

Synthesis of Porous Coordination Polymers Comprising Mixed Ligands of Triazole and Amino Triazole under Magnetic Fields and Its Effects in Enhance CO₂ Adsorptivity¹

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Synthesis of Porous Coordination Polymers Comprising Mixed Ligands of Triazole and Amino Triazole under Magnetic Fields and Its Effects in Enhance CO₂ Adsorptivity¹

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Abstract—A series of porous coordination polymers of Zn-oxac-ATaz (oxac : oxalic acid and ATaz: 3-amino,1,2,4-triazole), Zn-oxac-Taz (Taz: 1,2,4-triazole), and combination of ATaz–Taz ligand with 0.5 molar ratio *X* of Zn-oxac-Taz/ATaz were synthesized under magnetic fields. There is no even significant XRD pattern change of Zn-oxac-Taz 6T but there are noticeable changes in the morphology of irregular agglomerates at zero field to rectangular-prism crystals with smooth surfaces. Compared to the zero field, it is obvious that magnetic fields also bring significance morphology and crystal orientation change of Zn-oxac-ATaz 4T and Zn-oxac-Taz/ATaz 4T with growth to prolong rectangular morphology. Zn-oxac-Taz/ATaz 0T adsorbed more CO₂ (135 mg g⁻¹) at 303 K after heating in a vacuum at 333K for 12 h. It is suggested that the integration effects of pore space and amine group presences inside the frameworks induce to enhance carbon dioxide adsorption amount. Furthermore, due to magnetic fields the CO₂ adsorption by Zn-oxac-Taz/ATaz 4T also increases to 155.46 mg g⁻¹ and Langmuir surface areas developed is 467 m² g⁻¹. Magnetic fields cause interesting phenomena observed in XRD pattern and through morphological changes inducing the enhancement carbon dioxide (CO₂) capture in these porous coordination series.

Keywords: porous coordination polymers, ATaz–Taz ligands, CO₂ adsorption

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Porous coordination polymers (PCPs), organometallic complexes compound constructed by coordinating bonds between multi-dentate ligands and metal atoms, became new attractive porous materials possessing highly selective gas adsorption capability [1–6]. In [7–11] it was reported that porous coordination polymers might become a promising adsorbents for CO₂ separation. In the last few years, considerable efforts have been made to synthesize PCP materials. They are mainly synthesized by hydrothermal or solvothermal methods and the interesting technique in the synthesis of the material based on the selection of metal centers and design of synthesis ligands. Different combinations of metal center and ligands

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based on rational design ideas generate PCP material with various structures and preferential pore properties. Besides large surface areas and pore volumes, many PCP materials are well known to have specific interaction of CO₂ with functionalized aromatic molecules. They have shown effectiveness in introducing specific polar substituent groups, e.g., –OH, –COOH, –CH₃ or –NH₂, on the aromatic ligand in increasing the CO₂-ligand affinity [11].

Synthesized as amine functionalized PCP of Zn₂(C₂O₄)(C₂N₄H₃)₂·(H₂O)_{0.5} comprising of Zn²⁺, oxalic acid (oxac), and 3-amino,1,2,4-triazole (ATaz) [11], refers to Zn-oxac-ATaz, three dimensional frameworks of which are built from the pillar of Zn-amino triazole layers by

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¹ The text was submitted by the authors in English.

the oxalate groups. The oxalate ligands bind to zinc in a bidentate mode through two oxygen atoms from different carboxylate groups. The amino triazole ligand binds to trigonal bipyramidal zinc centers of three N-atoms of the ring, while the amino groups remain. This structure, apparently, is not porous, which indicates the absence of appreciable uptake of N₂, Ar, or H₂ under comparable conditions. This is caused by the presence of free amine groups within the pore, which impede the penetration of the guest molecule into the framework. Interestingly, the presence of amine groups inside the framework creates a significant amount of carbon dioxide in the frameworks. The potential of amine groups interacting with CO₂ molecules is currently well studied [11].

Magnetic fields (*H*, *T*) modify seriously structures and properties of materials, especially colloidal systems [12–16]. Homogeneous magnetic fields affect them thermodynamically via magnetic energy, $(1/2)\chi H^2$, which may induce the magnetic orientation, phase transition, etc. Magnetic field gradient also causes magnetic force, which causes convection, mass-transport, concentration change, etc. Previously, PCPs of $[Zn_2(Oxac)(Taz)_2] \cdot (H_2O)_x$, referred to ZOTW_x [17, 18], was synthesized from water-methanol solutions of Zn₅(CO₃)₂(OH)₆, 1,2,4-triazole (Taz), and oxalic acid (Oxac) at 453 K under various magnetic fields of upto 6 T [18]. Magnetic fields changed markedly the morphology of the irregular agglomerates at zero field to rectangular-prism crystals with smooth surfaces at 6 T, although its monoclinic crystal structure was only slightly deformed to tri-clinic one.

As-synthesized ZOTW_x(H) prepared under magnetic fields induced the formation of hydrophobic micropores having a lower water content and very uniform size [18]. However, there are a limited number of reports on frameworks having more than two kinds of ligands. In [9] it was reported on ligand mixing system synthesized as Zn₄O(bdc)_x(abdc)_{3-x}, in which the terephthalic linkers are partially substituted by 2-aminobenzene-1,4-dicarboxylate (abdc). Otherwise, the potential of the mixed-ligand approach in the framework of Z₄O(L)₃ (L = terephthalic acid derivatives) was extended by using of a high-throughput technique. In the frameworks of the structure, various terephthalic acid derivatives can be incorporated to create a porous structure, and one of them has eight terephthalic acid derivatives in the crystal structure. This fact suggests that the matching of the interval of ligands and comparable strengths of coordination bonds are the key to integrate the different ligands in the structure [19].

In the study, we reported of the effect of magnetic field on synthesis of PCP comprising of Zn²⁺, oxalic acid (oxac), and 3-amino,1,2,4-triazole (ATaz), Zn-oxac-ATaz, and PCP that contain 1,2,4-triazole (Taz), Zn-oxac-Taz, which have similar structure but it is necessary to compare the magnetic fields effects at the presence and absence of amine group in the frameworks. To investigate the effects of amine group interaction with CO₂ molecules by increasing pore space, herein we also report on the modified PCPs with 2 kind of ligands Zn-oxac-Taz/ATaz. The ligand of 1,2,4-triazole (Taz) with no amine groups, which can create a larger space, and 3-amino,1,2,4-triazole (ATaz) ligand with amine groups inside the pore are combined with 0.5 molar ratio under magnetic fields. The presence of amine groups (due to ATaz ligand) and larger space (due to Taz ligand) will serve to increase the CO₂ capture because of strong interaction of CO₂ molecules with –NH₂ groups under available space for guest molecules such as CO₂. Also we study how the CO₂ capture is enhanced in the frameworks synthesized under magnetic fields.

EXPERIMENTAL

Materials. Zn₅(CO₃)₂(OH)₆ was purchased from Alfa Aesar, Co. Ltd and used as sources of Zn(II). Oxalic acid, 1,2,4-triazole (Taz) and 3-amino,1,2,4-triazole (ATaz) were purchased from Wako Pure Chemical Industries, Ltd.; WPCI). Methanol (99%) and distilled water of commercial grade quality were used as solvents.

Synthesis of PCPs. Zn₂(C₂O₄)(C₂N₃H₂)₂·(H₂O)_{2.5} or Zn-oxac-Taz were prepared by following procedure [17]. Zn₅(CO₃)₂(OH)₆ (0.4 g), Oxac (0.4g of), and Taz (1.4 g) were added to a mixed solvent (12 mL of methanol and 2 mL of distilled water). The dispersion was transferred to a Teflon cell which was set in an autoclave vessel made of non-magnetic stainless steel (SUS316). The vessel was put in the center of magnetic fields (2, 4, and 6 T) of a JASTEC JMTD-6T150E1 superconducting magnet, and heated in the homogeneous magnetic field region within ±3% at 453 K for 12 h. Ther the system was cooled down to room temperature under magnetic fields; crystals prepared were filtered and washed with the solvent, followed by drying them in air at room temperature. Zn₂(C₂O₄)(C₂N₄H₃)₂·(H₂O)_{0.5} or Zn-oxac-ATaz also was synthesized according to report [11] using metal ion of Zn₅(CO₃)₂(OH)₆ (Alfa Aesar, Co. Ltd.). Amount Zn₅(CO₃)₂(OH)₆ (0.4 g) (Alfa Aesar, Co. Ltd.), oxalic

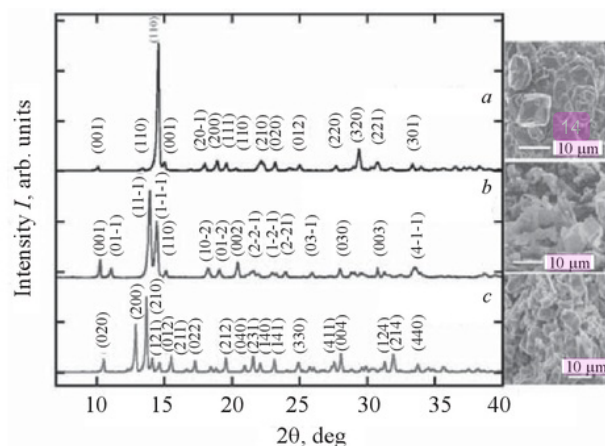


Fig. 1. XRD patterns and SEM images of as-synthesized (a) Zn-oxac-Taz, (b) Zn-oxac-Taz/ATaz, and (c) Zn-oxac-ATaz at 0T magnetic field.

acid (Oxac) (0.4 g) (Wako Pure Chemical Industries, Ltd.; WPCI), and 3-amino-1,2,4-triazole (ATaz) (1.6 g) (Wako Pure Chemical Industries, Ltd.; WPCI) were added to a mixed solvent (12 mL of methanol (WPCI) and 2 mL of distilled water). The preparation of mixed ligands of Zn-oxac-Taz/ATaz was also prepared by the similar procedure with use of 0.4 g of $Zn_3(CO_3)_2(OH)_6$, 0.4 g of $H_2C_2O_4$, 0.7 g of 1,2,4-triazole, and 0.8 g of 3-amino-1,2,4-triazole. These 3 of PCPs were synthesized under 2, 4, and 6 T magnetic fields.

Characterization. Crystals prepared were characterized by a Rigaku X-ray diffractometer (XRD) Multiflex with $CuK\alpha$ at 40 kV and 20 mA and a JEOL scanning electron microscope (SEM) JSM-7600F. Crystal parameters were measured by using of EXPO 2013 software [20] and analyzed directly from XRD patterns of each sample. The elemental analysis of C, H, and N was carried out with a Thermo Electron Flash EA1112 elemental analysis equipment. N_2 and CO_2 adsorption were measured at 77 and 303 K, respectively, by a custom made volumetric adsorption system, after each sample of about 100 mg was pretreated at 333 K and 1 mPa for 1 h and then 383 K for 12 h.

RESULTS AND DISCUSSION

White crystals of Zn-Oxac-Taz complexes were synthesized in the yield of ca. 70%. The composition was estimated as $[Zn_2(C_2O_4)(C_2N_3H_2)_2] \cdot (H_2O)_{2.5}$, which is referred to Zn-oxac-Taz, from the results of TG and elementary analyses (C : H : N = 18.02 : 2.27 : 21.02%). This crystal structure is similar with other PCPs of

$[Zn_2(C_2O_4)(C_2N_4H_3)_2] \cdot (H_2O)_{0.5}$ or Zn-oxac-ATaz which reported in [11]. Even though XRD patterns (Fig. 1) and solvent contents were slightly different from each other, different crystal systems formed as orthorhombic for Zn-oxac-ATaz and monoclinic for Zn-oxac-Taz were estimated by EXPO 2013 program [20]. On the other hand, by mixing the ligand of Taz and ATaz with 0.5 molar ratio referred to Zn-oxac-Taz/ATaz, a triclinic crystal system formed. XRD patterns exhibited the new crystal structure, indicated mixed ligands bonds formed on this complex and it was estimated by elemental analysis, that this PCPs refers as $[Zn_2(C_2O_4)(C_2N_4H_3)_{0.3}(C_2N_3H_2)_{1.7}] \cdot (H_2O)_{2.5}$.

The same molar fraction of reactants for both of these Taz and ATaz ligands induce more content of Taz ligand (6 times) bind with Zn metal center comparing to ATaz ligand. The framework formed should be characterized by more space in comparison with only Zn-oxac-ATaz framework, whereas both of the PCPs contain amine group inside of the frameworks. Synthesis under 4T and 6T magnetic field induced a slightly XRD intensity change in Zn-oxac-Taz and Zn-oxac-Taz/ATaz patterns, but synthesis Zn-oxac-ATaz under 2T and 4T magnetic field bring a significance change (Fig. 2).

The crystal structures estimated by the EXPO 2013 program [20] were changed for Zn-oxac-Taz 4T and 6T to triclinic and for Zn-oxac-Taz/ATaz were changed from orthorhombic (0T) to triclinic (4T) and monoclinic (6T). Otherwise, only Zn-oxac-ATaz changes into triclinic at 4T, and orthorhombic crystal system formed at 6T was the same as for 0T crystal. SEM image (Fig. 3) confirmed homogenous particles formed from mixed Taz and ATaz

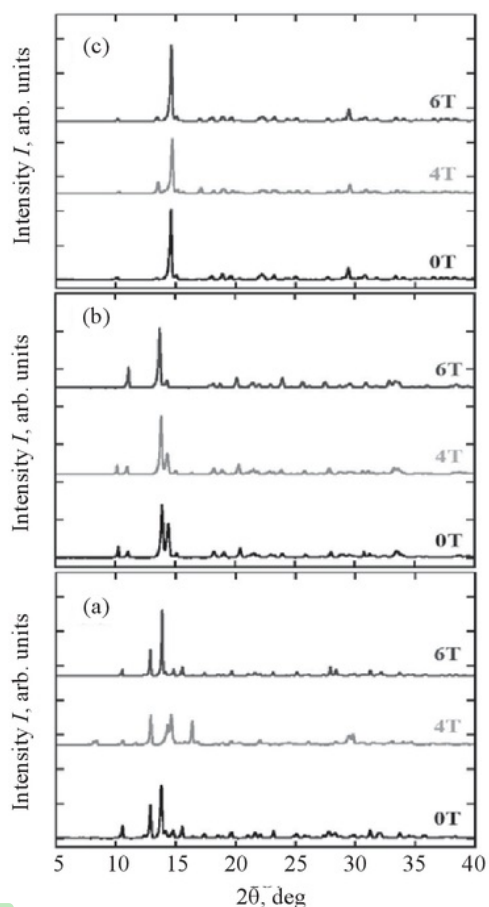


Fig. 2. XRD patterns of as-synthesized (a) Zn-oxac-ATaz, (b) Zn-oxac-Taz/ATaz ($X = 0.5$); and (c) Zn-oxac-Taz at $H, T = 0, 2, 4$, and 6 .

PCPs. The morphology also detects differences in each other indicating the different properties of these three PCP systems. Magnetic fields more than 2T also induces the morphology change in irregular agglomerates at zero field to rectangular-prism crystals with smooth surfaces and larger size at Zn-oxac-Taz, as seen in the SEM images (Fig. 3) [18].

Interestingly, the same phenomenon is also observed in morphology of Zn-oxac-ATaz growth to more prolonged rectangular morphology prepared for 4T magnetic field (Fig. 3). The same morphology change at 4T also observed in Zn-oxac-Taz/ATaz which was not detected at 6T crystal. Synthesis under 4T magnetic field of Zn-oxac-ATaz and Zn-oxac-Taz/ATaz bring the significant change in morphology and crystal orientation. Magnetic fields

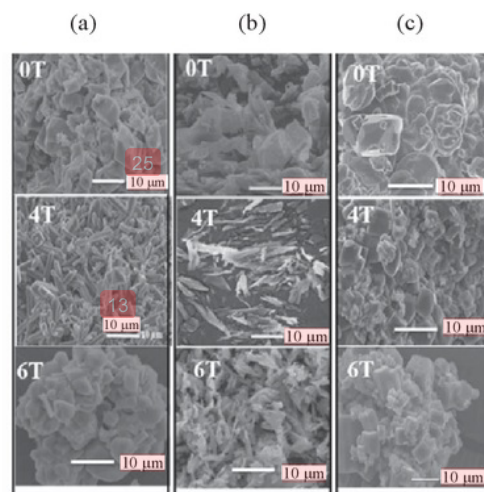


Fig. 3. SEM images of (a) Zn-oxac-ATaz, (b) Zn-oxac-Taz/ATaz ($X = 0.5$), and (c) Zn-oxac-Taz in various magnetic fields; $H, T = 0, 4$, and 6 .

of 2T and 4T induced morphology change in irregular agglomerates at zero field to rectangular-prism crystals at Zn-oxac-ATaz and Zn-oxac-Taz. This also results in a smooth surfaces and larger sizes at 6T crystal, as seen in the SEM images (Fig. 3). It indicates that the magnetic field effects appear from the depression of the nucleation process on the crystal surfaces. The transport of reactants due to convection and diffusion is depressed because of the gradients in the magnetic field and magnetic susceptibility on the solution. The crystal grows slowly to make the smoothly surface due to the interaction of magnetic fields with water [16] thus affecting crystal formation via hydrated reactants [15, 18].

Figure 5a shows that an as-synthesized Zn-oxac-Taz has little amount of N_2 adsorbed at 77 K, indicating that the micropores are filled with solvent. Since TG/DTA curves (Fig. 4) showed that solvent molecules included were desorbed between 330 and 388 K, the as-synthesized Zn-oxac-Taz for gas adsorption was pretreated at 383 K. Heating the as-synthesized Zn-oxac-Taz at 383 K, its weight decreased gradually until 3 h, rapidly between 4 and 8 h, and then reached a saturation value of solvent off (Fig. 5).

After short pretreatment the crystals still include some water to be expressed as $[Zn_2(Oxac)(Taz)_2] \cdot (H_2O)_x$ or Zn-oxac-Taz, where $x = 2.5 - \Delta$ (Δ is the amount of H_2O dehydrated). The pretreatment shorter than 3 h brought about little change in adsorption amount of N_2 , but over

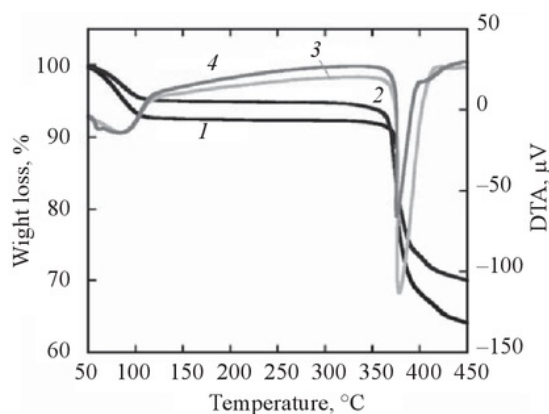


Fig. 4. TG analysis of (1) Zn-oxac-Taz, (2) Zn-oxac-ATaz; DTA analysis of (3) Zn-oxac-Taz, (4) Zn-oxac-ATaz.

4 h adsorption amount increased significantly from low pressure of Type I adsorption isotherms indicating an appearance of micropores due to removal of H₂O. The Zn-oxac-Taz pretreated over 12 h showed the saturation adsorption amount of ca. 190 mg g⁻¹ (0.23 mL g⁻¹) from the DR-plot and the pore diameter of 0.84 nm from the *t*-plot.

On the other hand, even with a similar structure with Zn-oxac-Taz, the presence of amine group inside the frameworks, induce almost no N₂ uptake on Zn-oxac-ATaz after pretreatment at 110°C for 12 h (Fig. 6a). This result also corresponds with data published in [11], which did not show any appreciable uptake of N₂, Ar or H₂. Also, CO₂ adsorption at 303 K was significant and consisted 72 mg g⁻¹ at 760 torr after pretreatment at 110°C for 12 h which contributed to CO₂ and -NH₂ interaction inside the frameworks

The presence around 10% ATaz at Zn-oxac-Taz/ATaz mixed of Taz and ATaz system as ligand with amine group inside the frameworks not strong enough to block the pore from the interpenetrate of gas molecules that shows significant amount of nitrogen adsorbed: around 160 mg g⁻¹ at 0.1 *p/p*₀ after pretreatment at 110°C for 12 h (Fig. 6a). As the water content of Zn-oxac-Taz/ATaz lower than Zn-oxac-Taz (Fig. 5), CO₂ molecule could adsorb in framework: around 79.5 mg g⁻¹ at high pressure region (760 torr) even no pretreatment time (0 h) (Fig. 6b). The integration effects of pore space and the amine groups presences inside frameworks are more explicable from carbon dioxide adsorption at 303 K. Despite PCPs of Zn-oxac-Taz/ATaz has lower space than Zn-oxac-Taz which is indicated by lower amount of N₂ adsorbed (Fig. 6a),

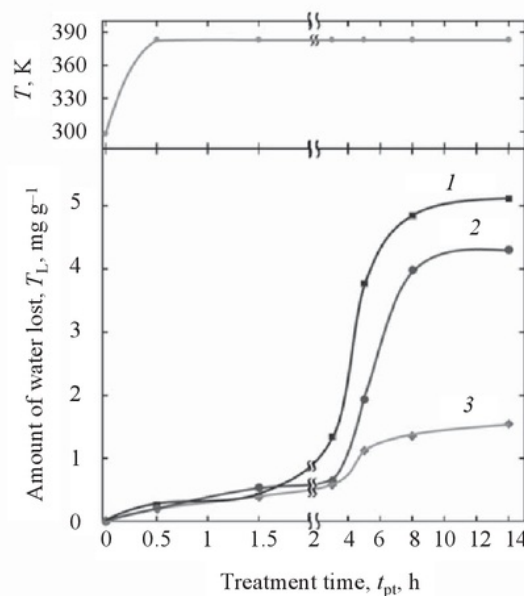


Fig. 5. Amount of water lost from as-synthesized (1) Zn-oxac-Taz, (2) Zn-oxac-Taz/ATaz (*X*: 0.5), and (3) Zn-oxac-ATaz at 0T magnetic field by heating at 298 K in first 10 min and subsequently to 383 K (upper).

but the presence a few amine group inside the pore induces strong interaction between CO₂ molecules with amine group to make larger access for CO₂ molecules in the pore. It shows preferential CO₂ adsorption, to 135 mg g⁻¹ at high pressure region (760 torr).

Figure 6a shows the nitrogen (N₂) adsorption after pretreating at 110°C during 12 h for all PCPs which were prepared at 0 and 4T (ATaz ligand and molar ratio 0.5) and at 0 and 6T for Taz ligand. Although Zn-oxac-ATaz prepared for 4T induces significance change in XRD patterns and interesting change in morphology, there are almost no changes in the amount of adsorbed nitrogen compared to synthesized 0T crystal. Preparing under magnetic fields has not enough effect for opening pores blocked by amine group inside the frameworks. Zn-oxac-Taz and mixed system of Zn-oxac-Taz/ATaz show increasing amount of nitrogen adsorbed at crystal which was prepared under magnetic field. Zn-oxac-Taz 6T showed the highest surface area and also Zn-oxac-Taz/ATaz 4T adsorbed higher nitrogen amount than Zn-oxac-Taz/ATaz 0T. The presence of 10% amount of ATaz ligand could create more space in the framework to allow penetration of nitrogen gases in the pores.

Although, amount of nitrogen adsorbed at Zn-oxac-Taz/ATaz is lower than that at Zn-oxac-Taz in lower

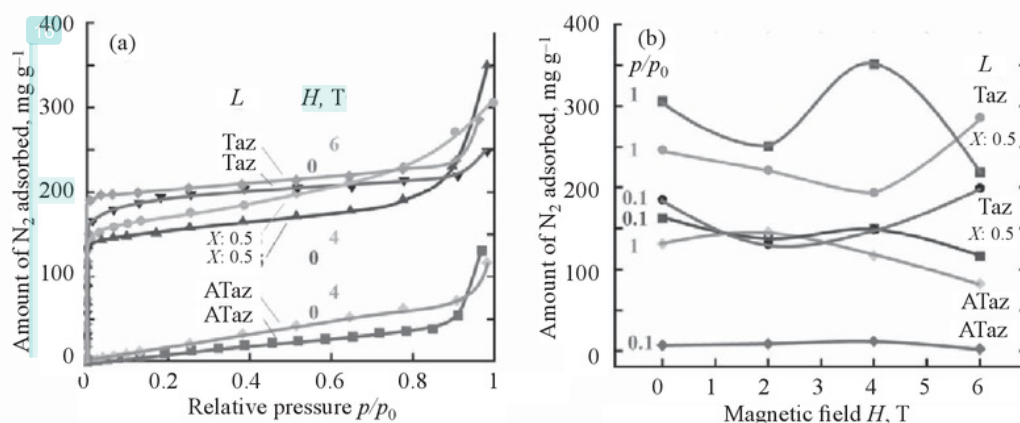


Fig. 6. (a) N_2 adsorption isotherms at 77 K of all PCPs after pretreating at 110°C for t_{pt} : 12 h at ATaz ligand and $X = 0.5$ ($H, T = 0, 4$) and at Taz ligand ($H, T = 0, 6$); (b) Magnetic field intensity as function of nitrogen adsorption amount at 0.1 and 1 of p/p_0 .

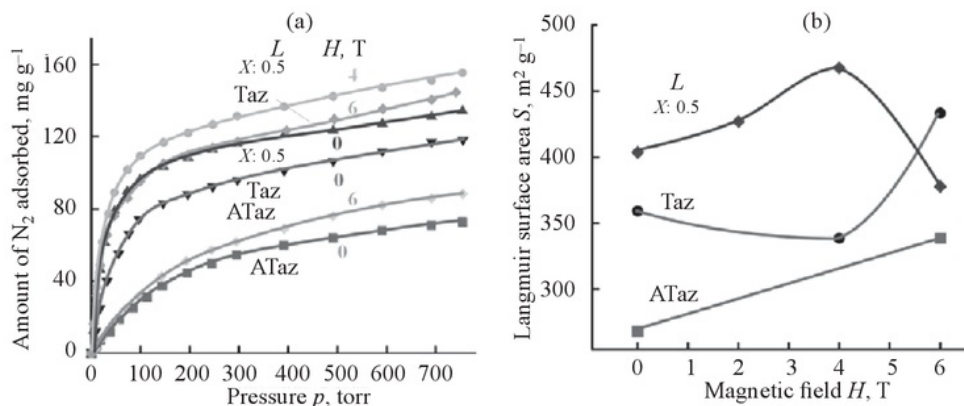


Fig. 7. (a) CO_2 adsorption isotherms at 303K of all PCPs after pretreating at 110°C for t_{pt} : 12 h at ATaz ligand and Taz ligand ($H, T = 0, 6$), and $X = 0.5$ ($H, T = 0, 4$). (b) Magnetic field intensity as function of Langmuir surface area.

pressure, but in high pressure, around $1 p/p_0$, Zn-oxac-Taz/ATaz 0T and 4T have higher adsorbed amount of nitrogen. It suggests that the presence of a few ATaz ligand induces opening pores due to interaction of introduced nitrogen gas to the frameworks with amine group inside the framework under high pressure. Zn-oxac-Taz 6T shows the highest surface area and Zn-oxac-Taz/ATaz 4T have the highest space pore at the high pressure ($1 p/p_0$) (Figs. 6a and 6b). Interestingly, all magnetic field crystals, such as Zn-oxac-ATaz 6T, Zn-oxac-Taz/ATaz 4T, and Zn-oxac-Taz 6T, show a higher level of CO_2 adsorption compared with each of the zero field crystals (Fig. 7a). According to adsorption isotherms CO_2 adsorption by Zn-oxac-Taz/ATaz is higher than that by

Zn-oxac-Taz (Fig. 7a) indicating that interaction between CO_2 molecule with amine group affects adsorbing CO_2 .

Combination of available space for CO_2 molecule to introduce into the pore from Taz ligand is also supported by ATaz ligand due to due to strong interaction of CO_2 molecules with amine group of ATaz ligand. In this mixture ligand system, magnetic field 4T not only induces the prolonged growth of rectangular crystal, they also can enhance CO_2 adsorption and create $467 \text{ m}^2 \text{ g}^{-1}$ Langmuir surface area. The magnetic field not only controls the growth of crystals: it must be slow to make the inner and outer surfaces of the crystal smooth. However, this effect on the morphology is also accompanied by a change in the adsorption properties within the framework. This suggests

that the hydrophobic surface of micropores with a lower water content in the PCP, as previously reported in [11], obviously creates conditions in the form of non-polar molecules in a crystal obtained in a magnetic field for convenient access to CO₂. Figure 7b demonstrates how strong magnetic field effects on enhancing Langmuir surface area. As it can be seen Zn-oxac-ATaz and Zn-oxac-Taz prepared under 6T magnetic field have higher Langmuir surface area, 339 m² g⁻¹ and 433.16 m² g⁻¹, respectively, than their 0T samples.

CONCLUSIONS

A PCP magnetic field crystals with hydrophobic micropores cause an increase in CO₂ adsorption. Also we success to create PCPs containing three kinds of ligands: oxalic acid, amino triazole, and triazole, combination of which allows the increase in the space available for CO₂ molecules penetration, thereby enhancing CO₂ adsorption by interaction of amine group with CO₂. Magnetic fields also enhance the CO₂ adsorption of Zn-oxac-Taz/ATaz 4T upto 155.46 mg g⁻¹ and create 467 m² g⁻¹ Langmuir surface area. Analysis of XRD patterns shows that magnetic fields bring an interesting phenomena and morphology change, which induce the enhancement of carbon dioxide capture in these porous coordination series.

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