Perovskite Nanocrystal Synthesis as Undergraduate Laboratory Practice: Methodology Analysis

Daniil Petrakov

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Department of Chemistry, Douglas College

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Introduction

Overview
Lead halide perovskite nanocrystals (LHP NCs) with the $CsPbX_3$ structure (X = Cl, Br, or I) exhibit exceptional photoluminescent properties and have attracted extensive attention for their optoelectronic applications. Of particular interest is their potential in chemical education. The emission wavelength of LHP NCs is tunable, enabling light emission across the entire visible spectrum (410–700 nm) under UV light. This makes LHP NCs excellent candidates for visually striking demonstrations and appealing synthetic targets for inorganic chemistry laboratory courses that are also tied to modern materials science research.

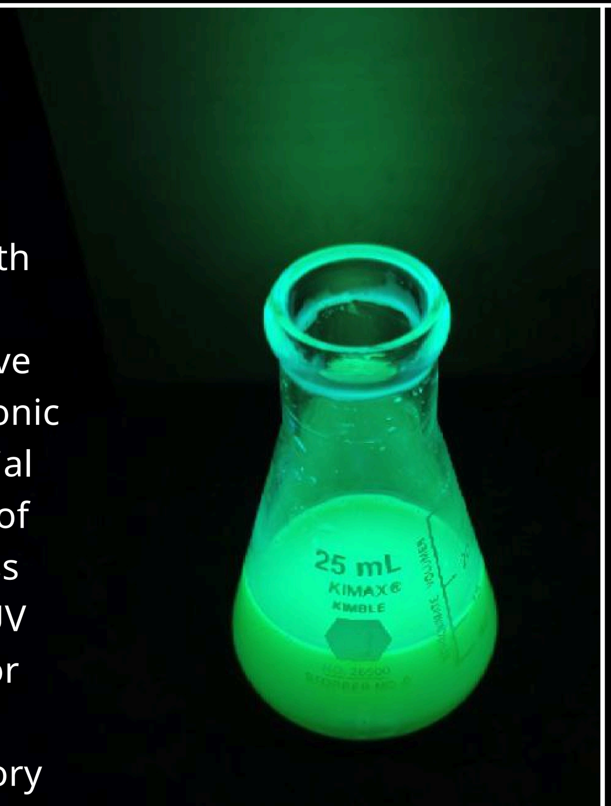


Figure 1. CsPbBr3 solution in toluene under UV light

Photoluminescence (PL)
Photoluminescent materials can absorb light of a certain wavelength, raising the energy level of the electrons inside of them. When these excited electrons inevitably fall back to lower energy levels, they emit this energy back in the form of light – but at a longer wavelength

The wavelength a material can absorb and the wavelength it emits back depend on the chemical composition of the material. In the case of LHP NCs, UV light is absorbed, and visible light is emitted. By varying the ratio of halogens (Cl/Br/I) in the structure, LHP NCs can be made to emit light at any wavelength in the visible spectrum.

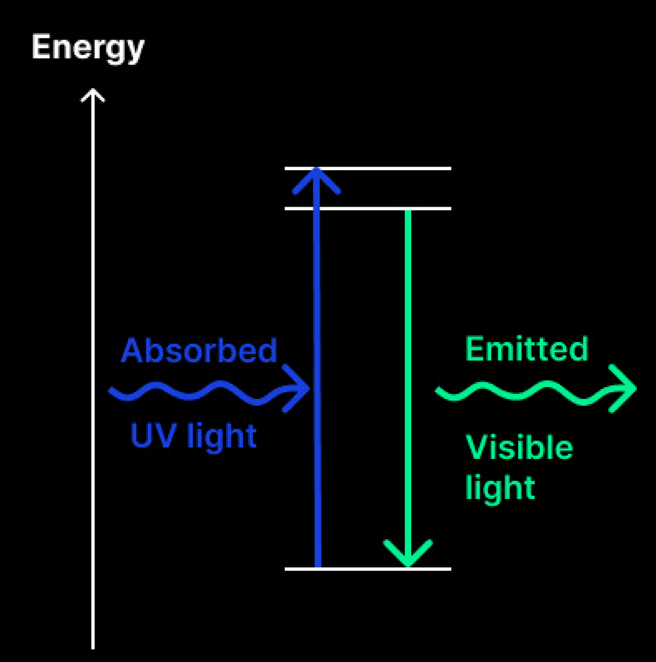


Figure 2. Diagram illustrating the basics of photoluminescence

Applications
Inorganic LHPs have a broad range of optoelectronic applications including LEDs, photodetectors, solar cells, and lasers. LHP-containing solar cells have been shown to reach efficiency levels of up to 30%, surpassing the traditional silicon semiconductor cells by over 10%. [1]

Methods and materials

All of the reagents were acquired directly from Sigma-Aldrich and Fischer Scientific and used without any purification or drying. Technical grade reagents were used to ensure minimal cost.

After literature review, we have identified the three most prospective methods for LHP NCs synthesis (LARP, hot injection, and phase conversion). CsPbBr3 was chosen as it is the most stable and easiest to synthesize out of the three (Cl/Br/I). The procedures were modified for lower scale and synthesis in air as opposed to inert conditions (see Fig. 3)

Cost per run and procedure complexity were assessed to determine the most fit method. Fluorometry at 405 nm was used to confirm the production of CsPbBr3 and confirm the identity and purity of the product.

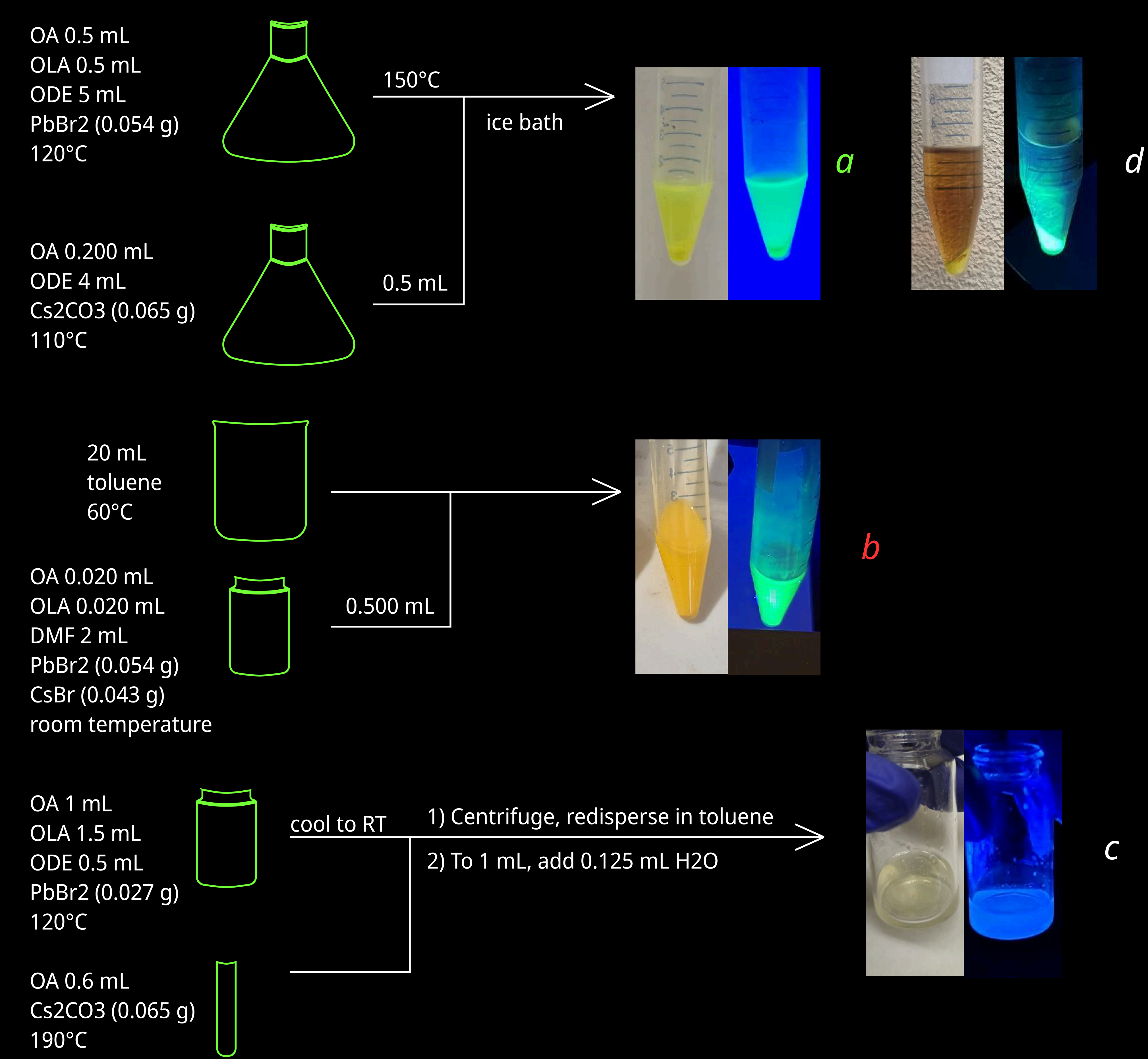


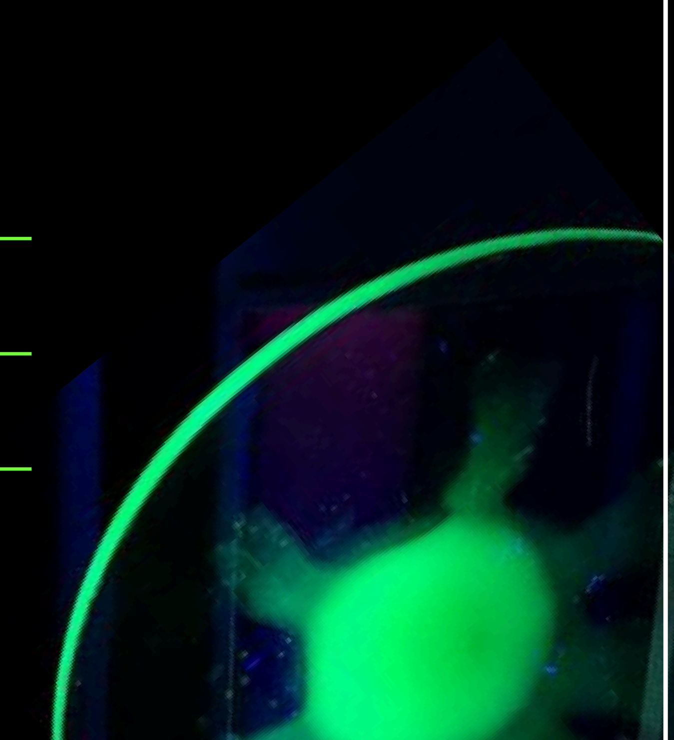
Figure 3. Methods of CsPbBr3 nanocrystals synthesis and product pictures (under daylight/UV). OA - oleic acid, OLA - oleylamine, ODE - 1-octyldecene, DMF - N,N-dimethylformamide.

a) CsPbBr3 nanocrystals synthesized using hot injection method
b) CsPbBr3 nanocrystals synthesized using LARP method
c) Product of the phase conversion method
d) CsPbBr3 nanocrystals synthesized using hot injection method out of thermally compromised precursor solutions

Findings

Table 1. Relative rankings of sythesis methods (1 is best, 3 is worst)

Method	Synthesis success	Cost per run	PL intensity	Procedure complexity
Hot injection	Yes	3	1	2
LARP	Yes	1	2	1
Phase conversion	No	2	3	3



Findings, continued

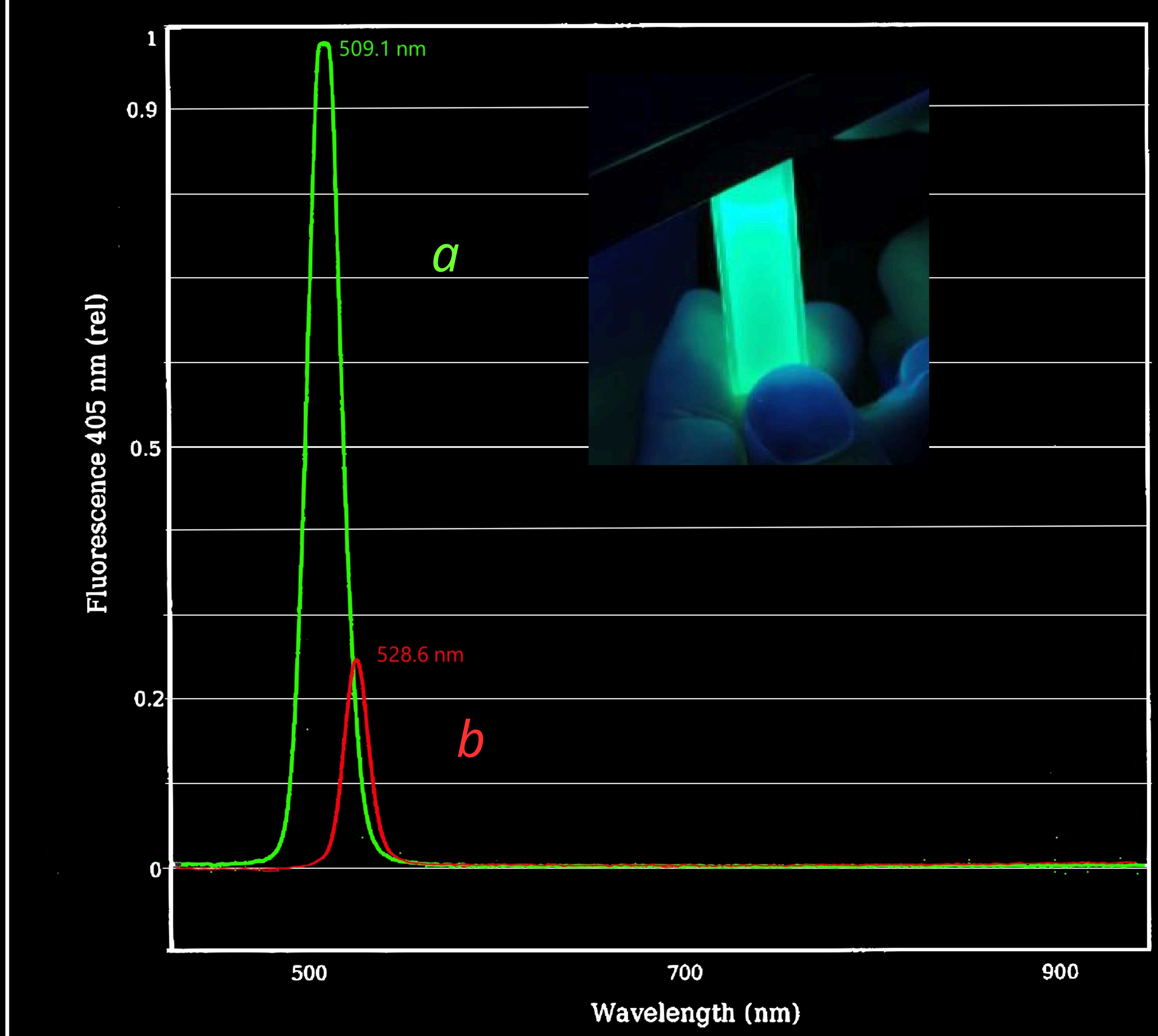


Figure 4*. Emission at 405 nm of synthesized CsPbBr3 solutions in toluene measured using Vernier's Go Direct® SpectroVis® Plus Spectrophotometer through LabQuest 2.*

a - hot injection (509.1 nm, 0.97 rel. intensity, pictured on the right)
b - LARP (528.6 nm, 0.25 rel. intensity)

The hot-injection method yielded highly luminescent NCs that formed a colloidal solution when redispersed in toluene (Fig. 3a). Furthermore, viable CsPbBr3 NCs were produced even when the stock solutions were deliberately heated over the recommended temperatures (Fig. 3d)

The LARP method was calculated to be cheaper and less complex due to using less overall glassware and reagents. However, it produces nanocrystals with less PL intensity as seen in Fig. 4.

As the phase conversion method did not produce any results (blue color in Fig. 3c is due to reflected UV light), we pronounce hot injection the most viable method of CsPbBr3 synthesis for an undergraduate lab.

Future work

Our success in selecting a reliable method of synthesizing CsPbBr3 in suboptimal conditions opens the path for further investigation. To further demonstrate the optoelectronic properties of perovskites, Cl or I could be introduced by anion exchange with PbCl2 and PbI2 respectively, shifting the emission wavelength. Doping with transition metals can also be investigated.

References

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