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Communication

## Two-dimensional cobalt metal-organic frameworks for efficient C<sub>3</sub>H<sub>6</sub>/CH<sub>4</sub> and C<sub>3</sub>H<sub>8</sub>/CH<sub>4</sub> hydrocarbon separation

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## ABSTRACT

A Co-based two-dimensional (2D) microporous metal-organic frameworks, [Co<sub>2</sub>(TMTA)(DMF)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>] $\cdot$ NO<sub>3</sub><sup>-</sup>·DMF (UPC-32) has been synthesized based on 4,4',4''-(2,4,6-trimethylbenzene-1,3,5-triyl)tribenzoic acid (H<sub>3</sub>TMTA). UPC-32 features a 2D microporous framework exhibits high adsorption of H<sub>2</sub> (118.2 cm<sup>3</sup>/g, 1.05 wt%, at 77 K), and adsorption heat (Q<sub>st</sub>) of CO<sub>2</sub> (34–46 kJ/mol). UPC-32 with narrow distance between layers (3.8 Å) exhibits high selectivity of C<sub>3</sub>H<sub>6</sub>/CH<sub>4</sub> (31.46) and C<sub>3</sub>H<sub>8</sub>/CH<sub>4</sub> (28.04) at 298 K and 1 bar. It is the first 2D Co-MOF that showed selective separation of C<sub>3</sub> hydrocarbon from CH<sub>4</sub>.  
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C1 to C3 light hydrocarbons (CH<sub>4</sub>, C<sub>2</sub>H<sub>2</sub>, C<sub>2</sub>H<sub>4</sub>, C<sub>2</sub>H<sub>6</sub>, C<sub>3</sub>H<sub>6</sub> and C<sub>3</sub>H<sub>8</sub>) are important energy resources and raw chemicals. For example, natural gases, whose main component is CH<sub>4</sub>, have been considered as the most promising alternative fuel for future vehicle transportation, while C<sub>3</sub>H<sub>8</sub> are important chemical for the manufacture of fine chemicals such as acrylonitrile, acrylic acid, allyl alcohol, propylene oxide, isopropanol, glycerol as well as other organic chemicals. In order to fully utilize these light hydrocarbons, high quality and purity of them are essential [1–3], thus, separations of these light hydrocarbons, especially the separation of C<sub>2</sub> and C<sub>3</sub> hydrocarbons from CH<sub>4</sub> are very important industrial processes [4,5]. The traditional separation technology such as the cryogenic distillation and the pressure swing adsorption, which are based on their different vapor pressures and boiling points, are energy-intensive. Compared to the traditional separation technologies, adsorptive separation is one of the most promising low-cost and energy-efficient route. However, the traditional adsorbents, such as zeolites, have low separation coefficient owing to their less-varying porosity and low surface area. Hence, developing new-type and more effective adsorbents is critical for the effective separation of light hydrocarbons [6–9].

Metal-organic frameworks (MOFs), which are comprised of metal ions or clusters coordinated with organic ligands, have been regarded as a novel class of crystalline microporous materials with periodic network structures [10,11]. Owing to the pores within

such porous MOFs can be adjusted to maximize their size-selective sieving effects and the pore surfaces can be functionalized to direct specific recognition of small molecules, porous MOFs have been intensively investigated for applications in traditional gas storage and separation, such as N<sub>2</sub>, H<sub>2</sub>, CO<sub>2</sub>, and so on [12–16]. Recently, MOFs start to show ability in separation of light hydrocarbons [17–20]. For example, FJI-C1 [21] and FJI-C4 [22] have been reported to display high C<sub>3</sub>H<sub>8</sub>/CH<sub>4</sub> separation performance (78.7 and 293.4). The sizes and shapes of the pores in MOFs sorbents are the foremost for their separation performance. The pore size of an adsorbent which is comparable to or slightly larger than the kinetic diameters of the adsorbate will significantly promote the separation selectivity of these light hydrocarbons. Hence, the design and synthesis of MOFs with narrow pores close to 4.0 Å are crucial for the separation of light hydrocarbons (C1–C3) as their kinetic diameters range from 3.3 Å to 4.4 Å. In addition, based on the adsorbate-surface interactions, tailoring pore surface function, such as the immobilization of polar functional groups is another effective strategy to improve the MOFs' separation.

In this communication, we present an unprecedented 2D cobalt MOF, [Co<sub>2</sub>(TMTA)(DMF)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>] $\cdot$ NO<sub>3</sub><sup>-</sup>·DMF (denoted as UPC-32) by using a functional 1,3,5-tris(4-carboxyphenyl)benzene ligands H<sub>3</sub>TMTA. UPC-32 exhibits 2D layered structure with permanent porosity, high adsorption of H<sub>2</sub>, and high adsorption heat (Q<sub>st</sub>) of CO<sub>2</sub>. In addition, UPC-32 shows porosity-dependent C<sub>3</sub>H<sub>6</sub> and C<sub>3</sub>H<sub>8</sub> uptakes and selective C<sub>3</sub>H<sub>6</sub> and C<sub>3</sub>H<sub>8</sub> adsorption over CH<sub>4</sub>.

The purple block-shaped crystals of UPC-32 were synthesized by the solvent thermal reaction of Co(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O and H<sub>3</sub>TMTA. The synthesis details are shown in Supporting information. Single

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crystal X-ray diffraction (SCXRD) of UPC-32 revealed that it crystallizes in the monoclinic system with the  $P2_1/c$  space group. There are two  $\text{Co}^{2+}$  ions (Co1 and Co2), one  $\text{TMTA}^{3-}$  ligand, two coordinated DMF molecules and two coordinated water molecules in the asymmetric unit of UPC-32. Co1 and Co2 are connected by three carboxylic groups to form a  $\text{Co}_2(\text{COO})_3$  paddle-wheel cluster (Fig. 1a). The Co1 is coordinated by three oxygen atoms from three different carboxylic groups and one oxygen atom from water molecule with the average Co1–O bond length of 2.024 Å. The Co2 is coordinated by three oxygen atoms originating from three  $\text{TMTA}^{3-}$  ligands, two oxygen atoms from coordinated DMF molecules and one oxygen atom from coordinated water molecule with the average Co2–O bond length of 2.039 Å. The  $\text{Co}_2(\text{COO})_3$  clusters are connected by three carboxylic groups of  $\text{TMTA}^{3-}$  ligands to form a 2D double-layer framework. The single neighboring layer of UPC-32 stack on top of each other by  $\pi$ - $\pi$  interactions (3.8 Å) (Fig. 1b) to form a 3D supramolecular architecture with large pores (15.8 × 15.8 × 3.8 Å<sup>3</sup>) (Fig. 1c). The potential void calculated by the PLATON [23] software is 53.7% of the total volume (2395.5 Å<sup>3</sup> out of the 4456.3 Å<sup>3</sup> unit cell volume). The phase purity for the bulk materials of UPC-32 has been confirmed by the PXRD analysis (Fig. S1 in Supporting information). Thermogravimetric analysis (TGA) curve display approximately 30.1% weight loss in the temperature range from 40 °C to 308 °C (Fig. S2 in Supporting information), which are assigned to the release of two coordinated DMF molecules, two coordinated water molecules and one guest DMF molecule for UPC-32. Above 308 °C, the frameworks start to decompose.

The establishment of permanent porosity is one of the important goals in MOF research [24]. The as-synthesized crystals of UPC-32 were solvent exchanged three times with dry acetone, then the samples were degassed at 298 K for one night and at 353 K for 12 h with the outgas rate of 5 mmHg/min to produce the activated samples for the gas adsorption measurements. The active phases are highly crystalline, and remain almost the same as its as-synthesized phase (Fig. S1 in Supporting information). The  $\text{N}_2$  gas adsorption curve of UPC-32 at 77 K is recorded to check their porosity (Fig. 2). UPC-32 show type I  $\text{N}_2$  adsorption isotherms, suggesting permanent micro-porosity. The  $\text{N}_2$  gas uptake is 332 cm<sup>3</sup>/g at 77 K and 1 bar. The pore volume calculated is 0.41 cm<sup>3</sup>/g, which is smaller than the theoretical pore volume (0.50 cm<sup>3</sup>/g) owing to the structural contractions during the activation. The Brunauer-Emmett-Teller (BET) surface area calculated is 1345 cm<sup>2</sup>/g.

The  $\text{H}_2$  adsorption experiments show 118.2 cm<sup>3</sup>/g (1.05 wt%) and 83.6 cm<sup>3</sup>/g (0.75 wt%) uptakes around 1 bar at 77 and 87 K, respectively (Fig. 3a). The moderate  $\text{H}_2$  uptake for UPC-32 at 77 K is comparable to PCN-131 (0.84 wt%) and PCN-19 (0.95 wt%) [25,26]. Moreover, the adsorption heat of  $\text{H}_2$  calculated by the

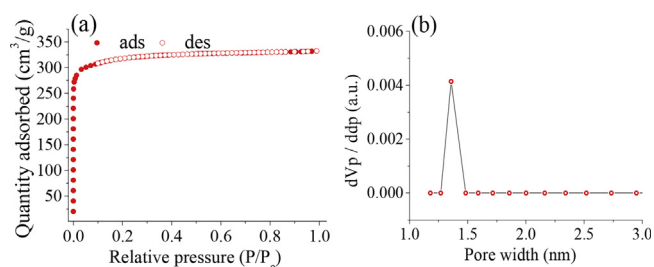


Fig. 2. (a) The  $\text{N}_2$  sorption isotherms at 77 K, and (b) pore size distribution for UPC-32.

Clausius-Clapeyron equation is 8.5 kJ/mol at zero coverage and decreases slowly with increasing  $\text{H}_2$  loading (Fig. 3c). These values are higher than those of some famous MOF materials, such as HKUST-1 (6.6 kJ/mol) [27], MOF-5 (5.2 kJ/mol) [28], and NOTT-122 (6.0 kJ/mol) [29].

Since  $\text{CO}_2$  is a dominant component of greenhouse gas and a main contaminant of natural gas, it is meaningful to investigate the capacity for  $\text{CO}_2$  and selectivity of  $\text{CO}_2/\text{CH}_4$ . Low pressure  $\text{CO}_2$  adsorption isotherms are also measured at 273 K and 298 K (Fig. 3b). The amount of  $\text{CO}_2$  uptake for UPC-32 is 102.8 cm<sup>3</sup>/g (20.2 wt%) and 65.7 cm<sup>3</sup>/g (12.9 wt%) under 1 bar at 273 K and 298 K, respectively. Compared with the enormous MOFs, the amount of MOFs which exhibit over 20.0 wt %  $\text{CO}_2$  uptakes at 273 K and 1 bar is relatively small [30,31]. The  $Q_{\text{st}}$  for  $\text{CO}_2$  in UPC-32 is 46 kJ/mol calculated by the Clausius-Clapeyron equation (Fig. 3c and Table S1 in Supporting information). The predicted  $\text{CO}_2/\text{CH}_4$  selectivity (for equimolar gas-phase mixtures) by IAST at 298 K under 1 bar are calculated as 6.6 for UPC-32 (Fig. 3d and Table S2 in Supporting information). It should be noted that these values are lower than Mg-MOF-74 ( $\text{CO}_2/\text{CH}_4$ : 105), UTSA-16 ( $\text{CO}_2/\text{CH}_4$ : 30) [32] and SIFSIX-3-Zn ( $\text{CO}_2/\text{CH}_4$ : 231) [33], but still comparable to ZIF-79 ( $\text{CO}_2/\text{CH}_4$ : 5.4) [34], SIFSIX-2-Cu ( $\text{CO}_2/\text{CH}_4$ : 5.3) and PCN-88 ( $\text{CO}_2/\text{CH}_4$ : 5.3) [35], making it qualified considerable candidate for  $\text{CO}_2$  capture and separation from natural gas.

Considering the small pore size and intrinsic permanent porosity of UPC-32, we have investigated its potential application for light hydrocarbons adsorption and separation. Although many MOFs with excellent gas sorption capacity (such as  $\text{H}_2$ ,  $\text{CO}_2$  and  $\text{CH}_4$ ) have been reported, only a few MOFs show high adsorption capacity and selectivity toward light hydrocarbons. To examine the adsorption and separation of light hydrocarbons for UPC-32, single component gas adsorption isotherms of UPC-32 for various light hydrocarbons ( $\text{CH}_4$ ,  $\text{C}_2\text{H}_2$ ,  $\text{C}_2\text{H}_4$ ,  $\text{C}_2\text{H}_6$ ,  $\text{C}_3\text{H}_6$ , and  $\text{C}_3\text{H}_8$ ) are performed at both 273 K and 298 K. As expected, UPC-32 can uptake a high amount of  $\text{C}_3\text{H}_8$  (104.3 cm<sup>3</sup>/g),  $\text{C}_3\text{H}_6$  (110.1 cm<sup>3</sup>/g),  $\text{C}_2\text{H}_6$  (80.2 cm<sup>3</sup>/g),  $\text{C}_2\text{H}_4$  (74.1 cm<sup>3</sup>/g), and  $\text{C}_2\text{H}_2$  (85.0 cm<sup>3</sup>/g), but a relatively lower amount of  $\text{CH}_4$  (31.3 cm<sup>3</sup>/g) at 273 K and 1 bar (Fig. 4a). It should be noted that the sorption capacity of UPC-32 for  $\text{C}_3\text{H}_8$  (84.9 cm<sup>3</sup>/g),  $\text{C}_3\text{H}_6$  (98.2 cm<sup>3</sup>/g),  $\text{C}_2\text{H}_6$  (66.1 cm<sup>3</sup>/g),  $\text{C}_2\text{H}_4$  (57.2 cm<sup>3</sup>/g),  $\text{C}_2\text{H}_2$  (60.6 cm<sup>3</sup>/g), and  $\text{CH}_4$  (18.9 cm<sup>3</sup>/g) at 298 K and 1 bar are higher than those of UTSA-35a and UTSA-36a (Fig. 4b and Fig. S5 in Supporting information) [36,37]. The magnitude of the adsorption heats reveals the affinity of the pore surface toward adsorbents, which plays a significant part in determining the adsorptive selectivity [38]. To evaluate the affinity of such light hydrocarbons in UPC-32, the adsorption heats are calculated by the Clausius-Clapeyron equation. The  $Q_{\text{st}}$ s for  $\text{CH}_4$ ,  $\text{C}_2\text{H}_2$ ,  $\text{C}_2\text{H}_4$ ,  $\text{C}_2\text{H}_6$ ,  $\text{C}_3\text{H}_6$ , and  $\text{C}_3\text{H}_8$  are 13.1, 6.3, 8.3, 6.6, 13.3, and 13.8 kJ/mol at zero coverage, respectively (Fig. 4c).

The C3 light hydrocarbons with higher adsorption heat may provide stronger affinity with a skeleton, which results in these

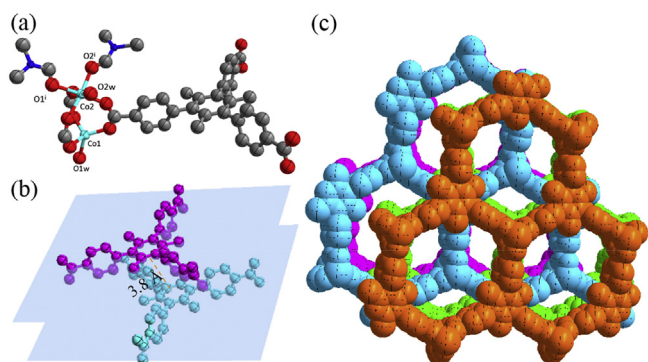
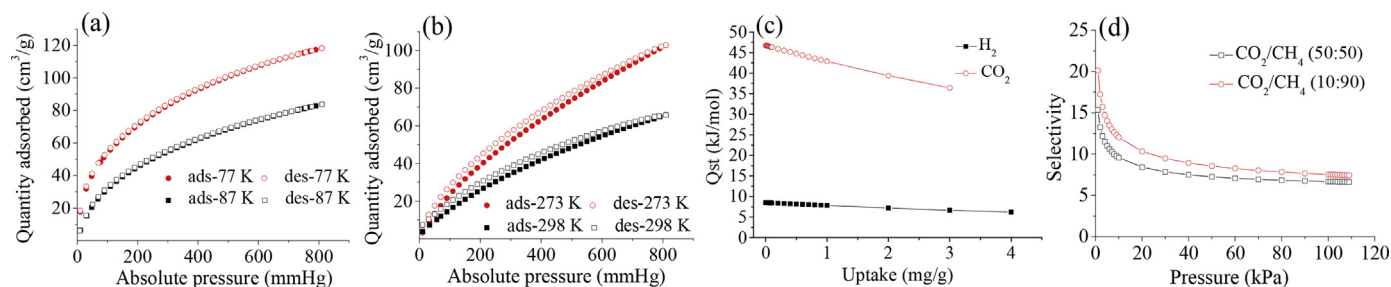
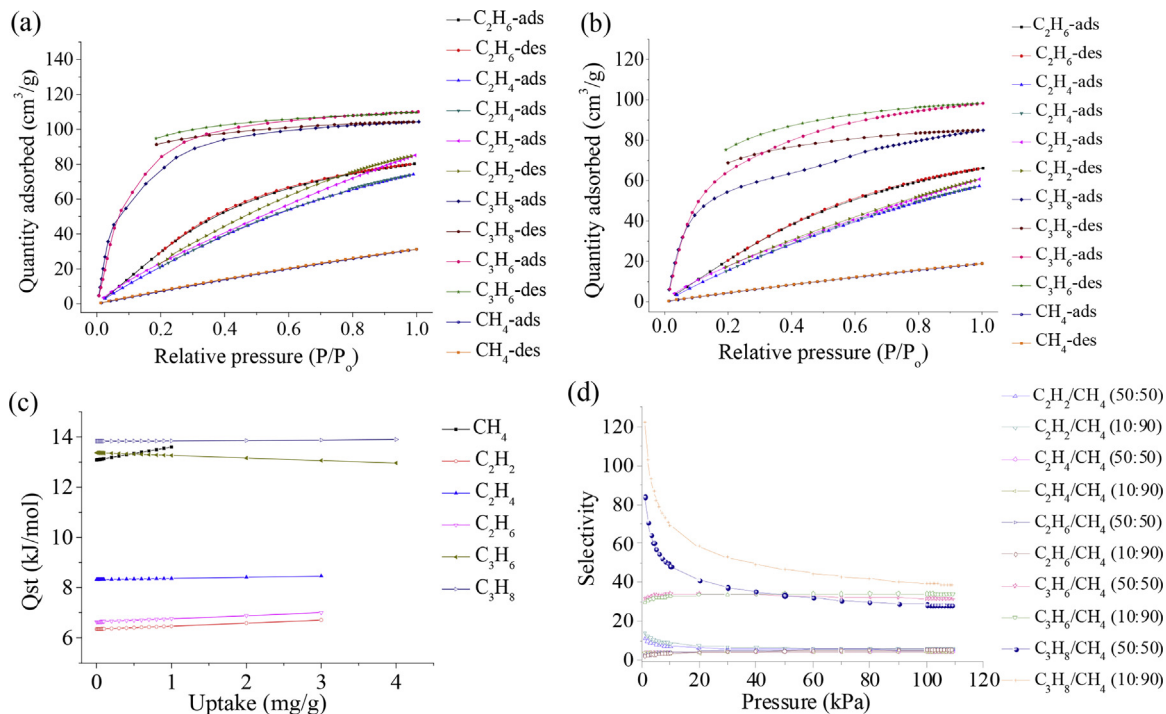


Fig. 1. Structure of UPC-32: (a) The  $\text{TMTA}^{3-}$  ligand and Co cluster. (b) The distance between the two layers. (c) ABAB stacking of 2D layers.



**Fig. 3.** (a) The H<sub>2</sub> sorption isotherms at 77 K and 87 K; (b) The CO<sub>2</sub> sorption isotherms at 273 K and 298 K; (c) The adsorption heat ( $Q_{st}$ ) of H<sub>2</sub> and CO<sub>2</sub> for UPC-32, calculated by the Clausius-Clapeyron equation; (d) The CO<sub>2</sub>/CH<sub>4</sub> (v/v: 50/50 and 10/90, respectively) selectivity at 298 K, calculated by the IAST method.



**Fig. 4.** The CH<sub>4</sub>, C<sub>2</sub>H<sub>6</sub>, C<sub>2</sub>H<sub>4</sub>, C<sub>2</sub>H<sub>2</sub>, C<sub>3</sub>H<sub>8</sub> and C<sub>3</sub>H<sub>6</sub> adsorption isotherms at 273 K (a) and 298 K (b) for UPC-32; (c) The  $Q_{st}$  for CH<sub>4</sub>, C<sub>2</sub>H<sub>6</sub>, C<sub>2</sub>H<sub>4</sub>, C<sub>2</sub>H<sub>2</sub>, C<sub>3</sub>H<sub>8</sub> and C<sub>3</sub>H<sub>6</sub>; (d) The C<sub>2</sub>H<sub>6</sub>/CH<sub>4</sub>, C<sub>2</sub>H<sub>4</sub>/CH<sub>4</sub>, C<sub>2</sub>H<sub>2</sub>/CH<sub>4</sub>, C<sub>3</sub>H<sub>8</sub>/CH<sub>4</sub> and C<sub>3</sub>H<sub>6</sub>/CH<sub>4</sub> (v/v: 50/50 and 10/90, respectively) selectivity at 298 K, calculated by the IAST method.

gases being preferentially adsorbed on a skeleton of UPC-32. Thus, it may have high selectivity of C3 light hydrocarbons with respect to CH<sub>4</sub>. Therefore, the potential for separation of CH<sub>4</sub> from C3 light hydrocarbons has been appraised by ideal solution adsorbed theory (IAST) for binary quimolar components (Fig. 4d). At 1 bar and 298 K, the selectivities of C<sub>3</sub>H<sub>8</sub> and C<sub>3</sub>H<sub>6</sub> with respect to CH<sub>4</sub> are 28.0 and 31.4, which are higher than C<sub>2</sub>H<sub>2</sub>, C<sub>2</sub>H<sub>4</sub>, and C<sub>2</sub>H<sub>6</sub> with respect to CH<sub>4</sub> for 5.2, 4.3, and 5.8, respectively. The results indicate that UPC-32 is a prospective adsorbent for effectively selective adsorptive separation of CH<sub>4</sub> from C3 hydrocarbons at room temperature. The high adsorption selectivity of C3/CH<sub>4</sub> could be attributed to the narrow distance between layers, which is match well with the kinetic diameters of C3 light hydrocarbons.

In summary, we have developed and characterized a new microporous 2D Co-MOF (UPC-32) based on a pre-designed functional ligand. UPC-32 exhibits high adsorption of H<sub>2</sub> and high adsorption heat of CO<sub>2</sub>, it has the right pore size to maximize interactions between gases and frameworks, so it exerts high separation selectivity for C3 light hydrocarbons with respect to CH<sub>4</sub>, as shown by single component gas adsorption and selectivity calculations. These results indicate that UPC-32 could be a

promising candidate for fuel gas purification and separation of light hydrocarbons in the future.

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## Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at <https://doi.org/10.1016/j.ccllet.2017.11.020>.

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