Two new inorganic-organic hybrid compounds: photocatalytic, luminescent property and treatment activity on polycystic ovary syndrome

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ABSTRACT
Two new mixed-ligand-based compounds, namely [Co(1,3-pde)(4-bpmh)]ₙ (1) and [Zn₃(3,3'-bpdc)₃(4-bpmh)₁.₅]ₙ (2) (1,3-H₂pde = 1,3-phenylenediacetic acid, 3,3'-H₂bpdc = biphenyl-3,3'-dicarboxylic acid, 4-bpmh = N,N-bis-pyridin-4-ylmethylene-hydrazine) have been prepared under hydrothermal conditions. The obtained compounds were determined by single crystal X-ray diffraction analyses, elemental analyses, powder X-ray diffraction analyses and thermogravimetric analyses. Moreover, the photocatalytic activity of 1 and luminescent property of 2 have also been investigated. This presented research estimate their treatment activity of the polycystic ovary syndrome, also the mechanism was measured as well. Firstly, a relative expression of the estrogen receptor on the ovarian epithelial cells was determined with real time RT-PCR. In addition, the amount of progesterone of the plasma was measured with ELISA detection kit after indicated treatment.

1. Introduction
Polycystic ovary syndrome, one of the endocrine diseases, is very typical for childbearing-age women. It belongs to a clinical syndromes group characterized by reproductive disorders, endocrine abnormalities, metabolic disorders and mental problems [1]. Polycystic ovary syndrome not only affects the patient’s fertility, but also affects the health of its pregnancy. The etiology of polycystic ovary syndrome is complex, plus the pathogenesis is still unclear, and possibly can be influenced by gene as well as environment [2].

Coordination polymers (CPs) have become research hotspot at present owing to their diverse architectures and promising potentials as structural polymers in luminescence, gas sorption as well as separation, magnetism, heterogeneous catalysis and biomedicine. [3–6]. The latest developments in coordination polymer crystal engineering have innovated many synthetic techniques with high efficiency toward synthesizing coordination polymers with required frameworks and characteristics [7–9]. A successful strategy for synthesizing...
the coordination polymers is to use the multi-type ligands technique for the construction of 3D extended framework [10–12]. The use of two complementary ligands with different coordination functional groups provides an additional level in controlling over the framework structure and regulating the pH of reaction mixture. The most widely used mixed-ligand strategy for synthesizing the functional coordination polymers is combining a polycarboxylate ligand with a N-donor ligand such as 4,4’-bipydine, 2,2’-bipyridinie, 1,3-di(4-pyridyl)propane, 1,2-di(4-pyridyl)ethane, 1,4-bis(N-imidazolyl)butane, etc. [13–16]. According to previous reports, it is observed that the main structural mode produced by using mixed-ligand method is the formation of neutral metal carboxylate layer motif, which is then connected or pillared by auxiliary N-donor ligands into 3D pillar-layer frameworks with high thermal stabilities [17]. Using this strategy, the frameworks and topologies of the coordination polymers can be easily controlled by altering the polycarboxylate ligands or N-donor ligands. Furthermore, metal ions could also result in the structure and property of the final coordination polymers via their different coordinative geometries and electronic properties [18].

Considering the above mentioned, in the present study, we also selected this following method based on the mixture of carboxylate ligand with N-donor auxiliary ligand to construct new functional compounds (Scheme 1). Through self-assembly reaction under hydrothermal conditions, two new compounds have been produced, namely [Co(1,3-pde)(4-bpmh)]n (1) and [Zn3(3,3’-bpdc)3(4-bpmh)1.5]n (2) (1,3-H2pde = 1,3-phenylenediacetic acid, 3,3’-H2bpdc = biphenyl-3,3’-dicarboxylic acid, 4-bpmh = N,N-bis-pyridin-4-ylmethylene-hydrazine). The properties investigations indicate that compound 1 exhibits outstanding photocatalytic activity for MB degradation under UV light, besides, compound 2 shows high luminescence under condition of room temperature. In this paper, Serial biological experiments were conducted to investigate the application values for compounds 1 and 2 on the polycystic ovary syndrome, and the mechanism was explored as well.

2. Experiments

2.1. Materials and instrumentation

Two starting materials containing the organic ligands, metal salts, and DMF were normally purchased through Aladdin company also have no require to further purificate as well. This study conducted elemental analyses (C, H and N) through an analyzer in type
of Vario EI III. PXRD (Powder X-ray diffraction) analysis was conducted by using PANalytical X'Pert Pro powder diffractometer with Cu/Kα radiation (λ = 1.54056 Å) with 0.05° as step size. TGA (Thermal gravimetric analysis) of compounds 1-2 was conducted by applying NETSCHZ STA-449C thermoanalyzer by setting the heating rate at 10°C per minute under air atmosphere within the interval of 30-800°C. We evaluate the luminescence feature for compound 2 by using Edinburgh Analytical instrument FLS920.

2.2. Synthesis of compound [Co(1,3-pde)(4-bpmh)]n (1) and [Zn3(3,3'-bpdc)3(4-bpmh)1.5]n (2)

A mixture of Co(NO₃)₂·6H₂O (0.176 g, 0.2 mmol), 1,3-pde (0.109 g, 0.2 mmol), 4-bpmh (0.2 mmol) and H₂O (10 mL) was stirred for 30 min in air. The resulting solution was transferred into 20 mL Teflon-lined stainless steel, then the pH value was adjusted to approximately 7.0 with the dilute NaOH solution, and heated at 140°C for 72 h, and then cooled to room temperature naturally. Red block crystals of 1 were obtained in 47% yield. Anal. Calcd. (%) for C₂₂H₁₈CoN₄O₄: C 57.23, H 3.90, N 12.14; Found (%): C 57.27, H 3.93, N 12.09.

In this procedure, we mixed equal volume of 0.2 mmol Zn(NO₃)₂·6H₂O (0.176 g), 3,3'-H₂bpdc (0.109 g), 4-bpmh and 10 mL H₂O and stirred the mixture for 30 minutes under air. We transferred the stirred solution into a 20 mL Teflon-lined stainless steel, the next is to adjust the pH value to approximately 7.0 by mixing dilute NaOH solution, then the heating process lasted 72 hours under 140°C, finally the last step is to cool down to room temperature naturally. Crystals without color in block size of 2 were collected and the yield was 48%. Anal. Calcd. (%) for C₆₀H₃₉N₆O₁₂Zn₃: C 58.44, H 3.17, N 6.82; Found (%): C 58.48, H 3.15, 6.85.

2.3. X-ray crystallography

The singe crystal dataset of compounds 1-2 were obtained under temperature of 293 K by applying Oxford Xcalibu E diffractometer controlled by computer using graphite–monochromated Mo–Kα radiation (λ = 0.71073 Å). To solve the frameworks, the dual direct method using ShelxT was applied, also the results was optimized by full-matrix least square method based on F² through the SHELXL-2014 [19]. We used SADABS to perform sorption correction [20]. Table 1 lists the data of crystallography and optimizations of structures for compound 1-2. Table S1 shows selected bond lengths (Å) and angles (°) of 1.

2.4. Photocatalytic experiment

Firstly, add 50 mg of compound 1 to 200 mL of MB (Methyl Blue) solution with a concentration of 30 mg/L, and then stir the suspension under dark environment for about 6 h so that an equilibrium of sorption and desorption can be established. After that, the suspension was irradiated with ultraviolet radiation from a 175W high-pressure mercuric vapor light source. Within the designed time range, termly took aliquots of the mixed reactants out and analyzed it with an ultraviolet–visible spectrophotometer under ambient temperature. As for control group, repeated this procedure without compound 1.

2.5. Real time RT-PCR

In the present research, the real time RT-PCR detection was conducted to measure the relative expression of the estrogen receptor on the ovarian epithelium when compounds 1 and 2 was
used for treatment. Guided by the instructions, this procedure was conducted strictly requiring only a little change. In brief, female rats aged 9 days after birth were selected, and testosterone propionate was injected subcutaneously at the back of the neck at a dose of 1.25 mg per rat. The animals at the age of 21 days were routinely raised after weaning, and they were free to drink and eat. Vaginal smears were continuously taken for 11 days since the age of 70 days of the model animals, and the model was successful if the persistent keratinization of the vaginal epithelium was observed. Then, with a concentration of 5 mg/kg, compounds 1 and 2 were offered. Finally, we collected these ovarian epithelial cells as well as all total RNAs in the cells were extracted. After determining the RNA concentration, cDNAs were generated by all the RNAs. Finally, we evaluated the expression levels of the estrogen receptor by applying a real time RT-PCR, using gapdh as the internal control.

2.6. ELISA assay

To determine the content of the progesterone in the plasma after compound treatment, we conducted the ELISA detection for the presented study. This experiment was conducted entirely according to the instructions requiring a few changes. Shortly, these female rats aged 9 days after birth were selected, and testosterone propionate was injected subcutaneously at the back of the neck at a dose of 1.25 mg per rat. The animals at the age of 21 days were routinely raised after weaning, and they were free to drink and eat. Vaginal smears were continuously taken for 11 days since the age of 70 days of the model animals, and the model was successful if the persistent keratinization of the vaginal epithelium was observed. Then, with a concentration of 5 mg/kg, compounds 1 and 2 were offered. Finally, we collected these ovarian epithelial cells as well as all total RNAs in the cells were extracted. After determining the RNA concentration, cDNAs were generated by all the RNAs. Finally, we evaluated the expression levels of the estrogen receptor by applying a real time RT-PCR, using gapdh as the internal control.

3. Results and discussion

3.1. Crystal framework of compound 1

Singe crystal X-ray characterization reveals that compound 1 belongs to the monoclinic $P2_1/n$ space group and demonstrates a columnar structure of three dimensional with 6-
linked \textbf{pcu} topological network. One fundamental component of 1 consists of 1 Co(II) ion, 1 1,3-pde ligand, as well as 1 4-bpmh bridging ligand. From what Figure 1a demonstrates, Co1 shows an octahedral coordination structure that was constructed by carboxylate oxygen atoms (O2, O1b, O3a and O4a) offered by three kinds of 1,3-pde ligands in equatorial plane and both the nitrogen atoms (N1 and N4c) was offered by 4-bpmh ligands of apexes. Table S1 lists the bond lengths (Å) of Co-O and Co-N which are in range of 2.011(3)-2.315(4) and 2.184(3)-2.232(3). Each 1,3-pde ligand connects three different Co(II) ions containing one carboxyl group in a chelating mode as well as another carboxyl group in a double monodentate mode (Figure S1a). Both close Co(II) with the separation of 4.06 Å are connected by two carboxyl groups in double monodentate mode together, generating a [Co2(COO)2] unit with double nuclear (Figure 1b). The chelating carboxylate groups of 1,3-pde ligands further linked the binuclear [Co2(COO)2] units into a 2D layer motif which extends along crystallographical \textit{bc} surface (Figure 1c). Finally, the adjoining 2D layers becomes columnar through the 4-bpmh ligands, affording a columnar structure in three dimensional of 1 (Figure 1d). In this 3D pillar-layer framework, every binuclear [Co2(COO)2] unit is linked to six adjoining other units through 1,3-pde as well as 4-bpmh ligands, with amount of 4 and 2 respectively. If every dinuclear [Co2(COO)2] subunit can be simpliﬁed as a 6-linked point, then the 1,3-pde and 4-bpmh ligands can be constructed as linear connectors. From topological perspective, the whole structure of 1 represents a 6-linked \textbf{pcu} topological grid and the point symbol of 1 is \{4\^{12}·6\^{3}\} (Figure 1e). Further analysis the structure of 1 via the SOLV operation embedded in the software PLATON indicates that there is no solvent accessible volume in its framework.

\subsection*{3.2. Crystal structure of compound 2}

X-ray diffraction shows the crystallization of compound 2 happens in the monoclinic crystal system of \textit{P}2\textsubscript{1}/c space group. The dissymmetric unit of 2 contains two independent
moieties: a part that includes a Zn(II) ion, a 3,3'-bpdc ligand and a half 4-bpmh ligand; the other including two Zn(II) ions, two 3,3'-bpdc ligands and a 4-bpmh ligand. From the Figure 2, three crystallographically independent Zn(II) ions are penta-coordinated with twisted tetragonal pyramid structures defined by carboxyl oxygen atoms (O1, O6, O4a, O7b for Zn1, O2, O5, O3a, O8b for Zn2, O9, O10e, O11d, O12f for Zn3) from four different 3,3'-bpdc ligands constituting the equatorial plane and a nitrogen atom (N1 for Zn1, N4c for Zn2) from one 4-bpmh ligand occupying the vertex. In 2, all bond length (Å) of Zn-O and Zn-N are within interval of 2.021(4)-2.062(4) and 2.014(4)-2.047(5) (Table S1), which are at the average level comparing to existed literature [21]. Three crystallographically independent 3,3'-bpdc ligands are all in $\mu_4$-bridging mode but with different dihedral angles of 30.62°, 29.79°, 34.52° between two benzene rings (Figure S1b). As shown in Figure 2b, both adjoining Zn(II) ions are connected to the $[\text{Zn}_2(\text{COO})_4]$ building block in shape of paddle with Zn...Zn length of 2.9 Å by four double monodentate groups. Besides, the $[\text{Zn}_2(\text{COO})_4]$ building blocks are linked with 3,3'-bpdc ligands to offer a 2D layer parallel to crystallographical bc plane (Figure 2c). At last, these neighboring layers then become pillars by the 4-bpmh ligands to generate a columnar layer form (Figure S2). Interestingly, the two independent moieties in the asymmetric unit grow into the same 3D pillar-layer framework by the bridging organic ligands. Viewed from the crystallographical c direction, there are large 1D channels. They are so large that additional two identical 3D pillar-layer structure can fit in. Therefore, compound 2 has 3-fold interpenetrating 3D columnar-layer structure (Figure 2d). In 2, each paddlewheel shaped $[\text{Zn}_2(\text{COO})_4]$ unit is linked with each hexa neighbor ligands through four 3,3'-bpdc ligands and two 4-bpmh ligands, and each organic ligand (3,3'-bpdc or 4-bpmh) links two different $[\text{Zn}_2(\text{COO})_4]$ units. By topological simplification, we can simplified
compound 2 as a 3-fold interpenetrating pcu topology with \( \{4^{12}\cdot6^3\} \) point symbol by looking \([\text{Zn}_2(\text{COO})_4]\) units as 6-linked points and organic ligands (3,3’-bpdc and 4-bpmh) as linear linkers (Figure 2e).

### 3.3. Powder X-ray diffraction patterns (PXRD) and thermogravimetric analyses (TGA)

The satisfactory matches between PXRD pattern of the obtained sample and the simulation result of the single crystal diffraction data indicates a large number of samples of 1 are pure phases, which was also supported by the elemental analyses (Figure S3).

The thermostability of compounds 1-2 was evaluated by TGA experiment in N\(_2\) atmosphere and within 30-800 °C. Figure 3 plots the TGA curve of compound 1-2. The plot of compound 1 shows no obvious weight loss before 330 °C, and dramatically weight loss occurs between 330-480 °C due to the organic ligands has disassembled, and the remainder left was CoO (obsd:16.14%, calcd:16.24%). The plot of compound 2 shows no significant weight decrease before 320 °C, and after which temperature, the skeleton of 2 begins to collapse for the investigation of the thermostability of compounds 1-2, thermogravimetric because the organic ligands decomposed. 19.67% of the last remainders could be powdery ZnO (calcd: 19.72%).

### 3.4. Photocatalytic property of compound 1

Co(II)-based coordination polymers is widely recognized as an ideal photocatalysts to decompose organic dyes [22, 23]. This study chose methylene blue (MB), one of the refractory organic dyes, to be a pollutant basis to measure the photocatalytic ability of compound 1. From what is demonstrated in Figure 4a, as the irradiation time increases, the maximum sorption of MB solution of 662 nm constantly reduces in the presence of compound 1 as photocatalyst. The changes of \( C/C_0 \) for MB solution against reaction time were plotted in Figure 4b. The MB degrades inconspicuously for the blank control group, and the effective rate is only 4.2% after 100 min. When compound 1 was added as photocatalyst, the degradation efficiency of MB solution can reach to 95.3% with the irradiation time of 100 min. After photocatalytic experiments, the sample of compound 1 was separated by centrifugation to investigate its photostability. From Figure S3a, the PXRD pattern is perfectly congruent with the original sample of compound 1. In addition,
concentration of the Co(II) ion after the photocatalytic progress has been studied via the inductively coupled plasma atomic emission spectrometer (ICP-AES), which reflects that only 2.3 mg/L of Co(II) ion was present in the filtrate, indicating little Co(II) ion was leaking during the photodegrade the MB progress. The conclusions of compound 1 inform that it can be a desired and steady photocatalyst to photodegrade the MB with ultraviolet condition.

3.5. Luminescence property of compound 2

Due to the excellent luminescence of d\(^{10}\) transition metal coordination polymers [24], the luminescence characteristics of compound 2 and free organic ligands (3,3' H\(_2\)bpdc and 4-bpmh) were evaluated under room temperature, and Figure 5 shows the corresponding luminescence spectra. When excited at 350 nm, the luminescence spectrum of compound 2 shows one emission band with the supreme emission top value at 469 nm, and the emission spectra for 3,3' H\(_2\)bpdc and 4-bpmh exhibit maximum emission at 456 nm (\(\lambda_{ex} = 350\)nm), 417 nm (\(\lambda_{ex} = 350\)nm), respectively, which was caused by the \(\pi^*-\pi\) or \(\pi^*-\pi\) charge transfer [25]. It is well known that the Zn(II) center has difficulty in oxidizing or to reducing, thus the luminescence of compound 2 can be assigned to intraligand charge transfer or between ligands transfer. The obvious redshift of compound 2 is possibly because the ligand is coordinated to metal centers that improve the stiffness of ligands with high efficiency.

3.6. Compound 1 could significantly reduce the relative expression of the estrogen receptor on the ovarian epithelial cells

Since synthesized compounds 1 and 2, the application values for the polycystic ovary syndrome were determined. Thus, the relative expression of the estrogen receptor on the ovarian epithelial cells was measured by using real time RT-PCR. From plots listed of Figure 6, it can be summarized that the relative expression of the estrogen receptor on the ovarian epithelial cells was distinctly reduced in the model group, which is obviously different from the control experiment. Afterwards, compound 1 was treated, then the relative expression of the estrogen receptor on the ovarian epithelial cells was increased, but
3. Compound obviously inhibited the levels of progesterone in the plasma

In the above experiments, compound 1 is able to obviously increase the estrogen receptor expression on the ovarian epithelial cells. In addition to this, the influence of the compound 2 displayed slightly affect the relative expression of the estrogen receptor on the ovarian epithelial cells.

3.7. Compound obviously inhibited the levels of progesterone in the plasma

In the above experiments, compound 1 is able to obviously increase the estrogen receptor expression on the ovarian epithelial cells. In addition to this, the influence of the
compounds on the levels of progesterone in the plasma was also measured with ELISA detection kit. The results in Figure 7 showed that compound 1 could also increase the levels of progesterone in the plasma. According with above experiments, compound 2 also demonstrated a slight influence for the levels of progesterone in the plasma.

4. Conclusions

To summarize, this study designed reasonably two latest mixed-ligand-based inorganic-organic hybrid compounds as well as the synthesis procedure have been conducted in hydrothermal environment with great success. The obtained two compounds display three-dimensional columnar layer structure with 6-linked pcu topological network. What is different is that compound 1 is non-interpenetrating 3D framework, while compound 2 is 3-fold interpenetrating structure. The photocatalytic investigation for compound 1 indicates that it can be used as high quality and steady photocatalyst to photodegrade the MB with ultraviolet condition. The solid-state luminescent spectrum shows that compound 2 shows high-level luminescent capability under room temperature. The output data by real time RT-PCR demonstrated that compound 1 is capable to distinctly increase the relative expression for the estrogen receptor on the ovarian epithelial cells, but not compound 2. Moreover, compound 1 could lead greater decrease of the progesterone levels of plasma compared to compound 2. At last, it can be concluded that compound 1 could be an excellent candidate for the polycystic ovary syndrome by regulating the expression of estrogen receptor.

Disclosure statement

No potential conflict of interest was reported by the author(s).
**Data availability**

Selected bond lengths (Å) and angles (°) for compound 1 (Table S1), the coordination modes of the organic carboxylate ligands (a) for 1,3-pde (b) for 3,3′-bpdc (Figure S1), the single framework of 2 (Figure S2), the PXRD patterns (a) for compound 1 and (b) for compound 2 (Figure S3), the information could be found in the supporting information file.

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