Enhanced Two-Photon-Pumped Emission from In Situ Synthesized Nonblinking CsPbBr$_3$/SiO$_2$ Nanocrystals with Excellent Stability

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1. Introduction

Perovskites have emerged as a class of cutting-edge photovoltaic and light-emitting materials. However, poor stability due to high moisture sensitivity and undesirable blinking severely limits their further application. Here, to solve these problems without destroying optoelectronic performance, a simple process for the fabrication of nonblinking CsPbBr$_3$ quantum dots (QDs) is investigated. By embedding CsPbBr$_3$ QDs into waterless silica spheres, the blinking of QDs can be strikingly suppressed, with an effective improvement of the moisture resistance and enhanced photostability. The silica sphere can also prevent anion exchange of different halide elements between perovskite QDs. Ultrastable amplified spontaneous emission (ASE) from QDs/SiO$_2$ with no degradation for at least 12 h is observed under continuous laser irradiation ($4 \times 10^7$ continuous intense laser shots), with almost no ASE degradation evident after 60 d of storage under ambient conditions. Most notably, the ASE threshold ($P_{th}$) of CsPbBr$_3$ QDs is decreased by 50% and the relative efficiency increased by 388%. The perovskite QDs coated by the waterless SiO$_2$ shell provide a novel platform for realizing perovskite nanomaterials with improved operational stability, nonblinking properties, and enhanced emission all at the same time, which is especially attractive for photovoltaic and light-emitting device applications.
and render them water-soluble, which has frequently been of use in biological applications that exploit the capability for surface conjugation with several hydrophilic groups such as thiols, amines, and carboxyl groups. However, even though a lot of effort has been put into improving the photostability of silica-coated perovskite QDs, most attempts have failed due to the high sensitivity of the perovskite QDs to the surrounding environment and the presence of water during the formation of the silica coating on the perovskite QDs, which will induce the quenching of the perovskite QDs. Therefore, reducing the exposure to water during SiO2 coating becomes one of the critical problems in the field of silica-coated perovskite QDs; for example, the stability of silica-coated CH3NH3PbBr3 QDs has been significantly enhanced by using toluene (with a H2O content of 0.0184%) during the silica-coating process. However, so far, it has not been possible to completely eliminate the presence of water in the fabrication of silica-coated perovskite QDs. Moreover, the core-shell structure of CsPbBr3/SiO2 cannot be obtained by using the toluene-based silica-coating method.

Here, to both eliminate water present in the fabrication of the silica coating and suppress the undesirable blinking of perovskite QDs, a facile process for the in situ growth of silica spheres onto all-inorganic perovskite QDs has been developed without using any water. Microsized silica spheres with a smooth surface have been developed to accompany the crystallization of perovskite QDs, enabling fabrication of CsPbBr3/SiO2 core/shell structures without any damage to the QDs. Subsequently, the −OH functional group of 2-methoxyethanol has been introduced to improve the hydrophilicity of perovskite QDs, which can further strengthen the water resistance of perovskite QDs. The formation of the protective waterless silica shells that isolate the QDs from moisture in the air leads to significantly improved photostability of perovskite QDs, even after exposure to an atmosphere with a relative humidity (RH) of 70%.

Undesirable photoluminescence (PL) quenching and anion exchange between perovskite QDs with different halide compositions in solution was also found to be prevented for the silica-coated QDs. In addition, after being embedded into the waterless silica sphere, the blinking characteristic of perovskite QDs was suppressed significantly at the single-particle level. Moreover, enhanced one- and two-photon pumped ASE was realized in an ambient atmosphere at room temperature for CsPbBr3 embedded into SiO2, with a relatively lower threshold and more stable emission compared with pure CsPbBr3 QDs. These results indicate that the stability and light-emission performance of perovskite QDs can be significantly improved by embedding into a silica sphere, which is desirable for optical gain media used for frequency up-conversion, providing novel opportunities for nonlinear lasers and photoelectric devices.

2. Results and Discussion

Colloidal cesium lead halide perovskite QDs embedded in silica spheres. Pure CsPbBr3 colloidal QDs were synthesized by the hot-injection method; after initial formation of the perovskite QDs, the silica precursor tetraethoxysilane (TEOS) was injected immediately for the in situ growth of silica shells. After reaction for several minutes, the amorphous SiO2 layers were successfully formed onto the surface of the CsPbBr3 QDs. Compared with an average size of ≈19 nm for a pure CsPbBr3 QD (as shown in Figure S1 in the Supporting Information), the CsPbBr3/SiO2 core/shell structures exhibit a smooth surface with sizes ranging from 0.8–1.6 μm, as observed by the transmission electron microscope (TEM) (Figure 1a). The purification of perovskite QDs prepared by the hot-injection method is generally recognized to be difficult. Here, the micrometer size of the CsPbBr3/SiO2 core/shell structure enables the particles to be easily collected at relatively low centrifugal speed. For the same amount of precursor (0.38 mmol of PbBr2), the production weight of CsPbBr3 QD powder is ≈12.5 mg, while the production weight of CsPbBr3/SiO2 powder increases to ≈57.2 mg (as shown in Figure S2 in the Supporting Information). Therefore, the silica spheres can facilitate efficient gathering of CsPbBr3 QDs.

As shown in Figure 1b, a large amount of perovskite QDs are aggregated in the silica sphere, which was further confirmed by the magnified TEM image showing the CsPbBr3/SiO2 core/shell structure. As shown in Figure 1c, an abundance of cubic CsPbBr3 QDs are embedded inside the silica spheres, with only a few QDs attached onto the surface of the spheres. The corresponding high-resolution TEM (HRTEM) image for a typical single CsPbBr3 QD at the edge of the CsPbBr3/SiO2 core/shell structure (the QD denoted by the dotted red line) further demonstrates a lattice plane distance of 5.8 Å, which is consistent with that of pure CsPbBr3 QDs (Figure S3, Supporting Information). In addition, the X-ray diffraction (XRD) pattern of CsPbBr3/SiO2 NCs (Figure 1e) is in full agreement with that obtained for CsPbBr3 QDs (Figure S4, Supporting Information), which indicates that the in situ growth of the SiO2 shell does not affect the cubic crystal structure of the CsPbBr3 core. Moreover, as shown in Figure 1f, both CsPbBr3 QDs and CsPbBr3/SiO2 NCs show similar PL spectra in solution following excitation at 350 nm, with a narrow green emission peak at 533 nm and full-width at half-maximum (FWHM) of 18 nm, which further demonstrates that the in situ growth method for the silica shell does not influence the crystal quality and optical properties.

The heightened hydrophilicity and stability of CsPbBr3/SiO2 NCs. In general, perovskite QDs are typically protected by the initial surfactant such as oleic acid and oleylamine, and consequently demonstrate hydrophobic properties. To alter the hydrophilicity of the NCs, 2-methoxyethanol was used as a ligand to modify the surface. Figure 2a shows the corresponding schematic diagram for the modification of this method. The hydrophilic terminal −OH functional group of 2-methoxyethanol can bind with silica precursors, heightening the hydrophilicity of CsPbBr3/SiO2 NCs. The formation of Si−O bonds were confirmed by the Fourier transform infrared spectroscopy (FTIR) spectrum (Figure S5, Supporting Information) for CsPbBr3/SiO2 NCs, which showed an intense peak at 1090 cm−1 and a weaker peak at 796 cm−1. As shown in Figure S5 in the Supporting Information, the broad O−H stretching band at ≈3447 cm−1 and the H−O−H bending band at ≈1634 cm−1 may be due to 2-methoxyethanol or absorbed water molecules. To verify the water solubility and stability of the pure perovskite and perovskite/SiO2 structures, a solvent containing...
Toluene and water (ratio 4:1) was used to disperse CsPbBr$_3$ QDs and CsPbBr$_3$/SiO$_2$ NCs, respectively. CsPbBr$_3$-QD and CsPbBr$_3$/SiO$_2$ NC solutions were excited by a 365 nm UV-lamp, and bright green light emission was observed from both solutions. The CsPbBr$_3$ QDs dispersed only in toluene and were completely insoluble in water, as demonstrated by the clear PL/liquid interface in the water–toluene solvent (Figure 2b, left figure on the top). In contrast, the PL interface disappeared in the CsPbBr$_3$/SiO$_2$ solution, which indicated that CsPbBr$_3$/SiO$_2$ NCs could disperse in both toluene and water (Figure 2b, top right figure). The increased water solubility of the perovskite QDs has the potential to broaden optoelectronic and biological applications.

Although all-inorganic CsPbBr$_3$ QDs exhibit excellent optical properties, they can be very unstable due to their sensitivity to moisture.$^{[14]}$ As shown in Figure 2b, the green light emission from the pure CsPbBr$_3$ QDs solution completely disappeared after 12 h in air, whereas the CsPbBr$_3$/SiO$_2$ solution maintained a bright green emission, indicating the ultrahigh water stability and water-dispersion properties of CsPbBr$_3$/SiO$_2$ NCs. To confirm the stability related to water resistance further, samples were placed in an environment with constant RH as high as 75% at room temperature. As shown in Figure 2c, a CsPbBr$_3$/SiO$_2$ film shows a slower PL decay rate compared to a CsPbBr$_3$ film, which confirms the enhanced moisture resistance due to the silica shell. After being stored for 12 h, the PL...
intensity of the CsPbBr$_3$ film decreased to 66.6% of the initial value, whereas the CsPbBr$_3$/SiO$_2$ film maintained a higher PL intensity with a smaller decrease to 73.8% of the initial value, suggesting that the SiO$_2$ shell can effectively protect CsPbBr$_3$ QDs from degeneration due to moisture. In addition, both pure CsPbBr$_3$ and CsPbBr$_3$/SiO$_2$ films were heated to 60 ºC in atmosphere to evaluate their thermo-stabilities. As shown in Figure 2d, after 15 h, the relative PL intensity of CsPbBr$_3$ film almost decreased to zero with weak light emission observed. In contrast, the PL intensity from the CsPbBr$_3$/SiO$_2$ film decreased much more slowly, with ≈36.4% of the initial PL intensity maintained after 15 h at 60 ºC. Therefore, the silica shell can provide effective protection to the perovskite QDs against degeneration due to high humidity and high temperature.

Recently, the halide-anion exchange (Cl$^{-}$, Br$^{-}$ and I$^{-}$) between perovskite QDs with different compositions has
been shown to be an additional critical problem hindering optoelectronic applications, especially applications in white light-emitting diodes.[31–34] It is recognized that silica coating is an efficient approach to isolate the direct contact between perovskite QDs with different compositions.[24] As shown in Figure 2e, immediately after mixing for 10 min, the PL spectrum of the CsPbBr$_3$ + CsPb(Br/I)$_3$ solution changed dramatically compared to the combined PL spectra of individual CsPbBr$_3$ QDs (green solid line) and CsPb(Br/I)$_3$ QDs (orange solid line). Specifically, the orange emission from CsPb(Br/I)$_3$ decreased remarkably and the green emission from CsPbBr$_3$ QDs showed a redshift and clear increase, which can be attributed to the exchange of Br$^-$ in CsPbBr$_3$ QDs for I$^-$ in CsPb(Br/I)$_3$ QDs. In contrast, no clear shift was observed for the PL spectra of the CsPbBr$_3$/SiO$_2$ and CsPb(Br/I)$_3$/SiO$_2$ mixture solution, as shown in Figure 2f. The corresponding optical photographs after 10 min are shown in Figure S6 in the Supporting Information. All these data suggest that the SiO$_2$ shell can be used to effectively protect the perovskite QDs from anion-exchange by avoiding direct contact.

Nonblinking characteristics of CsPbBr$_3$/SiO$_2$ NCs. To further study the efficient protection of the perovskite core by the SiO$_2$ shell, detailed analyses of the dynamic optical properties were carried out for a typical single CsPbBr$_3$ QD and CsPbBr$_3$/SiO$_2$ NC. For the measurement of single nanocrystals, diluted solutions of CsPbBr$_3$ QDs and CsPbBr$_3$/SiO$_2$ NCs were dispersed in toluene for comparison. A bright-field microscope was utilized to locate the nanocrystals for further observation (Figures S7 and S8, Supporting Information).

Figure 3 shows the PL-tracking data measured under continuous wave laser excitation at 405 nm for one single CsPbBr$_3$ QD (Figure 3a) and CsPbBr$_3$/SiO$_2$ NC (Figure 3b) dried on a glass slide. For the pure CsPbBr$_3$ QDs (Figure 3a), the fluorescence intermittency randomly oscillated between two states, the relatively high state (on) and low state (off), which can be attributed to blinking caused by nonradiative recombination.[35–37] On the other hand, the fluorescence intensity also showed a decrease under continuous exposure to laser light for 180 s. In contrast, the single CsPbBr$_3$/SiO$_2$ NC showed nonblinking character. The typical time trace displayed no obvious decrease in fluorescence intensity or oscillation, with the continuous ON state showing a minor decrease following a Poisson intensity distribution.[37] The corresponding real time monitoring of CsPbBr$_3$ QDs (Movie S1, Supporting Information) and CsPbBr$_3$/SiO$_2$ NCs (Movie S2, Supporting Information) for 142 s are also supplied as Movies in the Supporting Information. These observations further indicate that the silica shell can passivate the surface sensitivity and effectively suppress the blinking property of CsPbBr$_3$ QDs.

Time-resolved PL decay was used to study the mechanism for the nonblinking property of the CsPbBr$_3$/SiO$_2$ core/shell structure. As shown in Figure 3c, the total fluorescence lifetime for pure CsPbBr$_3$ QD (black curve) was calculated to be $\approx 39.8$ ns, which can be extracted by using a biexponential decay fit with two component lifetimes of $\tau_r$ (12.7 ± 0.4 ns, 78%) and $\tau_{nr}$ (60.1 ± 0.6 ns, 22%) ($\tau_r$ radiative, $\tau_{nr}$ nonradiative). Compared to pure CsPbBr$_3$ QDs (black curve), CsPbBr$_3$/SiO$_2$ (red curve) NCs exhibited a longer lifetime of 48.3 ns with longer $\tau_r$ (18.5 ± 0.3 ns, 82%) and $\tau_{nr}$ (80.2 ± 0.2 ns, 17%) (Table S1, Supporting Information). Therefore, it can be conclude that the observed suppression of fluorescence fluctuation in CsPbBr$_3$ QDs is consistent with an increased contribution from the radiative recombination component.[13]

Enhanced ASE from CsPbBr$_3$/SiO$_2$ NCs under two-photon excitation. As reported previously, all-inorganic CsPbX$_3$ perovskites demonstrate excellent potential as candidate materials for lasing and two-photon-excited up-conversion devices because of

**Figure 3.** Suppressed blinking property of the perovskite/SiO$_2$ structure. Fluorescence intensity as a function of time (binning time, 10 ms) with optical excitation at 405 nm for a) single CsPbBr$_3$ QD, the inset shows a histogram for the intensity distribution, b) single CsPbBr$_3$/SiO$_2$ NC, the inset histogram shows the Poissonian intensity distribution. c) Corresponding time-resolved photoluminescence decay of CsPbBr$_3$ QDs (black) and CsPbBr$_3$/SiO$_2$ NCs (red).
their large two-photon absorption cross-section.\[35,38\] Here, we focus on a comparison study of the emission performance from CsPbBr3 and CsPbBr3/SiO2 under both two- (800 nm) and one-photon (400 nm) excitation. All of these measurements were performed at room temperature in an air environment. For ASE under two-photon excitation, 800 nm femtosecond laser pulses with a repetition rate of 1 kHz and pulse duration of 100 fs were used as the optical pump. The pump-intensity dependence of the laser-induced emission is shown in Figure 4. The threshold ($P_{th}$) for two-photon pumped ASE for the CsPbBr3 is determined to be 480.8 $\mu$J cm$^{-2}$, whereas the threshold for CsPbBr3/SiO2 is only 230.8 $\mu$J cm$^{-2}$; therefore, the two-photon pumped ASE threshold for coated QDs is less than half of the value measured for uncoated QDs. As shown in Figure 4a,c, at lower excitation intensity, spontaneous emission can be obtained for both CsPbBr3 and CsPbBr3/SiO2 centered at 534 nm with a FWHM of 24 nm (Figure S9, Supporting Information, black). When the pump intensity is increased above the $P_{th}$, both samples show a sharp redshifted peak (537 nm for both CsPbBr3 and CsPbBr3/SiO2), which is in agreement with previous reports,\[39,40\] with bright green light emission clearly observed in air (inset Figure 4c). Figure 4b,d shows the dependence of the output intensity and linewidth on the pump intensity under 800 nm excitation. In both samples, the emission intensity increases dramatically when the pumping intensity is increased above the $P_{th}$, and a sharp decrease in the FWHM (from 24 to 5 nm for both CsPbBr3 and CsPbBr3/SiO2) was also observed. These similar results for both CsPbBr3/SiO2 and CsPbBr3 imply that the SiO2 shell coating process does not impair the emission performance of the CsPbBr3 QDs. Furthermore, a detailed comparison of the output intensity of CsPbBr3 and CsPbBr3/SiO2 shows that the output emission intensity, $P_{out}$ of CsPbBr3 QDs at 1.5 times $P_{th}$ (721 $\mu$J cm$^{-2}$) is 1.8 times the output intensity at $P_{th}$, while the $P_{out}$ of CsPbBr3/SiO2 NCs at 1.5 times $P_{th}$ (346 $\mu$J cm$^{-2}$) is as high as 8.8 times that of $P_{out}$ at $P_{th}$. Therefore, following coating by the SiO2 film, the ASE threshold of CsPbBr3 is decreased by 50% and the relative efficiency increased by 388%. From the lifetime data shown in Figure 3c, we consider that the longer lifetime for the upper state will be greatly advantageous for realizing population inversion in the optical gain medium, which can, thus, enhance the ASE performance. The lower threshold and high optical conversion efficiency of CsPbBr3/SiO2 NCs indicate promising potential for application in light-emitting devices.

Similar ASE results under one-photon pumping were obtained for CsPbBr3/SiO2 and CsPbBr3, as shown in Figure S10 in the Supporting Information. The ASE threshold for CsPbBr3/SiO2 is determined to be $\approx$95.8 $\mu$J cm$^{-2}$ (Figure S10d, Supporting Information), which is lower than the threshold (143.5 $\mu$J cm$^{-2}$) of pure perovskite CsPbBr3 QDs (Figure S10b, Supporting Information). The incident power dependence of the emission intensity (Figure S10a,c, Supporting Information) and gain-induced linewidth narrowing (Figure S10b,d, Supporting Information) are described in detail in the Supporting Information.

3. Discussion

CsPbBr3/SiO2 and CsPbBr3 exhibit similar light-emission features under both two-and one-photon excitation; however,
was very simple: a silica precursor was inserted into freshly acquired CsPbBr3 QDs and CsPbBr3/SiO2 NCs, a mixture of 200 mg of Cs2CO3, 1.2 mL of OA and 8 mL of octadecene (ODE) were degassed under nitrogen flow in a three-neck flask at 120 °C for 1 h until all the Cs2CO3 reacted with the OA. Then, 10 mL ODE and 0.38 mmol of PbBr2 were mixed in a three-necked flask which was placed in a heating jacket with nitrogen. This three-necked flask was heated to 120 °C under nitrogen flow. The heater temperature was then set up to 165 °C. Subsequently, 1 mL oleylamine and 1 mL OA were rapidly injected into the PbBr2 crude solution.

Characterization of CsPbBr3 QDs and CsPbBr3/SiO2 NCs: The crystal phases of all samples were characterized by XRD with CuKα radiation (XRD-6100, SHIMADZU, Japan). The transmission electron microscopy (TEM) images were recorded using a ZEISS LIBRA 200FE microscope. The optical absorption was measured by a Scan UV–vis spectrophotometer (UV–vis: UV-2100, Shimadzu, Japan) at room temperature over a wavelength range of 200–800 nm. The PL spectra were measured by a fluorescence spectrophotometer (PL: Agilent Cary Eclipse, Australia). PL lifetime measurements were carried out using a fluorescence spectrometer Edinburgh Instruments (F55-TCSPC).

A. Characterization: For the two-photon-pumped ASE experiments, a commercially supplied Ti:sapphire regenerative amplifier (800 nm, 35 fs, 1 kHz) was used as the pumping source. For one-photon excitation, a UV-laser pulse centered at 400 nm was used, which was generated by an ytterbium-doped fiber laser. The pulse duration was 130 fs, the repetition rate was 80 MHz, and the output power was 20 mW. The ASE was compared for CsPbBr3 (black) and CsPbBr3/SiO2 (green) under continuous excitation using 800 nm femtosecond laser pulses with pump intensity of 600 μJ cm−2 in air over 12 h (up to 4.32 × 105 excitation cycles). As shown in Figure 4f, after 12 h continuous excitation, the light emission from the CsPbBr3/SiO2 film was expected to experience multiple scattering over a much longer distance and hence experience much more gain before light output as ASE. [41,44] which can also decrease the ASE threshold and increase the ASE efficiency.

Since most perovskite materials are subject to environmental instability due to their susceptibility to hydrolysis from moisture in the atmosphere, photostability under intense laser excitation is critically important for further application in the nanolaser field. Here, to demonstrate the stability of CsPbBr3/SiO2 NCs for application in future lasing devices, the extended operation of ASE was compared for CsPbBr3 (black) and CsPbBr3/SiO2 (green) under continuous excitation using 800 nm femtosecond laser pulses with pump intensity of 600 μJ cm−2 in air over 12 h (up to 4.32 × 105 excitation cycles). As shown in Figure 4f, after 12 h continuous excitation, the light emission from CsPbBr3/SiO2 NCs maintains >95% of the original emission intensity, whereas the emission for pure CsPbBr3 QDs drastically decreases to 85%, which indicates a relatively long operating lifetime for perovskite QDs coated with silica spheres under atmospheric conditions. After exposure to the atmosphere for a 2 month period, the optical properties of the CsPbBr3 QDs remained stable ASE (see inset in Figure 4f). Notably, the extended light-emission operation under intense laser excitation in an atmospheric environment is encouraging for the prospective realization of perovskite-SiO2 in further practical optoelectronic applications.

5. Conclusion

By the in situ embedding of all-inorganic perovskite CsPbBr3 QDs into completely waterless silica spheres, both the photo- and light emission properties of CsPbBr3 QDs were enhanced at the same time. The fabrication process used was very simple: a silica precursor was inserted into freshly synthesized samples to accompany the crystallization of perovskite QDs. The effective isolation was provided by the anhydrous silica shell from the moisture in the air led to a dramatically enhanced stability of the CsPbBr3/SiO2 NCs compared with the pure perovskite QDs. In addition, the blinking problem at the single-particle level was effectively suppressed, and the undesirable anion-exchange between perovskite QDs was efficiently prevented. Surprisingly, by embedment into waterless SiO2 spheres, the ASE threshold of CsPbBr3 QDs decreased by 50% and the relative light-output efficiency increased by 388%, which is mainly attributed to an enhanced Rayleigh scattering intensity. In addition, the waterless silica shell can be regarded as not only a protective layer to guard CsPbBr3 QDs against degradation but also as an enhancement factor to improve the photoinduced emission. We expect that the perovskite/SiO2 structure, with its enhanced stability and relative hydrophilicity, can open a novel route for the realization of frequency up-conversion lasers and photoelectric devices.
by frequency doubling in a 200 μm thick beta barium borate (type 1, Δθ = 29.2°) crystal. In both experiments, the laser beams were focused onto samples by a cylindrical lens with a focal length of 10 cm. The stripe length of the spot size was precisely controlled by an adjustable slit. The ASE was detected by a fiber spectrometer (Ocean Optics) with a spectral resolution of ~1 nm.

PL Intensity Trace Measurements for Single QDs: All samples were studied under ambient conditions by a home-built wide-field fluorescence microscope. For the PL intensity trace measurement of single QDs, we diluted a sample solution to a low concentration until isolated bright spots in the images were obtained (Figure S11, Supporting Information). The 514 nm laser line from an Ar+ CW laser was used as the optical excitation. The excitation power was adjusted by a set of neutral density filters. The ASE was collected by an oil immersion objective lens (Olympus UPlanFLN 60×, numerical aperture (NA) = 1.25) and imaged onto a CCD camera (Pro-EM: 512B, Princeton Instruments) after passing through a long-pass filter (HQ675lp, Chroma Technology Corp.). A transmission grating (150 lines per mm) was placed in front of the CCD camera for the spectral measurement giving a resolution of ~8 nm. The PL intensity transients contained 1000 frames with an exposure time of 100 ms per frame.

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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Conflict of Interest

The authors declare no conflict of interest.

Keywords

amplified spontaneous emission, perovskites, quantum dots, SiO2, two-photon excitation


