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RESEARCH ARTICLE



2D network structure of zinc(II) complex: A new easily accessible and efficient catalyst for the synthesis of pyrazoles

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Abstract

A Zn(II) coordination polymer { $[Zn_3(paz)(btc)_2(H_2O)_8] \cdot 4H_2O\}_n$ (noted as **ZnPB**), where paz = pyrazine, H₃btc = trimesic acid, was synthesized and characterized by single-crystal X-ray diffraction, elemental analysis, infrared spectroscopy and powder X-ray diffraction analyses. **ZnPB** shows a (3,3)-connected 2D network with a point symbol of $(6^3)_2$. Hydrogen bonds and $\pi \cdots \pi$ interactions connect adjacent 2D network to form a 3D packing structure. This compound exhibits excellent catalytic activity in the reaction of condensation and cyclization of sulfonyl hydrazides with 1,3-diketones for the synthesis of pyrazoles. This solvent-free and halogen-free catalytic system represents an effective economic and environmentally friendly method for the construction of pyrazoles.

K E Y W O R D S

2D network, condensation reaction, hydrazine, pyrazoles, zinc(II) complex

1 | INTRODUCTION

Nitrogen-containing heterocycles are key core structures that form the basis of many natural products, pharmaceutical and agrochemical.^[1-7] Among them, pyrazole moiety is synthetic unit of utmost importance in the pharmaceutical industry,^[8,9] which is known to have abundant of strong biological activity such as antipyretic,^[10] antibacterial,^[11] and pesticidal.^[12] Nowadays, as privileged structure, pyrazole core skeleton are found in many drugs, such as celecoxib (A),^[13] lonazolac (B),^[14] and penthiopyrad (C),^[14] which are already booming in the market (Figure 1). Given its extreme

importance, various synthetic approaches have been developed for the synthesis of pyrazoles over the past decades. It can be synthesized via 1.3-dipolar cycloaddition of diazo compounds,^[15] reaction of acetylenic ketones,^[16] *N*-sulfonylhydrazones,^[17] or chalcones^[18] and hydrazines.^[19] But the direct condensation of hydrazines with 1,3-diketones is a more efficient method to successfully obtain pyrazole derivatives, it represents the most straightforward and effective method owing to significantly high overall reaction efficiencies and improved atom economy.^[20] In this regards, various catalysts have been explored to catalyze this reaction with water as sole H_2SO_4 ,^[10] $Sc(OTf)_{3}$,^[21] by-product. For instant,



FIGURE 2 Coordinating environment of (a) ZnPB, (b) btc ligand, and (c) paz ligand

 $P_2O_5 \cdot SiO_2$, ^[22] nano-SSA, ^[3] (CH₃PO₃)_{1.2}(O₃PC₆H₄SO₃H)_{0.8}], ^[24] PSSA,^[23] [a-Zr and $(C_2H_8N)_{12}Na_2[H_{10}{Ce(H_2O)_5}_2(Te_2W_{37}O_{132})]\cdot 39H_2O^{[25]}$ have been developed for the preparation of pyrazoles via condensation of hydrazines with 1,3-diketones. Although these reported procedures have their own merits, they also have suffered from various drawbacks, such as harsh reaction conditions, inconvenient operation, large loading, and difficult preparations of the catalyst. And the reaction was usually carried out in homogeneous solution thus far. Thus, product-catalyst separation remains a big challenge, as homogeneous catalysts are difficult to be separated from the product mixtures for recycling, which strongly hamper their extensive application. Nonetheless, despite the significance of these reports, it is highly desirable to develop a facile, efficient approach to synthesize pyrazoles using an inexpensive heterogeneous catalyst, with excellent activity, in an operationally simple and environmentally friendly manner.

As part of ongoing research on green catalysis chemistry,^[26-31] we focus on the coordination polymers (CPs) constructed by transition-metal ions and organic ligands, which had been studied widely in the past decades due to their multifaceted chemical and physical properties as well as the potential application in gas/ion adsorption, catalysis, conductivity, luminescence, sensor, and so on.^[32] CPs constructed by Zn(II) ions or clusters show advantages in heterogeneous catalysis due to the d¹⁰ electron configuration and conjugative effects. For instance, Tran et al. reported the synthesis of 2-benzylidenemalononitrile catalyzed by ZIF-8.^[33] Arai et al. reported a novel trinuclear Zn(II) complex and catalyzed asymmetric iodolactonization in up to 99.9% ee.^[34] Markad et al. synthesized two new Zn(II) CPs for the catalyze of Michael addition of malononitrile to 2-enoylpyridines with 99% conversion.^[35] All above mentioned indicated that Zn complexes have a good role in catalytic organic synthesis.

Hence, we reported a Zn(II) CP {[Zn₃(paz) (btc)₂(H₂O)₈]·4H₂O}_{*n*} (**ZnPB**), which was constructed by cheap pyrazine and trimesic acid in conventional aqueous solution. Single-crystal X-ray analysis reveals that **ZnPB** shows a (3,3)-connected 2D network with a point symbol of $(6^3)_2$; hydrogen bonds and $\pi \cdots \pi$ interactions connect adjacent 2D network to form a 3D packing structure. This compound exhibits excellent catalytic activity in the reaction of condensation and cyclization of sulfonyl hydrazides with 1,3-diketones for the synthesis of pyrazoles. This solvent-free and halogen-free catalytic

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system represents an effective economic and environmentally friendly method for the construction of pyrazoles (Scheme 1).

2 | RESULTS AND DISCUSSION

2.1 | Crystal structure

Single-crystal analysis shows that **ZnPB** has a (3,3)connected 2D network with P-1 space group. The



FIGURE 3 (a) The 2D network of ZnPB and (b) schematic depiction of the (3,3)-connected 2D network



FIGURE 4 Schematic depiction of the (a) hydrogen bonds and (b) $\pi \cdots \pi$ interactions in **1**





FIGURE 5 Recyclability of **ZnPB** in direct condensation reaction of **1a** and **2a** under the optimized reaction conditions

asymmetric unit of **ZnPB** consists of two Zn(II) atoms, half paz ligand, one btc ligand, four coordinating water molecules, and two solvent water molecules. Zn1 is coordinated with three carboxylate oxygen atoms from two btc ligands (O1/O3#1/O4#1), one nitrogen atom from one paz ligand (N1), and two coordinating water molecules (07/08) with a distorted octahedral geometry. Zn2 is located in the center of a slight distorted octahedral geometry, coordinated with two carboxylate oxygen atoms (O5/O5#2) from two btc ligands and four water molecules (09/010/09#2/010#2) (Figure 2a). The btc ligands in **ZnPB** show monodentate mode in two carboxylate group (O1O2/O5/O6) and monochelating mode for O3O4. Thus, one btc ligand coordinates with three Zn(II) atoms (Zn1/Zn2/Zn1#4) (Figure 2b). The paz ligand shows a C_2 symmetry and coordinates with two Zn1 atoms (Zn1/Zn1#3) (Figure 2c).

Topologically, each Zn1 is coordinated with two btc ligand and one paz ligand, which can be simplified as a three-connected node. The btc ligands in **ZnPB** can also

TABLE 1 Crystallographic data of Z	ZnPB
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Complex	ZnPB
CCDC No.	2052269
Formula	$C_{22}H_{34}N_2O_{24}Zn_3$
Fw	906.62
T/K	293.15
Crystal system	Triclinic
Space group	P-1
<i>a</i> (Å)	7.6645(11)
<i>b</i> (Å)	10.1903(14)
<i>c</i> (Å)	10.7263(15)
α (°)	87.218(2)
β(°)	89.250(2)
γ (°)	82.928(2)
$V(\text{\AA}^3)$	830.4(2)
F(000)	462
Ζ	1
$ ho_{ m calcd} ({ m g \ cm^{-3}})$	1.813
$\mu (\mathrm{mm}^{-1})$	2.249
Reflections collected	6615
Unique reflections	3541 ($R_{\rm int} = 0.0144$)
Parameter	248
GOF	1.053
$R_1^{a} (I \ge 2\sigma(I)]$	0.0300
wR_2^{b} (all data)	0.0921

 ${}^{a}R_{1} = \Sigma(||F_{0}| - |F_{c}||)/\Sigma|F_{0}|.$

 ${}^{b}wR_{2} = \Sigma [w(F_{0}^{2} - F_{0}^{2})^{2}] / \Sigma [w(F_{0}^{2})^{2}]^{1/2}.$

be simplified as a three-connected node. Both Zn2 and paz ligand are simplified as two-connected nodes because they only connect two btc ligands or Zn1 atoms. Thus, the three-connected Zn1 atoms and btc ligands are connected by two-connected Zn2 and paz ligands to form a 2D network (Figure 3a). The 2D structure of **ZnPB** can be simplified as a (3,3)-connected network with a point symbol of $(6^3)_2$ (Figure 3b).

TABLE 2 Evaluation of reaction

parameters

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		Chemistry		
	ONHNH2 S-NHNH2 O + 1a	<u>Conditions</u> 2a		, >
Entry	Catalyst (mol%)	Temperature (°C)	Time (min)	Yield ^a (%)
1	_	r.t.	15	29
2 ^b	ZnSO ₄	r.t.	15	41
3	ZnPB	r.t.	15	63
4	ZnPB	40	15	69
5	ZnPB	60	15	75
6	ZnPB	80	15	82
7	ZnPB	80	30	93
8	ZnPB	80	60	99
9 ^c	ZnPB	80	60	92

Note: Reaction conditions: naphthalene-2-sulfonohydrazide **1a** (1 mmol), 1,3-diketone **2a** (1 mmol), **ZnPB** 5 mol%, EtOH (0.1 ml).

^aThe yields were determined by GC with biphenyl as the internal standard.

^bThe loading of ZnSO₄ based on Zn²⁺.

^cThe loading of **ZnPB** 3 mol%.

Four coordinated water molecules and two solvent water molecules in **ZnPB** connect adjacent 2D network with hydrogen bonds and $\pi \cdots \pi$ interactions to form a 3D packing structure (Figure 4a,b). The distances of donator and acceptor of hydrogen bonds are 2.763(4) Å for O7 \cdots O1#1, 2.692(3) Å for O8 \cdots O6#3, 2.731(3) Å for O9 \cdots O4#4, and 2.786(3) Å for O10 \cdots O2#5. And the solvent water O12 also connects adjacent 2D network with hydrogen bonds with the distances of 2.788(4) Å for O12 \cdots O2#6 and 2.795(4) Å for O12 \cdots O5#7. The benzene rings of btc ligands from adjacent 2D network have $\pi \cdots \pi$ interactions with the distance of 3.924 and 4.134 Å for the centers of benzene rings. The FT-IR spectrum and PXRD patterns of **ZnPB** is shown in Figures S1 and S2.

2.2 | Catalytic activity

To the best of our knowledge, Zn CPs has excellent Lewis acid catalytic activity. In order to explore the catalytic performance of **ZnPB**, in the preliminary experiments, we performed condensation cyclization reaction with naphthalene-2-sulfonohydrazide **1a** and 1,3-diketone **2a** as the substrates and screened a series of reaction conditions. First, the control experiment indicated the reaction could be occurred without a catalyst (Table 2, Entry 1). The catalytic performance of ZnSO₄ as the raw material for the synthesis of **ZnPB** was evaluated, and the yield

was only 41% (Table 2, Entry 2). When the **ZnPB** (5 mol %) was employed as catalyst under the identical conditions, the desired product **3a** was obtained in 63% yield (Table 2, Entry 3). Afterward, the reaction temperature, the reaction time, and the loading of **ZnPB** were explored (Table 2, Entries 4–9). The results clarified that both the increase of reaction temperature and the extension of reaction time could make the reaction proceed smoothly. Using 5 mol% **ZnPB** at 80°C, 60 min produced the best outcome (Table 2, Entry 8).

The substrate scope was explored under optimized conditions by using 5 mol% ZnPB as the catalyst. Table 3 shows the results of ZnPB-catalyzed condensation cyclization of sulfonyl hydrazides with 1,3-diketones. Naphthalene-2-sulfonohydrazide 1a reacted with 1,3-diketone 2a under the optimized reaction conditions, giving 99% yield of the desired pyrazole product 3a (Table 3, Entry 1). Benzenesulfonyl hydrazide derivatives with functional groups are suitable for this reaction with a slight adjustment of reaction temperature and reaction time. Both electron-withdrawing and electron-donating substituted groups in benzene ring of benzenesulfonyl hydrazide reacted with acetylacetone, producing the desired products in excellent yields (Table 3, Entries 2-8). To our delight, benzenesulfonyl hydrazide bearing halo substituted groups such as Cl and Br could also converted to the desired pyrazoles (Table 2, 3e-3h), which provides the potential for further functionalization via cross

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TABLE 3 Substrate scope of ZnPB-catalyzed condensation cyclization reaction



TABLE 3 (Continued)



Note: Reaction conditions: hydrazide **1** (1 mmol), 1,3-diketone (1 mmol), **ZnPB** (5 mol%), EtOH (0.1 ml). ^aIsolated yield.

coupling. Furthermore, the difference in yields of 3e-3g indicated the same substituents at different positions had slight influence on the efficiency of this transformation (1e > 1f > 1g). Subsequently, the scope of the reaction was further explored using a variety of 1,3-diketones. 3-Methylpentane-2,4-dione **2b** and 3-chloropentane-2, 4-dione **2c** could also react well with various benzenesulfonyl hydrazide, delivering an excellent yields of 96% to 99% (Table 3, Entries 9–12).

Moreover, the gram-scale synthesis was investigated under identical conditions (Scheme 2). To our delight, the present protocol was found to be scalable and practical. The 97% and 93% yields of corresponding products were obtained, respectively, when the reaction was conducted on 5 mmol scale.

In addition, the facile separation of the product and the recyclability of the catalyst are important features of the designed catalyst system. The recycling experiments of naphthalene-2-sulfonohydrazide **1a** and **1**,3-diketone **2a** were carried out by using **ZnPB** as the catalyst under the optimal reaction conditions. The results shown in Figure 5 indicated that the reaction did not exhibit any significant change in yield after six cycles, the catalyst was stable under the reaction conditions, and **ZnPB** could be used at least for six times without significant loss of activity.

3 | CONCLUSIONS

In summary, a new (3,3)-connected Zn(II) CP **ZnPB** has been synthesized and fully characterized. **ZnPB** can be used as Lewis acid catalyst for the condensation cyclization to synthesize pyrazoles in high yields with low catalyst loadings and short reaction time. Importantly, this reaction could be scaled up, and the heterogeneous catalyst was used at least for six times. Investigations on the synthesis and potential catalytic performance of Zn(II) CPs are in progress.

4 | EXPERIMENTAL SECTION

4.1 | General methods and materials

All reagents were purchased and used without further purification. FT-IR data were obtained on a Bruker VERTEX 70 FT-IR spectrophotometer in the 4000- to 500-cm⁻¹ region. Powder X-ray diffraction (PXRD) was performed on a D/MAX-3C diffractometer with Cu-Ka radiation ($\lambda = 1.5406$ Å) at room temperature. The products were isolated by column chromatography on silica gel (200–300 mesh) using petroleum ether (60° C to 90° C) and ethyl acetate. All compounds were characterized by ¹H NMR, ¹³C NMR and mass spectroscopy, which were consistent with those reported in related literatures. NMR spectra were determined on Bruker Ascend 500 in CDCl₃. ¹H NMR chemical shifts were referenced to residual solvent as determined relative to CDCl₃ (7.26 ppm). The ¹³C NMR chemical shifts were reported in ppm relative to the carbon resonance of CDCl₂ (central peak is 77.0 ppm). ¹H NMR peaks were labeled as singlet (s), doublet (d), triplet (t), and multiplet (m). The coupling constants, J, are reported in Hertz (Hz). GC analyses were performed on a Shimadzu GC-2014C equipped with a capillary column (HP-5, 30 m \times 0.25 µm) using a flame ionization detector.

4.2 | X-ray crystallography

The single-crystal X-ray diffraction data were collected on Bruker D8 Smart Apex II diffractometer with graphite monochromated Mo K α radiation ($\lambda = 0.71073$ Å). Intensities were collected by ω -scan and reduced on *APEX II* (Bruker AXS Inc., 2009) and a multiscan absorption correction was applied.^[36] The positions of hydrogen atoms were determined with theoretical calculation. The structures were solved and refined on *Olex2* using *SHELXTL* package.^[37–39] Parameters of the crystal data collection and refinement are given in Table 1. Selected bond lengths and bond angles are given in Tables S1 and S2. Crystallographic files can be obtained via http://www. ccdc.cam.ac.uk/data_request/cif with the CCDC number of 2052269.

4.3 | Synthesis of ZnPB

ZnSO₄·7H₂O (0.12 mmol, 0.0345 g), H₃btc (0.06 mmol, 0.0126 g), and paz (0.12 mmol, 0.01 g) were dissolved in 20-ml H₂O, and the mixture was adjusted to pH = 7 with dilute NaOH solution. The mixture was stirred for 10 min and then filtered. The filtrate keeps stand still

at room temperature. Colorless block crystals of **ZnPB** were collected after one month. Anal. calc. for $C_{22}H_{34}N_2O_{24}Zn_3$ (1): C, 29.15; H, 3.78; N, 3.09%; found: C, 28.97; H, 4.01; N, 2.89%. IR (cm⁻¹): 3129 (m), 1606 (m), 1518 (m), 1470 (w), 1430 (s), 1355 (vs), 1109 (w), 1059 (w), 980 (w), 928 (w), 814 (w), 752 (s), 710 (vs), 654 (s), 607 (s), 561 (s).

4.4 | Typical procedure of ZnPBcatalyzed condensation reaction

To a 4-ml reaction vial, benzenesulfonyl hydrazide (1 mmol), 1,3-diketone (1 mmol), and **ZnPB** (5 mol%) were added. Then, the reaction was carried out in screwcap vials with a Teflon seal at 80°C for the desired time. After the reaction, the mixture was purified by column chromatography (petroleum ether/EtOAc) to afford the desired products. However, some products could also be obtained by recrystallization. In addition, the reacted catalyst can be washed three times with EtOAc and EtOH, respectively, and can be recycled after being dried under vacuum at 50°C for 3 h.

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AUTHOR CONTRIBUTIONS

Gang-Ming Cao: Data curation; investigation; methodology. Guo-Dong Zeng: Data curation; investigation. Ke Li: Formal analysis; funding acquisition; validation. Yu-Feng Liu: Funding acquisition; methodology. Xiao-Ling Lin: Data curation; methodology. Guo-Ping Yang: Funding acquisition; methodology; project administration.

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available in the supplementary material of this article.

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