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Ionic Covalent Organic Frameworks: Design of a Charged Interface Aligned on 1D Channel Walls and Its Unusual Electrostatic Functions

Ning Huang⁺, Ping Wang⁺, Matthew A. Addicoat, Thomas Heine, and Donglin Jiang^{*}

Abstract: Covalent organic frameworks (COFs) have emerged as a tailor-made platform for designing layered two-dimensional polymers. However, most of them are obtained as neutral porous materials. Here, we report the construction of ionic crystalline porous COFs with positively charged walls that enable the creation of well aligned yet spatially confined ionic interface. The unconventional reversed AA-stacking mode alternately orientates the cationic centers to both sides of the walls; the ionic interface endows COFs with unusual electrostatic functions. Because all of the walls are decorated with electric dipoles, the uptake of CO_2 is enhanced by three fold compared to the neutral analog. By virtue of sufficient open space between cations, the ionic interface exhibits exceptional accessibility, efficiency, and selectivity in ion exchange to trap anionic pollutants. These findings suggest that construction of the ionic interface of COFs offers a new way to structural and functional designs.

Covalent organic frameworks (COFs) are crystalline porous polymers with designable primary- and high-order structures.^[1,2] Recent progress in the chemistry of COFs had significant effect on enhancing their structural diversity and complexity.^[3] However, most of COFs still rely on neutral skeletons. Integration of ionic modules into the frameworks has a high probability of generating ionic interfaces to control the interactions with molecules and ions that would induce novel functions distinct from those of neutral skeletons.^[1,4] In this sense, the wall surface of the one-dimensional (1D) channels of COFs is of great interest because it constitutes the interface between molecules and COFs.

To implant ionic interface on the pore walls requires the use of ionic building blocks, which however, prevent π - π stacks owing to strong charge repulsion and usually result in low crystallinity and porosity.^[4] Therefore, there is a contra-

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diction between the charged layers and the crystallinity and porosity of COFs. Here, we report a general strategy for designing ionic COFs with high crystallinity and porosity, through the combination of ionic linkers and neutral knots for the construction of ionic interfaces that are well aligned yet spatially confined on the pore walls. The COFs assume an unconventional reverse AA-stacking mode in which the cationic benzimidazolium linkers^[4d] are alternately orientated and aligned on both sides of the pore walls. We highlight that such an alternate alignment exerts profound effects on the ionic interface and triggers unusual electrostatic functions of COFs. By virtue of reduced charge repulsion between layers, the ionic COFs possess high crystallinity and porosity. With electric dipoles on both sides of the walls, the COF enhances the CO_2 adsorption by even three fold. The alternately aligned layers offer sufficient open space around cationic centers and the ionic interfaces exhibit exceptional accessibility, efficiency, and selectivity in trapping and removing anionic pollutants.

We synthesized 4,4',4",4"'-(pyrene-1,3,6,8-tetrayl) tetraaniline (PyTTA) as neutral knot and 5,6-bis(4-formylbenzyl)-1,3-dimethyl-benzimidazolium bromide (BFBIm, see the Supporting Information) as cationic linker for the construction of imine-linked positively charged COFs in which the benzimidazolium cationic sites were exposed to the wall surface (Figure 1a, PyTTA-BFBIm-iCOF). The reaction was carried out in a mixture of o-dichlorobenzene and n-butanol in the presence of acetic acid catalyst under solvothermal conditions at 120°C for 3 days (see the Supporting Information). PvTTA-BFBIm-iCOF was obtained as vellow powder at an isolated yield of 82%. A variety of methods were employed for structural characterizations. The infrared spectrum revealed a stretching vibration band at 1624 cm⁻¹ that was assigned to the C=N bonds (see Figure S1 in the Supporting Information). Elemental analysis corroborates well with the theoretical values of infinite 2D sheet (Table S1). Field emission scanning electronic microscopy revealed that PyTTA-BFBIm-iCOF adopts micrometer-scale belt morphology (Figure S2). High-resolution transmission electron microscopy enables the direct visualization of pores (Figure S3). PyTTA-BFBIm-iCOF is thermally stable up to 450 °C under nitrogen (Figure S4).

PyTTA-BFBIm-iCOF is a highly crystalline polymer with strong signals in powder X-ray diffraction (PXRD). PyTTA-BFBIm-iCOF exhibited diffraction peaks at 3.48°, 5.14°, 7.06°, 10.60°, 14.35°, and 23.88°, which were assignable to the (110), (020), (220), (040), (060), and (001) facets, respectively (Figure 1 f, red curve). The presence of (001) facets indicates that PyTTA-BFBIm-iCOF has periodic orders in all three dimensions. The Pawley-refined pattern (black curve) with R_w

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Figure 1. a) Schematic representation of the synthesis of PyTTA-BFBIm-iCOF. b) Top and c) side views of the reversed slipped AA-stacking mode of PyTTA-BFBIm-iCOF (gray: C, orange: Br⁻, blue: N, white: H); d) Top and e) side views of slipped AA-stacking mode. f) PXRD patterns of PyTTA-BFBIm-iCOF of experimentally observed (red), Pawley refined (black), their difference (green), simulated curves for reversed slipped AA-stacking mode (blue), slipped AA-stacking mode (purple), and staggered AB-stacking mode (orange). Units cells of g) reversed slipped AA, h) slipped AA, and i) staggered AB-stacking modes.

and R_{WP} values of 5.57% and 7.23%, respectively, confirmed the correctness of peak assignments as evident by negligible deviation from the observed PXRD patterns (green curve).

Structural simulations were conducted by using the selfconsistent charge density functional tight binding (SCC-DFTB) method with starting structures created by AuTo-GraFS and pre-optimized using a topology-preserving force field (see the Supporting Information). We optimized the monolayer and then various layered framework structures with different stacking modes. In PyTTA-BFBIm-iCOF, the reversed slipped AA-stacking mode is the most stable structure in the total energy among the various stacking modes, including slipped AA- and staggered AB-stacking modes (Figures S5 and S6, Tables S2 and S3). The reversed slipped AA-stacking mode avoids the direct overlap of benzimidazolium cationic centers on each other between neighboring layers and reduces the repulsion between layers (Figure 1 b,c). The reversed slipped AA-stacking mode yielded a PXRD pattern (Figure 1 f, blue curve) that was in good agreement with the experimentally observed PXRD profile. PyTTA-BFBIm-iCOF assumes the space group of *P*mm2 with a = 24.46 Å, b = 28.61 Å, c = 7.44 Å, and $a = \beta =$ $\gamma = 90^{\circ}$ (for atomic coordinates see Table S4). On the other hand, the staggered AB-stacking mode could not reproduce the PXRD pattern (Figure 1 f, orange curve). In contrast to the slipped AA-stacking mode that gives rise to an orientation of the benzimidazolium cationic centers to only one side of the pore walls (Figure 1 d,e), the reverse slipped AA-stacking mode distributes the benzimidazolium cationic centers on both sides of the pore walls (Figure 1 b,c).

The high crystallinity of PyTTA-BFBIm-iCOF intrigues us to evaluate its porosity. PyTTA-BFBIm-iCOF exhibited reversible type IV sorption curves (Figure 2a), which are



Figure 2. a) Nitrogen sorption curves of PyTTA-BFBIm-iCOF at 77 K (filled circles for adsorption, open circles for desorption). b) The pore size distribution and pore volume of PyTTA-BFBIm-iCOF.

characteristics of mesoporous materials. The Brunauer— Emmett–Teller (BET) surface area was calculated to be as high as $1532 \text{ m}^2 \text{g}^{-1}$. To the best of our knowledge, this surface area is the highest among ionic COFs reported to date.^[4] We evaluated the pore size and its distribution by using the nonlocal density functional theory method (Figure 2b). PyTTA-BFBIm-iCOF exhibited only one type of pore at 2.3 nm, which is close to its theoretical value. The total pore volume was estimated to be $0.7 \text{ cm}^3 \text{g}^{-1}$.

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Chemical stability is important for ionic materials. With the high crystallinity and porosity in mind, we treated the PyTTA-BFBIm-iCOF samples in different solvents, including N,N-dimethylformamide (DMF), boiling water, and aqueous HCl (3M) and NaOH (3M) solutions at 25 °C for 24 h. The COF samples were collected, washed with water and THF, dried under vacuum at 120 °C for 12 h, and subjected to PXRD (Figure 3a) and N₂ sorption isotherm measurements



Figure 3. a) PXRD patterns and b) N_2 sorption curves of PyTTA-BFBImiCOF upon 24-hour treatment in different solvents (color in b is the same as that in a).

(Figure 3b). Surprisingly, all these samples exhibited intense PXRD patterns without changes in the peak position and intensity, indicating that the high crystallinity is retained under these harsh conditions (Figure 3a). Moreover, the BET surface areas were also retained under these conditions (Figure 3b).

To date, a number of porous materials have been developed for CO_2 capture and separation.^[5] These investigations revealed that the nature of chemical functionality along with the textural properties of polymers is an important parameter to tailor CO_2 affinity of these materials. In the case of COFs, introduction of polar groups such as amine and carboxylic acid units has shown positive effects on CO_2 adsorption.^[5] Although ionic surface is promising to enhance the CO_2 affinity through dipole–quadruple interactions, however, to what extent an ionic interface of COFs will affect the CO_2 adsorption remains unprecedented.

PyTTA-BFBIm-iCOF uptakes CO_2 with capacities of 93 mgg⁻¹ at 298 K and 177 mgg⁻¹ at 273 K and 1 bar (Figure 4a). The ionic COF is superior to COF-5 (5.9 wt%, at



Figure 4. a) CO_2 (circles) and N_2 (triangles) adsorptions at 273 K (red) and 298 K (blue). b) Q_{st} value for CO_2 adsorption at low coverage.

273 K and 1 bar, the same at below),^[6a] **COF-102** (1.21 wt %),^[6a] (7.6 wt %),^[6a] ILCOF-COF-103 (6.0 wt %),^[6b] (9.2 wt %),^[6c] 1 TDCOF-5 TpPa-1 (15.6 wt %),^[6d] and even carboxylic acid functionalized $[HO_2C]_{100\%}$ -H₂P-COF (17.4 wt %).^[5b] As a control, we synthesized PyTTA-TPhA-COF that is a neutral analog to PyTTA-BFBIm-iCOF without ionic interface on the pore walls (Figures S7 and S8). PyTTA-TPhA-COF exhibits a high BET surface area of $1754 \text{ m}^2 \text{g}^{-1}$ (Figure S9). However, PyTTA-TPhA-COF uptakes CO2 with capacities of only $65\ mg\,g^{-1}$ at 273 K and 36 $mg\,g^{-1}$ at 298 K and 1 bar (Figure S10). Therefore, the ionic interface in the COFs enhances the CO₂ adsorption by almost three fold. The $Q_{\rm st}$ value was calculated from the isotherms to be 30.2 kJ mol⁻¹, which is moderate in COFs (Figure 4b).

The alternate orientation of cationic centers to both sides of the walls leaves enough space between cationic sites. Such a spatial alignment greatly enhances the accessibility of the cationic sites by guest molecules. Herein, we developed the ionic interface to trap ionic pollutants through ion exchange, by dispersing insoluble PyTTA-BFBIm-iCOF powder (5 mg) to the ethanol/water solution (1/1 by vol., 15 mL) of anionic methyl orange (MO, 200 mg L^{-1}) at room temperature. The trapping process is easily monitored by electronic absorption spectroscopy (Figure 5a). Time-dependent spectral change shows that the absorption band of MO at 465 nm decreased with time. The PyTTA-BFBIm-iCOF can rapidly capture MO; after 10 h the residual MO concentration in the solution was smaller than 0.1 mg L^{-1} . This result suggests that over 99.9% of MO was removed by PyTTA-BFBIm-iCOF. The ion exchange between the anionic MO and the bromide anions occur on the wall surfaces of PyTTA-BFBIm-iCOF, leading to the trap of MO.

We investigated the removal capacity by keeping the amount COF and changing the initial concentration (Ci) of the MO solutions. As a result, the adsorption capacity versus concentration plot reveals a sharp increase at low concentration (0-30 mg L^{-1}) and saturated after 50 mg L^{-1} (Figure 5b). Surprisingly, the maximum removal capacity of PyTTA-BFBIm-iCOF was as high as 553 mg g^{-1} (see the Supporting Information). This maximum capacity is equivalent to 0.81 MO anion per benzimidazolium cation, suggesting the high accessibility of benzimidazolium cationic sites to the MO anions. To the best of our knowledge, the capacity of PyTTA-BFBIm-iCOF is the highest among adsorbent materials reported to date and is much higher than those of benchmark porous materials, for example, nanosize SiO₂- Al_2O_3 mixed oxides (381 mgg⁻¹),^[7a] nanostructured protoncontaining H- δ -MnO₂ nanoparticles (427 mg g⁻¹),^[7b] alkaliactivated multi-walled carbon nanotube CNTs-A (149 mg g^{-1}) ,^[7c] and NH₂-MIL-101(Al) (199 \text{ mg g}^{-1}).^[7d] The equilibrium adsorption isotherm can be perfectly fitted with Langmuir model to yield a high correlation coefficient ($R^2 >$ 0.999) (Figure 5c, see the Supporting Information). This linear curve reflects that trapping MO by PyTTA-BFBImiCOF is based on Langmuir adsorption model.

To investigate the kinetics of the trapping process by dispersing PyTTA-BFBIm-iCOF (5 mg) in the ethanol/water solution of MO (1/1 by volume, 30 mg L^{-1} , 50 mL), we

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Figure 5. a) Time-dependant electronic absorption spectral change (from top: 0 min, 1 min, 2 min, 3 min, 5 min, 10 min, 12 min, 15 min, 20 min, 30 min, 1 h, 2 h, 3 h, 5 h, 10 h) of an ethanol/water solution (1/1 by volume, 15 mL) of MO at initial concentration of 200 mg L⁻¹ upon addition of PyTTA-BFBIm-iCOF (5 mg). b) Adsorption isotherm of MO. c) Linear regression using Langmuir adsorption model. d) Effect of contact time on the MO adsorption at initial concentration of 30 mg L⁻¹. e) Pseudo-second-order kinetics of MO adsorption. f) Capture efficiency of MO at different pH values. g) Capture efficiency of different dyes. h) Cycle performance.

evaluated the contact time-dependent adsorption profiles. As shown in Figure 5 d, the adsorption was quick and completed within 100 min. By using the pseudo-second-order kinetics (Figure 5 e, correlation coefficient R = 0.998), the kinetic constant (k) was evaluated to be $5.32 \times 10^{-2} \text{ gmg}^{-1}\text{min}^{-1}$. Notably, this k value is larger than those of other porous materials, including activated carbon ($2.17 \times 10^{-4} \text{ gmg}^{-1}\text{min}^{-1}$),^[7e] MOF-235 ($7.67 \times 10^{-4} \text{ gmg}^{-1}\text{min}^{-1}$),^[7f] and PED-MIL-101 ($2.75 \times 10^{-3} \text{ gmg}^{-1}\text{min}^{-1}$).^[7e]

By virtue of skeleton stability in acidic and basic solutions, we further investigated the removal of MO under different pH conditions. PyTTA-BFBIm-iCOF exhibited the removal efficiency of 99% at the pH values of 1 and 4 (Figure 5 f). To our surprise, the efficiencies were as high as 95% and 91% at pH values of 10 and 13, respectively. The slight decrement under basic condition originates from the enhanced OH⁻ anions that compete with the MO anions. High performance over a wide pH range is important for applications. In this sense, PyTTA-BFBIm-iCOF is much superior to porous silicas that deteriorate under harsh conditions.

Interestingly, the ionic surface can also trap dianions efficiently. For example, when the dianionic indigo carmine acid blue 74 (IC-74) was used, PyTTA-BFBIm-iCOF can remove IC-74 with a high efficiency of 98.5% (Figure 5d and Figure S11). By contrast, PyTTA-TPhA-COF cannot trap neutral and cationic molecules (Figure 5d). Indeed, neutral nile red (NR) or coumarin 6 (C6), and cationic rhodamine B (RB) exhibited very low trapping levels of only 1.6%, 4.3% and 2.4%, respectively (Figure 5g, Figures S12–S14). These contrastive results indicate that the ionic interfaces recognize the opposite charges and the electrostatic interactions on the wall surfaces enable a distinct discrimination between anions, cations, and neutral species.

After trapping anionic pollutants, the ionic interface can be simply regenerated by treating the COF samples with aqueous NaBr solution (1.0 M) at room temperature for 48 h. The MO anions can be desorbed nearly quantitatively. This high regeneration yield of COFs makes the repetitive use possible. It is worthy to mention that PyTTA-BFBIm-iCOF retained 92% of the original capacity even after six cycles (Figure 5 h). All the above results indicate that the ionic COF sets a new benchmark that combines capacity, efficiency, durability, selectivity, and reusability in removing anionic pollutants.

In summary, we have shown that COFs offer a wonderful extended scaffold for creating ionic interfaces that are well aligned and spatially confined on the pore walls of onedimensional channels. We elucidated two fundamental aspects of ionic interface, that is, electric dipole and electrostatic interactions through the findings of their unusual functions in CO_2 adsorption and ion exchange to remove anionic pollutants. It becomes clear that ionic interfaces can induce new functions or boost physiochemical properties. With the availability of various types of charged species as well as the possibility to design COFs, the creation of ionic interfaces provides a new chemical platform for structural and functional designs.

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Conflict of interest

The authors declare no conflict of interest.

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Communications

Carbon Capture

N. Huang, P. Wang, M. A. Addicoat, T. Heine, D. Jiang* _____ IIII--IIII

Ionic Covalent Organic Frameworks: Design of a Charged Interface Aligned on 1D Channel Walls and Its Unusual Electrostatic Functions



A scaffold for ionic interfaces: Covalent organic frameworks were synthesized. They bear ionic interfaces that are well aligned and spatially confined on the one-dimensional channel walls. The ionic interfaces exert profound effects on the frameworks and trigger unusual electrostatic functions, such as the adsorption of CO_2 and the selective removal of anionic pollutants.

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