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# Exfoliation of Covalent Organic Frameworks into Few-Layer Redox-Active Nanosheets as Cathode Materials for Lithium-Ion Batteries

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ABSTRACT: Covalent organic frameworks (COFs) have attracted growing interest by virtue of their structural diversity and tunability. Herein, we present a novel approach for the development of organic rechargeable battery cathodes in which three distinct redox-active COFs were successfully prepared and delaminated into 2D few-layer nanosheets. Compared with the pristine COFs, the exfoliated COFs with shorter Li<sup>+</sup> diffusion pathways allow a significant higher utilization efficiency of redox sites and faster kinetics for lithium storage. Unlike diffusion-controlled manners in the bulk COFs, the redox reactions in ECOFs are mainly dominated by charge transfer process. The capacity and potential are further engineered by reticular design of COFs without altering the underlying the topology. Specifically, DAAQ-ECOF exhibits excellent rechargeability (98% capacity retention after 1800 cycles) and fast charge-discharge ability (74% retention at 500 mA g<sup>-1</sup> as compared to at 20 mA g<sup>-1</sup>). DABQ-EOF shows a specific capacity of 210 mA h g-1 and a voltage plateau of 2.8 V.

Organic materials with redox-active sites are promising candidates for the next generation of rechargeable lithiumion (Li<sup>+</sup>) batteries, since they are heavy-metal free and can be prepared from widespread resources and feature high rate performance stemmed from simple redox reaction.<sup>[1]</sup> Great efforts have been devoted to linking redox monomers to polymers to enhance their cycling performance<sup>[1,2]</sup>, however, in most cases, capacity fading at high rates are inevitable due to the partial dissolving or side reactions. Covalent-organic frameworks (COFs),<sup>[3]</sup> an emerging class of crystalline and porous polymeric materials, can serve as potential alternatives for battery applications. Unlike traditional polymers, COFs allow precise integration of the redox-active building blocks into two- or three-dimensional (2D or 3D) polymeric frameworks with long-range ordered skeletons and nanopores. The diversity of building units together with atomically controllable arrangements make it easy to tailor the redox properties of COFs. Moreover, COFs provide confined

open channels in nanoscale for ionic transportation to reach the active sites, and also charges are allowed to rapidly move in the pre-organized pathways including both eclipsed stacked  $\pi$  columns and  $\pi$ -conjugated intralayer skeletons.<sup>[4]</sup>

Several pioneering works have been reported where 2D COFs can either be utilized as active materials<sup>[5]</sup> or the host for active materials<sup>[6]</sup> in rechargeable Li<sup>+</sup> battery. However, since 2D extended polygons tend to closely pack in an eclipsed fashion in COFs due to the strong  $\pi$ - $\pi$  interactions, the interior active sites deeply buried inside the 1D channels are difficult to reach due to the long ion transportation path, especially at a high charge/discharge rate. The sluggish Li<sup>+</sup> migration in 1D channels will inevitably lead to the insufficient utilization of the redox-active sites and thus lowering their capacity and rate performance.

Herein, we propose a strategy to accelerate the diffusioncontrolled redox reactions in COFs by the delamination of layered redox active COFs into 2D few-layer nanosheets (Scheme 1). The eclipsed stacking of the planar sheets in 2D COF through non-covalent van der Waals force between interlayers gives opportunities to delaminate them into fewlayer structures. The exfoliated COFs structurally resemble graphenes with the addition of adjustable atomic structures and periodical channels. Compared with COFs featured multiple-stacking structures, the exfoliated COFs (ECOFs)<sup>[7]</sup> with limited few layers are able to shorten the ion/electron migration length and facilitate the ionic/electronic diffusion. A large portion of Li storage sites are exposed on the surface or near-surface region of the ECOFs, which is favorable for proceeding fast charge/discharge cycles and fully utilizing the redox-active sites.

As a proof of concept, we first prepared a redox-active ECOF (DAAQ-ECOF) with approximately 5 nm thickness by delaminating anthraquinone based COF (DAAQ-TFP-COF)<sup>[8]</sup> via ball milling. Evaluating as a charge storage material for the cathode in rechargeable Li batteries, DAAQ-ECOF shows a high reversible capacity of over 145 mA h g<sup>-1</sup> at 20 mA g<sup>-1</sup>, which delivers 96% of its theoretical value. At a high current density of 500 mA g<sup>-1</sup>, DAAQ-ECOF displays a

reversible capacity over 107 mA h g<sup>-1</sup> and 98% capacity retention after 1800 cycles of deep discharge-charge. The capacity of DAAQ-ECOF is doubled at 500 mA g<sup>-1</sup> and tripled at 3000 mA g<sup>-1</sup> compared with the pristine COF material without exfoliation. To further demonstrate the tunability of COF materials and the applicability of our exfoliation strategy, we further designed and prepared two new COF materials, DABQ-TFP-COF and TEMPO-COF (Scheme 1a, Figure S2 and S3), with higher theoretical capacity and voltage plateau. To the best of our knowledge, the overall performance of these ECOFs rivals the state-of-art quinone-based Li<sup>+</sup> cathodes.

Scheme 1. (a) Chemical structures of DAAQ-TFP-COF, DABQ-TFP-COF and TEMPO-COF. (b) Schematic illustration for the exfoliation of 2D redox-active COFs into exfoliated COFs as cathodes for Lithium-ion battery.



DAAQ-ECOF was prepared via a facile and scalable mechanical milling method. Micron-sized DAAQ-TFP-COF was put into a milling pot and then vibratory ball milled at room temperature without additional exfoliating agents. Different vibration frequency and time were tuned to optimize the thickness and battery performance of exfoliated COF (details are provided in the ESI, Figure S4 and S5). In order to illustrate the morphology changes after the mechanical exfoliation, field-emission scanning electron microscopy (FE-SEM), transmission electron microscopy (TEM) and atomic force microscopy (AFM) were performed. Compared

with the DAAQ-TFP-COF precursors, DAAQ-ECOFs exhibit sheet-like morphology with smaller size and thinner thickness (Figure 1, S6 and S7). TEM images indicate that DAAQ-ECOFs possess ultrathin, transparent and slightly wrinkled layer structure, whereas the pristine DAAQ-TFP-COF shows a bulky and opaque morphology. The thickness of DAAQ-ECOFs prepared under a vibration frequency of 50 Hz for 0.5 h are ranging from 3 to 5 nm. Considering that the distance between adjacent layers is about 3.4 Å, it suggests that DAAQ-ECOFs consist 10-15 atomic layers (Figure 1). These results together with the Tyndall effect for DAAQ-ECOF (Figure S8) confirmed DAAQ-TFP-COF has been successfully exfoliated into very thin 2D layers.



**Figure 1.** TEM photo, AFM topographical image and height profile of DAAQ-ECOF.

The chemical composition and structure of DAAQ-ECOF was determined by the Fourier transform infrared attenuated total reflection (FTIR-ATR), <sup>13</sup>C cross-polarization magic angle spinning (CP-MAS), solid-state NMR spectroscopy and elemental analysis (EA). The strong absorptions at 1250 and 1550 cm<sup>-1</sup> in the FTIR-ATR spectrum of DAAQ-ECOF are assigned to C-N and C=C stretching vibration, respectively, indicating the few-layered COF still appears in the keto form rather than the enol form (Figure S9a).<sup>[3c,9]</sup> In the <sup>13</sup>C NMR spectrum of DAAQ-ECOF, the signals at 146 ppm are corresponding to the enamine carbon (=CNH) (Figure S9b). The consistence of DAAQ-ECOF before and after exfoliation in the EA (Table S1), FT-IR and <sup>13</sup>C NMR results demonstrate that the chemical composition as well as the bonding mode remains intact after the mechanical ball milling. These results indicate that the ball-milling process prefers to break apart the  $\pi$ - $\pi$  interactions between the layers rather than destroy the chemical structure of the polymeric layers reinforced by irreversible β-ketoenamine-based covalent bond together with H-bond.

In order to further characterize the crystalline structure of the exfoliated DAAQ-ECOF, powder X-ray diffraction (PXRD) measurements were carried out. DAAQ-ECOF exhibits an intense peak at 3.5°, revealing that DAAQ-ECOF retains the periodic and extended hexagonal polygons in the planar sheets after exfoliation (Figure S9c). The BET surface area of DAAQ-ECOF calculated from its N<sub>2</sub> sorption isotherm at 77 K is 216 m<sup>2</sup> g<sup>-1</sup>, it is attributed to the random displacement of the 2D layers to some extent (Figure S10).

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The electrochemical performance of DAAQ-ECOF was evaluated by combining it with super-P and PVDF (6:3:1) as cathode and then assembling into coin cell versus lithium anode (details are given in the ESI).

Firstly, we conducted cyclic voltammogram (CV) analyses. As shown in Figure S11, DAAQ-ECOF exhibits highly reversible lithiation/delithiation reactions, with a pair of symmetric redox peaks at 2.34 and 2.48 V,<sup>[10]</sup> providing a practical voltage range comparable with many emerging cathode materials, such as sulfur (~2.1 V), Prussian blue analogues (2~3 V), etc. The reduction potential of DAAQ-ECOF is 0.4 V higher than that of the monomer (DAAQ), originating from the extended conjugation within the polymeric layer.<sup>[11]</sup> Actually, there are two steps (AQ/AQ<sup>--</sup> and AQ<sup>--</sup>/AQ<sup>2-</sup>, Figure S11) involved in the electrochemical redox reaction and even more steps in a conjugated structure; however, the small gap between these redox potentials makes it difficult to distinguish these individual steps.<sup>[12]</sup>

The cathodic and anodic peak current of DAAQ-ECOF grows linearly with an increase in the sweep rate (Figure S12a-b and Figure S13-14), revealing that the redox reaction is a surface and/or near-surface process and its kinetics is mainly determined by charge transfer rather than mass transfer.<sup>[2a, 2b]</sup> On the contrary, for DAAQ-TFP-COF with multiple layers, there is a linear relation between its peak current and the square root of scan rate, demonstrating that the redox-reaction kinetic is dominated by Li-ion diffusion in the bulk materials. Therefore, DAAQ-ECOF with limited few layers are endowed with the ability for ultrafast discharge/charge process and fully utilization of its active sites.

These coin cells were proceeded charge/discharge cycles between 1.5 and 4 V at a rate of 20 mA  $g^{-1}$  (Figure S12d). The plateaus showing in the galvanostatic charge-discharge profiles agree well with their CV curves, corresponding to the reversible oxidization/reduction of the carbonyl groups (Figure 12c and S16). Remarkably, the DAAQ-ECOF shows a capacity of 145 mA h g-1 after 70 cycles, achieving 96% of its theoretical capacity. As comparison, DAAQ-TFP-COF only delivers 73% (110 mA h g<sup>-1</sup>) of its theoretical value, resulting from that the redox sites deep inside the 1D channels are difficult to participate the electrochemical process. DAAQ-based cathode exhibits an initial capacity of 197 mA h g<sup>-1</sup> and rapidly fades to 75 mA h g<sup>-1</sup> within 20 cycles, owing to the dissolution problems (Figure S17). In contrast, both DAAQ-TFP-COF and DAAQ-ECOF are chemically and thermally robust (Figure S18) and are completely insoluble in the electrolyte, ensuring their excellent cycle performance.

We further evaluated the rate performance of these materials (Figure 2a and 2c). Relative to the maximum capacity of 145 mA h  $g^{-1}$  at a rate of 20 mA  $g^{-1}$ , the capacity retention for DAAQ-ECOF is 93, 89, 86, 74, 68, 60 and 50% at increased current rates of 50, 100, 200, 500, 1000, 2000 and 3000 mA  $g^{-1}$ , respectively. As shown in Figure 4c, the capacity retentions for DAAQ-TFP-COF are much lower at higher current

rates, due to the ion-diffusion controlled electrochemical process. It's noteworthy that the capacity of DAAQ-ECOF retains 107 and 76 mA h g<sup>-1</sup> at 500 and 3000 mA g<sup>-1</sup>, respectively, which almost doubles and triples the values for DAAQ-TFP-COF at the same current rate. Remarkably, DAAQ-ECOF shows a lithium ion diffusion coefficient  $(D_{\text{Li}}^+)$  of  $6.94 \times 10^{-11}$  cm<sup>2</sup> s<sup>-1</sup>, which is almost three times that of DAAQ-TFP-COF ( $2.48 \times 10^{-11}$  cm<sup>2</sup> s<sup>-1</sup>) and is much higher than those of many commercial electrode materials (Table S3). Rapid Li<sup>+</sup> transport along shortened diffusion paths is crucial for achieving good rate performance.



**Figure 2.** (a) Rate cyclability. (b) Capacity retention of DAAQ-ECOF and DAAQ-TFP-COF compare with their capacities measured at 20 mA  $g^{-1}$ . (c) Long-term cyclability and Coulombic efficiencies measured at a current density 500 mA  $g^{-1}$ .

The cycling properties of these coin cells were studied by repeated charge/discharge experiments at a current density of 500 mA g<sup>-1</sup> (Figure 2b and Figure S20). The reversible capacity of the DAAQ-ECOF was retained at 104 mA h g<sup>-1</sup> after 1800 cycles, with a mild activation process during the initial several cycles possibly due to the penetration of electrolyte. The Coulombic efficiency is close to 99% during the whole test. This remarkable cycle performance is benefited from the stable framework reinforced by covalent and H bonding and high redox reversibility of quinone groups.

The potential and redox behavior of COF can be easily tuned. To increase the theoretical gravimetric capacity, we used 2,5-diamino 1,4-benzoquinone (DABQ), a smaller structural unit, to substitute 2,6-diamino-anthraquinone (DAAQ) on the edges of DAAQ-TFP-COF. Direct preparation of DABQ-TFP-COF could only afford amorphous solid. Therefore, we first condensed 2,5-diamino 1,4dihydroxybenzene dihydrochloride (DABH) and 1,3,5tris(4-formylphenyl)benzene (TFP) to obtained DABH-TFP-COF and then subjected it to sequential oxidization to yield DABQ-TFP-COF (Figure S2). The fully oxidization of hydroxyl to carbonyl groups was confirmed by IR and EA analysis results (Figure S23 and Table S1). Furthermore, in order to enhance the voltage plateau, TEMPO-COF was prepared by post-synthetic modification (Figure S3). The intense PXRD diffraction peaks for DABQ-TFP-COF and

TEMPO and their exfoliated products (Figure S27) agree well with the simulated diffractions of their packing structures constructed using eclipsed stacking modes (Figure 3a-d, S25-26). It should be noted that the discharge voltage of DABQ-ECOF is 0.5 V higher than that of DAAQ-ECOF due to electronic effect. TEMPO-ECOF displays two voltage plateaus at 3.6 and 2.7 V attributed to the reduction of oxo-ammonium cation to nitroxide radical and then to aminoxy anion (Figure 3e). The capacities of DABQ-ECOF and TEMPO-ECOF are 35% and 53% higher than their pristine materials at 20 mA g<sup>-1</sup>, respectively (Figure 3f). Especially for DABQ-ECOF, its capacity reaches 210 mA h g<sup>-1</sup>. The improved capacity and discharge voltage will lead to higher energy density for practical applications.

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**Figure 3.** (a-d) Experimental and simulated PXRD patterns of DABQ-TFP-COF (a) and TEMPO-COF (b) and their unit-cell structures (c-d). (e) Discharge curves at 20 mA  $g^{-1}$ . (f) Capacities of pristine and exfoliated COFs at 20 mA  $g^{-1}$ .

In summary, we have successfully exfoliated 2D covalent organic frameworks into few-layered polymeric sheets rich in fully accessible and reversible redox-active sites. The Li<sup>+</sup> ion transportation path is significantly shortened in the ECOFs, and consequently, the ion-diffusion controlled electrochemical process in the pristine COF-based cathodes was turned to charge-transfer dominated process. DAAQ-ECOF delivers as high as 96% of its theoretical capacity at 20 mA g<sup>-1</sup>, and exhibits a capacity of 104 mA h g<sup>-1</sup> at 500 mA g<sup>-1</sup> after 1800 cycles without any apparent fading. The battery performance of DAAQ-ECOF, including capacity, cycling stability and rate behavior, is significantly improved compared with its monomer and pristine COF counterparts. Through molecular design, we can increase the discharge voltage to 3.6 V and improve the capacity to 210 mA h g<sup>-1</sup>. The atomically controllable and highly crystalline features of COFs enable fine tuning of capacity and potential and provide a platform for better understanding of diffusion mechanisms and electrochemical process of active sites. The present exfoliation strategy paves ways to achieve the next generation of highperformance and sustainable energy-storage devices.

# **Supporting Information**. The Supporting Information is available free of charge on the ACS Publications website. Experimental details and data (PDF).

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#### Notes

The authors declare no competing financial interests.

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