Highly Luminescent, Stable, and Red-Emitting CsMg Pb Quantum Dots for Dual-Modal Imaging-Guided Photodynamic

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Highly Luminescent, Stable, and Red-Emitting $CsMg_xPb_{1-x}I_3$ Quantum Dots for Dual-Modal Imaging-Guided Photodynamic Therapy and Photocatalytic Activity

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ABSTRACT: In this study, for the first time, red-emitting $CsM g_x Pb_{1-x}I_3$ quantum dots (QDs) are prepared by doping with a ganesium (Mg) ions via the one-pot microwave pyrolysis technique. The X-ray diffraction and X-ray photoelectron spectroscopy results have confirmed partial substitution of Pb^{2+} by Mg^{2+} inside the $CsPbI_3$ framework. The as-synthesized $CsM g_x Pb_{1-x}I_3 QDs$ have exhibited excellent morphology, higher quantum yield (upto B9%), better photostability and storage stability than undoped $CsPbI_3$. Next, the bioavailability of as-synthesized hydrophobic $CsM g_x Pb_{1-x}I_3 QDs$ is improved by encapsulating them into gadolinium-conjugated plur Bsymbolinium Conjugated Plur <math>Bsymbolinium Csymbolinium Csymboli



Gd nanoagents are demonstrated by employing T_1 and T_2 studies, which validated that PQD@Gd nanoagents had superior MR contrasting elect with a r_2/r_1 ratio of 1.38. In vitro MRI and fluorescence imaging analyses have shown that the PQD@Gd nanoagents are internalized into the cancer cells via a caveolae-mediated endocytosis pathway. The PQD@Gd nanoagents have exhibited excellent biocompatibility even at concentrations as high as 450 ppm. Interestingly, the as-prepared PQD@Gd nanoagents have eleiently produced cytotoxic reactive oxygen species in the cancer cells under 671 nm laser illumination and thereby in the cancer cells under 671 nm laser illumination and thereby in the cancer cells under 671 nm laser illumination. The organic pollutants under visible light irradiation. The organic pollutants rhodamine b, methyl orange, and methylene blue were degraded by 92.11, 89.21, and 76.21%, respectively, under 60, 8 and 100 min, respectively, irradiation time. The plausible mechanism for the photocatalytic activity is also elucidated. Overall, this work proposes a novel strategy to enhance the optical properties, stability, and bioapplicability of PQDs. The multifunctional PQD@Gd nanoagents developed in this study could be the potential choice of components not only for cancer therapy due to dual-modal imaging and photodynamic therapeutic properties but also for organic pollutant or bacterial removal due to excellent photocatalytic properties.

KEYWORDS: CsM q_x Pb_{1-x}I₃ QDs, multifunctional nanoagent, reactive oxygen species, MR imaging, and photocatalytic activity

1_INTRODUCTION

Owing to their exceptional optoelectronic character 57 is such as bright display, high absorption coellient rate, near-unity frescence quantum yield (QY), and narrow excitonic bands, perovskite nanocrystals (PNCs) have attracted interest in practical applications including photocatalysis, $^{1-3}$ solar cell fabrication, 4,5 white-light-emitting diodes, $^{6-8}$ and fluorescent photodetection. $^{9-12}$ The three-dimensional perovskite crystal structure (AMX3) is made up of a comer-shared octahedral unit [MX6] $^{-4}$, in which the M-site cation [lead (Pb $^{2+}$), mangan 143 Mn $^{2+}$), tin (Sn $^{2+}$)] is coordinated by six X-site anions (CI $^{-}$, Br $^{-}$, and I $^{-}$) and the A-site cation [cesium (Cs $^{+}$), methylamine (CH3NH3 $^{+}$, MA), ammonia (NH3 $^{+}$)] is embedded in a void surrounded by octahedral unit cells. 13,14

Furthermore, colloidal metal halide nanocrystals have an interesting band arrangement that is defect-tolerant and enables incorporation of atoms of 70 ns into the host structure. 7,14,15 Besides, to adjust the perovskite lattice structure, alleviate crystallographic phases, and achieve diverse optical and electrical properties, the doping strategies that can bring suitable elements into the crowd nanocrystals framework

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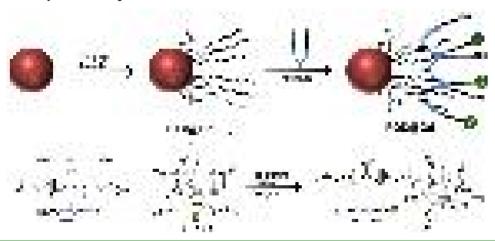






Scheme 1. Schematic Illustration of the Encapsulation of the CsM $g_xPb_{1-x}I_3$ QDs by PF127-Gd Micelle Structure to Produce Water-Soluble a PQD@Gd Nanoagent

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are widely deployed. It has been reported that integrating a M²⁺-site cation into PNCs could correct the Pb coordination structure and increase the short-range lattice order by correcting the Pb-X octahedral alteration and eliminating surface traps by suppressing halide vacancies, which prevents them from achieving high QY. 16-22 For example, Chen and coworkers²³ recently used Cu²⁺ doping to stabilize orthorhombic-phase CsPbI3. The Cu2+-doped PNCs exhibited not only high formation energy but also high stability than undoped PNCs. Tao et al. produced Sn-doped perovskite (MA₃Sb₂I₉) structures using a hydrothermal synthesis approach to construct a high-performance solar cell device. However, this PNC import frequently employed in photovoltaic applications might be due to the high exciton binding energy and transient oxidation state of Sn ions $(Sn^{2+} \square Sn^{4+})$. Recently, Rakesh et. al reported that Ni²⁺-integrated CsPbI₃ could exhibit intense emission spectra and improved stability. Even though the produced NCs had strong restraint ability and good photovoltaic performance, Ni²⁺ is a Shannon radii cation.²⁵ These findings suggest the existence of space to propose new divalent cations that can lift the stability and photophysical properties of the red perovskites upon doping. Also, current approaches could help to stabilize the nanocrystals to some extent, but still, there is requirement of robust strategies. Accordingly, herein, we propose that doping with metal ions having comparable ionic radii and stable oxidation states, such as magnesium (Mg), can improve the luminescent quality of freshly produced perovskite quantum dots (PQDs) by simultaneously improving the energy band gap and resolving the Shannon elect.

After successfully employing the hot injection technique by Kovalenko and collaborators, ²⁶ the advancement of adjustable synthetic procedures for obtaining lead halide PNCs takes a big stride forward. This approach consisted of a two-step process that ³⁰ an with the preparation of a Cs-oleate precursor and subsequent injection of the Cs-oleate precursor into a lead-containing flask at high temperatures and under an inert atmosphere. Although the nanocrystals produced owned excellent crystallinity and stability, the intricate arrangement and inert environment posed major drawbacks. ^{27–30} M icrowave irradiation techniques can address the issue of inhomogeneous heating by providing a scalable platform for

producing exceptional NCs with improved morphological properties. $^{31-33}\,$

D espite the fact that PNCs are the most explored material in the production of WLEDs, solar cells, and sensors, they have received less attention in the nanomedicine field because their crystal structure is highly unstable when exposed to air, aqueous conditions, and light irradiation. 27,28,34 As a result, adopting a strategy to improve PNCs' structure stability in polar solvents is particularly desirable, as it will o der a lot of possibilities for using their ultrahigh-resolution imaging in cancer cell diagnostics and combining it with other therapeutic activities. Polymer embedding matrix, 35 inorganic encapsulation,³⁶ core—shell formation,³⁷ and chemical transformation³⁸ are the most commonly employed techniques to convert hydrophobic perovskite QDs into water-soluble QDs. The polymer embedding technique would be more preferable as it can order a strong hydrophobic protective layer on the PNCs' red wall surface, and similarly, the incorporation of the silica matrix is also critical to achieving stable interaction with water via hydrogen bonding with the alkoxyl group. SiO 2-integrated PQDs, on the other hand, had better morphology and luminescence properties; the silica covering hindered interfacial carrier transfer, which further weakened the performance of PNCs.^{1,39} As a result, we were interested in finding an amphiphilic polymer that can simultaneously provide hydrophobic support to the PQD structure while also having a strong tendency to dissolve in water. In this scenario, pluronic (PF127), a triblock copolymer with amphiphilic properties due to poly(ethylene glycol) (a hydrophilic segment) at both ends and poly(propylene glycol) (hydrophobic segment) at the central block, would be an attractive candidate for fabricating water-stable red-emitting QDs. More importantly, because of its amphiphilic nature, PF127 undergoes self-assembly into micelle structures in a wide range of solutions, allowing for loading of drug or other therapeutic agents. 40 Although a recent study demonstrated that water-phase PQD can be used as a bioimaging probe for cancer cell treatment, 441 the combination of PQD's fluorescence properties with a theranostic agent has never been reported before. It would be important to combine phototherapeutic and contrast agents in a single platform to pinpoint tumor areas and improve cancer therapeutic e□tiency. Magnetic resonance imaging (MRI) has shown to be an e□tient diagnostic tool due to its higher resolution and tissue penetration than existing fluorescence techniques. PNCs' luminescent features make them ideal for use with MRI contrast agents like Gd imaging modalities. Therefore, PF127 polymer micelles with hydrophilic—hydrophobic segments could be an excellent carrier for incorporating a Gd contrast agent into micelles, as well as PQDs, which could be a viable technique for making dual-modal imaging-guided nanoagents.

In this regard, we have established quick and single-step microwave synthesis of high-quality and controllable morphology red-emitting Mg-doped CsPbI3 QDs for the first time. Thus, the as-prepared $CsMg_xPb_{1-x}I_3$ NCs exhibit superior optical properties with excellent crystallinity and enhanced photostability as compared to the nondoped CsPbI3 NCs. Moreover, the incorporation of Mg²⁺ enhances the tolerance factor, resulting in prolonged stability without a⊡acting the crystalline morphology of the host structure. Herein, for the first time, we demonstrate the fabrication of a water-soluble red-emitting and dual image-guided therapeutic agent (PQD@ Gd nanoagent). Gadolinium (Gd), an MRI contrast agent, was combined with PF127 amphiphilic polymer via a DMAP/DCC cross-linker to encapsulate and transfer CsM $g_xPb_{1-x}I_3$ QDs into the aqueous phase, as shown in Scheme 1. Under 671 nm laser irradiation, PQD@Gd nanoagent produces a significant amount of reactive oxygen species (ROS). Due to the presence of Gd3+, the PQD@Gd nanoagent exhibits a distinct MRI contrasting response. The phototherapeutic elects and MRI response of the PQD@Gd nanoagent have been demonstrated for in vitro cancer cells. Furthermore, the photocate tic activity of the PQD@Gd nanoagent was also studied for the decomposition of the organic dyes upon visible light treatment. The results suggest that PQD@Gd could be utilized as an e□cient nanoagent for MRI-guided photodynamic therapy and outstanding fluorescent imaging capabilities.

2. EXPERIMENTAL SECTION

2.1. Chemicals. Lead chloride (PbCl₂, 975), lead iodide (PbI₂, 99.9%), octadecene (ODE, ≥99.8%), and oleylamine (OAm, 80-90%) were obtained from Acros Organics. Lead bromide (PbBr₂, 99.98%) and diethylenetriamine penta-acetic acid (DTPA-Gd, ≥99.9%) were found from Alfa Aesar. Toluene (99.8%, Baker Analyzed) was acquired from J.T. Baker. Cesium acetate (CsAc, 99.99%), magnesium sulfate (MgSO 26 9%), rhodamine B (RhB, 95%), trioctylphosphine oxide (T O P, 90%), 5,5-dimethyl-1-pyrroline N-oxide (DMPO, \geq 97%), oleic acid 1411c, 65.0-88.0%), 2,2,6,6-tetramethylpiperidine (TEMP, \geq 99%), 4',6-diamidino-2-phenylindole (DAPI, 98%), ascorbic acid (AA, 99.10%), silver nitrate (≥99%), p21)níc F127 (PF127), o-phenyldiamine (ÓPD, 98%), isopropanol (IPA, ≥99.5%), methylene blue (MB, ≥82%), methyl orange (MO, 85%), anhydrous ethanol (99.5%), ethylenediamine tetraacetic acid (EDTA, 99%), nitro blue tetrazolium (NBT 55 8%), minimum essential medium Eagle (MEME), high-glucose Dulbecco's modified Eagle's medium (DMEM), 2,7-dichlorofluorescin diacetate (DCFH-DA, ≥97%), sodium bicarbonate (NaHCO₃, 99.5—100.5%),1,3-dicyclohexyl carbodiimide (DCC, 5,%), sodium pyruvate, and 4-dimethylaminopyridine (DMAP, ≥99%) were bought from Sigma-Aldrich. WST-1 (4-[3-(4-Iodophenyl)-2-[4-nitrophenyl]-2H-5-tetrazolio]-1,3-benzene disulfonate) was procured from Roche Applied Science. H exane (C₆H₁₄) was obtained 2m UNI-ONWARD (Taiwan). Dimethyl sulfoxide (DM SO) and 1,4-diphenyl-2,3benzofuran, (DPBF, 99.995%) were purchased from J.T. Baker. All

reagents were of analytical grade and were utilized without additional

2.2. Synthesis of CsPbI₃ and CsMg_x _{671-x}I₃ QDs. CsAc (0.1 mmol), 0.3 mmol PbI₂, 0.04 mmol MgSO₄, 0.5 mL of OAm, 1 mL of OAc, and 10 mL of ODE were added and mixed for 3 min by ultrasonication (130 g5 60%) to obtain a uniform solution. Then, TOP (2 mL) was added to the resulting solution to aid in the breakdown of the lea 66 recursor, and the mixture was subjected to microwave heating (200 °C for 15 min). Afterward, the resulting solution was card to room temperature before being subjected to centrifugation at 6000 rpm for 25 min. The supernatant was removed, and the precipitate was dissolved in 10 mL of hexane and centrifuged again (5000 rpm, 4 min) to remove any bigger particles. The supernatant solution (denoted CsMg_xPb_{1-x}I₃) was then collected for further investigation. Except adding a MgSO₄ precursor, the same experimental procedure was followed to produce undoped CsPbI₃ nanocrystals.

The same experimental technique was employed to fabricate mixed halide perovskite QDs CsM $g_vPb_{1-v}X_3$ (X = Cl, Br, I) by varying the amounts of PbCl $_2$ (0.05, 0.1, 0.2, and 0.3 mmol), PbBr $_2$ (0.05, 0.1, 0.2, and 0.3 mmol) precursors with a fixed amount of the CsAc (0.1 mmol) precursor.

- 2.3. Synthesis of PF127-Gd Carriers. The PF127-Gd conjugates were synthesized by adapting the recently reported protocol. ⁴⁰ Briefly, PF127 (100 mg in 10 mL) and DTPA-Gd (25 mg/5 mL) were dispersed in DM SO separately. To act 76 carboxylic groups, DCC (0.05 mmol) and DM AP (0.1 mmol) were added to the DTPA-Gd solution and agitated for 30 min. Subsequently, the 16 yeated DTPA-Gd and PF127 solutions were mixed and reacted for 24 h at room temperature. Finally, the PF127-Gd conjugates were purified by dialysis (Mw cut-o⊡ 12 000 Da) against deionized (DI) water for 3 days and lyophilized.
- 2.4. Encapsulation of Cs 122 $^{\circ}$ $^$
- 2.5. Examination of Reactive Oxygen Species (ROS) Generation. The potential of the PQ D@Gd nanoagent to generate radical oxygen species under laser irradiation was investigated using a DBPF test, a frequently used reagent for ROS detection. In this, 0.4 mL of the PQ D@Gd nanoagent (100 μ g/mL) and 20 μ L of DPBF (1.5 \times 10⁻³ M) solution were mixed in a quartz cuvette and illuminated under a 671 nm light sour \$35\$ a power density of 2.0 W/cm². Then, the PL spectra of DPBF were recorded at an excitation wavelength of 410 nm after every 5 min irradiation for a total of 30 min. The changes in the PL intensity at 460 nm were compared with those before irradiation (0 min).
- 2.6. Characterization of Generated ROS. Electron spin resonance (ESR) was used to analyze the type of ROS produced by PQ D@Gd. The experiment was performed to detect ${}^{1}O_{2}$, ${}^{4}O_{2}$, and ${}^{4}OH$ RO Sby radical trappers 100 mM TEMP (in DMSO) and 100 mM DM PQ Tall th DM SO and ${}^{4}OH$, respectively. In this, 0.9 mL of PQ D@Gd solution was mixed with 0.1 mL of spin trapper (either TEMP or DMPO) and then illuminated for 10 min with a 671 nm laser (2.0 W/cm²) and ESR spectra were recorded.

The generation of ${}^{1}O_{2}$ was further confirmed by analyzing the PL spectra at 1270 nm that we corded after irradiation. The mixture was treated using a 671 nm light source at a power density of 2.0 W/cm² after saturating 3 mL of PQD@Gd (100 µg/mL in $D_{2}O$) with oxygen gas for 10 min. The fluorescence spectra were monitored at around 1270 nm (λ_{2} mm = 671 nm)

around 1270 nm ($\lambda_{excitation}$ = 671 nm). The generation of $^{\circ}O_2^{-}$ radical was examined by the NBT assay following the reported protocol. About 200 μ L of 100 $\frac{32}{2}$ NBT was mixed with 400 μ L of PQD@Gd (100 $\frac{6}{2}$ mL) in a quartz cuvette and treated with a 671 nm light source (2.0 W/cm²) for 5 min. The absorbance $\underline{\text{of}}$ the solution following laser irradiation was monitored at 260 nm.

The generation of *OH radical was investigated using the OPD solution. The *OH radical oxidizes OPD to yellowish 2,3-diaminophenazine (DAP), which exhibits absorbance at 450 nm. Thus, 100 μ L of OPD (10 mM in DI water) was sonicated with 460 μ L of PQD @Gd (100 μ g/mL) and illuminated [40 min with a 671 mm laser (2.0 W/cm²). Then, ultraviolet—visible (UV—vis) absorption spectrometry was employed to measure the absorption spectra of oxidized OPD at 450 nm.

2.7. Photocatalytic Activity of the PQD@Gd Nanoagent. The photocatalytic performance of the PQD@Gd nanagent was examined using the degradation of organic pollutants modamine B (RhB), methyl orange (MO), and methylene blue (MB) under visible lamp irradiation. To attain adsorption—desorption reaction equilibrium between the catalyst and the MO solution, 1 mg/mL PQD@Gd nanoage13 owder was dissolved in 20 mL of MO solution (1 mM in ethanol) and the suspension was stirred for 10 min in the dark. 58 suspension was then illuminated under 671 nm visible light at a distance of 5 cm between the light sour 72 nd the suspension liquid and centrifuged for 10 min at 5000 rpm to remove the photocatalyst nanoparticles. The degradation of MO was detected using UV −vis spectroscopy at 460 nm. The same experimental procedure was followed for the degradation study of 1 mM MB and 1 mM RhB. The absorption spectra of MB and RhB at 664 and 550 nm, respectively, were monitored every 10 min to determine their degradation. The degradation e□tiency was calculated as follows

degradation efficiency =
$$\frac{(A_o - A)}{A_o} \times 100\%$$

where A_{\circ} and A denote the initial concentration and the concentration after the time of irradiation, respectively. The reusability of the PQD@Gd nanoagent was tested toward MO, RhB, and MB organic dyes.

2.8. Detection of Active Species. To identify the role of various types of photocatalytic-dependent radicals and reaction pathways, a series of trapping tests were accomplished. Prior to the addition of the PQD@Gd photocatalyst, silver nitrate (AgNO₃), ascorbic acid (AA), isopropyl alcohol 21 A), ethylenediamine tetraacetic acid (EDTA), and β-carotene were used as radical trappers for holes (h⁺), superoxide radicals (*O₂-), hydroxyl radicals (*OH), electrons (e⁻), and single 23 tygen (¹O₂), respectively. The mixture was exposed to a 671 nm light source at a power source of 2.0 W/cm² and then centrifuged to ren 103 photocatalyst nanoparticles. UV —vis spectroscopy was utilized to determine the concentration of the supernatant solution. The photodegradation was estimated and compared to a control experiment in which no radical trapping reagent was utilized.

2.9. Evaluation of In Vitro Biocompatibility of the PQD@Gd Nanoagent. The WST-1 test was employed to examine in vitro cell viability. The biocompatibility investigations were performed on human liver carcinoma (HepG2) cells, mous 64 elanoma (B16F1), and human cervical cancer (HeLa) cells. First, the cells were cultured to confluence at 37 °C and 5%CO2 in EMEM (for HeLa) or DMEM (for B16F10 30 d HepG2) medium containing 1% antibiotic—antimycotic, 1% nonessential amino acids, 1% L-glutamine, 1% sodium pyruvate, and 10% FBS.

After that, the cells were washed and harvested from culture plates. About 1 × 10⁴ cells/well were placed in each 96-well flat-bottomed plate and cultured for 24 h. Then, 100 µL of fresh medium was supplied along wit 34 arious concentrations of the PQD@Gd nanoagent (0, 100, 200, 3(26,400, and 450 µg/mL) and kept for predetermined time points (24, 48, and 72 h). At each time point, 10 µL of WST-1 reagent was 50 ded to each well and kept for 2 h. To quantify cellular viability, the absorbance at 450 nm was determined using a model 680 XR microplate reader (Bio-Rad). By equating the absorbance value of treated cells with that of the control experiment (untreated cells), the relative cell viability (%) was computed. For every test, the experiment was performed three times.

2.10. Evaluation of PDT-Mediated Cell Toxicity. In this experime 14 cancer cells were cultured in a 96-well plate at 1.0×10^4 per 17 for 24 h at 37 °C and 5% CO $_2$. After that, the old medium was washed with phosphate-bullared saline (PBS), and 100 μ L of fresh cell medium having various concentrations of PQD@Gd was added to each well followed 72 incubation for 6 h for cell internalization. Then, the gells were treated with a 671 nm light source (power density of 2.0 W/cm²) for 5 min. The irradiated cells were incubated for another 24 h, and the cell viability was determined by the WST-1 assay.

2.11. Analysis of In Vitro MR Imaging. To explore the contrasting elect of the as 7 epared PQD@Gd nanoagent, the concentration of Gd³+ ions was assessed by inductively coupled plasma atomic emiss 35 pectroscopy (ICP-AES). Cancer cells were seeded for 1 d₂ in a 50 mL serum bottle with 5 mL of medium added. The old medium was then removed and rinsed using PBS, and fresh medium containing the PQD@Gd nanoagent at various concentrations was added. Cells were then trypsinized, rially and resuspended in PBS after a 24 h incubation period. A Bruker MedSpec 7T MRI machine with a birdcage head coil was utilized to acquire MR pictures of cells inside 0.2 mL tubes.

2.12. Examination of Intracellular Imaging. HepG2, B1 and HeLa cells were cultured for 24 h on glass slides assembled six-well glass plate containing 2 mL of culture medium. After that, the cells were rinsed twice with PBS, fresh cell medium comprising the PQ D@Gd nanoagent at varying concentrations was added to the wells, and the plate was placed in an incubator for 6 h. The cells were harvested by removing the cell medium along with the non-internalized material. Then, the cell nuclei were marked with DAP1 based on the manufacturer's instance on after treatment with 2 mL of ethanol (75% purity). Finally, confocal laser scanning microscopy (CLSM, Leica, Germany) was employed to capture the fluorescence pictures of the cells.

2.13. Endocytosis Tra Lcking Assay. HeLa cells were cultured on glass slides that were fixed in a six-well plate. Next, the cells were pretreated with a culture medium diluted w 77 various inhibitors including amiloride hydrochloride (2.5 mM), 79 tatin (50 μg/mL), genistein (100 μg/mL), and M βCD (16 mM) for 1 h. Following this, the medium was wi 70 drawn and fresh medium containing the PQD @ Gd nanoagent was added and incubated for 3 h. Then, the cells were cultured with DAPI (blue) reagent for 20 min to mark the nucleus structure. In the end, confocal microscopy was used to track nanoagent internalization.

By varying the incubation temperature, an energy-dependent mechanism of cellular ingestion was demonstrated. HeLa cells were cultivated as mentioned above and supplied with nanoagents at various temperatures suc 32 4 and 37 °C. The glass slides were made as previously described, and the images were obtained using confocal microscopy.

2.14. Intracellular ROS Detection. HeLa cells were grown for 1 day as indicated in Section 2.12. After that, fresh medium containing the PQD 73 d nanoagent at various concentrations was supplied to the cells followed by incubation for 6 h. The medium was then removed from each well, and the wells were 2 sed twice with PBS to eliminate any non-ingested material. Then, the cells were incuba 61 with 10 µL of DCFH-DA for 2 h and exposed to 671 nm light (2.0 W/cm²) for 5 min. Finally, the cell images were recorded using confocal microscopy.

2.15. Characterization Techniques. O ptical characterization of the as-prepared material was performed by ultraviolet—visible absorption spectrometry (UV—vis; model FP-6500, Jasco Co. Ltd.), photoluminescence spectrometry (PL; model Fluorolog-3, Horiba Jobin Yvon), and time-resolved photoluminescence [69] PL; model NanoHarp, Pico Quantity). The morphological study was carried out by transmission electron microscopy (TEM; m [62] Tecnai F20 G2 FETEM, Philips; model JEM-2100F, JEOL). X-ray photoelectron spectroscop [14] XPS) was conducted using an ESCALAB 250 (VG Scientific). X-ray dicitation (XRD) was performed using a Bruker D8 Discover dicitactometer equipped with monochromatized Cu Ka radiation (wavelength 1.54 Å). Fourier transform infrared (FTIR)

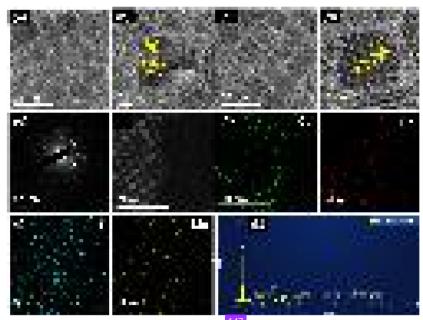


Figure 1. TEM images of (a) $Cs^{m}g_{x}Pb_{1-x}I_{3}$ QDs. (b) $Cs^{m}g_{x}Pb_{1-x}I_{3}$ QDs. HRTEM images of (c) $Cs^{m}g_{x}Pb_{1-x}I_{3}$ QDs. (e) SAED image of $Cs^{m}g_{x}Pb_{1-x}I_{3}$ QDs. (f) HAADF-scanning transmission electron microscopy (STEM) image and elemental mapping images of (g) Cs, (h) $Cs^{m}g_{x}Pb_{1-x}I_{3}$ QDs. (k) EDS spectrum for $Cs^{m}g_{x}Pb_{1-x}I_{3}$ QDs. (b) $Cs^{m}g_{x}Pb_{1-x}I_{3}$ QDs. (c) $Cs^{m}g_{x}Pb_{1-x}I_{3}$ QDs. (c) $Cs^{m}g_{x}Pb_{1-x}I_{3}$ QDs. (d) $Cs^{m}g_{x}Pb_{1-x}I_{3}$ QDs. (e) $Cs^{m}g_{x}Pb_{1-x}I_{3}$ QDs. (e) $Cs^{m}g_{x}Pb_{1-x}I_{3}$ QDs. (f) $Cs^{m}g_{x}Pb_{1-x}I_{3}$ QDs. (g) $Cs^{m}g_{x}Pb_{1-x}I_{3}$

spectrometry was performed using an FTS-3500 (Bio-Rad). Ultraviolet photoelectron spectrometry (UPS) (ESCALab250Xi, Thermo Scientific) was used to estimate the valence band (VB) of QDs. A Zetasizer 20 61 M ALVERN) was utilized to examine the ζ-potential. The average size of the nanomaterials was computed by dynamic light scattering (DLS) (model Nano-ZS90, Malvern). The viable cell measurement was performed b 33 pploying an ELISA reader (model Power Wave XS, BioTek). Inductively coupled plasma atomic emission spectroscopy (ICP-AES; Horiba JY2000-2) was utilized to calculate the amount of Gd in PQD@Gd nanocomposites. Electron spin resonance (EPR) spectra were acquired at 19 using a Bruker Elexsys E-580 spectrometer (Bruker, Germany). Magnetic resonance imaging (MRI) was performed on a Bruker MedSpec 78 whole-body system (Ettlingen, Germany) with a birdcage head coil. Confocal laser scanning microscopy (CLSM) under a TCS SP2 device (Leica, Germany) was employed to capture the fluorescence and cellular images. Rhodamine B dye (QY = 68.9% in 95% ethanol) was used as the standard reference to calculate the QY of red-emitting QDs,

 $\mathrm{QY_{NCs}} = \mathrm{QY_{ref}} imes rac{a_{ref}}{a_{NCs}} imes rac{A_{NCs}}{A_{ref}} imes \left(rac{n_{NCs}}{n_{ref}}
ight)^2$, where the notions N C s and ref represent the as-prepared perovskite material and standard reference dye, respectively, a is the absorbance adjusted in between 0.05 and 0.059, and A refers to the integrated area of the PL intensity for both N C s and the reference dye.

80 3. RESULTS AND DISCUSSION

3.1. Preparation and Characterization of CsPbI $_3$ and CsMg $_x$ Pb $_{1-x}I_3$ QDs. The highly luminescent red-emitting CsMg $_x$ Pb $_{1-x}I_3$ NCs were synthesized using the microwave heating technique, as shown in Figure S1. In a standard experiment, CsAc, PbI $_2$, MgSO $_4$ (source of the Mg dopant), and a capping ligand as the stabilizer were mixed appropriately and subjected to optimized reaction conditions (200 $^{\circ}$ C, 15 min). The amount of Mg in CsMg $_x$ Pb $_{1-x}I_3$ NCs was varied using di $_1$ erent molar ratios of MgSO $_4$. The undoped CsPbI $_3$

NCs were synthesized using the same procedure by avoiding the use of M $\mbox{gSO}_4.$

The structural morphology of the as-synthe CsPbI3 and $CsMg_xPb_1$ $\longrightarrow 1180$ Ds was determined using X-ray dilutaction (XRD) and transmission electron microscopy (HRTEM). The TEM images of $CsPbI_3$ (Figure 1a) and $CsMg_xPb_{1-x}I_3$ (Figure 1b) indicated that both the fabricated NCs had the same cubelike morphology and well-oriented lattice fringes with estimated sizes of 14 and 15 nm, respectively. The cubic geometry of the NCs remained unchanged even after doping with M g²⁺ ions. The DLS test was employed to estimate the size distribution of $CsPbI_3$ and $CsMg_xPb_{1-x}I_3$ QDs, and the obtained results validated that both $CsPbI_3$ and $CsM g_xPb_{1-x}I_3$ Name had average sizes of 14.79 and 15.62 nm, respectively (Figure S2a,b). The HRTEM image of CsM $g_xPb_{1-x}I_3$ QDs (Figure 1d) showed that the interplanar distance of the (221) lattice plane was 0.56 nm and well matched with the crystalline plane of pristine CsPbI₃ NCs (Figure 1c). However, the interplanar distance was 0.55 nm (Figure S2c) for 0.08 mmol Mg²⁺-doped CsPbI₃ QDs. The representative selected area electron dillaction (SAED) pattern for CsM $g_xPb_{1-x}I_3$ QDs shown in Figure 1e illustrated that the perovskite cucture possessed a high degree of crystallinity. Using elemental mapping and energy-dispersive X-ray spectroscopy (EDS) techniques, the integration and distribution of compositional ions, including the guest cation (M g²⁺), were demonstrated. We noticed that the EDS scans were carried out in regions with a high number of NCs in an attempt to 87 her clinically significant information. Figure 1f depicts a magnified highangle annular dark-field (HAADF) image showing that $CsMg_xPb_{1-x}I_3$ QDs owned cubic morphology. Figure 1g-jdisplays the elemental mapping results, which reveal that each individual ion, such as Cs, Pb, I, and Mg, is evenly distributed across the lattice structure. Furthermore, no phase segregation

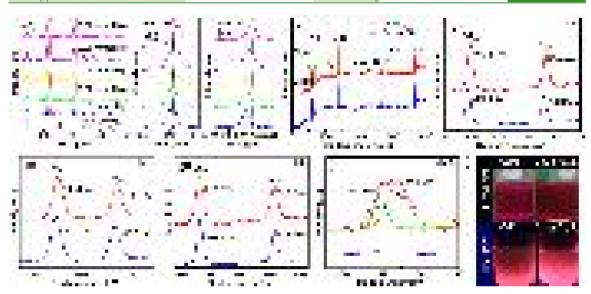


Figure 2. (a) XRD pattern of $CsPbI_3$ QDs doped with dilarent molar ratiod (0, 0.02, 0.04, 0.06, and 0.08 mmol) of M g^{2+} ions. Magnified view of the (b) 220 and (c) 221 XRD peaks with a systematic shift in the peak position with a gardinary of CsPbI3 and CsM $g_xPb_{1-x}I_3$ QDs: (d) survey spectra of $CsPbI_3$ and $CsM g_xPb_{1-x}I_3$ QDs and $g_xPb_{1-x}I_3$ (right) QDs taken at daylight and under a UV lamp, respectively.

or surface preference were identified, suggesting that the M g²⁺ ion distribution was uniform. Inductively coupled plasma atomic emission spectroscopy (ICP-AES) measurements were performed to further validate the practical concentration of M g²⁺ in C sM g_xPb_{1-x}I₃ Q D s. It was observed that 0.011, 0.032, 0.051, and 0.069% M g²⁺ ions were present in C sM g_xPb_{1-x}I₃ QDs synthesized using 0.02, 0.04, 0.06, and 0.08 mmol M gSO 4, respectively (Table S1), suggesting that a substantial quantit 120 M g²⁺ ion was integrated into the QD structure. These results are completely in agreement with the data obtained using EDS mapping.

The XRD peaks of CsPbI₃ and CsM g_v Pb_{1-v}I₃ QDs shown in Figure 2a were in line with those of standard data (JCPDS#79-0874), and they were mismatched to each other due to Mg doping. The di⊡action pattern demonstrated that $CsMg_xPb_{1-x}I_3$ with the Pm3m space group was highly crystalline and adopted a cubic phase. Moreover, di derent strong peaks were observed at 0, 15.38, 21.67, 31.6, 34.58, 36.9, and 44.3°, which could be well oriented to 110, 220, 211, 310, 221, and 112 crystal faces of the cubic perovskite phase, respectively. In the XRD scans, no additional impurity peaks or new phases attributed to Mg ions were observed, suggesting that doping with M g ions did not alter the crystal arrangement of pristine CsPbI3. However, further analysis of peaks of 220 and 211 planes (Figure 2b,c) revealed that doping with M g²⁺ ions caused peak shifts, which became larger as the concentration of M $\rm g^{2+}$ increased. The shift in peak positions could be created to lattice shrinkage caused by the exchange of larger Pb ions ($r_{Pb} = 0.133$ nm) with smaller M g^{2+} ions (r_{Mg} = 0.087 nm). Precisely, as the M g²⁺ concentration increased from 0 to 0.08 mmol, the di daction peaks drifted toward a higher angle, implying that smaller M g²⁺ cations replaced Pb²⁺ cations in octahedral positions and resulted in progressive lattice contraction. The shifting panel of the (220) and (221) peaks in Figure 3b,c indicated that M g²⁺ enters the host by

occupying lead lattice sites in the perovskite crystal structure. Moreover, the tolerance factor (TF) was computed using the following equation 21

$$TF = \frac{r_{A} + r_{X}}{\sqrt{2} \left(r_{B} + r_{X} \right)}$$

The TF value of CsM $g_xPb_{1-x}I_3$ N Cs was deduced as 0.93, which is better than that of undoped CsPbI $_3$ (0.80), suggesting that doping with smaller-sized metal ions such as M g^{2+} could enhance the phase stability of CsM $g_xPb_{1-x}I_3$ over CsPbI $_3$ QDs. 42 /A $_3$

The distribution of ions in $CsPbI_3$ and $CsMg_xPb_{1-x}I_3$ Q Ds was investigated by employing X-ray photoelectron spectroscopy (XPS). As displayed in Figure 2d, the complete XPS scan \mathbf{g} ectra of both CsPbI_3 and $\mathsf{CsMg}_\mathsf{x}\mathsf{Pb}_{1-\mathsf{x}}\mathsf{I}_3$ exhibited peaks corresponding to Cs 3d, Pb 4f, and I 3d at around 725, 138, and 628 eV, respectively. A new peak at around 53 eV corresponding to M $_{113}$ p was observed in C sM $\rm g_xPb_{1-x}I_3$ spectra, confirming the doping of M g ions into the host lattice. Be 24se of capping ligands, additional signal peaks relating to N 1s, O 1s, C 1, 2p, and P 2p were observed in the XPS spectra. The peaks of Cs 3d, Pb 4f, and I 3d orbitals in QD samples were close-fitting with identical dominating peaks, signifying that M g²⁺ doping does not a Ect the local binding environment. The changes in the binding energies were analyzed using high-resolution XP (HRXPS). The Cs 3d peak of CsPbI₃ NCs deconvoluted into two peaks at binding energies of 738.2 and 724.3 eV, which could be ascribed to $3d_{3/2}$ and $3d_{5/2}$ signals, respectively; however, the binding energies of these signals in CsM g_xPb_{1-x}I₃ QDs positively shifted by 0.4 and 0.6 eV, respectively (Figure 2e). The Pb 4f peak of CsPbI₃ NCs exhibited two integrated peaks for 4f_{5/2} and $4f_{7/2}$ at B.E. values of 142.8 and 137.9 eV, respectively (Figure 2f); however, the binding energies of Pb 4f_{7/2} and 4f_{5/2} in $CsM q_v Pb_{1-v} I_3$ QDs shifted to 143.1 and 138.1 eV,

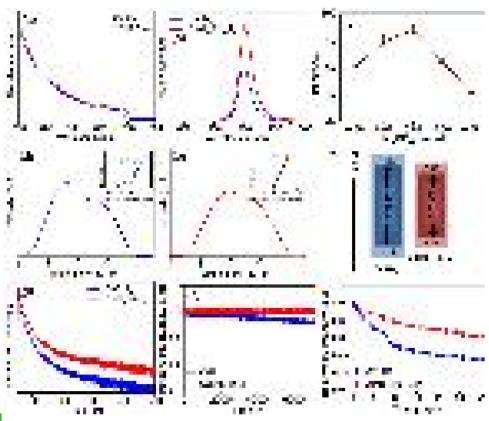


Figure 3. (a) Absorption spectra of $CsPbI_3$ and $CsMg_xPb_{1-x}I_3$ QDs. (b) PL intensity of $CsPbI_3$ and $CsMg_xPb_{1-x}I_3$ QDs measured using an excitation wavelength ($\lambda_{bccitation}$) of 370 nm. (c) PLQY variation properties of $CsMg_xPb_{1-x}I_3$ QDs versus diLaerent molar ratios of the MgSO₄ precursor. UPS spectroscopic analysis 107 d) $CsPbI_3$ and (e) $CsMg_xPb_{1-x}I_3$ QDs. (f) Schematic diagram of the band gap energy level diagrams for $CsPbI_3$ and $CsMg_xPb_{1-x}I_3$ QDs. (g) Time-resolved PL decays for excitonic luminescence of $CsPbI_3$ and $CsMg_xPb_{1-x}I_3$ QDs. (h) Photostability performance of $CsPbI_3$ and $CsMg_xPb_{1-x}I_3$ QDs after continuous excitation at 370 nm wavelength. (i) Relative PL intensity properties for $CsPbI_3$ and $CsMg_xPb_{1-x}I_3$ N Cs measured at dil arent storage days.

respectively. T hese outcomes in plied that the addition of M g^{+2} to the lattice could prevent the formation of I vacancies in CsPbI₃ NCs and influence the electron density surrounding the host cations of Cs 3d and Pb 4f. The decompluted XPS spectra of I 3d in CsPbI₃ QDs demonstrated two peaks at 628.4 and 618.3 eV, corresponding to $3d_{5/2}$ and $3d_{3/2}$ signals, respectively, as shown in Figure 2g; nevertheless, in $CsM g_xPb_{1-x}I_3$ N Cs, these peaks shifted positively by 0.2 eV for $3d_{5/2}$ and 0.3 for $3d_{3/2}$ eV, which could be because of the formation of Mg-I cross bonds in the octahedral position. After doping with Mg2+ ions to host CsPbI3 NCs, the positive shifts the B.E. of Cs $(3d_{5/2}$ and $3d_{3/2})$, Pb $(4f_{7/2}$ and $4f_{5/2}$), and I ($3d_{5/2}$ and $3d_{3/2}$) suggested the existence of a halide-rich chemical environment around the [PbI₆]4octahedra. The HRXPS of Mg showed two distinct peaks at B.E. values of 51.52 and 53.89 eV (Figure 2e), which could be ascribed to the $2p_{1/2}$ and $2p_{3/2}$ peaks of core Mg²⁺, respectively, validating the integration of M g⁺² ons into the host NCs. These observations further verified that the Mg²⁺ ions integrated into the CsPbI₃ perovskite matrix replaced the Pb²⁺ sites, that the Mg²⁺ 9ns had a greater binding a□hity with I- than Pb2+, and that the interactions between extranuclear and core electrons in both these neighboring ions (Pb²⁺ and

I⁻) were partially hindered due to M g's lower electronegativity than Pb. Similar trends were identified in Ni-doped CsPbCl_xBr_{3-x} and Cd-z₄ grated CsPbCl₃ NCs. ^{44,45} The obtained XPS and XRD results, which are in good agreement with each other, indicated the partial substitution of Pb²⁺ octahedral ions in PbI₆ by M g²⁺ in the CsM g_xPb_{1-x}I₃ Q Ds and increased phase stability and uniformity of the PNCs upon doping with M g.

3.2. Photophysical Properties of CsMg_xPb_{1-x}I₃. The photophysical characterizating of CsPbI₃ and CsMg_xPb_{1-x}I₃. QDs were monitored using UV—vis absorption and fluorescence spectroscopy techniques. The as-prepared resulting NCs dispersed in hexane emitted dark red luminescence light in daylight and bright red light under UV irradiation, as shown in Figure 2h. The UV—vis absorption specifically of CsMg_xPb_{1-x}I₃ NCs demonstrated a slight variation in the first excitonic peak located at 683 nm and the absorption band compared to those of CsPbI₃ NCs (Figure 3a), revealing that Mg²⁺-doped QDs were slightly larger in size. The absence of subsequent structural alteration after successful integration of the Mg²⁺ ions was could be noted by the presence of a weak band edge (Urbach tail), which further implies that both pristine and Mg-doped samples had superior crystallinity with

a sharp absorption peak and no substantial variations in spectral shapes. Figure 3b depicts the emission spectra of $C\, sP\, bI_3$ and $C\, sM\, g_x Pb_{1-x}I_3$ Q Ds measured at an excitation wavelength ($\lambda_{\text{excitation}}$) of 370 nm. It was observed that CsM g_xPb_{1-x}I₃ Q Ds exhibited a significantly higher PL intensity than pristine CsPbI₃ Q Ds under similar conditions, with very minor red shifts from 699 to 702 nm. The narrow peak spectra of the QDs $_{\overline{\mbox{\scriptsize 93}}}\mbox{\scriptsize gested}$ that QDs with uniform sizes were synthesized. Moreover, the full width at half-maximum (FWHM) of 0.04 mmol Mg-doped QDs was measured to be 38 nm, which is slightly higher than that of undoped QDs (36.2 nm). The increase in FWHM and the slight shift in absorption spectra could be ascribed to the increased size of QDs upon doping with Mg²⁺, which is in good agreement with recent reports on Zn2+- or Ni2+-doped CsPbI3 NCs.21,46 Additionally, the PLQY of resulting Mg-doped QDs was plotted against the MgSO₄ feeding ratio (Figure 3c), showing that the PLQY increased progressively from 60.2% (undoped QDs) to 89.1% for 0.04 mmol Mg-doped QDs and then decreased to 44.6% for 0.08 mmol Mg-doped QDs. Henceforth, CsM g_xPb_{1-x}I₃ Q Ds means CsPbI₃ N Cs doped with 0.04 mmol MgSO₄. To further confirm the minor variation in the absorption spectra of $CsPbI_3$ and $CsMg_xPb_{1\rightarrow x}I_3QDs$, we used UPS to analyze their band gap di rence. Thus, the valence band may for undoped and M g-doped Q Ds were observed at 2.19 and 1.98 eV, respectively. The optical band gap energies of $CsPbI_3$ and $CsMg_xPb_{1-x}I_3$ QDs were computed by extrapolating 134ar regions from (ahv)2 plots against photon energy (hv), as shown in Figure S3. By integrating the optical band gap energies, the panduction band minima of CsPbI₃ and $CsMg_xPb_{1-x}I_3$ Q Ds were calculated to be 1.35 and 1.22 eV, respectively. These experimental results confirmed that M g²⁺ cations were successfully doped into the CsPbI3 framework

To acquire a better understanding of the remarkable improvement in PLQY and adiative recombination rate of M g²⁺-doped CsPbI₃ Q Ds, time-resolved PL (TRPL) lifetime measurements were performed using TCSPC techniques. The time-resolved PL decay lifetimes of pristine and M g²⁺-doped CsPbI₃ NCs were studied at an excitation wave 137 h of 378 nm. The biexponential decay function equatio 21 t) = A_1e^{-t/T_1} + A 2e-t/T2 was used to fit the PL decasturves, where I(t) refer to the PL function of time (t) and A1 and A2 represent the amplitudes of shorter T_1 and longer T_2 lifetimes, respectively. The average PL lifetime (τ_{ave}) was estimated from A_i and τ_{i} values by the following equation: $\tau_{ave} = \frac{(A_1 \tau_1^2 + A_2 \tau_2^2)}{(A_1 \tau_1 + A_2 \tau_2)}$. Table S2 provides the propriate fitting parameters and their respective amplitudes. The average decay lifetime of CsPbI₃ QDs was computed to be 30.43 ns, with τ_1 and τ_2 of 11.55 and 49.68 ns, respectively, whereas the $CsMg_xPb_{1-x}I_3$ QDs exhibited an average PL lifetime of 51.89 ns, with a τ_1 of 17.24 ns and a τ_2 of 86.54 ns. The PL decay lifetime of CsPbI₃ NCs increased to 51.78 from 30.12 ns after doping with M g²⁺, suggesting that Mg doping can promote exciton recombination via the radiative pathway while suppressing nonradiative decay and surface recombination, thereby improving the PLQY of CsM $g_xPb_{1-x}I_3$ QDs by up to $\square 89\%$. From this, the plausible explanation of the e⊡ect of M g concentration on PLQY was deduced as follows: (applied on a small concentration of Mg into the CsPbI3 host lattice could lift the order in the local chemical binding of lead and promote short-range ordering without making a new recombination channel that would

encourage radiative recombination by reducing surface trap states, thereby leading to an enhancement in PLQY. (2) However, an excess amount of M g^{2+} ions could produce more surface trap states, which causes a reduction in the average lifetime and radiative decay rate of QDs and thereby decreases the PLQY of resulting $C\,sM\,g_xP\,b_{1-x}\,I_3$. Additionally, the presence of H $_2O$ molecules and $SO\,_4^{2-}$ ions in the Mg precursor is responsible for ionic instability and aggregation of PNC structures, respectively, resulting in low PLQY.

N ext, the stability of the $CsPbI_3$ and $CsMg_xPb_{1-x}I_3$ N Cs to light and region was investigated. Figure 3h shows the relative PL stability of the as-preparate perovskite QDs under continuo (12) V lamp irradiation at an excitation wavelength of 370 nm. After 2 h of continuous UV lamp irradiation, the PL intensity of the CsPbI₃ QDs decreased by 9.5%, whereas that of the CsM $g_xPb_{1-x}I_3$ Q Ds remained unchanged, indicating that $Mg_xPb_{1-x}I_3$ had greater photostability than the undoped QDs. The elect of moisture on the stability of QDs was examined by measuring the PL intensity of QDs after one month of storage. Figure 3i depicts that CsPbI₃ and $CsMg_xPb_{1-x}I_3$ QDs retained 40 and 70%, respectively, of their initial PL intensities, suggesting that $C sM g_x Pb_{1-x}I_3 Q D s$ had more storage stability than CsPbI₃ QDs. The sulfate ions present on the surface of CsM $g_xPb_{1-x}I_3$ QDs could create steric hindrance and protect QDs from air or moisture, empowering long-term stability.

To investigate the underlying mechanism for the enhanced optical properties of $CsMg_xPb_{1-x}I_3$ NCs, various control experiments were conducted. Upon confirming that Mg⁺² could increase the PL intensity, the $e^{\Box e}$ ct of M g^{+2} concentrations on PL intensity was examined by varying to feeding ratio of the Mg precursor from 0 to 0.08 mmol. As shown in Figure S4a, the PL intensity of resulting M g-doped NCs increased as the feeding ratio of MgSO₄ increased from 0 to 0.04 mmol; however, it diminished upon a further increase in the MgSO4 feeding ratio. The dopant precursor MgSO4. 2H ₂O comprised M g²⁺ ions, SO ₄²⁻ iong nd 2 moles of water molecules, so further investigation was carried out to study the elect of each ion/molecule on the PLQY of CsM $g_xPb_{1-x}I_3$ NCs. In the literature reports, it has been mentioned that a small amount of the cation dopant or additive can significantly improve the optical properties of QDs as well as their morphological structures. For instance, recent studies have found that M -site-doped perovskite structures (substitution of Pb²⁺ ions), such as Bi-doped CsPb[36]nd Ni-doped Cs_xPb_{1|x}J₃, resulted in an increased PLQY of perovskite QDs due to the elimination of the surface trap state and increased radiative decay rate. 21,46,47 M gI $_2$ was used in place of M gSO $_4$ 2H $_2$ O as the Mg precursor to figure out the elect of Mg+2 ions on PLQY. The PLQY of resulting C sM $g_xPb_{1-x}I_3$ Q D s increased to 73.4% for 0.1 mmol M gI₂ and then decreased upon a further increase in MgI₂ (Figure S4b), indicating that Mg²⁺ ions are crucial for PL enhancement of CsMg_xPb_{1-x}I₃ NCs. Similarly, sulfobetaine and phosphocholine were used as zwitterionic capping ligands in the fabrication of CsPbBr₃ QDs, resulting in enhanced chemical stability and high quantum yield (90%). In another report, a combination of an anionic ligand and PbBr₂ reduces the surface traps, stabilizing the PbX₆ octahedral structure and restoring the high QY performance. 15 Subsequently, 10 µL of H₂SO₄ was employed as the additive in the synthesis of PNCs to mimic the SO₄²⁻ ions of MgSO₄. The PLQY of resulting CsPbI₃ NCs (68.9%) was higher than that of pristine CsPbI₃ NCs (60.2%) (Figure S4c). These outcomes

proved that both M g^{2+} ions and SO g^{2-} ions play key roles in the augmented PLQY of CsM g_x Pb $_{1-x}I_3$ QDs. This study has also proved that incorporating SO $_4^{2-}$ ions into perovskite synthesis improves stability by causing a steric hindrance elect and eliminates surface traps, resulting in improved optical characteristics with long-term durability. In recent studies, CsPbBr₃ QDs with improved PL properties and photostability were synthesized using water additives. 49 Therefore, CsPbI₃ NCs were synthesized using water molecules as additives; however, the PL intensity of as-synthesized NCs was found to be water-intolerant, and the emission peak diminished as the amount of water was increased, indicating that H₂O molecules in the MgSO₄·2H₂O precursor did not contribute to the enhanced optical properties of CsM g_xPb_{1-x}I₃ QDs. Taking this discussion into consideration, the improvement in PLQY of CsM $g_xPb_{1-x}I_3$ QDs could be credited to the synergistic elect of incorporated Mg^{2+} and SO_4^{2-} ions. The role of each ion could be plained as follows: (1) M g²⁺ ions could reduce the surface defects to some extent, which was supported by increased radiative decay rates, resulting in enhanced PLQY. (2) SO₄²⁻ surface passivation could create a stronger steric elect between the capping ligands and eliminate surface trap states, which is more beneficial to produce high-quality NCs. The other possible mechanism for the improved PLQY of $CsMg_xPb_{1-x}I_3$ QDs is most likely owing to Mg-induced structural stability. Thus, $CsMg_xPb_{1-x}I_3$ QDs have a Goldschmid tolerance factor of 0.93, which is greater than that of CsPbI₃ QDs. As a consequence, using a smaller Mg²⁺ dopant in the Pb2+ site increases the tolerance factor, which is beneficial for exciton radiative recombination (resulting in a higher PLQY) and improved phase stability. Regardless of radiative recombination, there is a non-radiative relaxation phenomenon that develops in energy loss either by hole trapping or electron trapping. Hence, this could be the reason for the low PLQY of CsM gxPb1-xI3 QDs with excess MgSO4 dopant. Therefore, the increase in PL lifetime suggests that the addition of a small amount of M gSO₄ in CsPbI₃ QDs reduces the density of non-radiative recombination centers and surface trap states, simultaneously increases the radiative recombination rate, and eliminates surface trap states as well as the M g²⁺ and SO₄²⁻ ion incorporation improves the phase stability and leads to a higher steric hindrance e dct, respectively, finally resulting in improved PLQY with prolonged stability.

Besides, various 138 (X: CI, Br, and I) compounds in dildrent proportions were combined with fixed amounts of CsAc and MgSO₄ precursors to produce mixed freque perovskite NCs with wavelength-tunable PL intensity in the visible region, as shown in Figure S5. Mg-doped CsPbX3 QD colloidal solution emitted strong light under UV lamp illumination that covers the entire visible spectrum from blue to greenson red, as illustrated in Figure S5a. Figure S5b,c depicts the absorption and PL spectra of M g-doped CsPbX 3 NCs, which covered the 20 ble spectral range from 393 to 710 nm, exhibiting a higher absorption coe □cient, FWHM (from 14 to 45 nm), and QY (up to 89%). The narrow FWHM values indicate that the NC distribution is homogeneous and that the materials are monochromatic. Moreover, because of the high quantum configurent of NCs, a significant Stokes shift was detected when comparing the absorptic 20 pectra with the PL emission spectra. This was obtained from radiative recombination of free exciton or bound exciton recombination. This method is most likely the facile and most straightforward

technique to produce highly emissive perovskite $\ensuremath{\mathsf{QDs}}$ with excellent optical properties.

3.3. Synthesis and Characterization of the POD@Gd Nanoagent. Based on the above optical properties, it is clear that the as-synthesized CsM $g_x Pb_{1-x}I_3$ QDs (hereafter referred to as PQDs) can be used as phototherapeutic and imaging agents. However, their solubility in aqueous media is one of the major concerns. To address the increasing demands for imaging-guided therapeutic agents and improve the bioavailability and properties of the CsM $g_xPb_{1-x}I_3$ QDs, we designed and assembled the PQD@Gd theranostic platform, as illustrated in Scheme 1. The PQD@Gd nanoagent was prepared through an encapsulation mechanism at room temperature. First, the PF127-Gd carrier was synthesized by an esterification reaction between the hydroxyl group of PF127 and the carboxylic group of the DT PA-Gd contrast agent using the DCC/DMAP cross-linker, as indicated in Scheme 1. The resulting PF127-Gd conjugates could be employed for encapsulation of hydrophobic drugs due to the amphiphilic nature and MRI response because of the conjugated Gd³⁺ contrast agent. PF127 could self-assemble into a robust micelle structure because of its amphiphilic characteristics, in which the PPO segment (hydrophobic section) of PF127 acts as the core of the micelle via hydrophobic interactions. Thus, the assynthesized CsM $g_{\mbox{\tiny X}}\mbox{Pb}_{1-\mbox{\tiny X}}\mbox{I}_3$ Q Ds were loaded into the hydrophobic core of the PF127-Gd micelle via hydrophobic interactions between the PPO segment of PF127 and a longchain capping ligand such as OAm, OAc, or TOP (Scheme 1), and the resulting nanoparticles were named as PQD@Gd nanoagents. The hydrophobic interaction was possibly developed. The fabricated PQD@Gd nanoagent exhibited excellent solubility in aqueous solutions by taking advantage of the hydroxyl end of PEO, which is located at the outer edge of the micelle. The as-synthesized PQD@Gd nanoagent demonstrated a spherical micelle or shell-like structure, as shown in TEM profile (Figure S6a). As shown in Figure S6a, several perovskite QDs clustered together inside a micelle structure and maintained their cubic form after polymer embedding, suggesting that the PF127-Gd carrier successfully shielded $CsMg_xPb_{1-x}I_3$ QDs from the aqueous environment. The size distribution of a nanoagent was further analyzed usi measurements (Figure S6b), and the results indicated that the average hydrodynamic diameter of PQD@Gd was 315 nm (polydispersity index, PDI = 0.41). The FTIR spectra were recorded to study the surface properties of CsM gxPb1-xI3 QDs before and after encapsulation. The results shown in Figure S6c demonstrated that $CsMg_xPb_{1-x}I_3$ NCs had strong IR sorption bands at 3400, 2989, 1720, 1212, and 586 cm⁻¹, which could be assigned to stretching frequencies of -OH/-NH, C \rightarrow H, C \square O/C \square N, C \rightarrow O/C \rightarrow N, and P \square O interactions, respectively, due to the presence of the capping ligand. M oreover, the IR peak shown at 1450 $\rm cm^{-1}$ is ascribed to $\rm S\square$ O bonding, confirming that SO₄²⁻ acts as a capping ligand to stabilize the surface of perovskites QDs. After encapsulation, because the PF127-Gd polymer is rich in hydroxyl and amine groups, 127 QD@Gd nanoagent exhibited a broader IR peak at 3500 cm-1 is ascribed to the C-H stretching frequency, $C \square O/C \square N$ stretching, $C \square C$ vibration, C - O / C - N stretching, and C - O / CC—C stretching were identified in the IR spectra at 1700, 1498, 1389, and 1054 cm⁻¹, respectively. Afterward, the surface charge properties of the PQD@Gd nanoagent were investigated using a zetasizer. The presence of PO_4^{-3} , COO_7 , and

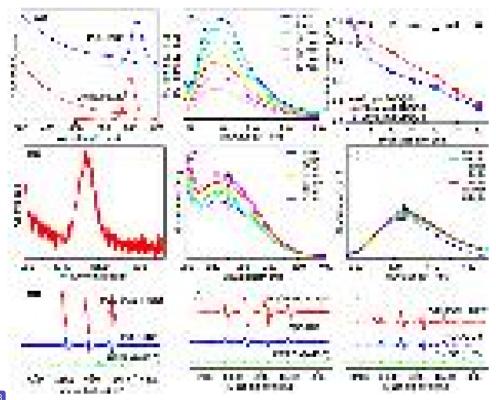


Figure 4. (a) Absorption and PL emission spectra of $CsMg_xPb_{1-x}I_3$ QDs and PQD@Gd nanoagents. (b) PL intensity of 1.5×10^{-3} [34] PBF solution ($\lambda_{excitation} = 410$ nm, $\lambda_{emission} = 460$ nm) in the presence of 100 [45] n PQD@Gd nanoagent at dilarent time intervals upon 671 nm laser irradiation (2.0 W/cm²). (c) Relative PL intensity of DPBF at 460 nm in the presence of various concentrations of the PQD@Gd nanoagent at dilarent laser irradiation times. (d) Emission spectrum of $^{1}O_2$ measured at 1270 nm using the PQD@Gd nanoagent (dissolved in D $_2$ O) after 671 nm laser irradiation. Absorption spectra of (e) 100 mM N BT and (f) 10 mM O PD coupled with the PQD@Gd nanoagent upon irradiation with nm laser at various time intervals. ESR spectroscopic analysis of (g) TEMPO signals in the presence of $^{1}O_2$ obtained for the PQD@Gd nanoagent with and without laser irradiation. (h) ESR signal for DMPOO adduct formation in the PQD@Gd nanoagent solution with and without laser irradiation for $^{\bullet}O_2$ radical detection. (i) DMPOO signal responding to $^{\bullet}O$ H radicals in the presence of the PQD@Gd nanoagent with and without laser illumination.

SO₄²⁻ ions from the capping ligand conferred negative surface charge (-22.68 mV) to CsM $g_x P b_{1-x} I_3$ QDs before encapsulation, as shown in Figure S6d. The PQD@Gd nanoagent displayed a more negative surface charge (-29.09 mV) probably due to the hydroxyl ions of the PF127 polymer, proving that the $CsN_{73}Pb_{1-x}I_3$ NCs were successfully encapsulated. Figure 4a shows the absorption and PL spectra of CsM g_xPb_{1-x}I₃ QDs before and after encapsulation. The absorption spectra of the POD@Gd nanoagent exhibited a smooth absorption curve at 693 nm, which corresponds to the same position as in the spectra of $CsMg_xPb_{1-x}I_3$ QDs. However, in PL spectra, the PQD@Gd nanoagents demonstrated a considerable shift in the PL peak contained to free $CsMg_vPb_{1-x}I_3QDs$. This peak shift could be due to the QD aggregation and interdot energy transfer.⁵⁰ Furthermore, the PLQY of the PQD@Gd nanoagent was calculated as 53.4%, which is slightly lower than that of non-encapsulated CsM $g_v Pb_{1-x}I_3$ Q Ds (89.1%) suggesting that the encapsulated QDs preserve their intrinsic properties in an aqueous solvent. Time-resolved PL decay lifetimes of C sM $g_x Pb_{1-x}I_3$ Q D s and PQD@Gd nanoagent were further investigated under the same conditions and compared. The obtained results depicted in

Figure S6e indicated that excited electrons possess a relatively long lifetime. The average PL lifetime of the PQD@Gd nanoagent was 23.36 ns (inset of Figure S6e), which is lower compared to 51.89 ns of nonencapsulated CsM g_x Pb_{1-x}I₃ QDs, indicating that PF127-Gd micelles might result in loss of the surface ligand during the encapsulation mechanism. This would cause the formation of more surface trap states and additional nonradiative channels, thereby resulting in a reduced PL lifetime and lower QY electioncy.

Moreover, the photostability of $CsMg_xPb_{1-x}I_3$ and PQD@Gd nanoagents was investigated by applying UV lamp irradiation at specified time intervals. Figure S6 reveals that the relative PL intensities of $CsMg_xPb_{1-x}I_3$ NCs and PQD@Gd nanoagents were reduced by 34 and 45%, respectively, after 48 h, as compared to the initial PL intensity evaluated at 0 h. The $CsMg_xPb_{1-x}I_3$ (before and after encapsulation) produce bright color after being exposed to UV light for 48 h, as shown in the inset image. This demonstrates that the photoresistance of the $CsMg_xPb_{1-x}I_3$ NCs was comparable before and after encapsulation. Similarly, the moisture durability of $CsMg_xPb_{1-x}I_3$ QDs before and after encapsulation was examined using a 10% volume ratio of DI water. The

fluorescence intensity of CsM $g_x Pb_{1-x}I_3$ Q Ds deteriorated immediately, with only 12% of the initial intensity remaining 40 min, whereas the PQD@Gd nanoagent retained 41% of its initial intensity even after 24 h. Furthermore, as illustrated in the inset picture of Figure S6g, CsM $g_x Pb_{1-x}I_3$ NCs deteriorated in the presence of 10% DI water, while the PQD@Gd nanoagent shows a luminous image, demonstrating that encapsulated NCs retain their luminescent properties in the aqueous solvent. The results show that the aqua-resistant properties of CsM $g_x Pb_{1-x}I_3$ QDs enhanced after encapsulation.

3.4. Photodynamic Therapy of the PQD@Gd Nanoagent. The PNCs are ideal candidates to be employed as photosensitizers (PSs) in light-activated therapeutic activities, such as photodynamic therapy, because of their multiple excitonic generation properties. In PDT, the PS absorbs the light and excites to the triplet state; from there, it decomposes molecular oxygen to reactive oxygen species (ROS). 140 Thus, herein, we studied the suitability of the PQD@Gd nanoagent to be employed as a PDT agent by measuring the ROS generation under laser irradiation. The widely used DPBF probe was employed for the detection of ROS. The yellowish fluorescent DPBF degrades to colorless 1,2-dibenzoylbenzene (DBB) upon interaction with ROS species (Figure S7a); thus, emission spectra at 460 nm ($\lambda_{\text{excitation}} = 410$ nm) is helpful to evaluate the ROS generation. As shown 2 Figure S7b, the PL spectra of DPBF remained unchanged after 30 22 laser irradiation. However, the PL intensity of DPBF gradually declined as the irradiation time increased (Figure 4b) in the presence of 100 ppm PQD@Gd nanoagent, indicating that POD@Gd could serve as an elient ROS-generating PS agent. The influence of PQD@Gd nanoagent concentration on ROS production e□ciency was investigated, as shown in Figure S7c. At higher concentrations (200 ppm) of PQD@Gd, fluorescence intensity of DPBF declined rapidly due to the generation of more ROS. In the presence of 200 ppm of PQD@Gd, around 75% fluorescence intensity declined from the initial value within 15 min of irradiation, whereas 100 ppm PQD@Gd caused a 68.5% decline in the same irradiation time (Figure 4c), implying that ROS generation [117] ncentrationdependent. Next, to validate 102 radicals, the PL emission spectra were recorded at around 1270 nm ($\lambda_{\text{excitation}} = 671 \text{ nm}$). The PQD@Gd sample was first saturated with O2 gas for 10 min before being irradiated with a laser at 671 nm, and then, the PL spectrum was recorded at $\lambda_{\text{excitation}} = 671$ nm. A strong and intense peak at 1265 nm with a bandwidth of 20.8 nm was observed (Figure 4d), which confirms the generation of 102 radicals. The ROS produced from the light-activated POD@ Gd nanoagent was further and opposition of the state of t colorimetric assays to validate the presence of *O₂ and *O H radical species, respectively. In the UV -vis spectra of NBT and PQD@Gd mixture (Figure 4e), the intensity of the characteristic peak of reduced NBT at 260 nm gradually increased with increasing irradiation times and also the formation of gray formazan was observed (as shown 126 igure S8a). indicating the formation of *O₂ radicals via a charge transfer mechanism. 56 the UV -vis spectra of the mixture of PQD@Gd and OPD solution (as shown in Figure 4f), the absorbance of the peak at 450 nm, which is the characteristic peak of formed DAP (Figure S8b), increased with irradiation time, indicating that *O H radicals formed via an energy transfer mechanism. These experimental results confirmed that the PQD@Gd nanoagent predominantly produces ${}^{1}\mathrm{O}_{2}$ and ${}^{\bullet}\mathrm{O}_{2}^{-}$ radicals and a small amount of *OH radicals.

The ability of the PQD@Gd nanoagent to generate ROS was further examined qualitatively using ESR spectroscopy. In this regard, TEMP and DMPO were used as probes for detection of ¹O ₂ and [•]O ₂⁻/ [•]O H, respectively. Figure 4g shows that no signal peak was obtained for TEMP only after 10 min of laser irradiation. However, the combination of PQD@Gd with TEMP produced a set of characteristic TEMP-1-oxyl (TEMPO) signal peaks with a ratio of 1:1:1 in the absence of the laser. This phenomenon was significantly pronounced when the PQD@Gd nanoagent was exposed to 10 min laser irradiation, which was attributed to the generation of ¹O₂. Similar results were obtained when DM PO in DM SO was used for the detection *O₂-. No signals were observed for DMPO only with the laser and DMPO with PQD@Gd, as shown in Figure 4h. However, under 671 nm laser irradiation, the ESR spectra of DMPO and PQD@Gd exhibited signal peaks at 1:2:2:1 ratio due to the formation DMPOO adducts, confirming that the as-synthesized nanoagent generated lightinduced ${}^{\bullet}O_{2}^{-}$ radicals. For the detection of hydroxyl (${}^{\bullet}OH$) radicals, DMPO in water was used as the probe. Consistent with the above trends, neither the control experiment (DMPO in water) nor the PQD@Gd nanoagent produced an ESR signal without the laser (Figure 4i). The combination of the PQD@Gd nanoagent and laser irradiation generated a weak ESR peak (with a ratio of 1:2:2:1) due to the formation of DM POO, approving *OH radical generation. According to the results of ESR spectroscopy and colorimetric assay, the most abundant radical species generated by the PQD@Gd nanoagent were ${}^{1}O_{2}$ and ${}^{\bullet}O_{2}^{-}$ with a low proportion of ${}^{\bullet}OH$ radicals. To understand the mechanism of e□cient ROS generation from the PQD@Gd nanoagent, the multiple exciton property of perovskite QDs was reported. In the literature, it was reported that perovskite QDs owned higher light-harvesting property, which could find application in many photocatalysis 250 cesses. Therefore, the absorption of light by PNCs causes formation of an electron—hole pair (e-, h+), as well as the subsequent separation and recombination of electrons and holes is the most possible pathway for the lightactivated ROS generation property. Briefly, PNCs + hv \square e⁻ + h⁺, and then, the photogenerated electrons and holes undergo a self-trapping process to produce e_{TR}^- and h_{TR}^+ species, respectively. After that, the recombination of electron-hole pairs occurred, $e_{T\,R}^{\,-}\,+\,h_{T\,R}^{\,+}\,\,\square\,\,e_{C\,B}^{\,-}\,+\,heat,$ which is more favorable for oxygen-based radical species. The formation of electrons and holes in the system results in catalysis of molecular oxygen and/or water molecules to produce ROS via energy and charge transfer mechanisms, as shown in Figure S8c. O verall, our results are in good agreement with a recent report, approving that the PQD@Gd nanoagent could be used as a PS agent in PDT treatment of various cancer cells.¹/

3.5. Photocatalytic Evaluation of the PQD@Gd Nanoagent. The photocatalytic e□tiency of as-synthesized PQD@Gd nanoagents was demonstrated by deactivation of organic pollutants under visible light illumination. Redox processes mediated by photoinduced electrons (e⁻) and holes (h⁺) on the photocatalyst's surfact2 produce photocatalytic properties. S2,53 In light of this, UV—vis spectroscopy was used to demonstrate the degradation of MO, RhB, and MB dyes under last illumination.

As shown in Figure S9, there was no substantial change in the absorption spectra of dyes in the presence of only laser or only PQD@Gd nanoagent, implying that the PQD@Gd nanoagent is a light-activated catalyst. In the presence of the

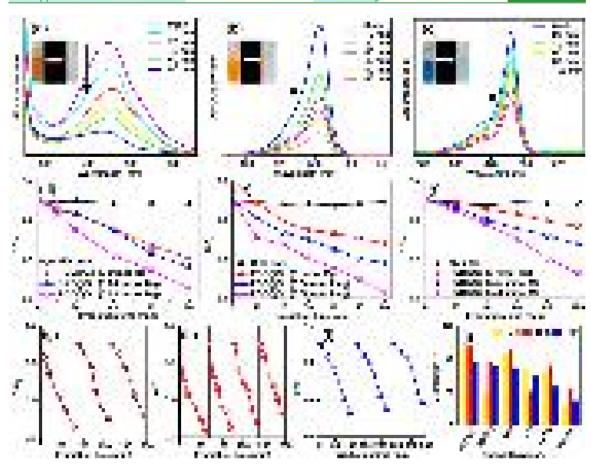


Figure 5. Absorption spectra of light-induced degradation of (a) 1 mM MO, (b) 1 mM RhB, and (c) 1 mM MB using the PQD@Gd nanoagent. Relative concentrations of (d) MO, (e) RhB, and (f) MB organic dyes upon combining with the PQD@Gd photocatalyst at dilarent Mg contents. Reusability performance of the PQD@Gd photocatalyst toward the decomposition of (g) MO, (h) RhB, and (i) MB organic dyes. (j) Photodegradation of Mo, RhB, and MB organic dyes by the PQD@Gd photocatalyst examined with dilarent radical scavengers. The control experiment was performed in the absence of a radical scavenger. The experiment was performed using 671 nm light illumination (2.0 W/cm²).

PQD@Gd nanoagent, the absorbance of characteristic peaks of MO, RhB, and MB at 460, 560, and 660 nm, respectively, gradually decressed as the irradiation time increased (Figure 5a–c). Also, the color of the solution changed to colorless for MO and RhB dye solutions and light blue for MB dye solution (inset in Figure 5a–c), indicating the complete degradation of MO and RhB due to the photocatalytic activity of the nanoagents. As a result, the amount of remaining dyes in the solution became lesser with increasing concentration of the nanoagent or irradiation time (Figure S10a–c), intimating that the catalytic activity depends on the laser irradiation time and PQD@Gd nanoagent concentration.

Next, the elact of Mg⁺² ion doping on photocatalytic activity was studied by employing the PQDs that were prepared with dilarent amounts of Mg precursors. As shown in Figure 5d−f, at any irradiation time point, the dye degradation rate increased with increasing Mg content in PQDs. After 80 min irradiation, 58.7, 67.9, and 88.8% of MO was degraded for PQDs with 0, 0.04, and 0.08 mmol Mg, respectively. Similarly, 44.1, 64, and 92.7% photodegradation of the RhB dye was observed for PQDs with 0, 0.04, and 0.08

mmol Mg, respectively, after 60 min laser irradiation. For MB, 26.4, 45.1, and 73.9% degradation occurred after 100 min of continuous laser irradiation. These findings validated that doping Mg⁺² ions into CsPbI₃ QDs significantly enhanced the catalytic activities of resulting $Q_{25} g_x P b_{1-x} I_3 Q D s$. Pradeev Raj et al. and Yousefi et al. recently reported that the photocatalytic activity of M g-integrated ZnO nanoparticles was increased, and the dye-degrading activity was dependent on the Mg dopant's concentration. 54,55 The minor shift in the absorption spectra of the $CsMg_xPb_{1-x}I_3$ QDs in Figure 3a could be due to the slightly grown crystal size, which is beneficial for the improved light-harvesting e□ciency caused by enhanced light scattering properties. Another argument is that after Mg²⁺ doping, the band gap di drence decreased (Figure 3f), causing electron hole recombination to accelerate radical generation, thereby triggering the photodegradation activity. The addition of M q²⁴ ions to the QD structure boosted the light absorption capacity, which could promote the formation of more radical species and increase the e□ciency of organic dye degradation.

The reaction mechanism for the pollutant so egradation is shown in Figure S11. The combination of a light source and

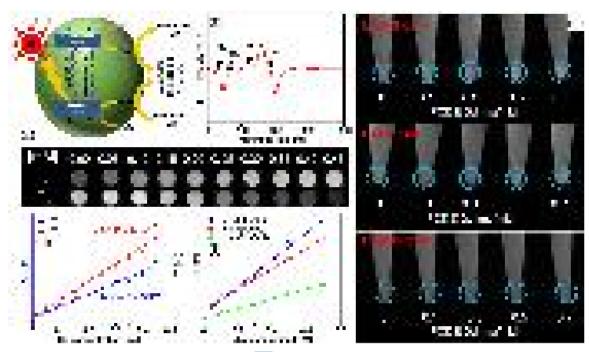


Figure 6. (a) Schematic illustration of a proposed mechanism 144 he photocatalytic activity of the PQD@Gd nanoagent. (b) ESR spectra of the PQD@Gd nanoagent m 59 red at 10 K. (c) Representative T_1 - and T_2 -weighted phantom pictures of the PQD@Gd sample at dicarent Gd 3 + concentrations. (d) Plot of water—proton longitudinal (1/ T_1) and transverse (1/ T_2) relaxation rates versus dicarent Gd 3 + concentrations based on panel (c). (e) T_1 - and T_2 -weighted MR pictures of B16F1, H eL.a, and H epG2 cancer cells cultured with various amounts of the PQD@Gd nanoagent overnight. (f) Plot of inverse relaxation rate (1/ T_1) versus dicarent Gd 3 + contents depending on panel (e). The slope of fitted lines was used to compute the relaxivity value.

the PQD@Gd nanoagent could result in the generation of radical species, which would activate the breakdown of the M O structure into two benzyl rings (Figure S11a). For the RhB dye, the radical species would possibly reduce it to rhodamine, as shown in Figure S11b. The RhB dye can decompose in a number of ways, including through an indirect reaction with powerful oxidants that were generated photocatalytically from water on the surface of the PQD@Gd nanoagent or explicitly through the decay of the RhB dye adsorbed to the surface of PQD@Gd. Because it contains four N-ethyl groups on both sides of the xanthene moiety, the RhB dye could bind to the surface of PQD@Gd via electrostatic coupling. In those ase of MB, the oxidative breakdown of MB to CO₂ or the reductive degradation of M B to the colorless Leucomethylene blue could be a plausible mechanism for MB degradation (Figure S11c). Furthermore, the durability of the PQD@Gd nanoagent (0.08 mmol Mg content) photocatalyst toward MO, RhB, and MB was also explored, as shown in Figure 5g-i. After three repeated cycles, the percentage of the dye degraded was slightly decreased to 75% from 89.01% for MO, 74% from 92.2% for RhB, and 63% from 74% for MB, proving that the PQD@Gd nanoagent had superior photocatalytic performance and comparable recyclability. To identify the type of radicals responsible for the degradation mechanism, we used a radical trapper such as isopropanol for OH, EDTA for e-, β-carotene for ${}^{1}\text{O}_{2}$, AgN O ${}_{3}$ for h⁺, and ascorbic acid for ${}^{\bullet}\text{O}_{2}^{-}$. Figure 5j shows that the degrading capacity of the PQD@Gd nanoagent (0.08 mmol Mg content) against MO and MB dyes decreased from 89.12% (without the trapper) to 22.12 and 29.8% for the

β-carotene reagent, respectively, indicating that the decomposition of M O and M B dyes was catalyzed by singlet oxygen ($^{1}O_{2}$). Using AgN O $_{3}$ and EDT A as radical trappers, the degradation ratio M O and M B dyes reduced by 45%, revealing that photogenerated electrons and holes accelerate the breakdown of the pollutants. The photodegrading capacity of PQ D @Gd for RhB reduced to 41% when ascorbic acid and β-carotene were used as radical trappers, confirming that $^{\bullet}O_{2}^{-}$ and $^{1}O_{2}$ radicals were preferable to inactivate RhB structures.

The underlying mechanism for the degradation of organic dyes can be explained as follows: the photocatalysis reaction could be triggered when a high-energy photon with su □cient energy (corresponding to the E_{bg} of the photocatalyst) was absorbed, and charge separation could occur when an electron (e) was driven from VB to CB, as illustrated in Figure 6a. In the VB and CB, a hole (h+) and an electron (e-), which are potential oxidizing and reducing agents, respectively, could be produced via the following reaction: photocatalyst + hv \square e⁻ + h+. The h+ could directly oxidize the pollutant or water to produce *OH radicals, which subsequently oxidized the pollutants and the resulting degraded products. The electron in the CB could reduce the molecular oxygen adsorbed on the photocatalyst surface, forming reactive superoxide radicals (*O₂-) that inactivated the organic molecules. The ESR data strongly supported this explanation, i.e., ${}^{1}O_{2}$, ${}^{\bullet}O_{2}^{-}$, and ${}^{\bullet}OH$ radicals are responsible for photocatalytic degradation of MO, RhB, and MB, respectively. In ESR spectroscopy, generation of ¹O₂ radicals and [•]O₂ and [•]O H radicals was detected using TEM P and DEM PO, respectively (Figure 4d-f). These results

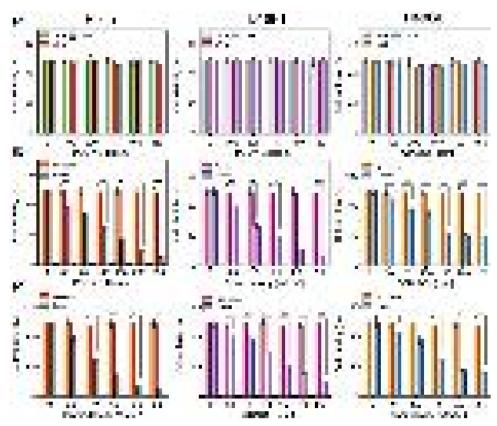


Figure 7. (a) In vitro biocompatibility of the PQD@ 32 nanoagent incubated at dilarent concentrations with HeLa, B16F1, and HepG2 cancer cells for 24, 48, and 72 h. (b) In vitro PDT treatment of HeLa, 616F1, and HepG2 cancer cells incubated with various amounts of the PQD@Gd nanoagent overnight and treated with a 671 nm laser power (2.0 W/cm²) for 5 min. (c) In vitro DT treatment of HeLa, B16F1, and HepG2 cancer cells incubated with 200 ppm of PQD@Gd nanoagent overnight and irradiated with a 671 nm laser source at various power densities (0.5, 1.0, 1.5, 2.0, and 2.5 W/cm²) for 5 min.

are consistent with those in Figure 5j, confirming that the assynthesized PQD@Gd nanoagents could act as photocatalyst agents in various wastewater treatment techniques or bacterial inactivation protocols.

3.6. MRI Measurements of the PQD@Gd Nanoagent. The presence of paramagnetic metal ions Gd³⁺ could endow the PQD@Gd nanoagent with MRI properties. According to the literature, the Gd3+ ion with seven unpaired electrons, outstanding magnosic properties, and a high electron-spin relaxation time could be used as a T₁-dominating contrast agent in various cancer treatments.^{56,57} Thus, ESR spectroscopy was employed to confirm the presence of unpaired electrons in Gd3+ of PQD@Gd. As displayed in Figure 6b, a single prominent peak and Zeeman interaction with $g_1 = 1.99$, $q_2 = 2.48$, $q_3 = 2.8$, and $q_4 = 3.00$ was observed, indicating that PQD@Gd nanoagents could be promising candidates for MR imaging. These findings are in accordance with earlier findings Gd-based MRI agents. 56,58 The Gd3+ ion can promote longitudinal (spin-lattice relaxation 40r transverse (spin —spin relaxation) relaxation, allowing it to be used as a T_1 or T_2 contrast agent. Thus, the relaxivities of PQD@Gd nanoagents in aqueous media 12 e measured to assess the feasibility of using them as dual 11 and 12 weighted contrast agents. Figure 6c depicts the T₁ and T₂-weighted MR images of PQD@Gd

nanoagents at various concentrations based on Gd contents, ating a concentration-dependent signal enhancement for both T_1 - and T_2 -weighted images. Briefly, the T_1 phantom weighted image displayed a more brighter contrast with increasing Gd3+ concentration, whereas under the same conditions, T₂ studies displayed arker spot images, implying that the PQD@Gd nanoagent could be used a dual-modal contrast agent in cancer diagnosis. Generally, the e□ciency of an MRI contrast agent is assessed by the following equation: $R_{1,2} = \frac{1}{5} + r_{1,2} \times C$, where $R_{1,2}$ is the measured relaxation rate (s^{-1}) of MRI contest agents, T is the observed relaxation rate (s^{-1}) before the addition of contrast agents, $r_{1,2}$ is the relaxivity coe \square cient (mM $^{-1}$ s $^{-1}$), and C is the concentration (mM) of the MRI contrast agents. Positive MR contrast agents with high r_1 ratios of 1–2 is optimal, while negative M R contrast agents with high r_2 values and r_2/r_1 ratios larger than 10 are preferred. Specifically, T₁ contrast agents help to strengthen the longitudinal regression value, resulting in a positive (bright) contrast in the selected spot, whereas T₂ contrast agents (darker contrast) boost the signal intensity in T₂-weighted pictures.^{59,60} The relaxivity coe \square cients (r_1 and r_2) were computed from T1 and T2 mapping images obtained using an inversion recovery sequence to evaluate the MR

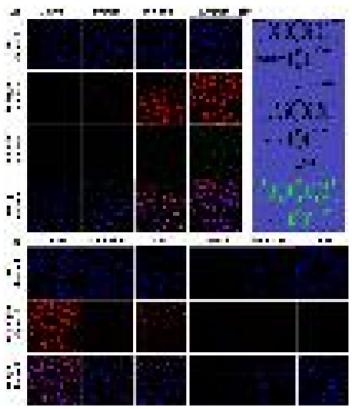


Figure 8. (a) Confocal images of HeLa cells incubated with various concentrations of the PQD@Gd nanoagent for 6 h at 37 °C and in a humidified atmosphere. The catalycells were stained with DAPI (blue dye) to track the nucleus. The green channel indicates the DCFH-DA oxidation to DCF• species. The species. The species were treated with the DCFH-DA reagent and illuminated under 671 nm laser power (2.0 W/cm²). (b) Hydrolysis and ROS-induced oxidation of DCFH-DA molecules to form DCF green fluorescent material. (c) Cellular imaging of HeLa cells incubated with 30 µL of dicerent in 56 tors (nystatin, genistein, amiloride, and M βCD) for 30 min and 20 56 m of PQD@Gd nanoagent 3 h. For the temperature-dependent study, HeLa cells were incubated with 200 ppm of PQD@Gd nate 1000 nt at 4 °C. The control experiment was performed without adding the inhibitor. The images were captured at excitation wavelengths of 405, 488, and 532 nm for blue, green, and red channels, respectively.

contrast elect, as shown in $_{58}$ ure 6d. The $_{1}$ and $_{2}$ values of the PQD@Gd nanoagent were estimated to be 27.12 and 37.45 mM $^{-1}$ s $^{-1}$, respectively. The ratio of $_{1}$ and $_{2}$ was calculated to evaluate the potential of the nanoagent in terms of $_{1}$ and $_{2}$ contrast elects. The $_{2}/r_{1}$ ratio was found to be 1.38, confirming that the PQD@Gd nanoagent could be a promising candidate in $_{1}$ -guided imaging application, but the higher relaxivity value ($_{2}$ = 37.45 mM $^{-1}$ s $^{-1}$) made them also preferable as $_{2}$ contrast agents. The results are in good agreement with a recent report. $_{2}$

After confirming the T_1 -dominating contrasting effect of the PQD@Gd nanoagent, in vitro MRI studies were performed using cancer cells to verify the ability of the PQD@Gd nanoagent as a dual T_1/T_2 MR agent in cancer diagnosis. In this regard, cancer cells were treated with a concentration gradient of the PQD@Gd contrast agent overnight, and subsequally, MRI measurements were performed. Figure 6e shows the longitudinal (T_1) - and transverse (T_2) -weighted images of the different cancer cells such as B16735 HeLa, and HepG2. The nontreated cells served as a control group. Compared with the control group, the B16F1 and HeLa cancer cells treated with the PQD@Gd nanoagent showed obvious T_1

MRI signals and the T₁ value increased with increasing PQD@ Gd concentrations, confirming the T₁-dominating elect, whereas HepG2 cancer cells treated with the PQD@Gd nanoagent did not show any significant change with increasing material concentration. These findings indicated that the PQD@Gd nanoagent o \square ered a superior positive T₁ contrast enhancement for in vitro MR imaging. This was further confirmed by plotting the relaxivity properties against di dirent concentrations of the PQD@Gd nanoagent (Figure 6f), proving that the relaxation value (r₁) increased with increasing D@Gd concentration for B16F1 and HeLa cancer cells. When the T_1 - and T_2 contrast signals were comparable, the dual contrast e dct probes may produce ideal contrasts on both T₁- and T₂-weighted images due to their synergistic combination of the two relaxation elects. The accumulation of the PQD@Gd nanoagent in the cancer cells via the endocytosis route led to the considerable change in the contrasting intensity.

3.7. In Vitro Demonstration of the PQD@Gd Na22-agent. In vitro cytotoxicity of the PQD@Gd nanoagent was assessed using the WST-1 assay. First, the cytocompatibility of the PQD@Gd nanoagent to HeLa, HepG2, and B16F1 cancer

cells was examined. As shown in Figure 7a, at all given concentrations the viability of cells treated with the PQD@Gd nanoagent was comparable to that of untreated cells, suggesting that the PQD@Gd nanoagent is cytocompatible. This could be because of the encapsulation into the PF127 micelle structure, which could protect the cell by preventing the release of harmful Pb2+ ions. Next, the anticancer PDT e Lect of the PQD@Gd nanoagent was invergigated under light illumination. To this end, the cancer cells were treated overnight with various concentrations of the nanoagent (0, 50, 75, 100, 150, 200, and 300) and irradiated using a 671 nm laser (2.0 W/cm²). Without light irradiation, the nanoagent alone did not induce cell toxicity even at a concentration as highers 250 ppm. On the other hand, under light irradiation, the cell viability decreased with increasing concentration of the nanoagent (Figure 7b) because the endocytosed PQD@Gd nanoagent generated ROS upon laser irradiation. The antica e Lacy of most substances is commonly expressed using the IC 50 value, which indicates the amount of a drug required to inhibit a biological process by 50%. Thus, the cell viability relative to the PQD@Gd concentration was fitted with a logistic function, and the IC_{50} values of PQD@Gd for HeLa, B16F1, and HepG2 were calculated as 100, 123, and 117 ppm, respectively. According to Figure 7b, PQD@Gd induced higher cell death in B16F1 and HeLa cells than that in HepG2 cells. At 250 ppm of PQD@Gd and 5 min of laser irradiation, the cell death was around 85% for B16F1 and HeLa cells, whereas the cell death for HepG 2 was 60%. The internalization of the PQD@Gd nanoagent is predicted to be stronger in B16F1 and HeLa cells, resulting in a stronger PDT impact. Moreover, as depicted Figure 7c, the experiments were carried out using various power densities (0.5, 1.0, 1.5, 2.0, and 2.5 W/cm²) and a constant PQD@Gd nanoagent concentration (200 ppm). The cell viability decreased as the power density increased due to the generation of more ROS at higher power densities. After being treated with the 200 ppm PQD@ Gd nanoagent and a 2.5 W/cm² laser power, the live cell numbers for HeLa and B16F1 cancer cells reduced to 8.9 and 9.1%, respectively, whereas 31.5% of HepG2 cells remained alive in the same circumstances, implying that the PDT eciency was concentration- and laser-dependent. The findings were in accordance with the qualitative analysis shown in Figure 4c, confirming that the as-synthesized PQD@ Gd nanoagent could be a potential anticancer agent.

3.8. In Vitro Fluorescence Imaging, The ionic instability and degradation of PQDs in water are the main hurdles in their applicability in cellular imaging. However, as the stability and water dispersibility of PQDs were improved by our strategy, the resulting PQD@Gd nanoagents might be used as luminescent probes for biological imaging. To investigate the applicability of the PQD@Gd nanoagent as fluorescent biomarkers in practical nanomedicine, cellular internalization of the PQD@Gd nanoagent was demonstrated using HeLa, B16F1, and HepG2 cancer cells. The cancer cells were supplied with various amounts of the PQD@Gd nanoagent overnight in a humidified atmosphere and then stained with DAPI to track the nucleus. The confocal images of HeLa cells treated with PQD@Gd shown in Figure 8a exhibited bright red fluorescence, confirming that PQD@Gd nanoagents were well absorbed into the cells. Moreover, the fluorescence intensity increased with increasing concentration of nanoagents, implying that the cellular uptake may be controlled by a concentration gradient. Similar results were obtained for

B16F1 and HepG2 cancer cells; however, the intensity was low in HepG2 compared to that in B16F1 cells under similar conditions (Figure S12), suggesting that the nanoagents were preferably uptaken by B16F1 and HeLa cells. Further, to determine the position of the nanoagent in the organelles of the cancer cells, confocal scans with Z-axis stacking were performers nder the same conditions. The three-dimensional confocal images of HeLa, B16, and HepG2 cells incubated with the PQD@Gd nanoagent obtained under the vertical scan mode along the Z-axis are shown in Figures S13-S15. The results indicated that PQ D@Gd was well distributed in the cell structure, as higher Q D signals were observed in the cytoplasm than on the cell surface. Therefore, the Z-stack images (from top to bottom) showed elective integration of PQD@Gd in HeLa, B16, and HepG2 cells and highlighted the di ☐ rences in FL signals between cell lines. The fluorescence intensities were quantified and compared using ImageJ analysis. Figure S16a shows that B16F1 and HeLa cells had higher QD signals, proving that PQD@Gd is well distributed in the cell, whereas HepG2 cancer cells showed lower signals under similar conditions due to the lower di□sion rate of the PQD@Gd nanoagent. The CLSM and MR imaging analyses validated that the PQD@Gd nanoagent is suitable for both MRI and bioimaging, enabling them as potential candidates for dualmode cancer cell diagnosis.

Intracellular ROS Detection. After the cellular uptake of PQD@Gd was validated, the ability of PQD@Gd to regulate the intracellular oxidative stress upon irradiation with a laser was examined use a DCFH-DA probe. Under intracellular oxidative stress, the non-fluorescent DCFH-DA converts to green fluorescent 2',7'-dichlorofluorescein (DCF), as shown in Figure 8b.⁶¹ The nucleus was stained with DAPI. According to the CLSM photographs shown in Figure 8a, the HeLa cells treated with only laser without the PQD@Gd nanoagent exhibited a weak green fluorescence (FL) signal. However, the cells neated with the laser and PQD@Gd nanoagent exhibited higher green fluorescence signals than the control cells, suggesting the generation of intracellular ROS due to the PDT elect of PQD@Gd. The green fluorescence signal became brighter with increasing PQD@Gd nanoagent concentration, validating that the PDT response was laserinduced and concentration-dependent. The distribution of PQD@Gd in the nucleus seems to be confirmed by the overlap of blue, red, and green signals in the provided overlay images. Moreover, in Figure 8a, the degree of ROS formation (green fluorescence) correlates with the cell toxicity results, suggesting that PQ D @Gd e⊡ectively targets HeLa cells. This quantitative result indicates that the POD@Gd nanoagent is preferentially uptaken by cancer cells, suggesting that it could be used as a multifunctional platform FL and MRI probe as well as a PDT agent for cancer diagnosis and therapy.

The mechanism of intracellular uptake was further in (123) ated with dicarent inhibitors, as mentioned in Section 2. In this study, HeLa cancer cells were cultured with dicarent inhibitors such as amiloride, nystatin, genistein, and M βCD for 30 min and then with 200 ppm of PQD@Gd nanoagent for 3 h. The CLSM images shown in Figure 8c demonstrated that the genistein inhibitors significantly suppressed the nanoagent uptake, indicating that the caveolae-mediated endocytosis pathway played a major role. Notably, the inhibitors amiloride and nystatin facilitated less nanoagent uptake than the M βCD inhibitors, which had approximately the same mean fluorescence intensity (Figure S16b) as the control experiment

(without the inhibitor). Remarkably, none of the inhibitors completely stopped the uptake process, suggesting that the nanoagents are endocytosed via multiple pathways simultaneously. In addition, the cells maintained at a lower temperature showed extremely limited uptake, suggesting that the endocytosis process is energy-dependent rather than di⊡sion-driven across the cell membrane.

4. CONCLUSIONS

In summary, we have prepared Mg⁺² ion-doped CsPbI₃ QDs $(CsMg_xPb_{1-x}I_3 QDs)$ with improved optical properties and controllable morphology via the microwave tegatique. The $CsM g_x Pb_{1-x}I_3 NCs$ had higher PLQY ($\square 89\%$) and improved phase stability than undoped CsPbI3 NCs because of the improved charge recombination rate and increased Goldschmidt tolerance factor caused by Mg doping. Subsequently, the as-synthesized CsM g_xPb_{1-x}I₃ NCs are further encapsulated into the micelle-like structure of PF127-Gd through hydrophobic—hydrophobic interactions to improve their bioapplicability. The resulting PQD@Gd nanoagen have demonstrated good water dispersibility due to the presence of hydrophilic PF127 on the surface. The capabilities of PQD@ Gd to generate ROS and degrade organic dyes photocatalytically were investigated under light irradiation. The T₁ and T₂ contrast measurement studies suggested that the PQD@Gd nanoagent (15)d be a promising theranostic agent in cancer diagnosis. I81 vitro results showed that the PQD@Gd nanoagents are taken up by the di⊡erent cancer cells via caveolae-mediated endocytosis pathways and electively damaged the cancer cells upon laser irradiation through ROS-mediated therapy. Confocal imaging and MRI proved that the nanoagent produced excellent fluorescent imaging and contrast ability, respectively. Taking all this together, the redemitting PQD@Gd multifunctional nanoagent could be employed as a fluor arent and M RI-guided PDT agent in future nanomedicine. The photocatalytic activity of the PQD @ Gd nanoagent sheds light on its use in dilarent antibacterial inactivation experiments.

29 ASSOCIATED CONTENT

* Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsami.1c19644.

M icrowave synthesis of $CsPbI_3$ and $CsM\ g_xPb_{1-x}I_3\ QDs$; optical band gap energies of $CsPbI_3$ and $CsM\ g_xPb_{1-x}X_3\ QDs$; PL emission spectra of $CsPbI_3$ and $CsM\ g_xPb_{1-x}I_3\ QDs$ in dicerent conditions; DLS measurements of $CsPbI_3$ and $CsM\ g_xPb_{1-x}I_3\ QDs$; fluorescence lifetimes of $CsPbI_3$ and $CsM\ g_xPb_{1-x}I_3\ QDs$; fluorescence lifetimes of $CsPbI_3$ and $CsM\ g_xPb_{1-x}X_3\ QDs$; TEM images, DLS analyses, FTIR spectra, and ζ -potentials of $CsM\ g_xPb_{22}I_3\ QDs$ and PQD@Gd nanoagents; photocatalytic degradation of M O, RhB, and M B organic dyes in the presence of the PQD@Gd nanoagent; class in the presence of the PQD@Gd nanoagent; and comparison of the fluorescence intensity of cellular imaging versus PQD@Gd concentration (PDF)

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Notes

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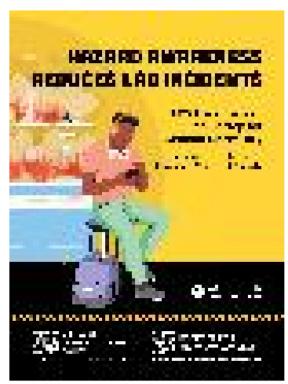
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