



Hierarchical NiCo-LDH@NiOOH core-shell heterostructure on carbon fiber cloth as battery-like electrode for supercapacitor



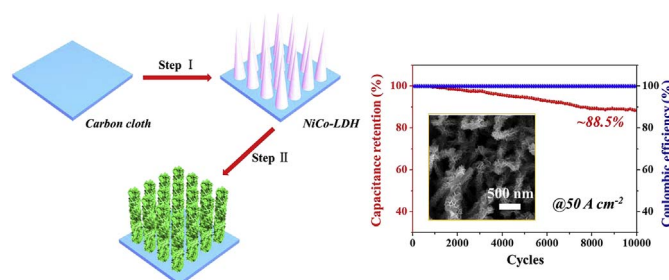
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HIGHLIGHTS

- A NiCo-LDH@NiOOH core-shell heterostructure is fabricated via a two-step method.
- The electrode shows an ultrahigh specific capacitance and excellent cycling performance.
- The assembled asymmetric supercapacitor exhibits a high energy density.

GRAPHICAL ABSTRACT



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ABSTRACT

Constructing rational structure and utilizing distinctive components are two important keys to promote the development of high performance supercapacitor. Herein, we adopt a facile two-step method to develop an in-situ heterostructure with NiCo-LDH nanowire as core and NiOOH nanosheets as shell on carbon fiber cloth. The resultant NiCo-LDH@NiOOH electrode exhibits a high specific capacitance of about 2622 F g^{-1} at 1 A g^{-1} and good cycling stability (88.5% remain after 10000 cycles). This reinforced electrochemical performance is benefit from the distinct core-shell structure, and takes advantage of the synergetic effect to supply more electrochemical active spots and pathways to accelerate electron and ion transport. Furthermore, the fabricated asymmetric supercapacitor of optimized NiCo-LDH@NiOOH//AC device displays a high energy density of 51.7 Wh kg^{-1} while the power density is 599 W kg^{-1} and presents a satisfying cycling performance.

1. Introduction

With the rapid growth of energy consumption in the world, high-performance energy-storage devices have attracted more and more attention. Among these energy-storage devices, supercapacitors have become a hotspot due to the higher power density and longer cycle lifespan than conventional batteries recently. Supercapacitors, according to the energy-storage mechanism, can be classified as

electrochemical double layer capacitors (EDLCs) and pseudocapacitors [1–5]. The contribution of pseudocapacitance is much higher than that of EDLCs, because of the dominated Faradaic redox reactions provided by the active materials [6]. Usually, the pseudocapacitive electrode is made up of transition metal oxides (TMOs), and there are many relevant researches. For instance, Wei et al. develop a mesoporous hybrid $\text{NiO}_x\text{-MnO}_x$ electrode, which delivers a specific capacitance of 1218 F g^{-1} and good cycling performance of 2.9% loss [7]. Kim's group

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reports a ZnO/MnO₂ core-shell electrode and fabricates the planar supercapacitor. The planar supercapacitor shows an excellent electrochemical with comparable long term cyclic stability [8]. Pang synthesizes porous NiO nanospindles by calcination and the resultant material has an excellent performance [9]. Raj's group synthesizes NiCo₂O₄ aggregates and the power density could be up to 10.6 kW kg⁻¹ [10]. Other TMOs are also reported such as V₂O₅ and CuO [11,12]. However, the low energy density of TMOs has limited their electrochemical performance [13]. Hence, finding another substitute and optimizing the structure of electrode are two urgent issues to overcome this difficulty.

Recently, transition metal layered double hydroxides (LDHs) have aroused more interests for the application in supercapacitors due to their superior anion exchanging, tunable chemical composition, high redox activity and intercalating capability [14,15]. Many researches have reported to enhance the electrochemical performance of LDHs. Zhang et al. [16] have reported a 3D hierarchical NiAl-LDH electrode through a liquid-phase deposition method. The obtained electrode shows good electrochemical performance. Jagdale et al. [17] take a facile electrodeposition method to gain CoMn-LDH with an excellent reversibility. NiMn-LDH [18] and CoAl-LDH [15] are also investigated and proved to be promising candidate for application. Particularly, NiCo-LDH has been a focus material in the study of supercapacitor electrodes owing to its rich oxidation states, environmental friendliness, satisfactory stability, and rapid reactions caused by the effective diffusion of electrolyte [19–21]. However, LDHs still have some weaknesses that have limited the sufficient utilization, e.g. they are unable to reach the theoretical capacity [22]. To break this bottleneck, enormous efforts have been devoted. The rational design of structure is the most considerable and efficient way. Particularly, the core-shell heterostructures have been proved to be an efficient approach to complete the challenge by combining the unique properties of different pseudocapacitive materials [23,24]. In most cases, one-dimensional material as core, such as nanowires and nanorods, can provide an efficient path for electron transport. Meanwhile, two-dimensional nanosheets acting as shell can ensure larger surface areas and more electro-active sites [25–29]. Plenty of composite structures have been designed, which have been reported to present excellent electrochemical performance. Jun's group reports a facile strategy to produce MnCo-LDH@Ni(OH)₂ core-shell heterostructure with ultrahigh specific capacitance [22]. Li designs a NiO@FeCo-LDH electrode, which has enhanced areal capacitance and rate capability. The constructed ASC displays ultrahigh energy and power density [30]. Among several candidates to composite with LDHs, the nickel hydroxides may be the ideal one, because their actual capacity is very close to theoretical one, and they have good cycling stability [31,32]. Moreover, in-situ growing corresponding (hydro) oxides is the simplest and effective strategy. In addition, the electrode materials directly grown on the substrates can reduce the influence of organic binders on the capacity performance for supercapacitors. The carbon fiber substrate seems to be a promising approach, due to its, low cost, high conductivity, and electrochemically stable [33]. Jun et al. [34] synthesizes amorphous MoS_x thin-film-coated carbon fiber paper, which shows extremely small resistance. Yu's group deposits bare PEDOT on flexible 3D carbon fiber cloth. The symmetric supercapacitor exhibits significantly power density of 40.25 kW kg⁻¹ and delivers long device stability with capacitance retention of ~86% after 12000 cycles [35].

Herein, we report a facile strategy to synthesize NiCo-LDH@NiOOH core-shell heterostructure grown on carbon fiber cloth, where NiCo-LDH NWs acted as core and two-dimensional NiOOH acted as shell. Because of the synergistic effect of multiple components, the as-fabricated electrode showed outstanding electrochemical performance. The resultant NiCo-LDH@NiOOH electrode showed a high specific capacitance. Furthermore, an asymmetric supercapacitor was assembled, using NiCo-LDH@NiOOH and activated carbon (AC) as positive and negative electrode individually. The supercapacitor showed a high energy density of 51.7 Wh kg⁻¹ at a power density of 599 W kg⁻¹.

2. Experimental section

2.1. Synthesis methods

2.1.1. Synthesis of NiCo-LDH nanowire arrays (NiCo-LDH NWAs)

Typically, the synthesis of NiCo-LDH grown on carbon fiber cloth was carried out following the hydrothermal method. 1.4 g CoCl₂·6H₂O, 0.7 g NiCl₂·6H₂O, and 1.1 g urea were dissolved in 60 mL of deionized water (DIW). A piece of carbon fiber cloth (CFC, 2 × 5 cm) was sonicated and rinsed with 10% HCl solution, ethanol and deionized water to remove the impurities. The carbon fiber cloth was immersed into the as-prepared solution. The homogeneous solution was transferred into a 100 mL Teflon-lined stainless-steel autoclave, and kept at 120 °C for 8 h. The obtained product was rinsed with deionized water and ethanol for three times. The sample was dried at 60 °C under vacuum condition for further use.

2.1.2. Synthesis of NiCo-LDH@NiOOH core-shell heterostructure

The NiCo-LDH@NiOOH core-shell heterostructure was synthesized via chemical bath deposition. To gain NiOOH on NiCo-LDH nanowire, 26.3 g NiSO₄·6H₂O and 5.4 g K₂S₂O₈ were dissolved in 180 mL DIW under strong stirring to form a homogeneous solution. The 20 mL of aqueous ammonia was added into the solution. With a continuous stirring for another 10 min, the NiCo-LDH/CFC was immersed into the aqueous solution for 20 min. The as-prepared sample was dried at 60 °C for 12 h. The mass loading is about 5 mg cm⁻². NiOOH nanosheets directly grown on carbon fiber cloth were prepared under the same method.

2.2. Material characterization

The crystal structures of the materials were characterized by X-ray diffraction (D/max 2550). The morphologies and microstructures were observed by scanning electron microscope (HELIOS NanoLab 600i) and transmission electron microscope (Tecnai G2 F30). The chemical properties of the materials were studied with X-ray photoelectron spectrometer (ESCLAB 250Xi).

2.3. Electrochemical measurements

The electrochemical properties of synthesized materials were studied using three-electrode cells, where Pt was used as the counter and Hg/HgO as reference electrodes. 6.0 M KOH solution was used as the electrolyte. Cyclic voltammetry (CV) and galvanostatic charge/discharge (GCD) curves were conducted on an electrochemical workstation (CHI760E). Electrochemical impedance spectroscopy (EIS) analysis was conducted using the electrochemical workstation (PARSTAT 4000A), ranging from 100 kHz to 0.01 Hz with amplitude of 5 mV.

2.4. Assembly and electrochemical measurements of the asymmetric supercapacitor

The carbon electrode was made of active carbon, carbon black and PVDF with a mass ratio of 8:1:1 in N-methylpyrrolidinone (NMP). The mixture was coated on carbon fiber cloth uniformly, and then was dried at 120 °C for 3 h before used. The mass loading of both electrodes was following equation (1). The corresponding power density (P) and energy density (E) were calculated according to equations (2) and (3).

$$m_+/m_- = \Delta V_- C_- / \Delta V_+ C_+ \quad (1)$$

$$E = \int I \times V(t) dt / (3.6 m) \quad (2)$$

$$P = 3600 \times E / \Delta t \quad (3)$$

Where C represents specific capacitance (F g⁻¹), ΔV represents the

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