

Br-Doped CsPbI₂Br PEROVSKITE SOLAR CELL INVESTIGATIONS

Natthapong Wongdamnern¹, Nutchapon Chotigkrai²,
Taweewat Krajangsang³, Cheewita Suwanchawalit⁴, Surawut
Chuangchote^{5,6}, Sakchai Laksee⁷, Supakij Suttiruengwong⁸ and Narit
Triamnak^{8,*}

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Abstract

Organic-inorganic hybrid halide perovskite solar cells (PSCs) have gained significant attention recently. However, the moisture and thermal instability have still been their intensive weakness to prohibit their commercialization. Substituting organic cations in PSCs with inorganic ones is the most promising solution to this deterioration problem due to their moisture endurance characteristics. In this study, the inorganic halide perovskite solar cell CsPbI₂Br was investigated for the improvement of the performance stability via bromide doping. The bromide doping concentration was varied within a range of 0 to 0.05 ml through the hydrobromic acid (HBr) addition to observing the effect of bromide doping content on the solar cell performance. The film samples were prepared using the spin coating technique and structurally investigated using the X-ray diffraction (XRD) technique and Raman Spectroscopy. In addition, UV-Vis spectroscopy and energy band gap were also examined. It was found that the hydrobromic acid addition effectively prolonged the deterioration of the perovskite layer.

Keywords: Inorganic; Perovskite Solar Cells; CsPbI₂Br; Bromide Doping

¹ Faculty of Science and Technology, Rajamangala University of Technology Suvarnabhumi, Nonthaburi, 11000, Thailand.

² Department of Chemical Engineering, Faculty of Engineering and Industrial Technology, Silpakorn University, Nakhon Pathom, 73000, Thailand.

³ Solar Photovoltaic Research Team (SPVT), National Energy Technology Center (ENTEC), National Science and Technology Development Agency (NSTDA), Pathum Thani, 12120, Thailand.

⁴ Department of Chemistry, Faculty of Science, Silpakorn University, Nakhon Pathom, 73000, Thailand

⁵ Department of Tool and Materials Engineering, King Mongkut's University of Technology Thonburi (KMUTT), Bangkok, 10140, Thailand.

⁶ Research Center of Advanced Materials for Energy and Environmental Technology (MEET), King Mongkut's University of Technology Thonburi (KMUTT), Bangkok, 10140, Thailand

⁷ Nuclear Technology Research and Development Center, Thailand Institute of Nuclear Technology (Public Organization), Nakhon Nayok, 26120, Thailand.

⁸ Department of Materials Science and Engineering, Faculty of Engineering and Industrial Technology, Silpakorn University, Nakhon Pathom, 73000, Thailand. E-mail: triamnak_n@su.ac.th

* Corresponding author

Introduction

Over the past few years, organic-inorganic hybrid perovskite solar cells (PSCs) have been widely interested in photovoltaic applications due to their distinctive photovoltaic properties, such as tunable bandgap, high light absorption coefficient, low exciton binding energy, long-range carrier diffusion, and simple and low-cost manufacturing process (Jeon *et al.*, 2015; Zheng *et al.*, 2015; Liu *et al.*, 2016; Yang *et al.*, 2016; Luo *et al.*, 2018; Liu *et al.*, 2019; Wongdamnarn *et al.*, 2023). However, organic-inorganic hybrid perovskite solar cells are volatile in terms of thermal and humidity, prohibiting them from practical commercialization (Liu *et al.*, 2019). Moreover, hybrid perovskite solar cells often employ expensive organic hole-transport layers (HTL) and use noble metals for electrodes, which affects the fabrication cost. To solve this problem, substituting organic cations in the organic-inorganic hybrid PSCs with inorganic ones is the most promising solution to this deterioration problem due to the moisture endurance characteristics of inorganic elements.

Substituting Cs¹⁺ to the organic site like Methylammonium (MA) or Formamidinium (FA) of hybrid PSCs, in which both substituting and substituted cations have one positive charge, could conduct pure inorganic PSCs with more excellent stability (Liu *et al.*, 2019). Also, cooperating with using carbon for the electrode layer instead of high-cost noble metals could lower the cost ceiling of fabrication (Chang *et al.*, 2016; Liang *et al.*, 2016; Duan *et al.*, 2018a; Li *et al.*, 2018). Therefore, these bring much more attention to this kind of PSC.

In practical uses of pure inorganic PSCs, CsPbX₃ (X = Br, I) is one of the most excellent pure inorganic systems often used to produce perovskite solar cells. CsPbBr₃ is an inorganic compound with outstanding stability to high temperature and moisture. Unfortunately, it comes with a wide band gap (2.3 eV), low crystallinity, and poor morphologies (Kulbak *et al.*, 2015; Duan *et al.*, 2018b; Liu *et al.*, 2019), so it cannot absorb long wavelengths of light spectrum around 500 to 600 nm. In contrast, CsPbI₃ has a relatively narrow band gap (1.73 eV) (Sa *et al.*, 2022; Tai *et al.*, 2019) but is unstable in environments. Thus, this research aims to study the stability of inorganic perovskite solar cells in terms of balancing the band gap wideness and environmental stability by mixing Br and I into the X₃ site as CsPbI₂Br. Moreover, the hydrobromic acid concentration is also added up and varied in a range of 0, 0.01, 0.02, 0.03, 0.04, and 0.05 ml in CsPbI₂Br to study the development of morphological and optical properties as well as the efficiency of perovskite solar cell performance

because the hydrobromic acid can improve solubility and reduce the crystallinity rate resulting in regular and homogenous grains with less pores on the films (Yu *et al.*, 2016).

Experimental Procedures

Chemicals and materials

Precursor materials used in this study were comprised of Caesium bromide (CsBr ≥ 99.9%) and Lead (II) iodide (PbI₂ ≥ 99.9%) from Acros Organics, while Hydrobromic acid (HBr ≥ 49wt%), Dimethyl Sulfoxide (DMSO ≥ 99.9%) and Isopropanol (IPA ≥ 99.9%) were obtained from Carlo Erba Reagents. Besides, Deionized water (DI water) and ethyl alcohol were received from RCI Labscan Co., Ltd., and Acetone was received from Univar Solutions.

Synthesis of CsPbI₂Br solution

To synthesize the CsPbI₂Br solution, 2 mmol of PbI₂ and CsBr were added to 2 mL of dimethyl sulfoxide (DMSO), and then the solution was stirred for 30 minutes. Subsequently, the different concentrations of hydrobromic acid (HBr) with a volume of 0, 0.01, 0.02, 0.03, 0.04, and 0.05 mL were added into the stirred solution with continuous stirring for 30 minutes. Then, the yellow solution was finally obtained, indicating the formation of CsPbI₂Br.

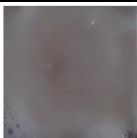
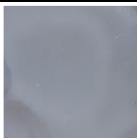
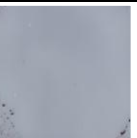
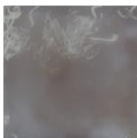
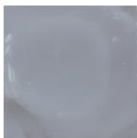
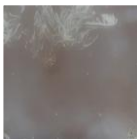
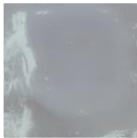
Film deposition

The conducting glasses, F-doped SnO₂, were cleaned by an ultrasonic cleaner sonicator with deionized water, ethanol, and isopropanol for 15 minutes each, respectively. Then, those conducting glasses were dried in N₂ gas and treated with UV-ozone for 20 minutes. After that, the CsPbI₂Br solution was operated under the one-step spin coating technique at 500 rpm for 10 seconds and 2500 rpm for 30 seconds and then annealed at 250°C for 10 minutes to form the perovskite films.

Characterizations

The crystal structure of the films was detected by the X-ray diffraction technique (XRD) of Shimadzu LabX XRD-6100. The chemical bonding of films was achieved by the Raman spectrometer of the Bruker Senterra series. Finally, the optical properties and further calculated energy band gap were conducted via the ultraviolet and visible (UV-vis) absorption spectroscopy by a Shimadzu UV-1800.

Table 1. Deterioration of CsPbI₂Br films with different concentrations of HBr acid at various exposure times

Samples Times	No HBr	0.01 HBr mL	0.02 HBr mL	0.03 HBr mL	0.04 HBr mL	0.05 HBr mL
initial						
30 min.						
1 hour.						
2 hours.						
4 hours.						
8 hours.						
12 hours.						
7 days.						

Results and Discussion

Physical appearance

In order to further understand the effect of the addition of HBr acid on the perovskite films, the films with different concentrations of HBr acid at 0, 0.01, 0.02, 0.03, 0.04, and 0.05 mL were prepared and investigated. After being successfully prepared, the films with different HBr acid concentrations were exposed to the ambient environment (50% RH and 25 °C) and left as a development of time, as

shown in Table 1. Please note that right after successful preparation, the films were brought to anneal at 250°C for 10 minutes before being exposed to an ambient environment. The as-annealed films with no and low concentrations of HBr acid (0 and 0.01 mL of HBr) showed a light-brown color on the film surface, while the films with higher concentrations of HBr acid appeared as milky white (0.02-0.05 mL of HBr). This implies

that HBr acid concentration directly affects the film color. With higher HBr acid concentration, brighter milky white is obtained. After 30 minutes passed, the light-brown films appearing in 0 and 0.01 mL of HBr condition started the transformation, as obviously seen by the appearance of white stripes on the surface. In contrast, the films with higher HBr acid concentrations had much fewer white stripes. These white stripes possibly indicated the deterioration of the films. However, as longer time passed, the deterioration in all film conditions became more and more obvious sequentially, as could be observed from the appearance of more and bigger white stripes in all film conditions. Comparatively, the appearance of white stripes in higher HBr acid concentration films was less than those of lower concentration ones. This suggests that a higher concentration of HBr acid could prolong the film's deterioration. Finally, after exposure to the ambient environment for 7 days, all film surfaces have paled to almost pure white, indicating complete deterioration.

X-ray diffraction

To confirm the crystal structure of CsPbI₂Br at different concentrations of HBr acid, the XRD patterns of the films were collected, as shown in Figure 1(a) - Figure 1(g). It can be seen that, at 0 hours of exposure, all films with different HBr acid concentrations revealed the diffraction peaks corresponding to the monoclinic structure (γ -phase) of CsPbI₂Br (ICDD no. 00-018-0364) (Yang *et al.*, 2019), while after exposed for 12 hours, all films wholly transformed to the orthorhombic structure (δ -phase) of CsPbI₂Br (ICDD no. 0376-018-00) (Wu *et al.*, 2020). Please note that the monoclinic perovskite structure is more suitable for solar cell applications because it has a considerably narrow energy band gap (1.92 eV) compared to that of the orthorhombic structure, which has a broader energy band gap (2.85 eV) (Mariotti *et al.*, 2018).

Noticeably, the diffraction patterns of the films at 0 HBr showed the peaks of CsPbI₂Br perovskite structure even without HBr acid, as seen in Figure 1(a). This indicates that the films have a monoclinic structure since the beginning. Anyway, after 0.5 hours of exposure in 0 HBr acid condition, the new diffraction peaks appeared at $2\theta = 26.2, 27.0, \text{ and } 32.0$. By referring to ICDD card no. 0376-018-00, these three diffraction peaks correspond to the CsPbI₂Br orthorhombic phase. Thus, the perovskite structure of the films with no HBr acid gradually transformed to a new phase after 0.5 hours. With further development over time, the CsPbI₂Br orthorhombic phase became more and more dominant. Interestingly, while the film structure of 0 HBr acid started transforming after

0.5 hours, the structure of the films with 0.01 and 0.02 HBr acid began changing to a new phase after 1 hour, as shown in Figure 1(b) - Figure 1(c). For 0.03, 0.04, and 0.05 HBr acids, the film structure started transforming after 2 hours, as displayed in Figure 1(d) - Figure 1(f). This strongly confirms that the HBr acid prolongs the phase transformation, which means it could help in retarding the film deterioration and maintaining the film stability.

To further understand the effect of adding HBr acid on the CsPbI₂Br perovskite, the XRD patterns with different HBr acid contents were analyzed comparatively, as illustrated in Figure 1(g). It can be observed that the peaks shifted to a higher angle when the concentration of HBr acid increased. This is attributed to the addition of HBr acid to the CsPbI₂Br structure, causing Br⁻ to replace the I⁻ site, resulting in a change of the I:Br ratio (Duan *et al.*, 2017). According to Shannon's Effective Ionic Radii, Br⁻ (1.96 Å) is smaller than I⁻ (2.2 Å), hence causing the peak shifting to a blue shift following Bragg's law as shown in the equation (Straus *et al.*, 2019).

$$n\lambda = 2d\sin\theta$$

where n is the diffraction order, λ is the x-ray wavelength, d is the spacing of the crystal layers (path difference), and θ is the incident angle (the angle between the incident ray and the scattering plane).

Therefore, this suggests that an increase in HBr acid content could reduce the size of a unit cell of the crystal structure.

Raman spectroscopy

Chemical bonding analysis of the CsPbI₂Br perovskite with different HBr acid concentrations was examined on 0, 0.01, 0.02, 0.03, 0.04, and 0.05 mL of HBr acid by Raman spectroscopy after 0, 0.5, 1, 2, 4, 8 and 12 hours of exposure to the environment and shown in Figures 2(a) - (f). It was found that after annealing, all film compositions still possessed the monoclinic structure (γ -CsPbI₂Br), which could be identified by the Raman peak at 63 cm⁻¹ (Satta *et al.*, 2021). This is also corresponding to the XRD results. As time passed for 0.5 hours, the films with HBr acid concentrations of 0, 0.01, 0.02, and 0.03 mL gradually occurred bonding vibration, which means the structure is starting to change from monoclinic to orthorhombic phase. Since the Cs-I bonding distance is about 3.95 Å, while the bonding distance of Pb-I is about 3.24 Å, thus making Pb-I bonding stronger. The vibration of Cs-I bonding, therefore, occurs at lower frequencies compared to those of Pb-I bonds. Hence, the peaks at 63 cm⁻¹ were for Cs-I bonding.

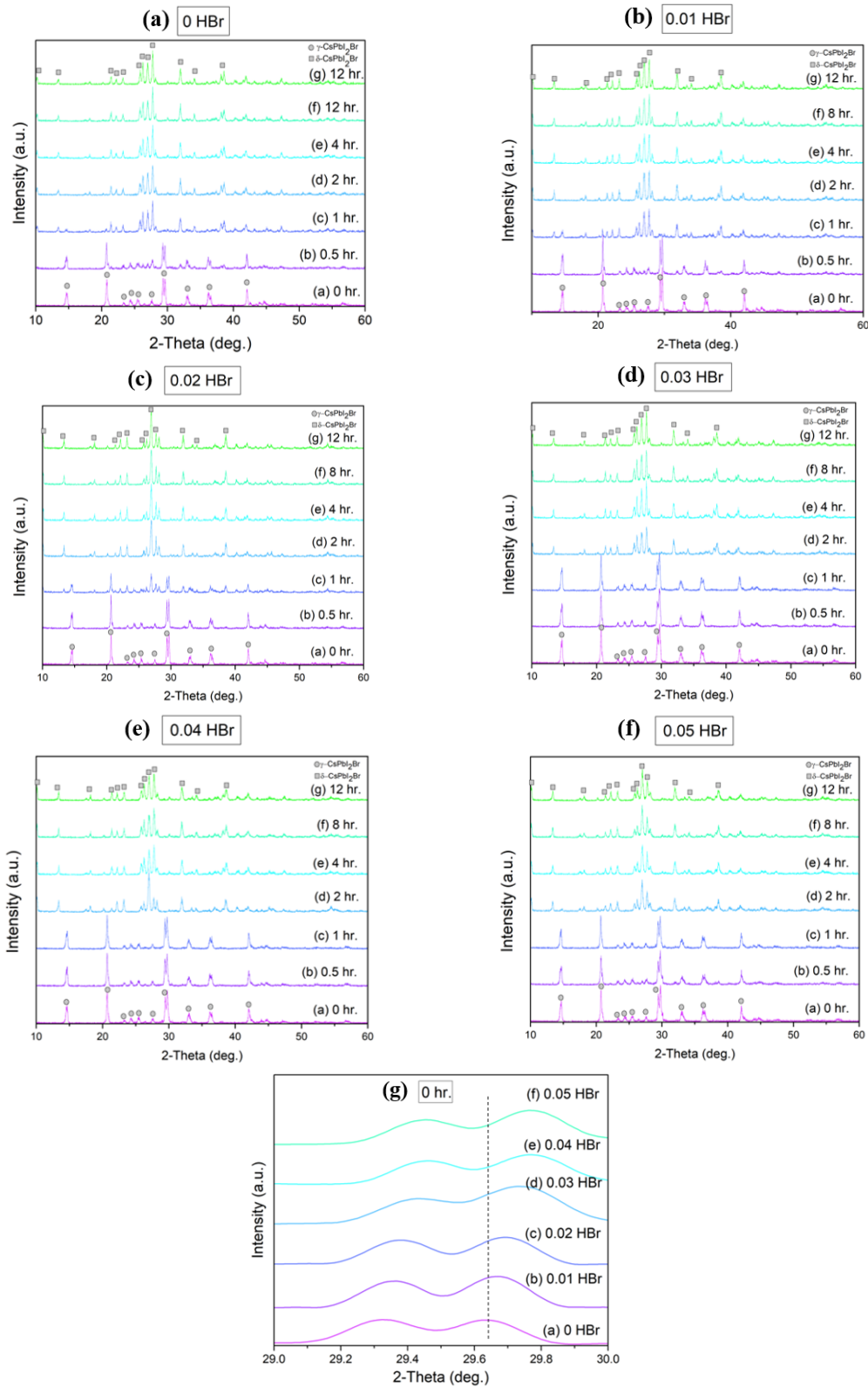


Figure 1. The XRD Patterns of annealed CsPbI_2Br films with different exposure times and HBr acid concentrations: (a) 0 mL HBr acid, (b) 0.01 mL HBr acid, (c) 0.02 mL HBr acid, (d) 0.03 mL HBr acid, (e) 0.04 mL HBr acid, (f) 0.05 mL HBr acid, and (g) magnified XRD Patterns of the films in a 2θ range of 29.0-30.0 with different HBr acid concentrations at 0 hr.

After 0.5 hours of exposure, the peak at 63 cm⁻¹ of the perovskite films at 0, 0.01, 0.02, and 0.03 mL of HBr acid disappeared, as seen in Figure 2(a)- Figure 2 (d). In contrast, the peak at 63 cm⁻¹ still remained in the perovskite films with 0.04 and 0.05 mL of HBr, as shown in Figure 2(e) - (f), and then completely disappeared after 1 hour. The four new peaks that appeared at 73 cm⁻¹, 88 cm⁻¹, 117 cm⁻¹, and 130 cm⁻¹ were from vibrations of structure transformation, indicating an orthorhombic structure (Satta *et al.*, 2021). The peaks at 73 cm⁻¹ and 88 cm⁻¹ were assigned to the

shearing of the double octahedra along the axial and equatorial directions. The peak at 117 cm⁻¹ was assigned to the vibration of the I-Pb-I frame, with the I-Pb-I stretching resulting in an expansion of the double octahedra. The peak at 130 cm⁻¹ was caused by vibration between Pb-O bonds due to unstable lead in ambient conditions, which may cause oxidation and become PbO (Yang *et al.*, 2020; Satta *et al.*, 2021). The remaining 63 cm⁻¹ peak at 0.04 and 0.05 mL of HBr conditions conform to the XRD results and confirm that HBr acid could help stabilize the perovskite performance.

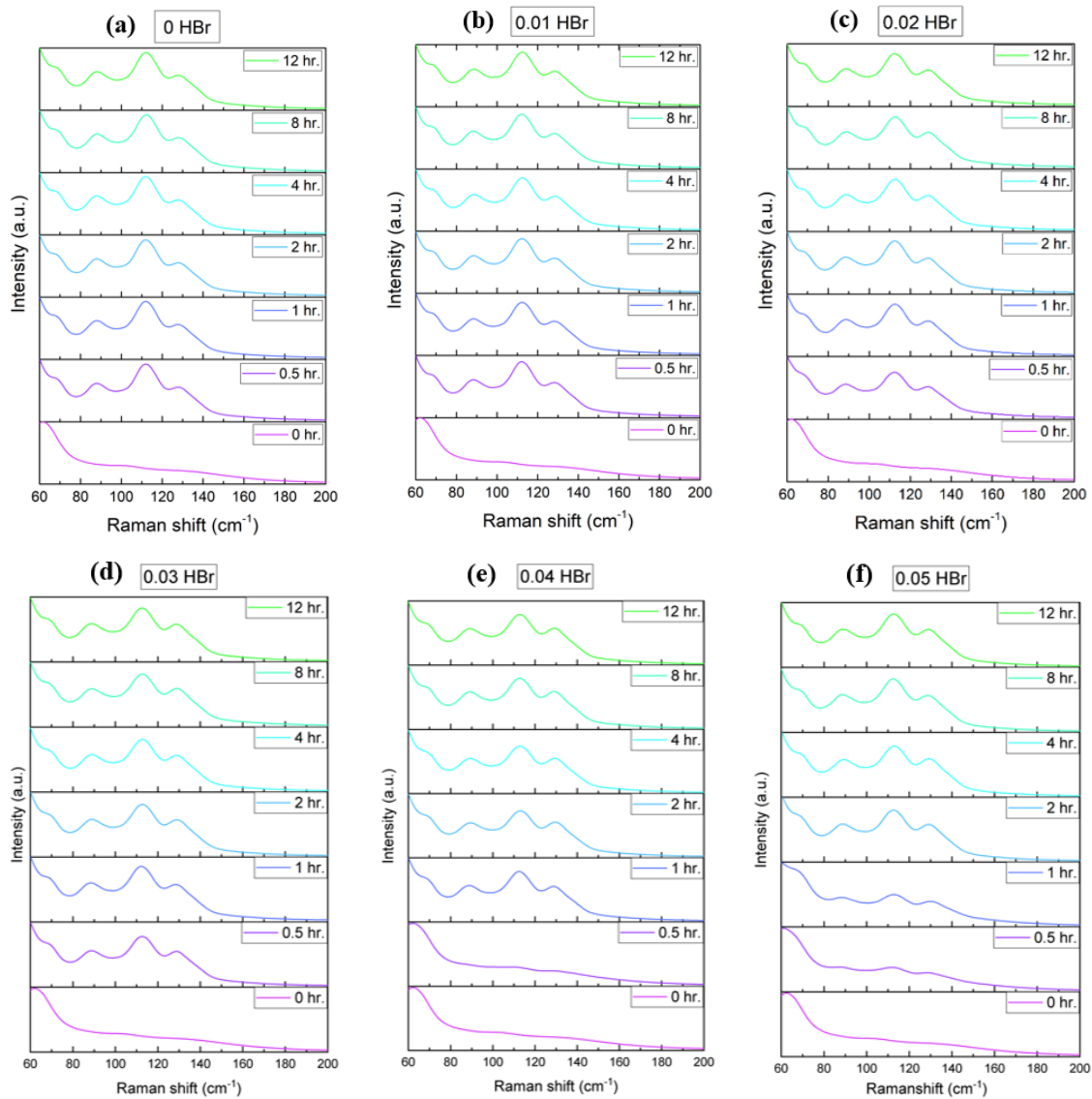


Figure 2. Raman spectra of CsPbI₂Br films with different concentrations of HBr acid (a) without HBr acid, (b) 0.01 mL of HBr acid, (c) 0.02 mL of HBr acid, (d) 0.03 mL of HBr acid, (e) 0.04 mL of HBr acid, and (f) 0.05 mL of HBr acid at various exposure times

UV-visible spectrophotometer

To study the optical properties of perovskite films with different contents of HBr acid, the annealed films were observed by UV-visible spectroscopy in the visible wavelength range between 400-700 nm, as shown in Figures 3(a) - (f). It was found that all film conditions could absorb light in the visible wavelength and had an absorption edge from 400-640 nm. With careful consideration, it can be noticed that the absorption

of the films with 0.02 and 0.03 HBr acid concentrations is reduced obviously, indicating that the absorbance of these films decreases at short wavelengths. Nevertheless, all film compositions still performed light absorption effectively in the same wavelength range even after 12 hours. This means the absorption performance of perovskite films has still remained stable even though their crystal structure has gradually transformed.

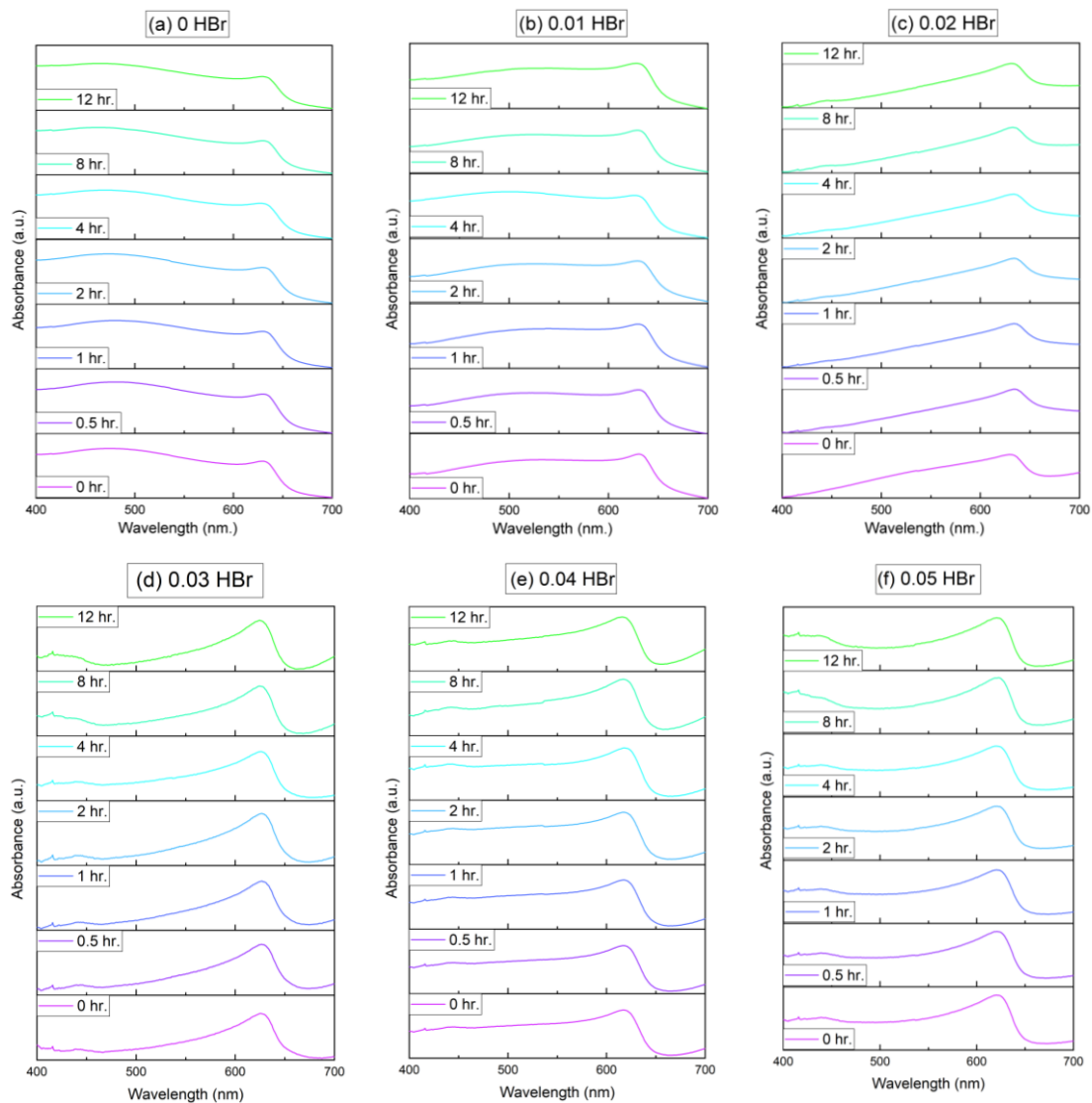


Figure 3. Absorbance of CsPbI₂Br films with different concentrations of HBr acid (a) without HBr acid, (b) 0.01 mL of HBr acid, (c) 0.02 mL of HBr acid, (d) 0.03 mL of HBr acid, (e) 0.04 mL of HBr acid, and (f) 0.05 mL of HBr acid at various times

The energy band gap of CsPbI₂Br films with different contents of HBr acid for 0-hour exposure was calculated representatively and shown in Fig. 4(a-b). To study the optical properties of CsPbI₂Br films with structural development and the influence of HBr acid addition, the energy band gap was determined by Tauc plots (Makula *et al.*, 2018).

$$\alpha h\nu = A(h\nu - E_g)^n$$

where α is the absorption coefficient, h is the Planck constant, ν is the photon's frequency, E_g is the energy band gap, and A is a constant. The n factor depends on the nature of the electron transition and is equal to 1/2 or 2 for the direct and indirect transition band gap, respectively.

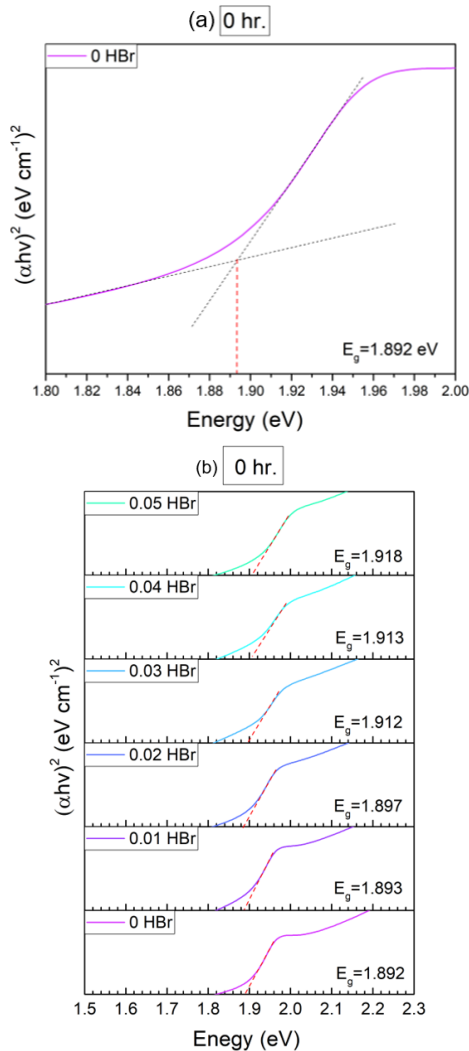


Figure 4. Energy band gap of CsPbI₂Br films (a) with no HBr acid and (b) with different concentrations of HBr acid at 0-hour exposure

Figure 4(a) showed the energy band gap of perovskite CsPbI₂Br film with no HBr acid right after annealing. The energy band gap was 1.892 eV. With increasing HBr acid contents from 0 to 0.05 mL, the energy band gap gradually widened. This is due to an increase in HBr acid contents affecting the ratio of I:Br (Duan *et al.*, 2017). With higher HBr acid concentration, more phase transformation into an orthorhombic structure with a wider energy band gap occurred. This agrees with the results in Figure 4(b).

Conclusions

In summary, the synthesis of CsPbI₂Br perovskite films with different concentrations of hydrobromic (HBr) acid was successfully fabricated. The addition of HBr acid to the perovskite could shift the diffraction peak of 2θ in a range of 29° - 30° to the higher angle due to the smaller ion substitution of Br⁻ into the I⁻ site. The perovskite films gradually changed from the monoclinic to orthorhombic structure with an increase in the content of HBr acid. On Raman spectroscopy investigation, the peak of all annealed films with 0-hour exposure appeared at 63 cm^{-1} , corresponding to the γ -CsPbI₂Br (monoclinic phase). With an increase in exposure time, the crystal phase gradually transformed to δ -CsPbI₂Br (orthorhombic phase), which was a signal of film deterioration. However, the films with higher concentrations of HBr acid could effectively prolong the deterioration. Raman spectroscopy could confirm the phase transformation with the time passed, and higher HBr acid content could delay the formation of a new phase. The energy band gap gradually increased with increasing HBr acid concentration. Anyway, light absorbance in the visible wavelength was still effective through the environment exposure in all film conditions. Therefore, HBr acid could effectively prolong the deterioration of the films with an insignificant reduction in photovoltaic conversion performance.

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