Highly luminescent MAPbI₃ perovskite quantum dots with a simple purification process via ultrasound-assisted bead milling†

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Organic–inorganic hybrid lead halide perovskite quantum dots (QDs) have various excellent optical properties, and they have drastically enhanced the field of light-emitting diode (LED) research. However, red-emissive CH₃NH₃ (MA) PbI₂ QDs have worse optical properties compared with those of green-emissive MAPbBr₃ QDs due to their instability under high-moisture and high-temperature conditions. Therefore, it is quite difficult to prepare MAPbI₃ QDs with good optical properties via bottom-up methods using conditions involving high temperature and high-solubility solvents. On the other hand, top-down methods for preparing MAPbI₃ QDs under an air atmosphere have attracted attention; however, there are issues, such as PL emission with a wide FWHM being obtained due to the wide particle-size distribution. In this research, red-emissive MAPbI₃ QDs were prepared via an ultrasound-assisted bead milling (UBM) method, and the MAPbI₃ QDs were purified using various carboxylate esters. As a result, we solved the issue of the wide particle-size distribution unique to top-down methods via purifying the MAPbI₃ QDs, and they achieved the following excellent optical properties: a FWHM of 44 to 48 nm and a PLQY of over 60%. Notably, a fabricated LED device with MAPbI₃ QDs purified using methyl acetate showed a PL peak at 738 nm and a FWHM of 49 nm, resulting in an excellent EQE value of 3.2%.

1. Introduction

Organic–inorganic hybrid lead halide perovskite (APbX₃; A = CH₃NH₃ (MA) or NH₂CHNH₂ (FA); X = Cl, Br, or I) quantum dots (QDs) have attracted attention as novel light sources for use in light-emitting diodes (LEDs) owing to their high photoluminescence quantum yields (PLQYs), narrow full-width at half-maximum (FWHM) values, and tunable emission wavelengths upon adjusting the halide component.¹⁻⁵ Notably, green-emissive MAPbBr₃ QDs exhibit narrow emission with a FWHM of approximately 25 nm, a PLQY of over 90%, and structural stability, maintaining PL emission over 3 months.⁶⁻⁸ In contrast, the optical properties of MAPbI₃ QDs with PL emission in the red and near-infrared regions are inferior to those of green-emissive MAPbBr₃ QDs. This is because MAPbI₃ QDs containing iodide at the X site are more sensitive to moisture, heat, and solvent polarity due to the weak ionic bonding between the inorganic PbI₂ framework and organic MA⁺ cations.⁹⁻¹¹ As a result of the instability of these MAPbI₃ QDs, they rapidly decompose under ambient conditions,¹² which has prevented the advancement of fundamental studies and future applications. Therefore, it is significantly important to demonstrate stable MAPbI₃ QDs that can retain the perovskite structure under air.

To date, the synthesis of highly luminescent iodide-based (red-emissive) perovskite QDs has been achieved using two bottom-up methods: the hot injection method¹³,¹⁴ and the ligand-assisted reprecipitation (LARP) method.¹⁵,¹⁶ In hot injection, a perovskite precursor is injected into a hot solution containing the other precursor, capping ligands, and a high-boiling solvent under a nitrogen atmosphere. After injection, rapid nucleation occurs and perovskite QDs are generated. However, the synthesis of perovskite QDs using hot injection generally requires a high temperature of over 100 °C, and this high temperature decomposes MAPbI₃ into PbI₂, NH₃, and CH₃I.¹⁷ On the other hand, in the LARP method, the perovskite precursors are dissolved in a polar solvent (e.g., dimethyl sulfoxide or N,N-dimethylformamide), and then the precursor solution is injected into a nonpolar solvent (e.g., toluene, or octane) to prepare colloidal perovskite QDs. The LARP method makes it easy to prepare perovskite QDs without requiring some conditions, such as high temperature and a nitrogen atmosphere. The challenge when using the LARP method to prepare iodide-based perovskite QDs is that the polar solvent used...
during synthesis strongly coordinates the PbI₂ framework, forming Pb–O bonds. As a result, a perovskite structure is not formed or the structure is destroyed rapidly after formation. As another method for overcoming the problems faced when using bottom-up processes, such as the hot injection method and LARP method, the preparation of perovskite QDs via top-down methods has attracted attention in recent years. A typical top-down method is the ball-milling method, which uses centrifugal force as the driving force. This method makes it possible to prepare red-emissive MAPbI₃ QDs without needing a high temperature of over 100 °C or polar solvent; however, there are some issues, such as a low PLQY of below 10%, a wide particle size distribution, and PL emission with a wide FWHM based on unreacted perovskite precursors. Recently, to overcome the issues associated with previous top-down methods, an ultrasound-assisted bead milling (UBM) method was developed. In UBM, a combination of ultrasound cavitation and mechanical shear force promotes the production of perovskite QDs. In detail, through ultrasound irradiation in a reaction vessel containing the perovskite precursors, zirconia beads, and capping ligands, the intensity of collisions between zirconia beads and the precursors accelerates and, finally, the particle size was minimized on the nanoscale to improve the PLQY. However, the problem of PL emission with a wide FWHM from red-emissive MAPbI₃ QDs has still not been solved, resulting in difficulties for LED applications. To achieve LEDs, the purification of MAPbI₃ QDs is critically important to remove the impurities such as unreacted perovskite precursors that cause PL emission with a wide FWHM. According to a previous report on the purification process, an ester solvent like ethyl acetate can be added into the as-synthesized perovskite QD dispersion, resulting in the division of perovskite QDs by particle size. However, structurally unstable ionic MAPbI₃ QDs are sensitive to a high dielectric constant, so the selection of a purifying solvent for purifying MAPbI₃ QDs is significantly important to achieve high purity and PL emission with a narrow FWHM.

In this work, MAPbI₃ QDs synthesized through the UBM method were purified using various carboxylate esters due to differences in the dispersibility of MAPbI₃ QDs in each solvent, and narrow emission with a FWHM of 44 to 48 nm was obtained. This is because unreacted precursors and small MAPbI₃ QDs, which were affected by quantum confinement effects, were completely removed. As a result, we synthesized highly luminescent MAPbI₃ QDs with a PLQY of 63% without using high temperature or polar solvents. MAPbI₃ QD thin films made using MAPbI₃ QDs purified with methyl acetate showed a high PLQY of 48%. An LED containing MAPbI₃ QDs purified using methyl acetate exhibited a high external quantum efficiency (EQE) of over 3%.

2. Experimental methods

2.1. Materials

Lead(II) iodide powder (PbI₂ 99.0%), oleic acid (OAc, 90.0%), and n-octylamine (OAm, 99.0%) were purchased from Aldrich. Hydroiodic acid (HI, 57 wt%), toluene (99.5%), and methyl acetate (98.0%), and butyl acetate (99.0%) were purchased from Wako Pure Chemical Industries. Propyl acetate (98.0%) was purchased from Tokyo Chemical Industry. Ethyl acetate (99.3%) was purchased from Kanto Chemical Co., Inc. YTZ® balls (ZrO₂ beads 50 μm in diameter) were purchased from Nikkato Co., Ltd. Poly(3,4-ethylenedioxythiophene):poly(styrenesulfonate) (PEDOT:PSS, A14083), poly(4-butylphenyl-diphenyl-amine) (poly-TPD), and tris-[1-phenyl-1H-benzimidazole] (TPBi) were purchased from Clevious and American Dye Source and e-Ray Optoelectronics Technology Co., Ltd. All chemicals were used without purification.

2.2. Synthesis of methylammonium halide (MAX)

Methylammonium halide salts (MAX, X = Br and I) were synthesized following a previously reported method. As an example, 7 ml of HI solution was reacted with 30 ml of methylamine solution (in methanol) in a 250 ml round-bottom flask under stirring for 2 h at 0 °C using an ice bath. Then, the solution was evaporated and the white color precipitate was collected. We purified the product via recrystallization using methanol and diethyl ether. The obtained product was dried under vacuum overnight.

2.3. Preparation of MAPbX₃ QDs

MAPbI₃ QDs were prepared via ultrasound-assisted bead milling with modifications. 92.0 mg of PbI₂ (0.20 mol), 25.5 mg of MAI (0.16 mol), and 23.5 g of zirconia beads (Φ = 50 μm) were loaded into a glass vessel. 350 μl of OAc, 25 μl of OAm, and 20 ml of toluene were added into the vessel. Then, an ultrasound homogenizer was inserted into the vessel and ultrasound irradiation was carried out for 3 h. The temperature of the solution was controlled at 0 °C with a chiller to avoid undesirable heating as a result of ultrasound. After ultrasonic irradiation, the as-synthesized MAPbI₃ QD dispersion was centrifuged at 6000 rpm for 5 min to remove ZrO₂ beads and the supernatant was collected. MAPbBr₃ and MAPb(Br/I)₃ were prepared using the corresponding halide precursors. The moles of precursors used were fixed based on the experimental conditions for MAPbI₃ QDs.

2.4. Purification process of MAPbX₃ QDs

MAPbX₃ QD powder was collected via evaporating 4 ml of QD dispersion. The QD powder was dispersed in 300 μl of carboxylate ester (methyl, ethyl, propyl, and butyl acetate) as a purifying solvent. Then, the QDs in carboxylate ester were centrifuged at 16 500 rpm for 5 min, and the obtained precipitate was dispersed in octane.

2.5 Fabrication of MAPbI₃ QD LEDs

PEDOT-PSS (55 wt% containing A14083) was spin-coated onto a cleaned indium tin oxide (ITO) substrate and annealed at 150 °C for 10 min, resulting in a 40 nm-thick layer. Poly-TPD was dissolved in chlorobenzene at a concentration of 4 mg ml⁻¹. This solution was spin-coated onto the PEDOT-PSS layer and annealed at 100 °C for 10 min. Colloidal MAPbI₃ QDs
using a Keithley 2400 source measure unit and a Minolta CS200
voltage and luminance
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precursors and small MAPbI3 QDs, a low-dielectric-constant
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added as a purifying solvent to the synthesized MAPbI3 QD
MAPbI3 QDs is shown in Fig. 1. Table 1 shows that MAPbI3 QDs
 dispersion for puri

\[ \text{active area of the device was } 2 \text{ mm}^2. \]

2.6 Characterization
X-ray diffraction (XRD) patterns of samples were obtained based
on out-plane diffraction and were measured using Rigaku Smart
Lab apparatus (using Cu Kα radiation at 45 kV and 200 mA). The
samples were observed using a JEOL JEM-2100F transmission
electron microscope (TEM) (accelerating voltage of 200 kV).
Visible absorption spectra of the samples were obtained using
a JASCO V-670 spectrophotometer (detecting wavelength range
of 400 to 900 nm). Photoluminescence (PL) spectra and photo-
luminescence quantum yields (PLQYs) of samples were ob-
lained with a JASCO FP-8000 luminescence spectrometer
(excitation wavelength of 400 nm and detection wavelength
range of 400 to 900 nm). PL lifetimes were obtained using
Hamamatsu C11367 Quantaurus-Tau apparatus. Electrolumi-
nescence (EL) spectra were recorded using a Hamamatsu
PMA-11 photonic multichannel analyser. The current–density–
voltage and luminance–voltage characteristics were measured
using a Keithley 2400 source measure unit and a Minolta CS200
luminance meter, respectively.

3. Results and discussion
Colloidal MAPbI3 QDs capped with OAc and OAm have been
successfully synthesized via an UBM method with a few modi-
fications, using MAI, PbI2, toluene, and ZrO2 beads. After
sufficient ultrasonic irradiation, a dark red suspension was
obtained. To remove impurities, including unreacted perovskite
precursors and small MAPbI3 QDs, a low-dielectric-constant
carboxylate ester (methyl, ethyl, propyl, or butyl acetate) was
added as a purifying solvent to the synthesized MAPbI3 QD
dispersion for purification. A schematic illustration of the perovskite QD synthesis and purification process for obtaining
MAPbI3 QDs is shown in Fig. 1. Table 1 shows that MAPbI3 QDs
were collected via centrifugation from all samples dispersed by
different purifying solvents after the evaporation of the solvent.
The differences in MAPbI3 QD dispersibility are attributed to
interactions between the ligands on the MAPbI3 QD surface and the
purifying solvent, such as methyl acetate.13,23,27 The MAPbI3
QD surface capped with OAc and OAm as ligands is hydrophobic,
and the MAPbI3 QDs are greatly dispersed in a solvent
with a low dielectric constant and hydrophobic groups, such as
toluene. On the other hand, upon using a carboxylate ester with
the hydrophilic –COO– group as a dispersion solvent, interactions
between the ligands on the MAPbI3 QD surface and the
carboxylate ester occurred as follows: some MAPbI3 QDs were
aggregated, and they can be easily collected via centrifugation.

As shown in Fig. 2, the PL spectrum of the prepared MAPbI3
QDs before purification shows not only a main PL emission
peak at 739 nm but also a double-shoulder PL emission peak
from 600 to 700 nm, resulting a wide FWHM of 60 nm. This
shoulder peak can be caused by emission from PbI2 nano-
crystals or from MAPbI3 QDs with a particle size of less than
6 nm, which show quantum size effects (Fig. ES1†).18,26 In
contrast, MAPbI3 QDs purified using a carboxylate ester showed
narrower PL emission, with a FWHM of 44 to 48 nm, than
before purification as a result of not having a PL shoulder peak
attributed to impurities. The narrowing of the PL spectrum was
confirmed for other types of perovskite QDs, such as MAPbBr3
QDs and MAPb(Br/I)3 QDs (Fig. ES2†). This implied that the
purification process was adaptable to other types of perovskite
QDs. However, it must be noted that the optimal purifying
solvent depends on the type of perovskite QDs. Based on UV
spectra, it was confirmed that the absorbance of the MAPbI3
QDs gradually improved upon increasing the dielectric constant
of the purifying solvent from 5.01 to 6.68 (Fig. 2b and ES3†).
This implied that upon decreasing the hydrophobicity of the
purifying solvent, the MAPbI3 QDs were more strongly aggre-
gated, resulting in the collection of more of them. In addition,
the PLQY of the MAPbI3 QDs was slightly improved from 57% to
a maximum value of 63%, which implied that PbI2 with weak PL
emission was completely removed (Table ES1†).

To investigate the MAPbI3 QD crystallinity and morphology,
XRD patterns and TEM images were obtained (Fig. 3 and 4).
From the XRD patterns, all the MAPbI3 QDs showed the
following diffraction peaks: 13.9° (110); 28.4° (220); 31.6° (114);
40.4° (321); and 42.9° (402), which were attributed to perovskite
crystal structure.18,27,28 Focusing on the XRD pattern of the
MAPbI3 QDs before purification, diffraction peaks at 12.6°,
25.9°, and 39.5° were also observed, which were attributed to
the perovskite precursor PbI2. These results clearly indicated
that unreacted PbI2 was removed from the prepared MAPbI3 QD
dispersion through this purification process. Diffraction peaks
attributed to ZrO2 beads and MAI, which is another perovskite
precursor, were not seen (Fig. ES4†). The TEM images of the
MAPbI3 QDs before purification confirmed that there are two
types of particles: cubic-shaped particles of about 10 nm and
dot-shaped ones of less than 5 nm. On the other hand, the dot-
shaped particles disappeared in all the MAPbI3 QD samples
after purification. Moreover, the average MAPbI3 QD particle
size before purification is 7.2 nm; however, after purification
using methyl, ethyl, propyl, and butyl acetate, the average sizes

![Fig. 1 A schematic illustration of the synthesis and purification processes for obtaining MAPbI3 QDs.](image-url)
are 11.3 nm, 11.1 nm, 11.0 nm, and 11.2 nm, respectively, showing an increase in the average particle size (Fig. ES5†). This indicates that the purification of MAPbI3 QDs was critical for removing the small particles. The time-resolved PL decay curves of the prepared MAPbI3 QDs are shown in Fig. 5. The average PL lifetime was determined before purification to be 32.0 ns, while after purification, the lifetimes were 46.3 ns (methyl acetate), 41.8 ns (ethyl acetate), 45.6 ns (propyl acetate), and 46.1 ns (butyl acetate), showing a drastic improvement. According to previous reports,18,29 the lifetime of MAPbI3 QDs is related to the particle size, and the PL lifetime can be determined based on this. Therefore, it is possible to easily predict that the increase in the PL lifetime was related to the removal of the small-sized particles. In addition, the PLQY was slightly improved when comparing QDs before and after purification. This suggests that the number of non-luminescent sites, which are short-lived components, decreased, resulting in an increase in the PL lifetime.30,31 From the above results, the unreacted precursors and small MAPbI3 QDs causing PL emission with a wide FWHM

**Table 1** The structures and dielectric constants of methyl, ethyl, propyl, and butyl acetate, and photographs of MAPbI3 QDs (after centrifugation: MAPbI3 QDs after dispersion in purifying solvent and centrifugation; after purification: MAPbI3 QDs after the completion of the purification process)

| Solvent      | Structure | Dielectric constant | After centrifugation (room light) | After purification |
|--------------|-----------|---------------------|-----------------------------------|--------------------|
| Methyl acetate | ![Structure](image) | 6.68                | ![Photo](image)                   | ![Photo](image)    |
| Ethyl acetate | ![Structure](image) | 6.02                | ![Photo](image)                   | ![Photo](image)    |
| Propyl acetate | ![Structure](image) | 6.002               | ![Photo](image)                   | ![Photo](image)    |
| Butyl acetate | ![Structure](image) | 5.01                | ![Photo](image)                   | ![Photo](image)    |

**Fig. 2** (a) PL spectra and (b) absorption spectra of MAPbI3 QDs before purification and MAPbI3 QDs purified using methyl, ethyl, propyl, and butyl acetate.

**Fig. 3** TEM images of MAPbI3 QDs (a) before purification and after purification with (b) methyl acetate, (c) ethyl acetate, (d) propyl acetate, and (e) butyl acetate.

**Fig. 4** XRD patterns of MAPbI3 QDs before purification, MAPbI3 QDs purified using methyl, ethyl, propyl, and butyl acetate, and the precursor PbI2.
were successfully removed and, notably, it was clearly proved that the best purifying solvent is methyl acetate.

Finally, in order to confirm the effectiveness of the purification process for LED applications, we demonstrated a MAPbI₃ QD LED with the following structure: indium tin oxide (ITO)/poly(3,4-ethylenedioxythiophene):poly(styrenesulfonate) (PEDOT:PSS; 40 nm)/poly(4-butylphenyl-diphenyl-amine) (poly-TPD; 20 nm)/MAPbI₃ QDs (10 nm)/tris-(1-phenyl-1H-benzimidazole) (TPBi; 50 nm)/lithium 8-quinolate (Liq; 1 nm)/Al (100 nm), as shown in Fig. 6a. Before evaluating the LED device characteristics, the optical properties of the MAPbI₃ QD thin film were clarified. The prepared MAPbI₃ QD thin film shows a lower PLQY of 48% than the dispersion sample, which indicates the denser packing of MAPbI₃ QDs on the substrate causing energy transfer to non-radiative recombination sites.²²,²³ Fig. 6b shows the PL spectrum of purified MAPbI₃ QD thin film prepared via a spin-coating method and the EL spectra of the LED device with MAPbI₃ QDs as an emission layer. As seen in Fig. 6b, the EL spectrum of the fabricated LED has an EL peak at 738 nm and a FWHM of 49 nm, which is almost the same as the PL emission spectrum during photoexcitation (a PL peak at 741 nm and a FWHM of 48 nm). In addition, there are no differences in the EL spectrum shape at different current values (Fig. ES6a†). As shown in Fig. 6c, the turn-on voltage is 3.8 V (at 0.1 cd m⁻²) and the maximum luminance is 5.6 cd m⁻² at 6.2 V. The maximum EQE of the prepared LED is 3.2%, as shown in Fig. 6d; as far as we know, the EQE of these MAPbI₃ QDs is a new record value.²⁴,²⁵ As a result, it can be seen that the suggested MAPbI₃ QD purification process utilizing the UBM method has a significant effect on the optical properties and LED device characteristics.

4. Conclusions

In summary, MAPbI₃ QDs with good optical properties, e.g., a PLQY of over 60%, were successfully prepared via an UBM method, purifying the QDs using methyl acetate. This is because unreacted perovskite precursors and unsuitably sized MAPbI₃ QDs were removed, and the FWHM of PL emission became narrower as a result. The dielectric constant of the carboxylic acid ester used as a purifying solvent affected the absorbance (collected amount) of MAPbI₃ QDs, and a linear relationship was confirmed. This was caused by interactions between the alkyl ligands capping the MAPbI₃ QD surface and the carboxylic acid ester, especially after a decrease in the hydrophobicity of the carboxylic acid ester. In addition, the MAPbI₃ QD thin film based on MAPbI₃ QDs purified using methyl acetate showed a PLQY of 48%. A fabricated LED showed brightly luminescent EL at 738 nm with a FWHM of 49 nm, luminance of 5.6 cd m⁻², and maximum EQE of 3.2%, which, as far as we know, is a new record EQE for MAPbI₃ QDs. Interestingly, the preparation of MAPbI₃ QDs via UBM and the purification process can all be performed under air, without needing a glove box. These results indicate that the process is a simple and practical approach for optoelectronic device applications.

Conflicts of interest

There are no conflicts to declare.

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