

## Research Article

# Sonochemical Syntheses of a One-Dimensional Mg(II) Metal-Organic Framework: A New Precursor for Preparation of MgO One-Dimensional Nanostructure

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Nanostructure of a Mg<sup>II</sup> metal-organic framework (MOF), {[Mg(HIDC)(H<sub>2</sub>O)<sub>2</sub>].1.5H<sub>2</sub>O}<sub>n</sub> (**1**) (H<sub>3</sub>IDC = 4,5-imidazoledicarboxylic acid), was synthesized by a sonochemical method and characterized by scanning electron microscopy, X-ray powder diffraction, IR spectroscopy, and elemental analyses. The effect of concentration of starting reagents on size and morphology of nanostructured compound **1** has been studied. Calcination of the bulk powder and nanosized compound **1** at 650°C under air atmosphere yields MgO nanostructures. Results show that the size and morphology of the MgO nanoparticles are dependent upon the particles size of compound **1**.

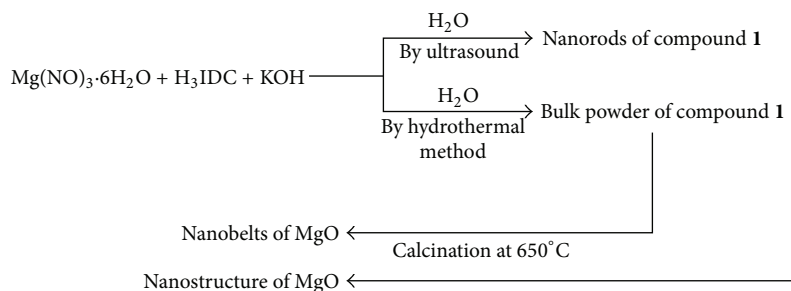
## 1. Introduction

Metal-organic frameworks (MOFs) constructed by metal ions and multifunctional organic linkers have attracted a great deal of interest in recent years [1–7]. Their high internal surface area, light weight, high porosity, and low volumetric density give them great opportunity for the potential applications in ion exchange [8], chemical sensor [9–13], catalysis [14–17], gas storage [18–21], and separation [22]. Recently, 4,5-imidazoledicarboxylic acid (H<sub>3</sub>IDC) was used to synthesize various functional metal-organic hybrid frameworks [23–29]. This ligand has three pH-dependent abstractable protons and six donor sites, which can be exploited for the synthesis of a variety of hybrid solids through a number of flexible coordination modes [30]. However, most of the MOFs are constructed from transitional metals but rare earth metals and alkali or alkaline-earth metals based MOFs are scarcely investigated [31–34]. Compared to the transitional metals, alkali metal units are lighter, which can offer a lower framework density and lead to an increase in gravimetric gas sorption capacity [35–37]. Among the s-block metals, the large polarizing power of Mg<sup>II</sup> can provide a strong

coordination bond with oxygen. There are few porous hybrid frameworks based on Mg<sup>II</sup> that have been reported [38–43].

Generally, porous materials are synthesized by slow diffusion, hydrothermal, and solvothermal synthesis methods [44–46]. In many cases a long reaction times, high reaction temperatures and pressures are required. To date a more efficient synthetic approach to MOFs still remains a challenge. Recently, a microwave assisted hydrothermal method is applied to prepare MOFs. This method is a highly efficient route to MOFs, although some reactions finish within several hours, but high reaction temperature and pressure are still needed [47, 48]. In the past two decades, sonochemical methods have been widely used in organic synthesis [49]. Compared with traditional techniques, sonochemical method is more convenient and easily controlled. A large number of organic reactions have been carried out under ultrasound irradiation in high yields within a short reaction time. To date application of ultrasonic method for the construction of MOFs remains largely unexplored [50].

In the present work, a Mg<sup>II</sup> metal-organic framework with ligand H<sub>3</sub>IDC, {[Mg(HIDC)(H<sub>2</sub>O)<sub>2</sub>].1.5H<sub>2</sub>O}<sub>n</sub> (**1**) (H<sub>3</sub>IDC = 4,5-imidazoledicarboxylic acid) was synthesized



SCHEME 1: Materials produced and synthetic methods.

under ultrasonic irradiation. Conversion of the compound **1** into nanostructured magnesium oxide (MgO) by calcination at 650 °C was also investigated. Synthesis of magnesium oxide nanostructures has been given much attention due to its applications in catalysis, toxic waste remediation, and as an additive in refractory, paint, and superconductor products [51–53].

## 2. Experimental

**2.1. Materials and Physical Techniques.** All reagents for the synthesis and analysis were commercially available from Merck Company and used as received. Doubly distilled water was used to prepare aqueous solutions. Ultrasonic generators were carried out on a SONICA-2200 EP, input: 50–60 Hz/305 W. Melting points were measured on an Electrothermal 9100 apparatus. Microanalyses were carried out using a Heraeus CHN-O-Rapid analyzer. The infrared spectra were recorded on a Nicolet Fourier Transform IR, Nicolet 100 spectrometer in the range 500–4000  $\text{cm}^{-1}$ , using the KBr disk technique. The simulated XRD powder patterns based on single crystal data were prepared using Mercury software [54]. X-ray powder diffraction (XRD) measurements were performed using a Philips X'pert diffractometer with monochromated  $\text{Co } k\alpha$  radiation ( $k = 1.78897 \text{ \AA}$ ). The samples were characterized by a scanning electron microscope (SEM) (Philips XL 30 and S-4160) with gold coating.

**2.2. Synthesis of  $\{[\text{Mg}(\text{HIDC})(\text{H}_2\text{O})_2] \cdot 1.5\text{H}_2\text{O}\}_n$  (**1**).** The compound **1** was prepared according to the reported method [55]. A mixture of  $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  (0.5 mmol, 0.128 g),  $\text{H}_3\text{IDC}$  (0.5 mmol, 0.078 g), and KOH (1 mmol, 0.056 g) in a molar ratio of 1:1:2 was dissolved in distilled water (8 mL) and stirred for 1 h in air. The solution was transferred into 23 mL Teflon-lined Parr autoclave and heated at 160 °C for 24 h. The mixture was allowed to cool to room temperature and the resulting colorless crystals were filtered off, washed with distilled water and ethanol, and air-dried [46] ((0.084 g yield: 69.4%), m.p. > 300 °C. (Anal. calcd for  $\text{C}_5\text{H}_9\text{MgN}_2\text{O}_{7.5}$ : C, 24.87; H, 3.76; N, 11.60. Found: C, 24.89; H, 3.28; N, 11.65%). IR ( $\text{cm}^{-1}$ ) selected bands: 3414 (w), 1579 (s), 1471 (s), 1396 (s), 1247 (m), 1105 (m), 837 (m), 674 (m)).

**2.3. Synthesis of  $\{[\text{Mg}(\text{HIDC})(\text{H}_2\text{O})_2] \cdot 1.5\text{H}_2\text{O}\}_n$  (**1**) Nanostructure by Using Sonochemical Method.** To prepare nanosized

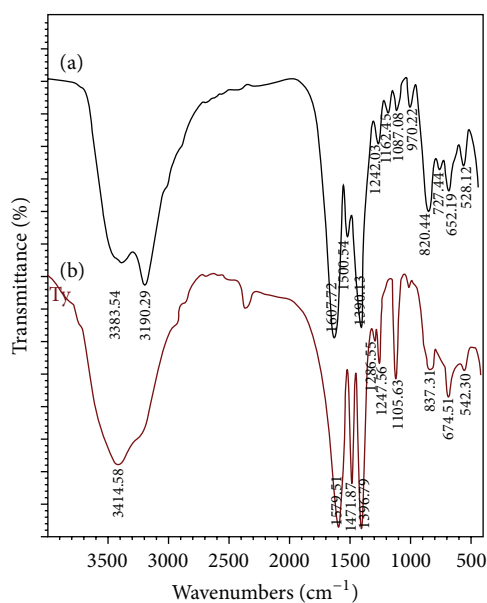


FIGURE 1: IR spectra of (a) nanostructured compound **1** produced by sonochemical method in concentration of initial reagents  $[\text{HL}^{2-}] = [\text{Mg}^{2+}] = 0.05$  and (b) bulk powder of compound **1**.

$\{[\text{Mg}(\text{HIDC})(\text{H}_2\text{O})_2] \cdot 1.5\text{H}_2\text{O}\}_n$  (**1**), 20 mL of an aqueous solution of the ligand  $\text{H}_3\text{IDC}$  (0.05 M) and potassium hydroxide (0.1 M) was positioned in a high-density ultrasonic probe, operating at 50 Hz with a maximum power output of 305 W. Into this solution 20 mL of an aqueous solution of magnesium nitrate (0.05 M) was added dropwise. The obtained precipitates were filtered off, washed with water and ethanol, and air-dried (m.p. > 300 °C. (Found: C, 24.84; H, 3.22; N, 11.67%). IR ( $\text{cm}^{-1}$ ) selected bands: 3383 (w), 3190 (w), 1607 (br), 1500 (m), 1390 (s), 1242 (m), 820 (m), 652 (m)).

For studying the effect of concentration of initial reagents on size and morphology of nanostructured compound **1**, the above processes were done under the following concentration condition of initial reagents:  $[\text{HL}^{2-}] = [\text{Mg}^{2+}] = 0.025 \text{ M}$ .

## 3. Preparation of MgO Nanostructures

For preparation of MgO nanostructure and also for investigating the size effect of precursor **1** on the size and morphology of the MgO nanostructures, calcination of bulk

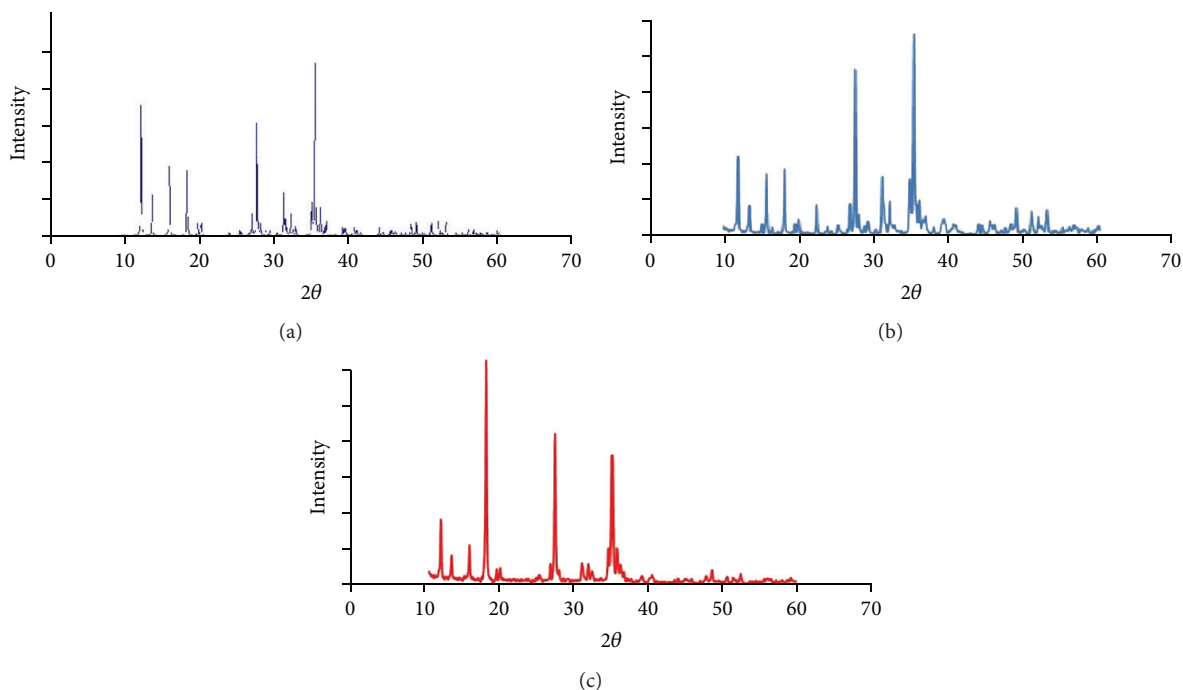


FIGURE 2: XRD patterns; (a) simulated pattern based on single crystal data of compound **1**, (b) bulk powder of **1**, and (c) nanostructured compound **1** prepared by sonochemical process in concentration of initial reagents  $[\text{Mg}^{2+}] = [\text{L}^{3-}] = 0.05 \text{ M}$ .

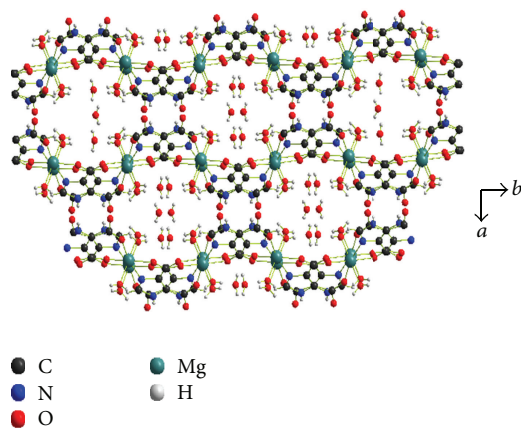


FIGURE 3: A fragment of the 3D framework in compound **1**, viewed along  $c$  direction.

powder and nanosized compound **1** was done at  $650^\circ\text{C}$  in static atmosphere of air for 4 h. IR spectrum and powder XRD diffraction show that calcination was completed and the entire organic compound was decomposed.

#### 4. Results and Discussion

The reaction between 4,5-imidazolecarboxylic acid ( $\text{H}_3\text{IDC}$ ) and potassium hydroxide with magnesium nitrate led to formation of a metal-organic framework  $\{[\text{Mg}(\text{HIDC})(\text{H}_2\text{O})_2] \cdot 1.5\text{H}_2\text{O}\}_n$  (**1**). Nanostructure of compound **1** was obtained in aqueous solution under ultrasonic irradiation,

while the bulk powder of compound **1** was obtained under hydrothermal condition. Scheme 1 gives an overview of the methods used for the synthesis of  $\{[\text{Mg}(\text{HIDC})(\text{H}_2\text{O})_2] \cdot 1.5\text{H}_2\text{O}\}_n$  (**1**) using the two different routes.

The elemental analysis and IR spectra of the nanostructure produced by the sonochemical method and of the bulk material produced by the hydrothermal method are indistinguishable (Figure 1). The symmetric and asymmetric vibrations of the carboxylate group are observed in the regions of  $1531\text{--}1654$  and  $1396\text{--}1471 \text{ cm}^{-1}$ , respectively. The  $\Delta(\nu_{\text{as}} - \nu_{\text{sym}})$  values indicate bidentate chelate coordination mode of  $\text{HIDC}^{2-}$  in **1**. The strong and broad absorption band in the range of  $3400\text{--}3500 \text{ cm}^{-1}$  indicates the presence of hydrogen-bonded water molecules [55].

Figure 2 shows the comparison of XRD patterns, one simulated from single crystal X-ray data (Figure 2(a)) against the bulk powder of compound **1** (Figure 2(b)) and that of a typical sample of compound **1** prepared by the sonochemical process, respectively (Figure 2(c)). The comparison between these XRD patterns indicates acceptable matches with slight differences in  $2\theta$ . This finding proves the formation of compound **1** under hydrothermal and sonochemical processes.

Compound **1** is a 3D supramolecular metal-organic framework and crystallizes in the monoclinic space group  $C2/c$  and consists of a 1D coordination chain of  $\text{Mg}^{\text{II}}$  linked by  $\text{HIDC}^{2-}$ . 1D chains are engaged in hydrogen bonding interactions with coordinate water molecule and carboxylate oxygens forming 2D supramolecular sheets. These sheets are further linked by hydrogen bonds through uncoordinated imidazole nitrogen and carboxylate oxygen, forming a 3D supramolecular framework with 1D channels occupied by the

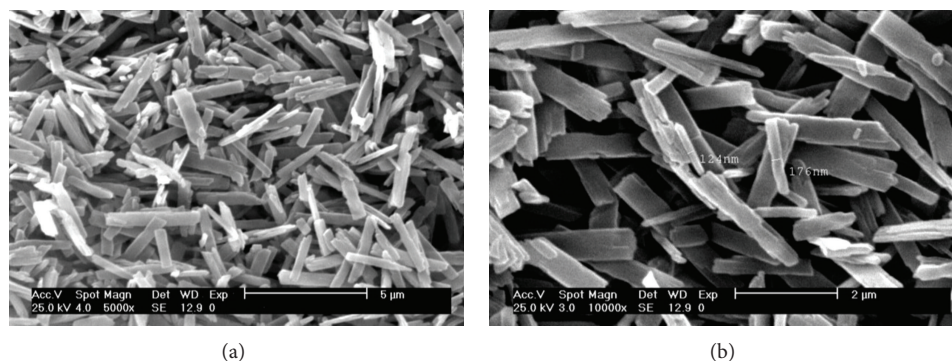


FIGURE 4: SEM images of compound 1 nanorods prepared by sonochemical process in concentration of initial reagents  $[Mg^{2+}] = [L^{3-}] = 0.05$  M.

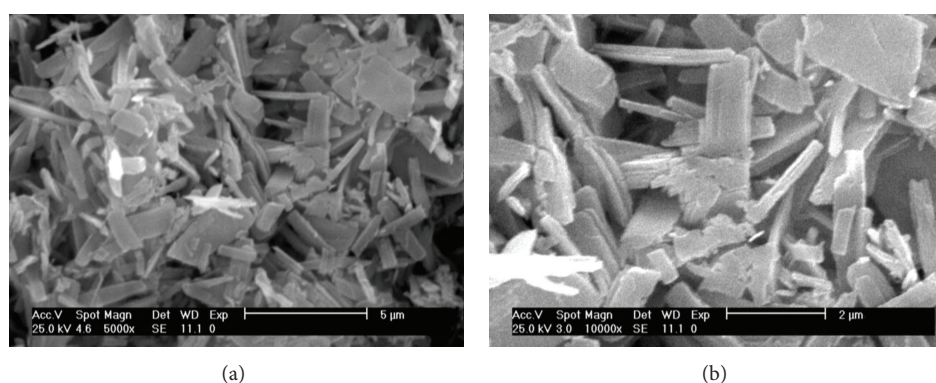


FIGURE 5: SEM images of compound 1 nanorods prepared by sonochemical process in concentration of initial reagents  $[Mg^{2+}] = [L^{3-}] = 0.025$  M.

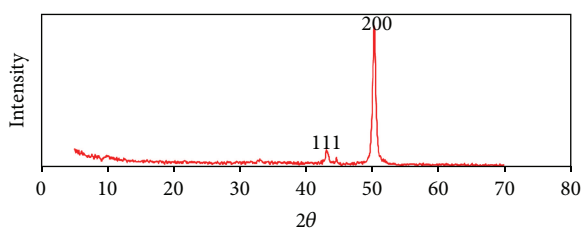


FIGURE 6: XRD pattern of MgO nanostructure prepared by calcination of compound 1 at 650°C.

water molecules. Figure 3 shows a fragment of framework 1 along the crystallographic  $c$  axis showing that Mg atoms are six coordinate [55].

The reaction between 4,5-imidazoledicarboxylic acid ( $H_3IDC$ ) and potassium hydroxide with magnesium nitrate under ultrasonic irradiation provided a crystalline nanostructure of the general formula  $\{[Mg(HIDC)(H_2O)_2] \cdot 1.5H_2O\}_n$  (**1**). The morphology and size of the compound **1** prepared by the sonochemical method were characterized by scanning electron microscopy (SEM) showing that it is composed of nanorods with sizes about 100 nm. Figure 4 shows the SEM images of the compound **1** obtained under 0.05 M concentration of  $HL^{2-}$  and  $Mg^{2+}$ . To investigate

the role of the concentration of initial reagents on the nature of products, reactions were performed with different concentrations of magnesium nitrate (0.025 M) and 4,5-imidazoledicarboxylic acid ( $H_3IDC$ ) aqueous solutions (0.025 M). The resultant SEM images are illustrated in Figure 5. Nanorods of compound **1** were obtained under both concentrations of initial reagents (Figures 4 and 5). Comparison of IR spectra and XRD patterns shows that the reaction at both concentrations of initial reagents produces the same product. However, size of the nanorods is dependent on the concentration of initial reagents as the nanostructure obtained at higher concentration of initial reagents has more uniform morphology and smaller size.

TGA data of compound **1** indicates a continuous weight loss below 650°C; this can correspond to thermal removal of the solvent molecules, decomposition of ligand, and the formation of MgO.

There is no extra weight loss above 650°C, which indicates that the compound **1** is completely transformed to MgO materials following the heat treatment process at 650°C [46]. Figure 6 provides the XRD pattern of the residue obtained from calcination of compound **1**. The obtained pattern matches with the standard pattern of cubic MgO with the lattice parameters  $a = 4.2112$  Å,  $Z = 4$ , and S.G = Fm3m which is the same as the reported values (JCPDS card number

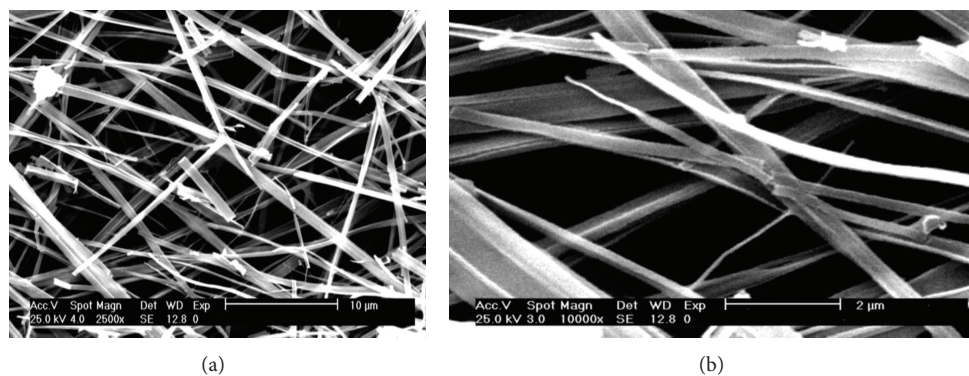


FIGURE 7: SEM image of MgO nanobelts prepared by calcination of bulk powder of compound **1** at 650°C.

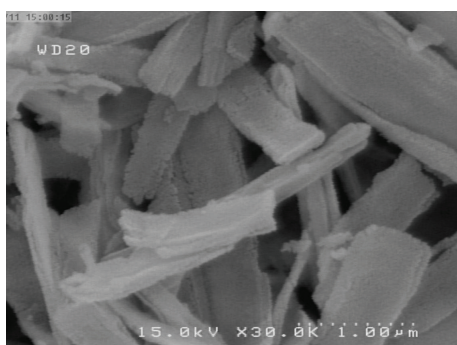


FIGURE 8: SEM image of MgO nanostructure prepared by calcination of compound **1** nanostructure at 650°C.

45-0946). No characteristic peaks of impurities are detected in the XRD pattern.

Figure 7 shows the SEM images of MgO nanobelts obtained from calcination of bulk powder of compound **1** at 650°C. As the calcination process was successful for the preparation of MgO nanobelts, the nanostructured compound **1** prepared by the sonochemical process was also calcinated at 650°C. Figure 8 shows the SEM image of the resulting MgO nanostructure. The XRD pattern of the residue shows that the resulting residue was again MgO with the same lattice parameters which are mentioned above. Comparison between resulting SEM images (Figures 7 and 8) indicates that the size of the coordination polymeric precursor correlates to the size and morphology of the formed MgO nanostructures.

## 5. Conclusions

In conclusion, we have exhibited an efficient, low cost, and environmentally friendly route to produce a 3D supramolecular metal-organic framework (MOF) based on Mg<sup>II</sup>,  $\{[\text{Mg}(\text{HIDC})(\text{H}_2\text{O})_2] \cdot 1.5\text{H}_2\text{O}\}_n$  (**1**) ( $\text{H}_3\text{L} = 4,5\text{-imidazole-dicarboxylic acid}$ ) by using ultrasonic method. Nanocrystals of compound **1** were prepared by using ultrasonic method and characterized by scanning electron microscopy, X-ray powder diffraction, IR spectroscopy, and elemental analyses. To prepare the nanostructure of compound **1**, two different

concentrations of initial reagents were tested. Nanorods of compound **1** were obtained under both concentrations. Results show an increase in the nanostructure size as the concentration of initial reagents has decreased. Calcination of compound **1** at different sizes produced nanostructures of MgO. Size and morphology of the MgO nanostructures depend on the initial particles size of compound **1**.

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