Two New Zn(II)/Cu(II) Compounds: Photoluminescent and Photocatalytic Properties, and Protective Activity on Parkinson Disease

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Abstract
Two coordination polymers, that is [Zn(pdc)(im)(H2O)]n (1) and [Cu(pdc)(im)2]n·n(H2pdc) (2) (H2pdc = terephthalic acid, im = imidazole), were hydrothermally synthesized via the reactions of H2pdc and im in combination with Zn(II) or Cu(II) ions. Compound 1 shows intense blue luminescence and compound 2 shows good photocatalytic activity for the methyl violet degradation under the irradiation of ultraviolet light. In addition, the assessment of the two compounds’ application values against Parkinson’s disease were carried out and their specific mechanism was tested simultaneously. First of all, the real time RT-PCR was implemented and the relative expression levels of N-methyl-D-aspartic acid receptor receptor on neurons were measured. Besides, the Annexin V-FITC/PI apoptosis assay was utilized for the assessment of the influence of the compounds on the dopaminergic neuron death rate. The hemolysis toxicity detection was conducted to detect the biocompatible of the compounds.

Keywords Cu(II) compound · Supramolecular framework · Luminescence · Photocatalysis · Parkinson’s disease

Introduction
Parkinson’s disease (PD) is a type of common neurodegenerative disease in the middle-aged people and elderly. And the clinical characteristics of this disease are muscle stiffness, static tremor, bradykinesia, postural instability together with the lack of movement [1]. For the PD, the principal pathological change is the dopaminergic neurons loss in midbrain substantia nigra. Aspartic acid is an endogenous excitatory amino acid, which reveals a significant effect in the central nervous system of mammals [2, 3]. There is evidence that Parkinson’s disease is related to the excessive accumulation of excitatory amino acids in the brain. To clarify the pathogenesis of PD will lay the experimental foundation for the development of PD targeted therapies.

Recently, coordination polymers with novel compositions and topological frameworks have attracted great attention mainly owing to their functional properties that make them can be served as potential hybrid materials in catalysis, luminescence, magnetism, gas separation and storage, and other areas [4–7]. For creating the coordination polymers, one of the most effective method is the technology of hydrothermal or solvothermal self-assembly that requires intelligent selection of organic ligand with appropriate central metal ions and coordination positions having the definite coordination structures [8–11]. During the past few decades, organic carboxylate ligands as one kind of organic building backbone have been widespread exploited for producing the metal–organic coordination polymers with charming topological networks and attractive performances owing to their a variety of coordination manners of the carboxyl groups along with strong coordination affinity to metal centers [12–16]. Terephthalic acid (H2pdc), as an inexpensive ligand, is a rigid aromatic carboxylate ligand and has four potential coordination sites. In the construction of coordination polymers, it usually acts as linear connectors, which is helpful for us to obtain the compounds with expected structures [17–19]. To date, numerous of single-nuclear or polynuclear metal-based coordination polymers with H2pdc or H3pdc and N-donor auxiliar ligands as organic backbone have been reported, which show excellent properties in gas storage, catalysis, and luminescence, etc. [20–22]. In order
to continue to develop new hybrid functional materials, in this work, terephthalic acid was selected to self-assemble with Zn(II) or Cu(II) ions using imidazole as the organic base to tune the reaction pH, successfully obtaining two coordination polymers, that is [Cu(pdc)(im)2]n·n(H2pdc) (2) and [Zn(pdc)(im)(H2O)]n (I) (H2pdc = terephthalic acid, im = imidazole). In the following, the syntheses, crystal structures, thermal behaviors, luminescent and photocatalytic properties will be reported. Their treatment effect on the PD was explored and the relevant mechanism was also explored.

Experimental

Materials and Instrumentation

All of the raw chemical reagents employed in our investigation were acquired from Jinan Henghua Company, and they could be utilized directly. Vario EL III analyzer was employed for the analysis of Hydrogen, Nitrogen and Carbon elements for 1–2. The diffractometer of PANalytical X’Pert Pro was applied for the analysis of PXRD with 0.05° step size using the Cu/Kα radiation (with λ of 1.54056 Å). The two compounds’ TGA were finished through employing the NETSCHZ STA-449C under flow of nitrogen at 10 °C min−1 heating rate in the temperature range of 30–800 °C. The photocatalytic experiments were carried out in a Persee TU-1950 UV–vis spectrophotometer. The luminescent spectra was measured using the fluorescence spectrophotometer of Edinburg FLS920 TCSPC under ambient temperature.

Synthesis of [Zn(pdc)(im)(H2O)]n (1) and [Cu(pdc) (im)2]n·n(H2pdc) (2)

The mixture generated from 0.2 mmol Zn(NO3)2·6H2O, 0.2 mmol H2pdc, 0.2 mmol imidazole, and 8 mL H2O was sealed to the stainless steel vessel (25 mL) lining with Teflon and the acquiring product was heated for three days under 160 °C temperature. Subsequently, it was naturally cooled to ambient temperature, the 1’s colorless massive crystals were gained with 46 percent yield on the basis of Zn(NO3)2·6H2O. Anal. calcd. (%) for the C11H10N2O5Zn: N, 8.87, C, 41.83 and H, 3.17. Found (%): N, 10.55, C, 49.79 and H, 3.42.

X-Ray Crystallography

The architectural data of single crystal for the two compounds were harvested through the graphite–monochromated Mo–Kα radiation (with λ of 0.71073 Å) via exploiting the Mercury CCD controlled via the computer at ambient temperature. By utilizing the dual direct means, the compounds’ architectures can be solved with ShelXS, and then the SHELXL-2014 is applied for the refinement of the structures via full-matrix least square technique on the basis of R2 [23]. The data of crystallography and the optimization of structures for the complexes were concluded in the Table 1. The chose bond angles (°) and lengths (Å) of the compounds are exhibited in the Table S1. The detailed parameters of H-bond for the complexes are displayed in the Table S2.

Real Time RT-PCR

In our present investigation, the real time RT-PCR was finished to determine the expression levels of NMDA receptor on the neurons after the treatment of compounds. This conduction was implemented completely in accordance with instructions with only minor modification. In short, forty mice (4–5 weeks, 20–22 g) were offered via the Model Animal Reach Center of Wuhan University (Wuhan, China). All of the mice applied in this research were maintained between 20 and 25 °C, under the environment of free food and water. After that the PD animal model was established by the injection of β-amyloid, and treatment was performed with the two compounds at 5 mg each kg concentration. After that the neurons were separated, and then the TRIZOL was applied for the extraction of the entire RNA in cells. After measuring the entire RNA concentration, this concentration could be reverse transcripted into the cDNA. Ultimately, the relative expression of NMDA receptor against the neurons was checked with the real time RT-PCR, in which the gapdh was employed as an internal control. This study was implemented for 3 times.

Annexin V-FITC/PI Apoptosis Assay

After β-amyloid stimulation, the evaluation of novel compounds’ inhibitory influence against the apoptosis levels of neurons was conducted with Annexin V-FITC/PI apoptosis assay. All the implementation in our experiment was carried out on the basis of instructions. In general, in the phase of exponential growth, the neurons could be harvested and subsequently they were seeded with 2 × 10^5 cells per well destiny into the plates of 6-well, and they were cultivated...
in 5 percent of CO₂ humidified surroundings overnight at 37 °C. Subsequently, the cells were treated through the two compounds with 5 mg per mL concentration for one day. Dimethyl sulfoxide (0.1 percent, DMSO) together with the apoptosis inducer kit (Beyotime, Shanghai, China) were exploited as a negative and positive control, respectively. Each of the group has 3 parallel wells. After the above treatment, in each group, the cells were collected and after that they were labeled via the application of propidium iodide (PI, 5 μL) along with Annexin V-FITC (5 μL) for thirty minutes. The determination of stained cells in each group was carried out by flow cytometry (BD, NJ, USA) at 488 nm excitation wavelength and 625 nm emission wavelength. The study requires to be finished at least 3 times.

**Hemolysis Toxicity Determination**

The hemolysis toxicity detection was performed in this present research to measure the biocompatible of compounds 1 and 2. This experiment was carried out strictly under the guidance of the instructions with some modifications. Briefly, the New Zealand white rabbits used in this experiment were obtained from the Changchun Jingcheng Model Animal Technology Co., Ltd, the blood used for hemolysis experiment were harvested from adult male New Zealand white rabbits. The collected blood was placed in a tube, then compound 1 or 2 was added and incubated in an incubator at the condition of 37°C. The supernatant was aspirated and transferred into a cuvette, and the absorbance at 545 nm of each sample was measured with a spectrophotometer. Evaluation of the in vitro hemolysis of the compound was determined by measuring the degree of red blood cell lysis and hemoglobin release. This experiment was performed in triplicate, and the results were presented as mean ± SD.

**Results and Discussion**

**Crystal Structure of Compound 1**

The architectural research of X-ray single crystal indicated that the 1’s architecture reflects an one-dimensional infinite chain based on {ZnO₃N} nodes and pdc²⁻ linkers. And its fundamental unit is constructed from a Zn(II) ion, a ligand of pdc²⁻, a terminal ligand of im together with a coordinated molecule of water. As illustrated in the Fig. 1, each of Zn(II) ion exhibits a coordination structure of tetrahedron, which is defined via 2 carboxylic acid O atoms in 2 diverse ligands of pdc²⁻, a N atom originated from a terminal ligand of im and a coordinated molecule of water, and the bond separation of Zn–O/N are between 1.962(3) and 1.992(3) Å, and it is in a normal range [24]. These tetrahedral Zn(II) ions exhibits a coordination structure of tetrahedron, which is defined via 2 carboxylic acid O atoms in 2 diverse ligands of pdc²⁻, a N atom originated from a terminal ligand of im and a coordinated molecule of water, and the bond separation of Zn–O/N are between 1.962(3) and 1.992(3) Å, and it is in a normal range [24]. These tetrahedral Zn(II) ions are linked through the ligands of pdc²⁻ by the carboxylic acid groups with the uniform monodentate manner to provide an one-dimensional infinite zigzag chain architecture (Fig. 1b), and the Zn⋯Zn separated by pdc²⁻ ligand is 11.01 Å. The H atoms of the suspended terminal im ligands in the chain
can be suitably accepted via the carboxylic acid O atoms in the neighboring chains, producing the intermolecular H-bonds of N–H…O. These H-bonds of N–H…O linked the one-dimensional chains into a 2D layer motif with (4,4) grids (Fig. 1c). Interestingly, two adjacent hydrogen-bonded 2D layers interpenetrated with each other, forming an interpenetrated bi-layer structure (Fig. 2d). Also, the H donors of coordinated water molecules in these bi-layer structure can be appropriately accepted through the carboxylic acid O atoms derived from the consecutive bi-layer architecture.
As a result, this bi-layers are in-depth H-bonded into a three-dimensional supramolecular skeleton (Fig. 1e). The corresponding hydrogen bond parameters can be found from the Table S2.

Crystal Structure of Compound 2

The diffraction of X-ray single crystal confirmed that the 2’s fundamental unit is composed of 2 Cu(II) ions with the occupation ratio of 0.5, and a ligand of pdc$^{2-}$, two terminal im ligands, as well as one free H$_2$pdc molecule. As displayed in the Fig. 2a, the Cu1 ion and Cu2 ion are both tetracoordinated via 2 carboxylic acid O atoms in 2 diverse ligands of pdc$^{2-}$ and 2 N atoms come from 2 terminal on a basal level, and the spacing of Cu…O/N are between 1.9436(14) and 1.9759(17), which is also in a normal range. On account of the well-known John–Teller effect of Cu(II) ion with d$^9$ electronic configuration, the coordination geometries of Cu(II) ions can be described as elongated octahedrons with the axial sites taken over via the 2 carboxylic acid O atoms in 2 distinct ligands of H$_2$pd, and the weak distances of Cu…O are from 2.870 to 2.970 Å. The pdc$^{2-}$ ligands in 2 act as μ$_2$-bridges linking Cu(II) into an one-dimensional chain architecture (Fig. 2b), and the separation of Cu…Cu is 10.80 Å. Owing to the weak Cu…O interactions, the 1D chains and free H$_2$pd ligands are extended into a two-dimensional layer architecture (Fig. 2c). In this layer, we can found that multipoint N–H…O hydrogen bonds further consolidate this 2D layer. Finally, such two-dimensional layers are stacked with the parallel manner through the weak forces of Van der Waals, providing a stacked three-dimensional supramolecular skeleton (Fig. 2d).

Powder X-Ray Diffraction (PXRD) Patterns and Thermogravimetric Analyses (TGA)

The compounds phase purities purity was demonstrated via the analysis of PXRD, displayed in Fig. S1. Significantly, the pattern of experiment was in accordance with that of simulation created by the data of single crystal diffraction, clearly reflecting the excellent purities of massive samples. Considering the following bioactivity tests, it is necessary to study the framework stability of complexes 1 and 2 in the dispersing solvent DMSO and the simulated in vivo condition phosphate buffered saline (PBS). Due to complexes 1 and 2 could not be dissolved in the organic solvents and water, we used their stock solutions in the following bioactivity tests according to the literature methods [17, 18].

With this in mind, about 100 mg of the as-prepared crystalline samples of 1 were was taken in a mortar, respectively. They were then being ground manually for 30 min by using apestle. The produced fine powders were soaked in 20 mL PBS or DMSO and subjected to the ultrasonic treatments for 2 h to obtain the well dispersed solution. After standing for two days, the fine powders could be recovered via centrifugation and their PXRD measurements show that the PXRD profiles of the treated samples show good matches with those of the as-prepared samples, reflecting their good stability in the above conditions (Fig S1). The sizes of the treated samples have been further studied by scanning electron microscopy (SEM) studies that are obtained by drop-casting a DMSO-dispersed solution on a glass surface. Complexes 1 and 2 adopt almost square morphologies in order to minimize the interfacial free energy between the particle and the solvent molecules. Dynamic light scattering (DLS) studies are carried out to prove further the nanostucture formation. The results show that the hydrodynamic radius of all the treated complexes falls in the nano regime (~150 nm) (Fig. S2).

Moreover, the compounds’ thermal behaviors under nitrogen atmosphere were also characterized via the TGA experiments, and the TGA results are displayed in Fig. 3. In the TGA curve of 1, the loss of the coordinated molecules of water appeared between 96 and 118 °C temperature (with the calculated and observed values of 5.70% and 5.72%), and subsequently, no remarkable weightlessness was found when the temperature less than 287 °C, and then, the framework collapse appeared in the range of 287–416 °C that is on account of the organic ligand decomposition. In the compound 2’s TGA curve, there is no evident weightlessness appeared when the temperature less than 290 °C, and subsequently, rapid weightlessness occurred in the range of 290–433 °C, revealing the framework collapse resulted from the organic ligand decomposition.

Photoluminescent Property of 1

The luminescent spectra and free ligand of H$_2$pd and the complex 1 were checked at environmental temperature. As displayed in the Fig. 4a, it can be found that the free ligand of H$_2$pd reveals an emission band with the maximum peak at 436 nm (with $\lambda_{ex}$ of 340 nm) that is owing to the orbital charge transitions of $\pi^*$ to n or $\pi$ [25], and compound 1 shows a much strong emission band, and at 447 nm, there exist the maximum peak ($\lambda_{ex}$ = 340 nm). Owing to the d$^{10}$ electronic configuration of Zn(II) ion, the 1’s luminescence should principally derive from the $\pi^*$ to n or $\pi$ orbital charge transitions of pdc$^{2-}$ ligand [26]. Compared to that of the H$_2$pd ligand, a red-shift of 11 nm probably resulted from the H$_2$pd ligand deprotonation and the pdc$^{2-}$ ligand coordination to the central metal ions. By calculation, it can be found that the CIE chromaticity coordinate of 1 is (0.1689, 0.1176) at the 1931 CIE chromaticity diagram (Fig. 4b), indicating that compound 1 may be served as an excellent blue fluorescent material.
Photocatalytic Property of 2

The photocatalytic performance of 2 was evaluated by the degradation of methyl violet (MV) under UV light irradiation. As shown in Fig. 5a, it is clear that the absorption peak at about 580 nm of MV solution decreased gradually owing to the photodegradation of MV with the increase of irradiation time. After the irradiation time of 80 min, it can be calculated that 70.2% MV were successfully photodegraded by the photocatalyst of 2 (Fig. 5b). In addition, the control investigation without complex 2 was also implemented under the identical conditions, and the MV degradation reaction was relatively slow, and after 80 min, the leaving undegraded MV accounted for 84.9%. Such results indicated that complex 2 shows good activity for the MV photocatalytic degradation under the irradiation of ultraviolet light. Compared to the reported coordination polymers, the photocatalytic activity of 2 is little lower [27]. After photocatalytic reaction, compound 2 keeps the structural integrity that was proven via the patterns of PXRD (Fig. S1b).
Compound Significantly Reduce the Expression Levels of the NMDA Receptor on Neurons

Aspartic acid is an endogenous excitatory amino acid, which has a remarkable influence in the central nervous system of mammal, and the excessive accumulation of excitatory amino acids in the brain could lead to the PD development. Thus, in this present research, after the compound treatment, the expression levels of NMDA receptor on neurons were measured. As the outcomes illustrated in the Fig. 6, there was an evidently enhanced NMDA receptor expression level in contrast to control group. Nevertheless, after treated with 1, expression levels of the NMDA receptor on neurons were decreased significantly, but 2 exhibited nearly no effect on the NMDA receptor expression.

Compound Obviously Inhibited the Levels of Neurons Apoptosis

In former investigation, we have suggested that compound 1 could remarkably down-regulate the expression levels of NMDA receptor on neurons. However, the influence of the compound on the neurons’ apoptosis was still need to be explored. The outcomes in the Fig. 7 revealed that in model

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Fig. 5  a Ultraviolet–visible absorption spectra for degradations of MV in the presence of 2 as photocatalyst. b The degradation rate of MV under UV light with or without 2 as photocatalyst

Fig. 6  Significatly decrease NMDA receptor expression levels on the neurons after treating with compound. The PD animal model was created in our experiment, then the treatment of the two compounds was carried out with 5 mg per kg concentration. The expression levels of NMDA receptor on neurons were assessed through the real time RT-PCR
The neurons apoptosis levels were much higher than the control group. Compound 1 evidently decreased the apoptosis levels of neurons, which is much stronger than compound 2.

**Compound have Good Biocompatible Application**

In Figs. 6 and 7, we revealed that the compound has excellent application values on the PD therapy by reducing the expression levels of NMDA receptor on neurons, and inhibiting the apoptotic neurons levels. The biological activity of compound 1 was much stronger than that of compound 2. For further clinic application, the biocompatible of the new compounds were still need to be assessed (Fig. 8). The results of the hemolysis toxicity assay showed that both compounds 1 and 2 showed no influence on the red blood cells integrity, there were no differences among those groups. This result suggested that the both compounds 1 and 2 have excellent application value on PD disease treatment.
Conclusions

On the whole, we have produced two fresh coordination polymers of [Zn(pdc)(im)(H2O)]n (1) and [Cu(pdc)(im)2]n(H2pdc) (2) (H2pdc = terephthalic acid, im = imidazole). Both 1 and 2 have 1D chain structures. For 1, the 1D chains are firstly linked via the N–H…O H-bonds into a 2D layer, which is interpenetrated by another adjacent identical one to form a bi-layer motif, and such bilayers are ultimately extended into a three-dimensional supramolecular skeleton via the connection of Ow–H…O H-bonds. For 2, the 1D chains are firstly connected through the N–H…O H-bonds and weak Cu…O bonds into a 2D layer, and these two-dimensional layer are ultimately stacked into a three-dimensional supramolecular skeleton in a parallel mode. The intense blue luminescence of 1 and good photocatalytic activity of 2 indicate that they can be served as promising hybrid materials. Through the real time reverse transcription-polymerase assay, it can be found that the compound 1 possesses stronger inhibitory effect on decreasing the NMDA receptor expression levels than the 2. Moreover, the 1 could evidently decrease the neurons apoptosis levels, which is better than the activity of compound 2. Through the hemolysis toxicity assay, we proved that the compounds both have good biocompatible application values on the disease treatment. In conclusion, compound 1 has good protein activity and is expected to be a candidate drug for the treatment of PD.

Supplementary Information The online version contains supplementary material available at https://doi.org/10.1007/s10895-021-02815-z.

Author Contributions NW and FJH synthesized and characterized the compounds; MSZ and BXS performed the activity assay experiments; YW and LYQ designed the study and prepared the manuscript.

Data Availability Selected bond lengths (Å) and angles (°) for compounds 1–2 (Table S1); The detailed hydrogen bond parameters for 1–2 (Table S2); The PXRD patterns (a) for compound 1 and (b) for compound 2 (Fig. S1); The DLS and SEM study of complexes 1 and 2 (Fig. S2), the information could be found in the supporting information file.

Declarations

Ethical Approval BALB/c mice (4–5 weeks, 20–22 g) were used for PD animal model construction. Ethics Committee approval was obtained from the Institutional Ethics Committee of Model Animal Reach Center of Wuhan University to the commencement of the study.

Consent to Participate This experiment does not involve clinical cases and informed consent.

Consent for Publication The authors agreed for the article publication.

Conflict of Interest The author(s) declare(s) that there is no conflict of interest regarding the publication of this paper.

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