

Electronic Supplementary Information

Facile synthesis of $\text{Li}_2\text{C}_8\text{H}_4\text{O}_4$ -graphene composites as high-rate and sustainable anode materials for lithium ion batteries

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Experimental Details

Preparation of Graphene

All the experimental chemicals were analytical grade and used without further purification. Graphene was reduced from graphene oxide (GO) by heating treatment. The GO was exfoliated from the graphite oxide prepared using Hummers method with a little modification. The details are as follows: firstly, 0.4 g of natural graphite powder and 0.35 g of NaNO_3 were placed in a three-necked flask with a stirrer chip followed by adding 30 mL of 98 % H_2SO_4 slowly. The mixture was stirred in an ice water bath environment for 1h. Then 1.8 g of KMnO_4 (purity 99%) was added gradually under slowly stirring for about 3h. The as-formed mixture was reacting for seven days at room temperature. Afterwards, 40 mL of 5 wt% H_2SO_4 aqueous solution was added dropwise and stirred for 1h followed by adding 1.2 mL of 30wt% H_2O_2 aqueous solution and stirred for another 1h. This solution was washed thoroughly with a mixed aqueous solution of 3 wt% $\text{H}_2\text{SO}_4/0.5$ wt% H_2O_2 and then deionized (DI) water several times. The resultant mixture was centrifuged and a brown-black graphite oxide dispersion was obtained. The graphite oxide dispersion was sonicated at 50 °C for 5 h to form a stable GO dispersion. Finally, the GO was put in a quartz boat in the center of a tube furnace. After flowing 25% H_2 -75% Ar combination gas for about 10 mins, then temperature ramp rate for the oven is 50 °C min^{-1} , the furnace was heated up to 900 °C for 2h. After the furnace was cooled below 50 °C, graphene was obtained.

Preparation of PTAL

For preparation of PTAL, 1.66 g terephthalic acid was dissolved in 50 mL ethanol and 0.90 g LiOH·H₂O was dissolved in 25 mL DI water. Then the terephthalic acid solution was added into LiOH solution dropwise under slow stirring conditions at 80 °C for 24 h. Then, the solution was centrifuged, washed, and dried in vacuum at 80 °C for 12 h to obtain a white powder called PTAL.

Preparation of PTAL-G

PTAL-G was prepared through a dispersing-depositing process: 0.8 g PTAL and 0.04 g graphene was dispersed in 8 mL DI water. The mixture was stirred for 10 mins and sonicated for 2 h. The mixture was put in a quartz boat in the center of a tube furnace. After flowing Ar gas for about 10 mins, the furnace was heat up to 425 °C for 5 h. Finally, a black PTAL-G was obtained.

Characterization

The structure of the as-prepared samples were characterized by powder X-ray diffraction (XRD, Bruker D8 Advance Germany) with Cu-K α radiation and Fourier transformed infrared spectroscope (FTIR, Burker Tensor 27). The morphology was observed with a scanning electron microscope (JSM-6380, Japan) and transmission electron microscope (JEM-2100HR, Japan). Thermogravimetric analyses (TGA) were carried out on a Perkin-Elmer TGA 7 thermogravimetric analyzer with a heating rate of 10 °C/min under nitrogen atmosphere.

Electrochemical characterization

Coin-type cells (CR2025) consisting of a working electrode and a lithium foil counter electrode separated by a Celgard 2400 microporous membrane were assembled in an argon-filled glove-box. The working electrodes were prepared as follows: 40 wt% as-prepared samples (PTAL, PTAL-G and graphene) with 50 wt% acetylene black and 10 wt% polytetrafluoroethylene binder were mixed in N-methylpyrrolidinone (NMP), coated and compressed onto a copper foil current collector. The average load of active material on the electrode is about 1 mg per cm². The electrolyte was 1M LiPF₆-EC+DMC (V_{EC}:V_{DMC}=1:1). The charge/discharge experiments were performed by using a LAND cell test system (Land CT 2001A) in the potential range of 0.5~3 V versus Li/Li⁺ at different current rate. Cyclic voltammograms (CVs) and electrochemical impedance spectroscopy (EIS) measurements were obtained on PGSTAT-30 (Autolab), CV was tested in the potential range of 0.5~3V at a scanning rate of 0.05 mV/s, EIS was tested at the open-circuit voltage in a frequency range of 10⁵-0.01Hz with an amplitude of 5 mV.

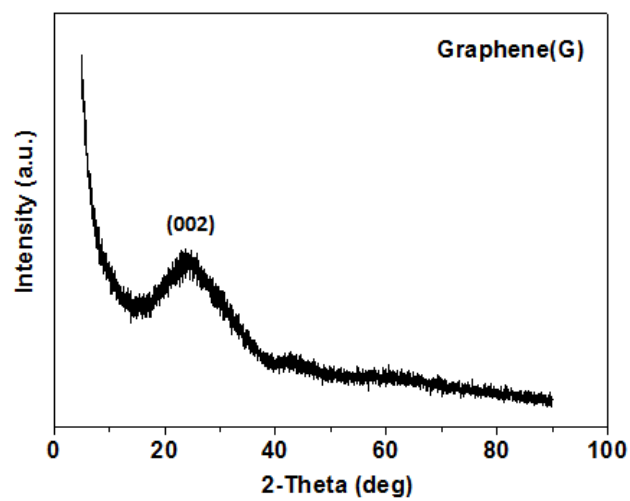


Fig. S1. XRD pattern of as-synthesized graphene

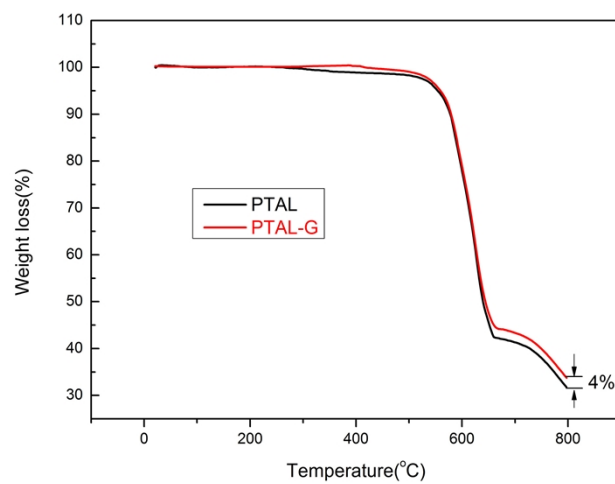


Fig. S2. The TG curves of the PTAL and PTAL-G.

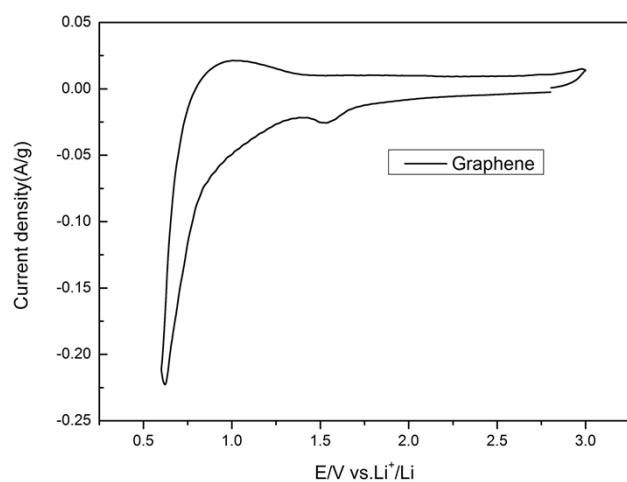


Fig. S3. Cyclic voltammogram of the graphene, scan rate: 0.05 mV/s

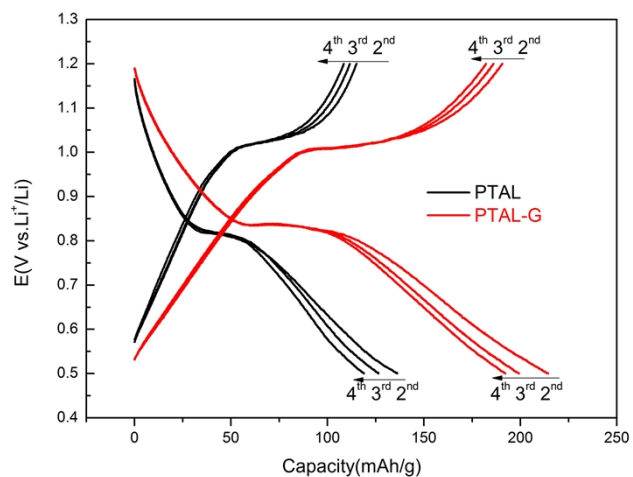


Fig. S4. The charge/discharge curves of the PTAL and PTAL-G at 0.1 C between 0.5 and 1.2 V

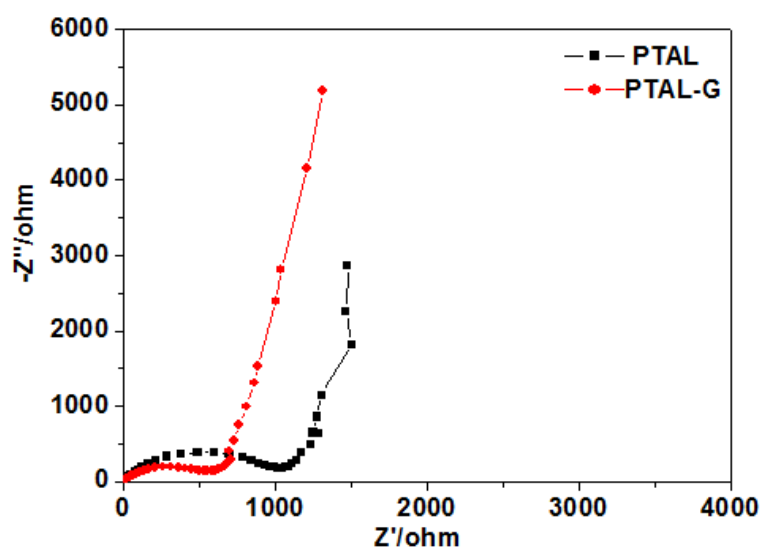


Fig. S5. Nyquist plots of the PTAL and PTAL-G before cycle

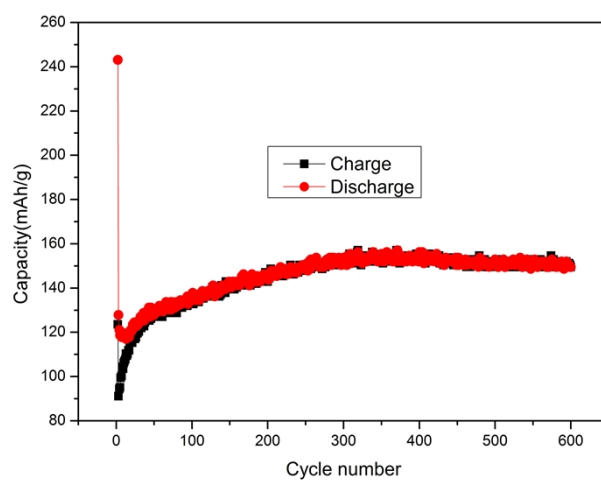


Fig. S6. The cyclic capacity of the graphene at 10C

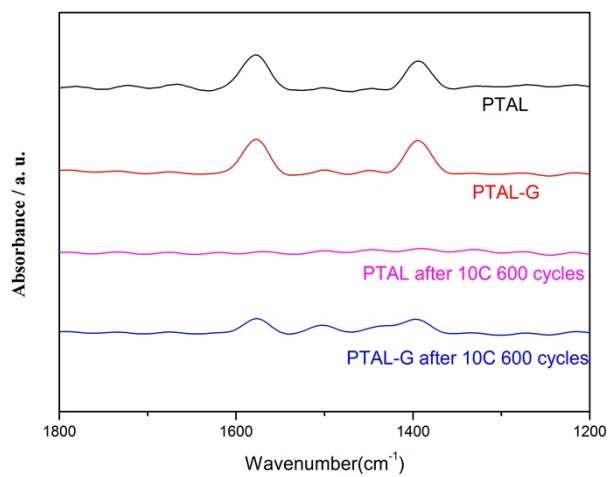


Fig. S7. The FT-IR spectra of the PTAL and PTAL-G electrodes

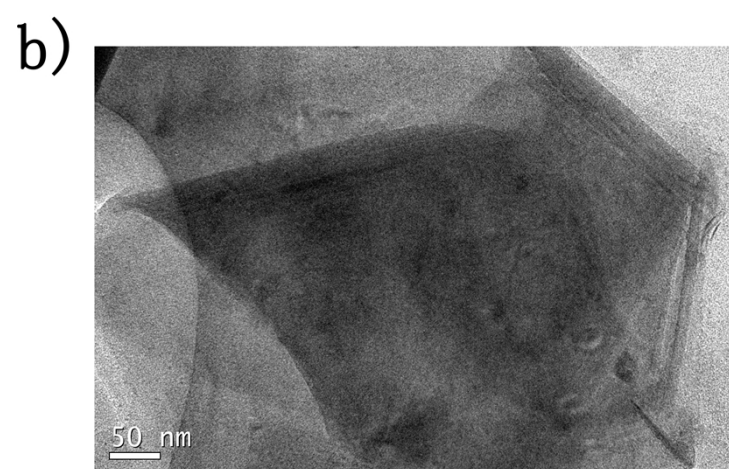
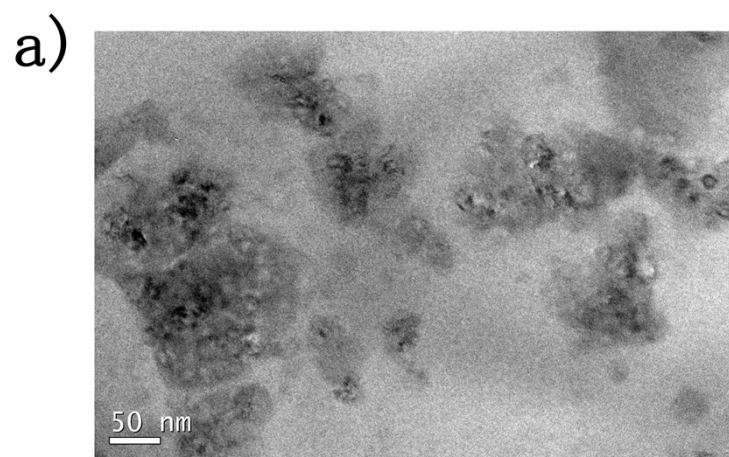


Fig. S8. (a) HRTEM image of the PTAL; (b) HRTEM image of the PTAL-G.