**BODIPY-Based Conjugated Porous Polymer and Its Derived Porous Carbon for Lithium-Ion Storage**

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**ABSTRACT:** Conjugated porous polymers (CPPs) possess great potential in the energy storage aspect. In this work, a boron-dipyrromethene (BODIPY)-conjugated porous polymer (CPP-1) is achieved by a traditional organic synthesis route. Following this, a carbonization process is employed to obtain the carbonized porous material (CPP-1-C). The two as-prepared samples, which are characterized by doping with heteroatoms and their porous structure, are able to shorten the lithium-ion pathways and improve the lithium-ion storage property. Then, CPP-1 and CPP-1-C are applied as anode materials in lithium-ion batteries. As expected, long-term cyclic performances at 0.1 and 1 A g⁻¹ are achieved with maintaining the specific capacity at 273.2 mA h g⁻¹ after 100 cycles at 0.1 A g⁻¹ and 250.8 mA h g⁻¹ after 300 cycles at 1 A g⁻¹. The carbonized sample exhibits a better electrochemical performance with a reversible specific capacity of 675 mA h g⁻¹ at 0.2 A g⁻¹. Moreover, the capacity is still stabilized at 437 mA h g⁻¹ after 500 cycles at 0.5 A g⁻¹. These results demonstrate that BODIPY-based CPPs are capable of being exploited as promising candidates for electrode materials in the fields of energy storage and conversion.

**INTRODUCTION**

The past decades have witnessed the problems of growing natural resource consumption and emergency requirement of environmental protection.¹⁻² Therefore, green and sustainable energy storage systems have become a research hot spot.³⁻⁸ Fortunately, lithium-ion batteries (LIBs) have been successfully used in most mobile systems owing to their merits of high energy density, environmental friendliness, and long cyclic stability. In fact, anode materials largely affect the electrochemical performance of LIBs. Although graphite has been widely used as a commercially available anode material, it still possesses a low theoretical capacity of 372 mA h g⁻¹ and a poor rate capacity, which cannot satisfy the demand for high-power and high-energy density LIBs. As alternatives, various types of anode materials with novel structures and properties have been developed, such as silicon/carbon composites,⁹ metallic oxides,¹⁰⁻¹¹ metallic sulfides,¹²⁻¹³ metal alloys,¹⁴ carbonaceous materials, and their composite materials.¹⁵⁻¹⁶ Among these, carbon-based materials with a porous structure and doped with heteroatoms have attracted extensive attention in energy storage system because of their merits of reproducible nature, low price, and wide availability of raw materials.¹⁸ Lithium-ion diffusion can be promoted by the shortening of length benefited from the porous structure. Meanwhile, the doping heteroatoms could enhance the electronic conductivity by enriching the defects for electric transport.

So far, conjugated porous polymers (CPPs) have presented versatile applications in the fields of heterogeneous catalysis, energy storage, gas separation, and hydrogen storage.¹⁹⁻⁻²⁴ These porous materials with high surface area and rich intrinsic porosity are able to provide adequate interfaces for function. A highly stable structure and synthetic diversity render them ideal alternatives to other materials. In addition, the unique π-conjugated frameworks endow these polymers with certain degrees of electron-transition capacity.²⁵ Therefore, this type of materials presents great potentials in energy storage application. Several works on CPPs as electrode materials of high-performance LIBs have been reported recently.²⁶⁻⁻²⁹ BODIPY-based CPPs have risen as a novel type of porous material and shown efficient heterogeneous photocatalytic activity, gas separation, and iodine capture capability in recent years.³⁰⁻⁻³² Apart from rich porosity and excellent physico-chemical stability, similar to other CPPs, the polymer backbone is doped with heteroatoms such as boron (B), nitrogen (N), and fluorine (F) because of the incorporation of...
Scheme 1. Synthesis Route of CPP-1 and CPP-1-C

Figure 1. High-resolution XPS spectra of B 1s, C 1s, N 1s, and F 1s for CPP-1 (a–d) and CPP-1-C (e–h).
the BODIPY moiety. The application of BODIPY-based CPPs in energy storage fields just stays at the early stage. We envisage whether BODIPY-based CPPs can be served as electrode materials in the energy storage system. However, raw CPPs as electrode materials in LIBs normally suffer relatively low capacity, which limits their further application. As is well known, the carbonization strategy is a typical and efficient postmodification method to create novel functional materials. The resulting carbonized material might exhibit some advantages such as rich defects, better conductivity, and controllable porous structure. Recent reports demonstrate the potential application of porous carbon that stemmed from CPPs in the energy storage aspect. In this way, we aim to investigate and compare the electrochemical property of BODIPY-based CPPs before and after the carbonization process as electrode materials in a LIB system.

To address the abovementioned consideration, a novel conjugated porous polymer CPP-1 is derived from the BODIPY derivative BDP-1 and 1,3,5-triethynylbenzene (TEB) by Sonogashira coupling reaction. Following this, a conventional carbonization procedure on CPP-1 is performed, and the resulting product CPP-1-C is obtained as dark brown powder. Given the high surface area and permanent nanopores, both CPP-1 and CPP-1-C can provide massive sites for the intercalation of lithium ions. In this way, CPP-1 and CPP-1-C are exploited as anode materials in LIBs. As for CPP-1, long-term cyclic performances at 0.1 and 1 A g$^{-1}$ are achieved with maintaining the specific capacity at 273.2 mAh g$^{-1}$ after 100 cycles at 0.1 A g$^{-1}$ and 250.8 mAh g$^{-1}$ after 300 cycles at 1 A g$^{-1}$. The carbonized sample exhibits better electrochemical performance with a reversible specific capacity of 675 mAh g$^{-1}$ at 0.2 A g$^{-1}$. Moreover, the capacity is still stabilized at 437 mAh g$^{-1}$ after 500 cycles at 0.5 A g$^{-1}$.

## RESULTS AND DISCUSSION

### Structure and Morphology Analysis

The resulting product was prepared according to the synthesis routes (Scheme 1). Details about the synthesis processes and characterization data are described in the Experimental Section. The solid-state 13CP/magic angle spinning (MAS) NMR spectrum was recorded to investigate the molecular structure of the obtained CPP-1 (Figure S1). The wide band between 150 and 110 ppm at the aromatic region might be attributed to the extended conjugated framework of the polymer, whereas the peaks at 40 and 15 ppm might be generated from the methyl groups of the BODIPY units. X-ray photoelectron spectroscopy (XPS) is employed to characterize the structural information of the as-prepared CPP-1 and CPP-1-C. In the detailed 1s curve (Figure 1a), peaks centered at 190.5 and 192.7 eV are observed, which are assigned to B–N and B–F bonds, respectively. While the simulation of high-resolution C 1s peak reveals the interaction of C–C≡N–B–N bond around 284.4 eV and that of C–N bond centered at 286.3 eV (Figure 1b). In the N 1s spectra (Figure 1c), two distinct peaks around 398.0 and 399.8 eV are present, which are attributed to the interaction of B–N and C–N, respectively. The 1s spectra reveal two potential components around 686.4 and 685.6 eV, which are assigned to two separate B–F bonds (Figure 1d). Therefore, the specific chemical bonds in the monomers BDP-1 and TEB have been transferred to polymer successfully, from the XPS results. As for the high-resolution XPS spectra of CPP-1-C, B–N, C–C/≡C–C, and C–N are observed as well, which is similar to CPP-1. The detected O–C==O peak around 289.0 eV can be attributed to the absorption of CO$_2$ in the air among the nanopores. We speculate that the obtained CPP-1-C could behave as a platform for CO$_2$ uptake so that a limited amount of CO$_2$ can be detected by XPS characterization. However, the F 1s spectrum is too weak to be detected after carbonization from the XPS spectra, indicating that F is mostly decomposed.

The polymerization is further confirmed by the Fourier transform infrared (FT-IR) spectrum (Figure 2). First, the corresponding peak located at 2201 cm$^{-1}$ is ascribed to the C≡C vibration arisen after polymerization, whereas the terminal C≡C–H stretching vibration at 3270 cm$^{-1}$ disappears. Second, the appearances of obvious peaks at 1630 and 2970 cm$^{-1}$ are, respectively, assigned to the C–N and C–H vibrations from the BODIPY moiety, revealing that the polymerization is successful. However, the carbonized product CPP-1-C exhibits few broad vibration peaks, and the corresponding characteristic peaks of CPP-1 vanish. This result might be ascribed to high carbonization of the CPP-1 accompanied by the break of covalent bonds and the collapse of frameworks in the polymer during the pyrolysis process.

In order to elaborate the morphologies of the obtained samples, scanning electron microscopy (SEM) and transmission electron microscopy (TEM) experiments are performed. As shown in Figure 3a,d, CPP-1 and CPP-1-C display a similar honeycomb-like morphology composed of spherened nanoparticles. Energy-dispersive spectroscopy (EDS) mappings of CPP-1 (Figure 3b1–b4) demonstrate that boron (B), carbon (C), nitrogen (N), and fluorine (F) are well distributed in the substrate. However, as shown in Figure 3e1–e4, fluorine (F) almost disappeared after the heat treatment, which is consistent with Feng’s observation. The rich porous characteristic is further verified by TEM investigation. The images reveal uniform alternately dark and bright wormlike porous microstructures (Figure 3c,f). High-resolution transmission electron microscopy (HR-TEM) indicates that both CPP-1 and CPP-1-C are amorphous materials, which agreed well with the X-ray diffraction (XRD) results (Figure S2).
Nitrogen adsorption−desorption isotherm experiments at 77 K were employed to further investigate the porous properties. As demonstrated in Figure 4a, both CPP-1 and CPP-1-C possess reversible sorption profiles. A rapid uptake at a low relative pressure ($P/P_0 < 0.05$) reveals the existence of microporous architectures. Moreover, the hysteresis in the high pressure region is attributed to the mesoporous structure. Indeed, both micropores and mesopores are available from the pore size distribution results (Figure 4b). The Brunauer−Emmett−Teller (BET) surface area of CPP-1 is calculated to be 502 m$^2$ g$^{-1}$, whereas CPP-1-C shows a higher surface area of 683 m$^2$ g$^{-1}$. The average pore sizes of CPP-1 and CPP-1-C are determined to be 20.5 and 6.9 nm, respectively. The shrinkage of the average pore size after the pyrolysis process may be attributed to the collapse of the relatively thermolabile parts of polymer frameworks under high temperatures. Hence, the hierarchical porous architectures with a high specific surface area of the two samples are confirmed. Such porous structures are beneficial to ion transportation by shortening the diffusion distance.

**Electrochemical Property of CPP-1.** Figure 5a shows the cyclic voltammetry (CV) curve of CPP-1, in which the irreversible peak can be detected around 0.7 V at the first cycle and vanished in the following cycles, which is associated with the formation of a solid electrolyte interface resulting from the decomposition of electrolyte during the initial discharge process. In addition, the long plateau below 0.2 V was attributed to the lithiation process of carbonaceous materials. The charge−discharge profiles at different cycles are displayed in Figure 5b, manifesting its superior cyclic stability with a retention of 85% after 100 cycles at 0.1 A g$^{-1}$. The relatively low initial Coulombic efficiency is due to the high specific surface area and the formation of LiF. As for the EIS result, the values of $R_p$, $R_i + R_{ct}$, and $W_o$ are 10.0, 36.5, and 7.9 Ω, respectively, which are in favor of lithium-ion transport in electrolyte. We demonstrate the electrochemical performance at different current densities from 0.05 to 5 A g$^{-1}$ in Figure 5c as well. To our delight, the sample shows a specific capacity of 138.8 mA h g$^{-1}$ even at 5 A g$^{-1}$, and the value remains at 262.6 mA h g$^{-1}$ when the current goes back to 0.1 A g$^{-1}$. As illustrated in Figure 5d,e, long-term cyclic performances at 0.1 and 1 A g$^{-1}$ are achieved with maintaining the specific capacity of CPP-1 at 273.2 mA h g$^{-1}$ after 100 cycles at 0.1 A g$^{-1}$ and 250.8 mA h g$^{-1}$ after 300 cycles at 1 A g$^{-1}$. The heteroatom doping of CPPs with superior rate performance and long-term cyclic stability can be a promising way when it comes to LIB anodes.

**Electrochemical Property of CPP-1-C.** To evaluate the anode property in LIBs comprehensively, the electrochemical properties of the as-prepared carbonized sample (CPP-1-C) are also investigated. A similar CV curve with CPP-1 can be found in Figure 6a. The discharge−charge profiles in the first three cycles at 50 mA g$^{-1}$ are shown in Figure 6b, which experience a short plateau centered at 0.7 V and a long plateau
below 0.2 V. This result is consistent with the CV profiles. In addition, the initial discharge and charge capacities are as high as 1675 and 933 mA h g\textsuperscript{−1}, respectively. As a result, the initial Columbic efficiency (ICE) is 55.7%. It is worth mentioning that the ICE is highly correlated with the formation of SEI on the porous electrode. After the initial cycle, the charge-discharge curves show good repeatability, delivering charge specific capacities of 910 and 885 mA h g\textsuperscript{−1} at the second and third cycles, respectively. The high capacity can be attributed to the fact that the heteroatoms inside the CPP-1-C skeleton can act as redox-active units, providing abundant energy storing modules. The rate performance in Figure 6c demonstrates that the as-prepared sample shows great advantages with high specific capacity and cyclic ability even at high rates. It can still maintain a reversible capacity of 222 mA h g\textsuperscript{−1} at 5 A g\textsuperscript{−1}. Moreover, when the current recovers back to 0.1 A g\textsuperscript{−1}, the specific capacity is able to return to 580 mA h g\textsuperscript{−1}, which indicates that the prepared sample shows good ability to bear the artificial change of current density in a wide range.

The cyclic performance of CPP-1-C at 0.2 A g\textsuperscript{−1} is displayed in Figure 6d. It can be seen that the sample delivers excellent cyclic stability even after 200 cycles. Particularly, the capacity decreases from 675 to 521 mA h g\textsuperscript{−1} in the first 50 cycles and remains stable in the following cycles. The capacity decay during the first 50 cycles might be attributed to the high specific area and porous character, which leads to the difficulty in forming a stabilized SEI film. After 200 cycles, the specific capacity remained at 517 mA h g\textsuperscript{−1}. An average capacity decay ratio from 50 to 200 cycles can be calculated to be 0.025 mA h g\textsuperscript{−1} per cycle. Even after 500 cycles at 0.5 A g\textsuperscript{−1} (Figure 6e), the charge capacity remained at 436 mA h g\textsuperscript{−1} with a retention of 68%. Note that the profile shows the same trend as that at 0.2 A g\textsuperscript{−1}, showing a decay at first decades (from 641 to 490 mA h g\textsuperscript{−1} in the first 25 cycles), followed by a stable level (from 490 to 436 mA h g\textsuperscript{−1} in the last 475 cycles). In addition, the cyclic performance at 0.5 A g\textsuperscript{−1} for 300 cycles after rate test (Figure S3) and long-term cyclic properties (Figure S4) further confirm the unique electrochemical performance of the prepared material. After cycling, the integrity of the pole piece can be maintained (Figure S5). The superior long-term cyclic stability and high rate performance are benefited from the stable porous character of the prepared sample, which facilitates lithium-ion transportation and storage. In addition, the N, B heteroatom doping enhances the conductivity of the sample, which can promote the electron transport especially at high rate. As a result, the rate capability can be improved.

**CONCLUSIONS**

In summary, BODIPY-incorporated CPP-1 has been successfully prepared and thoroughly characterized. The N, B codoped carbon material CPP-1-C was realized from the
abovementioned CPP-1 via a direct carbonization process. The as-prepared CPP-1 and CPP-1-C show excellent porous property with high surface area, which promoted the transportation and storage of lithium ions. Besides, the heteroatoms can behave as redox-active units that provide abundant energy storing modules. As a result, the assembled LIBs composed of the CPP-1 anode material exhibit high lithium-ion storage property, including high specific capacity, outstanding cycle stability, and superior rate performance, which is comparable to the reported state-of-the-art CPPs. In addition, obvious enhancement of electrochemical performance was detected after the carbonization treatment, which may arise from the better conductivity and structural stability of CPP-1-C. From these results, we believe that the CPPs can serve as promising candidates for the next-generation energy storage devices. Further work on this aspect to design functional CPP-based electrode materials is ongoing in our laboratory.

**EXPERIMENTAL SECTION**

**Materials.** All chemical reagents are obtained from the commercial suppliers and used without further purification unless otherwise stated. All reactions are carried out in three-neck flasks under the protection of N₂. N,N-dimethylformamide (DMF) and trimethylamine (TEA) are dried by refluxing in the presence of calcium hydroxide. Analytical thin-layer chromatography is employed to determine the reaction process. Column chromatography is applied to purify the crude products with silica gel (200−300 mesh) as the stationary phases. Deionized water is obtained from an ultrapure water system, HITECH Smart-S15.

**Instruments.** ¹H NMR and ¹³C NMR experiments are performed on the Bruker Avance spectrometer at 400 and 100 MHz at 298 K, respectively. Solid-state cross-polarization MAS (¹³C CP/MAS NMR) spectra are obtained from the Bruker Avance III 400 MHz spectrometer. All chemical shifts are reported in parts per million (ppm) by using tetramethylsilane as an internal reference. High-resolution mass spectra were recorded on the Bruker Daltonics Apex IV spectrometer. XRD (Rigaku, Rint-2000) is employed to characterize the structural evolution before and after the heat treatment from 10 to 40° at a scanning rate of 10° min⁻¹. SEM images and EDS measurements are carried out with a JSM 6390LV microscope. A JEOL JEM-2100F microscope is exploited to obtain the HR-TEM images. FT-IR spectra are collected on a Nicolet 6700 FT-IR (scimitar series) spectrometer in the 500−4000 cm⁻¹ region. The cumulative apparent surface areas for N₂ are calculated on Micro ASAP 2020 using the Brunauer−Emmett−Teller (BET) model range from 0.01 to 0.1 bar for all samples. Microporous volumes are calculated using the t-
plot method, and the total porous volumes are obtained from the N$_2$ isotherm at P/P$_0$ = 0.99. Pore size distributions are derived from the N$_2$ adsorption isotherms using density functional theory methods. All samples followed typical pretreatment procedures to be degassed at 120 °C for 8 h before the measurements. XPS spectra are recorded on an ESCALAB 250Xi.

**Synthesis of Compound 1.** The synthesis of compound 1 was according to the literature.$^{51}$ $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 8.57 (s, 1H), 8.03 (d, $J$ = 8.4 Hz, 2H), 7.91 (d, $J$ = 8.8 Hz, 2H), 7.47 (m, 4H), 5.89 (s, 2H), 2.63 (s, 6H), 0.65 (s, 6H).

**Synthesis of BDP-1.** Compound 1 (200 mg, 0.47 mmol) and an excess amount of iodine (1.0 g, 3.9 mmol) were dissolved in anhydrous tetrahydrofuran (100 mL). Aqueous mixture solvent (DMF/TEA = 1:1, 8.0 mL). The mixture was degassed by three freeze-thaw cycles again, and then the solution was heated to 100 °C for 4 h. After that, the solvent was removed under reduced pressure. The crude product was washed with water, and the organic portion was dried over MgSO$_4$. Then, the precipitate was filtered, and the organic solvent was evaporated. The residue was purified by silica gel chromatography with DCM/PE (4:1, v/v) as eluents to a red brown solid (270 mg, 85%). $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 8.62 (s, 1H), 8.06 (d, $J$ = 8.5 Hz, 2H), 7.81 (d, $J$ = 8.6 Hz, 2H), 7.51 (t, $J$ = 7.6 Hz, 2H), 7.44 (t, $J$ = 6.8 Hz, 2H), 2.71 (s, 6H), 0.67 (s, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 157.8, 157.1, 145.1, 144.3, 132.8, 131.3, 128.5, 127.4, 125.9, 124.7, 120.2, 82.0, 29.7, 22.7, 15.6, 14.1. ESI-MS: calcd for C$_{27}$H$_{22}$BF$_2$I$_2$N$_2$ [M + H]$^+$, 675.9855; found, 675.9868.

**Synthesis of CPP-1.** BDP-1 (300 mg, 0.44 mmol) and TEB (67 mg, 0.44 mmol) were dissolved in an anhydrous mixture solvent (DMF/TEA = 1:1, 8.0 mL). The mixture was degassed by three freeze–pump–thaw cycles to remove the residual oxygen, and then Pd(PPh$_3$)$_4$ (25 mg, 0.016 mmol) and CuI (7.0 mg, 0.04 mmol) were added successively into the mixture. The mixture was degassed by three freeze–pump–thaw cycles again, and then the solution was heated at 100 °C and kept stirring for 96 h under a N$_2$ atmosphere. After the mixture was cooled down to room temperature, the resulting precipitate was collected by filtration. The crude product was washed with chloroform, acetone, and dichloromethane. The resulting CPP-1 was added to a tube furnace, which was then heated to 700 °C and held for 3 h under an Ar atmosphere at a heating rate of 5 °C min$^{-1}$. After cooling down to ambient temperature, the residue CPP-1-C was collected as black powder and employed as electrode materials. The elaborate synthesis route is shown in Scheme 1.

**Electrochemical Measurements.** As a typical route, the working electrodes were prepared by mixing CPP-1 or CPP-1-C (80%) as active materials, super P (10%) as a conductive agent, and poly(vinylidene fluoride) (10%) as a binder to make slurry with the addition of N-methyl pyrrolidinone. The slurry was stirred for 0.5 h, then uniformly pasted onto a Cu foil, and dried in an oven at 120 °C for 12 h. The loading mass of the electrode materials was controlled at 0.7–0.9 mg cm$^{-2}$. The CR2025 coin-type cells were assembled in a glove box filled with Ar. Lithium metal and Celgard 2500 were employed as a counter electrode and a separator, respectively, while 1 M LiPF$_6$ in EC/DEC/DMC (1:1:1 in v/v/v) was employed as an electrolyte. Then, the as-prepared cells were laid aside for at least 8 h and tested on a Neware battery test system within the voltage range of 0.01–3.0 V at different current densities. CV experiments were carried out with a CHI660d electrochemical work station.

**ASSOCIATED CONTENT**

**S Supporting Information**

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acsomega.8b01128.

Solid-state NMR spectra, XRD pattern, element content by XPS analysis, cyclic stability, SEM morphological analysis before and after cycling, and NMR and mass spectra (PDF)

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**Author Contributions**

G.C.L and J.-F.Y. contributed equally. All authors have given approval to the final version of the manuscript.

**Notes**

The authors declare no competing financial interest.

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