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Research paper

Environmentally-friendly synthesis of highly luminescent cesium lead halide perovskite nanocrystals using Sn-based halide precursors

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ABSTRACT

In the last several years, cesium lead halide perovskite ($CsPbX_3$, X = Cl, Br, I) nanocrystals (NCs) have attracted much attention as promising kinds of optoelectronic and photonic materials. Via anion exchange reactions, the photoluminescence spectra of the $CsPbX_3$ NCs can be tuned over nearly the entire visible spectral region. However, the preparation of the anion-exchange sources, mainly Pb-based halide precursors or oleylammonium halide precursors, would use large amounts of PbX_2 (X = Cl, Br, I), trioetylphosphine (TOP) and HX (X = Cl, Br, I). These chemicals are toxic and environmentally-hazardous. To solve this problem, we report the synthesis of $CsPbX_3$ (X = Cl, Br, I or mixed halides) NCs through modified anion exchange reactions. Using two kinds of Sn-based halide precursors as the anion-exchange sources, we can finely tune the chemical compositions and optical properties of pre-synthesized $CsPbX_3$ NCs. Considering no PbX_2, TOP and HX have been used in the preparation of the Sn-based halide precursors, we can determine this two-pot synthetic scheme as a step toward a green-chemistry approach for synthesizing high quality $CsPbX_3$ NCs.

1. Introduction

In the last several years, the organic-inorganic lead halide ($CH_3NH_3PbX_3$, X = Cl, Br, I) perovskite materials have been demonstrated to be amazing semiconductors with an record high power conversation efficiencies for solar cells [1–5]. By preparing mixed-halide perovskites [6,7] or tuning the average crystallite size [8], the photoluminescence (PL) spectra can be tuned over nearly the entire visible spectral region. However, these organic-inorganic lead halide perovskites are extremely unstable when exposed to the environment of oxygen and moisture. Compared to hybrid organic-inorganic lead halide perovskites, the cesium lead halide all-inorganic perovskites ($CsPbX_3$, X = Cl, Br, I) are much more stable [9]. $CsPbX_3$ NCs have many excellent properties such as high optoelectronic quality, small exciton binding energy, high thermal stability and balanced electron and hole mobility lifetime [6,9–11]. The PL spectra of the $CsPbX_3$ NCs can also be tuned over nearly the entire visible spectral region through the same way of hybrid organic-inorganic lead halide perovskites [12–16]. Therefore, the $CsPbX_3$ NCs have much great potentials in applications, including high-efficiency photovoltaic cells, light-emitting-diodes, lasers and photodetectors [17–26]. Recent studies of the $CsPbX_3$ NCs have been made mainly in synthesizing high-quality perovskite nanocrystals with well-defined morphologies and components [27–34].

Anion exchange reactions have been proven to be a powerful method to tune the optical properties of $CsPbX_3$ NCs [35–37]. Kovalenko et al. reported the fast and low-temperature anion exchange reactions in highly luminescent $CsPbX_3$ (X = Cl, Br, I) NCs for the first time [12]. As-prepared $CsPbX_3$ NCs exhibit a wide color gamut and possess uniform diameters. Yang et al. synthesized composition tunable and highly luminescent $CsPbX_3$ (X = Cl, I) nanowires through anion-exchange reactions [16]. Their anion exchange routes solve the problems of directly synthesized $CsPbX_3$ (X = Cl, I) nanowires, such as low nanowire yield, poor diameter control and undesirable optical properties. However, these reactions were applied to $CsPbX_3$ NCs by reacting with certain amounts of Pb-based halide precursors [12] or ODA-X, OLAM-X, TBA-X [38], TOA-X halide precursors [39]. The preparation of these halide precursors would use large amounts of PbX_2 (X = Cl, Br, I) salts, trioetylphosphine (TOP) and HX (X = Cl, Br, I). As these chemicals are toxic, it's not environmentally-friendly to use them in the anion exchange reactions. And, the research into the preparation of environmentally-friendly halide precursors is still rare. It remains a challenge to choose the appropriate environmentally-friendly halide precursors for the anion exchange reactions in $CsPbX_3$ NCs.

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To overcome the disadvantages of the halide precursors mentioned above, here in this report, for the first time, we introduce a facile anion exchange approach using Sn-based halide precursors to achieve the purpose of green chemistry. In our experiments, we found that the preparation of the Sn-based halide precursors does not need to use toxic chemicals such as PbX_2 salts, TOP and HX. Anion exchange reactions were observed to be very fast and could be completed in several seconds at room temperature. The PL spectra of the resulting NCs could be controlled to cover the entire visible spectra region (410–681 nm) by changing the amounts of added Sn-based halide precursors. The anion exchange reactions do not change the cubic perovskite structure, even the size and shape of the original $CsPbX_3$ NCs are preserved. In addition, the exchanged $CsPbX_3$ ($CsPbCl_3$ and $CsPbI_3$) NCs still hold the ability to be used as anion-exchange sources, by simply mixing them with $CsPbBr_3$ NCs together. The synthesis routes of the previous researchers are totally optimized to conform to the environmentally-friendly concept with a product of similar quality to that of the original synthesis. Based on our research, we can predict that some other metal halogenide such as MgX_2, ZnX_2, MnX_2, etc. can also be used to prepare lead-free halide precursors for the anion exchange reactions in $CsPbX_3$ NCs.

2. Experimental section

2.1. Materials

Cesium carbonate (Cs_2CO_3, 99%), lead bromide ($PbBr_2$), 1-octadecene (ODE, ≥90%), oleic acid (OA, 90%), oleylamine (OAm, 80–90%) and cyclohexane (CYH, ≥99%) were purchased from Aladdin. $SnCl_2$ (97.5%, anhydrous), $SnBr_2$ (99%) and SnI_2 (99%) were purchased from Stream Chemicals. All chemical reagents were used directly without any further purification.

2.2. Preparation of Cs-oleate

0.2666 g of Cs_2CO_3 was mixed with 10 mL of ODE and 0.835 mL of OA in a 100 mL 3-neck flask. The mixture was degassed and dried under vacuum at 100 °C for an hour to remove the water, and then heated to 150 °C under Ar flow for half an hour until all Cs_2CO_3 reacted with OA.

2.3. Synthesis of $CsPbBr_3$ NCs

10 mL of octadecene (ODE), 1 mL of OAm, 1 mL of OA, and 0.1321 g of $PbBr_2$ were loaded into a 100 mL three-neck flask and dried under vacuum at 120 °C for 30 min. After complete solubilization of $PbBr_2$ salt, the temperature was increased to 170 °C under Ar flow and 1 mL of Cs-oleate solution (prepared as described above) was injected. After 10 s, the reaction mixture was cooled to room temperature using a water bath. The NCs were isolated by centrifugation at 7000 rpm for 5 min. After centrifugation, the supernatant was discarded and the precipitate was dispersed in cyclohexane and centrifuged again. The obtained precipitate was re-dispersed in cyclohexane forming colloidally solutions for further use in anion-exchange reactions. Considering cyclohexane is less toxic than the hexane and toluene used by other researchers, we choose it as the solvent for the perovskite NCs in our search. We also find that the cyclohexane is a good solvent for the perovskite NCs.

2.4. Anion-exchange reactions

Exchange reactions were performed under air-free conditions. SnX_2 ($SnCl_2$, $SnBr_2$ or SnI_2) as the anion exchange source was mixed with 5 mL of ODE in a 3-neck flask and kept under vacuum at 100 °C for 20 min. Certain amount of surfactants (Table 1) were injected at 100 °C under Ar flow. After complete dissolution of the anion source, the temperature was lowered to room temperature and $CsPbX_3$ NCs dispersed in cyclohexane were injected to initiate the anion-exchange reactions. After reaction, the NCs were isolated by centrifugation at 7000 rpm for 5mins. After centrifugation, the supernatant was discarded and the precipitate was dispersed in cyclohexane and centrifuged again. The obtained precipitate was re-dispersed in cyclohexane forming colloidally solutions for further analysis.

2.5. Characterization

X-ray diffraction (XRD) patterns were measured using a BRUKER D8 ADVANCE X-ray diffractometer with a CuKα source. Transmission electron microscope (TEM) images were obtained on a JEOL-JEM 1200 TEM at an accelerating voltage of 100 kV. High-resolution TEM (HRTEM) images were taken with a FEI Tecnai TEM at an accelerating voltage of 200 kV. Photoluminescence (PL) spectra were collected using an Edinburgh FLS 980 fluorescence spectrophotometer. Ultraviolet and visible absorption (UV–vis) spectra were performed by a Shimadzu UV-2550 ultraviolet–visible spectrometer with an integrating sphere.

3. Results and discussion

Fig. 1 shows the XRD patterns of the parent $CsPbBr_3$ NCs and the anion-exchanged products. By direct synthesis at 170 °C, $CsPbBr_3$ NCs are formed in the cubic phase (space group *Pm*3*m*, ICSD 29073). The patterns of the almost fully exchanged NCs correspond to those recorded on directly synthesized $CsPbCl_3$ (space group *Pm*3*m*, ICSD 29072) and $CsPbI_3$ (space group *Pm*3*m*, ICSD 181288) NCs. The anion exchanges do not affect the cubic perovskite crystal structure as a result of the rigidity of the cationic framework. So the XRD patterns of the partially exchanged NCs still show the cubic phase. We can also observe that there is only a slight shift in the XRD patterns after the anion exchange reactions. Because, the combination of Cl^- ions leads to the shrink of the cell and all the peaks move to higher angles, while the combination of I^- ions expands the cell and all the peaks move to lower angles. The results are consistent with the anion exchange reactions using Pb-based halide precursors or oleylammonium halide precursors.

As shown in Fig. 2(a), $CsPbBr_3$ NC has a cubic structure, which is based on the corner-shared $PbBr_6^{4-}$ octahedra locating in the center of Cs^+ formed framework. In the $CsPbBr_3$ structure, the $PbBr_6^{4-}$ octahedra are binding together by sharing their Br^- ions and the Cs^+ ion is owned by eight Cs^+ formed frameworks at the same time. This kind of structure leads to the rigidity of the cubic framework and the basis of the anion exchange nature. The lattice distance between the Cs^+ ions is 5.82 Å, which is verified by the HRTEM image of a single $CsPbBr_3$ NC (Fig. 2b). This lattice distance corresponds to the (1 0 0) plane set in the XRD pattern. Based on the results of the analysis of the XRD patterns, this plane set should

Table 1
Reaction parameters of the anion-exchange reaction. All anion-exchange reactions were conducted at room temperature for several seconds. X = Cl, Br, I.

Starting NCs (mmol)	Halide Precursor (mmol)	OA (mL)	OAm (mL)
0.02	SnX_2 – 0.006	0.2	0.2
0.02	SnX_2 – 0.012	0.4	0.4
0.02	SnX_2 – 0.018	0.6	0.6
0.02	SnX_2 – 0.024	0.8	0.8
0.02	SnX_2 – 0.030	0.8	0.8

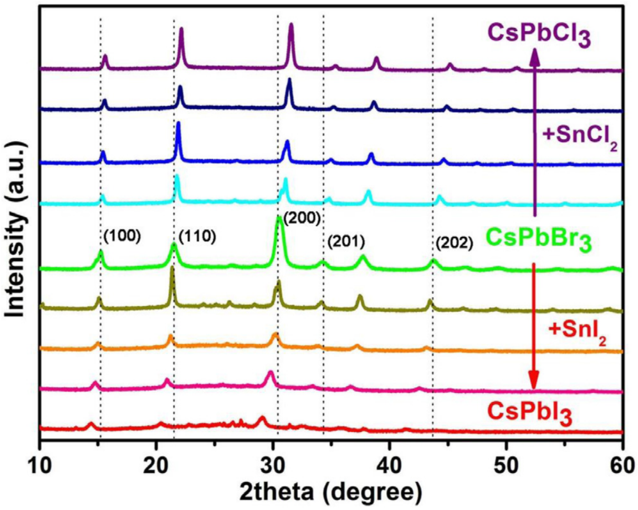

Fig. 1. XRD patterns of the parent $CsPbBr_3$ NCs and the anion-exchanged products (using $SnCl_2$ or SnI_2 as halide sources).

shrink with the combination of Cl^- ions and expand with the combination of I^- ions. So we can observe that the lattice distance of this plane set shrink to 5.69 Å in the HRTEM of Cl^- ion exchanged samples (Fig. 2c) and expand to 5.94 Å in the HRTEM of I^- ion exchanged samples (Fig. 2d). The HRTEM images of the Cl^- and I^- ion exchanged samples also indicate that the high crystallinity of the parent $CsPbBr_3$ NC is preserved during the anion exchange reactions. We can also deduce from the XRD patterns and the HRTEM images that the cation exchange reaction between Pb^{2+} and Sn^{2+} ions is not obvious. In the previous research, Ward van der Stam and his coworkers have reported a method for the partial cation exchange of Pb^{2+}ions (with Sn^{2+} ions) in colloidal $CsPbBr_3$ NCs [40]. However, their partial cation exchange reactions would cost about 16 h, which is much longer than the several seconds of the anion exchange reactions in our research. So there is likelihood of extremely small amount of Sn^{2+} ions to be incorporated into the NCs during the anion exchange reactions in our research and their effect can be neglected.

The nanostructures of the as-prepared samples were obtained with TEM. TEM images in Fig. 3 show that the shape of the parent NCs is preserved in the course of the anion-exchange reactions with the Sn-based precursors. We can also observe that the size of the NCs changed a little in the course of the anion-exchange reactions, whereas the reaction with $SnCl_2$ leads to a slight decrease in size and the exchange with SnI_2 leads to a slight increase in size. This phenomenon is also caused by the shrinking and expanding of the structure cell during the anion exchange reactions.

The anion exchange reactions of $CsPbBr_3$ NCs with different amounts of Sn-based halide precursors were monitored by measuring UV–vis absorption and PL spectra (Fig. 4a, b). The parent $CsPbBr_3$ NCs have an emission peak at 520 nm, which gradually shifted to blue with increasing amounts of $SnCl_2$ added as exchanging halide source and reached a final value of 410 nm with FWHM peak width of 12 nm. When the $SnCl_2$ was replaced with SnI_2, the PL peaks of $CsPbBr_3$ NCs red shifted to longer wavelengths and

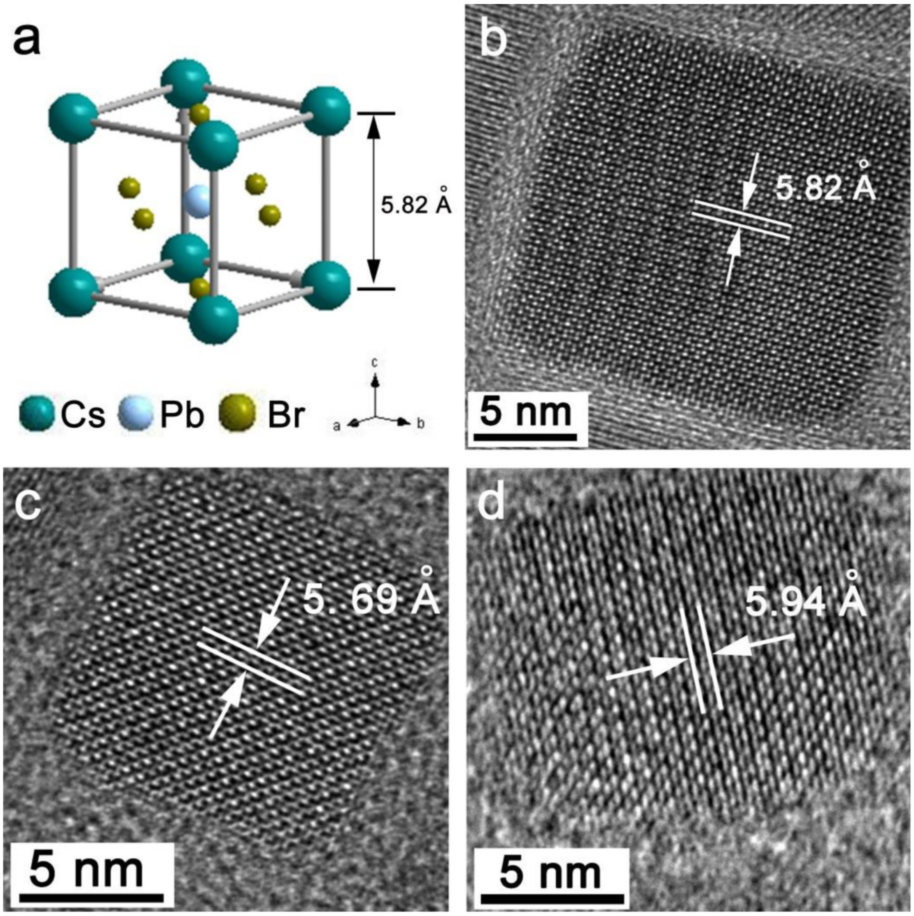

Fig. 2. (a) Crystal structure of $CsPbBr_3$. (b) HRTEM image of a single $CsPbBr_3$ NC. (c) HRTEM image of Cl^- and (d) I^- exchange NC.

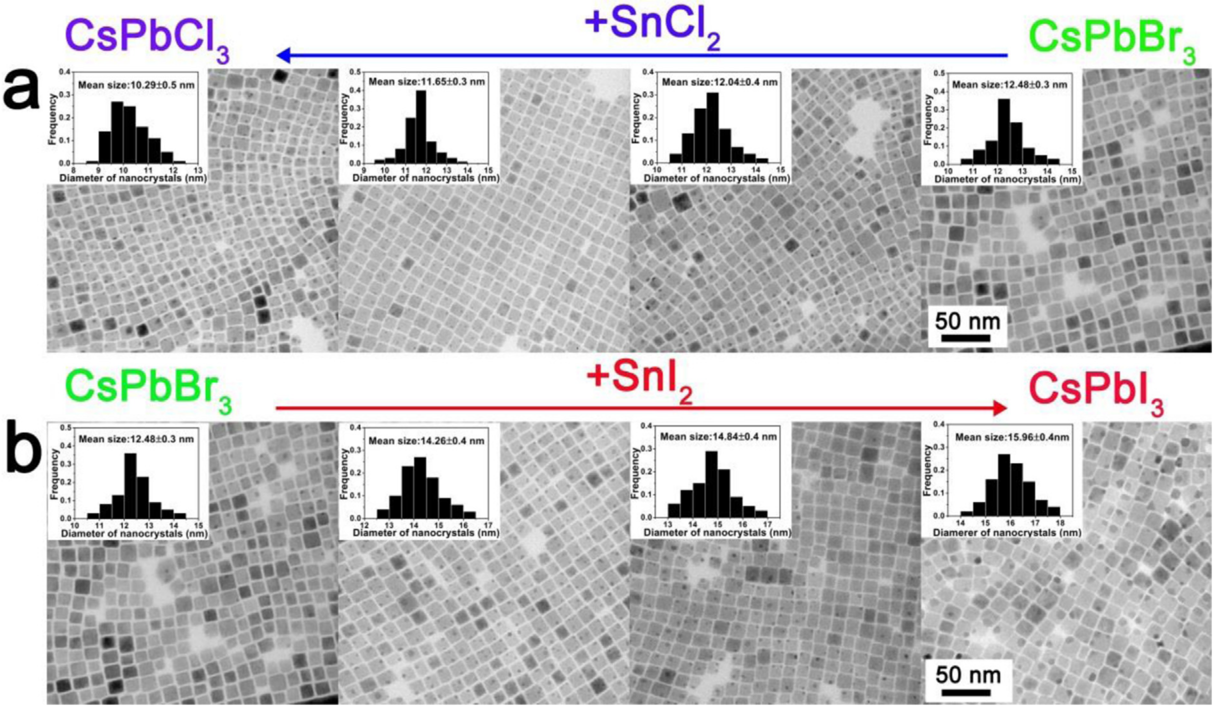

Fig. 3. Transmission electron microscope (TEM) images of $CsPbX_3$ NCs after treatment with various quantities of (a) $SnCl_2$ precursors and (b) SnI_2 precursors.

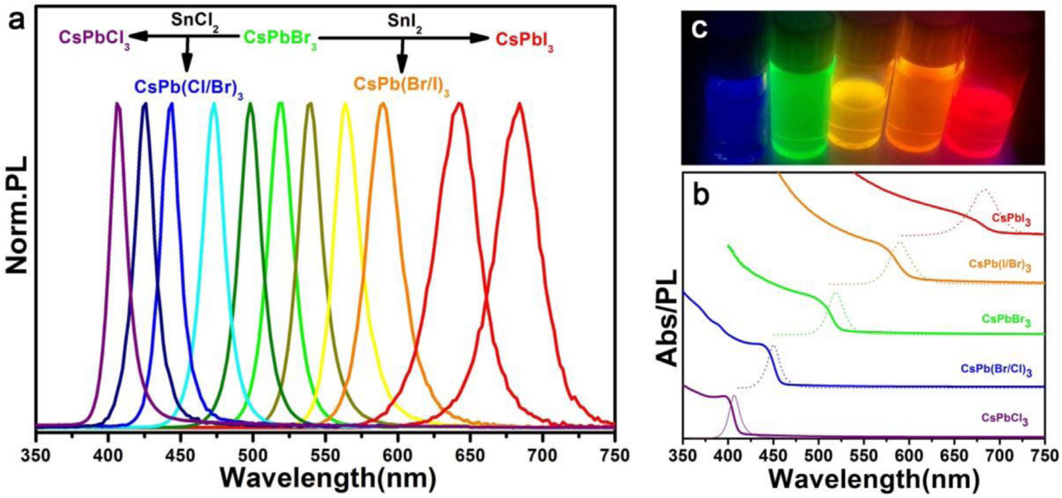

Fig. 4. (a) Photoluminescence spectra of $CsPbBr_3$ NCs exchanged with calculated amounts of $SnCl_2$ and SnI_2 precursors (b) Evolution of the UV–vis absorption (dashed lines) and PL (solid lines) spectra of the anion-exchanged NCs (c) Evolution of emission colors of specific anion-exchanged NCs (under a UV lamp, λ = 365 nm).

eventually reached about 680 nm with FWHM peak width of 40 nm (Fig. 4a). The PL spectra of the exchanged NCs are Stokes-shifted in regard to the UV–vis absorption spectra (Fig. 4b). The corresponding NCs dispersed in cyclohexane can emit light in the range from violet to purple to red under 365 nm light excitation as shown in Fig. 4c. Besides the tunable color, their high quantum yields are preserved in the anion-exchange reactions with a value of 10% for $CsPbCl_3$ and 80% for $CsPbI_3$.

We find that the NCs prepared by the anion-exchange reactions with Sn-based halide precursors can also be used as halide sources. The anion exchange reactions can be initiated by mixing $CsPbBr_3$ and $CsPbCl_3$ NCs or $CsPbBr_3$ and $CsPbI_3$ NCs, forming $CsPb(Cl:Br)_3$ or $CsPb(Br:I)_3$ NCs. The process took several minutes before the anion exchange between the NCs reached a steady state, as the ions migrate from one NC to another is a much more difficult process. The UV–vis absorption and PL spectra of the generated NCs are intermediate between those of the parent NCs (Fig. 5a). The PL spectra of such exchanged NCs are also Stokes-shifted with respect to the UV–vis absorption spectra and have only one peak. After the mixing with $CsPbBr_3$ NCs, the PL peak of $CsPbCl_3$ NCs shifted to longer wavelengths, and the PL peak of $CsPbI_3$ NCs shifted to shorter wavelengths. These kinds of anion-exchange reactions can be verified by the XRD patterns (Fig. 5d). The XRD patterns of the generated samples indicate that the halide ions are well dispersed in these NCs. What' more, the TEM images of the size and shape of the NCs are almost the same after the mixing (Fig. 5b, c). This means that these kinds of anion-exchange reactions do not affect the size and shape of the NCs.

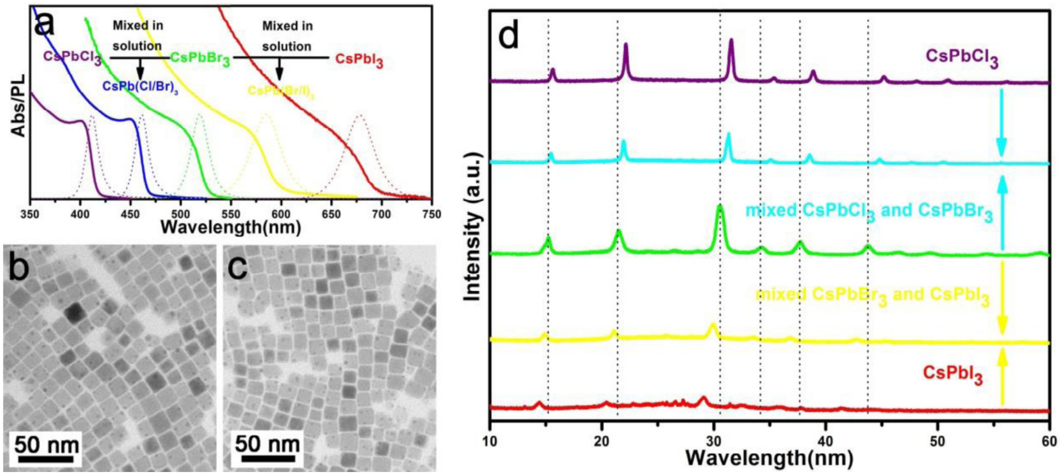

Fig. 5. (a) UV–vis absorption and PL spectra of samples synthesized by mixing $CsPbBr_3$ NCs with $CsPbCl_3$ or $CdPbI_3$ NCs (b) and (c) TEM images of $CsPb(Cl:Br)_3$ and $CsPb(Br:I)_3$ NCs. (d) XRD patterns of the parent $CsPbCl_3$, $CsPbBr_3$ and $CsPbI_3$ NCs and of the samples after mixing. Pattern for orthorhombic $CsPbBr_3$ with ICSD no. 97851 is provided for reference.

4. Conclusion

In conclusion, we have synthesized a range of $CsPbX_3$ perovskite NCs via environmentally-friendly anion-exchange reactions. In the preparation of the halide precursors, no toxic chemicals are accessible. The PL of the exchanged NCs can be tuned to cover nearly the whole visible region by tuning the amounts of Sn-based halide precursors. The shape and size of the parent $CsPbX_3$ NCs are preserved during the anion-exchange process. We have also found that the exchanged $CsPbX_3$ ($CsPbCl_3$ and $CsPbI_3$) NCs still hold the ability to be used as anion-exchange sources. Therefore, the synthesis routes of the previous researchers are totally optimized to conform to the environmentally-friendly concept with a product of similar quality to that of the original synthesis. Based on our research, we can predict that some other metal halogenide such as MgX_2, ZnX_2, MnX_2, etc. can also be used to prepare lead-free halide precursors for the anion exchange reactions in $CsPbX_3$ NCs.

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