Two novel 2D coordination polymers constructed from 5-aminoisophthalic acid and 4,4′-bipyridyl ligands: Syntheses, crystal structure, and photocatalytic performance

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A B S T R A C T
Two coordination polymers, [Zn(5-aip)(bpy)0.5]·H2O (1), and [Ni(5-aip)(bpy)0.5(H2O)]·2H2O (2), constructed by 5-aminoisophthalic acid (5-H2aip), 4,4′-bipyridine (bpy), and corresponding ions, have been synthesized under hydrothermal conditions, and characterized by single crystal X-ray diffraction, Fourier transform infrared spectroscopy (FTIR), CHN elemental analysis and UV–Visible diffuse reflectance spectra (UV–Vis DRS). Both 1 and 2 were built up of two-dimensional [Zn(5-aip)(bpy)0.5] and [Ni(5-aip)(bpy)0.5(H2O)] sheets, respectively. The UV–Vis DRS results revealed that the Eg values of 1 and 2 are 3.4 and 2.4 eV, respectively. The photocatalytic activities of degradation of methyl orange (MO), methylene blue (MB) and MO&MB mixture over 1 and degradation of MB over 2 under UV light irradiation were investigated. The results revealed that 1 exhibited good performance to photocatalytically decompose MB, MO and MB&MO mixture, while 2 can only degrade MB efficiently.

1. Introduction
Owing to their diverse structures [1–3] and many potential applications like gas storage [4–6], catalysis [7,8], carbon dioxide capture [9] and gas separation [10,11], coordination polymers (CPs) have attracted tremendous attentions over the past decades years. The application of CPs as photocatalyst is emerging as an interesting topic since their cavity dimensions sit exactly between molecular bioenzymes and porous solid catalysts. In recent years, CPs have been used to solve environmental problems, for example, acting as a photocatalyst to conduct organic pollutants degradation, CO2 reduction and Cr(VI) reduction upon irradiation of UV/visible light [12–14]. However, most CPs (including MOFs) photocatalysts suffer from relatively poor stability in the presence of water, which limits their practical applications in solar energy utilization [12]. Organic carboxylate ligands were proposed to prepare water stable CPs, therefore the benzene-polycarboxylate ligands, as good candidates for the construction of coordination polymers, have aroused a good deal of interest from chemists [15]. For example, 5-aminoisophthalic acid as a derivative of the isophthalic acid has been widely used in the synthesis of various coordination polymers. Quite different from isophthalic acid, the introduction of an amino group in 5-aip2- provides more coordination mode to coordinate with metal ions, and creates conditions for the formation of hydrogen bond [16–20].

With this paper, we present two CPs, namely [Zn(5-aip)(bpy)0.5]·H2O (1), [Ni(5-aip)(bpy)0.5(H2O)]·2H2O (2), constructed from 5-aminoisophthalic acid (5-aip) and 4,4′-bipyridine (bpy), as shown in Scheme 1. The crystal structures, optical energy gap, along with the photocatalytic performance towards organic dyes were studied.

2. Experimental
2.1. Materials and instruments
All chemicals were commercially available reagent grade, and used without further purification. CHN elemental analyses were
obtained using an Elementar Vario EL-III instrument. FTIR spectra, in the region (400–4000 cm⁻¹), were recorded on a Nicolet 6700 Fourier Transform infrared spectrophotometer. UV–Vis diffuse reflectance spectra of solid samples were measured from 200 nm to 800 nm by PerkinElmer Lambda 650S spectrophotometer using BaSO₄ as the standard with 100% reflectance. Powder X-ray diffraction (PXRD) patterns of the solid samples were determined by a Dandonghaoyuan DX-2700B diffractometer in the range of 2θ = 5°–50° with Cu Kα radiation.

2.2. Synthesis of [Zn(5-aip)(bpy)₀.₅H₂O] (1)

A mixture of 0.0818 g (0.6 mmol) ZnCl₂ (>98%, J&K Chemicals), 0.0543 g (0.3 mmol) 5-aip (>98%, J&K Chemicals) and 0.0469 g (0.3 mmol) bpy (>98%, J&K Chemicals) with a molar ratio of 2:1:1 was sealed in a 25 mL Teflon-lined stainless steel Parr bomb containing deionized H₂O (15 mL) and 2.5 mL NaOH solution (0.1 mol/L), heated at 160 °C for 72 h, and then cooled down to room temperature. Orange rod-like crystals were isolated and washed with deionized water and ethanol (yield 89% based on ZnCl₂). Anal. Calcd. for C₁₃H₁₁N₂O₅Zn: C, 45.8; N, 8.2; H, 3.2. Found: C, 46.0; N, 8.1; H, 3.2. IR (KBr)/cm⁻¹: 3422(w), 3302(s), 3253(w), 3151(s), 3035(s), 2928(s), 1611(s), 1536(s), 1489(m), 1417(m), 1391(s), 1329(m), 1220(s), 1214(m), 1156(w), 1108(w), 1078(m), 1047(m), 966(w), 857(w), 810(s), 777(w), 727(m), 643(s), 634(s), 571(w), 497(w).

2.3. Synthesis of [Ni(5-aip)(bpy)₀.₅(H₂O) ]·2H₂O (2)

Green rod-like crystals of 2 (yield 75% based on NiCl₂) were synthesized from a mixture of 0.1426 g (0.6 mmol) NiCl₂ (>98%, J&K Chemicals), 0.0543 g (0.3 mmol) 5-aip (>98%, J&K Chemicals) and 0.0469 g (0.3 mmol) bpy (>98%, J&K Chemicals) in 2:1:1 M ratio under the same conditions as 1. Anal. Calcd. for 2, C₁₃H₁₅N₂O₇Ni: C, 42.2; N, 7.6; H, 4.1; Found: C, 42.0; N, 7.2; H, 4.0. IR (KBr)/cm⁻¹: 3276(m), 3167(m), 1612(m), 1564(s), 1520(s), 1488(m), 1442(m), 1417(m), 1391(s), 1329(m), 1223(w), 1136(w), 1108(w), 1078(m), 1048(w), 1003(w), 975(m), 934(w), 894(w), 822(w), 804(m), 800(m), 729(m), 681(m), 639(m), 596(w), 555(w), 522(w), 473(w), 444(w), 409(w).

2.4. X-ray crystallography

X-ray single crystal data collection for CPs 1 and 2 was performed with a Bruker Smart 1000 CCD area detector diffractometer with graphite-monochromatized Mo Kα radiation (λ = 0.71073 Å) using o-scan mode at 298(2) K. The SMART software [21] was used for data collection and the SADABS software [22] for data processing. Empirical absorption corrections were performed with the SADABS program [23]. The structures were solved by direct methods (SHEXLXS-97) [24] and refined by full-matrix-least squares techniques on Fl with anisotropic thermal parameters for all of the non-hydrogen atoms (SHEXLX-97) [24]. The hydrogen atoms of the organic ligands were added according to theoretical models, and those of water molecules were assigned with common isotropic displacement factors and included in the final refinement by use of geometrical restraints. The H atoms attached on O(W) in the water molecules were restrained with O–H = 0.85 Å. All structural calculations were carried out using the SHELX-97 program package [24]. Crystallographic data and structural refinements for CPs 1 and 2 are summarized in Table 1. Selected bond lengths and angles are listed in Table 2.

2.5. Photocatalytic experiments

The photocatalytic performance to degrade MO, MB and their mixture over CPs 1 and 2 under UV light irradiation was evaluated in a photocatalytic reaction system (Beijing Aulgite Co. Ltd.). Fifty micrograms of the CPs photocatalysts were added to 200 mL organic dye (10 mg/L) aqueous solution in a 300 mL breaker. The suspension was magnetically stirred in the dark for 30 min to achieve adsorption/desorption equilibrium before photocatalysis. During the photocatalytic degradation experiment, 1 mL sample was extracted using a 0.45 μm syringe filter (Shanghai Troody) at regular intervals for analysis. A Laspec Alpha-1860 spectrometer was used to monitor the dye concentration changes determined by the maximum absorbance at the corresponding wavelength, i.e. 463 nm for MO and 664 nm for MB.

3. Results and discussion

3.1. Structure of CPs 1 and 2

In CP 1, the Zn1, adopting a distorted tetrahedron geometry, is four-coordinated by one nitrogen atoms (N1 and N2) from a bis-

<table>
<thead>
<tr>
<th>Table 1</th>
<th>Details of X-ray data collection and refinement for CPs 1 and 2.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Formula</td>
<td>C₁₃H₁₁N₂O₅Zn</td>
</tr>
<tr>
<td>M</td>
<td>340.61</td>
</tr>
<tr>
<td>Crystal system</td>
<td>Monoclinic</td>
</tr>
<tr>
<td>space group</td>
<td>P2₁/c</td>
</tr>
<tr>
<td>a (Å)</td>
<td>12.3612(9)</td>
</tr>
<tr>
<td>b (Å)</td>
<td>7.6711(6)</td>
</tr>
<tr>
<td>c (Å)</td>
<td>16.1694(15)</td>
</tr>
<tr>
<td>α (°)</td>
<td>90</td>
</tr>
<tr>
<td>β (°)</td>
<td>108.949(2)</td>
</tr>
<tr>
<td>γ (°)</td>
<td>90</td>
</tr>
<tr>
<td>V (Å³)</td>
<td>1473.6(2)</td>
</tr>
<tr>
<td>Z</td>
<td>4</td>
</tr>
<tr>
<td>μ(Mo, Kα) (mm⁻¹)</td>
<td>1.688</td>
</tr>
<tr>
<td>Total Reflections</td>
<td>7127</td>
</tr>
<tr>
<td>Unique</td>
<td>2605</td>
</tr>
<tr>
<td>Fl(000)</td>
<td>692</td>
</tr>
<tr>
<td>Goodness-of-fit on Fl²</td>
<td>1.060</td>
</tr>
<tr>
<td>R₁</td>
<td>0.0475</td>
</tr>
<tr>
<td>wR₂</td>
<td>0.0503</td>
</tr>
<tr>
<td>R₁ (all data)</td>
<td>0.01455</td>
</tr>
<tr>
<td>wR₂ (all data)</td>
<td>0.1609</td>
</tr>
<tr>
<td>Largest diff. Peak and hole(e/Å³)</td>
<td>1.857, -0.403</td>
</tr>
</tbody>
</table>

Scheme 1. Structural formulante of 5-aip (a) and bpy (b).
monodentate bpy ligand, one nitrogen atoms from the amino group attached to a 5-aip ligand, and two oxygen atoms (O1 and O3) from two different bridging 5-aip ligand, as illustrated in Fig. 1a. The Zn–O distances are in the range of 1.942(3) – 1.985(3) Å, and the Zn–N distances are in the range of 2.038(4)–2.135(4) Å (Table 2), respectively, which matched well with the typical Zn–O and Zn–N bond length in other literature [18,25]. As illustrated in Fig. 1b and Scheme 2a, the completely deprotonated 5-aip acts as tri-dentate linker to coordinate three Ni2+ atoms and chelating mode (Ni–O = 2.098(3) Å and 2.193(3) Å) respectively, and the N atom of its amine group also link a Ni2+ atoms (Ni–N = 2.112(3) Å). The Ni–O and Ni–N bond distances are all as expected in the previously reported CPs [28–30]. As a result, the 2D framework can be reduced to new 1,2,3,5-connected net with Schläfi symbol (6^2:8^2:10^2:6^2)2(8) by analysis of TOPOS 4.0 [27], which is almost identical to CP 1 (Fig. 3c). Similar to CP 1, the adjacent [Ni(5-aip)(bpy)0.5(H2O)]2H2O sheets are connected into a three-dimensional structure through hydrogen bonds interactions [N1–O4: (-x+1, y+1/2, -z+3/2) 3.084 Å; N1–O2: (-x+1, y+1/2, -z+3/2) 2.994 Å], as shown in Fig. 4 and Table 3.

3.2. Infrared spectra & powder X-ray diffraction patterns

Broad bands observed at 3061 cm\(^{-1}\) for 1 and 3276, 3167 cm\(^{-1}\) for 2 can be assigned to the O–H stretching vibration, showing the presence of the lattice water molecules [31], respectively. Sharp bands at 1611, 1536 and 1489 cm\(^{-1}\) (for 1), 1612, 1564 and 1488 cm\(^{-1}\) (for 2) are attributed to the asymmetric and symmetric vibrations of the carboxylate groups attached to 5-aip\(^2+\) ligand. The characteristic absorption peaks of the bpy ligand are observed at 1416, 1220, 1047 and 727 cm\(^{-1}\) for 1, as well as 1417, 1223, 1048 and 729 cm\(^{-1}\) for 2. Powder X-ray diffraction (PXRD) was conducted to confirm the phase purities of 1 and 2 used in the experiments. The measured PXRD patterns agree well with those calculated from the X-ray single crystal diffraction data, indicative the good purity of the CPs 1 and 2 powder.

3.3. Optical energy gap

To explore the electrical conductivities of the CPs 1 and 2,
UV–vis DRS was used to obtain their corresponding band gap $E_g$ Refs. [32,33]. The plots of Kubelka-Munk function $F$ versus energy $E$ for these CPs are shown in Fig. 5, where steep absorption edges are displayed. The $E_g$ values were obtained as 3.4 and 2.4 eV for CPs 1 and 2, respectively, implying that both these CPs show selective absorption in the visible and ultraviolet spectrum region. This result can be interpreted as the different metal ions and coordination modes in CP 1 and CP 2. In CP 2, [Ni(bpy)$_{0.5}$]$^{2+}$ acts as sensitizer to reduce the energy required for electronic excitation and can be induced by UV light [34].

3.4. Photocatalytic activity

Considering their high surface areas and high light absorption abilities, CPs have attracted a lot of attentions for their application as photocatalysts in degrading organic pollutants [12,30,35–37]. MO, MB and the MO & MB mixture were selected respectively to assess the photocatalytic activities of CP 1 under UV light irradiation (500 W Hg lamp), while MB was used to test the photocatalytic activities of CP 2. Control experiment on degradation of MO and MB without addition of any photocatalysts was also performed. The photocatalytic performances of CPs 1 and 2 were monitored by measuring the maximum absorbance intensity at $\lambda = 463$ nm (for MO) and $\lambda = 664$ nm (for MB) to determine the residual concentration of MO and MB, respectively.

3.4.1. Photocatalytic degradation of individual MO and MB with CP 1

The photodegradation of individual MB and MO were carried out over CP 1 under UV irradiation, respectively. As shown in Fig. 6(a) and (b), when the simulated wastewater samples of MB and MO were irradiated under UV light, the maximum absorption peaks of MB and MO decreased obviously with the reaction time in the presence of CP 1. As illustrated in Fig. 7a, it can be seen that the
degradation of MB increased from 25.86% in control experiment without any photocatalysts to 77.65% under UV light irradiation within 120 min, and the MO degradation efficiency increased from 13.83% (without CP 1 photocatalyst) to 92.83% under the same conditions up to 60 min, which was comparable to other reported MOFs [12,37–39]. Additionally, both the photodegradation reactions of MB and MO over CP 1 photocatalyst followed pseudo-first-order kinetics model with $R^2 = 0.910$ and $R^2 = 0.941$, respectively, as evidenced by the linear plot of $\ln(C/C_0)$ vs. reaction time $t$. The pseudo-first-order rate constants for the photocatalytic degradation of MB and MO over CP 1 photocatalyst were 0.0041 and 0.018 min$^{-1}$, respectively. Furthermore, no other new peaks are observed in Fig. 6a (b), indicating that no new pollutants occurred during the process of degradation.

To explore the extent of CP 1 of use quantitatively during the photocatalytic process, the effect of different dose catalysts on the MO degradation was investigated. As shown in Fig. 7b, the MO degradation efficiency was 87.0%, 68.5% and 58.0% after 1 h’s UV light irradiation when 40 mg, 30 mg and 20 mg photocatalysts (CP 1) were added to the 200 mL MO solution (10 mg/L). It indicated that the dose of photocatalyst could significantly influence the organic dyes degradation efficiency.

3.4.2. Photocatalytic degradation of the mixture of MO and MB with CP 1

Further study of photocatalytic degradation activity was done on the MB&MO mixture. After 20 min of UV irradiation, the intensity of the absorption peak at 446 nm of MO was observed to decrease sharply, while intensity of absorption peak at 664 nm of MB reduced gradually, as illustrated in Fig. 8(b). Compared with the degradation efficiency of individual MO and individual MB, MO in the MB&MO mixture was decomposed more quickly within the first 40 min, while MB in the MB&MO mixture was degraded more slowly, demonstrating that MO was preferred to be decomposed.  

3.4.3. Photocatalytic degradation of individual MB with CP 2  

As shown in Fig. 9, it can be seen that the photocatalytic degradation of MB over CP 2 was as high as 68.2% under UV light irradiation for 120 min. Moreover, the photocatalytic degradation reactions of MB dye in CP 2 followed pseudo-first-order kinetics model with $R^2 = 0.969$, as evidenced by the linear plot of $\ln(C/C_0)$ versus reaction time $t$. The pseudo-first-order rate constants ($k$) for the photocatalytic degradation of MB in CP 2 photocatalyst was 0.0086 min$^{-1}$. Due to the different central atoms and coordination modes in CPs 1 and 2, their response to UV light is different, hence the photocatalytic performance of CP 1 was different to CP 2.

Table 3
Hydrogen bonds for CPs 1 and 2 [Å and °].

<table>
<thead>
<tr>
<th>D–H</th>
<th>d(D–H)</th>
<th>d(H..A)</th>
<th>&lt;DHA</th>
<th>d(D..A)</th>
<th>A</th>
</tr>
</thead>
<tbody>
<tr>
<td>(1)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>N1–H1A</td>
<td>0.900</td>
<td>2.222</td>
<td>164.53</td>
<td>3.099</td>
<td>01</td>
</tr>
<tr>
<td>N1–H1B</td>
<td>0.900</td>
<td>2.222</td>
<td>154.00</td>
<td>3.057</td>
<td>04</td>
</tr>
<tr>
<td>O5–H5C</td>
<td>0.850</td>
<td>2.002</td>
<td>179.38</td>
<td>2.852</td>
<td>02</td>
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<tr>
<td>O5–H5D</td>
<td>0.850</td>
<td>2.049</td>
<td>178.83</td>
<td>2.899</td>
<td>04</td>
</tr>
<tr>
<td>(2)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>N1–H1A</td>
<td>0.900</td>
<td>2.271</td>
<td>150.23</td>
<td>3.084</td>
<td>04</td>
</tr>
<tr>
<td>N1–H1B</td>
<td>0.900</td>
<td>2.131</td>
<td>160.26</td>
<td>2.994</td>
<td>02</td>
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<tr>
<td>O5–H5C</td>
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<tr>
<td>O5–H5D</td>
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<td>O5–H5D</td>
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<td>2.746</td>
<td>05</td>
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<tr>
<td>O7–H7D</td>
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<td>1.968</td>
<td>156.34</td>
<td>2.768</td>
<td>06</td>
</tr>
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</table>

Fig. 2. 3D framework built with the aid of hydrogen-bonding interactions along the b-axis for 1 and highlights of the hydrogen-bonding interactions.
Fig. 3. (a) Ortep view of [Ni(5-aip)(bpy)0.5(H2O)] in CP 2 and coordination environments around Ni(II) ion. Lattice water molecules and H atoms are omitted for clarity. (b) and (d) 2D structure of [Ni(5-aip)(bpy)0.5(H2O)] layer. (c) Simplified topology structure of CP 2, hydrogen atoms are omitted for clarity.

Fig. 4. 3D framework built with the aid of hydrogen-bonding interactions along the b-axis for 2 and highlights of the hydrogen-bonding interactions.
More researches should be continued to clarify what contributed to their different photocatalytic activities.

3.4.4. The proposed mechanisms for the photocatalytic reaction over CPs 1 and 2

Generally, the CPs’ band gap is closely related to the HOMO-LUMO gap, and energy transfer can take place from the organic linker to the metal within some CPs (including MOFs) under light irradiation [43]. The HOMO is mainly contributed by O and/or N 2p bonding orbitals, and the LUMO is mainly contributed by empty M (like Zn and Ni in this study) orbitals. The electron of the excited state in the LUMO is normally easily lost, while the HOMO intensively needs one electron to return to its stable state [30, 44, 45]. Therefore, one electron was captured from water molecules, which was oxygenated into the \( \cdot OH \) active species. Then, the \( \cdot OH \) could degrade MO or MB efficiently to complete the photocatalytic process, which was confirmed by \( \cdot OH \) trapping experiments [46, 47]. The results revealed that the addition 1 mM isopropanol (IPA) as radical scavenger inhibited greatly the degradation efficiencies of MO (decrease from 92.8% to 63.6% over CP 1 photocatalyst) and MB (decrease from 77.65% to 52.34% over CP 1 photocatalyst and from 68.2% to 56.8% over CP 2 photocatalyst) under UV light irradiation, which suggested that \( \cdot OH \) radical was the main active species in...
this system. A similar mechanism had been proposed recently for the degradation of organic dyes in the presence of similar CPs [12,38].

4. Conclusions

In all, the synthesis of two new coordination polymers based on 5-aminoisophthalic acid, and 4,4’-dipyridyl ligands has been achieved via hydrothermal method. CPs 1 and 2 had been characterized using X-Ray single crystal diffraction analysis, FTIR, CHN element analysis, UV-vis DRS. The crystal structure analyses revealed that CPs 1 and 2 were built up of two-dimensional [Zn(5-aip)(bpy)0.5]·H2O and [Ni(5-aip)(bpy)0.5(H2O)]·2H2O, respectively. It was assessed that both CPs 1 and 2 has selective absorption in the ultraviolet region, as the $\varepsilon_g$ values of both CPs 1 and 2 are 3.4 and 2.4 eV, respectively. And CP 1 showed efficient photocatalytic degradation of MO, MB and MO&MB mixture under UV light irradiation, while CP 2 exhibited good performance to photocatalytically degrade MB. In addition, a possible photocatalytic mechanism was proposed and discussed, which was proved by trapping experiments of radicals with the addition of isopropanol as a radial scavenger. More researches should be conducted to explore the photocatalytic activities on other organic pollutants.

Acknowledgements

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Supplementary materials

CCDC 1434331 and 1434332 contain the supplementary crystallographic data for compound 1 and 2. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.

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