



Synthesis and structural characterization of five zinc bisphosphonate compounds



Ai-Yun Ni, Jie Pan, Zhen-Zhen Xue, Song-De Han, Jin-Hua Li, Guo-Ming Wang*, Zong-Hua Wang

College of Chemistry and Chemical Engineering, Collaborative Innovation Center for Marine Biomass Fiber Materials and Textiles, Qingdao University, Shandong 266071, China

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ABSTRACT

The hydrothermal reaction of zinc ions with 1-hydroxyethylidene-1,1-diphosphonic acid (HEDP) in the presence of additives such as 1,4-diazabicyclo[2.2.2]octane (DABCO), 1,3-propanediamine, imidazole and isonipecotic acid, afforded five metal phosphonates, namely, $[\text{H}_2\text{DABCO}]\{\text{Zn}_3[\text{CH}_3\text{C}(\text{OH})(\text{PO}_3)_2]_2\}$ (**1**), $[\text{C}_3\text{H}_{14}\text{N}_2]\{\text{Zn}_3[\text{CH}_3\text{C}(\text{OH})(\text{PO}_3)_2]_2\}$ (**2**), $[\text{C}_3\text{H}_5\text{N}_2]_2\{\text{Zn}_3[\text{CH}_3\text{C}(\text{OH})(\text{PO}_3)_2]_2\}$ (**3**), $\{\text{Zn}_2[\text{CH}_3\text{C}(\text{OH})(\text{PO}_3)_2][\text{C}_3\text{H}_4\text{N}_2]_2\}$ (**4**) and $\{\text{Zn}_2[\text{CH}_3\text{C}(\text{OH})(\text{PO}_3)_2][\text{C}_6\text{H}_{11}\text{NO}_2]_2[\text{H}_2\text{O}]_2\cdot\text{H}_2\text{O}$ (**5**). Compounds **1** and **2** are isostructural and adopt a pillar-layered 3D structure. Compound **3** is a 3D framework in which $[\text{ZnO}_4]$ tetrahedra are connected by HEDP molecules with tetradentate mode. The $[\text{ZnO}_3\text{N}]$ tetrahedra in **4** shares corners with the $[\text{CPO}_3]$ tetrahedra into a 3D framework with twelve-membered ring. It is also found that **4** displays interesting left-handed helical chains along $[100]$ direction. In **5**, HEDP molecules exhibit tetradentate coordination fashion to combine Zn atoms into a complicated 2D hybrid layer, resulting in the coordinated isonipecotic acid rings being sandwiched between the layers. All the compounds have been characterized by infrared spectra (IR), elemental analyses, powder X-ray diffraction (PXRD) and thermogravimetric (TG) analyses.

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1. Introduction

Metal phosphonates have been the focus of research due to their structural and compositional diversity, as well as their thermal and chemical stability for potential applications in the field of optics, catalysis, magnetism, gas storage/separation, and so on [1–10]. Compared with the carboxylic, pyridyl, imidazolyl groups broadly used in the generation of functional coordination polymers (CPs) [11–14], the phosphonic group $-\text{PO}_3\text{H}_2$ has three potential coordinating sites which allows it to bind to metal centers with more flexible coordination modes [1–10]. However, owing to the presence of insoluble phases in the assembly process, it is challenging to prepare crystalline metal phosphonates with high quality for acquiring their precise molecular structures and determining their properties [15–17].

During the past few years, a common strategy has been carried out to improve the solubility and crystallinity of metal

phosphonates by modifying phosphonic acids with functional groups, such as hydroxyl, carboxyl, methyl, amino, pyrazine, sulfonyl and thiophene [18–23]. As a result, many intriguing metal phosphonates have been synthesized and structurally characterized, for instance, two zinc phosphonates containing a potential capability for sensing UV radiation [24], a series of uranyl phosphonates exhibiting typical green-light emission originating from the metal centers [25], as well as two new layered zirconium phosphonates functionalized with amino groups displaying unprecedented topologies [26]. On the other hand, to fabricate new metal phosphonates with novel structures and properties, SDAs (structure-directing agents) or coligands such as aliphatic amine, imidazole, pyridine and their derivatives have usually been used [11,27–29]. Considering the steric and electronic effects together with the differential coordination tendency of the SDAs or coligands for metal centers, the resulting metal phosphonates directed or decorated by SDAs or coligands may present unique structures and/or captivating properties [30–32].

Taking inspiration from the aforementioned points, we decided to extend our research to 1-hydroxyethylidene-1,1-diphosphonic acid (HEDP). Besides one methyl and one hydroxyl, HEDP

* Corresponding author.

E-mail address: gmwang_pub@163.com (G.-M. Wang).

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