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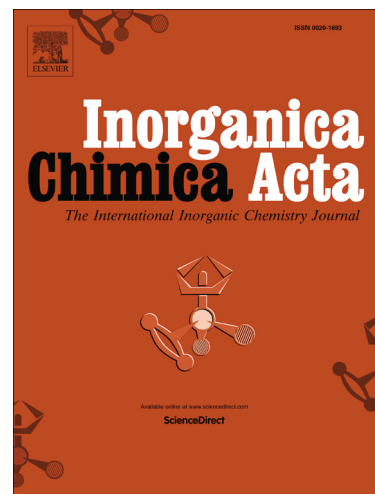
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Two novel magnesium-based metal–organic frameworks: structure tuning from 2D to 3D by introducing the auxiliary ligand of acetate

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ABSTRACT

By the reaction of 4,4',4''-benzene-1,3,5-triyl-tribenzoate and magnesium nitrate in a mixture of DMA/H₂O, a 2D coordination network was solvothermally synthesized. When an auxiliary ligand of acetate was introduced into the reaction system for the 2D complex, a 3D metal–organic framework was successfully obtained. The layered structure features a linear Mg₃ cluster as node which is terminated by four coordinated DMA molecules. However, the DMA molecules were substituted by acetate with stronger coordination ability, resulting in an infinite Mg(COO)₂ chain. Then a 3D architecture was finally formed. These two compounds also exhibit strong solid fluorescent emissions.

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1. Introduction

In the past two decades, metal–organic frameworks (MOFs) or porous coordination polymers (PCPs) have been attracting a great deal of attention because of their aesthetically pleasing structures [1–3], and promising application in gas storage [4,5], separation [6,7], drug delivery [8,9] and catalysis [10,11]. Obviously, these properties can be ascribed to their distinctive structures. Herein, 3D frame structures are undoubtedly their outstanding features which can easily afford appropriate inner spaces and active sites [12]. However, organic ligands and metal ions sometimes inevitably self-assemble into some 2D structures. So how to construct 3D stable architectures from 2D layers has become the study topic of some researchers.

In this regard, there are many approaches to forming the 3D structures. For example, the use of different solvent mixture and stoichiometry of reactants [13], changing the reaction temperature [14], altering the synthetic procedure [15], and many other factors [16] can all achieve this goal. Exploring suitable conditions to have 2D networks polycatenate, polyrotaxane, or interpenetrate is also an often used method [17–20]. But the more effective strategy is making 2D layers pillared by a rigid ligand, which can better show the experimenter's design intention [21–23]. Inspired by the employment of mixed linker ligands in pillaring strategy [24], we thought if some kind of small molecule or ion with good coordination ability was introduced into the reaction system, it would probably change the coordination environment for 2D sheets. Then 3D architectures might be constructed with the mixed ligands.

To this end, we selected two ligands and tried to make them react with a metal cation. One ligand is a long and rigid trimesic acid derivative, 4,4',4''-benzene-1,3,5-triyl-tribenzoic acid (H₃BTB). It is a good linker for

constructing metal coordination polymers, which has been demonstrated in many literatures [26–30]. Another ligand is a small carboxylate of acetate. It has a great affinity to various metal ions and so most likely changes coordination circumstance of metal ions [31]. As a type of low-cost, non-toxic and abundant metal ion, magnesium(II) ion was selected as connectors for constructing MOFs. Upon using such a light cation, the formation of lightweight materials is expected with improved gas storage capacity [32]. Nevertheless, examples of Mg-based MOFs are very limited compared with transition metal ones [33].

In this work, we adopt H₃BTB to react with magnesium nitrate, in pure or acetate-contained *N,N'*-dimethylacetamide (DMA). As a result, two different crystal products, [Mg₃(BTB)₂(DMA)₄]·4DMA·2H₂O (**1**) and [Mg₃(BTB)(CH₃COO)₃(H₂O)₃]·3.5DMA·1.5H₂O (**2**), were successfully obtained under the above two conditions respectively. Compound **1** possesses 2D network whereas **2** presents 3D framework.

2. Experimental

2.1 Materials and methods

All commercial reagents were used without further purification. The precursor H₃BTB was prepared according to the previously reported procedure [34]. IR spectra were recorded from KBr pellets in the range of 4000–400 cm⁻¹ on a VECTOR 22 spectrometer. Elemental analyses were performed with a Perkin-Elmer 240C elemental analyzer. Thermogravimetric analyses were carried out on a NETZSCH STA 449F3 instrument from room temperature to 1000 °C with a heating rate of 20 °C/min in N₂ atmosphere. Powder X-ray diffraction patterns were obtained on a Bruker D8 Advance X-ray diffractometer using Cu K α radiation (0.15418 nm), in which the X-ray tube was operated at 40 kV and 40 mA. UV–Vis absorption spectra were taken on a U-4100

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