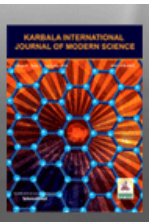
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Photoluminescence Intensity Enhancement and Stability in CdTe/SiO2 Quantum Dots through Water Molecule Adsorption and Trap Passivation

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Abstract

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Keywords

Photoluminescence intensity enhancement; nanoparticles; Nanocrystals; CdTe/SiO2; Traps

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RESEARCH PAPER

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Abstract

The study of the luminescence photostability for colloidal nanocrystals is an important task since the understanding of the corresponding physical processes advances new electronic devices based on semiconductor nanoparticles as well as other important applications such as biomarkers. In this paper, we provide the first study and comprehensive analysis of the photostability of the luminescent properties for colloidal CdTe/SiO_2 core/shell quantum dots prepared by an aqueous-based method. The quantum dots were exposed to continuous laser radiation during two time intervals with prolonged break in between. The photoluminescence intensity of the quantum dots increased over time under continuous laser irradiation. Also, some processes occurred in the quantum dots in the dark (during the break), leading to a further increase in the photoluminescence intensity after turning on the laser radiation in the second time interval. The observed continuous photoluminescence intensity enhancement of the CdTe/SiO_2 quantum dots was attributed to adsorption of water molecules on the surface of the nanocrystals and, as a consequence, to a decrease in the probability of nonradiative transitions. The positions of the photoluminescence intensity maximum and the colorimetric characteristics have been found to be stable against prolonged laser irradiation. This has been explained by the fact that for the CdTe core capped with a large-bandgap SiO_2 shell (with respect to the bandgap of CdTe), photocorrosion, which is often responsible for the PL blueshift, is a slow process. The results of our work can be used in the development of optoelectronic and nanophotonic devices based on colloidal CdTe/SiO_2 nanostructures.

Keywords: Photoluminescence intensity enhancement, Nanoparticles, Nanocrystals, CdTe/SiO_2, Traps

1. Introduction

Colloidal semiconductor nanocrystals such as quantum dots (QDs) [1–8], nanowires [9–11], nanoplatelets [12–16], and nanoscrolls [17,18] have attracted much attention due to the quantum confinement effect, which provides them with unique linear and nonlinear properties [19–23], which are governed by the average size of the nanocrystals [24,25]. QDs are of particular interest since excitons are confined in three dimensions. Colloidal nanoparticles are used in various fields of science and technology, and have been shown to be a strong counterpart to organic and organometallic phosphors [26–28] in many applications, e.g. LEDs [29,30]. They are also promising for lasers [31,32], biosensors [33,34], and photonic devices [35,36]. However, for the correct functioning of electronic

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devices based on colloidal nanoscrystals and, in particular, QDs, various factors (temperature, electric field, and temporal behaviour) should be considered and the mechanisms that lead to changes in the optical and colorimetric properties of QDs should be unveiled. To date, it has been established that changes in temperature can lead to shifts in photoluminescence (PL) spectra [37,38], which directly affects the emission color. An external electric field can lead to both enhancement [39–41] and quenching [42] of the PL intensity, as well as to the PL redshift due to the Stark effect [42]. Also, different changes in the optical properties of colloidal nanocrystals can occur over time. For example, PL photodegradation can be observed [43–45]. Effect of the environment is also a crucial problem which has been addressed in many studies. Thus, the photostability of nanocrystals has been studied in air [46], in dry air [47], in an aqueous solution [48], in nitrogen [49], in dry nitrogen [50], and in argon [50]. In these studies, photodegradation processes have been discovered. The opposite effect of photodegradation is PL intensity enhancement [51]. PL enhancement is often achieved by additional passivation of the surface of nanostructures, which can eliminate surface defects which are nonradiative recombination centers [51]. There are a number of studies reporting an increase in the emission intensity of colloidal nanostructures on a glass substrate under continuous laser irradiation in air and in nitrogen [52–54]. The shell coating contributes to the increase in the photostability and quantum yield of colloidal nanoparticles due to the effective localization of an exciton at a certain distance from the nanocrystal surface [55]. The quantum yield of colloidal nanocrystals covered with a semiconductor shell can reach 85–95% [56]. However, the shell does not always provide complete elimination of various negative effects in nanocrystals. Thus, the study of luminescence photostability for colloidal nanocrystals is an important task since the understanding of the corresponding physical processes advances the efficiency of new electronic devices based on semiconductor nanoparticles. For example, in Ref. [57], almost complete photostability of the bandgap of CdTe/SiO_2 QDs at elevated temperatures has been found.

In the present study, we first address the photostability of PL intensity of CdTe/SiO_2 QDs under continuous laser irradiation to reveal the corresponding mechanisms responsible for their emission behavior.

2. Materials and methods

2.1. Synthesis

Colloidal synthesis of the QDs is based on the use of organic ligands with a thiol group and their subsequent substitution [58]. Tellurium precursor solution was obtained by mixing 0.25 mmol of a TeO_3 powder and 25 ml of water in a flask at room temperature. Then, a $NaBH_4$ powder was added into the flask until a pink-colored solution was obtained. This resulted in a solution of the tellurium precursor. The cadmium precursor was a salt of cadmium and 3-mercaptopropionic acid. For its synthesis, 0.25 mmol of $CdBr_2$ was dissolved in another flask containing 50 ml of water at 30 °C. Then, 0.675 mmol of 3-mercaptopropionic acid (3-MPA) was added. The solution became milky white. After that, the pH was increased to 10 by adding 1 mol/l of NaOH. In this case, the solution became transparent, indicating the formation of the Cd/3-MPA cadmium precursor. Then, the tellurium precursor solution was mixed with the Cd/3-MPA solution with constant stirring by a magnetic stirrer rotating at 300 rpm. The temperature of the solution during mixing was 30 °C. To ensure complete reaction, the prepared mixture was kept at 30 °C for 30 min with constant stirring. To remove the reaction products, acetone was added to the colloidal solution of the CdTe/3-MPA QDs in equal quantities (5 ml of acetone was added to 5 ml of CdTe QDs). The resulting mixture was centrifuged at 5000 rpm for 15 min and re-dissolved in 5 ml of distilled water. The purification procedure was repeated 3 times. The silica shell was formed on the surface of the QDs using an aqueous synthesis. The purified CdTe/3-MPA QDs were dissolved in 250 mL of distilled water and placed in a three-necked flask. To coat the QDs with a SiO_2 layer, an aqueous solution of 1 mmol of 3-mercaptopropyltrimethoxysilane was added to the QD solution. 3-mercaptopropyltrimethoxysilane was preliminarily dissolved in 50 mL of distilled water at 25 °C and kept for 5 h. After adding the 3-mercaptopropyltrimethoxysilane solution to the QDs, the mixture was stirred for 2 h at 25 °C. This provided the replacement of 3-MPA with 3-mercaptopropyltrimethoxysilane at the QD interface. Thus, a SiO_2 monolayer was formed. To form a thicker shell, 50 mmol of sodium metasilicate was then added to the QD solution and the solution was kept under constant stirring at 300 rpm with a magnetic stirrer at 25 °C for another 2 h. The product CdTe/SiO_2

QDs were purified from the reaction mixture using the method described above (precipitated with acetone and centrifuged).

2.2. Morphology and absorption measurements

The size distribution and morphology of the synthesized QDs were studied using a JEM-2100 transmission electron microscope (TEM; JEOL, Japan).

The absorption spectrum of the QD aqueous solution was acquired by a Lambda 45 UV/Vis spectrophotometer (Perkin-Elmer, USA) within the 200–600 nm wavelength range.

The X-ray diffraction pattern was measured within 2 Theta = 10°–70° using a DRON-4-07 diffractometer with Cu K_α radiation.

The FTIR spectrum was obtained with an FSM 2202 instrument operating within the 370–7800 cm^{-1} range at room temperature using a KBr substrate.

2.3. PL measurements

PL spectra were recorded at room temperature for QDs drop casted on a glass substrate using the Ocean Optics Maya Pro 2000 spectrometer with an LDH-C 400 pulsed laser (PicoQuant, Germany) used for excitation. The laser emission wavelength was 405 nm, the pulse duration was 75 ps, the average emission power was 5 mW, and the repetition rate was 40 MHz.

The PL quantum yield was measured by a relative method at room temperature using Coumarin 153 in ethanol as the standard [59].

3. Results and discussion

3.1. Morphology studies

The TEM image of the synthesized colloidal $CdTe/SiO_2$ QDs is presented in Fig. 1a. It can be seen that the nanocrystals have a spherical shape. The size distribution of the nanocrystals is given in

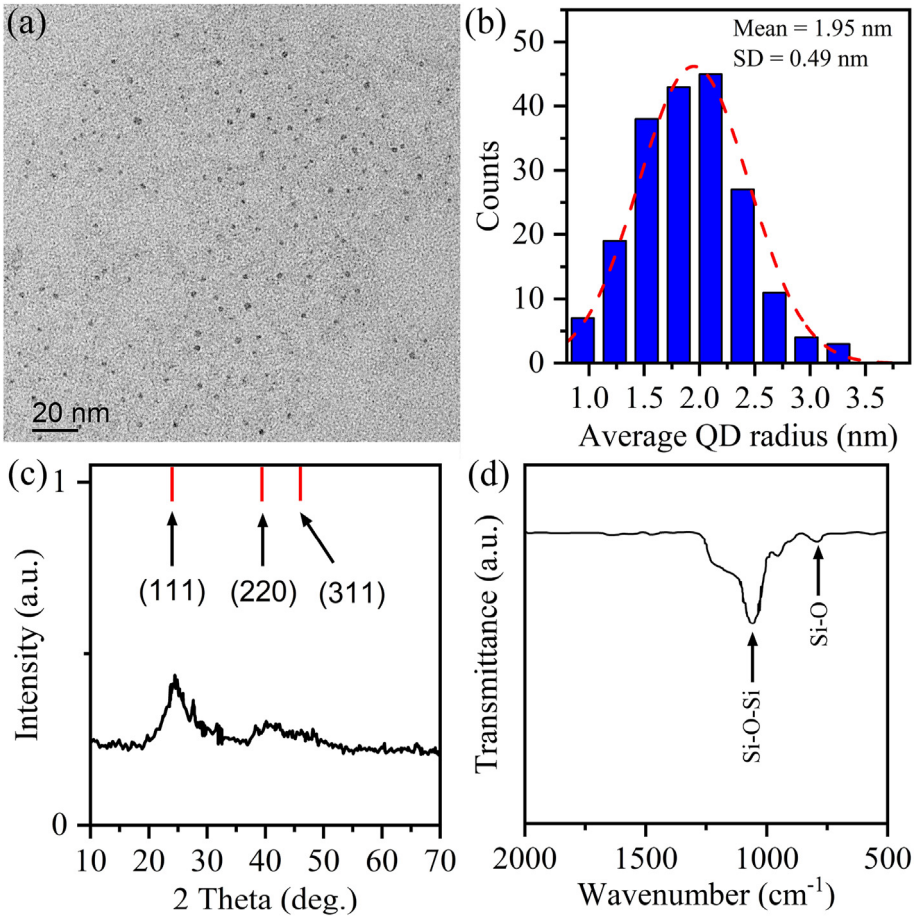

Fig. 1. (a) TEM-image of the synthesized colloidal semiconductor $CdTe/SiO_2$ QDs; (b) The size distribution of the synthesized QDs. The dashed curve is a Gaussian fit to the data; (c) XRD pattern for the $CdTe/SiO_2$ QDs; (d) FTIR spectrum the $CdTe/SiO_2$ QDs.

Fig. 1b. The dashed red curve is a Gaussian function $a \cdot \exp(-(R - Mean)^2 / 2 \cdot SD^2)$. Here a is the amplitude, R is the radius of the QDs, *Mean* is the average radius of the QDs, and *SD* is the standard deviation. The average radius of the colloidal CdTe/SiO_2 QDs was obtained to be 1.95 nm with SD = 0.49 nm. The XRD pattern shown in Fig. 1c was used to determine the phase structure of CdTe/SiO_2 QDs. The diffraction features at 24.03°, 39.47°, and 46.48° are due to the (111), (220), and (311) crystal planes and are attributed to the cubic CdTe (JCPDS Card No. 15–0770). The weak intensity of the diffraction peaks is due to the presence of the SiO_2 shell. Also, a FTIR analysis has been conducted (Fig. 1d). Bands at 1060 and 790 cm^{-1} were observed, which can be attributed to asymmetric Si-O-Si stretching vibrations and symmetric Si-O stretching vibrations specific to CdTe/SiO_2 core/shell systems [60,61].

3.2. Optical properties

The samples were studied by absorption and emission spectroscopies (Fig. 2a). The absorption band with a maximum at ~456 nm (Fig. 2a, blue curve) is associated with electronic transitions between the valence band and the conduction band of the QDs [62]. In the PL spectrum (Fig. 2a, red curve) of the CdTe/SiO_2 QDs, a band due to the interband transition is observed. The PL intensity maximum is at 521 nm, and the full width at half-maximum (FWHM) of the PL spectrum is 56 nm. At longer wavelengths, a noticeable broadening of the PL band is observed in the wing of the spectrum, which may be due to the very weak trap-state luminescence [63]. The quantum yield of the CdTe/SiO_2 QDs was measured to be 42%.

Using the wavelength (456 nm) of the first exciton peak in the absorption spectrum, we can estimate the average size of the QDs using the following relation [64]:

$$E_g^{QD} = E_g^{Bulk} + \frac{h^2}{8R^2}\left[\frac{1}{m_e} + \frac{1}{m_h}\right] - \frac{1.8e^2}{4\pi\varepsilon\varepsilon_0 R}.$$

Here E_g^{QD} is the QD bandgap, E_g^{Bulk} (1.475 eV [65]) is the bandgap of the bulk CdTe crystal, h is the Planck's constant, e is the elementary charge, $m_e = 0.11m_0$ and $m_h = 0.35m_0$ [65] are the effective masses of electrons and holes, respectively, m_0 is the electron rest mass, ε_0 is the electric constant, $\varepsilon = 7.1$ [64] is the dielectric constant of the CdTe QDs, and R is the average radius of the CdTe QDs. According to the calculations, the average radius of the QDs under study is 1.76 nm. The maxima of the absorption and PL spectra of the colloidal CdTe/SiO_2 QDs are shifted to the blue region relative to thicker

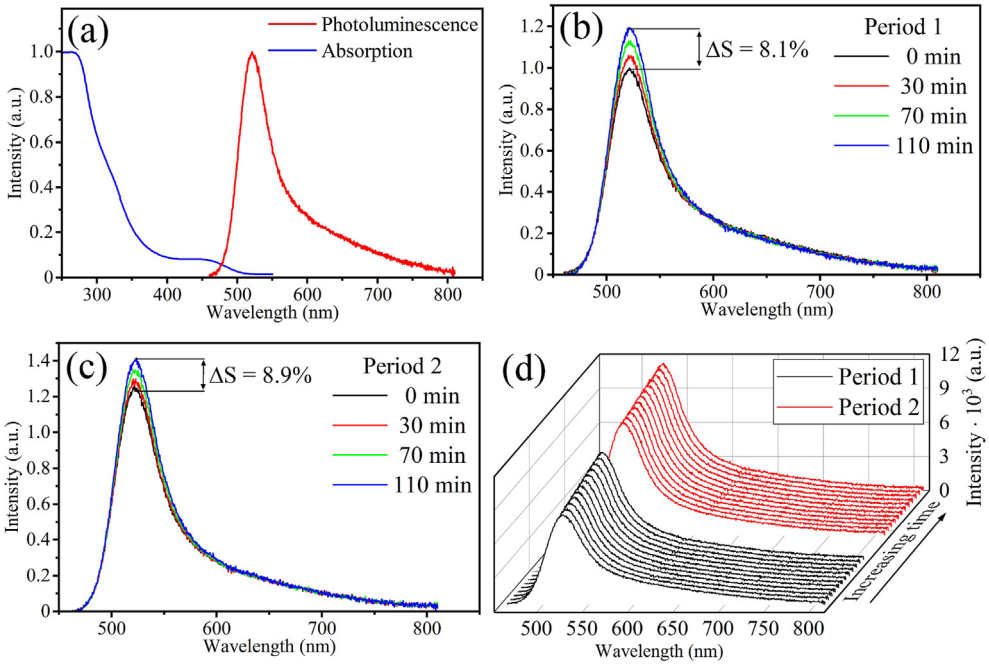

Fig. 2. (a) Absorption (blue curve) and PL (red curve) spectra of the CdTe/SiO_2 QDs at room temperature; (b) PL spectra of the CdTe/SiO_2 QDs obtained under continuous laser irradiation for "Period 1"; (c) PL spectra of the CdTe/SiO_2 QDs obtained under continuous laser irradiation for "Period 2"; (d) Representative series of PL spectra for "Period 1" and "Period 2". PL spectra for "Period 2" are normalized relative to the first spectrum obtained for "Period 1".

CdTe QDs and bulk CdTe crystal, which is due to the quantum confinement effect [64,66,67].

3.3. PL intensity enhancement

To measure the time dependence of the PL intensity of the CdTe/SiO_2 QDs under continuous laser irradiation, PL spectra were recorded every 10 min for 110 min. Then, the laser was switched off for 1 h and then turned on again for 110 min with the PL spectra registered every 10 min. The experiment carried out before the laser was turned off will be called "Period 1" and the experiment carried out after the laser was turned on again will be referred to as "Period 2". To quantify the observed effects, the integrated PL intensity was calculated using the following relation:

$$S = \int_{\lambda_1}^{\lambda_2} I(\lambda) d\lambda.$$

Here S is the integrated PL intensity for the CdTe/SiO_2 QDs, $I(\lambda)$ is the PL intensity as a function of wavelength, the integration limits are $\lambda_1 = 460$ nm and $\lambda_2 = 810$ nm.

The PL spectra of the CdTe/SiO_2 QDs at different time points recorded under continuous laser irradiation during "Period 1" are shown in Fig. 2b. It can be seen that the PL intensity increases with time, while the shape of the spectrum does not change significantly. For "Period 1", the integrated PL intensity increased by ~8.1%. After recording the spectra for "Period 1", the laser radiation was switched off for 1 h.

The PL spectra of the colloidal CdTe/SiO_2 nanocrystals at different time points recorded under continuous laser irradiation during "Period 2" are shown in Fig. 2c. These spectra are normalized relative to the first spectrum recorded for "Period 1" (Fig. 2b, black curve). It can be seen that the PL intensity is also increased for "Period 2". The shape of the spectra did not change significantly. The integrated PL intensity increased by about 8.9%. A representative series of PL spectra for "Period 1" and "Period 2" is shown in Fig. 2d.

The integrated PL intensity for the colloidal semiconductor CdTe/SiO_2 QDs continuously increases with time (Fig. 3a). The integrated PL intensity increased by about 22% over the entire experimental time range (Fig. 3b). The enhancement of the luminescence intensity can be related to the adsorption of water molecules on the surface of the QDs [49,51,52]. Consequently, passivation of defect states occurs, and thus a number of "dark" QDs

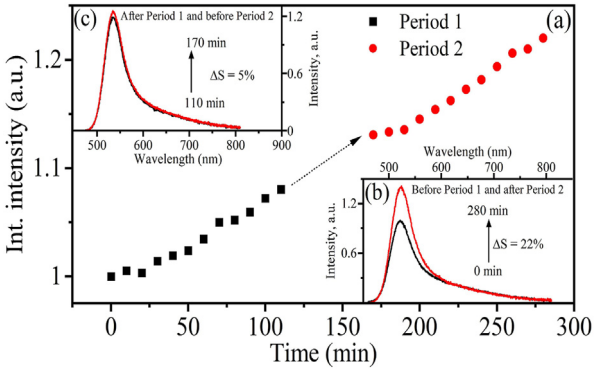

Fig. 3. (a) Time dependence of the integrated PL intensity for the CdTe/SiO_2 QDs; (b) PL spectra before "Period 1" (black curve) and after "Period 2" (red curve); (c) PL spectra after "Period 1" (black curve) and before "Period 2" (red curve).

[49,51] turn into "bright" QDs. In other words, the probability of nonradiative transitions in the QD ensemble decreases due to the trap passivation. Notably, in Ref. [51] core-only CdTe QDs have been studied under continuous laser irradiation and similar intensity enhancement has been observed. However, after approximately 200 min of irradiation, it turned into intensity quenching with a significant blueshift due to photocorrosion. None of the latter effects are observed in our case due to the protective effect of the SiO_2 shell, which will be discussed below. Moreover, after a 60 min "break" in the irradiation, the integrated PL intensity increased by ~5% (Fig. 3c) in contrast to the expected decrease [49]. The observed increase in the PL intensity after keeping the QDs in the dark suggests that the trap passivation process continues even in the absence of irradiation.

During the entire time of the experiment ("Period 1" + "Period 2"), the position of the PL intensity maximum for the colloidal CdTe/SiO_2 QDs is almost unchanged (Fig. 4a). This can be explained by the fact that for the CdTe core capped with a large-bandgap (~8.9 eV [68]) inorganic SiO_2 shell, photocorrosion, which is often responsible for the PL blueshift, is a slow process [52,53].

The color coordinates (x; y) calculated in the CIE 1931 space, dominant wavelength λ^*, color purity (CP), and correlated color temperature T_c for the investigated QDs are presented in Table 1 [69–71]. The color coordinates obtained for the PL of the CdTe/SiO_2 nanocrystals are almost at the edge of the chromaticity diagram (Fig. 4b) in the green region, indicating a high color purity ($CP = 81\%$). The dominant wavelength is $\lambda^* = 556$ nm. As a result of the luminescence intensity enhancement for 280 min, the color coordinates are slightly shifted to the blue region. At the same time, the color purity of

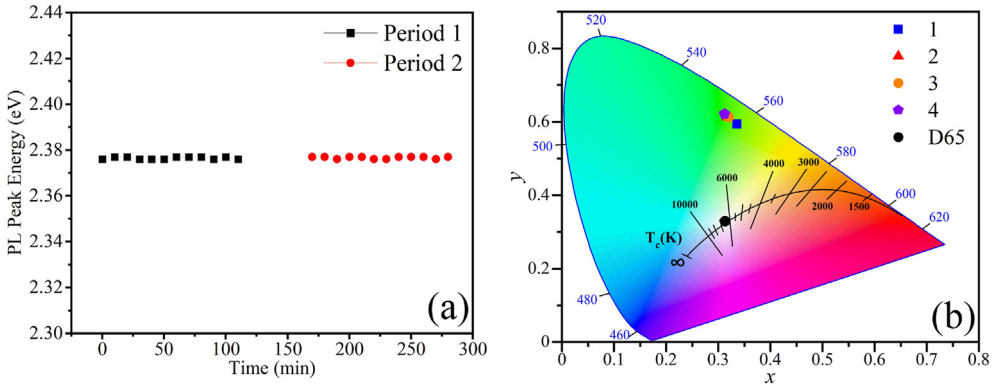

Fig. 4. (a) Time dependence of the PL intensity maximum for the CdTe/SiO_2 QDs under continuous laser irradiation; (b) Chromaticity diagram: 1 is PL of the QDs before "Period 1"; 2 is PL of the QDs after "Period 1"; 3 is PL of the QDs before "Period 2"; 4 is PL of the QDs after "Period 2"; D65 is the standard radiation (white light); solid black line is the Planck's curve; T_c (K) is the correlated color temperature.

Table 1. Color coordinates, dominant wavelength, color purity, and correlated color temperature for CdTe/SiO_2 QD emission.

Time	Color coordinates		λ^* (nm)	*CP* (%)	T_c (K)
	x	*y*			
1 – Before "Period 1"	0.336	0.594	556	81	5457
2 – After "Period 1"	0.319	0.613	553	83	5737
3 – Before "Period 2"	0.340	0.613	553	83	5716
4 – After "Period 2"	0.312	0.621	551	84	5837

the investigated QDs is increased by 3%. Hence, it is potentially possible to obtain PL with higher color purity at prolonged irradiation.

3.4. PL intensity enhancement mechanism

Let us consider the mechanisms of the PL intensity enhancement for the colloidal CdTe/SiO_2 semiconductor QDs (Fig. 5). The studied samples consist of an ensemble of QDs. At the initial moment of time (Fig. 5a), electrons (e_1 and e_2) in the ensemble of the QDs are excited (black solid arrow) under continuous laser irradiation, which can further relax from the conduction band to the valence band both radiatively (red arrow) and nonradiatively (black dashed arrow) through the

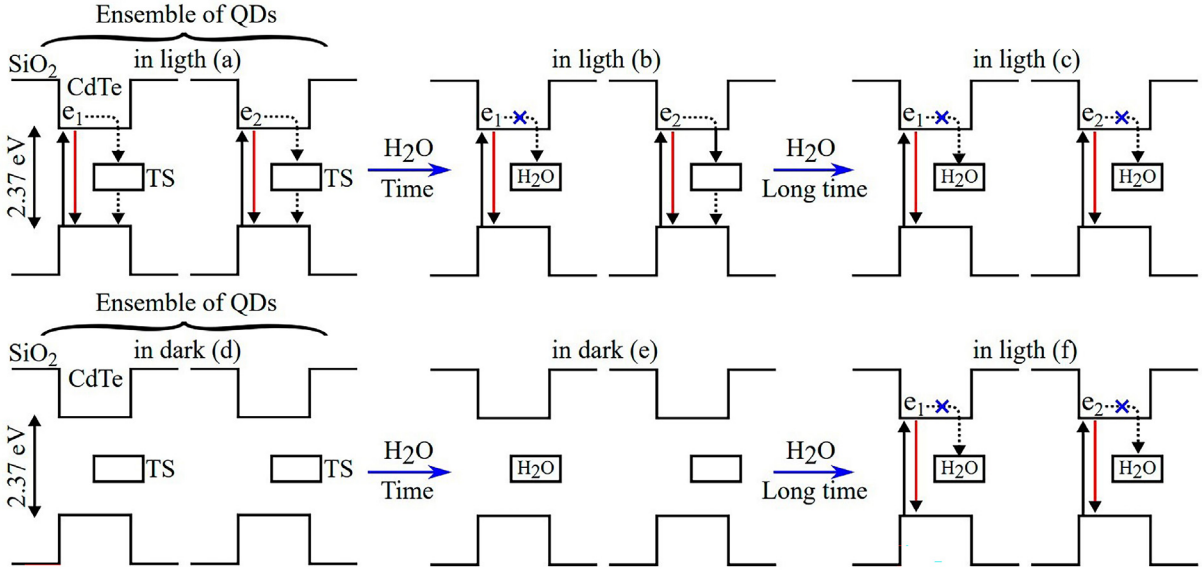

Fig. 5. Mechanism of the PL intensity enhancement for the CdTe/SiO_2 QDs under continuous laser irradiation (top) and without it (bottom): (a) Initial time point under continuous laser irradiation; (b) Some time after the start of irradiation; (c) Long time after the start of irradiation; (d) Initial time point in the absence of laser irradiation; (e) Some time after the beginning of the experimental phase when optical radiation is absent; (f) Moment of switching on the excitation radiation again.

trap state (TS). Water molecules are then adsorbed on the surface of several QDs and passivate the surface traps (Fig. 5b), thereby reducing the probability of nonradiative transitions and, consequently, increasing the QD PL intensity [49,51,52]. Eventually, the number of QDs with passivated traps increases in the ensemble (Fig. 5c). Thus, the number of radiative transitions increases, which results in an increase in the amplitude of the PL maximum (Fig. 2b–d) and integrated PL intensity (Fig. 3) under continuous laser irradiation. Let us now consider the possible mechanism of PL enhancement for the sample kept in the dark after irradiation. Let us assume that no transitions occur in the absence of laser radiation (Fig. 5d). In fact, spontaneous nonradiative transitions are possible both between the valence and conduction bands and through trap levels, but in our case such transitions do not contribute to the PL intensity. Therefore, they are absent in Fig. 5d. It is assumed that in the dark, the process of adsorption of water molecules on the surface of QDs does not stop and the passivation of traps continues (Fig. 5e). Thus, the number of QDs with passivated traps after storing them in the dark appears to be greater than the number of QDs with passivated defects at the beginning of the experimental phase when the laser is switched off. In other words, the probability of radiative transitions after storing the QDs in the dark before the second irradiation period is greater than that at the beginning of the experiment when radiation is absent. That is why, when the excitation radiation is switched on (Fig. 5f) after the QDs have been stored in the dark, the increase in the PL intensity (Fig. 3c) and the integrated PL intensity (Fig. 3a) with respect to the last spectrum registered for "Period 1" is observed.

4. Conclusions

In summary, the present study concerns the luminescent properties of colloidal CdTe/SiO_2 core/shell QDs under continuous laser irradiation at room temperature. It is revealed that PL of the investigated nanostructures is due to both interband and trap-state transitions. It is found that under continuous laser irradiation, there is an increase in the PL intensity for the QDs in time. The observed luminescence intensity enhancement is associated with adsorption of water molecules on the surface of the QDs and the resulting decrease in the probability of nonradiative transitions. The absence of the displacement of the PL intensity maximum can be explained by the fact that for the CdTe core capped with a large-bandgap inorganic SiO_2 shell, photocorrosion, which is often responsible for the PL blueshift, is a slow process. It is shown that the color purity of the PL increased for 280 min by 3% due to the discussed effects. The results of our work can be used in the development of optoelectronic and nanophotonic devices based on colloidal semiconductor CdTe/SiO_2 nanostructures. Moreover, the demonstrated stability of the PL peak position makes the studied QDs promising for biological marker application.

Disclosure statement

The authors report there are no competing interests to declare.

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Ethics information

This research is not related to ethical issues as it does not involve living organisms.

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