = 6.63$ ), *N,N*-diethylethylenediamine ( $pK_{a1} = 10.02$ ,  $pK_{a2} = 7.07$ ). The synthesis of sulfur quantum dots in the presence of the above amines and PEG-400 was carried out at 70 °C for 24, 48, 72 and 125 h, followed by etching of the polysulfides (Figure 1). The optical properties

of the obtained nano-sized structures were studied by UV-vis spectroscopy. Thus, the presence of polysulfide anions ( $S_x^{2-}$ ) on the surface of sulfur quantum dots was shown. In the absorption spectra of quantum dot samples synthesized in the presence of amines and PEG-400, an absorption band at  $\lambda = 303$  nm corresponding to  $S_2^{2-}$ -anions and an absorption band at  $\lambda = 370$  nm indicating the presence of  $S_8^{2-}$ -anions (Figure 2a) are observed in an alkaline environment, which is in good agreement with the data obtained in the study of classical sulfur quantum dots [13].

It is worth noting that the use of triethylamine and *N,N*-diethylethylenediamine as bases did not lead to the formation of fluorescent SQDs.

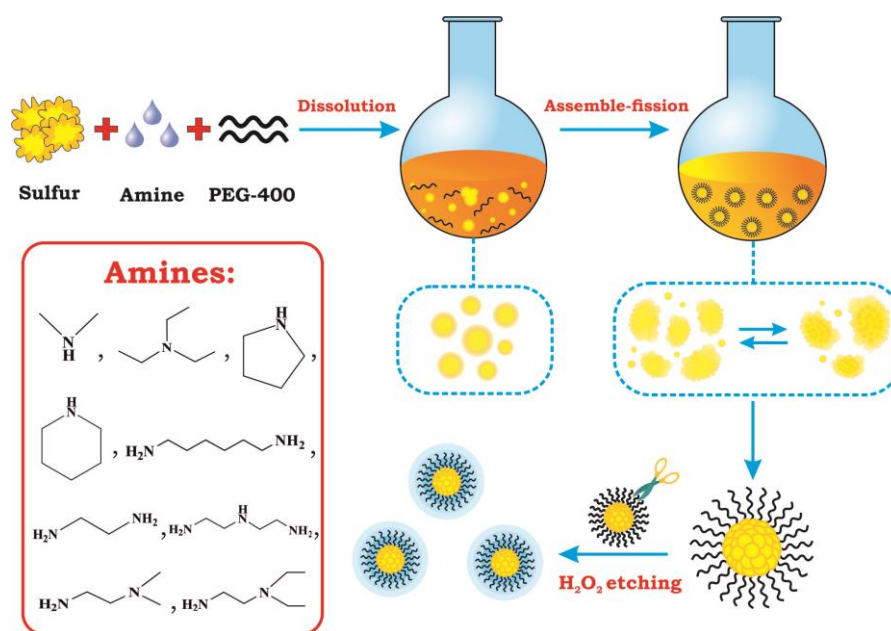


Figure 1 Scheme for the preparation of SQDs using a series of primary, secondary, and tertiary amines as a base.

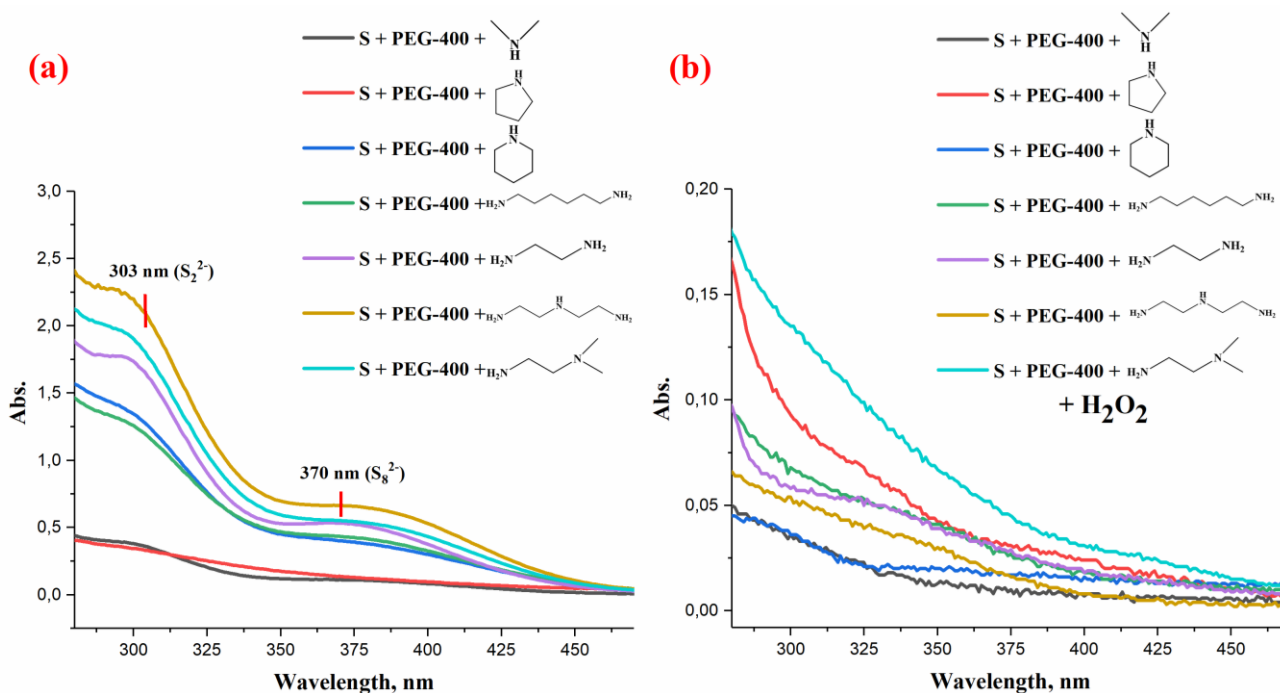


Figure 2 Absorption spectra of sulfur quantum dot samples synthesized in the presence of amines and PEG-400: in an alkaline environment (a); in a neutral environment (b).

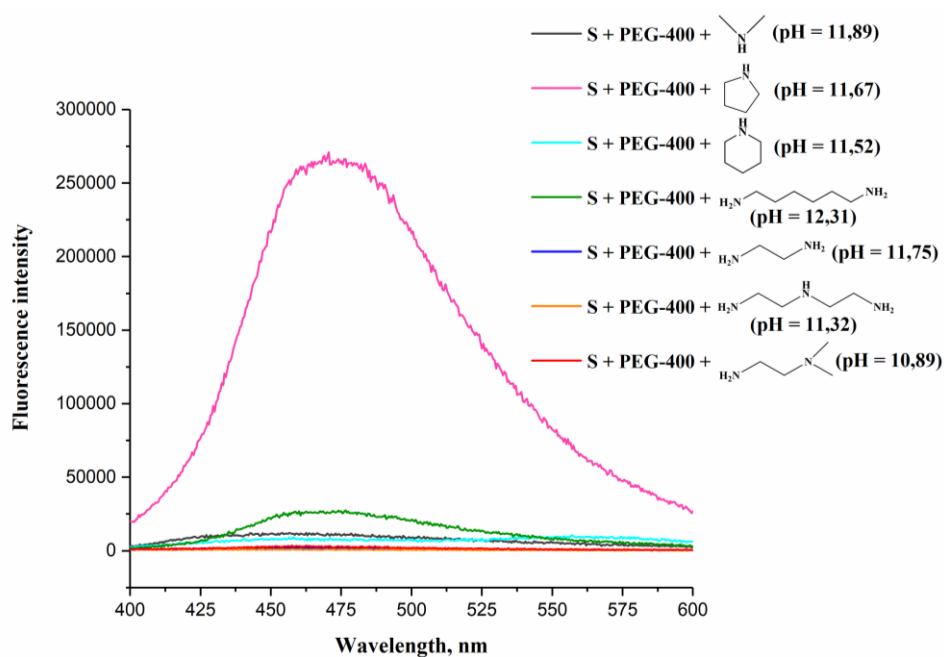
Henggang Wang et al. [14] proposed a «top-down» synthetic approach using  $H_2O_2$  to etch polysulfides to obtain highly luminescent color-controlled emission sulfur quantum dots in order to increase the luminescence quantum yield of SQDs. After adding hydrogen peroxide (30%) to the system, the previously observed absorption bands disappeared (Figure 2b), indicating that polysulfides were etched from the surface of the nanocrystals.

Then the optical properties of the obtained sulfur-based quantum dots were studied by fluorescence spectroscopy. It was found that with an increase in the synthesis time to 24, 48, 72, and 125 h, the fluorescence intensity of all the studied systems increases. However, in an alkaline environment, the highest emission intensity ( $I = 270955$  a. u.) is possessed by the quantum dot sample obtained after 125 h

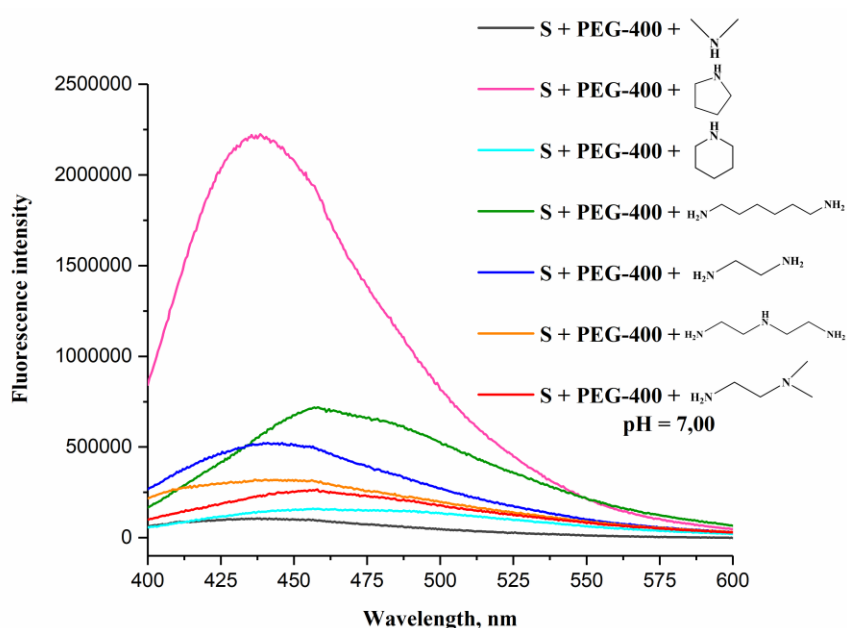
of reaction in the presence of pyrrolidine. The lowest fluorescence intensity values are exhibited by the samples synthesized for the same time in the presence of ethylenediamine ( $I = 2502$  a. u.) and diethylenetriamine ( $I = 1280$  a. u.) (Figure 3). This is probably due to the weak basic properties of the polyamines used.

The  $pK_a$  values for ethylenediamine (9.98) and diethylenetriamine ( $pK_{a1} = 10.101$ ,  $pK_{a2} = 9.386$ ,  $pK_{a3} = 4.889$ ) characterize them as weak bases. It is well known that the weaker the base chosen for the synthesis of sulfur quantum dots, the worse fluorescent properties it exhibits [10].

After etching polysulfides from the surface of quantum dots with hydrogen peroxide (30%) and bringing the environment to neutral using glacial acetic acid, the fluorescence intensity of all samples increases sharply (Figure 4).



**Figure 3** Fluorescence spectra of sulfur quantum dot samples synthesized in the presence of amines and PEG-400 in an alkaline environment.



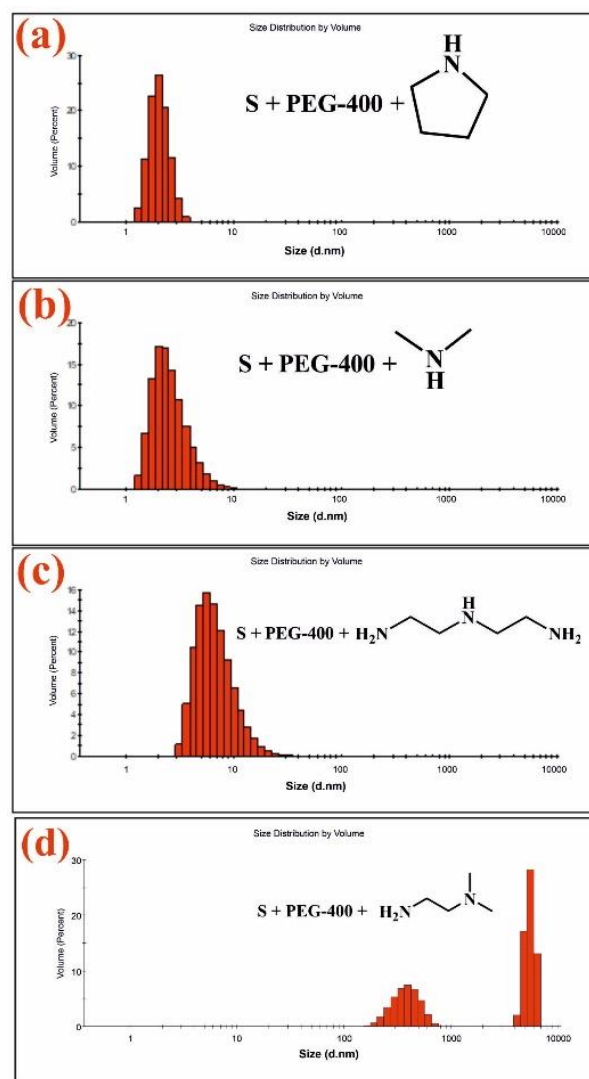
**Figure 4** Fluorescence spectra of sulfur quantum dot samples synthesized in the presence of amines, PEG-400 and  $H_2O_2$  in a neutral environment.

This fact is associated with a possible decrease in the size of the obtained nanomaterials after etching polysulfide anions. It is worth noting that for samples of sulfur quantum dots etched with hydrogen peroxide (30%), in a neutral environment, a shift in the emission maximum to a shorter-wavelength region of the spectrum is observed, which confirms the influence of the “size focusing” process on the fluorescent properties (Table 1). “Size focusing” means adjusting the size of the resulting particles by varying the synthesis conditions or by chemical or mechanical modification of the already prepared nanostructures [14, 15].

The presence of basic amines in the SQDs structure can allow regulating the size of the synthesized nanoparticles by the nature of the organic base. The self-association of SQDs synthesized in the presence of various amines was studied in an aqueous medium using the dynamic light scattering method. It was found that the smallest values of the SQDs size distribution are observed in the case of using pyrrolidine as a base (Figure 5) in the studied concentration range ( $10^{-3}$ – $10^{-6}$  M).

Thus, at a concentration of  $10^{-3}$  M, monodisperse associates with an average particle size of 3 nm and a polydispersity index (PDI) = 0.28 are formed (Figure 5a). In the case of dimethylamine, according to the DLS data, monodisperse associates with an average particle size of 6 nm and a PDI = 0.31 are observed (Figure 5b).

Also, SQDs up to 10 nm were observed in the S/PEG-400/hexane-1,6-diamine system, PDI = 0.29 (Figure 5c). It should be noted that in the case of all other systems, when using other amines, polydisperse systems were formed. It is important to note that systems with a polydisperse distribution with large particles do not have fluorescent properties (Figure 3, 5d). Apparently, this is due to the higher lipophilicity of amines and, as a consequence, the aggregation of the resulting SQDs.



**Figure 5** Particle size distribution by volume for systems: (a) S + PEG-400 + pyrrolidine; (b) S + PEG-400 + dimethylamine; (c) S + PEG-400 + hexane-1,6-diamine; (d) S + PEG-400 + *N,N*-dimethylethylenediamine.

**Table 1** Wavelengths at which the maximum fluorescence intensity is observed in alkaline and neutral media for sulfur quantum dot samples synthesized in the presence of some aliphatic amines.

Amine	Alkaline environment			Neutral environment		
	$\lambda_{ex}$ , nm	$\lambda_{em}$ , nm	Fluorescence Intensity, a.u.	$\lambda_{ex}$ , nm	$\lambda_{em}$ , nm	Fluorescence Intensity, a.u.
	360	455	12278	360	438	106946
	360	471	270955	360	439	2224161
	360	557	10634	360	554	160173
	360	477	27452	360	457	718732
	360	452	2502	360	441	522817
	360	447	1280	360	441	320299
	360	463	3577	360	458	266870

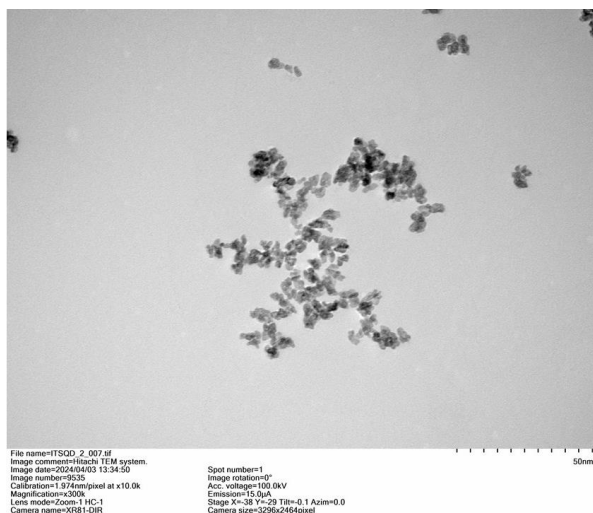
The results obtained allow us to conclude that pyrrolidine is the best of the considered bases for the formation of sulfur quantum dots with the smallest sizes and high fluorescence brightness.

The morphology of the obtained SQDs of the S/PEG-400/pyrrolidine system was determined by transmission electron microscopy (TEM) (Figure 6). According to TEM data, the SQDs S/PEG-400/pyrrolidine system has a morphology similar to the SQDs previously described in the literature [10]. Thus, in an aqueous solution of the SQDs S/PEG-400/pyrrolidine system, dense associates are formed, consisting of individual quantum dots with a shape close to spherical and an average diameter of 4 nm. It is worth noting that the results obtained by TEM are in good agreement with the DLS data, where SQDs S/PEG-400/pyrrolidine are monodisperse associates (PDI = 0.28) with an average particle size of 3 nm. The size difference of 1 nm is within the error limits of these methods.

We assume that the morphology and sizes of the obtained particles directly depend on the dissolution of sulfur, since the better the sulfur dissolves, the more homogeneous and monodisperse the particles will be at the final stage of the synthesis. Of all the aliphatic amines used in the synthesis, pyrrolidine is the strongest base ( $pK_a = 11.31$  at 25 °C), in which environment sulfur probably dissolves in the most favorable way for obtaining particles with the smallest sizes.

#### 4. Limitations

The development of a method for synthesizing sulfur quantum dots using polyfunctional amines was carried out with the aim of further expanding the areas of application of quantum dots. In this study, we developed a method for obtaining metal-free sulfur quantum dots. Dimethylamine, triethylamine, pyrrolidine, piperidine, hexane-1,6-diamine, ethylenediamine, diethylenetriamine, *N,N*-dimethylethylenediamine, *N,N*-diethylethylenediamine were used as a base in the synthesis of quantum dots.



**Figure 6** TEM images of aqueous solution of SQDs system S/PEG-400/pyrrolidine ( $1 \cdot 10^{-4}$  M).

Quite a long synthesis time requires further study of the methods for obtaining sulfur quantum dots. Optimization of the studied approach will allow to subsequently develop a method for obtaining sulfur quantum dots in a shorter period of time and with more outstanding luminescent characteristics. In the future, we plan to study the receptor properties of the obtained compounds that have amine groups in their structure, which will significantly expand the area of potential application.

#### 5. Conclusions

Thus, a method for obtaining sulfur quantum dots using primary and secondary amines was developed. The structure of all the obtained quantum dots was confirmed by a set of physicochemical methods: UV-vis and fluorescence spectroscopy, as well as dynamic light scattering and transmission electron microscopy. The fluorescence spectroscopy method showed that the fluorescence intensity changes depending on the reaction time. It was found that in the case of using pyrrolidine as a base ( $pK_a = 11.31$ ), after the reaction lasted for 125 h, the highest emission intensity ( $I = 270955$  a. u.) was observed. At the same time, the smallest values of the SQDs sizes (3 nm) with a polydispersity index of 0.28 were recorded. The fluorescent properties and sizes of SQDs are in good agreement with the characteristics of classical quantum dots [10]. Thus, the obtained results will allow expanding the range of available reagents for the synthesis of SQDs, regulating the size of the synthesized nanoparticles, and also, due to the framing of the shell with polyfunctional compounds, using SQDs more efficiently.

#### Supplementary materials

No supplementary materials are available.

#### Data availability statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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#### Author contributions

Conceptualization: D.N.S., I.I.S.  
Data curation: I.V.T., O.A.M.  
Formal Analysis: I.V.T., O.A.M.  
Funding acquisition: I.I.S.  
Investigation: D.N.S.  
Methodology: D.N.S.  
Project administration: I.I.S.  
Resources: D.N.S., I.I.S.  
Software: I.V.T., O.A.M.  
Supervision: I.I.S.  
Validation: I.V.T., O.A.M.  
Visualization: D.N.S.  
Writing – original draft: D.N.S., I.I.S., I.V.T.  
Writing – review & editing: D.N.S., I.I.S., I.V.T.

### Conflict of interest

The authors declare no conflict of interest.

### Additional information

Author IDs:

Dmitriy N. Shurpik, Scopus ID [56399350900](https://orcid.org/0000-9142-9000);

Ivan I. Stoikov, Scopus ID [6602887534](https://orcid.org/0000-9142-9000).

Websites:

Kazan (Volga region) Federal University, <https://eng.kpfu.ru>;

Institute of Chemistry, <https://kpfu.ru/eng/academic-units/natural-sciences/alexander-butlerov-institute-of-chemistry>.

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