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Fabrication of Hierarchical NiMoO₄/NiMoO₄ Nanoflowers on Highly Conductive Flexible Nickel Foam Substrate as a Capacitive Electrode Material for Supercapacitors with Enhanced Electrochemical Performance

Anil Kumar Yedluri^D, Tarugu Anitha and Hee-Je Kim *

School of Electrical Engineering, Pusan National University, Busandaehak-ro 63beon-gil, Geumjeong-gu, Busan 46241, Korea; yedluri.anil@pusan.ac.kr (A.K.Y.); anitha.tarugu@gmail.com (T.A.) * Correspondence: heeje@pusan.ac.kr; Tel.: +82-10-2295-0613; Fax: +82-51-513-0212

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Abstract: Hierarchical NiMoO₄/NiMoO₄ nanoflowers were fabricated on highly conductive flexible nickel foam (NF) substrates using a facile hydrothermal method to achieve rapid charge-discharge ability, high energy density, long cycling lifespan, and higher flexibility for high-performance supercapacitor electrode materials. The synthesized composite electrode material, NF/NiMoO₄/NiMoO₄ with a nanoball-like NF/NiMoO₄ structure on a NiMoO₄ surface over a NF substrate, formed a three-dimensional interconnected porous network for high-performance electrodes. The novel NF/NiMoO₄/NiMoO₄ nanoflowers not only enhanced the large surface area and increased the electrochemical activity, but also provided an enhanced rapid ion diffusion path and reduced the charge transfer resistance of the entire electrode effectively. The NF/NiMoO₄/NiMoO₄ composite exhibited significantly improved supercapacitor performance in terms of a sustained cycling life, high specific capacitance, rapid charge-discharge capability, high energy density, and good rate capability. Electrochemical analysis of the NF/NiMoO₄/NiMoO₄ nanoflowers fabricated on the NF substrate revealed ultra-high electrochemical performance with a high specific capacitance of 2121 F g^{-1} at 12 mA g^{-1} in a 3 M KOH electrolyte and 98.7% capacitance retention after 3000 cycles at 14 mA g⁻¹. This performance was superior to the NF/NiMoO₄ nanoball electrode (1672 F g⁻¹ at 12 mA g^{-1} and capacitance retention 93.4% cycles). Most importantly, the SC (NF/NiMoO₄/NiMoO₄) device displayed a maximum energy density of $47.13 \text{ W} \text{ h kg}^{-1}$, which was significantly higher than that of NF/NiMoO₄ (37.1 W h kg⁻¹). Overall, the NF/NiMoO₄/NiMoO₄ composite is a suitable material for supercapacitor applications.

Keywords: supercapacitor; hierarchical nanostructures; NiMoO₄/NiMoO₄; NiMoO₄; flexible supercapacitor electrode; excellent cycling stability

1. Introduction

In recent years, novel energy sources have become a major challenge for global sustainable development in the 21st century due to the exhaustion of fossil fuels and environmental pollution [1–4]. The concerns regarding global warming caused by the use of non-renewable fossil fuels have increased considerably [5]. Over the past decade, serious efforts have been made to exploit renewable energy, such as biofuels, wind power, and tidal sources, with the intention of partially replacing fossil fuels [6–8]. In this regard, considering the various energy storage devices available, the most promising and fast emerging storage device is the supercapacitor (SC) owing to its high energy and power density, rapid



charge-discharge capability, high flexibility, and sustained cycle life [9,10]. SCs can provide a clean and efficient emerging storage technology to help solve the global and environmental energy crises [11]. Generally, energy storage in SCs takes place via two methods: electrochemical double layer capacitors (EDLCs) and pseudocapacitors (PCs or redox capacitors) [12,13]. In EDLCs, the capacitance is due to ion adsorption and desorption at the surface of the carbon electrode and redox electrolyte, whereas in PCs, the energy is stored in reversible faradaic redox reactions taking place at the electrolyte-electrode interface [14]. PCs generally have a very high energy density and specific capacitance compared to EDLCs, owing to their excellent conductivity and electrochemical properties [15,16].

Of the different types of redox-active materials, ternary transition metal oxides (TMOs) are used widely as PC electrode materials because of their various oxidation states for faradaic redox reactions and high theoretical capacitance. Currently, ternary transition metal oxides, such as NiCo₂O₄, CoMoO₄, MnMoO₄, ZnCo₂O₄, MnCo₂O₄, CuCo₂O₄, and NiMoO₄, have been studied extensively as new types of energy storage materials owing to their excellent electrochemical performance and better conductivity [17–23]. Recently, few studies have examined the design of multicomponent nanostructured materials for SC applications that show excellent energy storage behavior. Lou et al. prepared NiCo₂O₄ structures exhibiting a nanorod and nanosheet morphology using a facile solution method, which delivered a specific capacitance (Cs) of 905 and 887.7 F g^{-1} at 2 A g⁻¹ [24]. Manickam et al. used a solution combustion synthesis approach to develop α -NiMoO₄ nanoparticles, exhibiting a high Cs of 1517 F g^{-1} at 1.2 A g^{-1} [25]. Furthermore, Zhang et al. reported a nanowire array of NiCo2O4 using a simple hydrothermal technique for SC applications, showing a super high Cs of 1283 F g^{-1} at 1 A g^{-1} [26]. On the other hand, combining two types of TMOs to form a unique nanostructure is an efficient way to improve the EC performance further. For example, Mai et al. synthesized MnMoO₄-CoMoO₄ nanowire structures using simple refluxing technology and achieved a high Cs of 181 F g^{-1} at 1 A g^{-1} [27]. Wang et al. used a solution method to synthesize NiCo₂O₄@MnMoO₄ nanocolumn arrays over a Ni foam substrate, exhibiting a high Cs of 1705.3 F g^{-1} at 5 mA cm⁻² [28]. Recently, Cheng et al. reported hierarchical core/shell NiCo₂O₄@NiCo₂O₄ nanocactus arrays for SCs applications, showing a very high Cs of 1264 F g^{-1} at 2 A g^{-1} [29].

Among the various TMOs available, nickel molybdate (NiMoO₄) has attracted enormous attention owing to its outstanding electrochemical conductivity, high theoretical capacity, cost-effectivity, rapid redox activity, low cost, earth abundance, and non-toxicity [30–35]. Until now, very few studies have evaluated NiMoO₄/NiMoO₄ as an electrode material for SC applications. The main challenge for potential real-time applications of SCs is to achieve high energy density, higher specific capacitance, sustained cycle life, rate capability, high flexibility, and fast charge-discharge capability [36]. Therefore, the development of a simple technique to fabricate nanostructured NiMoO₄/NiMoO₄ composite electrode materials with unique morphologies and high energy storage performance will be of great significance.

Based on the above ideas, an aligned compact of a NF/NiMoO₄ electrode and a NF/NiMoO₄/ NiMoO₄ composite on a nickel foam substrate were synthesized using a simple hydrothermal approach to produce highly efficient electrode materials. The use of nickel foam as a current collector increases the connection between NiMoO₄/NiMoO₄ nanostructures with the collectors. In addition, nickel foam (NF) also improves the conductivity, flexibility, and electron/ion transportation. This hierarchical NF/NiMoO₄/NiMoO₄ composite allowed the migration of electrolyte ions into the inner regions of the electrode, which could reduce the resistance of the electrolyte. The electrochemical behaviors of the hierarchical structured NF/NiMoO₄/NiMoO₄ composite electrode were investigated in a three-electrode system using a 3 M KOH aqueous electrolyte by cyclic voltammetry (CV), galvanostatic charge-discharge (GCD), and electrochemical impedance spectroscopy (EIS). The NF/NiMoO₄/NiMoO₄ composite, as the active electrode material, exhibited a high specific capacitance of 2121 F g⁻¹ at a current density of 12 mA g⁻¹, and excellent cycling stability (98.7% capacitance retention after 3000 cycles). The high performance observed was attributed to the synergism between NF/NiMoO₄ and NF/NiMoO₄. To the best of the authors' knowledge, the outstanding performance of the NF/NiMoO₄/NiMoO₄ composite was superior to that of the NF/NiMoO₄ electrode material, making it a promising candidate as a SCs material.

2. Experimental Methods

2.1. Chemicals and Materials

Sodium molybdate dihydrate [Na₂MoO₄.2H₂O], nickel nitrate hexahydrate [Ni(NO₃)₂.6H₂O], thiourea [CH₄N₂S], ammonium fluoride [NH₄F], potassium hydroxide [KOH], and Ni foam were obtained from Sigma-Aldrich Co., South Korea. Deionized (DI) water was used throughout the study. All chemicals used for the synthesis of the NF/NiMoO₄ nanoball structures and NF/NiMoO₄/NiMoO₄ nanoflower-like structures were of analytical grade and used as received.

2.2. Preparation of the NF/NiMoO₄ Nanoball Electrode on a Ni Foam Substrate

The composite NF/NiMoO₄/NiMoO₄ nanoflower electrode material was grown on Ni foam using a facile hydrothermal reaction. Before the synthesis process, the Ni foam $(1.5 \times 2 \text{ cm}^2)$ was treated with 3 M HCl for 40 min and were then cleaned with deionized (DI) water and ethanol for 30 min each. The host precursor materials, such as 0.03 mol of [Ni(NO₃)₂.6H₂O] and 0.06 mol of [Na₂MoO₄.2H₂O], were dissolved in 100 mL of distilled water and were stirred for 1 h. Subsequently, 0.4 mol thiourea [CH₄N₂S] and 0.04 mol ammonium fluoride [NH₄F] were added to the above mixture whilst stirring. The mixture was continuously stirred for 1 h until complete dissolution. The above solution and a piece of Ni foam were transferred to a Teflon-lined stainless steel autoclave. The hydrothermal reaction system was conducted at 140 °C for 12 h. After cooling to room temperature, the NF/NiMoO₄-loaded Ni foam electrode was cleaned with DI water and ethanol, and dried overnight in a vacuum oven at 100 °C. The collected NF/NiMoO₄ foams were then annealed at 200 °C for 5 h to obtain the final composite material.

2.3. Preparation of the NF/NiMoO₄/NiMoO₄ Nanoflower Composite Electrode on a Ni Foam Substrate

A secondary hydrothermal reaction was adopted; NF/NiMoO₄ was used as the scaffold on the nickel foam for further growth of the NF/NiMoO₄/NiMoO₄ structure using a similar process. Typically, 0.03 mol of [Ni(NO₃)₂.6H₂O] and 0.06 mol of [Na₂MoO₄.2H₂O] were dissolved in 100 mL of DI water with magnetic stirring for 20 min. The above solution was then transferred to a Teflon-lined stainless steel autoclave and the NF/NiMoO₄-deposited nickel foams were placed into the reaction mixture and maintained at 120 °C for 8 h. The autoclave was cooled naturally to room temperature and the as-obtained NF/NiMoO₄/NiMoO₄ composite material was washed with DI water and ethanol, and finally dried overnight at 80 °C. Finally, the samples were annealed in air at 250 °C for 5 h. The mass loading of the NF/NiMoO₄ and NF/NiMoO₄/NiMoO₄ nanostructures were around 7.0 and 10 mg cm⁻², respectively.

2.4. Characterizations of Nanoballs and Nanoflowers

All samples were examined by field emission scanning electron microscopy (FE-SEM, S-4800, Hitachi) at the Busan KBSI, high-resolution transmission electron microscopy (HRTEM, Jem 2011, Jeol corp. Tokyo, Japan), high resolution X-ray diffraction (XRD, Bruker D8 Advance, Tokyo, Japan) using Cu Kα operated at 40 kV and 30 mA, and X-ray photoelectron spectroscopy (XPS, VG scientific ESCALAB 250 system, Hitachi manufacturer, Tokyo, Japan).

2.5. Electrochemical Measurements

Electrochemical characterization of the as-prepared (NF/NiMoO₄ and NF/NiMoO₄/NiMoO₄) electrodes, such as CV, GCD measurements, and EIS, was carried out in a three-electrode configuration using a Biologic-SP150 workstation. A three-electrode glass cell setup was used in the experiment. The prepared samples, platinum wire, and saturated Ag/AgCl were used as the working, counter, and

reference electrodes, respectively, with a 3.0 M KOH aqueous solution as the supporting electrolyte. A charge-discharge study was carried out galvanostatically at 12–18 mA g⁻¹ from 0 to 0.4 V vs. Ag/AgCl and CV was recorded in the potential window of -0.2 to 0.5 V vs. Ag/AgCl by varying the scan rate from 1 to 30 mV s⁻¹. The specific capacitance, energy density and power density were calculated from the GCD curves at different current densities according to the following Equations (1)–(3) [37]:

$$C_s = \frac{I \times \Delta t}{m \times \Delta V} \tag{1}$$

$$E = \frac{C_s \times (\Delta V^2)}{2} \tag{2}$$

$$P = \frac{E}{\Delta t} \tag{3}$$

where *I* (A) represents current density; the potential range is depicted as ΔV . *m* (g) denotes the active material mass and discharge time is depicted as Δt (s) [38].

3. Results and Discussion

Preparation, Morphology, and Characterization of NF/NiMoO₄ and NF/NiMoO₄/NiMoO₄

The surface morphologies of the synthesized NF/NiMoO₄ and NF/NiMoO₄/NiMoO₄ were examined by FESEM. Figure 1a–f presents the FESEM images (lower and higher magnifications) of NF/NiMoO₄ and NF/NiMoO₄/NiMoO₄ synthesized on a nickel foam substrate using a facile hydrothermal method. Figure 1a–c shows SEM images of NF/NiMoO₄, revealing the formation of ball-like structures with rough NiMoO₄ nanostructures on the NF surface. The image revealed a large number of voids among the balls that were not distributed uniformly over the NiMoO₄ nanostructure on NF (Figure 1b), which is not a beneficial surface for electrochemical reactions (Figure 1c). In contrast, the FE-SEM image of NF/NiMoO₄/NiMoO₄ nanoflowers showed a three-dimensional interconnected, porous, and roughened surface structure without major voids, where ultrathin nanoballs of NF/NiMoO₄ were grown and were distributed uniformly over the surface of the NiMoO₄ nanostructure (Figure 1d,f). The three-dimensional nanostructures interconnected with each other to form a highly porous structure that was expected to enhance the surface area, increase the electrochemical conductivity of the working materials and facilitate rapid transportation between the active substance and electrolyte ion in supercapacitor electrodes.



Figure 1. Morphological characterization of Nickel foam NF/NiMoO₄ and NF/NiMoO₄/NiMoO₄. FE-SEM images of the prepared (**a**–**c**) NF/NiMoO₄ and (**d**–**f**) of NF/NiMoO₄/NiMoO₄ and their corresponding high magnification images.

Figure 2a,b shows different magnification HR-TEM and TEM images of NF/NiMoO₄, displaying similar structures, which are in good agreement with FE-SEM analysis. Figure 1b presents a TEM image with dark strips with a crumpled silk-like structure. The crystalline lattice fringe had a spacing of 0.31 nm and was assigned to the (220) plane of NiMoO₄. EDX analysis of NF/NiMoO₄ (Figure 2c) revealed the presence of Ni, Mo, and O elements without impurities. The atomic percentage of elements of Mo, Ni, O and C are obtained to be 13.34%, 44.30%, 37.51% and 4.82%, respectively. On the other hand, TEM images of the NF/NiMoO₄ nanoballs in the NF/NiMoO₄/NiMoO₄ electrode revealed a lattice fringe spacing of 0.352 nm, which was assigned to the (111) plane of NiMoO₄, as shown in Figure 2d,e. The NF/NiMoO₄/NiMoO₄ nanoflowers were comprised mainly of Ni, Mo, and O (Figure 2f) arising from the nickel foam, which confirmed the successful fabrication of NF/NiMoO₄/NiMoO₄. The atomic percentage of elements of Mo, Ni, O and C were obtained as 29.44%, 34.27%, 37.48% and 3.78%, respectively.



Figure 2. (**a**,**b**) HR-TEM and TEM images and (**c**) EDX pattern of NF/NiMoO₄ on nickel foam. (**d**,**e**) TEM images and (**f**) Energy-dispersive X-ray spectroscopy (EDX) pattern of NF/NiMoO₄/NiMoO₄ nanostructure on nickel foam.

The phase purity and crystal nature of the NF/NiMoO₄ and NF/NiMoO₄/NiMoO₄ electrodes were analyzed further by XRD. Both samples exhibited XRD peaks at 2θ angle of 14.3° , 25.3° , 28.8° , 32.6° and 43.9° 2θ (Figure 3), which were assigned to (1 1 0), (1 1 2), (2 2 0), (0 2 2), and (3 3 0) diffraction planes of the monoclinic phase of NiMoO₄ (JCPDS NO. 33-0948) [39]. Some other XRD peaks from the nickel foam substrate were observed. Overall, the XRD data concurred with the TEM observations.



Figure 3. XRD pattern of the fabricated NF/NiMoO₄ electrode and NF/NiMoO₄/NiMoO₄ composite electrode grown on NF.

The chemical compositions and oxidation states of the elements of NF/NiMoO₄/NiMoO₄ were analyzed further by XPS analysis and the results are shown in Figure 4. The survey spectrum of NF/NiMoO₄/NiMoO₄ revealed the presence of Mo 3d, Ni 2p, and O 1s. The XPS spectra of Mo 3d (Figure 4b) showed two major peaks centered at binding energies of 232 and 235 eV with a spin energy separation of 3 eV, corresponding to Mo $3d_{5/2}$ and Mo $3d_{3/2}$, respectively [40]. De-convolution of the Ni 2p spectrum (Figure 4b) revealed two prominent Ni $2P_{3/2}$ and Ni $2P_{1/2}$ spin-orbit peaks at 855.3 and 872.6 eV respectively; each were fitted with spin-orbit doublet characteristic of the Ni³⁺ and Ni²⁺ binding states [41]. Figure 4c presents the de-convolution of the O 1s spectra showing the oxygen contributions at 530.9 eV and 532 eV, which were assigned to s O₁ and O₂ associated with typical metal oxygen bonds [42,43].



Figure 4. XPS survey spectrum (**a**), Mo 3d (**b**), Ni 2p (**c**) and O 1s (**d**) of the NF/NiMoO₄/NiMoO₄ composite material.

The electrochemical performance of the as-obtained samples of NF/NiMoO₄ and NF/NiMoO₄/NiMoO₄ were examined by CV and GCD in a three-electrode cell configuration with a 3 M KOH aqueous electrolyte. Figure 5a presents the typical CV curves of the NF/NiMoO₄ and NF/NiMoO₄/NiMoO₄ electrodes at a scan rate of 5 mV s⁻¹ in the potential window of -0.2 to 0.5 V (vs. saturated calomel electrode (SCE)). Figure 5b,c presents the typical CV curves of the NF/NiMoO₄/NiMoO₄ and NF/NiMoO₄ material on nickel foam substrates at various scan rates from 1 to 30 mV s⁻¹ over the potential range of -0.2 to 0.5 V. All CV curves revealed a well-defined pair of redox peaks, which could be attributed to a Faradic redox reaction. The CV integrated area, oxidation, and reduction currents of the NF/NiMoO₄/NiMoO₄ electrode were much higher than those of the NF/NiMoO₄. Moreover, as the scan rate was increased from 1 to 30 mV s⁻¹ for NF/NiMoO₄, the cathodic and anodic peak positions shifted to a lower and higher potential, showing superior electrochemical performance and higher specific capacitance than the individual components.



Figure 5. (a) Comparison of the CV curves of the NF/NiMoO₄ and NF/NiMoO₄/NiMoO₄ at the same scan rate (5 mV s⁻¹). (b,c) CV curves of the NF/NiMoO₄/NiMoO₄ electrode and NF/NiMoO₄ electrode at various scan rates. (d) GCD curves of the NF/NiMoO₄ and NF/NiMoO₄/NiMoO₄ electrodes at a current density of 12 mA g⁻¹. (e,f) Galvanostatic charge-discharge curves of the NF/NiMoO₄/NiMoO₄ and NF/NiMoO₄ and NF/NiMoO₄ electrodes at different current densities.

GCD was conducted to examine the high charge-storage behavior of NF/NiMoO₄/NiMoO₄ electrode compared to other electrode of NF/NiMoO₄. Figure 5d compares typical GCD plots of the NF/NiMoO₄ and NF/NiMoO₄/NiMoO₄ samples at 12 mA g⁻¹ in the potential window of 0 to 0.4 V; significant differences were observed between the electrode materials. The NF/NiMoO₄/NiMoO₄ composite displayed a larger peak area than NF/NiMoO₄ electrode, highlighting its superior performance, which is in agreement with the CV result. Figure 5e,f presents GCD plots of NF/NiMoO₄/NiMoO₄ and NF/NiMoO₄ at different current densities from 12 to 18 mA g⁻¹ in the potential window from 0 to 0.4 V. The GCD results were in good agreement with the CV data.

The gravimetric specific capacitance of the NF/NiMoO4 and NF/NiMoO4/NiMoO4 electrode materials were determined from the discharge curves in a three-electrode configuration using Equation (1), as shown in the Figure 6a. The specific capacitance of the NF/NiMoO₄/NiMoO₄ and NF/NiMoO₄ electrodes were 2121, 2038, 1942, and 1806 F g^{-1} and 1672, 1358, 1260, and 1092 F g^{-1} , respectively, at a current density of 12, 14, 16 and 18 mA g^{-1} . The specific capacitance of the NF/NiMoO₄/NiMoO₄ electrode was significantly higher than that of the NF/NiMoO₄ electrode. This was attributed to the larger effective surface area, which provided improved accessibility to the electrolyte, enabled the effective transport of ions and reduced the charge transfer resistance. Furthermore, the as-obtained specific capacitance results of the NF/NiMoO₄/NiMoO₄ composite electrode in the present study is superior to those reported for NiMoO₄-based materials, such as hierarchical NiCo₂S₄@NiMoO₄ core/shell nanospheres (1714 F g⁻¹ at 1 A g⁻¹) [44], Co₃O₄@NiMoO₄ nanosheets (1526 F g^{-1} at 3 mA cm $^{-2}$) [45], CoMoO_4-NiMoO_4.xH_2O bundles (1039 F g^{-1} at 2.5 mA cm⁻²) [46], Co₃O₄@NiMoO₄ nanowires (1230 F g⁻¹ at 10 mA cm⁻²) [47], NiMoO₄@CoMoO₄ hierarchical nanospheres (1601.6 F g⁻¹ at 2 A g⁻¹) [48], and core-shell NiCo₂O₄@NiMoO₄ nanowires $(1325.9 \text{ Fg}^{-1} \text{ 2 mA cm}^{-2})$ [49]. To further investigate the long-practical applicability of the NF/NiMoO₄ and NF/NiMoO₄/NiMoO₄ electrodes for supercapacitor applications, a cycling stability test was carried out in a 3 M KOH electrolyte at 14 mA g^{-1} for 3000 cycles, as shown in Figure 6b. After 3000 cycles, the NF/NiMoO₄/NiMoO₄ electrode maintained approximately 98.7% of its initial capacitance, whereas the NF/NiMoO₄ electrode exhibited 93.4% capacitance retention. These results show that the NF/NiMoO₄/NiMoO₄ composite has outstanding electrochemical stability.



Figure 6. (a) Calculated specific capacitance at different current densities, (b) Long-term cycling stability, (c) Nyquist plots and (d) Ragone plot of the NF/NiMoO₄ and NF/NiMoO₄/NiMoO₄ electrode materials.

Figure 6c presents Nyquist plots of the NF/NiMoO₄/NiMoO₄ and NF/NiMoO₄ electrodes analyzed from the equivalent circuit from 0.01 Hz to 100 kHz. EIS data contains four categories: solution resistance R_s, charge transfer resistance R_{ct}, a pseudocapacitive element C_p, and Warburg element W. The R_s can be estimated that the axis intercepts of the high frequency region in both active electrodes were almost similar. The R_{ct}, which distorted to be much smaller in the diameter of the semicircle arc, revealed that the NF/NiMoO₄/NiMoO₄ electrode consisted of lower interfacial charge-discharge impedance. In addition, the NF/NiMoO₄/NiMoO₄ electrode delivered a more vertical line, recommending the W which is not a determining factor and this type of sample can store charge more effectively. As expected, the R_{ct} of NF/NiMoO₄/NiMoO₄ (0.05 Ω) was lower than that of NF/NiMoO₄ (0.09 Ω), which further confirmed the excellent conductivity of the NF/NiMoO₄/NiMoO₄ composite electrode. Figure 6d shows a Ragone plot (energy density vs. power density) of the as-obtained samples NF/NiMoO₄ and NF/NiMoO₄/NiMoO₄. The energy density and power density were calculated using Equations (2) and (3), respectively. The NF/NiMoO₄/NiMoO₄ device exhibited a very high energy density of 47.13 W h kg⁻¹, which was much higher than that of NF/NiMoO₄ (37.1 W h kg⁻¹), revealing excellent energy storage performance.

4. Conclusions

NF/NiMoO₄ and NF/NiMoO₄/NiMoO₄ nanostructures were prepared on a nickel foam substrate using a facile hydrothermal approach and evaluated as electrode materials for supercapacitor applications. The morphological and structural properties of the hierarchical NF/NiMoO₄/NiMoO₄ electrode material provides more active sites and large electrochemical surface area to enhance accessibility to the electroactive sites that improved the energy storing and transportation of ions and electrons compared to the NF/NiMoO₄ electrode. The electrochemical studies measured in the 3 M KOH electrolyte showed that this hierarchical NF/NiMoO₄/NiMoO₄ nanocomposite delivered

superior electrochemical behavior with a maximum specific capacitance of 2121 F g⁻¹ at 12 mA g⁻¹ than the NF/NiMoO₄ electrode (1672 F g⁻¹ at 12 mA g⁻¹), as well as outstanding cycling stability (98.7% and 93.4% capacitance retention after 3000 cycles at 14 mA g⁻¹). The NF/NiMoO₄/NiMoO₄ device possessed a higher energy density (47.13 W h kg⁻¹) than NF/NiMoO₄ (37.1 W h kg⁻¹). These features make the NF/NiMoO₄/NiMoO₄ composite electrode a potential candidate for next generation flexible energy storage systems.

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