Dataset of ligand-controlled synthesis of CsPbBr₃ nanoplatelets

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Abstract

Lead halide perovskites nanocrystals have emerged as next-generation materials in the application of photovoltaics and various optoelectronics owing to the controllable optoelectronic properties (achieved through varying the dimensionality and composition of the materials). The design and control to obtain the desirable optoelectronic properties of the halide perovskite nanocrystals, thus, remain paramount importance. The synthesis and stabilization of cesium lead bromide (CsPbBr₃) nanoplatelets through ligand-assisted reprecipitation protocol (LARP) can indeed enable the manipulation of the layer thickness over the resulting nanoplatelets. Herein, we have elucidated the role of ligand concentration and chain length effect on the crystal growth and mapped how these parameters affect the layer thickness (and crystal growth kinetics) of the corresponding nanoplatelets. Complex mapping the evolution of the average layer thickness of the CsPbBr₃ nanoplatelets provide a detailed perspective of the crystal growth with ligand shell assembly. Transmission electron microscopy (TEM) images directly measurable for the thickness of nanoplatelets along with photoluminescence (PL) emission spectroscopy have been employed to determine of the thickness of the nanoplatelets exhibiting

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thickness-dependent optical properties with different layer thickness nanoplatelets.

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Specifications Table

| Subject | Materials Science and Engineering / Inorganic chemistry |
|---------|------------------------------------------------------|
| Specific subject area | Lead halide perovskite nanoplatelets |
| Type of data | Scheme |
| How data were acquired | Transmission electron microscope, Photoluminescence emission spectroscopy |
| Data format | Raw |
| Parameters for data collection | Colloidal solution of cesium lead halide perovskite nanocrystal dispersed in toluene were recorded for photoluminescence emission spectra |
| Description of data collection | Photoluminescence emission spectra were recorded in the spectral range of 400-600 nm under the excitation wavelength of 360 nm using a Xenon arc lamp as the light excitation source |
| Data source location | Institution: Duksgun Women’s University |
| Data source location | City/Town/Region: Seoul |
| Data source location | Country: South Korea |
| GPS coordinates | Latitude and longitude (and GPS coordinates, if possible) for collected samples/data: |
| Data accessibility | https://data.mendeley.com/datasets/7hn8v6czkf/1 |
| Related research article | For a published article: J. Cho, S. Banerjee, Ligand-Directed Stabilization of Ternary Phases: Synthetic Control of Structural Dimensionality in Solution-Grown Cesium Lead Bromide Nanocrystals, Chem Mater. 30 (2018) 6144–6155. https://doi.org/10.1021/acs.chemmater.8b02730 |

Value of the Data

• The data shown herein explains the structural evolution for cesium lead halide perovskites as a function of varying the ligand concentration and ligand chain length.
• The data provides a fundamental insight on the layer thickness-control that is mediated through a surface-passivating ligand for stabilizing the cesium lead halide nanoplatelets.
• The data can be further used for designing experiments and advanced research in the field of synthesis of halide perovskite nanoplatelets and their application in various optoelectronic devices.

1. Data Description

The data presented here is related to experimental synthesis of cesium lead bromide nanoplatelets with various thickness of n = 2–10 that is accessible through the ligand-assisted reprecipitation (LARP) that is performed at room temperatures. A rapid change in solubility of precursor solution through transferring the solution from a polar solvent to a nonpolar solvent can indeed induce the rapid precipitation and recrystallization of cesium lead bromide. During the process, the alkylamine serving as surface-passivating ligands play a crucial role in stabilizing the different layer thickness of nanoplatelets. A conceptual scheme illustrated in Fig. 1 demonstrates the overall ligand-assisted reprecipitation process to obtain the colloidal cesium lead bromide nanocrystals as a function of varying the alkylamine concentration and chain length. Fig. 2
Fig. 1. (A) Schematic illustration for synthesis of cesium lead bromide nanocrystals through ligand-assisted reprecipitation method (LARP) wherein the solubility change in the precursor solution leads to the precipitation and recrystallization of corresponding colloidal nanocrystals at room temperatures. (B) Control over the lead octahedral layer thickness of nanocubes vs nanoplatelets through ligand-mediated synthesis using different chain-length of ligands from butylamine (C\textsubscript{4}) to oleylamine (C\textsubscript{18}).

Fig. 2. (A-C) Photoluminescent (PL) emission spectra acquired for cesium lead bromide nanocrystals stabilized with different alkylamine chain length from butylamine (C\textsubscript{4}) to oleylamine (C\textsubscript{18}) at the Pb:alkylamine=1:x (x = 0.5 (A), x = 1 (B), and x = 2 (C)). Inset to individual figure panels represent the digital photograph taken under UV lamp excitation of 365 nm. The dotted lines indicates the different layer thickness of nanoplatelets with n (n = 1–10).

Fig. 3 shows the transmission electron microscope (TEM) images for the cesium lead bromide nanoplatelets obtained using octylamine (C\textsubscript{8}) as a ligand. The PL emission spectra and emission color under hand UV excitation (365nm) can be used as a proxy to determine the layer thickness (and bandgap) of the nanoplatelets. Fig. 4 plots the 2D color contour mapping for the average layer thickness of cesium lead bromide nanoplatelets obtained at varying the alkylamine ligand concentration (x = 0.5–2) and chain length (C\textsubscript{4}–C\textsubscript{18}). Fig. 5 illustrates the photoluminescent emission spectra during the process of halide exchange reactions using the thick cesium lead bromide nanoplatelets (C\textsubscript{8} with x = 0.5) as a parent compound.
Fig. 3. (A) Low-magnification and (B) high-magnification transmission electron microscope (TEM) images acquired for the cesium lead bromide nanoplatelets stabilized using octylamine (C8) as a surface-capping ligands with a concentration of $x = 2$. In panel B, the individual thickness of the nanoplatelets is shown.

Fig. 4. Two-dimensional color contour plots mapping for the average layer thickness of cesium lead bromide nanoplatelets stabilized at varying the alkylamine ligand concentration ($x = 0.5$–2) and chain length (C4-C18). Color bar corresponds to average layer thickness (bluer is thinner while redder is thicker).
2. Chemicals

All chemicals were used without any further purification. Oleic acid (technical grade, 90%), oleylamine (technical grade, 70%), and 1-octadecene (technical grade, 90%) were purchased from Sigma-Aldrich. n-Octylamine (C8, >98.0%) was purchased from TCI-America. Toluene was purchased from EMD Millipore. Cesium carbonate (Cs$_2$CO$_3$, 99%), lead bromide (PbBr$_2$, 98+%), butylamine (C4, 99%), hexylamine (C6, 99%), dodecylamine (C12, 97%), dimethylformamide (DMF, 99%), and toluene (anhydrous, 99.8%) were purchased from Alfa-Aesar.

3. Experimental Design, Materials and Methods

The synthesis and data acquisition for colloidal cesium lead bromide nanoplatelets have been demonstrated in the previously reported publications [1–3]. Corresponding colloidal nanocrystals were stabilized through the ligand-assisted reprecipitation method (LARP) performed at room temperature [4]. Briefly, a precursor solution including Cs-oleate and lead bromide (PbBr$_2$) was made by mixing 37.5 μL of 0.4 M Cs-oleate, 0.030 mmol (0.011 g) of PbBr$_2$, and 0.2 mL of oleic acid which are consequently dissolved in 0.6 mL DMF with a stoichiometric amount addition of alkylamine ligands (C4-C18); 0.4M of Cs-oleate solution was already made by mixing and dissolving 2.5 mmol (0.4072 g) of Cs$_2$CO$_3$ in 2.5 mL of oleic acid and 10 mL ODE that were further heated at 180 °C under ambient condition. The stoichiometric ratio of Pb:alkylamine was systematically varied between x = 0.5 and 2. As-prepared mixed precursor solution was rapidly transferred to 10 mL of toluene with vigorous stirring which also leads to the formation of cesium lead bromide nanoplatelets. The crude solution was centrifuged at 5000 rpm for 10 min to obtain the precipitation, which was then re-dispersed in 5 mL of toluene for further characterizations. Halide exchange reactions were then performed using the resulting CsPbBr$_3$ nanocrystal dispersion in toluene as a parent solution by adding a stoichiometric amount of lead halide sources (such as PbCl$_2$ and PbI$_2$ that are already prepared with mixing with oleic acid, oleylamine, and octadecene and heating at 200 °C until entire dissolution of solids). Photoluminescence (PL) emission spectra were recorded with a Horiba PTI Quanta-Master series spectrofluorometer with

Fig. 5. Photoluminescent emission spectra obtained through halide exchange reaction using thick cesium lead bromide nanoplatelets (C8 with x = 0.5) as a parent compound. Inset to figure panel corresponds to the digital photographs for the four different PL spectrum.
a Xenon arc lamp as the light excitation source and a photomultiplier tube detector. The excitation wavelength of 360 nm was chosen to record the corresponding PL emission spectra. High- and low-magnification transmission electron microscopy (TEM) images were collected using a FEI Tecnai G2 F20 ST instrument operating at an accelerated voltage of 200 kV.

**Ethics Statement**

The study does not contain any research involving human participants and/or animals performed by any of the author.

**CRediT Author Statement**

**Junsang Cho:** Conceptualization, Methodology, Experiment, Data analysis, Writing-Original Draft Preparation, Investigation, Supervision, Software, Writing- Reviewing and Editing

**Declaration of Competing Interest**

The authors declare that they have no known competing financial interests or personal relationships which have or could be perceived to have influenced the work reported in this article.

**Data Availability**

Dataset of Ligand-Controlled Synthesis of CsPbBr3 Nanoplatelets (Original data) (Mendeley Data).

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