Low-Temperature Molten Salts Synthesis: CsPbBr₃ Nanocrystals with High Photoluminescence Emission Buried in Mesoporous SiO₂

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ABSTRACT: Using mesoporous SiO₂ to encapsulate CsPbBr₃ nanocrystals is one of the best strategies to exploit such materials in devices. However, the CsPbBr₃/SiO₂ composites produced so far do not exhibit strong photoluminescence emission and, simultaneously, high stability against heat and water. We demonstrate a molten-salts-based approach delivering CsPbBr₃/mesoporous-SiO₂ composites with high PLQY (89 ± 10%) and high stability against heat, water, and aqua regia. The molten salts enable the formation of perovskite nanocrystals and other inorganic salts (KNO₃−NaNO₃−KBr) inside silica and the sealing of SiO₂ pores at temperatures as low as 350 °C, representing an important technological advancement (analogous sealing was observed only above 700 °C in previous reports). Our CsPbBr₃/mesoporous-SiO₂ composites are attractive for different applications: as a proof-of-concept, we prepared a white-light emitting diode exhibiting a correlated color temperature of 7692K. Our composites are also stable after immersion in saline water at high temperatures (a typical underground environment of oil wells), therefore holding promise as oil tracers.

Nanocrystals (NCs) of lead halide perovskites (LHPs), with the chemical formula of APbX₃ (A = CH₃NH₃, HC(NH)NH₂, Cs and X = Cl, Br, I) have optimal optical properties, which include a high photoluminescence quantum yield (PLQY) and high color purity (narrow PL emission), making them promising candidates for different optoelectronic applications such as light emitting diodes (LEDs), displays, radiation detectors, and solar concentrators. However, the effective implementation of these NCs in industrial manufacturing processes is limited by their poor stability, which leads to their degradation when they are exposed to humidity, high temperature, and photoirradiation. On the other hand, metal oxides, thanks to their robustness, have the potential to ensure both thermal and water stability to LHPs, while preserving their high PL emission.

In order to solve this issue, in recent years different strategies aimed at protecting LHP NCs have been devised, with the most promising ones being their encapsulation in polymers, inorganic matrices (including metal oxide, e.g., SiO₂, TiO₂, Al₂O₃, and metal halide) or hybrid compounds (e.g., metal–organic frameworks, MOFs). The reported LHP/polymer nanocomposites are characterized by a high PLQY and good moisture/water resistance but a weak thermal resistance. MOFs and metal halides can provide thermal and photostability, but they do not offer protection against water. On the other hand, metal oxides, among the different metal oxides, mesoporous silica (m-SiO₂) is one of the best candidates for the encapsulation of LHPs, for the following reasons: (i) it is nontoxic, earth-abundant, and cheap; (ii) it has a high chemical and thermal stability; (iii) its surface can be easily functionalized (to make...
generating a white light with Commission Internationale de
l’Éclairage (CIE) color coordinates of (0.2985, 0.3076) and a
correlated color temperature (CCT) of 7692 K. Also, our
composites retain their luminescence after being exposed to
very harsh conditions of saline water (a mixture of NaCl,
CaCl2, MgCl2, Na2SO4, and NaHCO3) and high temperature
(90 °C) for 24 h. These conditions are essentially those of
crude oil extraction wells, suggesting that our composites can
be potential candidates as tracers for the oil-extraction
industry.

In a typical synthesis, CsBr and PbBr2 (i.e., the perovskite precursors) are mixed with a ternary mixture of molten salts,
namely KNO3:NaNO3:KBr in a 10:5:5 mmol ratio, and m-
SiO2 particles, and heated up to 350 °C under air in a furnace
for 60 min (Scheme 1). The powder, obtained after cleaning
the product with dimethyl sulfoxide, features a bright PL
emission peaked at 520 nm with a full width at half-maximum
of 21.5 nm (99.18 meV) and a PLQY as high as 89 ± 10%,
obtained by measuring the sample dispersed in DI water
(Figure 1a and 2). The X-ray diffraction (XRD) pattern of the
sample is characterized by the presence of peaks ascribable to
the orthorhombic CsPbBr3 (ICSD 98-009-7851), KBr, NaNO3,
and KNO3 phases (Figure 1d). A broad peak ranging from
15° to 35° is also present in the XRD pattern and is
ascribed to the amorphous SiO2 matrix (Figure S1a of the
Supporting Information). Given the high solubility in polar
solvents of all the inorganic salts employed here, the residual
salts detected by XRD analysis must have been encapsulated
in the pores of m-SiO2 particles together with the LHP NCs.

To reveal the morphology and the structure of our CsPbBr3/
m-SiO2 composites, we performed an in-depth transmission
electron microscopy (TEM) analysis. The starting m-SiO2
particles have a mean size of 0.6 μm, as emerged from our DLS
measurements (Figure S2) and are characterized by pores
arranged in a hexagonal framework, typical of MCM-41 silica,
with the pores having a mean diameter of 3.3 nm (Figure S1b).
Such mesoporous structure is only partially retained in the final
composites whose TEM appearance indicates that the
nanoparticles had grown inside the pores of SiO2 with the
concomitant collapse of most of the pores (Figure 1b). To gain
a deeper understanding of the nanostructure of the composites,
we performed high-resolution (HR) TEM, high-angle annular
dark-field (HAADF) scanning transmission electron microscopy
(STEM), and energy-dispersive X-ray spectroscopy (EDS) analyses.
These confirmed the partial collapse of the SiO2 mesoporous structure (Figure 1c, to be also compared with Figure 3a) and the formation of orthorhombic CsPbBr3 NCS inside the SiO2 particles (inset of Figure 1b), together with K, Na, and N-containing salts (with the K:Na ratio being close to 2:1) (Figure 1e), compatible with the XRD results.
In order to assess the stability of our composites, we exposed them to either high temperature ($180^\circ$C), to water, or to an acid + an oxidizing environment (aqua regia). Colloidal CsPbBr$_3$ NCs prepared via a standard hot injection approach were also tested in parallel.\(^1\) The thermal stability tests were carried out by monitoring the variation of the PLQY of the sample before and after annealing at $180^\circ$C for 3 h in argon atmosphere: the PLQY of our composite dropped from 89 to 85%, whereas that of colloidal CsPbBr$_3$ NCs dropped from 90% to 30% after annealing at $180^\circ$C in argon for 2 h (Figure 2a). The stability against water was assessed by dispersing and stirring the samples in DI water and monitoring the resulting PLQY over time. As shown in Figure 2b, the CsPbBr$_3$/m-SiO$_2$ composite was stable in water for 30 days with no visible drop in PL emission intensity, while the colloidal CsPbBr$_3$ NCs degraded quickly, with a complete quenching of the PL emission after only a few minutes. Most notably, our composite was stable when immersed in aqua regia for 30 days (Figure 2c). The decay in PLQY was not accompanied by any notable shift in the spectral position of the PL, not even after 70 days of immersion in aqua regia (Figure S3). Overall, these stability tests highlight the high stability of the CsPbBr$_3$/m-SiO$_2$ composite that stems from the complete embedment of the LHP NCs inside SiO$_2$. This is to be compared with previous works, in which CsPbBr$_3$ NCs had been grown inside m-SiO$_2$ without the use of molten salts and the resulting
compounds could not even sustain a washing step with water or with other polar solvents (Table 1).22−26,28

On the other hand, the stability of our samples is comparable to that observed by Zhang, et al., who prepared CsPbBr3/m-SiO2 composites via a solid state reaction in which CsBr, PbBr2, and m-SiO2 were annealed together at high temperatures (Table 1).27 In their case the collapse of the mesoporous form of silica was observed only when working above 700 °C and, therefore, was attributed to high reaction temperatures, which also led to the merging of SiO2 particles. Their resulting heavily sintered/aggregated composites had a reduced PLQY (63%) that could only be moderately increased to 71% by an HF treatment. Conversely, as demonstrated by DLS measurements, our molten salts synthesis does not lead to merging or aggregation of the CsPbBr3/m-SiO2 particles (Figure S2), whose PLQY, being already very high, makes it unnecessary to perform further (and possibly hazardous) treatments.

The pore collapse and sealing in our nanocomposites can be tentatively explained by the corrosiveness of alkali salts to various metal oxides, which has been known for decades.45,46 In fact, molten salts have been even used to produce mesoporous structures starting from nonporous metal oxides,45 and, in particular, from silica.32,47−50 To better understand the role of molten salts on our final composites, we performed a series of control experiments in which we systematically varied the molten salts composition and investigated the structural and optical properties of the corresponding products. When the synthesis was performed with KBr and KNO3, the product, consisting of m-SiO2 particles whose pores are filled with LHP NCs and KNO3 (Figure 3a,c), exhibited a high PLQY (89 ± 10%) and a low resistance against water and aqua regia (Figure 2a,b).51 Interestingly, this procedure did not affect the mesoporous structure of m-SiO2 which was completely retained in the composite (Figure 3a). Conversely, the use of NaN03 and KBr yielded composites having a low PLQY (42 ± 10%) and a high resistance against water and acid treatment (Figure 2a,b and Figure S4). The XRD and TEM analyses revealed that the product consisted of m-SiO2 particles filled with CsPbBr3 NCs and NaN03, which had partially lost their mesoporous structure (Figure 3b, d).

Overall, these control experiments indicate that the composition of the molten salts mixture employed has a profound impact on the structure of the final composites: (i) the use of NaN03 is responsible for the partial collapse of the porous structure of the m-SiO2 particles as this salt is probably more corrosive than KNO3 and KBr toward silica; (ii) the presence of KNO3 inside SiO2 leads to an optimal PL emission of CsPbBr3 NCs, for reasons that are unclear at present. These observations suggest that a ternary mixture of KNO3, KBr, and NaN03 is therefore essential to achieve both high PLQY and high stability, as experimentally observed by us. In another series of control experiments, in which we employed the ternary KNO3−KBr−NaN03 molten salts mixture and then systematically varied their relative composition, we also observed that the resulting emitting composites were stable in aqua regia (hence the LHP NCs were completely embedded inside the SiO2 particles) only when working with KNO3:NaN03:KBr ratios of 10:5:5, 8:7:5, 7:8:5, and 5:10:5 (Table S1 and Figure S4), with the final ratio also maximizing the PL emission of the product. These results indicate that both the composition and the stoichiometry of the molten salts

Table 1. Comparison of the Different LHP/m-SiO2 Composites Reported in the Literature

| ref           | reaction temperature | use of solvents | PLQY (%) | stability                                                                 |
|---------------|----------------------|-----------------|----------|---------------------------------------------------------------------------|
| Chen et al.   | 180 °C               | yes             | 68%      | Degradation and change in color after 15 min                               |
| Zhang et al.  | 700 °C               | no              | 63%      | ~100% PL retention after 50 days                                          |
| Wang et al.   | RT                   | yes             | ≤55%     | N/A                                                                       |
| Dirin et al.  | 150 °C               | yes             | 48%      | N/A                                                                       |
| Malgras et al.| 95 °C                | yes             | ≤5.5%    | N/A                                                                       |
| this work     | 350 °C               | no              | 90%      | ~95% PL retention after 30 days                                           |
mixture is of paramount importance in regulating the properties of the final composites.

Motivated by the optimal properties of our composites, we tested them in down converting LED (both on-chip and remote applications were tested). We also performed preliminary tests under conditions that are typical for oil tracing in the crude oil extraction industry, the latter requiring stability under high salinity conditions. For the fabrication of a white LED (on-chip application), a blue emitting LED (3 W, 3.2−3.4 V and wavelength: 445−450 nm) was covered by a mixture of our CsPbBr3/m-SiO2 composite (green emitting), K2SiF6:Mn (red emitting) powder, and TiO2 (light scattering agent) dispersed in poly(dimethylsiloxane). The white light emitted by the final device had CIE color coordinates of (0.2985, 0.3076) and a correlated color temperature (CCT) of 7692 K (Figure 4a,b). Being characterized by three distinct narrow emission peaks of blue, green, and red colors (Figure 4d), such white LED is promising as a light source in LCD applications. Indeed, commercially available green emissive phosphors have a very broad emission spectrum limiting the color gamut of displays.

Figure 4. (a) Emission spectrum of the fabricated W-LED (insets: photograph of the W-LED under operation) and the corresponding (b) CIE1931 color coordinate diagram. (c) Time-dependent normalized PL intensity of CsPbBr3/m-SiO2 polymer composites film and standard CsPbBr3 NCs-polymer composite film under high flux remote application test (inset: photograph of the (CsPbBr3/m-SiO2)-polymer film with blue LED chip (200 mW/cm2) under operation). (d) Color coverage of CsPbBr3 NCs-polymer composite film as compared to the standard Rec.2020 area.
physical properties, our composites were found to be promising as green emitting phosphors in LEDs: the resulting device emitted white light with CIE color coordinates of (0.2985, 0.3076), CCT of 7692 K and exhibited a highly stable device emitted white light with CIE color coordinates of (0.2985, 0.3076), CCT of 7692 K and exhibited a highly stable physical properties, our composites were found to be promising as green emitting phosphors in LEDs: the resulting device emitted white light with CIE color coordinates of (0.2985, 0.3076), CCT of 7692 K and exhibited a highly stable

ASSOCIATED CONTENT

Supporting Information
The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsenrgylett.1c00052.

Experimental Section, characterization of m-SiO2, DLS measurements, PL curves after the aqua regia treatment, KNO3::NaNO3:KBr molar ratio, tests of films employed for 10 days in remote configuration with a blue LED chip, test of composites exposed to brine, preparation of CsPb(Cl,Br), and CsPb(Br,I), based composites (PDF).

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Notes
The authors declare the following competing financial interest(s): M.N.A., S.P., L.M., L.S., O.B., L.D.T., and L.M. are inventors on patent application IT IT10202000018841 that covers the synthesis of nanocomposites and their properties. L.M. is a member of the Advisory Board of Quantum Solutions.

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