Hydrothermal Synthesis of a new Cd-MOF

Zeynab Golshani, Faranak Manteghi*

Department of Chemistry, Iran University of Science and Technology, Tehran, Iran

*f_manteghi@iust.ac.ir

Abstract

In this study, a new Cd-based Metal Organic Framework (MOF) is synthesized. Nanopowder of MOF was prepared using solvothermal method. To identify MOF, methods including powder X-ray diffraction (PXRD), scanning electron microscopy (SEM) and Fourier transform infrared spectroscopy (FT-IR) were applied.

1. Introduction

MOFs are constructed by metal ions/clusters and organic bridging molecules. They can be regarded as a subclass of coordination polymers, since the metal ions/clusters should be solely bridged by organic molecules (or ligands) via coordination into two-dimensional (2D) or three-dimensional (3D) porous networks in MOFs, whereas the bridging ligands may be either inorganic or organic in coordination polymers [1]. Exceptionally high porosity, crystallinity, compositional and structural variability, large surface area and acceptable thermal and mechanical stability of metal-organic frameworks (MOFs) make them ideal materials to satisfy the needs of various applications such as gas and vapour sorption, chemical separation, catalysis, magnetism, luminescence, drug storage and delivery [2,3]. There are many systematic and facile synthetic routes such as hydro/solvothermal technique, microwave, electrochemical and mechanochemical, etc. Hydrothermal method is used more than any other way due to its high speed [4]. Solvothermal synthesis is the most straightforward synthesis method; a substrate or particle is placed in the MOF precursor solution, and the synthesis progresses similarly to the standard synthesis for that MOF [5].

2. Experimental

2.1. Materials

In this study, $Cd(NO_3)_2 \cdot 4H_2O$, terephthalic acid ($BDCH_2$), pyridine-4-carboxylic acid (PCA), DMF and Ethanol were purchased from Merck Co. and used to prepare the sample.

2.2. Preparation of MOF

Synthesis of $[Cd(BDC)_{0.5}(PCA)]$ was prepared by reacting 1mmol $Cd(NO_3)_2 \cdot 4H_2O$, 1mmol pyridine-4-carboxylic acid (PCA), and 0.5 mmol benzene-1,4-dicarboxylic acid ($BDCH_2$) in 50 ml DMF−EtOH (1:1) by the solvothermal technique. The autoclave was heated under autogenous pressure to 120°C for 2 days and then cooled to RT in a 24 h period.

3. Results and Discussion

Fig.1 shows the XRD pattern of synthesized sample by solvothermal method. Due to the high intensity of the peaks and a peak that appears in 2Θ below 10 degrees, we concluded a high porosity of the obtained MOF.

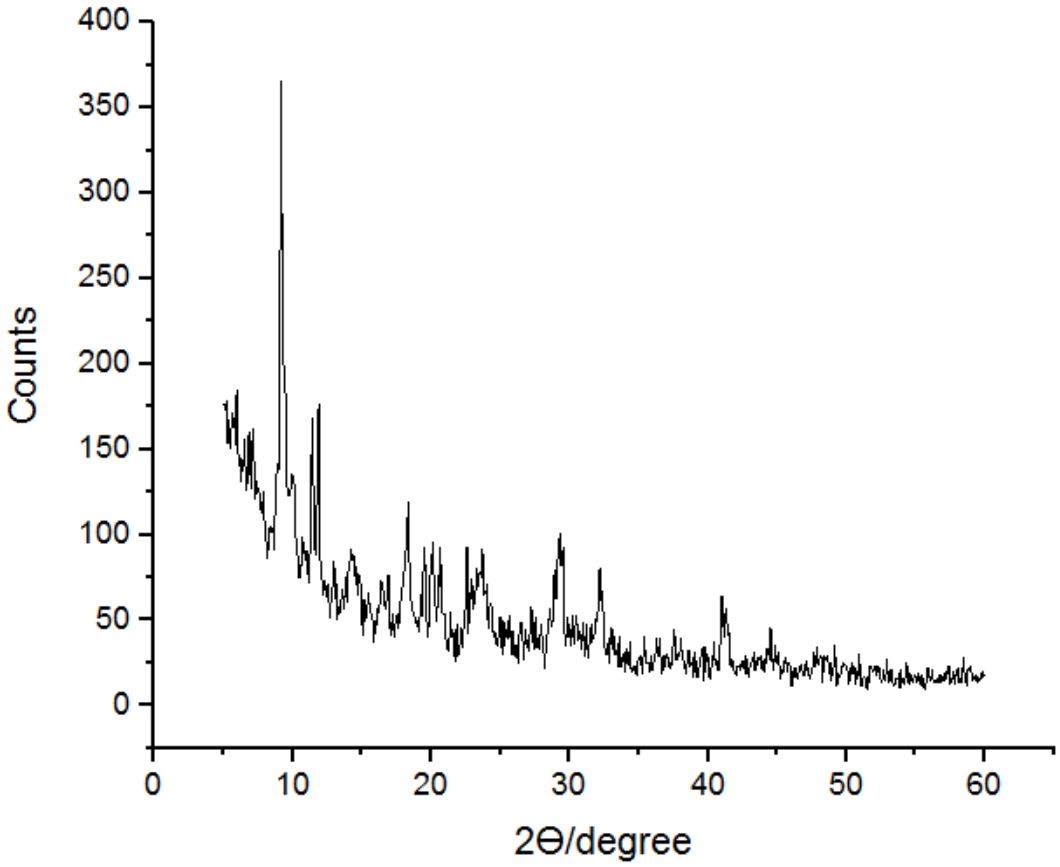
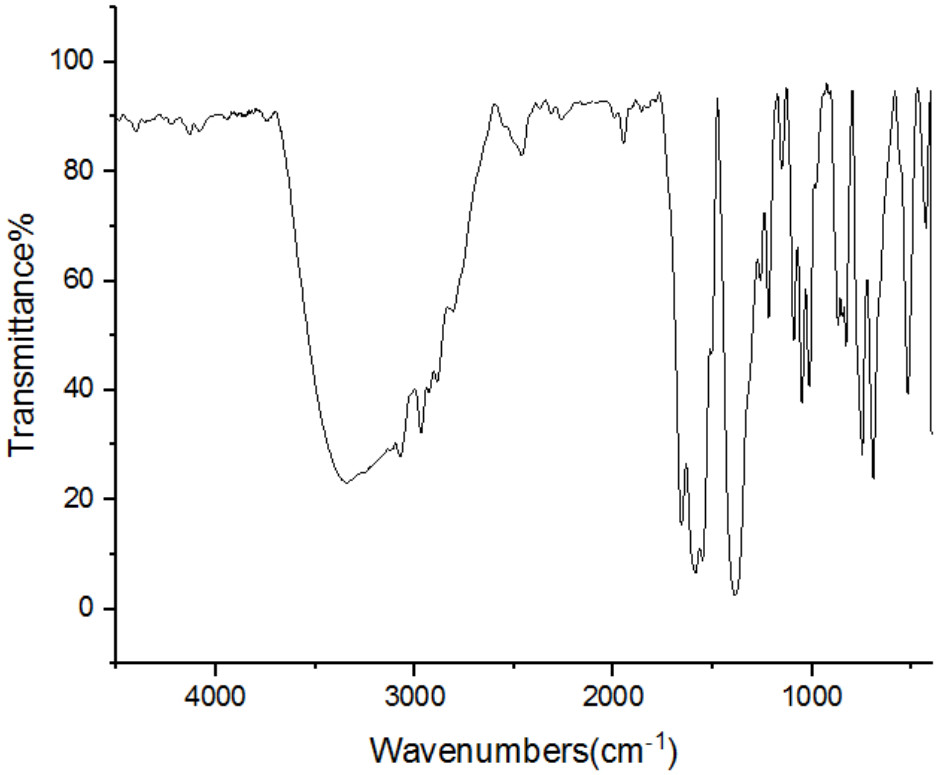


Fig.1. XRD pattern of the Cd-MOF

In Fig. 2, the FTIR spectrum of synthesized sample is shown**.** The broad peak at 3500-2800 cm^{-1} is attributed to C-H and O-H bonds of the two ligands. The characteristic C=O vibrations at 1650 cm^{-1} indicate the existence of carboxylate in PCA and BDC.

In Fig. 3, the SEM image of MOF particles is illustrated. The particles are bimorph, some of them seem to be hollow long microtubes with varying size of 0.84 to 1.35 μm, and others look like nuts, probably in microsize, too. It is notable that all numbers given in Fig. 3, are the diameters of tubes.



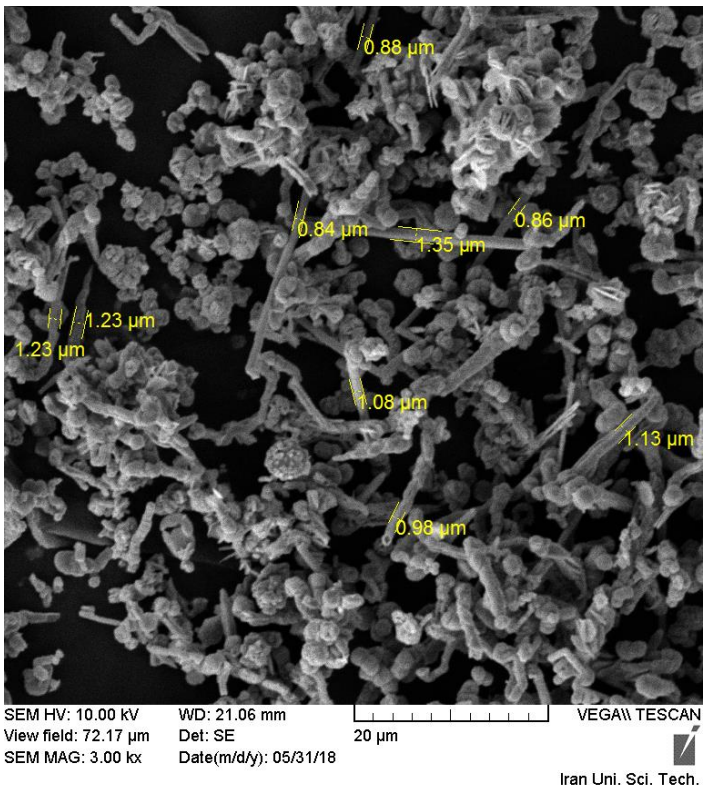


Fig. 3. SEM image of Cd-MOF

Conclusion

A new Cd-MOF was successfully synthesized by a facile solvothermal method. We have obtained the MOF by a cadmium salt and terephthalic acid and pyridine-4-carboxylic acid in DMF/ethanol. It is characterized by a variety of methods including PXRD, which proves the formation of MOF porous structure, SEM which shows the bimorph particles with micrometer size and FTIR which identifies the presence of organic ligands and metal to ligand bonds in the structure .

References

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