

Synthesis and Photoluminescence Quantum Yield Studies of CsPbI₃ Perovskite Nanocrystals in Liquid Paraffin as a Novel and Low-Cost Reaction Medium

Abbasali Ghorbani Kouhbanani

Shahid Bahonar University of Kerman

Vahid Saheb

vahidsaheb@uk.ac.ir

Shahid Bahonar University of Kerman

S. Mohhamad Ali Hosseini

Shahid Bahonar University of Kerman

Research Article

Keywords: CsPbI₃ perovskite nanocrystals, hot-injection synthesis, liquid paraffin medium, photoluminescence quantum yield

Posted Date: October 1st, 2026

DOI: <https://doi.org/10.21203/rs.3.rs-10715805/v1>

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Additional Declarations: No competing interests reported.

Abstract

Cesium lead iodide (CsPbI_3) perovskite nanocrystals (PNCs) have attracted significant attention as promising candidates for optoelectronic applications owing to their tunable bandgap and high photoluminescence efficiency. In this study, CsPbI_3 PNCs were synthesized via a modified hot-injection method using liquid paraffin (PaL) as the reaction medium, and their structural, morphological, and optical properties were systematically compared with those synthesized in the conventional octadecene (ODE) medium. The morphology, size distribution, and crystal structure of the PaL-synthesized PNCs were investigated using transmission electron microscopy (TEM) and X-ray diffraction (XRD). Statistical analysis of TEM micrographs revealed a mean particle size of 10.97 ± 2.16 nm ($n = 270$) with a polydispersity index (PDI) of approximately 19.7%, indicating a narrow and uniform size distribution. To evaluate the photoluminescence (PL) performance of the two media, a rigorous Integrated PL Intensity versus Absorbance approach was employed. The results showed that CsPbI_3 PNCs synthesized in PaL exhibited a regression slope approximately 60% higher than that of PNCs synthesized in ODE, indicating substantially superior photoluminescent performance. This enhancement is attributed to improved crystallinity and a reduced density of non-radiative defect sites, along with more favorable morphology and size distribution, in the PaL-synthesized PNCs. These findings establish liquid paraffin as an effective and reproducible synthesis medium for producing size-controlled CsPbI_3 PNCs with enhanced optical quality, offering valuable implications for their use in next-generation optoelectronic and photovoltaic devices.

1. Introduction

The global drive toward sustainable energy has accelerated innovation in conversion technologies, where low-cost, high-efficiency materials are essential for addressing climate challenges and energy security^[1–4]. While silicon photovoltaics have dominated for decades, their complex, high-temperature manufacturing limits scalability and cost reduction^[3, 5–8]. This has directed attention toward solution-processable materials with superior optoelectronic properties that enable low-temperature fabrication in devices such as LEDs, lasers, and photodetectors^[9–13].

Lead halide perovskite nanocrystals have emerged as outstanding candidates for next-generation energy and optoelectronic applications, combining strong quantum confinement, tunable band gaps, and high photoluminescence quantum yields (PLQYs)^[7, 10, 14–24]. Yet, maintaining phase stability—especially for CsPbI_3 nanocrystals—is a persistent challenge that depends heavily on reaction conditions, solvent environment, and precursor composition^[24, 25].

Since the reaction medium governs nucleation, morphology, and phase evolution, its choice is critical^[25, 26]. Nonpolar, high-boiling-point solvents often promote the formation of the desirable cubic phase with improved optical performance^[25]. This research work explores the feasibility of PaL as an inert reaction medium for CsPbI_3 nanocrystals—distinct from surface ligands, which solely serve passivation roles.

PaL offers both thermal stability and a significantly lower cost than conventional solvents like ODE, suggesting potential for scale-up if laboratory results are favorable [27–29].

Following synthesis in the paraffin medium, the nanocrystals are purified and dispersed in n-hexane, allowing precise separation between reaction and surface treatment steps. This study systematically evaluates the influence of paraffin on the phase, morphology, and photophysical behavior of CsPbI₃ and compares the results with conventional solvent systems [25, 30–32]. The findings highlight a low-cost, high-performance approach for improving the stability and optical quality of perovskite nanocrystals.

2. Materials and Methods

Materials All chemicals were purchased from commercial suppliers and used without further purification unless otherwise stated. Cesium carbonate (Cs₂CO₃, 99.9% purity), lead(II) iodide (PbI₂, 99.999% purity), oleic acid (OA, 90% purity), octadecene (ODE, 90% purity), oleylamine (OLA, 80–90% purity), methyl acetate, and n-hexane were obtained from Sigma-Aldrich, Acros, or ABCR. Liquid paraffin (PAL, CAS number: 8012-95-1 from Neutron company of Iran) was used as the high-boiling point solvent for the modified synthesis.

Preparation of Cesium Oleate (Cs-oleate) Precursor

The cesium oleate (Cs-oleate) precursor solution was synthesized by reacting cesium carbonate with OA in ODE. Specifically, 0.101 g of Cs₂CO₃, 0.3 mL of OA, and 5 mL of ODE were loaded into a three-neck flask equipped with a magnetic stir bar, under a nitrogen atmosphere. The mixture was heated to 120°C for 1 hour, followed by heating to 150°C until a clear, homogeneous solution was obtained, indicating the complete formation of Cs-oleate. The solution was then cooled and stored under nitrogen. For the synthesis, the Cs-oleate solution was preheated to 100°C prior to injection.

Synthesis of CsPbI₃ Nanocrystals: Two distinct synthetic routes were employed to prepare CsPbI₃ nanocrystals (NCs): a standard method using ODE and a modified method using PaL as the solvent which act as reaction medium.

Synthesis in ODE

CsPbI₃ NCs were synthesized via a hot-injection method [33]. A mixture of 0.087 g of PbI₂ and 5 mL of ODE was loaded into a flask and degassed at 120°C under vacuum for 1 hour to remove any trace of moisture. Subsequently, the temperature was raised to 160°C under a nitrogen atmosphere, and 0.5 mL of OLA and 0.5 mL of OA were injected. After the PbI₂ fully dissolved, the temperature was maintained at 160°C. A rapid injection of 0.4 mL of the preheated Cs-oleate solution (prepared as described in the Cs-oleate precursor preparation section) was then performed. The reaction was quenched after exactly 5 seconds by plunging the flask into an ice-water bath to control the size of the resulting CsPbI₃ NCs.

Synthesis in PaL: A modified procedure using PaL as the solvent was developed to potentially improve thermal stability or alter morphology. The precursor mixture consisted of 0.087 g of PbI_2 and 5 mL of PaL. This mixture was dried under vacuum at 120°C for 1 hour. Following the drying step, the temperature was raised to 140°C under nitrogen, and 0.5 mL of OLA and 0.5 mL of OA were added. After the PbI_2 dissolved, 0.4 mL of the preheated Cs-oleate solution was rapidly injected. The reaction was quenched after 5 seconds by immersing the flask in an ice-water bath. The reaction temperature was optimized for each solvent based on their distinct physicochemical properties. PaL, having significantly higher viscosity and slightly higher polarity compared to ODE, slows down solute diffusion and promotes earlier supersaturation. As a result, nucleation can be initiated at lower temperatures ($\approx 140^\circ\text{C}$). In contrast, in ODE the faster mass transport and stronger stabilization of Pb–oleate complexes delay precursor activation, requiring a higher thermal input ($\approx 160^\circ\text{C}$) to reach the nucleation threshold. Preliminary experiments confirmed this behavior: nanocrystal formation was observed at 140°C in PaL, whereas identical precursor compositions did not yield nanoparticles in ODE until the temperature was increased to 160°C. These findings are consistent with reports that higher-viscosity, slightly more polar hydrocarbon media facilitate low-temperature nucleation by modifying precursor solvation and controlling the reaction kinetics ^[34–36].

Purification of Nanocrystals

Following synthesis, the CsPbI_3 NCs were purified using a precipitation and washing procedure. For samples synthesized in ODE, the crude reaction mixture was precipitated by adding an excess of methyl acetate (approximately 3 volumes of methyl acetate to 1 volume of ODE)^[29, 37, 38]. The precipitate was collected by centrifugation at 7500 rpm for 5 minutes. The solid NCs were then redispersed in n-hexane, and the precipitation/centrifugation cycle was repeated twice to ensure thorough removal of residual reactants and ligands.

For samples synthesized in PaL, the high viscosity of the solvent necessitated an initial dilution step. The reaction mixture was diluted with an equal volume of n-hexane before proceeding with the methyl acetate precipitation. Subsequent centrifugation, redispersion in n-hexane, and washing steps were identical to those described for the ODE-based samples. The final purified nanocrystals were stored as a dispersion in n-hexane.

Comparative Evaluation of Photoluminescence Quantum Yield Using Integrated PL Intensity vs. Absorbance Method

To rigorously evaluate and compare the photoluminescence (PL) properties of CsPbI_3 PNCs synthesized in two distinct media—PaL and ODE—a Integrated PL Intensity vs. absorbance method was employed. This methodology ensures a fair comparison of PL intensities by mitigating artifacts arising from minor variations in concentration and light absorption ^[39–42]. For each synthesis medium, five serially diluted solutions were prepared, ensuring that the absorbance at the excitation wavelength ($\lambda = 450 \text{ nm}$) remained below 0.25. This critical step prevents the onset of phenomena such as the Inner Filter Effect

(IFE) and self-absorption of emitted light, thereby guaranteeing a linear correlation between PL intensity and NC concentration. Subsequently, the PL emission spectrum for each of the five diluted solutions was recorded. Concurrently, the absorbance of these solutions at 450 nm was measured using a UV-Vis spectrophotometer.

The integrated area under each PL emission curve, representing the total emitted energy, was plotted against the corresponding absorbance value at 450 nm. A zero-intercept linear regression was performed on these five data points for each sample. This procedure yielded two distinct regression lines, each characterized by a unique slope. The slope of these lines serves as a robust metric for the effective PL intensity per unit absorbance, effectively representing the relative quantum yield (Rel. QY) of the synthesized CsPbI₃ NCs.

3. Results and Discussion

3.1. Crystal Structure and Phase Identification by X-ray Diffraction

The crystal structure and phase purity of the CsPbI₃ nanocrystals (NCs) synthesized via hot-injection in liquid paraffin (PaL) were investigated by powder X-ray diffraction (PXRD). For sample preparation, a concentrated NC dispersion in *n*-hexane was deposited by drop-casting onto a clean glass substrate to form a thick NC film, which was subsequently air-dried prior to measurement. The diffraction pattern recorded over the 2θ range of 5°–90° is presented in Fig. 1.

The pattern exhibits a dominant, sharp reflection at $2\theta \approx 28.94^\circ$, accompanied by a series of weaker peaks at lower angles ($2\theta = 10^\circ\text{--}27^\circ$) and a broad, attenuated signal at higher angles ($2\theta > 35^\circ$). The overall diffraction profile is in good agreement with the cubic perovskite structure (space group *Pm3m*) of the photoactive black α -phase CsPbI₃ [43, 44], confirming that the hot-injection synthesis in liquid paraffin selectively yields the desired optically active phase rather than the thermodynamically stable but photoinactive orthorhombic δ -phase (yellow phase, space group *Pnma*) [45]. The stabilization of the metastable α -phase at room temperature is attributed to surface passivation by the organic ligand shell and the nanoconfinement effect — two well-established strategies for kinetically trapping the black perovskite phase.

The dominant reflection at $2\theta \approx 28.94^\circ$ is assigned to the (200) crystallographic plane of cubic α -CsPbI₃, which consistently appears as the strongest peak in diffractograms of this phase and serves as its primary structural fingerprint [46]. The weaker reflections in the 10°–27° angular range correspond to the (100), (110), and (111) low-index planes of the cubic perovskite unit cell, consistent with the systematic absences and relative intensities expected for the *Pm3m* space group. The absence of diffraction peaks attributable to PbI₂ ($2\theta \approx 12.7^\circ$) or CsI ($2\theta \approx 27.6^\circ$) confirms complete precursor conversion and high phase purity within the detection limit of laboratory PXRD [44].

A broad, diffuse scattering hump is evident underlying the Bragg reflections, most prominently between $2\theta = 15^\circ$ and 35° . Given the drop-cast sample geometry — wherein a thick NC film is deposited directly onto a glass substrate — this broad feature arises primarily from the amorphous glass support, which produces a characteristic diffuse scattering maximum in the 20° – 30° range. A secondary contribution from residual liquid paraffin retained on the NC surface following two washing cycles with *n*-hexane cannot be entirely excluded. This assignment is supported by the complete absence of any additional crystalline peaks indicative of a secondary amorphous CsPbI₃ phase or decomposition products.

The exceptionally high relative intensity of the (200) reflection at $2\theta \approx 28.94^\circ$ is noteworthy. In addition to the intrinsically high structure factor of this plane, the drop-cast deposition geometry may induce preferred crystallographic orientation, wherein the {100} facets of the cubic NCs align preferentially parallel to the glass substrate. Such texture effects are well documented in films of cubic nanocrystals prepared by solvent-evaporation methods and would systematically enhance the intensity of *h*00 reflections^[47]. The overall consistency of the pattern with α -CsPbI₃ is unaffected by this consideration.

3.2. Morphological Characterization, Particle Size Distribution, and Consistency with XRD Analysis

Transmission electron microscopy (TEM) was employed to characterize cesium lead iodide (CsPbI₃) perovskite nanocrystals synthesized in a PaL medium. Following a two-step washing procedure with *n*-hexane, the nanocrystal dispersion was drop-cast onto a 300-mesh carbon-coated copper grid. Figure 2 presents a typical TEM micrographs obtained using a LEO-912 microscope (ZEISS) operating at an accelerating voltage of 100 kV. Representative micrographs reveal that the QDs possess a well-defined cubic morphology, in agreement with the cubic crystallographic symmetry (Pm3m space group) of the photoactive black α -phase CsPbI₃ identified by XRD analysis (Section 3.1). The preferential exposure of the {100} facets, which are energetically stable under ligand-passivated conditions, is responsible for the cube-shaped morphology and is a characteristic structural feature of lead halide perovskite QDs synthesized via ligand-assisted methods^[10, 48].

A striking structural feature evident in the TEM micrographs is the spontaneous formation of large-area, multi-domain superlattices, wherein the cubic QDs self-assemble into highly ordered, square-packed two-dimensional arrays (Fig. 2). This long-range superlattice organization is a hallmark of monodisperse nanocrystal systems and is driven by the interplay between isotropic steric repulsion from the surface ligand shell and van der Waals attractive forces between adjacent QDs. The estimated center-to-center spacing between QDs within the superlattice (~ 9.5 nm) implies an inter-particle gap of approximately 3.8 nm, consistent with a bilayer of oleylamine/oleic acid ligands (~ 1.9 nm per side) passivating the QD surfaces. The coexistence of multiple superlattice domains with distinct orientational alignments, separated by grain boundary-like interfaces, suggests that the self-assembly process proceeds via kinetically controlled nucleation of ordered domains during solvent evaporation on the TEM grid.

Quantitative analysis of the particle size distribution was carried out by measuring the edge length of $n = 270$ individual, well-separated cubic QDs selected from multiple non-overlapping regions of the TEM micrographs, using ImageJ/Fiji software^[49, 50] with the scale bar precisely calibrated prior to measurement. The resulting size distribution histogram (Fig. 3) yields a mean edge length of $\bar{d} = 10.97 \pm 2.16$ nm (mean $\pm$ standard deviation), a median of 10.65 nm, and a 95% confidence interval of (10.71, 11.23) nm. The polydispersity index ($PDI = \sigma/\bar{d} \times 100 = 19.7\%$) falls within the range commonly reported for perovskite QDs prepared by heat-up or hot-injection routes in weakly coordinating media. The moderate PDI may be partly attributed to residual liquid paraffin coating on a fraction of the QDs — a consequence of the paraffin-based synthesis medium — which can overestimate the apparent particle size in TEM projection images for some nanocrystals. The distribution exhibits a moderate positive skewness ($\gamma_1 = 0.58$), with the majority of the population ($> 75\%$) confined to the 9.5–12.5 nm range and a low-abundance tail extending to ~ 17 nm, a feature that is characteristic of colloidal nanocrystal ensembles governed by Ostwald ripening kinetics^[51, 52] and is more accurately represented by a log-normal rather than a strictly Gaussian distribution.

To independently validate the TEM-derived particle dimensions, the average crystallite size was calculated from the X-ray diffraction data using the Debye–Scherrer equation^[53]:

$$D = K \lambda / (\beta \cos \theta)$$

where D is the average crystallite size, $K = 0.9$ is the dimensionless shape factor, $\lambda = 0.15406$ nm is the wavelength of the Cu K α radiation, β is the full width at half maximum (FWHM) of the diffraction peak expressed in radians, and θ is the Bragg angle. Applying this relation to the most intense and well-resolved diffraction peak at $2\theta = 28.94^\circ$ (corresponding to $\theta = 14.47^\circ$), the Scherrer analysis yields an average crystallite size of $D = 11.72$ nm^[53]. This value is in excellent agreement with the mean edge length determined by TEM ($\bar{d} = 10.97$ nm), with a relative deviation of less than 7%, which is well within the typical uncertainty of the Scherrer method arising from instrumental broadening, peak asymmetry, and microstrain contributions. The close correspondence between these two independent, complementary techniques constitutes strong evidence that each individual QD consists of a single coherent crystalline domain — that is, the physical particle size and the crystallite size are essentially equivalent — implying a low density of internal grain boundaries, dislocations, and extended structural defects within the nanocrystals. This single-crystalline character is a critical prerequisite for achieving high photoluminescence quantum yield (PLQY) and narrow emission linewidth in perovskite QD systems, as sub-crystalline grain boundaries and trap states are primary non-radiative recombination channels.

Taken together, the TEM morphological analysis, the particle size distribution statistics, and the XRD Scherrer sizing collectively confirm the successful synthesis of phase-pure, cubic, single-crystalline CsPbI₃ QDs with a mean size of approximately 11 nm (Table 1). At this length scale, the QDs reside in the moderate-to-strong quantum confinement regime, given that the exciton Bohr diameter of bulk CsPbI₃ is reported to be in the range of 12–24 nm^[54]. The resulting quantum confinement effect is expected to blue-shift the excitonic absorption onset and photoluminescence emission maximum relative to the bulk

bandgap (E_g , bulk ≈ 1.73 eV) ^[55], as will be discussed in detail in the following section on optical characterization (Section 3.3).

Table 1
Summary of XRD Structural Analysis Parameters.

Parameter	Value	Unit	Note
Primary peak (2 θ)	28.94	°	(200) reflection
Bragg angle (θ)	14.47	°	—
Phase	α -CsPbI ₃	—	Cubic, Pm3m
Shape factor (K)	0.9	—	Scherrer
λ (Cu K α)	0.15406	nm	—
Crystallite size D	11.72	nm	Scherrer eq.
TEM mean edge length	10.97 $\pm$ 2.16	nm	n = 270
XRD / TEM deviation	< 7%	—	✓ Consistent
Secondary phases	None detected	—	Phase pure
Sample preparation	Drop-cast / glass	—	Thick NC film

3.3. Photoluminescence Performance of CsPbI₃ PNCs Synthesized in Paraffin Liquid (PaL) Compared to Conventional ODE Medium

As described in “Material and Methods” section, an integrated PL intensity versus absorbance method was employed to evaluate and compare the PL properties of CsPbI₃ PNCs synthesized in two distinct media of PaL and ODE. The PL emission spectra for five diluted solutions are demonstrated in Fig. 4. The UV-Vis absorbance spectra of these solutions at, concurrently measured 450 nm, are depicted in Fig. 5. The integrated area under each PL emission curve is plotted against the corresponding absorbance value at 450 nm in Fig. 6. Our comparative analysis revealed that the slope of the regression line derived from CsPbI₃ NCs synthesized in PaL was approximately **60% higher** than that obtained for CsPbI₃ PNCs synthesized in ODE. This significant enhancement in PL intensity indicates superior luminescent performance for the CsPbI₃ PNCs prepared in the PaL medium.

This pronounced increase in PL intensity can be attributed to several factors intrinsically linked to the synthesis environment including enhanced crystallinity, reduced defects, favorable morphology and size distribution. The PaL medium appears to provide a more favorable environment for the growth of CsPbI₃ PNCs with a higher degree of crystalline order and a lower concentration of structural defects (e.g., dislocations, grain boundaries, point defects). Such defects often act as non-radiative recombination centers, effectively quenching the emissive pathways and diminishing PL intensity. Conversely, synthesis

in ODE may lead to a higher prevalence of defects within the CsPbI₃ PNCs structure. While initial analyses indicated comparable average sizes, subtle variations in nanocrystal morphology and size distribution, influenced by the synthesis medium, can impact their optoelectronic properties and consequently their luminescent efficiency. In conclusion, these PL results strongly suggest that employing PaL as the synthesis medium is a promising strategy for obtaining CsPbI₃ nanocrystals with superior optical quality and enhanced potential for applications in optoelectronic devices.

Conclusion

This research successfully demonstrated an optimized method for synthesizing CsPbI₃ perovskite nanocrystals using PaL as a reaction medium. By leveraging the unique properties of PaL, we achieved highly crystalline, cubic phase (α -CsPbI₃) nanocrystals with significantly improved photoluminescence (PL) properties. Specifically, the CsPbI₃ nanocrystals synthesized in PaL exhibited a 60% increase in relative quantum yield compared to those prepared using ODE. Characterization through XRD and TEM confirmed the formation of uniform, stable nanocrystals with an average size of approximately 11 nm. These findings clearly establish the effectiveness of PaL as a superior reaction medium for producing high-quality, stable CsPbI₃ nanocrystals. Furthermore, the cost-effectiveness of PaL presents a significant advantage for large-scale production and facilitates further experimental process development, making this optimized synthesis pathway a promising route for advancing the development and application of these materials in next-generation optoelectronic devices and sustainable energy technologies. Further investigations could explore the long-term stability under operational conditions and the scalability of this method for industrial applications.

Declarations

Declaration of interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Author Contribution

A.G.K.: investigation, syntheses, formal analysis, visualization, writing-original draft; V.S.: conceptualization, methodology, project administration, supervision, writing-review and editing; S.M.A.H.: methodology, supervision, writing-review and editing; N.J.Z.: syntheses, formal analysis, methodology.

Data availability

No datasets were generated or analyzed during the current study.

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Figures

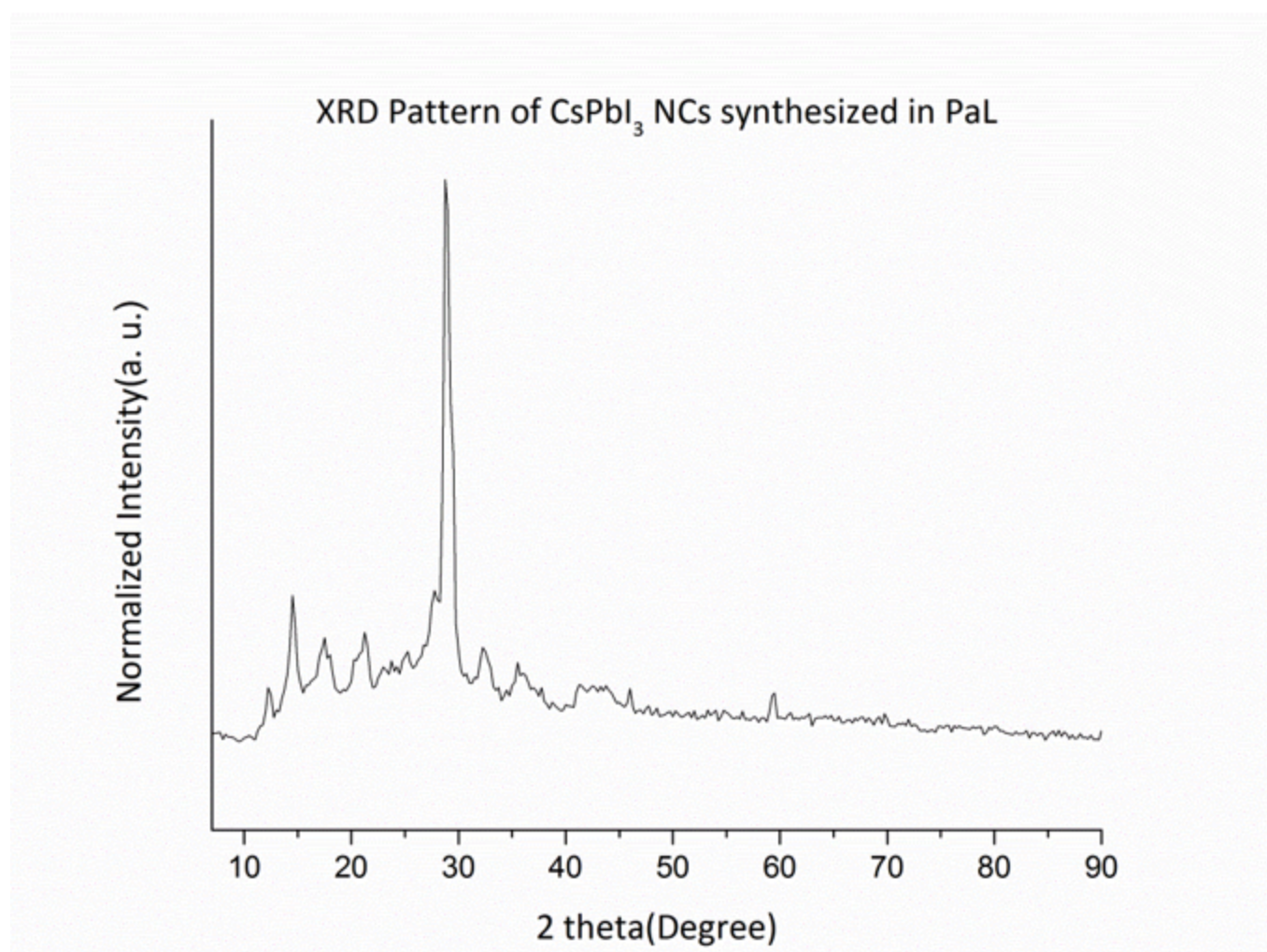


Figure 1

XRD Pattern of the CsPbI₃ NCs synthesized in Pal medium

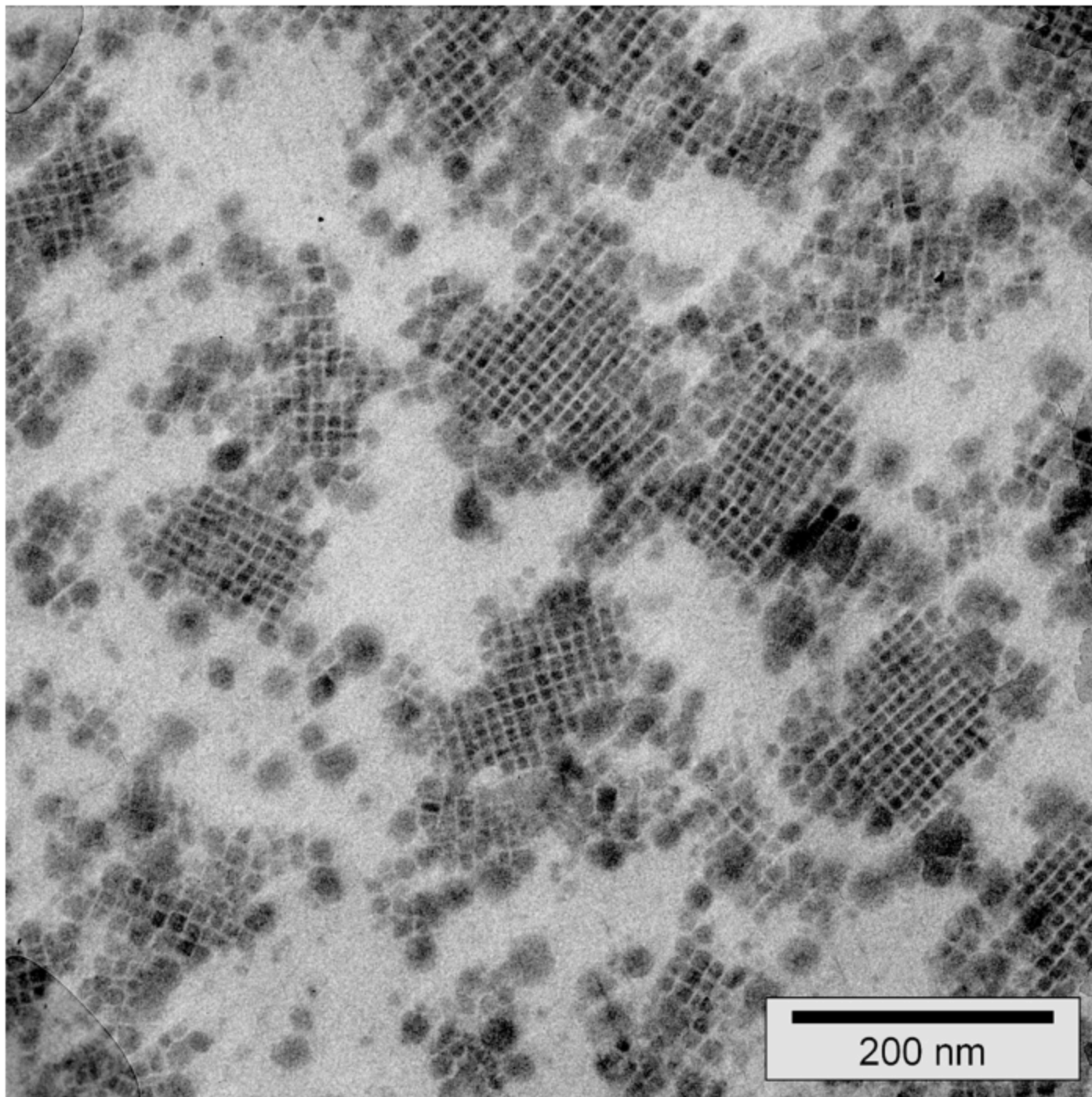


Figure 2

TEM Image of the CsPbI₃ PNCs synthesized in PaL medium.

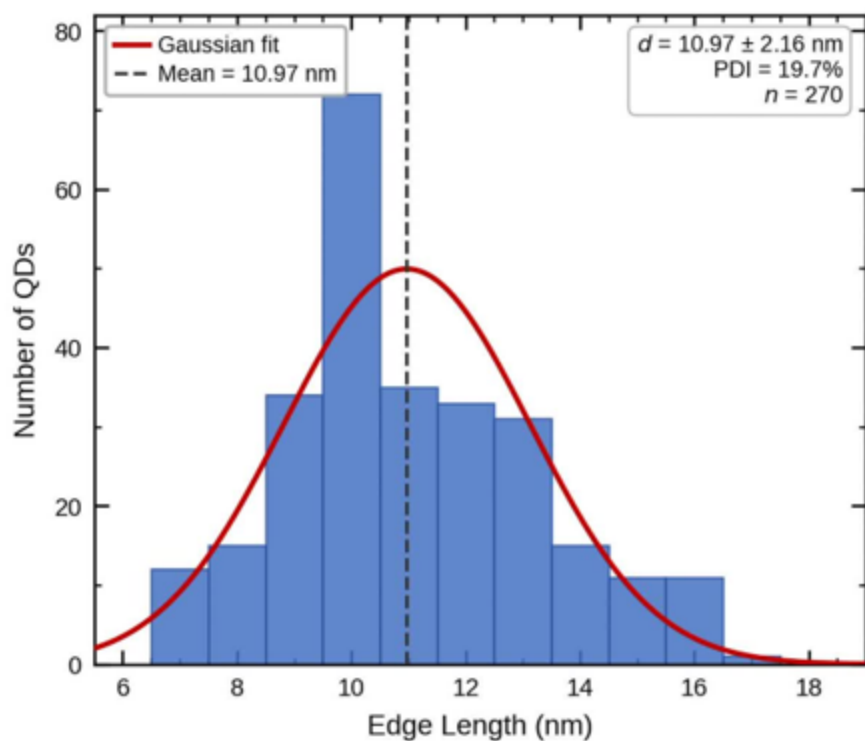


Figure 3

Histogram showing the size distribution of CsPbI₃ PNCs synthesized in PaL medium.

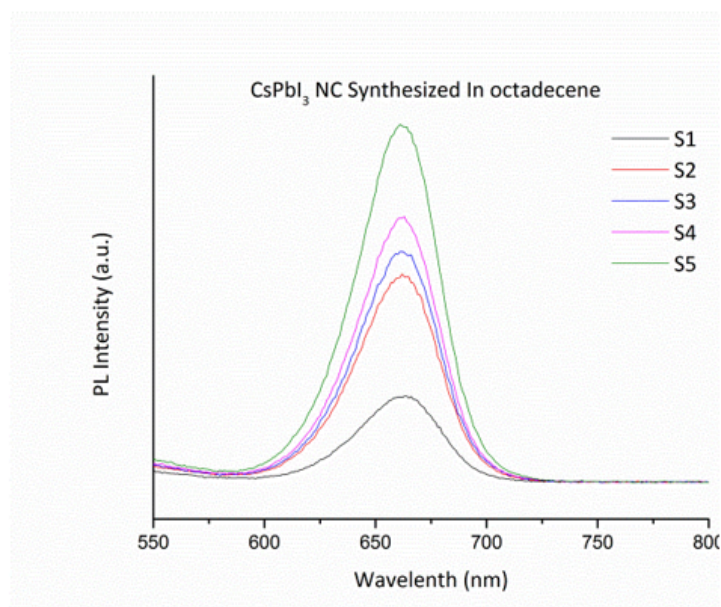
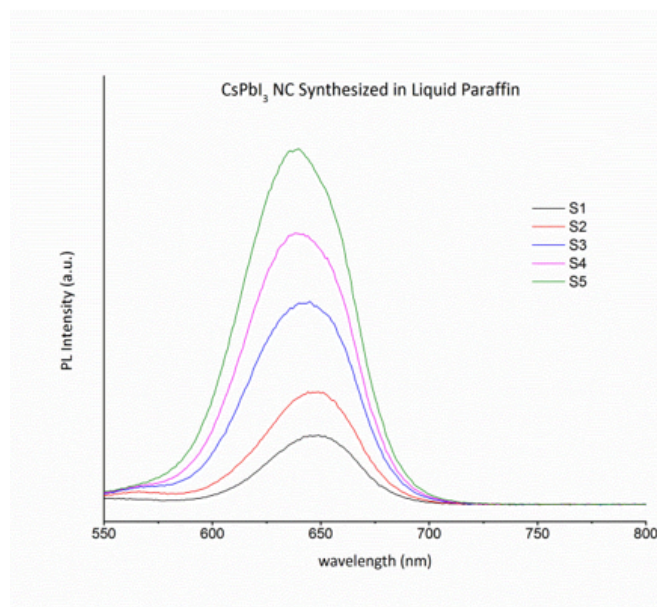


Figure 4

PL emission spectra of CsPbI₃ PNCs synthesized in (right: ODE medium and Left: PaL medium) of five serially diluted solutions S1, S2, S3, S4 and S5 in normal n-Hexane.

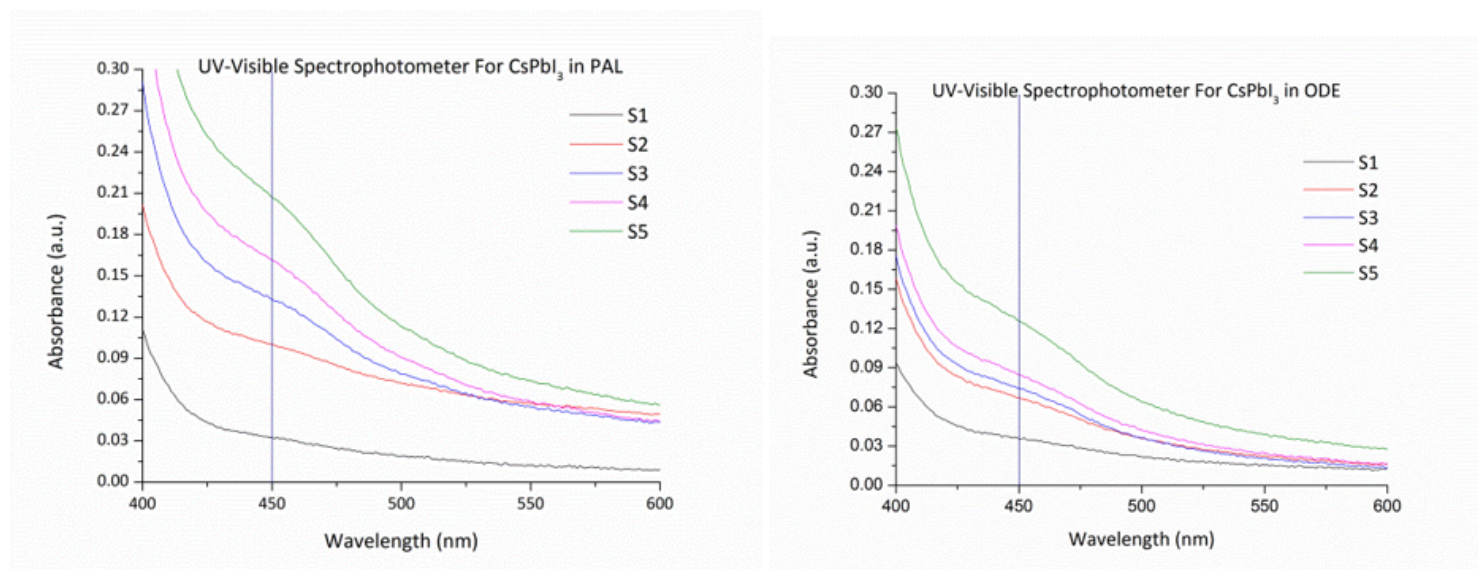


Figure 5

UV Absorption spectra of CsPbI₃ PNCs synthesized in PaL (Left) and ODE (Right) for five serially diluted solutions S1, S2, S3, S4 and S5 in normal n-Hexane.

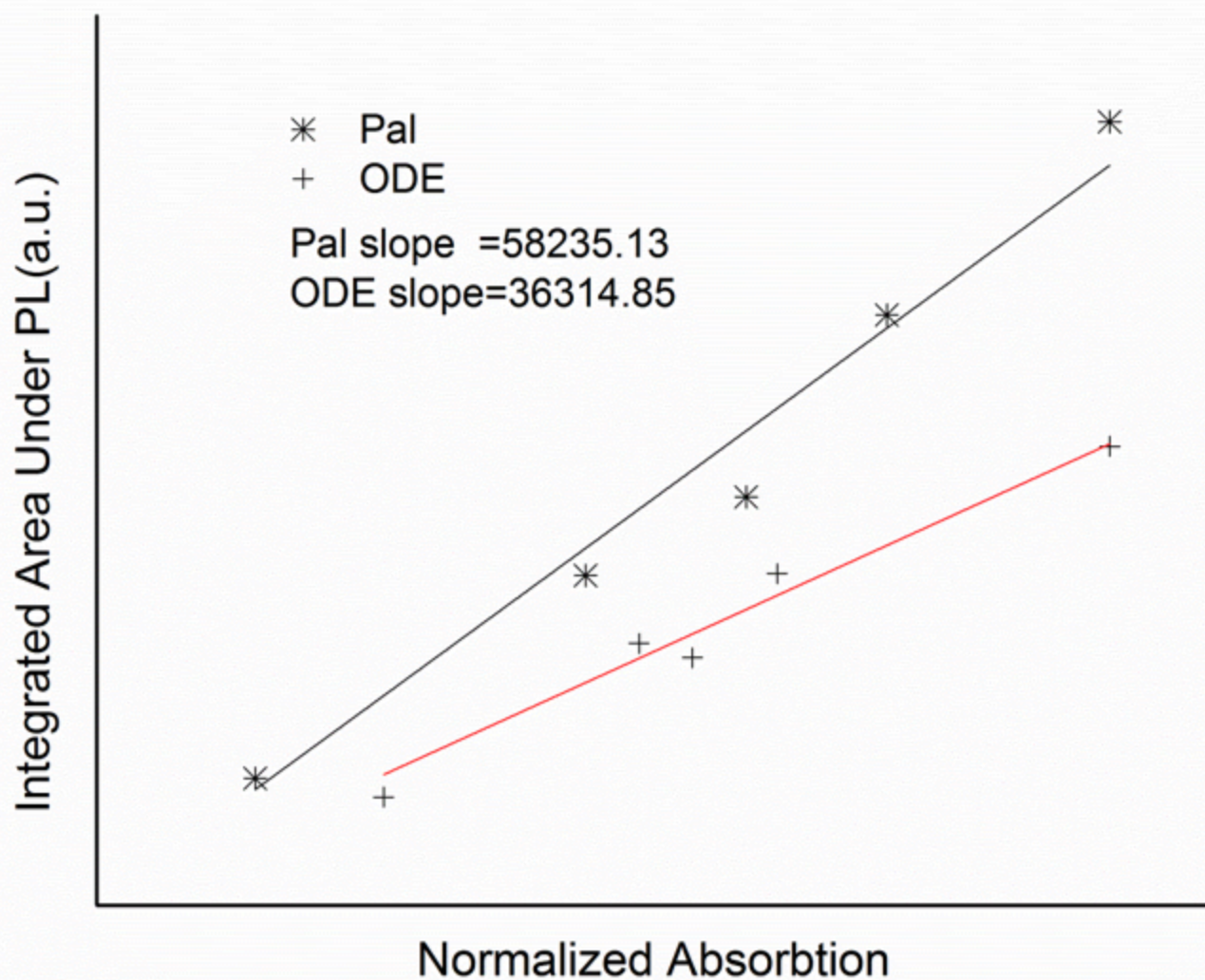


Figure 6

Linear fitting of integrated PL intensity versus absorbance at the excitation wavelength of 450 nm. Data were obtained from five serially diluted solutions. The slope of the fitted line represents the relative photoluminescence quantum yield (Rel. QY).