

Electronic Supplementary Information for

**A facile novel preparation of three-dimensional Ni@graphene by catalyzed
glucose blowing for high-performance supercapacitor electrode**

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Experimental details

NG Preparation

The NG was prepared by one-step method. The specific process is as follows: 2.38 g $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$, 2.00 g glucose and 2.00 g NH_4Cl were fully mixed and placed in a tube furnace. The mixture was heated at a rate of 3 °C/min under N_2 flow to 900 °C and maintained for 3 hours. The MCF was prepared by the above treatment method without adding $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$, while the BRF was prepared by the above treatment method without adding NH_4Cl . Besides, the E-NG and E-BRF were obtained by using 3 M HCl to etch NG and BRF at 80 °C, respectively.

Characterization

The structure of the samples was analyzed by powder X-ray diffraction (XRD, Bruker D/max 2500 PC), Raman spectroscopy (Raman, Renishaw RM1000-Invia), field emission scanning electron microscopy (FESEM, SAPHIRE SUPRA55) and transmission electron microscopy (TEM, JEOL JEM-2100). The Brunauer-Emmett-Teller surface area of as-synthesized samples was measured using an ASAP2010C surface aperture adsorption instrument (Micromeritics Instrument Corporation) by N_2 physisorption at 77 K. The content of Ni in NG was analyzed using inductively coupled plasma atomic emission spectrometry (ICP-AES, Analytik Jena AG, novAA 300) and thermogravimetric analyzer (TG, Rigaku Thermo 209 F3).

Electrochemical Measurements

Electrochemical measurements were performed in 2 M KOH solution in a three-electrode cell on a CHI 920C electrochemical working station. Platinum foil and a saturated calomel electrode (SCE) were used as the counter and reference electrodes, respectively. The as-prepared powder was mixed with 20 wt% acetylene black, 5 wt% polytetrafluoroethylene and a small amount of deionized water to produce a homogeneous paste, which was pressed onto nickel foam current collectors ($10 \times 10 \text{ mm}^2$) to make working electrode. The mass of active material was in a range of 3.8-8.4 mg.

DTG of a glucose-NH₄Cl-NiCl₂·6H₂O mixture

Fig. S1 DTG of glucose-NH₄Cl-NiCl₂·6H₂O mixture and glucose-NH₄Cl mixture.

The first mass loss at 107 °C results from the loss of water in the system. The adjacent strong endothermic peak at 137 °C should be attributed to melting of glucose. The endothermic peak appeared at 168 °C is dominated by Maillard reaction.¹ A strong endothermic peaks appeared at 243 °C, indicating NH₄Cl gradually decomposed to release HCl and NH₃ gases. In the following process, the NiCl₂ distributed on the surface of the thin wall of the cavities reacted with oxygen atoms on the surface of the glucose-derived polymers to generated NiO around 320 °C.² Subsequently, the NiO is converted into metallic Ni at 528 °C.^{3,4}

Effect of different amount of NH₄Cl on the morphology of the material

Fig. S2 FESEM images of Ni-C materials with 0 g (a), 0.5 g (b), 1.0 g (c) and 1.5 g (d) of NH₄Cl added; TEM image of etched Ni-C material with 1.5 g of NH₄Cl added (inset of d).

When no NH_4Cl was added, the distribution of the metal Ni is nonuniform, which easily leads to the agglomeration (Fig. S2a) and low specific surface of metal Ni, restricting the dissolution of C in Ni. When 0.5 g of NH_4Cl was used (Fig. S2b), the agglomeration of metal Ni was declining, but the utilization rate of Ni was still relatively low, which cannot play an effective catalytic graphitization role on carbon source. With the amount of NH_4Cl being increased respectively to 1.0 g and 1.5 g, the shaping performance of NH_4Cl was obvious, and the morphologies of 3D interconnected structures appeared in Ni-C materials (Fig. S2c-d). Nevertheless, the transparent graphene layer was not observed in the TEM image of etched Ni-C materials (with 1.5 g of NH_4Cl added) (the inset of Fig. S2d), suggesting the walls of the cavities was thick and the graphene structure was not formed.

XRD patterns of Ni-C composites prepared at different calcination temperature

Fig. S3 XRD patterns of Ni-C composites prepared at 600, 700, 800, 850, and 900 °C.

N_2 adsorption-desorption isotherms characterization of NG

Fig. S4 Nitrogen adsorption/desorption isotherm of NG.

TG characterization of NG

Fig. S5 TG curve of the NG composite.

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