



# Er<sup>3+</sup> loaded barium molybdate nanoparticles: IR to visible spectral upconversion

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## ABSTRACT

In this study, we report the infrared to visible upconversion luminescence behavior of Er<sup>3+</sup> loaded barium molybdate nanoparticles synthesized by microwave induced sol–gel route. Structural and morphological properties were analyzed and the upconversion response of the Er<sup>3+</sup> loaded samples (1 mol% and 5 mol%) was investigated by exciting the samples at 980 nm from a Continuous Wave Laser Diode (CWLD). Results showed that the green emissions around 528 and 546 and red emissions at 658 nm were observed, respectively, and the intensity of both emissions were found to be enhanced with increase in concentration from 1 mol% to 5 mol% of Er<sup>3+</sup> ions. Upconversion mechanism is discussed and this study gives the way to convert infrared energy to visible light in other molybdate scheelite structures.

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## 1. Introduction

Nanoengineering of upconversion materials has attracted the great deal of attention due to their important applications in various fields such as biomedical imaging, optical devices, data storage, photovoltaics and photocatalytic activities [1–3]. Among the upconversion materials, rare earth ions doped  $\beta$ -NaYF<sub>4</sub> is known as the most efficient upconverter due to its low phonon energy that minimizes the nonradiative pathways for upconverted energy states [4]. However, due to the poor chemical stability and toxicity induced by fluoride ions, its wide range of applications has remained limited [5]. Although a number of host materials exist for effective upconversion process, Oxides in particular, has attracted much attention due to their thermal and chemical stability, environmental nontoxicity and lower phonon energy [6]. Upconversion properties of rare earth ions doped binary oxides such as TiO<sub>2</sub>, CeO<sub>2</sub>, Y<sub>2</sub>O<sub>3</sub>, ZnO has been studied by different authors [7–10]. However, the upconversion of Er<sup>3+</sup> doped ternary oxides such as molybdates and tungstates have rarely been reported.

Barium molybdate is one of the most important members of molybdate family having scheelite-type tetragonal structure and

has potential applications in photoluminescence, solid-state lasers, photocatalysts and optical fibers [11]. In our earlier report [12], we investigated the photocatalytic activity of Er<sup>3+</sup> loaded barium molybdate nanoparticles and showed that the rare earth metal ion doped molybdate possesses enhanced photoactivity due to change in physicochemical and optical, in particular, upconversion properties; however, investigation of detailed upconversion properties were remained. Therefore, the scope of this study is to demonstrate the upconversion properties of Er<sup>3+</sup> loaded barium molybdate nanoparticles to better understand the mechanism and to establish this system for future applications.

## 2. Experimental

All the chemicals used in this study were of analytical grade and received from the Sigma Aldrich. The samples were prepared by adopting the procedure reported in our previous work [12]. Briefly, in a typical procedure, stoichiometric amount of Ba(NO<sub>3</sub>)<sub>2</sub> and H<sub>2</sub>MoO<sub>4</sub> were dissolved in 20 mL ethylene glycol in separate beakers and then mixed together. Aqueous solution containing 0 mol%, 1 mol% and 5 mol% of ErCl<sub>3</sub>·6H<sub>2</sub>O (99.9%, AR) was added to the resulting mixture separately and the pH 9 was maintained. The resulting precursor suspension was transferred to a teflon vessel for microwave assisted hydrothermal treatment (150 °C, 0.5 h, 200 W). Samples were washed with de-ionized water several

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times and finally calcined in air at 400 °C for 5 h. For the sake of convenience, the samples hereafter have been labeled as S-0, S-1 and S-5, respectively.

### 3. Characterizations

The phase and morphology were analyzed by performing powder X-ray diffraction (XRD), (Rigaku, D/MAX2200HR) and transmission electron microscopy (TEM), (JEM-2010, JEOL Ltd, Japan). The UC spectra were recorded by exciting the samples at 980 nm with 120–200 mW from a continuous wave laser diode (CWLD). The emitted signal was focused onto a PC-controlled SP-2357 spectrograph (Acton Research) and detected by a photomultiplier tube R955 (Hamamatsu). Samples were supported in 1 mm capillary tubes and measurements were done at room temperature.

### 4. Results and discussions

Fig. 1 shows the XRD patterns of the  $\text{Er}^{3+}$  loaded barium molybdate samples calcined at 400 °C for 5 h and confirmed that all the samples exist in similar phase. As we see the patterns, it is revealed that all the diffraction patterns could be indexed to scheelite-type tetragonal structure of  $\text{BaMoO}_4$  (JCPDS card no. 29-0193) and no signal for any other impurities or secondary phases were detected in the samples, implying that the charge difference induced by  $\text{Er}^{3+}$  ion doped into the bivalent site of host ions ( $\text{Ba}^{2+}$ ) did not affect the structural properties of the  $\text{BaMoO}_4$  [13]. However, as we compare the intensity of the doped samples, it is gradually decreased when the concentration of  $\text{Er}^{3+}$  ion increases from 0 mol% to 5 mol%. Moreover, we performed the peak profile analysis and calculated the crystallite size of each samples by using scherrer equation (please see [Supplementary information, S1](#)). Results showed that the crystallite size of the samples decreased with increasing the dopant concentration indicating that the degree of cationic ordering might be decreased by the doping with trivalent rare earth ions. Thus, XRD patterns confirmed the samples possessing the similar phase with slight variations in degree of crystallinity. Moreover, as synthesized products have oval shaped morphology with particle size ranging

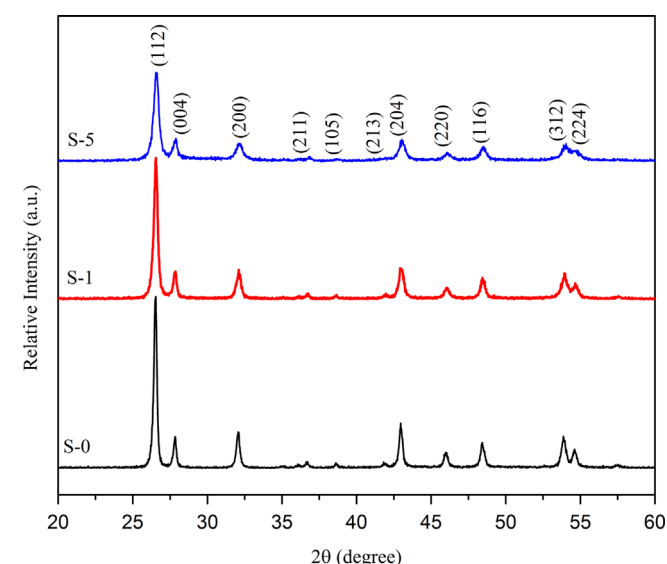


Fig. 1. XRD patterns of  $\text{Er}^{3+}$  loaded barium molybdate samples calcined at 400 °C for 5 h.

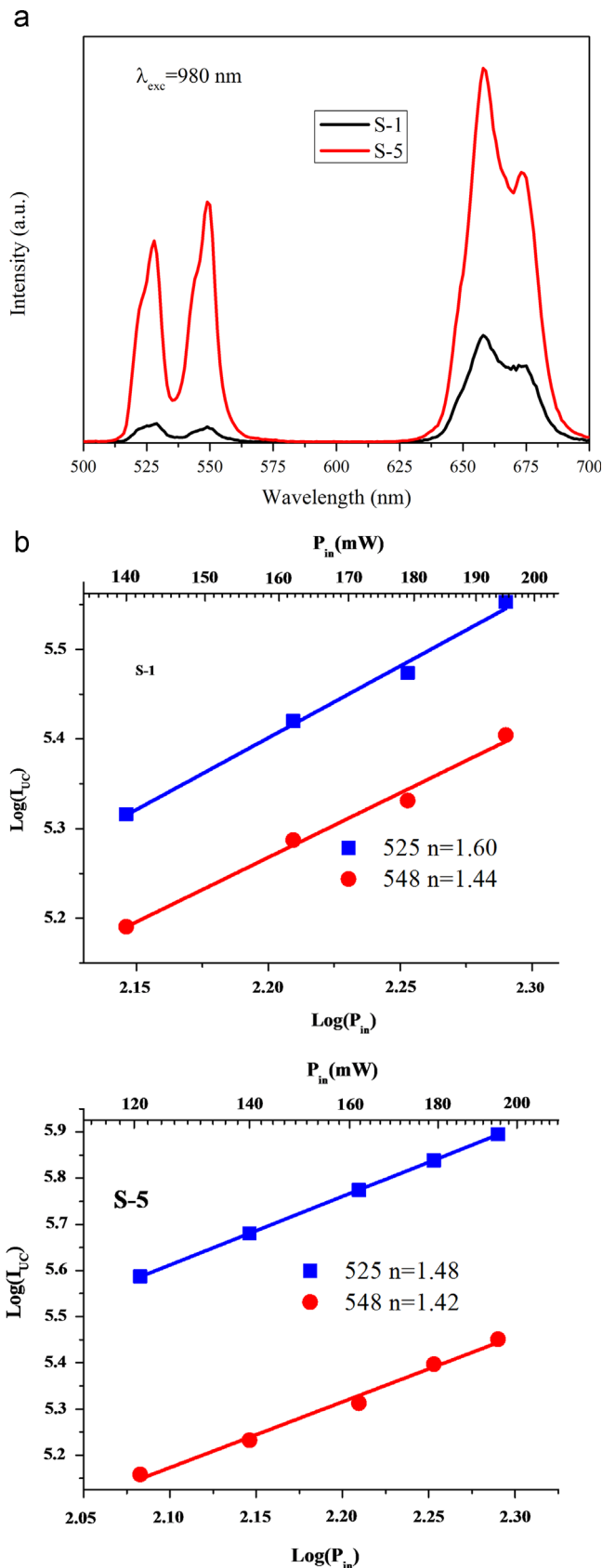


Fig. 2. (a) Upconversion emission spectra of the  $\text{Er}^{3+}$  loaded  $\text{BaMoO}_4$  nanoparticles upon excitation at 980 nm. (b) Dependence of the integrated (area under the curve) upconversion intensities on the pump power ( $P_{in}$ ). The straight lines are the fittings obtained by linear regression to the experimental data for each wavelength, S-1 (a) and S-5 (b).

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