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Novel Conjugated Copolymer as Promising Cathode for Rechargeable Lithium-Ion Battery

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Abstract: A novel conjugated copolymer has been synthesized and employed as an organic cathode material in rechargeable Lithium-ion batteries (LIBs). Due to the synergistic effects from conducting aniline and pyrene units, the resultant batteries based on the as-obtained copolymer can deliver a promising reversible specific capacity of 113 mAh g⁻¹ with a high voltage output of 3.2 V and a remarkable 75.2 % capacity retention after 180 cycles. Moreover, an excellent rate performance is also achieved with a fast recovery of the capacity at different current densities.

Introduction

Since the first discovery of Li-organic battery in 1969,^[1] organic electrode materials have attracted extensive interest. Although rechargeable lithium ion batteries (LIBs) with inorganic electrode materials have achieved great success in fundamental research and even commercial applications, the limited sources of critical elements (such as lithium and cobalt) and induced environment pollution have become serious issues for large scale applications of LIBs including electric vehicles and smart electricity grid.

Fortunately, the Li-organic batteries can provide an alternative way to resolve these issues and realize "green clean energy". In fact, organic rechargeable batteries have been considered as suitable candidates for next-generation sustainable energy storage systems due to their eco-friendly and potentially cost-efficient characteristics.^[2] It is well-established that redox reactions of inorganic materials is affected by the valence of metal ion. However, for organic materials, the redox process only depends on the charge state transformation of the redox-active organic units.^[3] Although various organic structures (e.g. carbonyl compounds, organosulfurs, organic salts, and conjugated polymers) and different redox mechanisms have been explored to enhance the electrochemical performance of organic batteries,^{[4],[5],[6],[7],[8]} However, before the commercialization, we need to tackle numerous challenges including poor electronic conductivity, low volumetric energy

density, and dissolution. The issues of poor conductivity is particularly important due to the electrical insulating nature of organic materials.^[9]

In contrast with metal oxides and organic small molecules/semiconducting polymers, conducting polymers possess higher conductivities, originated from the conjugation of adjacent π -electrons.^{[10], [11]} As one of the mostly investigated conducting polymer for electrochemical energy storage systems,^{[12],[13]} polyaniline (PANI) possesses several redox states, including the completely reduced leucoemeraldine, the intermediate emeraldine, and the completely oxidized pernigraniline, which are very important for conductivity and energy storage. Especially, the emeraldine salt form is believed to show the highest conductivity because of the extensive conjugation in the polymer chain, as well as the adjustable energy store ability associated with multiple redox states.^{[14],[15],[16]} Most applications has already demonstrated that the leucoemeraldine/emeraldine redox transformation can yield high energy and power. However, due to the fact that high potential of the oxidized pernigraniline (beyond 4 V vs. Li⁺/Li) could result in material decomposition,^[17] the practical specific capacity of PANI is often limited to about 150 mA h g⁻¹.^{[18],[19]} Thus, it is highly desirable to address this issue.

Although organic-inorganic hybrid materials based on polyaniline and transition metal oxides (e.g. PANI/V₂O₅,^[20] PANI/MoO₃,^[21] PANI/NiO,^[22] PANI-LiNi_{0.8}Co_{0.2}O₂ nanocomposites^[23]) have been widely used as active redox electrode materials in lithium batteries, these materials still face the volume change issues due to the swelling/shrinkage of PANI as well as the damage of the backbone of polymer during the insertion/deinsertion of counter ions.^{[17],[24]} Therefore, the construction of various copolymers containing polyaniline as electrode materials might be a promising strategy to address this issue and achieve an optimum battery performance with good cycling stability and high specific capacity.

Polyaromatic materials have been widely demonstrated as efficient anion-insertion cathodes.^{[3],[5],[10],[12],[25],[26]} In particular, polypyrene, as a redox-active polymer consisting of polycyclic aromatic hydrocarbons, has been considered as a promising candidate to replace graphite due to its good energy storage characteristic. In fact, Polypyrene has been demonstrated as an excellent organic positive electrode material for aluminium batteries.^{[26],[27],[28]} The space in-between molecular chains of polypyrene allows the easy penetration of the relatively larger ion electrolyte species as compared with lithium ions. Additionally, the porosity of polypyrene electrode can be controlled, therefore,

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polypyrene should be beneficial to specific applications in rechargeable batteries.

The combination of PANs and pyrene units might integrate the advantages of polyaniline and polypyrene and deliver an enhanced performance in LIBs.^{[29], [30]} In this research, we design a novel co-polymer by integrating pyrene species and dianiline units together through a facile one-step polymerization process. The as-prepared copolymers have been carefully characterized and used as cathodes in Li-ion batteries.

Results and discussion

1.1. Synthesis of PPDA-PYR copolymers

Figure 1. a): Synthetic route of the co-polymer poly-PPDA-PYR via the oxidative co-polymerization of *N*-Phenyl-*p*-phenylenediamine (N-PPDA) and pyrene (PYR) with iron chloride as an oxidant to produce Image of yellow pyrene flake (b), brown *N*-PPDA precursor (c) and copolymer poly-PPDA-PYR powder (d).

The targeted copolymer poly-PPDA-PYR was obtained as described in Figure 1. We copolymerized *N*-Phenyl-*p*-phenylenediamine (N-PPDA) and pyrene (PYR) in a 1:1 molar/molar ratio through oxidative polymerization with iron (III) chloride as the oxidant, the solution of N-PPDA and PYR was gradually changed into dark with a black precipitate generating upon the addition of oxidant agents.^[31] The detailed synthesis procedure is provided in the supporting information section. The as-prepared copolymer poly-PPDA-PYR is black powder (Figure 1d), which is much different from the starting materials: yellow pristine pyrene (Figure 1b) and brown sheet N-PPDA (Figure 1c).

Elemental analysis revealed that the co-polymer consists of C, 73.90 %; H, 3.34 %; N, 6.83 %, suggesting that pyrene and *N*-PPDA have been incorporated into the framework of the as-prepared copolymer with a molar ratio of 83:100 (m:n). The solid state ¹³C NMR (Figure S1) shows that aromatic carbon peaks locate at 129.4 ppm. As shown in Figure S2, the transmission FT-IR spectra of poly-PPDA-PYR, poly-PPDA, PPDA and PYR are exhibited for comparison. From the FT-IR spectrum of *N*-Phenyl-*p*-phenylenediamine, we can assign peaks 3400 cm⁻¹ and 3320 cm⁻¹ to N-H stretching vibration, 3300 cm⁻¹ and 3190 cm⁻¹ to C-H stretching vibration, 1600 cm⁻¹, 1510 cm⁻¹ and 1340 cm⁻¹ to C=C stretching vibration, C-C stretching vibration, =C-H inplane

vibration, respectively. The absorption at 1260 cm⁻¹ and 1180 cm⁻¹ indicates the C-N stretching vibration. As shown in the FT-IR of pyrene, 3030 cm⁻¹, 1590 cm⁻¹, 1430 cm⁻¹, 1180 cm⁻¹ are corresponding to C-H stretching vibration, C=C stretching vibration, C-H inplane vibration and C-C stretching vibration. The N-H stretching vibrations (3400 cm⁻¹ and 3320 cm⁻¹) and C-H stretching vibrations (3300 cm⁻¹, 3190 cm⁻¹) of *N*-Phenyl-*p*-phenylenediamine as well as C-H stretching vibration peak (3040 cm⁻¹, 3028 cm⁻¹) of pyrene were combined into broad peak in the range of 3200 cm⁻¹ after copolymerization. In the FT-IR spectrum of copolymer, 1590 (C=C stretching vibration), 1490 (C-C stretching vibration), 1290 (=C-H inplane vibration), 1180 (C-N stretching vibration), 1040 (=C-H inplane vibration). From the comparison spectrum, the spectra of the copolymer shows different IR signals compared with the respective monomers, pyrene and *N*-phenyl-*p*-phenylenediamine. The difference result from the interaction of pyrene and diamine units instead of simply physical mixing. In order to further confirm the successfully formation of copolymer, we compare the FT-IR signals of homopolymer poly-PPDA with copolymer poly-PPDA-PYR, the C=C stretching vibration shift from 1590 cm⁻¹ to 1570 cm⁻¹, C-C stretching vibration located at 1490 cm⁻¹, =C-H inplane vibration shift from 1290 cm⁻¹ to 1310 cm⁻¹, C-N stretching vibration shift from 1180 cm⁻¹ to 1150 cm⁻¹. Those FT-IR signal exhibited difference between poly-PPDA and poly-PPDA-PYR demonstrate that both *N*-Phenyl-*p*-phenylenediamine and pyrene units inserted into the copolymer molecule.

Figure 2. Raman spectra of the poly-PPDA-PYR, PPDA, and PYR samples.

As shown in Figure 2, the Raman spectra (excitation wavelength $\lambda = 1064$ nm) of copolymer poly-PPDA-PYR also exhibit much different signals from the two initial monomers PPDA and PYR. Copolymer exhibit strong Raman signal at 3650 cm⁻¹, 3400 cm⁻¹ (N-H stretching vibration), 3130 cm⁻¹, 2800 cm⁻¹ (C-H stretching vibration), but few other peak appear due to weak excitation energy source. While *N*-phenyl-*p*-phenylenediamine show N-H stretching vibration at 3320 cm⁻¹, C-H stretching vibration at 3050 cm⁻¹, C-C stretching at 1620 cm⁻¹, C-N stretching at 1220 cm⁻¹. In the Raman spectra of pyrene, 3050 cm⁻¹ and 3010 cm⁻¹ of pyrene

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are ascribed to C-H stretching vibrations, 1590 cm^{-1} is ascribed to C-C stretching vibration, 1400 cm^{-1} is assigned to C-C stretching coupling with weak ring-breathing vibration, 1240 cm^{-1} related to C-H vibration, 1070 cm^{-1} corresponding to C-H in-plane bending vibration.^[32]

The stability of the as-prepared copolymer was confirmed by Thermogravimetric analysis (TGA), it exhibit no obvious decomposition below $250\text{ }^{\circ}\text{C}$ as shown in Figure S3. All these characterization data confirm the successful formation of the as-prepared co-polymer.

Figure 3. Elemental mapping (a: Carbon element; b: Nitrogen element.) and SEM image (c) of poly-PPDA-PYR.

EDS (Energy dispersive X-ray spectrometry) elemental mapping confirms that carbon and nitrogen are the major elements (Figure. 3a, 3b). Field-emission scanning electron microscopy (FESEM) was performed to characterize the morphology of PDB. Fig. 3c shows that the polymer particles are in large micro-sized aggregations. No regular morphology can be observed and the diameters of the copolymer particles are in the range of $5\text{ }\mu\text{m}$ to $15\text{ }\mu\text{m}$.

Figure S4 shows the cyclic voltammetry (CV) profile of poly-PPDA-PYR from an assembled lithium cell. Two reversible redox peaks (3.4 V , 3.8 V vs. Li/Li^+) can be observed in the potential window of $2.0\text{--}4.2\text{ V}$ vs. Li/Li^+ , the reversibility is further displayed in the charge and discharge process. These two peaks are ascribed to the oxidation and reduction of the amine centres.^[18] The voltage difference regarding the two redox peaks demonstrates the electronic coupling interaction between the two redox amine centres. The charge and discharge profiles of poly-PPDA-PYR at different cycles are presented in Figure 4a. Poly-PPDA-PYR delivered a reversible capacity of 113 mAh g^{-1} at the initial discharge process with an average output voltage approximate to 3.2 V (Table S1). Such a high voltage could still be maintained, and a stable capacity of 85 mAh g^{-1} was achieved after 180 cycles. The stable performance of poly-PPDA-PYR has also been confirmed from Figure 4b since both a high capacity retention of 75.2% and a stable Coulombic efficiency (CE) of $\sim 99.2\%$ were observed after the 180th cycling. Different current densities were applied to confirm the rate stability of poly-PPDA-PYR. As shown in Figure 4c, poly-PPDA-PYR delivered the average capacities of 103, 94, 87, 78, 67, 64 and 58 mAh g^{-1} at the current densities of 20, 40, 100, 300, 800, 1000 and 1500 mA g^{-1} , respectively. To further assess the electrochemical stability of poly-PPDA-PYR, long-term cycling was performed as shown in Figure 4d. After 600 cycles of continuous charging and discharging, mild capacity deterioration was observed from poly-PPDA-PYR, which could still maintain a high capacity of 60 mAh g^{-1} , corresponding to an excellent capacity retention of 80% (with respect to the 1st discharge). Moreover, the average CE of poly-PPDA-PYR for the 600 cycles

was as high as about 99.12%. All these superior electrochemical properties indicate the great potential of poly-PPDA-PYR to be used as an effective organic cathode material for rechargeable LIBs. From the comparison of key properties of selected outstanding conducting polymers as electrode materials, the as-prepared copolymer also shows more pronounced electrochemical performance. (Table S2.)

Figure 4. Electrochemical performance of poly-PPDA-PYR between 4.2 and 2.0 V . a): Charge & Discharge profiles (the 1st, 2nd, 10th, 100th, & 180th cycles). b): Cycling Performance at 20 mA g^{-1} (for 180 cycles). c): Rate Performance (at different current densities). d): Long-term cycling at 500 mA g^{-1} (for 600 cycles).

Where $x + y = 1.48$

Figure 5. Proposed redox reactions at the anode and cathode of the copolymer-based battery.

The proposed mechanism is described in Figure 5. Lithium metal is used as a counter electrode as an anode. At the cathode, the anion species PF_6^- from the electrolyte salt is inserted into the polyaniline component in the copolymer upon charging and liberated from the copolymer during discharging. Accordingly, the Li^+ from the LiPF_6 salt is plated/stripped on the lithium-metal anode.

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Specifically, based on the reversible maximum specific capacity achieved (113 mAh g^{-1}), poly-DANI-PYR (with a molecular weight of 351 g mol^{-1} per repeating unit) could reversibly accept and release about 1.48 PF_6^- upon charging and discharging. On the one hand, PF_6^- anion species could be inserted into the aniline structure through a redox process. On the other hand, due to the large conjugated structure of pyrene for efficient π - π stacking effect, the space in-between the pyrene units can also store certain amount of PF_6^- . Hence, both anion insertion methods are favourable for the high specific capacity of poly-DANI-PYR. Therefore, the synergistic effect from the storage of anions by different organic motifs is proved to be an efficient method to improve the electrochemical performance of polymers.

Conclusions

We have prepared a novel copolymer via a one-step oxidation copolymerization. The as-obtained copolymer poly-DANI-PYR showed a good reversibility with a stable cycle life of 600 cycles. In addition, poly-DANI-PYR as a cathode material delivered a maximum capacity of 113 mAh g^{-1} with an average output voltage of 3.2 V . The high capacity of poly-DANI-PYR was attributed to the synergistic effects from aniline species and pyrene units. The presented promising electrochemical performance of the copolymer strongly stimulate the research interests in exploring other new high-performance copolymers through the substitution of conducting units. Particularly, pyrene would be a preferable choice to be used in combination with other conducting motifs. The other related works on the copolymers for electrochemical energy storage systems are currently in progress in our lab.

Conflicts of interest

There are no conflicts to declare

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Keywords: pyrene • polyaniline • copolymer • lithium-ion battery • Cathode

Experimental Section

Experimental Details.

Synthesis of PPDA-PYR:^[31]

The synthesis of target poly-DANI-PYR are described in **Fig. 1**. N-Phenyl-p-phenylenediamine monomer (184 mg, 1.0 mmol) and pyrene (254 mg, 1.0 mmol) were dissolved in the mixed solvents $\text{MeNO}_2/n\text{-Hexane}$ (v/v: 15 mL/15 mL), then the prepared solution of FeCl_3 (10 mmol) in MeNO_3 (10 mL) was added dropwise with strong stirring at 50°C . The mixture was continued at the same condition for 24 hrs. After the solution was cooled down to room temperature, the precipitate was collected by filtration, and washed with deionized water 4 times and then ethanol 2 times. The product was soxhlet extracted with acetone for 24 hrs and was dried at 60°C in vacuum overnight; the product was obtained as a black solid. Elemental Analysis: Found: C, 73.90; H, 3.34; N, 6.83.

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