Supplementary Material (ESI) for Chemical Communications
This journal is (c) The Royal Society of Chemistry 2013

Polyvinyl Pyrrolidone-assisted synthesis of Fe_3O_4/graphene composite with excellent lithium storage properties

Mochao Cai, Hang Qian, Zhikai Wei, Jiajia Chen, Mingsen Zheng* & Quanfeng Dong*
State Key Laboratory for Physical Chemistry of Solid Surfaces and Department of Chemistry, College of Chemistry and Chemical Engineering, Xiamen University, Xiamen, Fujian 361005, China.

Supplementary Information

Materials and Methods

Material preparation In a typical procedure, three embedded segment copolymers P123 ($EO_{20}PO_{70}EO_{20}$, Mw=5800 g mol^{-1}) was dissolved in ethylene glycol (EG) and then graphene oxide synthesized by the modified Hummers method [34] was dispersed in it. Then, 0.75 g ferric acetate and 0.16 g PVP were added into this solution. This sample was named as Fe_3O_4/GNS-PVP. Another sample was prepared without PVP for comparison and was named as Fe_3O_4/GNS. After stirring for 0.5 h, the mixture was transferred into a reflux unit and the temperature was maintained at 180 ℃ for 4 h. After incubation, the precursors were filtered and washed with acetone, deionized water and ethanol in sequence to remove the solvents and surfactants. The precursors were dried in a vacuum oven at 60 ℃ overnight and then sintered at 500 ℃ under Nitrogen atmosphere to obtain the final products.

Structure characterization The samples were characterized by XRD (Philips X'pert Pro Super X-ray diffractometer using Cu Kα radiation with λ=1.5418 Å). The crystal structure and surface morphologies were characterized by scanning electron microscopy (SEM, HITACHI S-4800) and transmission electron microscopy (TEM, F30). Raman spectra were recorded from 200 to 2000 cm^{-1} at room temperature using XploRa Raman spectrometer with an excitation line of 532 nm. Thermal behavior of samples was characterized by thermogravimetic analysis at a heating rate of 5 ℃/min from 25 ℃ to 600 ℃ in a dynamic atmosphere of air (TGA, Perkin Elmer, PyrisDiamond) .

Electrochemical characterization Electrochemical performances of the samples were evaluated using CR2016-type coin cells assembled in an Argon-filled glove box. The working electrode was fabricated by the composites mixing with water soluble polymer n-lauryl acrylate (LA, Chengdu, China) and acetylene black in a weight ratio of 8:1:1. 1 M LiPF6 in ethylene carbonate (EC), dimethyl carbonate (DMC) and ethyl methyl carbonate (EMC) (1:1:1 in volume) was used as electrolyte, and the separator was Celgard 2400 microporous film. Charge and discharge testing of the batteries were performed with a Neware BTS-5V 5 mA Tester (Neware Co., Ltd, Shenzhen, China) within 0.02-3.0 V at room temperature. The open-circuit voltage of the fabricated and aged cells (10 h) was about 3.0 V. Electrochemical impedance spectroscopies (EIS) were conducted over 0.01 Hz to 100 kHz using a Zahner Elektrik electrochemical workstation (IM6e, Zahner, Germany). The data were analysed using Z-view software (Version 2.2, Scribner Associates Inc., USA) to obtain the Nyquist plots.

Fig. S1 XRD images (a) and TG curves (b) of Fe3O4/GNS-PVP and Fe_3O_4/GNS.

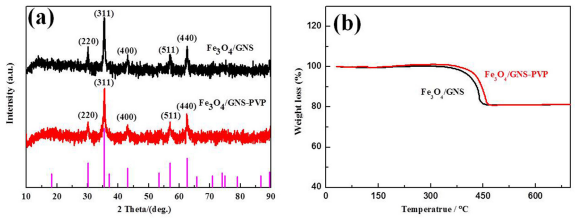

Fig. S2 The adsorption-desorption isotherm (a) and pore-size distribution (b) of Fe_3O_4/GNS-PVP and Fe_3O_4/GNS

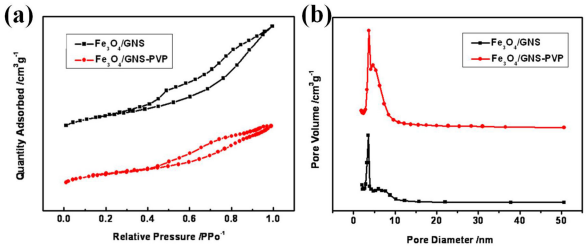

Fig. S3 Cyclic voltammograms of (a) Fe_3O_4/GNS-PVP and (b) Fe_3O_4/GNS from the first cycle to the third cycle at a scan rate of 0.2 mV s^{-1} in the voltage range of 0.01–3.0 V

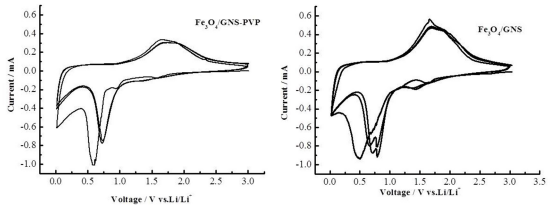

Fig. S4 FTIR spectra of (a) Fe_3O_4/GNS and (b) Fe_3O_4/GNS-PVP.

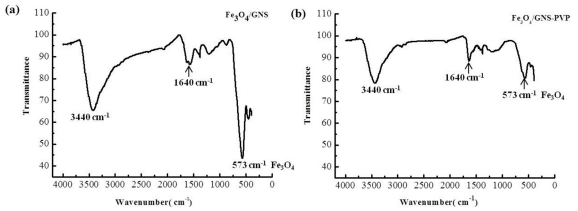

Fig. S5 SEM images of (a) Fe_3O_4/GNS-PVP and (b) Fe_3O_4/GNS