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High-performance hybrid supercapacitor with 3D hierarchical porous flower-like layered double hydroxide grown on nickel foam as binderfree electrode



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HIGHLIGHTS

- Layered double hydroxide is grown on nickel foam by liquid phase deposition method.
- The electrode shows excellent performance at high current density (50 A g^{-1}).
- The assembled hybrid supercapacitor exhibits good electrochemical performance.

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GRAPHICAL ABSTRACT



ABSTRACT

The synthesis of layered double hydroxide (LDH) as electroactive material has been well reported; however, fabricating an LDH electrode with excellent electrochemical performance at high current density remains a challenge. In this paper, we report a 3D hierarchical porous flower-like NiAl-LDH grown on nickel foam (NF) through a liquid-phase deposition method as a high-performance binder-free electrode for energy storage. With large ion-accessible surface area as well as efficient electron and ion transport pathways, the prepared LDH-NF electrode achieves high specific capacity (1250 C g⁻¹ at 2 A g⁻¹ and 401 C g⁻¹ at 50 A g⁻¹) after 5000 cycles of activation at 20 A g⁻¹ and high cycling stability (76.7% retention after another 5000 cycles at 50 A g⁻¹), which is higher than those of most previously reported NiAl-LDH-based materials. Moreover, a hybrid supercapacitor with LDH-NF as the positive electrode and porous graphene nanosheet coated on NF (GNS-NF) as the negative electrode, delivers high energy density (30.2 Wh kg⁻¹ at a power density of 800 W kg⁻¹) and long cycle life, which outperforms the other devices reported in the literature. This study shows that the prepared LDH-NF electrode offers great potential in energy storage device applications.

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1. Introduction

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The continuous increase in application of high-powered electric devices, depletion of fossil fuels, and intensifying global warming have motivated researchers to develop an advanced energy storage system with high energy density and high power [1-3]. As a rapidly rising class of energy storage devices, supercapacitors (SCs) have



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gained significant attention in the past decades because of their rapid charge and discharge rate, high power density, long lifespan, high reliability, and low maintenance cost [4,5]. In particular, because of the near surface redox reactions for charge storage, pseudocapacitive electrode materials can achieve much higher charge storage than electrochemical double-layer capacitors [6]. However, the disadvantages of pseudocapacitive materials, such as high cost of RuO₂ [7,8], low conductivity of MnO₂ [9,10], and low cycling stability of conductive polymers [11,12], causes unsatisfactory pseudocapacitor performance. Therefore, charge storage devices with high power and high energy densities are still urgently needed.

Layered double hydroxide (LDH) with a general formula of [M^{II}₁₋ $_{x}M^{III}_{x}(OH)_{2}]^{x+}[A^{n-}]_{x/n} \cdot mH_{2}O(M^{II} \text{ and } M^{III}, \text{ the layers of divalent and})$ trivalent metal cations, respectively; A^{n-} , the charge-balancing anion of valence n; $x = M^{III}/(M^{II} + M^{III}))$ has been extensively studied as promising positive electrode materials because of their relatively low cost, high redox activity, and environmentally friendly features [13,14]. Besides the electrical double-layer capacitance acquired by their abundant slabs, in particular, several Faradaic reactions of electrochemical active sites could occur on the surface or near the surface range for charge storage, leading to high energy storage and simultaneously maintaining a powerful performance comparable to that of supercapacitive electrode materials [13,14]. However, because of the high similarity of LDH-based battery-type materials to pseudocapacitive materials in terms of charge storage mechanism and high power performance. these types of electrode materials were once mistaken for pseudocapacitive materials [13,14].

From the future application viewpoint, developing advanced high-performance SCs with higher energy density without sacrificing power density and cycling stability is urgently needed [15]. However, low-rate performance and poor cycle life are the current limitations of utilizing LDH materials in next-generation hybrid SCs [16]. In most cases, the current density during the charge-discharge tests could not exceed 10 A g⁻¹ [17–19], while the specific capacity dropped dramatically at higher current densities, such as 18 A g⁻¹ [20]. In addition, restricted by the morphology of LDH-based materials reported in the literature, the long-term cycling stability of the electrodes were generally evaluated under 20 A g⁻¹ for no more than 2000 cycles [21–23], which was unsuitable for the high-rate performance application [24].

The intrinsically poor conducting nature of LDH materials is the main cause of their poor performance at high current densities [25]. For powdery LDH materials [13,14], a conductive agent and a polymer binder are commonly used during the preparation of SC electrodes, which not only increases contact resistance of the electrodes but also inevitably reduces the overall energy storage capacity and performance [26]. One effective method is to grow LDH directly on a conductive substrate to form a binder-free electrode with 3D interconnected network of LDH materials, allowing electrons and ions efficient transport during Faradaic redox reactions [22,27]. Nickel foam (NF) with a desirable 3D macro-porous structure, high specific surface area, and excellent physical strength, can be a promising substrate in LDH-based SCs [28]. For example, Pu et al. fabricated NiCo-LDH nanosheets on NF through a hydrothermal process [29]. The specific capacitance of the electrode was 1734 Fg^{-1} at 6 Ag^{-1} and 1146 Fg^{-1} at 30 Ag^{-1} . However, life stability was investigated only within 1000 cycles at 18 A g^{-1} with retention of 86%. To achieve improved cycling performance at high current density (up to 50 A g^{-1}), a new and facile growth strategy of porous LDH structure with excellent electrochemical performance on NF needs to be developed. Therefore, we report a simple and convenient strategy to deposit 3D hierarchical porous flower-like NiAl-LDH nanosheets on NF using the liquid-phase deposition (LPD) method.

The LPD method, originally developed as a route to synthesize oxide films, was first reported by Kawahara and Honda [30], and has been used to prepare various types of metal oxides [31] and hydroxide thin films [32] on planar surfaces. Thin films fabricated by the LPD method have a dense structure, good processability, and durability under continuous use [14,33]. In this study, this new and facile LPD method was developed for effective growth of 3D hierarchical porous flower-like NiAl-LDH nanosheets on 3D NF with high stability as the binder-free electrode, which displays high rate capability (1250.7 C g^{-1} at 2 A g^{-1} and 401 C g^{-1} at 50 A g^{-1}) after 5000 cycles of activation at 20 A g^{-1} and high cycling stability (76% retention after another 5000 cycles at 50 A g^{-1}). Moreover, to further evaluate the LDH-NF electrode for practical applications, a hybrid supercapacitor (HSC) was fabricated using LDH-NF as the positive electrode and porous graphene nanosheets coated on NF (GNS-NF) as the negative electrode. Compared with symmetric SCs, the HSC devices based on NF substrates (e.g., Co₃O₄@MnO₂-NF// MEGO [34] and Ni(OH)₂/MnO₂ on NF//rGO [35]) can extend the operating voltage window and achieve higher energy and power densities [36]. This study shows that our prepared HSC device exhibited high energy density (30.2 Wh kg⁻¹ at a power density of 800 W $kg^{-1})$ and long cycling stability (15000 cycles) with 70% retention at 10 A g⁻¹, which outperformed the reported devices in the literature [37,38].

2. Experimental section

2.1. Materials preparation

All chemicals were of analytical grade and were used as received without any further treatment.

2.1.1. Preparation of Ni parent solution

0.11 mol of Ni(NO₃)₂· $6H_2O$ was dissolved in 200 mL of water with vigorous stirring, and pH was adjusted to 7.5 with a certain amount of aqueous ammonia (28 vol%). The collected precipitate was repeatedly washed with water and dried at ambient temperature and then poured into 40 mL of 0.66 M NH₄F for 48 h under continuous magnetic stirring at ambient temperature. The dispersion was filtered to obtain the Ni parent solution.

2.1.2. Preparation of LDH-NF electrode by LPD method

Typically, a piece of NF is pretreated successfully with 6.0 M HCl. deionized water, and absolute ethanol, each for 15 min, to ensure a clean surface. The clean NF (10 \times 10 \times 1 mm, 110 PPI, 31 mg cm⁻²) was then immersed in a 100-mL Teflon autoclave with homogeneous solution of the freshly prepared Ni parent solution, 20 mL of 0.5 M H₃BO₃, 5 mL of 0.05 M Al(NO₃)₃ \cdot 9H₂O, and 5 mL of deionized water: the autoclave was heated in an oven at 120 °C for 10 h to allow the growth of NiAl-LDH on NF. The NF substrate, covered with NiAl-LDH, was washed with deionized water and ethanol to remove surface ions and molecules by using an ultrasonic bath cleaner, and then dried at 50 °C for 24 h to remove the adsorbed solvents. The obtained NF-based material was denoted as LDH-NF. The mass of NiAl-LDH on NF was determined by subtracting the weight before deposition from the weight after deposition. NiAl-LDH loading was approximately 1.8 mg for all electrodes. Meanwhile, the green precipitate in the autoclave was filtered, washed with deionized water and ethanol, and then dried at 50 °C for 24 h. The obtained powder was labeled as LDH.

2.1.3. Preparation of LDH-NF-H electrode by conventional hydrothermal method

A total of 3 mmol of Ni(NO₃)₂·6H₂O and 1 mmol of

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