2D→3D Polycatenated Cu(I) Coordination Polymer: Photocatalytic Property and Protective Activity on COPD by Reducing the INF-γ Production

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Received: 25 March 2021 / Accepted: 8 November 2021 / Published online: 3 January 2022
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Abstract
Reported here is a new \([\text{Cu}_4\text{I}_4]\) cluster-based coordination polymer, namely \([\text{Cu}_4\text{I}_4(\text{bib})_2]_{\infty}\cdot n(\text{DMF})\) (1, bib = 1,4-bis(imidazolyl)butane, DMF = N,N'-dimethylformamide), which was synthesized by the self-assemble reaction of CuI, bib and KI under solvothermal conditions. Remarkably, compound 1 shows promising photocatalytic performance toward the degradation of MB solution under visible light irradiation. For the COPD treatment, the ELISA detection kit was conducted to determine the content of INF-γ released by the respiratory tract mucosal epithelial cells. In addition to this, the activation levels of the NF-κB signaling pathway were still need to be assessed by the real time RT-PCR after the compound treatment.

Keywords Cu(I) compound · \([\text{Cu}_4\text{I}_4]\) cubane · Solvothermal synthesis · Photocatalysis · COPD · RT-PCR

Introduction
Chronic obstructive pulmonary disease (COPD) is one of the most common respiratory diseases, which is the fourth leading cause of death in the world, and it is expected to rise to the third place in 2025, becoming the fifth major disabling disease in the world, seriously threatening human health [1]. However, there are few studies on the early drug treatment of COPD, and there is no relevant report abroad [2]. More clinical studies are needed to provide evidence and feasible treatment options for early drug treatment of COPD.

Water pollution that was caused by the discharge of dye contaminants seriously affects human health in the developing countries [3, 4]. Therefore, how to efficiently remove dye contaminants has become a research hotspot at present because of its importance to ecology and environment [5, 6].

In order to achieve this goal, various physical and chemical methods such as precipitation, membrane filtration, adsorption and photocatalytic degradation have been developed and used to remove the pollutants, but most of them suffer from large investment, low efficiency, and long cycle [7–10]. Thus, it is very urgent to explore new highly efficient photocatalyst to degrade these dye contaminants from industrial wastewater.

As a new kind of functional materials, metal-organic frameworks (MOFs) are getting more and more attention owing to their intriguing structures and promising properties [11–16]. The structures of MOFs can be easily tuned by selecting appropriate organic ligands to absorb UV/visible/UV-visible light for photocatalytic applications. Recently, some copper-iodine cluster-based MOFs with different N-donor ligands have been reported with excellent photocatalytic activities for degradation of organic dyes under visible light irradiation [17–23]. For instance, Wen et al. have prepared two isomeric two-dimensional copper(I) coordination polymer materials based on an in situ generated 5-(3-pyridyl)tetrazole ligand, which showed high efficiency and high stability on the photocatalytic degradation of organic dyes [24]; Hou et al. prepared a copper(I)/copper(II)–salen coordination polymer based on the N,N'-bis-[(imidazol-4-yl)methylene]cyclohexane-1,2-diamine ligand, and they found that this coordination polymer...
displays promising visible-light-driven bimetallic catalytic activity in degrading various organic dyes [25]; Liu et al. group has successfully prepared a thermally and chemically stable Cu1-triazolate metal–organic framework which was demonstrated to exhibit high photocatalytic activity towards the degradation of dyes in the presence of H2O2 [26]. Given the above considerations, in this work, we selected flexible 1,4-bis(imidazolyl)butane (bib) as the bridging ligand to assemble with CuI and KI under solvothermal conditions, obtaining a new [Cu4I4] cluster-based compound formulated as [Cu4I4(bib)2]n·n(DMF) (1). Compound 1 features a 2D→3D polycatenated framework based on 4-connected sql plane net. Moreover, the photocatalytic property of 1 was also studied. Its application values on the COPD were determined and the related mechanism was explored at the same time.

**Experimental**

**Materials and Instrumentation**

The commercially available reagents and solvents were of analytical grade and used without further purification. Elemental analyses (C, H and N) were determined using an elemental Vario EL III analyzer. Powder X-ray diffraction (PXRD) analysis were recorded on a PANalytical X’Pert Pro powder diffractometer with Cu/Kα radiation (λ = 1.54056 Å) with a step size of 0.05°. Thermogravimetric analysis for compounds 1 were performed on a NETSCHZ STA-449 C thermoanalyzer with a heating rate of 10 °C/min under air atmosphere in the temperature range of 30−800 °C. The optical diffuse reflectance spectrum was measured at room temperature with a Perkin-Elmer Lambda 900 UV/vis spectrophotometer, a BaSO4 plate was used as a standard (100 % reflectance).

**Synthesis of Compound [Cu4I4(bib)2]n·n(DMF) (1)**

The mixture of CuI (0.1 mmol), bib (0.1 mmol), KI (0.3 mmol), DMF (3 mL) and CH3CN (2 mL) was placed in a 25 mL Parr Teflon-lined stainless steel vessel, and then heated at 150°C for 7 days. After cooling to the room temperature at a rate of 10°C/min. Yellow block crystals of 1 were isolated in 28 % yield based on CuI. Anal. calcd. for C23H35Cu4I4N9O (1215.40): C, 22.71; H, 2.88; N, 10.37 %. Found: C, 22.68; H, 2.85; N, 10.42 %.

**X-ray Crystallography**

Single crystal data of 1 was collected on a computer–controlled Oxford XcalibE diffractometer with graphite–monochromated Mo–Kα radiation (λ = 0.71073 Å) at T = 293(2) K. The structure of 1 was solved by the dual direct method and further refined with the full-matrix least squares technique based on F^2 using the SHELXL-2014 [27]. Crystallographic data of compound 1 are summarized in Table 1. Selected bond lengths (Å) and angles (°) of 1 are listed in Table S1.

**Photocatalytic Experiment**

Compound 1 (50 mg) was added into 150 mL of a methyl blue (MB) solution (10 mg/L), and then the suspension solution was stirred in the dark for about 30 min to establish the adsorption-desorption equilibrium. After that, the suspension solution was exposed to a 300 W daylight lamp. At a given interval, aliquots of the reaction mixture were periodically taken and analyzed by UV-vis spectrophotometer at ambient temperature. The blank comparison experiment in absence of 1 was also performed at the same conditions.

**ELISA Detection Kit**

ELISA detection was conducted in this present experiment to determine the content of INF-γ released by the respiratory tract mucosal epithelial cells after indicated treatment. This preformation was finished totally under the guidance of the instructions with only a little change. In brief, the 50 BALB/c mice used in this present research were purchased from the Experimental Animal Center of Wuhan University (Wuhan, China), with the laboratory animal certificate

| Table 1 | The Crystal data for 1 |
|---------|-------------------------|
| Formula | C23H35Cu4I4N9O         |
| Fw      | 1215.40                 |
| Crystal system | monoclinic             |
| Space group | Cc                  |
| a (Å)   | 13.3115(8)             |
| b (Å)   | 15.5688(12)            |
| c (Å)   | 17.3737(12)            |
| α°      | 90                     |
| β°      | 102.125(6)             |
| γ°      | 90                     |
| Volume (Å^3) | 3520.3(4)             |
| Z       | 4                      |
| Density (calculated) | 2.293                 |
| Abs. coeff. (mm⁻¹) | 5.924                 |
| Total reflections | 7963                  |
| Unique reflections | 4958                  |
| Goodness of fit on F^2 | 1.023                 |
| Final R indices [I>2σ(F²)] R = 0.0309, wR2 = 0.0607 |
| R (all data) | R = 0.0394, wR2 = 0.0651 |
| CCDC    | 2,071,748              |

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number of SCXK 2019-0032. Then, the mice were given intratracheal infusion with lipopolysaccharide to induce the COPD animal model. After that the compound was given for treatment at the concentration of 1, 2 and 5 mg/kg via intraperitoneal injection. The respiratory secretions were collected and the content of the INF-γ released by the respiratory tract mucosal epithelial cells was determined with indicated ELISA detection kit.

**Real Time RT-PCR**

After COPD animal model construction and indicated compound treatment, the real time RT-PCR was carried out to determine the activation of the NF-κB signaling pathway in the respiratory tract mucosal epithelial cells. Briefly, the mice were given intratracheal infusion with lipopolysaccharide to induce the COPD animal model. Then, the compound was given for treatment at the concentration of 1, 2 and 5 mg/kg via intraperitoneal injection. Subsequently, the respiratory tract mucosal epithelial cells were harvested and the total RNA in the mucosal epithelial cells was isolated with TRIZOL reagent. After measuring the concentration of the total RNA, which was then reverse transcripted into cDNA. Finally, the relative expression levels of the nf-κb and p53 in the mucosal epithelial cells was determined with real time RT-PCR, with gapdh used as the internal control gene. The \(2^{-\Delta\Delta\text{ct}}\) method was used for statistical analysis.

**LD50 Determination**

Take 8-10 mice and divide them into groups of 2 into 4-5 groups. Choose a series of doses with a larger distance between the groups. The compound was injected into the abdominal cavity. Observe the symptoms and record the number of deaths to find out the cause 0% and 100% mortality (at least find out the range of the dose that causes 20%-80% mortality). Within the range of 0% and 100% lethal doses obtained in the preliminary experiment, 5 doses were selected to increase or decrease proportionally; the mortality rate of half of the groups should be above 50%, and the mortality rate of the other half groups should be above 50%. There are 10 animals in each group, and the weight and sex of the animals should be evenly distributed. After completing animal grouping and dose calculation, the animals were administered intraperitoneally according to the group. According to the experimental design, the corresponding method is used to calculate LD50, generally using Reed-Muench method or Kou’s modified method karber method.

**Results and Discussion**

**Description of Crystal Structure for 1**

X-ray structural analysis shows that the structure of compound 1 features a 4-connected 2D layer with \([\text{Cu}_4\text{I}_4]\) cubanes as nodes and bib ligands as linkers. Its asymmetric unit consists of four Cu(I) ions, four I\(^-\) anions, two bib ligands and one lattice DMF molecule. A shown in Fig. 1, four I\(^-\) anions as \(\mu_3\)-bridges link four Cu(I) ions to forming a \([\text{Cu}_4\text{I}_4]\) cubane subunit with the Cu···Cu distances ranging from 2.6620(19) to 2.8366(9) Å, and all of the Cu(I) ions are tetrahedrally coordinated by three \(\mu_3\)-I\(^-\) anions and one nitrogen atom of bib ligand. The Cu-N and Cu-I bond lengths in the range of 1.998(8)-2.020(7) Å and 2.6293(15)-2.7732(5) Å are in agreement with that of reported \([\text{Cu}_4\text{I}_4]\)-based coordination polymers [28]. All isolated \([\text{Cu}_4\text{I}_4]\) cubanes are bridged by the bib ligands into a 2D undulated layer (Fig. 2a). Topologically speaking, this undulated 2D layer...
represents a 4-connected sql network with point symbol of \(4^4\cdot6^2\) by reducing [Cu4I4] cubanes as 4-connected nodes and bib ligands as linkers (Fig. 2b). Notably, there exists large quadrilateral windows in the undulated layer. Interestingly, these quadrilateral windows in the layer catenated with ones from the adjacent layer (Fig. 2c). Thus, these adjacent 2D layers are catenated with each other, giving a 2D→3D polycatenated framework (Fig. 2d). The lattice DMF molecules are filled in the channels of this framework.

**Powder X-ray Diffraction (PXRD) Pattern and Thermogravimetric Analysis (TGA)**

The phase purity of the bulk samples for 1 was characterized by the PXRD measurement, and the result is shown in Fig. 3a. It can be seen that the experimental pattern shows good agreement with the simulated pattern calculated from the single crystal data, demonstrating the high phase purity of the obtained samples.
In addition, we further performed TGA experiment to investigate the thermal stability of 1. As shown in Fig. 3b, compound 1 first undergoes a weight loss process of 5.98% in the temperature range of 105-149 °C, which can be attributed to the removal of the lattice DMF molecules (calcd: 6.01%), and then no significant weight loss occurred before 292 °C. After that the framework of 1 started to break down with a weight loss of 31.35% corresponding to the decomposition of bib ligands (calcd: 31.27%), leaving the final residues of 62.67% that may be cuprous iodide (calcd: 62.63%).

Photocatalytic Property of Compound 1

The solid-state UV–visible absorption spectrum shows that compound 1 has a broad absorption band in the visible-light region of 400–650 nm (Fig. 4a), and the band gap (Eg) of 1 was also calculated by Kubelka–Munk equation of $F = (1 - R)^2/2R$ ($R$ represents reflectance at a given wavelength), giving the $E_g$ value of 2.62 eV (Fig. 4b). The suitable band gap value of 1 inspired us to investigate the photocatalytic activity for the degradation of MB solution, which was selected as a model dye pollutant in this work. The 664 nm characteristic absorption peak of MB was selected to monitor the degradation process. As shown in Fig. 4c, the intensities of the 664 nm absorption peaks decreased significantly under visible light irradiation with the time from 0 to 25 min. When irradiated under visible light for 25 min, the degradation ratio of MB is up to 96% in the presence of 1 (Fig. 4d). For comparison, the contrast experiment in the absence of 1 was further carried out at the same conditions, showing that only 3% MB was photodegraded after irradiation of 25 min. The photocatalytic mechanism of 1 in this work may be similar to that of previously reported works [29]. The PXRD pattern after photocatalytic reaction perfectly coincides with that of the pristine samples (Fig. 3a), verifying the stability of the framework. Moreover, compound 1 as photocatalyst shows almost undiminished catalytic activity after three cycles in the same reaction time (Fig. S1).

![Fig. 4](image-url)

**Fig. 4** (a) The UV-visible absorption spectrum of 1 at room temperature. (b) Diffuse reflectance spectrum of Kubelka-Munk function versus energy for 1. (c) UV–visible absorption spectra for degradations of MB in the presence of 1. (d) The comparison of degradation efficiencies for MB with or without 1 as photocatalyst.
To determine the main active species that participate in the degradation process, trapping experiments were performed using the degradation of MB dye under the same conditions. Isopropyl alcohol (IPA) is a scavenger for •OH, benzoquinone (BQ) is a scavenger for •O₂⁻ and ammonium oxalate (AO) is a scavenger for positive holes (h+). The degradation efficiency of 1 reached only 34.37% after 25 min. In this case, IPA displays the most significant restraint for the degradation activity of 1 towards MB dye indicating that •OH radicals play a considerably important role in MB degradation (Fig. 5a). On the other hand, the degradation efficiencies of MB dye suffer slight effect by adding 20 mg of BQ or AO to the reaction medium. The catalytic degradation efficiencies of 1 in the presence of BQ or AO reached 84.27 and 76.18%, respectively. Thus, the photodegradation of MB dye is mainly caused through attack by •OH radicals. The following mechanisms can be suggested for the photodegradation process. During the irradiation process by UV light, electrons (e⁻) transfer from highest occupied molecular orbital (HOMO) or conduction band (CB) to the lowest unoccupied molecular orbital (LUMO) or valence band (VB) and they leave positive holes (h⁺) in the CB. These holes (h⁺) are possibly located at the metal ions, that is, they are present as Cu²⁺ ions in the framework of 1. The in situ formed Cu²⁺ species can undergo Fenton-like reactions to produce •OH (Fig. 5b). The •OH radicals can further destroy organic dyes efficiently.

Compound Significantly Reduce the Content of INF-γ Released by the Respiratory Tract Mucosal Epithelial Cells

After the synthesis of the new compound with novel structure, its treatment activity on the COPD was evaluated firstly. Thus, the content of INF-γ released by the respiratory tract mucosal epithelial cells after the compound exposure was determined. As the results showed in Fig. 6, we can see that there was a significant higher level of INF-γ in the COPD model group, which is significantly different from that of the control group. After the treatment of the new compound, the content of INF-γ released by the respiratory tract mucosal epithelial cells was obviously reduced. The inhibition of the compound showed a dose and time relationship.
Compound Obviously Inhibited the Activation Levels of the NF-κB Signaling Pathway

In the above experiment, we have proved that the compound could significantly reduce the content of INF-γ released by the respiratory tract mucosal epithelial cells. As the NF-κB signaling pathway regulate the releasing of the INF-γ. So, the activation of the NF-κB signaling pathway in the respiratory tract mucosal epithelial cells was detected. The results in Fig. 7 indicated that the abnormal high activation levels of NF-κB signaling pathway in the respiratory tract mucosal epithelial cells could be inhibited by the compound in a dose and time dependent manner.

LD50 Determination

Though the biological experiments, the biological activity of the new compound was confirmed. The compound has the potential to be an excellent candidate for the COPD treatment by regulating the INF-γ releasing by the respiratory tract mucosal epithelial cells. However, the toxicity of the new compound was still need to be explored. Thus, in this research, the LD50 value of the new compound on mice was determined. From this research, we can see that the LD50 value of the new compound was 132 mg/kg, which is significantly higher than the effective therapeutic concentration of the new compound. This result suggested that the new compound has no toxicity during therapy.

Conclusions

In summary, we have successfully synthesized and characterized a new Cu(I) coordination polymer with the chemical formula of [Cu₄I₄(bib)₂]ₙ·n(DMF) that was constructed by [Cu₄I₄] cubanes as nodes and bib as bridging ligands. The results indicated that compound 1 can be used as highly active photocatalyst with well catalytic recurrence and structural stability toward to the degradation of MB solution. The results of the ELISA detection showed that the compound could significantly reduce the content of INF-γ released by the respiratory tract mucosal epithelial cells. Besides, the activation levels of the NF-κB signaling pathway were also inhibited by the compound obviously. In the end, we draw this conclusion, the compound has the potential to be an excellent candidate for the COPD treatment by regulating the INF-γ releasing by the respiratory tract mucosal epithelial cells.

Supplementary Information The online version contains supplementary material available at https://doi.org/10.1007/s10895-021-02850-w.

Author Contributions Jun-Fei Zhu and Wei-Wei Yang carried out the experiments, analyzed the data and wrote the draft of this manuscript; Jian Yang and Li-Tong Jin designed the whole experiments and revised the draft.

Availability of Data and Material The table showing the bond lengths and angles for complex 1 (Table S1), Four cycling experiments for MB degradation in the presence of 1 (Fig. S1), information could be found in the supporting information file.

Code Availability The data that support the findings of this study have been deposited in NCBI Reference Sequence: NC_000002.12.

Declarations

Ethics Approval We try to replace live animals with unconscious experimental materials, or use lower animals to replace higher animals. we try to use the smallest number of animals to get the same amount of experimental data or use a certain number of animals to get more experimental data. We minimize the scope and extent of the impact of inhumane procedures on animals. All the preformation conducted in this present research was approved by the Animal Ethics Committee of Affiliated Hospital of Nanjing University (Nanjing, China) with the No. 20200233.
Consent to Participate Not applicable.

Consent for Publication Written informed consent for publication was obtained from all participants.

Conflict of Interest All authors declare that there is no conflict of interest regarding the publication of this paper.

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