

一个基于 $[\text{Cu}_4(\mu_2\text{-OH})_2\text{N}_{12}]$ 的10-连接3D配合物的合成、结构和光催化性质

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摘要: 通过扩散法合成了一个新的配位聚合物 $\{[\text{Cu}_2(\text{OH})(\text{btre})_{1.5}(1,2,4\text{-bte})] \cdot 13\text{H}_2\text{O}\}_n$ ($\mathbf{1} \cdot 13\text{H}_2\text{O}$) (btre=1,2-二(4*H*-1,2,4-三唑)乙烷, 1,2,4-bte=1,2,4-苯三甲酸根)。测试了 $\mathbf{1} \cdot 13\text{H}_2\text{O}$ 的晶体结构,并用红外光谱、元素分析、粉末X射线衍射对其进行表征。单晶X射线衍射表明 $\mathbf{1}$ 是基于四核铜簇 $[\text{Cu}_4(\mu_2\text{-OH})_2\text{N}_{12}]$ 构筑的10-连接的3D框架,其拓扑符号为 $3^{12} \cdot 4^{28} \cdot 5^5$ 。研究了 $\mathbf{1} \cdot 13\text{H}_2\text{O}$ 的热稳定性以及对甲基橙的催化降解作用。

关键词: 10-连接网络; 铜配位聚合物; 光催化降解; 1,2-二(4*H*-1,2,4-三唑)乙烷

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Synthesis, Structure and Photocatalytic Properties of a 10-Connected 3D Coordination Polymer Based on $[\text{Cu}_4(\mu_2\text{-OH})_2\text{N}_{12}]$

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Abstract: One new coordination polymer, $\{[\text{Cu}_2(\text{OH})(\text{btre})_{1.5}(1,2,4\text{-bte})] \cdot 13\text{H}_2\text{O}\}_n$ ($\mathbf{1} \cdot 13\text{H}_2\text{O}$) (btre=1,2-bis(4*H*-1,2,4-triazole)ethane, 1,2,4-bte=1,2,4-benzenetricarboxylate), was synthesized under room temperature condition and characterized by single-crystal X-ray diffraction, elemental analyses, IR spectroscopy and powder X-ray diffraction. Single-crystal X-ray structural analysis shows that compound $\mathbf{1}$ features a 3D framework based on the tetranuclear $[\text{Cu}_4(\mu_2\text{-OH})_2\text{N}_{12}]$. The 3D coordination network of $\mathbf{1}$ is a 10-connected network with $(3^{12} \cdot 4^{28} \cdot 5^5)$ topology based on the tetranuclear copper cluster nodes. The thermogravimetric (TG) analysis and photocatalytic degradation of methyl orange (MO) of $\mathbf{1} \cdot 13\text{H}_2\text{O}$ were studied. CCDC: 1526760.

Keywords: 10-connected network; copper(II) coordination polymer; photocatalysis; 1,2-bis(4*H*-1,2,4-triazole)ethane

0 Introduction

In recent years, the design and construction of functional coordination polymers, containing metal ions or metal clusters and bridging organic ligands,

have developed rapidly because of their potential applications in magnetism, photoluminescence, catalysis and interesting topological structure^[1-4].

Topology is a very important subject and a useful tool in the crystal engineering, which can simplify the

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complicated structure of coordination polymers (CPs). The simple 3-, 4-, and 6-connected nets are very common and can not meet the demand of complicated network topology. So, the study on synthesis, properties of complexes containing highly connected nodes is more attractive. But, up to now, only a few examples of 9-, 10- and 12- connected networks have been reported because that the central metal ions have limited coordination sites and most organic ligands have steric hindrance. In order to solve this problem, using metal clusters instead of transition metal ions as nodes can provide more coordination sites^[5-11]. Most of the clusters in coordination polymers are synthesized by oxo-centered metal clusters. For example, $[\text{Cu}_4(\mu_3\text{-OH})_2]$ tetranuclear copper(II) clusters are observed in several compounds^[12-14]. But, in some coordination polymers, hydroxyl group and carboxyl or triazole group induce the aggregation of some clusters with special properties, simultaneously, such as $[\text{Co}_4(\mu_2\text{-H}_2\text{O})_2(\text{COO})_6]$ cluster^[7], $[\text{Cu}_4(\mu_2\text{-H}_2\text{O})_2\text{N}_{20}]$ cluster^[15].

N-donor ligands play an important role in synthesis of CPs or metal-organic frameworks (MOFs). A large number of coordination polymers have been synthesized based on pyridine, imidazole and triazole derivative ligands in the past decades, such as 1,2-di(4-pyridyl)ethylene (bpe), 1,2-bis(imidazol-1-ylmethyl)benzene (bimb)^[16], 1,4-bis(2-methyl-imidazol-1-yl)butane (bib)^[17-18], 1,2-bis(1,2,4-triazol-1-yl)ethane (bte)^[19], 1,4-bis(1,2,4-triazol-1-yl)butane (btb)^[20], 1,4-bis(1,2,4-triazol-1-ylmethyl)-2,3,4,5-tetramethyl benzene (tmtz)^[21] and 1,4-bis(1,2,4-triazol-1-ylmethyl)benzene (bbtz)^[22]. Comparatively, the 4-substituted-1,2,4-triazole derivatives have more coordination sites. We have synthesized some coordination polymers based on the rigid 4-substituted-bis(1,2,4-triazole) ligand 1,4-bis(4*H*-1,2,4-triazole)benzene (btx)^[23], the flexible ligand 1,2-bis(4*H*-1,2,4-triazole)ethane (btre)^[24-26], 1,4-bis(4*H*-1,2,4-triazole)benzene (btrb)^[27] and a long and semi-rigid ligand bis(4-(4*H*-1,2,4-triazole)phenyl)methane (btpm)^[28].

With this background information, one new coordination polymer $\{[\text{Cu}_2(\text{OH})(\text{btre})_{1.5}(1,2,4\text{-btc})] \cdot 13\text{H}_2\text{O}\}_n$ (**1**·13H₂O) was synthesized (1,2,4-btc=1,2,4-benzenetricarboxylate). Compound **1** shows an unusual 10-con-

nected three-dimensional metal-organic framework based on the tetranuclear copper clusters $[\text{Cu}_4(\mu_2\text{-OH})_2\text{N}_{12}]$.

1 Experimental

1.1 Materials and methods

The ligand btre was synthesized according to the literature method^[29]. All other reagents (such as $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$, 1,2,4-benzenetricarboxylic acid, methanol) were of analytical grade and used without further purification. Elemental analyses for C, H and N was performed on a Perkin-Elmer 240C analyzer. The IR spectra was obtained using KBr pellets on a Nicolet iS10 spectrophotometer in the 4 000~400 cm^{-1} region. Powder X-ray diffraction (PXRD) were performed on a D/MAX-3C diffractometer with the Cu $K\alpha$ radiation ($\lambda=0.154\ 06$ nm, $U=40$ kV, $I=40$ mA) over the 2θ range of $5^\circ\sim 50^\circ$ at room temperature. TGA was carried out using a Thermal Analyst 2100 TA Instrument and SDT 2960 Simultaneous TGA-DTA Instrument in flowing dinitrogen at a heating rate of $10\ \text{^\circ C} \cdot \text{min}^{-1}$. UV-Vis spectrum was measured using a U-3900 spectrophotometer.

1.2 Preparation of $\{[\text{Cu}_2(\text{OH})(\text{btre})_{1.5}(1,2,4\text{-btc})] \cdot 13\text{H}_2\text{O}\}_n$ (**1**·13H₂O)

A solution of 1,2,4-benzenetricarboxylic acid (0.2 mmol) in 5 mL H₂O was adjusted to pH 6 with $1.0\ \text{mol} \cdot \text{L}^{-1}$ NaOH solution and btre (0.2 mmol) dissolved in 2 mL H₂O was added. The mixture was placed at the bottom of glass tube. $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ (0.4 mmol) dissolved in 5 mL methanol was placed on top of glass tube. The ligands (1,2,4-btc, btre) and CuCl_2 were separated by the mixture of methanol and H₂O (1:1, *V/V*). Two weeks later, the blue single crystals of **1**·13H₂O were obtained (0.090 8 g, Yield: 82% based on btre). Anal. Calcd. for $[\text{C}_{18}\text{H}_{16}\text{Cu}_2\text{N}_9\text{O}_7(\mathbf{1})+13\text{H}_2\text{O}]$ (%): C, 25.97; H, 5.05; N, 15.15. Found(%): C, 26.01; H, 4.98; N, 15.16. IR data (cm^{-1}): 3 429s, 3 121w, 1 582m, 1 487w, 1 384m, 1 198w, 1 170w, 1 128w, 1 079w, 1 023w, 772w, 647w.

1.3 Crystal structure determination

The diffraction data of **1**·13H₂O was collected on Rigaku Mercury CCD diffractometer with graphite monochromated Mo $K\alpha$ ($\lambda=0.071\ 073$ nm) radiation. Intensities were collected by the φ - ω scan technique. The structure was solved and refined by the SHELXTL

package^[30]. All non-hydrogen atoms were refined anisotropically and hydrogen atoms were determined with theoretical calculations and refined isotropically. The lattice water of **1**·13H₂O is highly disordered and could not be modeled properly and was removed by the SQUEEZE routine in PLATON^[31]. The number of lat-

tice water molecules for **1**·13H₂O was deduced from the TGA and elemental analysis. The parameters of the crystal data collection and refinement of **1** are given in Table 1. Selected bond lengths and bond angles of **1** are listed in Table 2.

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Table 1 Crystallographic collection and refinement parameters of **1**

Formula	C ₁₈ H ₁₆ Cu ₂ N ₉ O ₇	<i>Z</i>	4
Formula weight	597.48	<i>D_c</i> / (g·cm ⁻³)	1.003
<i>T</i> / K	293.15	<i>μ</i> / mm ⁻¹	1.11
Crystal system	Monoclinic	Reflection collected	31 322
Space group	<i>P</i> 2 ₁ / <i>n</i>	Unique reflection	7 223 (<i>R</i> _{int} =0.077 5)
<i>a</i> / nm	1.383 6(4)	Parameter	374
<i>b</i> / nm	2.251 1(5)	GOF on <i>F</i> ²	1.089
<i>c</i> / nm	1.397 6(4)	<i>R</i> ₁ , <i>wR</i> ₂ [<i>I</i> >2σ(<i>I</i>)]	0.065 5, 0.148 0
<i>β</i> / (°)	114.602(7)	<i>R</i> ₁ , <i>wR</i> ₂ (all data)	0.084 6, 0.155 6
<i>V</i> / nm ³	3.958 1(2)	(Δρ) _{max} , (Δρ) _{min} / (e·nm ⁻³)	460, -460
<i>F</i> (000)	1 204		

Table 2 Selected bond distances (nm) and angles (°) for **1**

Cu(1)-O(1)	0.196 9(3)	Cu(1)-O(7)	0.191 9(3)	Cu(1)-N(8)A	0.238 2(4)
Cu(1)-N(1)	0.199 3(4)	Cu(1)-N(4)	0.197 8(4)	Cu(2)-O7	0.192 2(3)
Cu(2)-O(5)B	0.199 1(3)	Cu(2)-N5C	0.235 1(4)	Cu(2)-N(9)D	0.198 1(4)
Cu(2)-N(2)	0.200 1(4)				
O(1)-Cu(1)-N(1)	88.46(15)	O(1)-Cu(1)-N(4)	92.73(15)	O(7)-Cu(1)-N(1)	86.46(14)
O(7)-Cu(1)-N(4)	91.89(14)	O(7)-Cu(1)-N(8)A	103.98(13)	N(1)-Cu(1)-N(8)A	94.92(17)
N(4)-Cu(1)-N(1)	176.96(17)	N(4)-Cu(1)-N(8)A	87.96(15)	O(5)B-Cu(2)-N(2)	89.72(15)
O(5)B-Cu(2)-N(5)C	84.04(14)	O(7)-Cu(2)-N(5)B	170.86(14)	O(7)-Cu(2)-N(2)	85.96(14)
O(7)-Cu(2)-N(5)C	104.38(13)	O(7)-Cu(2)-N(9)D	90.91(14)	N(2)-Cu(2)-N(5)C	95.65(17)
N(9)D-Cu(2)-O(5)B	92.73(14)	N(9)D-Cu(2)-N(2)	174.49(18)	N(9)D-Cu(2)-N(5)C	89.52(15)

Symmetry codes: A: 1/2+*x*, 1/2-*y*, 1/2+*z*; B: 3/2-*x*, 1/2+*y*, 3/2-*z*; C: 2-*x*, 1-*y*, 1-*z*; D: 3/2-*x*, 1/2+*y*, 1/2-*z*; E: 2-*x*, 1-*y*, 2-*z*.

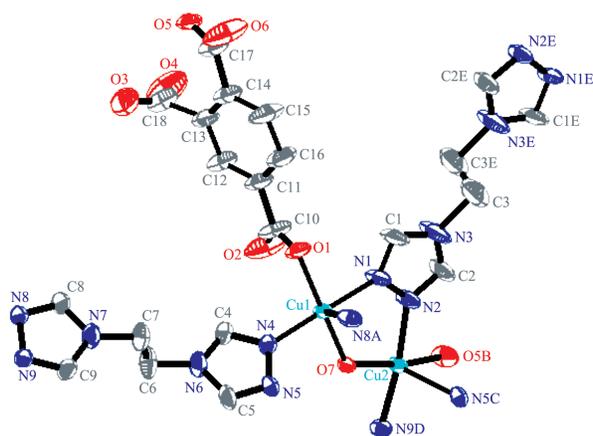
2 Results and discussion

2.1 Crystal structure description of [Cu₂(OH)(btre)_{1.5}(1,2,4-btc)]_n (**1**)

The asymmetric unit of **1** consists of two kinds of Cu(II) ions (Cu(1) and Cu(2)), one μ₂-OH group (O(7)), one 1,2,4-btc, one and half of btre ligands. Each Cu(II) ion is five-coordinated by one carboxylate oxygen atom from 1,2,4-btc and one μ₂-OH (O(7)) and three triazole nitrogen atoms from three btre ligands, and adopts a square-pyramidal geometry with a [CuO₂N₃] chromophore. N(8)A and N(5)C are located at vertex positions

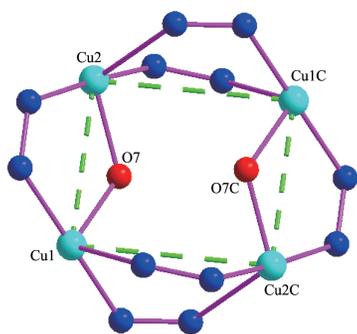
of the square-pyramidal geometry of Cu(1) and Cu(2), respectively. The Cu-O bond lengths are in a range of 0.191 9(3)~0.199 2(4) nm and the Cu-N bond lengths vary from 0.197 9(4) to 0.238 2(5) nm, which is similar to those observed in the other Cu-O/N compound constructed by btre ligand^[12] (Fig.1).

The Cu(1) and Cu(2) are bridged by one μ₂-OH group (O(7)) and one μ₂-triazole ring of btre ligand to form a dinuclear [Cu₂(μ₂-OH)N₂] cluster. Two dinuclear [Cu₂(μ₂-OH)N₂] units are bridged by four triazole rings giving a tetranuclear [Cu₄(μ₂-OH)₂N₁₂] (Fig.2). In tetranuclear cluster, the μ₂-OH group (O(7)) connects two



All H atoms are omitted for clarity; 50% thermal ellipsoids; Symmetry codes: A: $1/2+x, 1/2-y, 1/2+z$; B: $3/2-x, 1/2+y, 3/2-z$; C: $2-x, 1-y, 1-z$; D: $3/2-x, 1/2+y, 1/2-z$; E: $2-x, 1-y, 2-z$

Fig.1 Coordination environment of the Cu(II) atom in **1**



Symmetry code: C: $2-x, 1-y, 1-z$

Fig.2 Tetranuclear copper cluster in **1**

different crystallographic Cu (II) (Cu(1), Cu(2)) with bond lengths of 0.191 9(3) and 0.192 2(3) nm, respectively, and deviates from the Cu(II) ions plane (Cu(1), Cu(2), Cu(1)C, Cu(2)C) over 0.055 58 nm. The Cu(1)-O(7)-Cu(2) bond angle is $125.011(1)^\circ$. The distance between Cu(1) and Cu(2) is 0.340 74(7) nm. Cu(1) and Cu(2)C are bridged by two triazole groups with a distance of 0.395 37(7) nm.

There is one kind of 1,2,4-btc ligand in **1**. 1,4-Carboxylate groups (O(1)O(2), O(5)O(6)) of 1,2,4-btc ligand bridge two Cu(II) ions from two different tetranuclear Cu(II) clusters. The third carboxylate group (O(3)O(4)) is free. Each btre ligand exhibits the anti-conformation and bridges two tetranuclear copper(II) clusters. Each tetranuclear $[\text{Cu}_4(\mu_2\text{-OH})_2\text{N}_{12}]$ cluster connects ten adjacent clusters through four 1,2,4-btc and six btre ligands to yield an unusual 3D framework, as

shown in Fig.3 and 4.

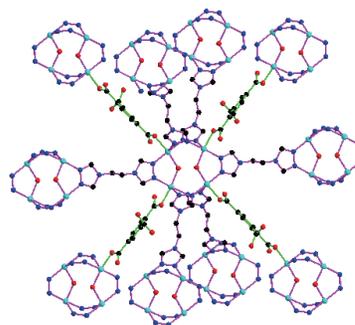


Fig.3 Tetranuclear copper cluster surrounded by ten neighboring tetranuclear copper clusters through six btre ligands and four 1,2,4-btc ligands

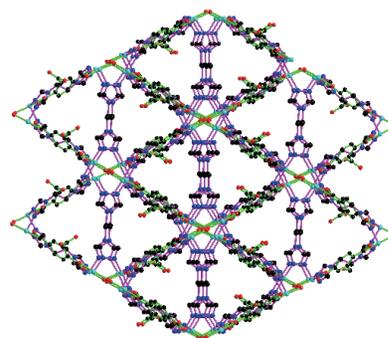
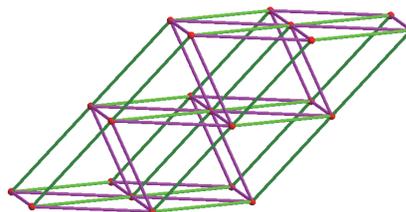


Fig.4 Three dimensional framework in **1**

As depicted in Fig. 3, each tetranuclear copper cluster is connected to ten adjacent tetranuclear copper clusters through four 1,2,4-btc and six btre ligands. Topologically, the tetranuclear copper cluster is simplified as one 10-connected node. The 1,2,4-btc and btre ligands are 2-connected linkers. The structure of **1** can be described as a 10-connected 3D network with the $3^{12}\cdot 4^{28}\cdot 5^5$ topology (Fig. 5). This network is identified by the code *bct*. The same topology has been reported in the literature^[8]. In addition, some of other 10-connected complexes have different topological struc-



Red ball: 10-connected copper clusters; Bright green and pink line: 2-connected 1,2,4-btc and btre ligands, respectively

Fig.5 Three 10-connected *bct* topological network in **1**

tures with Schläfli symbol of $3^6 \cdot 4^{34} \cdot 5^3 \cdot 6^2$ [7,32-33], $4^4 \cdot 8^{32} \cdot 12^9$ [34].

2.2 Thermogravimetric analysis of $1 \cdot 13\text{H}_2\text{O}$

In order to characterize the purity of compound $1 \cdot 13\text{H}_2\text{O}$, the as-synthesized sample was measured by X-ray powder diffraction at room temperature. As shown in Fig. 6, the peak positions of the measured patterns are in good agreement with the simulated patterns, indicating the high purity of the sample. In order to characterize the thermal stability of compound $1 \cdot 13\text{H}_2\text{O}$, the thermal behavior was investigated by TG in dry nitrogen atmosphere from room temperature to 600 °C with a heating rate of 10 °C \cdot min⁻¹. In the TG curve of $1 \cdot 13\text{H}_2\text{O}$ (Fig. 7), the weight lost in the range of 40~121 °C is attributed to the release of lattice water molecules (Calcd. 28.19%, Obsd. 28.18%). The anhydrous substance was stable upon heating to 211 °C. Then the weight decrease happened rapidly and did not end until to 425 °C. The framework of **1** was collapsed from 211 to 425 °C. The residue might be CuO (Calcd.:

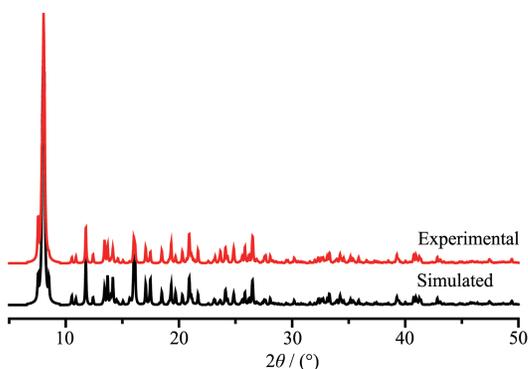


Fig.6 Simulated and experimental PXRD patterns of $1 \cdot 13\text{H}_2\text{O}$

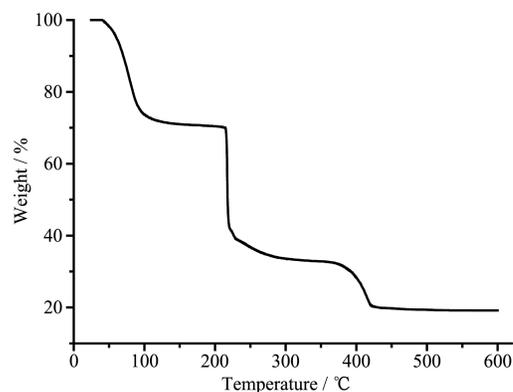


Fig.7 TG curve of $1 \cdot 13\text{H}_2\text{O}$

19.15%, Obsd. 19.16%).

2.3 Catalytic properties of $1 \cdot 13\text{H}_2\text{O}$

Photocatalytic degradation has attracted more and more research interest because of its potential application in the purification of wastewater [4,35-41]. The photocatalysis activity of compound $1 \cdot 13\text{H}_2\text{O}$ was evaluated in purifying waste water in the presence of small H_2O_2 (0.20 mL 30% H_2O_2 in 100 mL aqueous solution) by measuring the concentration of the models dye contaminant methyl orange (MO) under UV illumination. The photocatalytic reactions were carried out in a typical process. The degradation experiments of MO were tracked by spectroscopy (Fig. 8). The degradation efficiencies for MO were 11.6% in the presence of only H_2O_2 , and 19% in the presence of only catalyst $1 \cdot 13\text{H}_2\text{O}$ after 105 min. However, the degradation efficiencies of MO reached 86.7% for $1 \cdot 13\text{H}_2\text{O}$ in the presence of H_2O_2 after 105 min (Fig.9). Obviously, com-

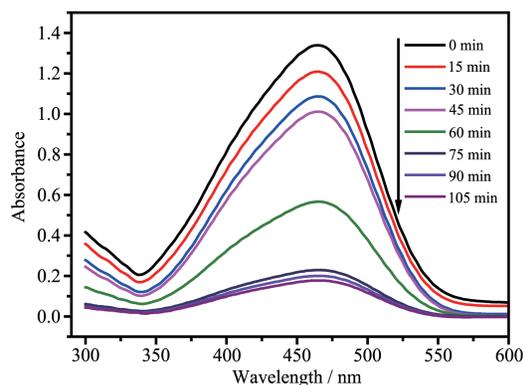


Fig.8 UV-Vis absorption spectra of MO solution degraded by photocatalyst $1 \cdot 13\text{H}_2\text{O}$ under irradiation at different time intervals

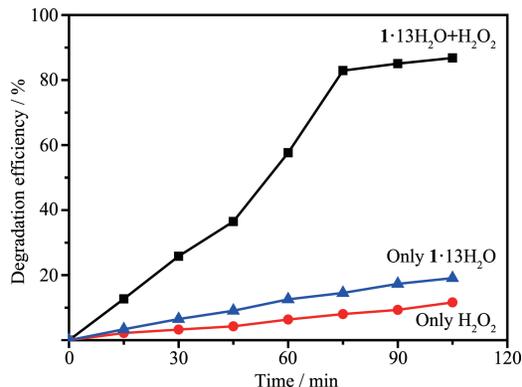


Fig.9 Photocatalytic degradation of MO solution under UV light using catalyst $1 \cdot 13\text{H}_2\text{O}$ in the presence or absence of H_2O_2 , and only H_2O_2

pound **1**·13H₂O shows good catalytic activity for the degradation of MO. The reaction mechanism should be well-known Fenton type mechanism^[37]. UV light can induce Cu(II) atoms with unsaturated square-pyramidal configuration to react with H₂O₂ to generate hydroxyl radicals. The ·OH radicals are highly active oxidizing species and can effectively decompose MO to complete the photocatalytic process^[4,42-44].

3 Conclusions

In summary, we successfully synthesized one unusual coordination polymer **1** using flexible 1,2-bis(4H-1,2,4-triazole)ethane and rigid multicarboxylate ligands. **1** exhibits an unusual 10-connected three-dimensional metal-organic framework based on [Cu₄(μ₂-OH)₂N₁₂]. **1**·13H₂O shows good photocatalytic activity in the degradation of methyl orange.

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