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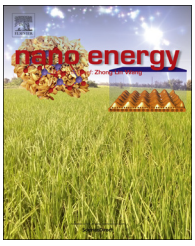

RAPID COMMUNICATION

High performance asymmetric supercapacitors: New NiOOH nanosheet/graphene hydrogels and pure graphene hydrogels

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Abstract
NiOOH nanosheet/graphene hydrogels (H-NiOOH/GS), with mesoporous NiOOH nanosheets uniformly dispersed within the highly interconnected 3D graphene network, are constructed and studied for the first time by a mixed solvothermal and hydrothermal reaction. The effect of solvent composition on the morphology, phase, dispersibility of nanocrystal and hydrogel strength is systematically studied. As binder-free electrodes of supercapacitors, H-NiOOH/GS delivers high capacitance of 1162 F g^{-1} at 1 A g^{-1} with excellent rate capability (981 F g^{-1} at 20 A g^{-1}). The charge-storage mechanisms of H-NiOOH/GS are in-depth investigated by quantifying the kinetics of charge storage, which reveals that NiOOH exhibits both capacitive effects and diffusion-controlled battery-type behavior during charge storage. Additionally, solvothermal-induced pure graphene hydrogels (H-GS) are also prepared and used as the negative electrode for the first time, which show an impressive specific capacitance of 425 and 368 F g^{-1} at 5 and 40 mV s^{-1}, respectively. Benefitting from the synergistic contribution of both positive and negative electrodes, the assembled H-NiOOH/GS//H-GS asymmetric supercapacitors achieve a remarkable energy density of 66.8 W h kg^{-1} at a power density of 800 W kg^{-1}, and excellent cycling stability with 85.3% capacitance retention after 8000 cycles, holding great promise for energy storage applications.
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Introduction

High-performance energy-storage and -conversion devices have attracted tremendous attention because of the rapid growth in energy consumption in the world [1]. Electrochemical capacitors (ECs) are one of the most promising devices due to their superior power density, ultrafast charging-discharging rate, long-cycle-life, and safety in use. However, supercapacitors suffer from relatively poor energy density, at least one order of magnitude lower than those of traditional batteries. To boost the energy density, one way is to rational design and explore novel-structured electrode materials with high specific capacitance [2]. Another solution is to develop asymmetric supercapacitors (ASCs) consisting of a battery-like Faradic electrode (as energy source) and a capacitor-like electrode (as power source), which have different potential ranges [3]. Compared with conventional symmetric ECs, the asymmetric configuration can greatly widen the operation-voltage window, resulting in a notable improvement in energy density.

As to the electrode material, a high-performance EC electrode generally requires high electrical conductivity, large ion-accessible surface area, fast ionic transport rate and good electrochemical stability [4]. In these regards, graphene-based composite materials, such as metal oxide/grapheme [5], metal hydroxide/grapheme [6] and polymer/grapheme [7], have been demonstrated to be very promising EC electrodes. However, driven by the strong π-π interaction, graphene sheets will readily parallel re-stack to form graphite-like powders during processing, leading to a severe reduction in specific surface area and becoming a serious obstacle to making full use of the unique properties of grapheme [8,9]. Moreover, polymer binder and/or conductive additives are usually required to be mixed with graphene-based active materials to make electrodes, which further reduce the specific capacitive performance.

Recently, three-dimensional graphene-based frameworks, especially graphene-based hydrogels, have received particular attention for potential applications in supercapacitors [7,10,11], supports for catalysts [12] and so on. The unique graphene gels consist of interconnected 3D porous frameworks with large specific surface areas, allowing multidimensional electron transport and rapid electrolyte ions diffusion [13]. In addition, the hydrogels can be used as binder-free electrodes, thus can effectively reduce the "dead surface" in traditional slurry-derived electrodes and facilitate more efficient charge and mass transportation [1,14]. For example, Duan et al. have demonstrated that a hydrothermally produced graphene hydrogel can give a specific capacitance of 190 F g^{-1} at 1 A g^{-1} [11]. Later, the same group further prepared functionalized graphene hydrogels (FGHs) by one-step chemical reduction of graphene oxide (GO) using hydroquinones as the reducing and functionalizing molecules simultaneously [13]. The FGHs showed a good specific capacitance of 441 F g^{-1} at 1 A g^{-1} in 1 M H_2SO_4 aqueous electrolyte, demonstrating exciting potential of the graphene hydrogels for energy storage applications. Although construction of graphene sheets into hydrogels has greatly improved its electrochemical performance, the specific capacitance of a pure graphene hydrogel is fundamentally limited by its electrical double layer (EDL) capacitance mechanism. To further improve their specific capacitance, many efforts have been made to composite graphene hydrogels with other pseudocapacitive materials (metal oxide (MnO_2 [5], Mn_3O_4 [15], V_2O_5 [16]), metal hydroxide ($Ni(OH)_2$ [6,10]), polymers [7], and so on) that have a higher theoretical specific capacitance. For instance, Ni $(OH)_2$ particles/GS hydrogels have been prepared by a hydrothermal method, which exhibited a specific capacitance of $\sim$1250 F g^{-1} at a scan rate of 10 mV s^{-1} [10]. Similarly, V_2O_5/GS hydrogels were also constructed by the hydrothermal reaction [16]. The composite hydrogel is capable of delivering a high specific capacitance of about 320 F g^{-1} at a current density of 1.0 A g^{-1}. Alternatively, Yan et al. employed a reduction-induced in situ self-assembly and constructed MnO_2/GS hydrogels, which delivered a capacitance of 242 F g^{-1} at 1 A g^{-1} and 92 F g^{-1} at 8 A g^{-1} [5]. Despite these encouraging results of graphene-based hybrid hydrogels, the reported methods are mostly based on chemical reduction or hydrothermal reaction, using H_2O as the reaction solvent. However, the influence of reaction solvents on the morphology, phase, nanocrystal dispersibility, and hydrogel strength of products has rarely been investigated. Moreover, the study of graphene-based hybrid hydrogels for asymmetric supercapacitors is also very limited despite their significant potential.

In this paper, we demonstrate that a mixed solvothermal and hydrothermal reaction (DMF and H_2O), instead of the commonly used hydrothermal reaction, can produce a new material, NiOOH nanosheet/GS hydrogels. Although many nickel-based materials (NiO, $Ni(OH)_2$) have been intensively investigated as the electrode materials for ECs, the research of NiOOH in this field is rather limited, and NiOOH/GS hydrogels have never been constructed and studied in previous work. Our study revealed that the composition of solvent played a key role in determining the final phase of the products. The effect of DMF/H_2O ratio on the morphology, dispersibility of nanocrystal and hydrogel strength was also systematically studied. As a binder-free electrode for ECs, the optimized NiOOH/GS hydrogels exhibited a specific capacitance of 1162, 1051 and 981 F g^{-1} at 1, 10 and 20 A g^{-1}, respectively, demonstrating excellent electrochemical performance. Furthermore, an ASC, based on as-prepared NiOOH/GS hydrogels as the positive electrode and solvothermal-induced pure graphene hydrogels as the negative electrode, has been successfully constructed. The ASC delivers a high energy density of 66.8 W h kg^{-1} at a power density of 800 W kg^{-1} and exhibits remarkable cycling stability with retention of 85.3% of specific capacitance after 8000 cycles at a large operating potential of 1.6 V. These findings highlight the importance of the solvent to final products, and open up the possibility of NiOOH/GS hydrogels for applications in high-voltage ASCs with high energy and power densities.

Experimental procedures

Material synthesis

GO was prepared from graphite powder (Alfa-Aesar) by the modified Hummers method [17].

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