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Polypyrrole Encapsulation on Flower-like Porous NiO for Advanced High-performance Supercapacitors

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Experimental Section

All of the reagents used in the present work were analytical grade and purchased from Aladdin.

Synthesis of NiO Nanoflowers: The NiO precursor nanoflowers were synthesized by hydrothermal method. Briefly, 0.530 g of $Ni(NO_3)_2 \cdot 6H_2O$ and 0.830 g of $CO(NH_2)_2$ were dissolved in 180 mL deionized water. The obtained homogeneous solution was further transferred into an autoclave reactor, which was heated in an oven at 180 °C for 4 h then cooled naturally to room temperature. The product was washed with DI water and ethanol for several times, respectively, then dried at 60 °C in an oven overnight. Finally, the product was calcined at 350 °C for 2 h to get the final product.

Synthesis of NiO@PPy Nanoflowers: The as-prepared NiO nanoflower was encapsulated by polypyrrole (PPy) via an *in situ* polymerization in water and alcohol mixture at 0-5 °C. 30 mL of anhydrous alcohol p-TSA (p-TSA concentration: 0.08 M) was mixed with 0.1 mL pyrrole monomer and stirred for 10 min to form a uniform mixture A. This step is allowed the pyrrole monomer to diffusion into the porous structure of the NiO nanoflower, forming a uniform distribution of the pyrrole monomer. At the same time, Solution B (volume of solution B: 20 mL) is 0.026 M of APS aqueous solution. Subsequently, solution B was added dropwise into the solution A (0.5 mL min^{-1}). The mixture was shielded from light for 24 h before rinsing with ethanol and deionized water successively to remove residues. In order to compare the performance of NiO@PPy with different thickness, a series of NiO@PPy with different molar ratio of the pyrrole monomer and the NiO are synthesized, which abbreviation for NiO@PPy-n ("n" is the molar ratio of py to NiO, here 0.5, 1.0, 1.5, 2.0 are used), the black products (figure S4) were washed with deionized water and ethanol by centrifuge separation, followed by drying at 80 °C in air, resulting in NiO@PPy-n nanoflower powder.

Synthesis of $Ni(OH)_2$-200@PPy and $Ni(OH)_2$@PPy Nanoflowers: $Ni(OH)_2$-200@PPy and $Ni(OH)_2$@PPy was synthesized via a same encapsulation process, except the calcination temperature change to 200 °C and without calcine (room temperature), respectively.

General Characterization: The NiO@PPy composites were characterized by X-ray diffraction (XRD, Rigaku Japan D/max 2400 diffractometer) using Cu Kα source with a wavelength of λ=0.154 nm and a scanning rate of 0.02 ° s^{-1}. The surface morphology, microstructure, and elemental composition of the composites were characterized by scanning electron microscopy (SEM, JEOL JSM-5900LV) at an accelerating voltage of 5 kV and transmision electron microscopy (TEM, Tecnai G2 F20 S-TWIN). X-ray photoelectron spectra (XPS, XSAM800) is used to characterize the elemental composition of the samples. Infrared spectra were obtained with a Nicolet 6700 Fourier Transform Infrared Spectroscopy

(FTIR) instrument from 4000 to 650 cm^{-1}. Thermal gravimetric analysis (TGA) test conducted at Q500 Thermogravimetric analyzer (TA instruments, USA) from 30 to 650 °C at air atmosphere (under 60 mL min^{-1} flow) with the heating rate of 10 °C min^{-1}.

Electrochemical Characterization: A three-electrode system was used to the electrochemical measurements. The composites materials were used as the working electrode, 6 M KOH aqueous solution as the electrolyte, Ag/AgCl as the reference electrode and a Pt mesh as the counter electrode, respectively. The active material, acetylene black and PTFE were mixed at a weight ratio of 80:10:10 in isopropanol to form a paste and then dropped onto the precleaned nickel foam, dried at 80 °C in air overnight to obtain the electrode. The electrochemical performance was characterized by cyclic voltammetry, galvanostatic charge-discharge tests, and electrochemical impedance spectroscopy measurements by an electrochemical working station (Chenhua CHI660E).

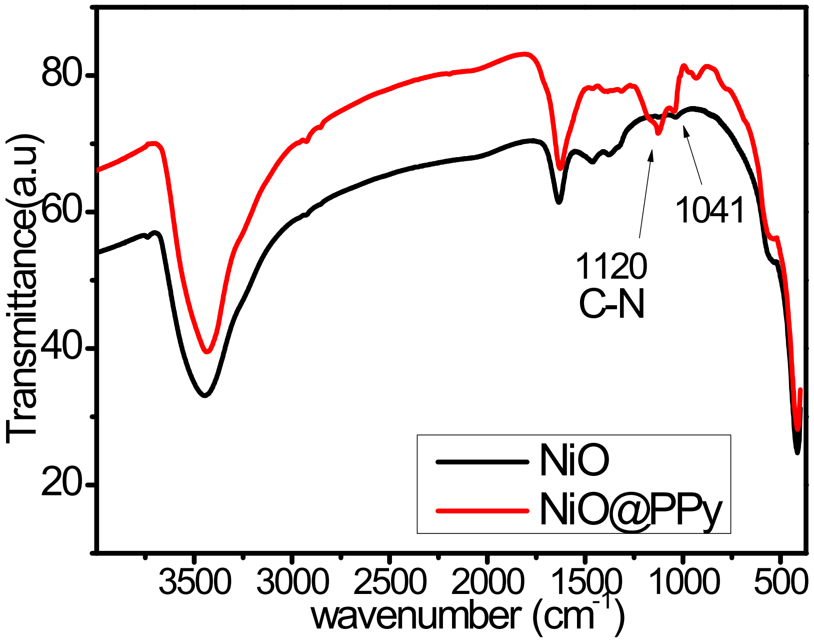

Figure S1 FT-Infrared spectra of the NiO@PPy, two new peaks located at 1120 cm^{-1} which belongs to the C-N bond, and 1041 cm^{-1} which belongs the pyrrole ring characteristic absorption peak are observed after the encapsulation process.

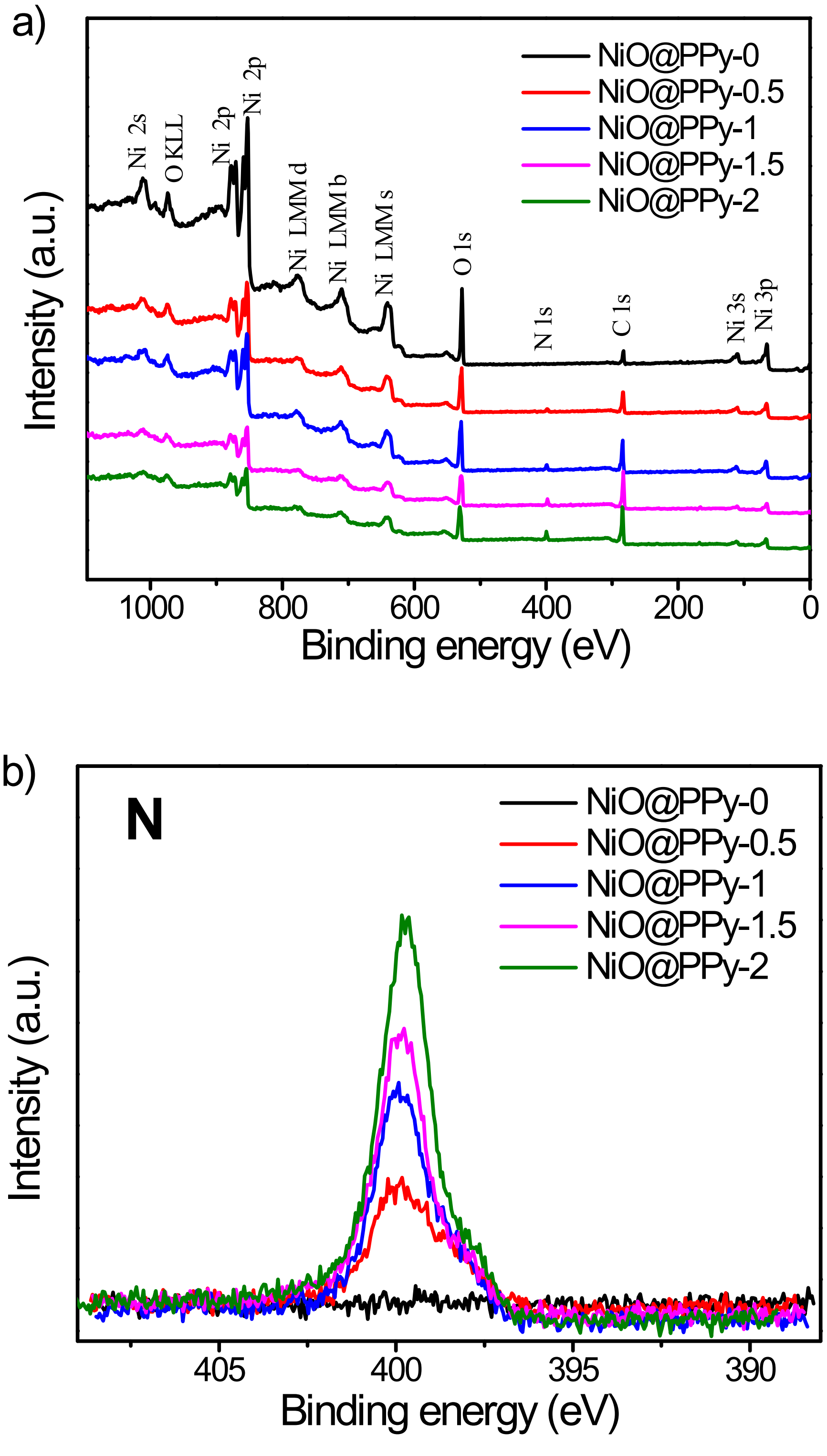

Figure S2 The XPS spectras of the NiO@PPy composites of a) full-scan and b) N 1s.

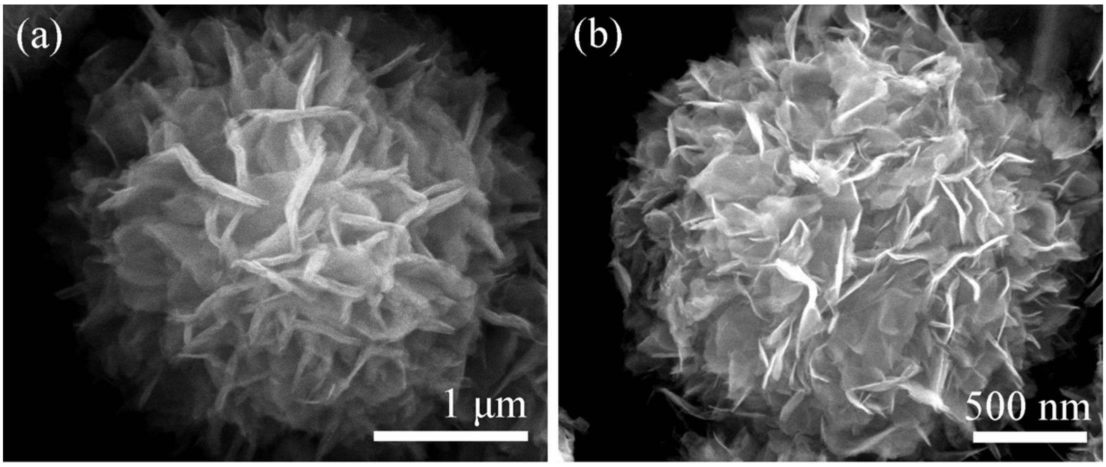

Figure S3 The SEM images of a) $Ni(OH)_2$ and b) NiO.

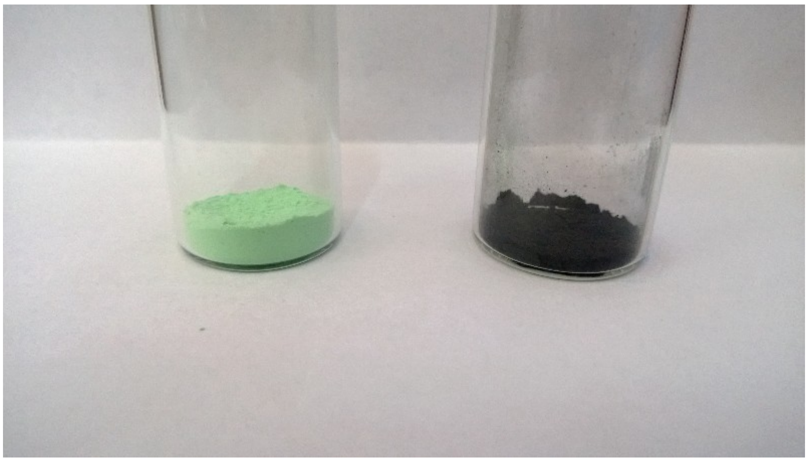

Figure S4 Photograph of the $Ni(OH)_2$ (left) and $Ni(OH)_2$@PPy (right). As the green powder turn to black after PPy coating process, indicating the successful encapsulation of PPy onto the $Ni(OH)_2$ surface.