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Colloidal Synthesis and Ultraviolet Luminescence of Rb_2AgI_3 Nanocrystals

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Abstract: Semiconductor nanocrystals (NCs) hold immense potential as luminescent materials for various optoelectronic applications. While significant progress has been made in developing NCs with outstanding optical properties in the visible range, their counterparts emitting in the ultraviolet (UV) spectrum are less developed. Rb_2AgI_3 is a promising UV-emitting material due to its large band gap and high stability. However, the optical properties of low-dimensional Rb_2AgI_3 NCs are yet to be thoroughly explored. Here, we synthesized Rb_2AgI_3 NCs via a hot injection method and investigated their properties. Remarkably, these NCs exhibit UV luminescence at 302 nm owing to self-trapped excitons. The wide-bandgap nature of Rb_2AgI_3 NCs, combined with their intrinsic UV luminescence, offers considerable potential for applications in UV photonic nanodevices. Our findings contribute to the understanding of Rb_2AgI_3 NCs and pave the way for exploiting their unique properties in advanced optoelectronic systems.

Keywords: Rb_2AgI_3 ; nanocrystals; ultraviolet luminescence; high stability



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1. Introduction

The rapid development of semiconductor light-emitting diodes (LEDs) has revolutionized lighting and display industries and has introduced a wide range of new technologies and applications [1–3]. In particular, ultraviolet (UV) LEDs have gained significant attention due to their applications in biological analysis, material processing, water purification, medical diagnosis, and computer data storage [4–10]. Traditional UV LEDs have predominantly relied on semiconductor materials with band gaps greater than 3.0 eV, such as GaN, ZnO, and SnO_2 [11–13]. GaN-based UV lasers rely on high-quality GaN grown via advanced growth techniques like metal–organic chemical vapor deposition (MOCVD) and vapor phase epitaxy [11]. However, these fabrication processes are complex and expensive due to the involvement of the epitaxial deposition of GaN on Si or sapphire substrates with intricate metal insulator semiconductor or quantum well structures [14]. They also require high-vacuum and high-temperature conditions, as well as stringent technical expertise in single-crystal synthesis [15]. Moreover, the presence of oxygen vacancies in the metal oxide lattice can introduce significant emissions from middle-gap states, leading to reduced spectral purity and efficiency [16]. Consequently, the pursuit of low-cost and high-quality UV LEDs remains a challenge.

Metal halide perovskites have emerged as a promising class of semiconductor materials, attracting significant attention in the past decade. Their versatility has led to wide-ranging applications in photoelectric detection, LEDs, and solar cells [17–20]. Recent advancements have focused on developing metal halide perovskites with unique structures, enabling tailored functional characteristics to suit specific applications [21,22]. Among these materials, silver-based metal halides, $\text{A}_x\text{AgX}_{(x+1)}$ ($\text{A} = \text{Rb}, \text{Cs}$; $\text{X} = \text{Cl}, \text{Br}, \text{I}$), have

shown immense potential as candidates for UV LEDs, thanks to their wide bandgap and exceptional stability [23]. Through the component tuning of $A_xAgX_{(x+1)}$, a broad range of spectra can be achieved, presenting exciting possibilities for the design of UV LEDs [24,25]. Notably, extensive research has demonstrated complete spectral tuning in the visible range via component adjustments [26]. However, in comparison to bulk materials, the study of Rb_2AgI_3 nanocrystals (NCs) is still in its early stage, particularly regarding their optical properties and how they differ from those of their corresponding bulk counterparts [23].

Here, we successfully synthesized phase-pure Rb_2AgI_3 NCs using a hot injection method. Their optical properties were studied via photoluminescence (PL), PL excitation (PLE), UV-vis absorption, transmission electron microscopy (TEM), X-ray diffraction (XRD), and XPS characterizations. The 1D crystal structure and soft crystal lattice of Rb_2AgI_3 facilitate the formation of self-trapped excitons, which generates luminescence at 302 nm with a large Stokes shift of 32 nm. These wide-band-gap Rb_2AgI_3 NCs with intrinsic UV luminescence have potential applications in short-wavelength photonic nanodevices. This research represents a crucial step towards harnessing the full potential of metal halide perovskite NCs for UV LEDs and advancing the understanding of their optical properties.

2. Materials and Methods

2.1. Materials

Rubidium acetate ($Rb(ac)$, 99.8%) was purchased from Aladdin. Silver acetate ($Ag(ac)$, 99.5%) was purchased from Macklin. 1-octadecene (ODE, 90%), oleylamine (OLA, 70%), and oleic acid (OA, 90%) were purchased from Sigma-Aldrich. All chemicals and solvents were used without further purification.

2.2. Synthesis of Rb_2AgI_3 NCs

OLA-I was synthesized by mixing 1.26 mL hydrogen iodide (HI), 13 mL octadecene (ODE), and 10 mL OLA in a 50-mL 3-neck flask. The mixture was degassed under nitrogen gas flow at 100 °C for 30 min to completely dissolve all powders. Then, 0.0867 g of CH_3COORb , 0.0501 g of CH_3COOAg , 1 mL of OLA, 1 mL of oleic acid, and 13 mL of ODE were added into a 50 mL 3-neck flask, and the mixture was degassed under nitrogen gas flow at 70 °C for 60 min. Then, 1.8 mL OLA-I solution was injected to obtain the Rb_2AgI_3 NCs. After 5 s, the mixture was cooled in an ice-water bath. The solution was centrifuged at 8000 rpm for 4 min, and then the precipitate was dispersed in 8 mL hexane. After a second centrifugation at 7000 rpm for 4 min, the supernatant was collected for characterization.

2.3. Characterizations

PL spectra and PLE spectra were measured at room temperature using an Edinburgh Integrated Fluorescence Spectrometer FS5 with a Xe lamp as the light source. TEM images were collected using an FEI Talos F200X TEM operating at 200 kV. UV-Vis absorption spectra were recorded via a UV-Vis spectrophotometer (UV-3600). XRD was performed using a Smartlab X-ray diffractometer. XPS was performed on a QUANTERA-II spectrometer equipped with a monochromatic Al K α source. The Fourier transform infrared spectroscopy (FT-IR) was tested in a Nicolet iS50 spectrometer. Thermogravimetric analysis (TGA) was tested using a TGA/DSC³⁺ STAR^e system with temperature range of 30–800 °C and heating rate of 15°/min in nitrogen gas. All characterizations were tested at room temperature except TGA. Dry samples were used in XRD, XPS, and TGA, and purified NCs solutions were tested in all other characterizations.

3. Results and Discussion

We synthesized the colloidal Rb_2AgI_3 NCs using the hot injection method (Figure 1). (This is specifically shown in the experimental part of the SI.) Rb_2AgI_3 NCs in the orthorhombic (space group Pbnm, no. 62) feature 1D $[AgX_4]_n^{3n+}$ chains separated by Rb^+ cations (Figure 2a,b). The anionic $[AgX_4]_n^{3n+}$ chains are made of corner-sharing $[AgX_4]^{3-}$ tetrahedra extending along the b-axis [27]. Because of the intrinsic 1D crystal structure,

Rb_2AgI_3 displays a much smaller band dispersion (especially for VBM) than that in 3D materials, for instance, CsPbX_3 ($X = \text{Cl}, \text{Br}, \text{I}$), Refs. [23,28], making excitons in Rb_2AgI_3 strongly confined to each $[\text{AgX}_4]_{\text{n}}^{3\text{n}+}$ chain with a high exciton binding energy.

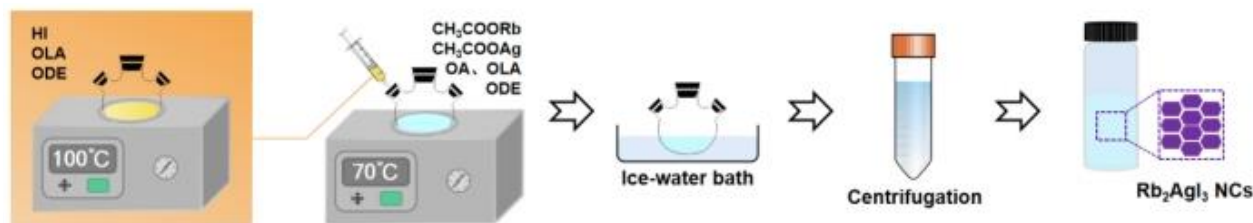


Figure 1. Schematic diagram of the preparation process of Rb_2AgI_3 NCs.

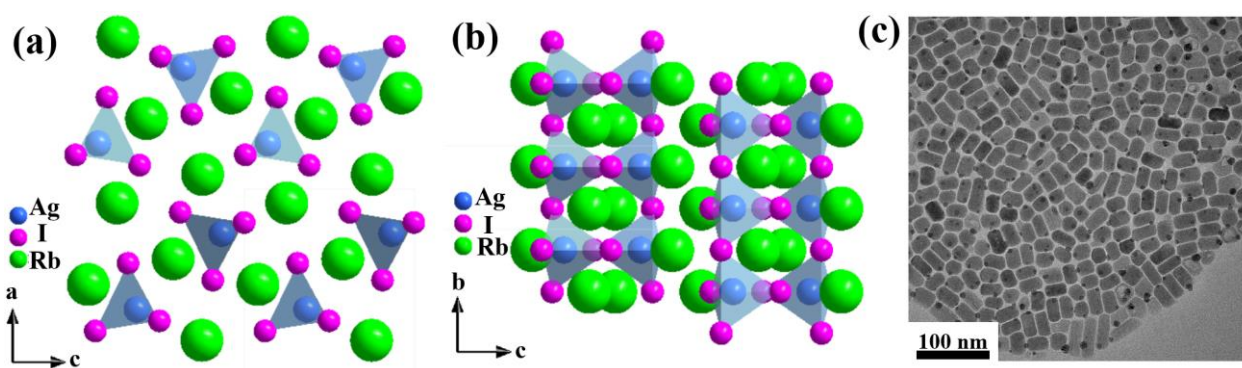


Figure 2. The crystal structure of Rb_2AgI_3 (a) along the b axis and (b) along the a axis; (c) TEM image of Rb_2AgI_3 NCs.

The TEM image (Figure 2c) reveals that the Rb_2AgI_3 NCs synthesized at 70 °C and reaction time of 5 s are in rod shape with an average size of 35.9×18.7 nm. The TEM image of a typical particle (Figure S1 in the Supporting Information) shows a clear lattice fringe, indicating high crystallinity of the particle, and the lattice spacing (0.71 nm) is consistent with that of the (2 1 0) planes of trigonal Rb_2AgI_3 .

The composition and crystal structure of the synthesized NCs were studied using X-ray diffraction (XRD) and X-ray photoelectron spectroscopy (XPS) (Figure 3). The complete XPS spectrum (Figure 3a) contains clear Rb, Ag, and I signals. The high-resolution XPS spectra (Figure 3b–d) further reveal the fine signals of Rb 3d_{3/2}, Rb 3d_{5/2}, Ag 3d_{3/2}, Ag 3d_{5/2}, I 3d_{3/2}, and I 3d_{5/2}. The electronic binding energies were offset-corrected with the C1s electronic binding energy (284.8 eV) of carbon contamination on the surface. In the Rb 3d spectrum, peaks of 110.5 eV and 109.0 eV can be assigned to Rb 3d_{3/2} and Rb 3d_{5/2}, respectively. Peaks of 373.8 eV and 367.7 eV in the Ag 3d spectrum can be assigned to Ag 3d_{3/2} and Ag 3d_{5/2}, respectively. In the I 3d spectrum, the binding energies of I 3d_{3/2} and I 3d_{5/2} are located at 630.2 eV and 618.7 eV, respectively. These characterizations definitely demonstrate that the synthesized NCs are well-crystallized Rb_2AgI_3 NCs. In addition, through the FTIR characterization results (Figure S2), it can be seen that the surface of Rb_2AgI_3 NCs bonds oleic acid and oleylamine organic ligands, which may promote their good dispersion in organic solvents. The XRD spectrum (Figure 3e) comprises a series of peaks that agree well with those of standard Rb_2AgI_3 data.

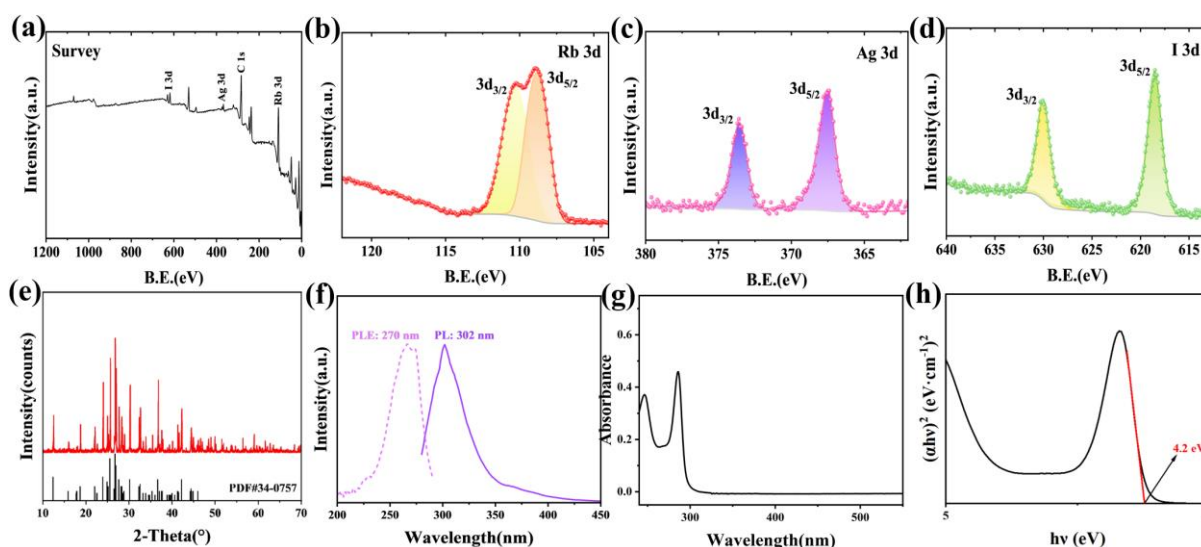


Figure 3. (a) XPS survey spectrum of Rb_2AgI_3 crystal; XPS spectrum of (b) rubidium, (c) silver, and (d) iodine in Rb_2AgI_3 NCs. (e) XRD pattern of Rb_2AgI_3 NCs. (f) The photoluminescence (PL) spectrum of Rb_2AgI_3 excited by 270 nm light and photoluminescence excitation (PLE) spectra measured at 302 nm; (g) UV–Vis pattern and (h) Tauc curve of Rb_2AgI_3 NCs.

The Rb_2AgI_3 NCs in hexane were further studied via PL and photoluminescence excitation (PLE) spectroscopies. The PL peak of Rb_2AgI_3 NCs is located at 302 nm (excitation: 270 nm) with a full width at half maximum (FWHM) of 38 nm and a Stokes shift of 32 nm. Typically, low-dimensional (1D and 0D) metal halide materials emit strong Stokes-shifted PL owing to self-trapped excitons (Figure S3). The PL results we observed on Rb_2AgI_3 NCs were markedly different for several reasons. First, Rb_2AgI_3 NCs are one of the few UV emitters with near-UV excitation and a relatively small Stokes shift (32 nm), which is advantageous, considering the small energy loss between the excitation and emission photons. From the UV-Vis spectrum, the direct bandgap of Rb_2AgI_3 NCs is calculated as 4.2 ± 0.1 eV (Figure 3h) based on the Tauc method according to the following equation:

$$(\alpha h\nu)^{1/n} = B(h\nu - E_g)$$

where α is the absorbance coefficient, h is Planck's constant, ν is the frequency of photons, B is a constant, and E_g is the semiconductor optical bandgap. The index n is related to the direct ($n = 1/2$) or indirect ($n = 2$) nature of the semiconductor. Therefore, by plotting $(\alpha h\nu)^{1/n}$ as a function of $h\nu$, the E_g can be determined by the intercept on the x -axis. Under different excitation wavelengths, the normalized PL spectra show the same characteristics in Figure S4a, while the normalized PLE spectra at different PL wavelengths from 290 nm to 320 nm show the same shape in Figure S4b. The similar PL and PLE spectra at different excitation wavelengths reveal that the UV emission of the Rb_2AgI_3 NCs is attributed to the relaxation of the same excited state rather than ionic luminescence or defective luminescence.

Controlling the synthesis conditions can adjust the composition, morphology, and size of nanoparticles, thereby affecting their physicochemical properties [29–32]. In order to obtain high-quality Rb_2AgI_3 NCs, we first adjusted the injection amount of the iodine precursor solution and characterized the composition and morphology of the product based on the stoichiometry ratio of each element in the product Rb_2AgI_3 . The XRD characterization results (Figure S5) showed that when the amount of iodine precursor (1.8 mL) injected just reached the stoichiometric ratio of Rb_2AgI_3 , the product matched well with the standard diffraction peak of Rb_2AgI_3 , and no other heterogeneous diffraction peaks appeared. Increasing the injection amount of iodine precursor to 2.4 mL deviated the stoichiometric ratio of rubidium, silver, and iodine to 2:1:4 and led to the formation of RbI .

When the injection amount was further increased to 3.0 mL (the stoichiometric ratio of rubidium, silver, and iodine was 2:1:5), the RbI diffraction peaks dominated the product with minimum diffraction peaks of Rb_2AgI_3 . In addition, the TEM characterization results (Figure S6) showed that when the amount of iodine precursor injection was 1.8 mL, the Rb_2AgI_3 nanocrystals showed a rod-like morphology with a relatively low length diameter.

In addition, in order to obtain purer-phase Rb_2AgI_3 NCs with uniform morphology, we explored the effect of rubidium salt and silver salt ratio (Rb/Ag) while fixing the amount of iodine injection. As shown in Figure S7, the XRD peaks of Rb_2AgI_3 NCs grown with Rb/Ag ratios of 3:1, 2:1, and 1:1 all corresponded to the standard diffraction peaks of Rb_2AgI_3 without impurity phase, indicating that the purity of Rb_2AgI_3 NCs is not significantly affected by the Rb/Ag ratio. However, the diffraction peak of the (221) crystal plane of Rb_2AgI_3 (marked in Figure S7) was significantly enhanced with Rb/Ag = 1:1, indicating that this reaction condition may help promote the growth of the (221) crystal face. The TEM images (Figure S8) showed that the Rb/Ag ratio could affect the morphology of Rb_2AgI_3 NCs, where Rb/Ag of 1:1 generated hexagonal NCs and Rb/Ag of two or three generated rod-shaped NCs. We further studied the effect of growth temperature on the Rb_2AgI_3 NCs, as shown in Figure S9. TEM images show that 70 °C is the optimal growth temperature that gives the best uniformity of NCs. Too low or too high temperatures generally produce NCs with large size and shape distributions (Figure S10).

Surface ligands have a significant effect on the nucleation growth of nanocrystals. We studied the effects of ligands by changing the ratio of OA and OLA ligands used in the precursor solution. TEM images (Figure S11) show that the Rb_2AgI_3 NCs obtained under the three OA/OLA volume ratios (1:2, 1:1, and 3:2) all exhibit rod-like morphology, but the length-to-diameter ratios are significantly different. The length-to-diameter ratio of obtained Rb_2AgI_3 NCs gradually increases with the OA/OLA ratio. Meanwhile, the particle size distribution of NCs also increased. Compared to the NCs obtained under OA/OLA conditions of 1:2 and 3:2, those obtained under the condition of OA/OLA of 1:1 have better size uniformity.

The optical properties of Rb_2AgI_3 nanocrystalline samples obtained under different reaction conditions were explored. The UV-Vis spectra characterization results are shown in Figure S12. When iodine precursor solutions are injected at 2.4 mL and 3.0 mL, the resulting product has a sharp peak at 222 nm, which belongs to the first exciton absorption peak of RbI. The sharper absorption peak at 287 nm belongs to the Rb_2AgI_3 component. Notably, the Rb_2AgI_3 nanocrystalline sample also showed a strong and broad peak at 247 nm, which, according to existing studies on Rb_2AgI_3 , is very close to the spectral position of the impurity Ag^+ bands in KI and RbI, which are connected with optical excitations of quasimolecular complex $(\text{AgI}_4)^{3-}$.

The PL spectra of the three products were tested by adjusting the absorbance at the excitation wavelength to the same value, as shown in Figure S13. Varying the amount of iodine precursor injection, the resulting samples all showed special UV emission at 302 nm, and samples with an iodine precursor injection of 1.8 mL showed the highest emission intensity, which may be attributed to the highest phase purity of the sample at the same incident light dose. Changing the feeding ratio of rubidium salt and silver salt in the reaction raw material, the emission peak position of the product did not change significantly, all at 302 nm, but the PL peak intensity of the product was higher when the ratio of rubidium salt and silver salt was 2:1. The characterization of the product obtained by changing the oleic acid and oleamine ratio showed that the sample with an oleic acid–oleamine ratio of 1:1 had significantly enhanced fluorescence emission, which may also be due to the relatively excellent size and morphological uniformity of the sample.

The environmental stability of NCs is a very important factor for the application of devices. The XRD data of Rb_2AgI_3 NCs stored in the air for 60 days is compared with that of fresh samples in Figure 4a. The XRD peaks remained almost unchanged without any new impurity peak after storage, indicating good environmental stability of Rb_2AgI_3 NCs. In addition, thermogravimetric analysis (TGA) suggests that, unlike most hybrid

organic-inorganic lead halide perovskites, Rb_2AgI_3 shows no significant weight loss up to 645.5°C (Figure 4b), which is in agreement with the reports of improved thermal stability of all-inorganic metal halides such as $\text{Cs}_3\text{Cu}_2\text{I}_5$, $\text{Rb}_4\text{Ag}_2\text{BiBr}_9$, and Cs_2SnI_6 [33–36]. The maximum decomposition rate temperature (T_{max}) is found to be 757.0°C (Figure 4c), where the maximum decomposition rate is $0.75\%/^\circ\text{C}$. Furthermore, the presence of monovalent Ag^+ in Rb_2AgI_3 makes it less prone to degradation in ambient air. This gives it a greater advantage in practical applications than Cu(I)-based halide perovskites, which are usually susceptible to oxidative degradation.

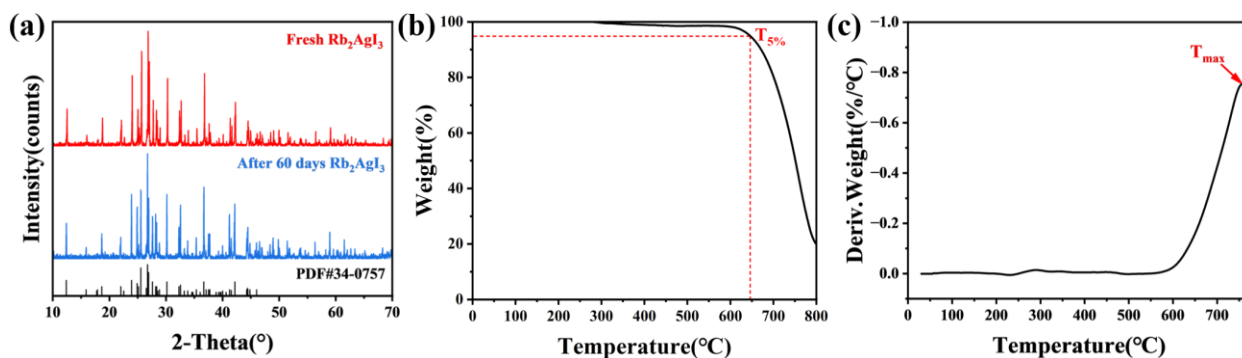


Figure 4. (a) XRD patterns of fresh Rb_2AgI_3 NCs and Rb_2AgI_3 NCs after 60 days in air; (b) TG and (c) DTG patterns of Rb_2AgI_3 NCs.

4. Conclusions

In summary, we have investigated the luminescence properties of the Rb_2AgI_3 NCs synthesized using the hot injection method. Different from bulk Rb_2AgI_3 that emits visible light, the Rb_2AgI_3 NCs exhibit pure UV luminescence with large Stokes shift originating from the self-trapped Frenkel excitons. Their optical spectra show clear evidence of the quantum confinement effect. In addition, Rb_2AgI_3 NCs are bonded with a large number of organic ligands on the surface, which makes them well dispersed in organic solvents. Considering the observation that such wide-gap Rb_2AgI_3 NCs have pure ultraviolet luminescence and high stability, we suppose that they may be applied in nanoscale optoelectronic devices such as flexible light-emitting diodes or photodetectors working in the near-UV spectral region.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/cryst13071110/s1>, Figure S1: HRTEM image of Rb_2AgI_3 NCs, Figure S2: FTIR pattern of Rb_2AgI_3 NCs. Vibrating bands at 1650 cm^{-1} and 1380 cm^{-1} can be assigned to counter-symmetric telescopic vibration and symmetrical telescopic vibration modes from oleic acid ligand $-\text{COO}^-$, respectively. Peaks around 1570 cm^{-1} indicate the bending vibration of the $-\text{NH}_2$ group N-H in oleamine. The vibration bands at 2930 cm^{-1} and 2860 cm^{-1} are attributed to the C-H bond tensile vibrations of $-\text{CH}_3$ and $-\text{CH}_2$, respectively, Figure S3: Schematic diagram of self-trapped exciton luminescence mechanism, Figure S4: Wavelength-dependent (a) PL and (b) PLE spectra of Rb_2AgI_3 NCs, Figure S5: XRD pattern of the product obtained by changing the amount of iodine precursor injection, Figure S6: (a–c) TEM image and (d–f) its particle size distribution of the product obtained by changing the amount of iodine precursor injection, Figure S7: XRD pattern of the product obtained by changing the feeding ratio of rubidium salt and silver salt in the initial solution, Figure S8: (a–c) TEM images and (d–f) its particle size distribution of the product obtained by changing the feeding ratio of rubidium salt and silver salt in the initial solution, Figure S9: (a–c) TEM images and (d–e) its particle size distribution of the product obtained by changing the reaction temperature (50°C , 70°C , 90°C), Figure S10: XRD pattern of the product obtained by changing the reaction temperature (50°C , 70°C , 90°C), Figure S11: TEM image of the Rb_2AgI_3 NCs obtained by changing the OA/OLA ratio, Figure S12: UV-Vis spectra of Rb_2AgI_3 NCs obtained under different reaction conditions, Figure S13: PL spectra of Rb_2AgI_3 NCs obtained under different reaction conditions.

Author Contributions: Conceptualization, H.L.; Methodology, Y.D.; Data curation, Y.Z.; Writing—original draft preparation, Y.D. and Y.Z.; Writing—review and editing, H.L., J.W. and F.L.; Resources, W.G., P.H. and G.J. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest: The authors declare no conflict of interest.

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