Changing the Dimensionality of Cesium Lead Bromide Nanocrystals by Reversible Postsynthesis Transformations with Amines

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Supporting Information

Cesium lead halide (CsPbX$_6$: X = I, Br, Cl) nanocrystals (NCs) were first synthesized by Protesescu et al. in 2015† and since then have increasingly attracted the attention of the scientific community. In the time span of less than 2 years, a plethora of reports on colloidal syntheses have appeared, enabling shape, size and composition control,‡−§ postsynthesis transformations,¶−¶ and applications in optoelectronics.‡¶−¶

In the last year, research has extended to various other types of cesium lead halide NCs, with a structure different from the original three-dimensional (3D) orthorhombic/cubic CsPbX$_6$, including CsPbBr$_x$ NCs,²⁰−²² and also the so-called zero-dimensional (0D) Cs$_4$PbX$_6$ phase.²³−²⁵ The terms “3D” and “0D” here refer to the coupling or decoupling of PbBr$_6$ octahedra in the crystal. In the 3D CsPbBr$_3$ structure, the lead hexahemidodecahedra share all corners and, hence, they are electronically coupled in the three directions of space, whereas in the 0D Cs$_4$PbBr$_6$ lattice the octahedra are fully decoupled (not sharing any corner), which results in drastically different optoelectronic properties. For example, our group has recently shown that 0D colloidal Cs$_4$PbBr$_6$ NCs can be synthesized with good control over the size distribution and we additionally demonstrated that these NCs can be transformed to bright-emitting CsPbBr$_3$ NCs by postsynthesis intercalation of PbBr$_2$.²⁵

Here we report how, starting from CsPbBr$_3$ NCs, it is possible to obtain 0D Cs$_4$PbBr$_6$ NCs by the simple addition of different amines at room temperature. This transformation is triggered by the extraction of PbBr$_2$ from the 3D CsPbBr$_3$ perovskite NCs operated by the excess amines, driven by the formation of complexes of amines with PbBr$_2$. Furthermore, we show that this process can also be carried out on dried NC films upon their exposure to vapors of a volatile alkylamine, such as butylamine (BuAm). Interestingly, the transformation can be inhibited if the NC ligands are first graphitized by X-ray irradiation.¹⁰ This was used to create luminescent patterns of CsPbBr$_3$ NCs surrounded by nonluminescent Cs$_4$PbBr$_6$ NCs. Eventually, upon mild thermal annealing, BuAm could be evaporated from the film and the NCs were reverted to the original luminescent 3D phase.

Monodispersed 8 nm CsPbBr$_3$ nanocubes (see Figure 1a) with an optical absorption onset around 500 nm (see Figure 1d, dotted black curve) and an X-ray diffraction (XRD) pattern (see Figure 2a) matching that of bulk orthorhombic CsPbBr$_3$ were synthesized following the work of Protesescu et al.† Upon exposure of a dispersion of CsPbBr$_3$ NCs in toluene to tetramethylethlenediamine (TMEDA) for a few minutes, a white precipitate was observed, consisting of ca. 50 nm rhombohedral shaped (in projection) polydisperse NCs (see Figure 1b). Unfortunately, the stability of such NCs in solution was poor, as the particles aggregated in the time span of a few minutes. However, their optical properties, measured immediately after the transformation, were drastically different from those of the starting CsPbBr$_3$ NCs, with a sharp absorption peak at 317 nm, no absorption in the visible range and no significant PL. These features are characteristic of 0D Cs$_4$PbBr$_6$ NCs,²³−²⁵ as reported previously by us and as corroborated by XRD analysis (see Figure 1e), although the absorption peak is slightly shifted from the expected value of 314 nm. This slight shift in absorption may be due to the fact the resulting NCs still contain a residual degree of coupling between PbBr$_6$ octahedra. The long tail in the extinction spectrum is most likely ascribable to scattering effects (due to particle aggregation) and to the presence of other impurity species in addition to the Cs$_4$PbBr$_6$ NCs, which are also responsible for minor reflections in the XRD pattern of the TMEDA treated sample (see Figure 1e). The much larger size of the 0D NCs compared to that of the original NCs suggests that the process cannot be based on an extraction of PbBr$_2$, followed by a structural reorganization of the NCs, as in this case the mean size of the particles should slightly decrease. Instead, the addition of amines, most likely, leads, after the extraction of PbBr$_2$, to the dissolution of the starting NCs followed by recrystallization to form the 0D Cs$_4$PbBr$_6$ NCs. The 3D to 0D transformation can be explained by considering that the PbBr$_2$ not employed in the formation of the 0D NCs (4CsPbBr$_3$ → Cs$_4$PbBr$_6$ + 3PbBr$_2$) forms stable complexes with the added TMEDA. Lead(II) halide salts are, indeed, known to form stable adducts with N-donor atom ligands.²⁶−²⁸ More precisely, stable mPbX$_2$·nL (L = amine) complexes have been isolated when working with many different amines. For example, TMEDA can form a m:n = 1:1 complex with PbBr$_2$, as shown by Wharf et al.²⁸ To verify this hypothesis, we performed infrared (IR) spectroscopy (see Figure S1 and Table S1 of the Supporting Information, SI) of pure TMEDA and TMEDA-treated NCs. The IR peaks positions of the TMEDA-treated NCs match very closely.
with those reported by Wharf et al. for the PbBr₂·nTMEDA complexes.

The reason for the poor colloidal stability of the 0D NCs obtained by reacting TMEDA with CsPbBr₃ NCs is most likely due to the strong coordination of the diamino group with Pb²⁺ ions, leading to a rapid transformation from 3D to 0D NCs. To decrease the speed of the reaction, we decided to use primary amines, which are known to have a lower affinity to PbBr₂. We tested oleylamine (OA) because it is routinely used in the synthesis of both 3D and 0D perovskite NCs. Indeed, upon exposure of the CsPbBr₃ NCs to OA, we observed the progressive disappearance of the exciton absorption peak at 500 nm, typical of the 3D phase, accompanied by the increase of the absorption peak at 314 nm, characteristic of the 0D phase (see Figure 1d and Figure S2 of the SI, where the extinction spectrum acquired after 1 h shows the presence of both CsPbBr₃ and Cs₄PbBr₆ phases). In this case, the transformation was much slower, requiring several hours for a complete conversion.

Motivated by these results, we tested the postsynthesis transformation of the original 3D structure into the 0D Cs₄PbBr₆ one by employing a short amine (i.e., butylamine, BuAm) on drop-cast films of CsPbBr₃ NCs on a substrate. The choice of such amine, rather than OA, was dictated by the possibility to perform the transformation with amine vapors at room temperature, as well as by the possibility to remove such amine, when necessary, by a mild annealing treatment, without significantly degrading the perovskite NCs film. Figure 2a,b displays the XRD pattern and the optical transmittance of the pristine CsPbBr₃ NC film (deposited on a quartz substrate). The film was then held for a few minutes on top of an open vial of BuAm at room temperature (BuAm is rather volatile, with a boiling point of 77 °C and a vapor pressure of 9.1 kPa). The exposure to BuAm quickly and almost fully converted the starting NCs to Cs₄PbBr₆ (see Video SI in the SI) as evidenced by the change in the XRD pattern (Figure 2c) and in the transmittance spectrum: the signal drop at approximately 510 nm, related to the presence of the 3D phase, disappeared while a new drop at 314 nm, typical for the 0D phase, became visible (Figure 2d). Another drop in transmittance was seen at around 390 nm. This is possibly coming from the concomitant formation of layered phases. Indeed, after longer exposure to BuAm gas (3 h), the XRD pattern of the film showed periodic diffraction peaks indicative of lamellar structures (see Figure S2 of the SI). The subsequent annealing of the film at 100 °C led to a rapid evaporation of the BuAm with the consequent release of the lead bromide salt. The lead bromide subsequently reacted with the Cs₄PbBr₆ phase, reforming the CsPbBr₃ structure (Figure 2e,f), in line with our recent results. Therefore, the “back” 0D to 3D transformation could be activated simply by thermal annealing (see Video S2 in the SI).

To understand better the evolution of the size and morphology of the CsPbBr₃ NCs deposited on a TEM grid, we reproduced the same postsynthesis protocol used for NCs film, but depositing the starting NCs on a TEM grid. The results of these experiments are illustrated in Figure 3. Upon exposure to the amine, the starting square-shaped (in projection) CsPbBr₃ NCs, exhibiting an orthorhombic crystal structure (see Figure 3a), transformed into rounded NCs of comparable size, but with a crystal structure that could be indexed as Cs₄PbBr₆ (see Figure 3b). Upon mild annealing of the TEM grid at 100 °C in air the 0D NCs
transformed into bigger irregular crystals, with orthorhombic 3D perovskite structure (see Figure 3c).

In recent works from our group,\textsuperscript{10,15,32} we have demonstrated how different reactions on NCs films can be inhibited when their ligands are partially graphitized by ionizing radiations. In those works, we showed that the irradiation of different nanocrystals with e-beams or X-rays under vacuum leads to the formation of C=C bonds (graphitization) between adjacent ligand molecules (oleylamine, oleic acid, or other long aliphatic molecules). Such cross-linking or graphitization results in a shielding of the NC cores against various external chemical species. This included also the inhibition of anion-exchange on films of CsPbX\textsubscript{3} NCs irradiated with X-rays. Here we used the graphitization of the ligands to inhibit the CsPbBr\textsubscript{3} to Cs\textsubscript{4}PbBr\textsubscript{6} transformation driven by BuAm, and we fabricated patterns of CsPbBr\textsubscript{3}/Cs\textsubscript{4}PbBr\textsubscript{6} exploiting the masked irradiation, as schematized in Figure 4 (top panel). Figure 4 also shows PL microscopy images and PL emission spectra corresponding to the 4 different steps of the overall process. First, CsPbBr\textsubscript{3} NCs were drop-cast on a silicon substrate (see Figure 4a) and irradiated through a mask (see Figure 4b). The irradiated regions had their PL partially quenched, as already reported by us.\textsuperscript{10} This drop in PL was found to be linked to surface trap states induced by the ligand graphitization rather than to the modification of the NC cores themselves. Upon exposure to BuAm vapors, the nonirradiated regions completely lost their PL (see Figure 4c). Eventually, upon annealing at 100 ºC for 30 min, the BuAm was evaporated and the Cs\textsubscript{4}PbBr\textsubscript{6} NCs were (at least partially) reverted to CsPbBr\textsubscript{3}, with the PL emission intensity being lower than that of the nontransformed regions, yet within the same order of magnitude (see Figure 4d). We further investigated the loss of PL upon different cycles of exposure to butylamine and subsequent annealing (on a nonirradiated film; see Figure S4) and found that the first cycle induces a loss of PL intensity of about 1 order of magnitude. This loss, however, was not ascribed to the structural 3D \textgreater{} 0D \textgreater{} 3D transformation, but rather due to annealing, as we found a similar PL drop on a reference 3D sample that was only annealed (i.e., without exposure to butylamine). After the first 3D \textgreater{} 0D \textgreater{} 3D cycle, we could perform three more cycles without significant loss of PL. At the fifth cycle, the PL intensity dropped again by about 1 order of magnitude and no further cycles were performed.

In summary, we have demonstrated an easy way to tune the structural and, consequently, the optical properties of cesium lead halide NCs in solution and in films by the simple addition of different amines at room temperature. This transformation could easily be reverted with a mild thermal annealing if a proper amine was used. Furthermore, we have shown that graphitization of the NC ligands inhibits the transformation, which can be used to create luminescent patterns of CsPbBr\textsubscript{3} surrounded by nonluminescent Cs\textsubscript{4}PbBr\textsubscript{6}.

**ASSOCIATED CONTENT**

*Supporting Information*

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.chemmater.7b00895.

Video of the transformation with BuAm gas (AVI)

Video of the back transformation via annealing (AVI)
Experimental details, infrared spectra of pristine solution, TMEDA and TMEDA-treated solution, relevant IR frequencies to evidence amino-lead bromide complexes, absorption spectrum of intermediate step in the OA treatment, XRD of BuAm-transformed NCs after 3 h, scaled PL spectra of samples treated with butylamine vapor and annealing (PDF)

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**Notes**
The authors declare no competing financial interest.

**ACKNOWLEDGMENTS**
The authors acknowledge G. La Rosa for support in transmittance spectra acquisition. The research leading to these results has received funding from the seventh European Community Framework Programme under Grant Agreement 614897 (ERC Consolidator Grant “TRANS-NANO”) and from framework programme for research and Innovation Horizon 2020 (2014-2020) under the Marie Skłodowska-Curie Grant Agreement COMPASS No. 691185.

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■ NOTE ADDED IN PROOF
During the review of our paper, a similar paper was published by Liu et al.\textsuperscript{33}