



# Sonochemical synthesis of $\text{CH}_3\text{NH}_3\text{PbI}_3$ perovskite ultrafine nanocrystal sensitizers for solar energy applications



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## ABSTRACT

The organic–inorganic hybrid perovskite  $\text{CH}_3\text{NH}_3\text{PbI}_3$  is becoming an interesting material in the field of energy harvesting. This material is one of the cleanest and cheapest components in solar cells which is available in ample amounts. However, most of the previous research work was done on thin film of this material. In the present work we describe the preparation of a powder containing nanoparticles of  $\text{CH}_3\text{NH}_3\text{PbI}_3$  using a sonochemical method. Characterization of the product was done by various methods, such as HRTEM, FTIR, PL, DLS and XRD. The particles were found to be highly crystalline (tetragonal crystal structure), polygonal in shape and having diameters of 10–40 nm.

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## 1. Introduction

Silicon cells are today's most popular commercialized solar cells, ruling all the photovoltaic industry with highest 24.7% efficiency for single-crystal silicon solar cells [1]. However, silicon solar cells are expensive for large scale production. It is known that the organic solar cells are much cheaper than the inorganic ones because of the low cost of the components and the ease of fabrication. In the past decade the perovskite-type compounds methylammonium lead trihalides  $\text{CH}_3\text{NH}_3\text{PbX}_3$  ( $\text{X} = \text{Cl}, \text{Br}, \text{I}$ ) gained more interest in solar-energy application ("perovskite solar cells"). The rapid advances and breakthroughs in the organic–inorganic hybrid photovoltaic materials make the field attractive and promising for the solar cell industry [2–7]. Recently, improvement in the efficiency of the perovskite solar cell has been reported [7,8]. The fabrication costs of the perovskite solar cells are much lower than those of any other photovoltaic device [6].  $\text{CH}_3\text{NH}_3\text{PbI}_3$  has a very high absorption coefficient in the visible region and a band gap of about 1.4–1.5 eV [9]. This material is of the crystal structure of perovskite  $\text{ABX}_3$  with distortion in the octahedral center, breaking the inversion center and resulting in an octahedral tilt of the system [3]. The Pb atom is located in the corner of the cage

whereas the iodine atom shares a bond between two Pb ions and the methyl amine ion, present at the center of the cage, acts as a rotating dipole. Solar cells made of this material work effectively in both mesoscopic and thin film configurations. The highest reported efficiency of this material, according to Seok, was 20.4% [10].  $\text{CH}_3\text{NH}_3\text{PbI}_3$  exhibits a very high charge carrier diffusion length of about 1 micron which is extremely good for photovoltaic cells [10]. A thin film of perovskite shows very high polarizability, ferroelectric property and ionic movement in the system. Current researches are focusing on finding an efficient fabrication method of the perovskite material to make the solar cells and light emitting diode (LED) devices cheaper and easier to produce [8].

Quantum dots (QD) of perovskite are also one of the targets of current research as an additional means for increasing the efficiency of the solar devices. Easy exciton formation and charge-separation are remarkably efficient in QD. These nanocrystals are mainly used in the areas of fluorescent labeling, probing, LED and solar points [11]. The fabrication of stable quantum dot of perovskites was previously reported [11–13] by several groups but commercial application of perovskite-QD is still under investigation. Few reports described the synthesis of  $\text{CH}_3\text{NH}_3\text{PbI}_3$  by one-step deep coating and/or other methods without using any catalyst [14–18]. Perovskite nanoparticles (NPs) have attracted attention due to their easy synthesis for highly luminescent colloidal quantum dot materials which are highly demanding in the field of LED devices and solar cell fabrication.

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Recently an ultrasound-assisted approach for the synthesis of the perovskite in the bulk form was reported [19]. However, a synthetic route of perovskite NPs in the nanometric scale is still required with good homogeneity, which is an essential parameter required for the fabrication of LED and solar cell devices. In the present work we report on a new sonochemical technique for the formation of  $\text{CH}_3\text{NH}_3\text{PbI}_3$  NPs in the size-range of 10–40 nm. The method involves ultrasonic irradiation of two materials,  $\text{CH}_3\text{NH}_3\text{I}$  and  $\text{PbI}_2$ , dissolved in isopropanol, without any catalyst. In addition, this study presents a detailed investigation of the effect of the sonication parameters on the physical and chemical properties of  $\text{CH}_3\text{NH}_3\text{PbI}_3$  NPs.

## 2. Experimental

### 2.1. Chemicals

Lead iodide ( $\text{PbI}_2$ ) and isopropanol were purchased from Sigma-Aldrich and used as received. Methyl ammonium iodide (MAI) was prepared according to a procedure published elsewhere [4,19,20].

### 2.2. Preparation procedure

The  $\text{CH}_3\text{NH}_3\text{PbI}_3$  ultrafine nanoparticles were prepared by sonicating together solutions of the two components,  $\text{CH}_3\text{NH}_3\text{I}$  and  $\text{PbI}_2$ , in isopropanol. First, 8 mL of a 0.25 M solution of  $\text{CH}_3\text{NH}_3\text{I}$  were transferred into a glass test tube and sonicated for 30 s. Then 6 mL of 0.25 M solution of  $\text{PbI}_2$  were added dropwise while sonication was continued for another 10, 20 or 30 min. A change in the color from yellow to dark brown was observed right after mixing the two precursors, indicating the occurrence of a chemical reaction. The sonication cell was immersed in a water bath and the temperature inside was constantly monitored by a thermocouple to be 40–44 °C.

To examine the effect of sonication, control experiments were performed in which the solutions of the two components were mixed by magnetic stirring at 40 °C, using either a regular laboratory stirrer (Fried Co. Israel) at 1000 rpm and or a high-speed stirrer (6000 rpm) Ultraturrax device (Leroy Somer, Digidrive SK, make ESCO-LABOR).

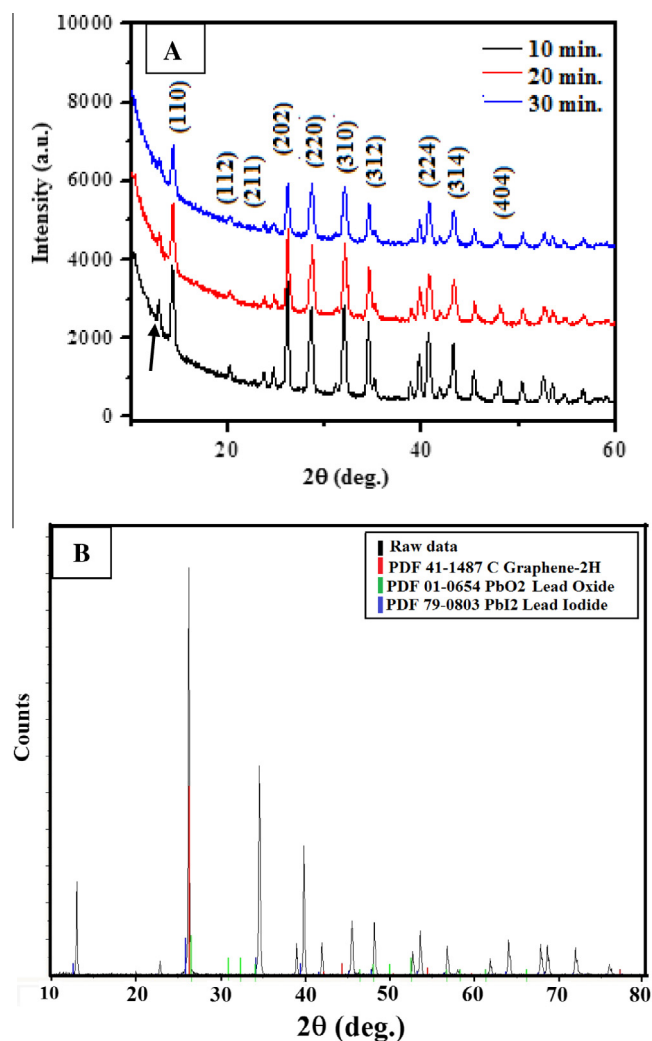
### 2.3. Equipment

The ultrasonic transducer (Sonics and Materials Inc., USA, model VCX 750, frequency 20 kHz, volt 230V AC) was operated at an amplitude of 60%.

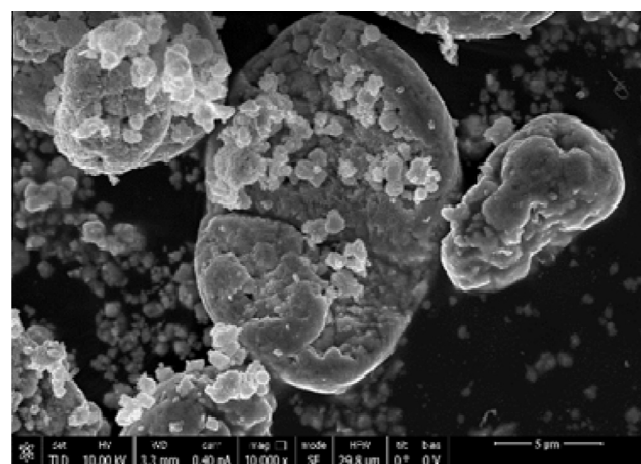
### 2.4. Analytical techniques

Inspection of the products was done with a JEOL 2100 high-resolution transmission electron microscope (HRTEM), operated at an accelerating voltage of 200 kV. The samples for TEM were prepared by suspending some  $\text{CH}_3\text{NH}_3\text{PbI}_3$  powder in isopropanol, using an ultrasonic cleaning bath. A few droplets of the suspension were applied on a carbon-coated TEM grid and dried in a vacuum chamber. Elemental analysis and elemental mapping were performed using the TEM Energy Dispersive X-ray Spectroscopy (EDS) unit. Scanning electron microscopy (SEM) was performed using an Inspect FEI microscope. Zeta potential and DLS measurements of the particles were performed on ZetaSizer Nano-ZS (Malvern Instruments Ltd., Worcestershire, UK). The photoluminescence of the  $\text{CH}_3\text{NH}_3\text{PbI}_3$  was measured using a fluorescence spectrophotometer (Varian Cary Eclipse). X-ray diffraction (XRD) was performed with a Bruker D8 Advance or with Philips

PW1050 X-ray diffractometer using  $\text{Cu K}\alpha$  radiation operating at 40 kV/30 mA with a 0.0019 step size and a 1.0 s step.



**Fig. 1.** XRD patterns of  $\text{CH}_3\text{NH}_3\text{PbI}_3$  perovskite ultrafine nanoparticles. (A) After sonication in isopropanol for 10, 20, and 30 min. The peak assigned to unreacted  $\text{PbI}_2$  is marked. (B) After 20 min sonication in water.



**Fig. 2.** SEM image of  $\text{CH}_3\text{NH}_3\text{PbI}_3$  particles obtained by reaction of  $\text{CH}_3\text{NH}_3\text{I}$  and  $\text{PbI}_2$  solutions under vigorous stirring (6000 RPM).

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