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Nitro Decorated Microporous Covalent Organic Framework (TpPa-NO₂) for Selective Separation of C₂H₄ from C₂H₂/C₂H₄/CO₂ Mixture and CO₂ Capture

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ABSTRACT: A nitro-decorated microporous covalent-organic framework (COF), TpPa-NO₂, has been synthesized in gram-scale with one-pot reaction. It can effectively selective separate C₂H₄ from C₂H₂/C₂H₄/CO₂ mixture and capture CO₂ from CO₂/N₂ based on IAST calculations and transient breakthrough experiments. Theoretical calculations demonstrated that the hydrogen atom of imine bonds, carbonyl oxygen and nitro groups show high affinity toward C₂H₂ and CO₂ and play vital roles in efficient separation.

In 2020, global energy demand is estimated to be 557.10 exajoules (EJ), and fossil fuel accounts for 83.152%.¹ The resultant global warming has led to sea level rise and more extreme weather events like heatwaves and heavy rainfalls.² Due to the COVID-19 pandemic and clean energy growing, the energy consumptions and carbon emissions have fallen by 4.5% and 6%, respectively. However, the rate of carbon emission decline is barely similar to what the world needs to do to meet the aims of the Paris Agreement. Two important methods for emission reductions are reducing energy consumption and carbon capture & sequestration. Statistics show that industrial separation processes, such as light hydrocarbon separation and carbon capture from flue gas, accounted for 10-15% of the world's total energy consumption in 2016.³ Light hydrocarbon (LH) separation rely on the high energy demand process, distillation. Carbon capture using aqueous amine solutions strategy suffers not only high energy consumption, but also solvent distillation, water use, and equipment corrosion. Hence developing alternative LHs separation and efficient CO₂ capture techniques with low energy consumption and mild operating conditions would decrease the global energy demand and carbon emission.

Because its energy efficiency, high working capacity and high selectivity, physical sorption separation utilizing porous solids materials offers a promising alternative to the established energy intensive processes for LHs separation/purification and CO₂

capture. One of the most pressing issues for this technology is lack of low-cost and efficient physisorbents. The similar kinetic diameters and boiling points of C₂H₂, C₂H₄ and CO₂, make the design of robust physisorbents with simultaneously high uptake and selectivity extremely challenging. Porous materials such as porous silica⁴, activated carbons,⁵ zeolites,⁶ porous aromatic frameworks (PAFs),⁷ metal-organic frameworks(MOFs),⁸⁻¹¹ metal-organic cages (MOCs),¹²⁻¹⁴ and covalent organic frameworks(COFs)¹⁵⁻¹⁹ etc. are potential candidates with excellent adsorption capacity and selectivity. Among them, COFs are a new type of crystalline framework materials built with strong covalent bonds between light elements (C, B, N, O, Si, B, etc.) using the design principles of 'reticular chemistry'.²⁰ They exhibited low density, remarkably high thermal stability, high surface area, readily adjustable pore size, and outstanding structural flexibility.²¹ COFs have been widely investigated for applications in gas storage and separation,^{4, 17, 22, 23} catalysis,^{24, 25} clean energy conversion,²⁶ sensing,^{27, 28} and energy storage.^{29, 30} As porous framework materials, COFs have been regarded as an ideal physisorbents for LH separation and^{17, 19, 31} CO₂ capture separation in the past decade.^{16, 23, 32, 33} For example, Zhu and co-workers synthesized a COF (PAF-110) which exhibit selective adsorption of C₂H₂ over C₂H₄ as its carbonyl oxygen provided higher affinity for acetylene than ethylene.¹⁹ Jiang's group introduced carboxylate groups onto the pore surface of COFs to increase the affinity towards CO₂.¹⁶ Lai and co-workers synthesized CAA-COF-1 and CAA-COF-2. The former one contain electron-rich chlorine atoms and enhanced CO₂ adsorption capacity (by almost 28–44%) compared to the latter one.²³ Han and co-workers synthesized a perfluorinated covalent triazinebased framework (FCTF-1). Compared with the unfluorinated CTF-1, FCTF-1 displayed significantly reduced pore size, from microporous to ultra-microporous (0.5 nm), and resulted in improved CO₂/N₂ selectivity in a mixed gas breakthrough test.³² These researches show that introducing functional groups to the pore walls of COFs can increase the host-guest interactions to improve the adsorptive selectivity and facilitate the separation processes. Banerjee group reported

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Electronic supplementary information (ESI) available: Experimental details, materials synthesis, additional structural figures, crystallographic refinement details, PXRD, TGA curves, gas adsorption results. See DOI: 10.1039/x0xx00000x

TpPa-NO₂, which possesses imine backbone and -NO₂ group functionalized pore walls. TpPa-NO₂ was first synthesized by means of a twin screw extruder,¹¹ which is difficult to obtain for common lab and unsuitable for small amount synthesis. In 2020, Luo and co-workers synthesized TpPa-NO₂ by solvothermal method at milligram-scale using solvothermal method.³⁴ However, the synthesis scale was too small to produce samples for transient column breakthrough separation.

Herein, we report the gram-scale synthesis of TpPa-NO₂ using one-pot reflux method, and investigation of its performance in C₂H₄ selective separation from C₂H₂/C₂H₄/CO₂ mixture as well as CO₂ capture from flue gas. Structure simulation and powder X-ray diffraction (PXRD) pattern demonstrate that the scale up synthesis produced crystalline and pure TpPa-NO₂ in AA packing mode. Single component gas adsorption experiments and theoretical calculations certify that introduction of -NO₂ can improve the adsorption selectivity and enhance the adsorbate-adsorbent interaction. The transient breakthrough experiments prove that TpPa-NO₂ can separate C₂H₄ from binary (C₂H₂/C₂H₄) and ternary (C₂H₂/C₂H₄/CO₂) mixtures while it can also effectively capture CO₂ from CO₂/N₂ (15:85) mixture simulating flue gas. The excellent C₂H₄ and CO₂ adsorption selectivity is attributed to the -NO₂ groups and the hydrogen atoms of the imine bonds based on theoretical calculations. Moreover, the outstanding durability, easy regeneration, and excellent thermal and chemical stability of TpPa-NO₂ suggest that it can be applied to industrial C₂H₄ purification process and CO₂/N₂ separation.

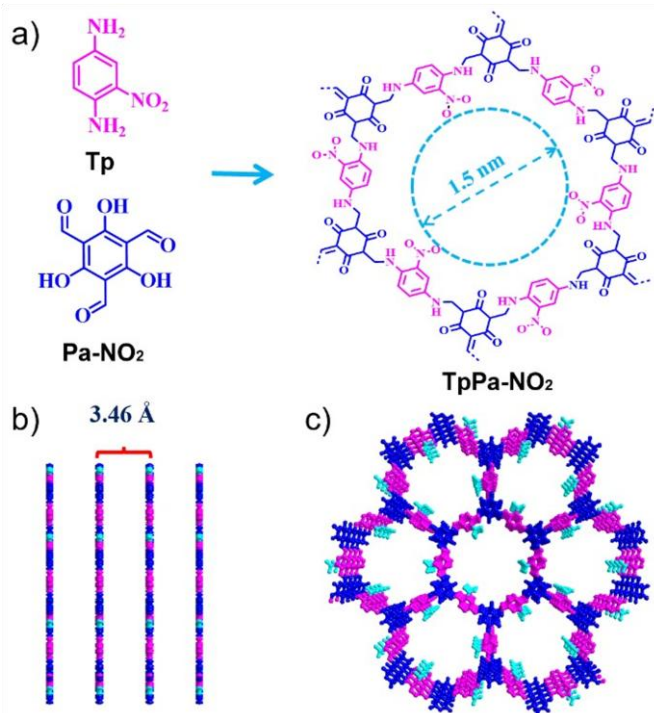


Figure 1. a) Synthetic scheme of TpPa-NO₂. Graphic view of the slipped AA stacking structure of TpPa-NO₂ (b, along a/b axis; c, along c axis) Hydrogen atoms are omitted for clarity. Color scheme: pink, Pa-NO₂; blue, Tp; turquoise, -NO₂ groups.

TpPa-NO₂ was synthesized using the one-pot reflux method by solvothermal reaction modified from the literature.³⁴ 2,4,6-triformylphloroglucinol (Tp) and 2-nitor-1,4-phenylenediamine (Pa-NO₂) were dissolved in 1,4-dioxane and 1,3,5-trimethylbenzene

with p-toluenesulfonic acid monohydrate as the template. The reaction mixture was refluxed under nitrogen protection at 120 °C for 72 hours (Figure 1a). The condensation produced scarlet powder in 87% isolation yield. The infrared (IR) spectrum of TpPa-NO₂ shows that the stretching vibration bands of C=N and C=O at 1617 and 1640 cm⁻¹,^{35, 36} respectively, proving the success of the aldoamine condensation reaction (Figure S1 in the Supporting Information). The morphology of TpPa-NO₂ was examined by scanning electron microscopy (SEM), which shows a large quantity of uniform nanofibers, thus implying its phase purity (Figure S2b). The structural simulation predicted that TpPa-NO₂ in AA packing mode has one-dimensional channels along the c-axis with a diameter of 15 Å while the layers stack with an interlayer distance of 3.46 Å (Figure 1). The framework of TpPa-NO₂ is stable in ambient air and retains its crystallinity after heating to 300 °C in air (Figure S3-4). It is also stable after soaking in a variety of organic solvents, concentrated acid (12 M HCl & 2 M H₂SO₄), or hot water (85 °C) for one week, while it can stay in basic aqueous solutions (2 M NaOH) for 48 hours (Figure S5). The specific surface area was determined to be 398 m² g⁻¹ from N₂ sorption isotherms at 77K, lower than the literature reported value (850 m² g⁻¹). Pore size distribution calculated by using the quenched solid density functional theory (QSDFT) method resulted in a pore size of 1.5 nm (Figure S6 & S7), matching the simulated AA-packing mode structure model.

Intrigued by its high crystallinity, microporous channels, excellent chemical and thermal stability, imine porous backbone, polar functional groups (-NO₂),³⁷ we expect that TpPa-NO₂ has high adsorption affinity for CO₂ and can make gas molecules diffuse differently, suitable for separation industrial gases with similar kinetic diameter such as CO₂ (3.3 Å), N₂ (3.64 Å), C₂H₂ (3.3 Å), C₂H₄ (4.16 Å).

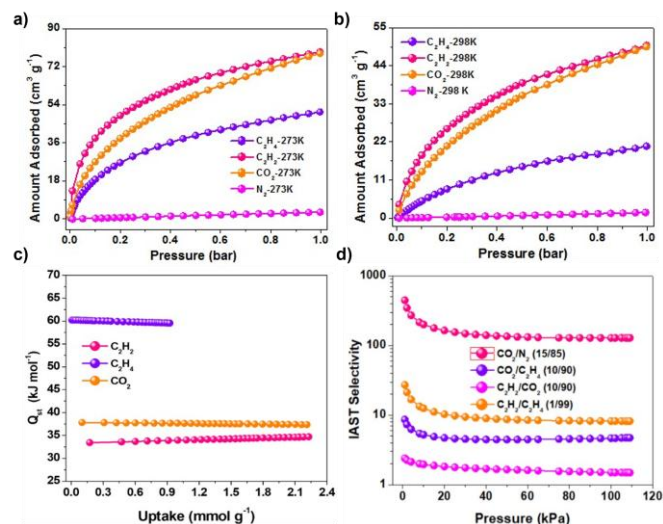


Figure 2. C₂H₂, C₂H₄, CO₂ and N₂ adsorption isotherms of TpPa-NO₂ at a) 273 and b) 298 K, respectively. c) Isothermic heat of adsorption (Q_{st}) of TpPa-NO₂ as a function of surface coverage. d) IAST selectivity of TpPa-NO₂ for different binary gas mixture at 298 K.

Single component adsorption isotherms of C₂H₂, C₂H₄, CO₂ and N₂ for TpPa-NO₂ were collected at 273 and 298 K, respectively (Figure 2a & b). The isothermic heat of adsorption (Q_{st}) plots were calculated using the Clausius-Clapeyron equation (Figure 2c Figure S8-10).^{38, 39} As expected, TpPa-NO₂ shows excellent CO₂ uptake under 273 K (78.3cm³ g⁻¹) and 298 K (49.52cm³ g⁻¹) at 1bar, similar

to those of C_2H_2 ($79.03 \text{ cm}^3 \text{ g}^{-1}$ at 273 K and $49.92 \text{ cm}^3 \text{ g}^{-1}$ at 298 K), respectively. The Q_{st} for CO_2 was $37.83 \text{ kJ mol}^{-1}$ at the zero coverage, and higher than that of C_2H_2 ($33.42 \text{ kJ mol}^{-1}$). For most porous materials the adsorption capacity and Q_{st} of C_2H_2 is always higher than those of CO_2 .^{10, 37} Due to the larger kinetic diameter (4.16 Å) and smaller quadrupole moment ($1.50 \times 10^{26} \text{ esu cm}^2$) of C_2H_4 compared with of CO_2 (3.3 Å and $4.30 \times 10^{26} \text{ esu cm}^2$),⁴⁰ the adsorption capacity of C_2H_4 ($50.57 \text{ cm}^3 \text{ g}^{-1}$ at 273 K and $20.74 \text{ cm}^3 \text{ g}^{-1}$ at 298 K) is much lower than that of CO_2 and C_2H_2 . N_2 keeps its consistent behavior of not being adsorbed under 273/298 K. To our surprise, the adsorption capacity of C_2H_4 seems to be more susceptible to temperature changes than C_2H_2 and CO_2 gases.

Increasing temperature can obviously reduce the uptake of C_2H_4 (Figure 2a & b), which implied that C_2H_4 may have the highest Q_{st} . Subsequent Q_{st} calculation ($60.22 \text{ kJ mol}^{-1}$) certified this speculation (Figure 2c). The adsorption selectivities of TpPa- NO_2 for binary mixtures of CO_2/N_2 (15:85 v/v), C_2H_2/C_2H_4 (1:99 v/v), C_2H_2/CO_2 (10:90 v/v), and CO_2/C_2H_4 (10:90 v/v) were calculated using the ideal adsorption solution theory (IAST) based on the single component adsorption data (Figure 2d).^{38, 39} From 0.01 to 1 bar at 298 K, TpPa- NO_2 presents excellent CO_2/N_2 selectivity (477.33-128.92), while its selectivity of C_2H_2/C_2H_4 (1:99 v/v), C_2H_2/CO_2 (10:90 v/v) and CO_2/C_2H_4 (10:90 v/v) were 27.15-8.22, 2.40-1.48 and 8.64-4.71, respectively.

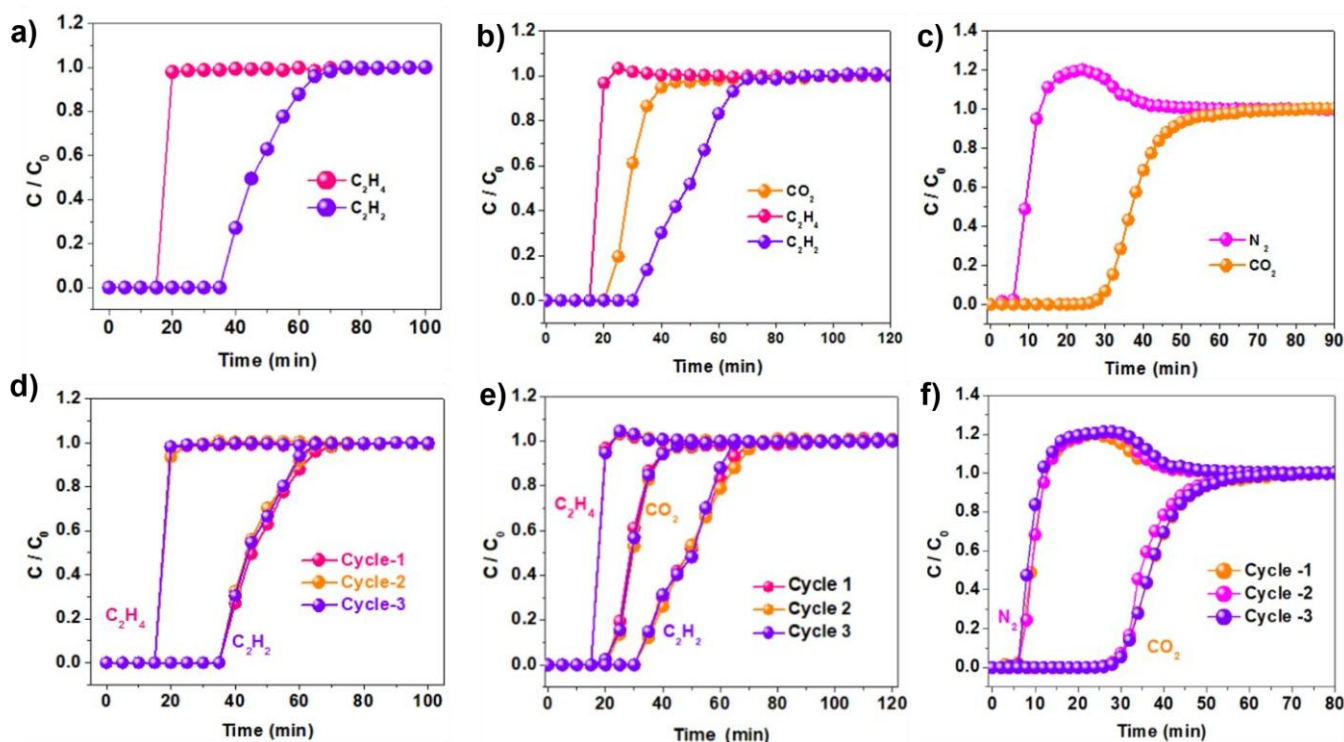


Figure 3. Transient breakthrough curves and cycling tests of TpPa- NO_2 for a/d) C_2H_2/C_2H_4 (1:99 v/v), b/e) $C_2H_2/C_2H_4/CO_2$ (1/90/9, v/v/v), and c/f) CO_2/N_2 (15/85, v/v) mixture at 298K and 1bar, respectively. (C/C_0 , outlet concentration/feed concentration)

To further confirm the separation performance of TpPa- NO_2 , transient breakthrough experiments for C_2H_2/C_2H_4 (1:99 v/v), $C_2H_2/C_2H_4/CO_2$ (1/90/9, v/v/v) and CO_2/N_2 (15/85, v/v) mixtures were carried out at 298 K (Figure 3). Firstly, binary gas mixtures C_2H_2/C_2H_4 (1:99 v/v) was tested. C_2H_4 broke through the packed bed and yielded purity C_2H_4 (>99.9%) at first only need 20 minutes after the start, whereas after 42 minutes C_2H_2 slowly eluted and reached equilibrium at 70 minutes (Figure 3a). For further examining the separation ability of TpPa- NO_2 in more complex gas mixture systems, ternary mixture was used. The outlet time of C_2H_4 (15 min) and C_2H_2 (30 min) maintained while CO_2 eluted as the second gas at 25 min (Figure 3b). The capture capacity of C_2H_4 , CO_2 and C_2H_2 were 1.39, 0.231 and 0.0416 mmol, respectively, Hence C_2H_4 can still be separated from the ternary mixtures. For simulated carbon capture in flue gas (CO_2/N_2 :15/85, v/v), N_2 eluted first as a result of weak adsorbate-adsorbent interaction; whereas CO_2 was captured in the column until its saturated sorption and then breakthrough, with the CO_2 capture capacity of 0.51 mol g^{-1} (Figure 3c). Furthermore, cycling breakthrough experiments on TpPa- NO_2 were carried out under the same conditions. The breakthrough curves for C_2H_2/C_2H_4 ,

$C_2H_2/C_2H_4/CO_2$, and CO_2/N_2 mixtures in 3 cycles tests were consistent, manifesting that TpPa- NO_2 has good stability and regenerability.

To understand the separation mechanism, first-principle density functional theory (DFT) calculations were employed to investigate the gas adsorption sites and host-guest interactions of TpPa- NO_2 . Generally, the gases are held by the weak van der Waals (vdW) force (the C...H, O...H and C...O interactions) at the channel aperture. As shown in Figure 4, the strongest interaction of C_2H_2 comes from the O...H interaction (2.27 Å) between the C_2H_2 and the carbonyl oxygen of TpPa- NO_2 (Figure 4a). For C_2H_4 , except the C...H interactions (2.83 Å) between the C_2H_4 and imine backbone, the O...H interactions (3.22 Å & 3.56 Å) provided by - NO_2 and carbonyl oxygen of TpPa- NO_2 also contribute to holding the C_2H_4 (Figure 4b). CO_2 was held by the O...H interactions (2.79~3.1 Å) between the H atoms of imine backbone and CO_2 , and the C...O interaction (2.97 Å) between the carbonyl oxygen of TpPa- NO_2 and carbon atoms of CO_2 (Figure 4c).

Conclusions

In summary, we have successfully achieved the gram-level synthesis of COF (TpPa-NO₂) in one-step synthesis using the one-pot reflux method. Its C₂H₄ selective separation and CO₂ capture performance has been systematically studied. TpPa-NO₂ exhibit high IAST selectivity of 8.22 and 128.92 for C₂H₂/C₂H₄ (1:99) and CO₂/N₂ (15:85) at 298 K and 1 bar, respectively. Breakthrough experiments further confirmed that TpPa-NO₂ can separated C₂H₄ from C₂H₂/C₂H₄ and C₂H₂/C₂H₄/CO₂ mixtures while it can also effectively capture CO₂ from CO₂/N₂. DFT calculations revealed that the H of imine group, carbonyl oxygen and -NO₂ imposed the strong influence for CO₂, C₂H₂ and C₂H₄. The outstanding durability, easy regeneration, excellent thermal and chemical stability of TpPa-NO₂ offer a promising application potential for high value-added light hydrocarbons separation & purification and carbon capture.

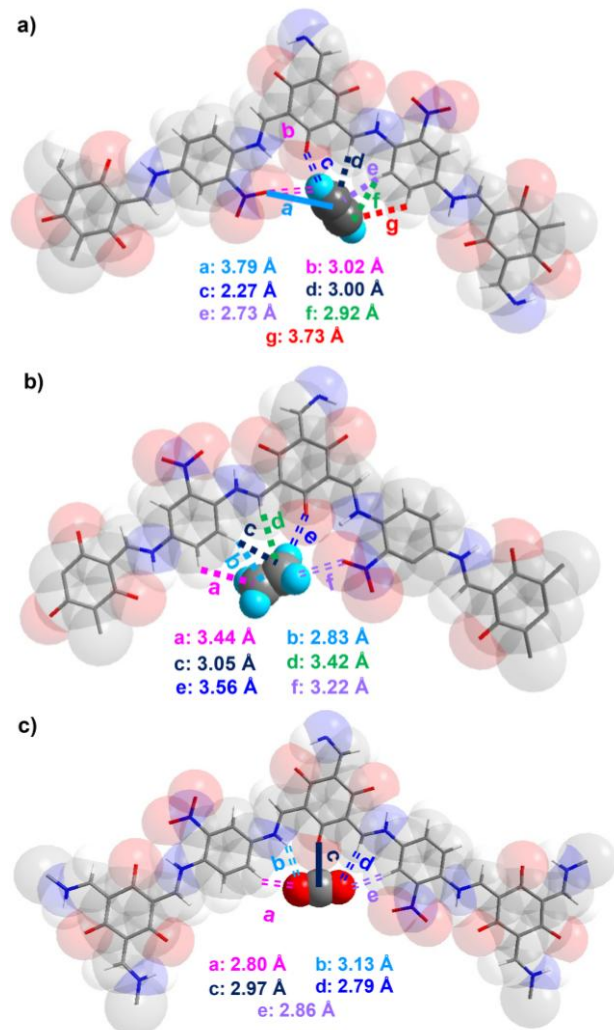


Figure 4. DFT-calculated TpPa-NO₂ binding configurations: a) C₂H₂, b) C₂H₄, c) CO₂. Double dotted-lines, H...O; dotted-lines, C...H interaction; solid line, C...O interaction.

Author Contributions

The manuscript was written through contributions of all authors.

Conflicts of interest

The authors declare no competing financial interest.

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