Electrochemical CO₂ Capture by a Quinone-based Covalent Organic Framework

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Abstract

Electrochemical CO₂ capture is an emerging technology that promises to be more energy efficient than traditional thermal- or pressure-swing processes. Herein the first evidence of electrochemical CO₂ capture using a covalent organic framework (COF) is presented. We hypothesized that the assembly of anthraquinone units into a well-defined porous framework electrode would lead to enhanced electrochemical CO₂ capture compared to previous approaches that grafted anthraquinones on carbon supports and suffered from low CO₂ capacities and stabilities. To test this, an anthraquinone-based COF is employed, and it is found that the quinones are electrochemical CO₂ uptake capacity > 2.6 mmol g^{-1}_{COF} , reaching half of the theoretical CO₂ capacity of the material, and surpassing the capacities of anthraquinone-functionalized carbons. The stability and CO₂ uptake rate issues encountered with ionic liquid system are also addressed by using aqueous electrolytes where we attained stable carbon capture for 500 cycles with 99.6% coulombic efficiency and an electrical energy consumption of 31 kJ per mol of CO₂. The use of covalent organic framework electrochemical CO₂ capture.

1. Introduction

Energy efficient CO_2 capture is vital for urgent climate change mitigation. As a promising alternative to traditional CO_2 capture methods, electrochemical CO_2 capture (eCC) employing switchable redox active carriers is gaining significant momentum.^{1–5} The research in this area is progressing in several directions including: (i) the development of stable capture systems with higher CO_2 uptake capacities and (ii) the discovery of new solid sorbents that can operate at high current densities and are compatible with aqueous electrolytes.^{5–9} On the first strand, by leveraging the redox behaviour of active molecular disulfides, bipyridyls, thiolates, and extensively studied quinones, a diverse range of capture systems have been developed.¹⁰ Even the simplest representatives of the quinone family have an appealingly high theoretical capacity of two CO_2 equivalents per molecule, compared to conventional amines which require two equivalents of amine to capture one equivalent of CO_2 .^{5–7} Importantly, eCC offers the benefit of conducting CO_2 capture and release without the need for external heating or heat removal, and has thus shown promising energy efficiencies.^{11–14}

Efforts to integrate solid CO₂ sorbents in eCC have seen remarkable initial successes. A notable example is the immobilization of polyanthraquinones on carbon nanotubes (CNTs) to build a semi-solid faradaic swing system that demonstrated impressive cyclic performance in both ionic liquid and water-in-salt electrolytes.^{15,16} Building on this work, we successfully grafted anthraquinone units onto conductive carbon substrates, achieving a 50% charge utilization of the loaded redox moieties for CO₂ capture. However, this system suffered from rapid CO₂ capacity loss over time, and it was challenging to control and characterise the quinone loading.¹⁷ Furthermore, existing redox-based eCC systems often face challenges related to structural integrity, slow CO₂ uptake kinetics, and low mass loadings, imposing optimization and operational constraints.^{2,14,18,19}

Motivated by the progress above, we hypothesized that the CO_2 affinity seen in molecular quinones could be replicated in quinone-based covalent organic framework (COF) materials. The electrochemical reduction of these materials would generate phenoxide anions which can then bind electrophilic CO_2 , while the subsequent electrochemical oxidation would liberate CO_2 and regenerate the quinone (Fig. 1). We propose that COF-based redox systems have a high density of well-defined active capture sites (the anthraquinone units), while the COF porosity would enable the required rapid CO_2 and electrolyte transport. Additionally, the diversity of constituent redox moieties and tuneability of possible COF structures offer possibilities to fine-tune CO_2 uptake performance, while the use of earth-abundant C, N, H and O elements is beneficial from a sustainability standpoint.^{20–22}



Fig. 1. Proposed mechanism of electrochemical CO₂ capture by an anthraquinone-based covalent organic framework.

2. Results and Discussion

To test our hypothesis, we synthesized a well-known anthraquinone-based COF (AQCOF) and employed it in electrochemical CO_2 capture experiments.²³ Details on the synthesis, characterization of the AQCOF, electrode preparation, and device assembly can be found in Supplementary Information Sections (S1-4 and Fig. S1-6, ESI†). In brief, our 2-electrode battery-like cells are equipped with an AQCOF-carbon composite as the working electrode, an activated carbon counter electrode to balance the charge, 1-butyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide ionic liquid ([Bmim][TFSI], IL) as the electrolyte, and a pressure sensor to monitor electrochemical sorption within the sealed CO_2 environment of the cell.

When charging the cells to negative voltages (*i.e.* by inducing the reduction of the AQCOF material), a CO₂ pressure drop was observed, supporting the electrochemical capture of CO₂ by the anthraquinone units (Fig. 2a). Subsequent discharge back to 0 V led to a pressure increase ascribed to the electrochemical oxidation of the quinone units and CO₂ release. Importantly, the changes in CO₂ pressure closely followed the applied cell voltage over repeated cycles, demonstrating that the cycle is reversible (Fig. S7, ESI[†]). From the observed pressure change an initial uptake capacity of ~1.5 mmol g⁻¹ CO₂ uptake per gram of AQCOF was calculated.

An improvement in conductivity and charge capacity of the material was achieved by growing AQCOF on carbon nanotubes (CNTs).²⁴ To implement this in the device, the composition with 7% CNTs following AQCOF synthesis (denoted hereafter as AQCOF@CNTs) with a specific surface area (SSA) of 325 m² g⁻¹ was selected for its minimal surface area difference with the pristine AQCOF (SSA 310 m² g⁻¹). Electrodes fabricated with the AQCOF-carbon nanotube composite exhibited improved charge storage capacities compared to the AQCOF alone (Fig. 2b) and

attained 1.09 e⁻ (74 mAh g⁻¹ at 5 mA g⁻¹) and ~0.85 e⁻ (58 mAh g⁻¹ at 10 mA g⁻¹) charge capacity per anthraquinone unit, indicating that quinone units remote from the conductive carbon additives might be electrically isolated and therefore not accessed.



Fig. 2. Electrochemical CO₂ capture performance of AQCOF-based capture systems using [Bmim][TFSI] as the electrolyte. (a) A typical electrochemical galvanostatic discharge curve showing CO₂ pressure changes (smoothed pressure curve, moving average every 100 seconds, red line) at 5 mA g⁻¹ constant current in static mode and with a 5-min voltage hold. (b) Electrochemical discharge capacities under CO₂ at different current densities and (c) CO₂ uptake capacities at low current densities, with data shown for electrodes made from pristine AQCOF and electrodes made from AQCOF@CNTs materials. (d) Effect of breaking the working voltage window into different voltage regimes on electrochemical CO₂ uptake capacity using AQCOF@CNTs.

Further insights into electrochemical CO₂ uptake performance were obtained by varying the current density during charge-discharge cycles (Fig. 2c). In general, high CO₂ uptake is observed only at low current densities. Notably, at a current density of 5 mA g⁻¹ CO₂ uptake reached ~2.6 mmol g⁻¹ for AQCOF@CNTs. This CO₂ uptake capacity is the best among all known capacitive or redox electrochemical capture systems where direct comparison is possible.^{17,25} Fast charging shows a decreasing trend in both the CO₂ uptake and the charge capacity while right-shifted pressure curves portray a delayed CO₂ uptake in response to the applied cell voltage. (Fig. S8a-c, ESI[†]). Moreover, CO₂ molecules captured per electron stored decrease progressively and the electrons: CO₂ utilization ratio at 5, 10, and 30

mA g^{-1} for CO₂ capture falls from 0.87, 0.64, to 0.34 electrons, respectively (Fig. S9, ESI[†]), inferring that CO₂ transport in COF channels can limit the CO₂ uptake in the cell.

The evidence of redox CO_2 capture by the anthraquinone units was gathered with measurements in different voltage windows, along with cyclic voltammetry (CV) scans from 2-electrode cells. The CV of the AQCOF@CNTs under CO_2 exhibits two distinct features (Fig. S10, ESI⁺). Within the voltage range of 0 to -1 V, a narrow rectangular capacitive feature devoid of redox activity appears. Afterward, as the voltage was increased from -1 to -2.25 V a broad peak becomes evident, assigned to anthraquinone reduction. This information combined with the galvanostatic charge-discharge profile presented in Figure 2a was then used to assess how voltage windows affect CO₂ uptake performance. As shown in (Fig. 2d), when the cell was operated within the purely capacitive range of 0 to -1 V no pressure change occurred (Fig. S11a, ESI[†]), suggesting that quinone redox is essential for driving electrochemical CO_2 capture by the AQCOF. Instead, when limiting the voltage between -1 to -2.5 V, *i.e.* in a range of prominent redox activity, the CO₂ pressure oscillated periodically in response to the applied voltage (Fig. S11b, ESI⁺). Nonetheless, uptake remained lower than what was recorded across the full 0 to -2.5 V range. Low CO₂ uptake at higher charging rates also indicated the need for more oxidizing potentials and more time to fully regenerate quinones to drive the next capture cycle. Furthermore, applying positive polarization from 0 to 2.5 V (i.e., positively charging the COF electrode) raised the pressure inside the cell with abrupt pressure changes suggesting degradation of the material (Fig. S12, ESI⁺). Subsequent operation of the cell within the 0 to -2.5 V showed no pressure change, indicating that the cell was no longer functional. Our previous experiments with an electrochemical cell employing porous carbon electrodes as both electrodes and the same ionic liquid electrolyte exhibited only very minor electrochemical CO₂ uptake,¹⁷ supporting the idea that the CO₂ capture process by the AQCOF is predominantly driven by anthraquinone redox.

To assess our system under more realistic conditions, we evaluated uptake capabilities in different gas mixtures. For 100% CO₂ and a 15% CO₂ mixture with N₂, CO₂ uptake capacities were within error of each other (Fig. S13a-d, ESI[†]). To investigate whether the pressure changes were indeed due to CO₂ capture, measurements were conducted under 100% N₂ and showed only very small pressure changes (Fig. S14, ESI[†]). While small periodic pressure changes were observed under 100% N₂, they remained the same in response to different current densities and quantified values fell within the measurement uncertainty. We attribute these small changes to the movement of electrolyte ions within the COF channels, and possible electrolyte density changes due to electrochemical charging. Finally, for stability evaluation, in long cycling experiments, the system was charged with an industry-relevant constant current of 100 mA g⁻¹. A 20% loss in CO₂ capacity after 50 cycles and a 60% loss after 100 cycles was noted (Fig. S15, ESI[†]). After 300 cycles only 20% of the initial uptake capacity was retained. Further limiting the voltage range between -1 to -2 V— a region of predominant redox activity— to minimize parasitic cell degradation at large voltages, led to lower CO₂ uptake, but improved capacity retention (~90%) after 100 cycles (Fig. S16a-d, ESI[†]). These stability issues in ionic liquids motivated us to explore alternate electrolytes.

Having identified key limitations of the capture process, we replaced the IL electrolyte with aqueous 1 M Na₂SO₄ electrolyte. Recently, studies have begun to tackle the challenge of performing eCC with quinones in aqueous

media.^{7,26–28} Excitingly, a typical eCC cycle (Fig. 3a) recorded using AQCOF@CNTs at 100 mA g⁻¹ in an aqueous cell exhibited a 4-fold increase in electrochemical CO₂ uptake compared to IL cells (IL ~0.2 mmol g⁻¹ and aq. ~0.8 mmol g⁻¹), with the pressure curves closely following the applied voltage. The aqueous cell utilizes *ca*. 50% of the stored charge to capture CO₂ with an energy consumption of 37 kJ mol⁻¹_{co2} at this current density, which is substantially lower than the binding enthalpy of traditional thermal amines processes.^{29,30} Without a voltage hold under pure CO₂, the uptake showed a negligible change (Fig. S17a-c, ESI[†]), but the electrical energy consumption of the capture process was further lowered to 31 kJ mol⁻¹ (Table S1, ESI[†]).

Importantly, cyclic voltammetry studies confirmed the presence of quinone redox processes in the studied voltage range (Fig. S18, ESI[†]), and we further note that our observed electrochemical CO₂ uptake capacity of ~0.8 mmol g⁻¹ $_{AQCOF@CNTs}$ is much larger than that observed in recently reported capacitive CO₂ capture processes,^{17,27,31,32} suggesting that quinone redox drives the electrochemical CO₂ capture process under aqueous conditions. In our previous work of a cell containing YP80F activated carbon as both the working and counter electrodes with the same aqueous electrolyte, we observed only ~0.1 mmol g⁻¹ CO₂ adsorption under identical conditions.²⁵ Moreover, when we positively charged the cell (0 to 1 V, i.e. oxidation of the AQCOF@CNTs electrode) no CO₂ uptake was observed, further supporting that the reduction of quinones drives CO₂ capture (Fig. S19, ESI[†]).



Fig. 3. Electrochemical CO₂ capture performance of AQCOF@CNTs system in 1 M Na₂SO₄ as electrolyte. (a) CO₂ capture-release cycle recorded at high current density of 100 mA $g^{-1}_{AQCOF@CNTs}$ (shown data is a smoothed pressure curve with moving average every 100 seconds, red) and 5-min voltage hold. (b) Comparison of discharge capacity under CO₂ and (c) CO₂ adsorption rate at different current densities measured in [Bmim][TFSI] and 1 M Na₂SO₄ as electrolyte. (d) Long cyclic stability in 1 M Na₂SO₄ as electrolyte. In all calculation net AQCOF@CNTs mass in electrode was used for the normalisation.

Electrochemical CO₂ uptake capacities at different current densities displayed contrasting trends when compared to the IL system (Fig. 3b, and Fig. S20a-c, ESI[†]). While the IL system shows a gradual decrease in uptake capacity as

the applied current density is increased, the aqueous system shows an increase in CO_2 capacity at first, followed by a decrease at higher currents, suggesting a different mechanism of action in aqueous media. With fast charging rates both CO_2 uptake and charge utilization are enhanced till 100 mA g⁻¹, with decreases then observed at higher currents.

To gauge the potential of the material for practical application, uptake rates (i.e., CO_2 capture capacities per unit time) were also determined. Fig. 3c shows comparable adsorption rates for the IL and aqueous electrolyte systems until 70 mA g⁻¹; beyond this point, rates improve further for the aqueous system. Similarly, improvements in Coulombic efficiencies were observed for both the IL and aqueous systems, rising from initial values of 83% and 95% at 5 mA g⁻¹ to 98% and 99.6% at 100 mA g⁻¹, respectively, indicating higher reversibility of electrochemical reactions in the aqueous system (Fig. S21, ESI†). The aqueous system also exhibited ultra-high stability with no obvious charge storage or CO_2 capacity loss over 500 cycles, and with a 99.6% coulombic efficiency (Fig. 3d). While the cell is selective towards CO_2 in a mixture of 85%N₂: 15%CO₂, (Fig. S22, ESI†) in an optimized cycle, without voltage hold operating between 0 to -0.8 V and with fast charging at 100 mA g⁻¹ (this voltage is considered industrially relevant) exceptionally low energy consumption of 28 kJ mol⁻¹ and superior adsorption capacity and adsorption rates were realized (Fig. S23, ESI†).

To try to better understand the underlying processes involved in CO₂ capture in aqueous cells, we explored different voltage regimes. While working in a 0 to -0.5 V window, a CO₂ uptake of ~ 0.2 mmol g⁻¹ was noted and was relatively constant at different current densities (Fig. S24a, ESI[†]). This behaviour is similar to the trend seen in recent supercapacitive swing adsorption experiments.^{31,32} In contrast, CO₂ uptake in the -0.5 to -1 V window showed a progressive increase as a function of current till 100 mA g⁻¹ where it reaches 0.4 mmol g⁻¹ (Fig. S24b, ESI[†]). The mechanism of electrochemical CO₂ capture by this system requires further study, and we note the possibility of both "direct capture" of CO₂ by the reduced anthraquinone units, as well as the possibility for a "pH swing" driven mechanism of uptake in the aqueous electrolyte used here.⁷

3. Conclusions

In conclusion, we have demonstrated the first example of using a covalent organic framework for electrochemical CO_2 capture. Electrochemical reduction of the anthraquinone units in the COF led to electrochemical CO_2 capture in electrochemical cells with both ionic liquid and aqueous electrolytes. While cells with ionic liquid electrolytes showed poor kinetic behaviour and low stability, cells with aqueous electrolytes showed greatly improved performance with higher CO_2 uptake rates and excellent long-term stability. Our measurements also indicate a difference in the uptake mechanism in the two electrolytes that requires further investigation. Ultimately, this study opens a new materials class for electrochemical CO_2 capture and may lead to further performance improvements as well as new understanding of how to control the thermodynamics and kinetics of this important process

Author contributions

M.A. Khan and A.C. Forse designed and managed the project. M. Muzammil, K. Amin, J. Ling, Z. Wei, and P.J.P. Milner synthesized and provided the COF materials. K. Amin, M. Muzammil, F. Salam, M. Munawar, Z. Xu, Z. Wei, and M.A. Khan

performed material characterization and data organization. M.A. Khan conducted electrochemical measurements, with assistance from Z. Xu, S. Bird, H.R.N.B. Enninful, N.A. Hartley, and J. Taylor. K. Hetze, S. Cao, Y. Lu, and M. Oschatz carried out electron microscopy. N.Z. Ali performed XRD analysis and fitting. Z. Xu, P.J.P. Milner, Z. Wei, M. Oschatz, and A.C. Forse contributed to data interpretation and manuscript review. M.A. Khan and A C. Forse collectively wrote the manuscript with the help of all the co-authors.

Conflicts of interest

There are no conflicts to declare.

Data availability

All data are available in the main text or the supplementary materials. All raw experimental data files will be available free of cost at the Cambridge Research Repository, Apollo.

Acknowledgements

This work is supported by HEC Pakistan NRPU 15273 to M. Abdullah Khan. M. Muzammil. and M. Munawar acknowledge HEC Pakistan for PhD studentship. This work is also supported by A.C.F. UKRI Future Leaders Fellowship (MR/T043024/1). This work was supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences under Award Number DE-SC0021000 (J.L., P.J.M.). We also acknowledge support from a Camille Dreyfus Teacher-Scholar Award to P.J.M. (TC-23-048).

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