

Synthesis, structure, and electrochemical properties of the $\text{Ni(OH)}_2/\text{NiMoO}_4$ composite on the rGO/NF template for hybrid battery-supercapacitor electrodes

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Abstract

The growing demand for renewable and sustainable energy has intensified research on advanced energy-storage systems that can deliver both high energy and power densities. In this study, a $\text{Ni(OH)}_2/\text{NiMoO}_4$ composite on a reduced graphene oxide/nickel foam (rGO/NF) template was synthesized through a hydrothermal and electrodeposition route to construct a hybrid battery-supercapacitor electrode. The unique hierarchical design combines the pseudocapacitive activity of $\text{Ni(OH)}_2/\text{NiMoO}_4$ with the excellent conductivity of rGO and the high surface area of NF, enabling efficient electron and ion transport. Structural and morphological analyses confirm the successful formation of the $\text{Ni(OH)}_2/\text{NiMoO}_4/\text{rGO/NF}$ architecture. The electrode exhibits a specific capacitance (C_s) of $\sim 358 \text{ F}\cdot\text{g}^{-1}$ at $5 \text{ mV}\cdot\text{s}^{-1}$, an energy density of $\sim 24 \text{ Wh}\cdot\text{kg}^{-1}$, a power density of $\sim 626 \text{ W}\cdot\text{kg}^{-1}$, a conductivity of $1.30 \text{ S}\cdot\text{m}^{-1}$ (0.01 kHz), and retains $\sim 86\%$ capacitance after 50 cycles. These findings demonstrate that the synergistic integration of multiredox $\text{Ni(OH)}_2/\text{NiMoO}_4$ with conductive rGO/NF enhances electrochemical performance, providing effective design strategy for developing hybrid battery-supercapacitor electrodes.

1. Introduction

Global energy consumption remains heavily dependent on fossil fuels, which accounted for 83% of total global energy use in 2023 [1]. Continued reliance on these resources accelerates environmental degradation and could increase global temperatures by $\sim 3^\circ\text{C}$ by the end of this century. To limit warning, carbon dioxide (CO_2) emissions must reach net zero by 2050 [2]. Moreover, fossil fuel reserves are finite, driving the need for sustainable energy technologies, including efficient energy storage systems [3].

Batteries, the dominant energy storage technology, offer high energy density at relatively low cost but suffer from low power density, limited lifespan, and performance degradation under high charge/discharge rates [4]. Supercapacitors, in contrast, deliver high power density, long cycle life, rapid charge/discharge, and low maintenance [5], but have lower energy density than batteries. Integrating both into a hybrid energy storage system can combine their advantages: in electric vehicles (EVs), for instance, the battery supplies long-range energy, while the supercapacitor buffers power surges during acceleration or braking, extending battery life and reducing degradation [4].

One promising effort to improve the performance of hybrid battery-supercapacitors is by using $\text{Ni(OH)}_2/\text{NiMoO}_4$ composites on a rGO/NF template. rGO is a graphene derivative with similar features as that of graphene to some extent and has the remaining oxygen functionalities [6,7]. rGO is commonly prepared from graphite which undergoes an oxidation process followed by reduction in which some of the oxygen

groups are removed, making rGO easily react with other materials [8,9]. rGO exhibits high electrical capacitance and surface area; therefore, it has potentially been used as an electrode material in batteries and supercapacitors. rGO stores charge predominantly via the electrostatic accumulation of ions at the electrode-electrolyte interface, a non-Faradaic process typical of electric double-layer capacitors (EDLCs) and symmetric charge storage systems. Its ability to store high energy is required in hybrid battery-supercapacitor systems [10].

Nickel-rich cathodes have emerged as a promising alternative in battery technology, offering structural stability and high operating potential for enhanced electrochemical performance [11]. Ni(OH)_2 , a typical pseudocapacitive or battery-type material, stores charge primarily through Faradaic redox reactions between Ni(OH)_2 and NiOOH in alkaline electrolytes and has been widely applied in asymmetric supercapacitor [12]. While the synthesis of Ni(OH)_2 nanosheets on NiMoO_4 nanorods has been reported [13], their integration with a symmetric EDLC-type material such as rGO remains unexplored. NiMoO_4 , a molybdate-based material, provides high capacitance, low cost, abundant resources, and environmental friendliness but suffers from limited rate capability and cycle life [14,15]. Therefore, combining NiMoO_4 with Ni(OH)_2 in a composite structure offers a promising strategy to enhance the overall performance of hybrid electrodes.

As a template, nickel-based substrates such as NF and nickel-conducting cloth serve as electrode supports due to their high density and large surface area [16]. NF can increase the bond between the active materials and facilitate efficient charge transfer [17]. In this study,

hydrothermal and electrodeposition methods are used to achieve the desired hierarchical structure on the electrode.

The novelty of this work lies in the strategic integration of $\text{Ni(OH)}_2/\text{NiMoO}_4$ composites with rGO on an NF template to form a hierarchical hybrid electrode structure. While $\text{Ni(OH)}_2/\text{NiMoO}_4$ composites have previously been explored in asymmetric supercapacitors [13], achieving a capacitance of $121 \text{ F}\cdot\text{g}^{-1}$ at $10 \text{ A}\cdot\text{g}^{-1}$ with 77% retention after 5000, their coupling with a symmetric charge storage component such as rGO within a single electrode architecture has not been reported. This design simultaneously combines (i) the multiple redox-active sites of Ni(OH)_2 and NiMoO_4 for enhanced pseudocapacitance, (ii) the high electrical conductivity and surface area of rGO for rapid charge transport, and (iii) the mechanical robustness and high surface area of NF for strong adhesion and efficient ion/electron pathways. This synergistic configuration results in improved specific capacitance, energy density, conductivity, and discharge time, making the electrode a promising candidate for next-generation hybrid battery-supercapacitor devices.

2. Experimental

2.1 Materials synthesis

The synthesis process is carried out in four stages, as illustrated in Figure 1. rGO was prepared from graphite (Dingshengxin ISO9001) by a modified Tour route. Details of the route can be found elsewhere [18]. The obtained rGO was then coated on NF to form the rGO/NF template. NiMoO_4 was fabricated by a hydrothermal method on the rGO/NF template inside an autoclave. Lastly, Ni(OH)_2 was prepared by electrodeposition process of $\text{Ni}(\text{NO}_3)_2\cdot 6\text{H}_2\text{O}$ onto $\text{NiMoO}_4/\text{rGO}/\text{NF}$.

2.1.1 Preparation of the rGO/NF template

NF with a size of $5 \text{ cm} \times 6 \text{ cm}$ was cleaned ultrasonically with acetone, 3 M hydrochloric acid (HCl, 37%, SAP chemicals), deionized (DI) water, and ethanol alternately for 10 min. Then, 10 mg of the prepared rGO was added to 30 mL of distilled water and then ultrasonicated for 90 min. 30 mg of ascorbic acid ($\text{C}_6\text{H}_8\text{O}_6$, Merck) was dispersed into the rGO solution and stirred for 10 min at room temperature to obtain an rGO suspension. The cleaned NF was soaked in the suspension and then heated in a water bath at 90°C for 6 h. The rGO/NF was cleaned with DI water and ethanol ($\text{C}_2\text{H}_6\text{O}$ 70%, SAP chemicals) to reach neutral pH and then dried in a vacuum at 60°C for 2 h.

2.1.2 Hydrothermal route for the synthesis of NiMoO_4 on the rGO/NF template

The process of making $\text{NiMoO}_4/\text{rGO}/\text{NF}$ begins by adding 40 mL of 0.1 M $\text{Na}_2\text{MoO}_4\cdot 2\text{H}_2\text{O}$ (99.5%, Himedia PCT0117) into 35 mL of 0.11 M $\text{Ni}(\text{NO}_3)_2\cdot 6\text{H}_2\text{O}$ (99%, Smart Lab) stirring them for 15 min to form a homogenous solution. Then, 0.185 g of ammonium fluoride (NH_4F , Merck) and 0.48 g of urea ($\text{CH}_4\text{N}_2\text{O}$, Merck) were added to the solution. The rGO/NF obtained from the previous stage was cut into three parts with a size of $1 \text{ cm} \times 2 \text{ cm}$ each, which were then put into the solution. It was then transferred into a Teflon-lines

autoclave (100 mL) to undergo a hydrothermal process at 160°C for 6 h. The result of $\text{NiMoO}_4/\text{rGO}/\text{NF}$ is washed with DI water and ethanol and then dried at 60°C overnight.

2.1.3 Electrodeposition of Ni(OH)_2 on $\text{NiMoO}_4/\text{rGO}/\text{NF}$

Electrodeposition was applied to attach Ni(OH)_2 to $\text{NiMoO}_4/\text{rGO}/\text{NF}$. 4 mmol potassium chloride (KCl 99%, SAP chemicals) was added slowly into 50 mL DI water. $\text{Ni}(\text{NO}_3)_2\cdot 6\text{H}_2\text{O}$ (1.75 g) was alternately added into the solution, then was magnetically stirred for 10 min. The previously prepared $\text{NiMoO}_4/\text{rGO}/\text{NF}$ was transferred into the solution, and then a potential of 5 V was applied for 120 s to deposit Ni(OH)_2 on $\text{NiMoO}_4/\text{rGO}/\text{NF}$. The resulting samples were cleaned with DI water and ethanol and then dried in a vacuum at 60°C .

2.2 Characterizations

X-ray diffraction (XRD, $\text{CuK}\alpha$, $\lambda = 1.54056 \text{ \AA}$) is used to analyze the obtained phase of each synthesis stage. The phase analysis was performed using Match! software. The functional groups, surface morphologies, and elemental compositions of samples were examined using Fourier transform infrared (FTIR, Nicolet iS10), scanning electron microscopy (SEM, INSPECT S50), and energy dispersive X-ray (EDX), respectively. Electrochemical tests were conducted on a half-cell with three-electrode setup. The capacitive performances of the $\text{Ni(OH)}_2/\text{NiMoO}_4/\text{rGO}/\text{NF}$ electrodes were evaluated by cyclic voltammetry (CV) within the potential range of 0 V to 0.7 V and scan rates of $5 \text{ mV}\cdot\text{s}^{-1}$, $10 \text{ mV}\cdot\text{s}^{-1}$, $25 \text{ mV}\cdot\text{s}^{-1}$, $50 \text{ mV}\cdot\text{s}^{-1}$, $75 \text{ mV}\cdot\text{s}^{-1}$, and $100 \text{ mV}\cdot\text{s}^{-1}$. The CV measurement was performed in a 2 M KOH electrolyte with graphite as a counter electrode and a saturated calomel electrode (SCE) as a reference. The specific capacitance (C_s), energy density (E), and power density (P) of electrodes were calculated using Equation (1) to Equation (3), respectively.

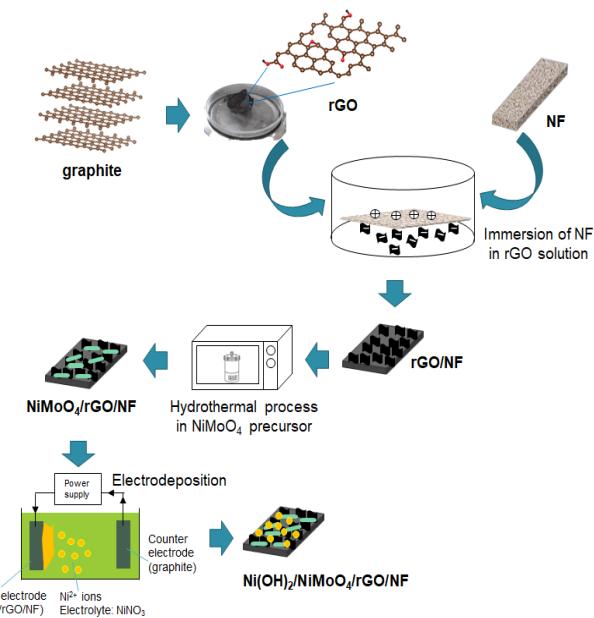


Figure 1. Schematic of the synthesis procedures of the $\text{Ni(OH)}_2/\text{NiMoO}_4$ on the rGO/NF electrode.

$$C_s = \frac{\int IdV}{2\Delta V m v} (\text{F}\cdot\text{g}^{-1}) \quad (1)$$

$$E = \frac{1/2 C_s (\Delta V)^2}{3.6} (\text{Wh}\cdot\text{kg}^{-1}) \quad (2)$$

$$P = \frac{E \times 3600}{t} (\text{W}\cdot\text{kg}^{-1}) \quad (3)$$

$\int IdV$ represents the area within the I-V curve, ΔV describes the potential window, m is the mass of the electrode, v is the scan rate, and t is the discharge time. The m was determined from the mass difference between the sample and the bare NF. The conductivity of the electrodes was analyzed with Electrochemical Impedance Spectroscopy (EIS) within the frequency range of 102 Hz to 106 Hz. Lastly, the galvanostatic charge/discharge (GCD) tests evaluated charge-discharge behaviors of the electrodes within the desired cycles. The discharged specific capacitance ($C_{s\text{-dis}}$) was calculated using Equation (4):

$$C_{s\text{-dis}} = \frac{I/m \times t}{\Delta V} (\text{F}\cdot\text{g}^{-1}) \quad (4)$$

where I/m is the current density ($\text{A}\cdot\text{g}^{-1}$), t is the discharge time, and ΔV is the potential window. The CV, EIS, and GCD measurements were performed using the same device (CS350 Potentiostat, Corrtest).

3. Results and discussion

3.1 Structural analysis

Figure 2(a) shows an illustration of the electrode evolution over the synthesis stages. The NF color changes to black when it is coated with rGO. A light green color is observed as NiMoO_4 is hydrothermally coated onto rGO/NF. The electrodeposition of Ni(OH)_2 on $\text{NiMoO}_4/\text{rGO}/\text{NF}$ within the area of 1 cm \times 1 cm results in a darker green color.

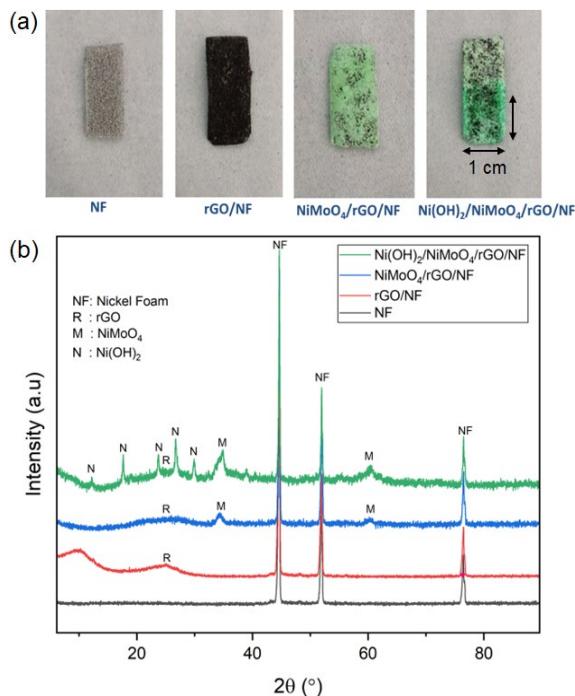


Figure 2. (a) Visualization, and (b) XRD patterns of the electrode over the synthesis steps.

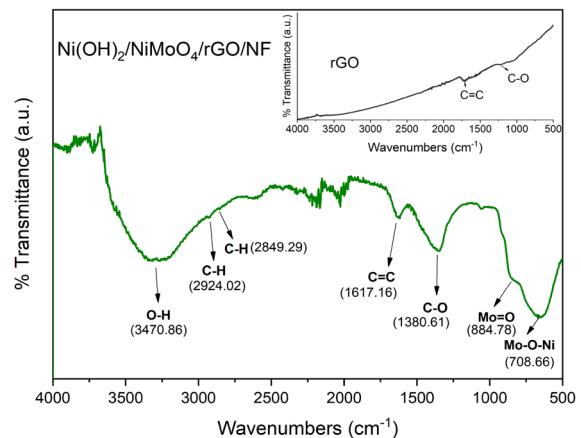


Figure 3. FTIR spectrum of the $\text{Ni(OH)}_2/\text{NiMoO}_4/\text{rGO}/\text{NF}$ electrode. Inset is the spectrum of rGO.

XRD patterns of the electrode over the synthesis procedures are shown in Figure 2(b). NF shows sharp peaks at $\sim 44.5^\circ$, $\sim 51.8^\circ$, and $\sim 76.4^\circ$, consistent with JCPDS card no. 04-0850 [19], which are dominant and remain over the procedures. A broad peak, typical of rGO, is observed at 20 of $\sim 24^\circ$ to $\sim 27^\circ$. Two additional peaks at $\sim 33.9^\circ$ and $\sim 60.2^\circ$ corresponding to NiMoO_4 (JCPDS card no. 45-0142) are seen in $\text{NiMoO}_4/\text{rGO}/\text{NF}$, which is consistent with that reported in [20]. The presence of Ni(OH)_2 is confirmed by XRD peaks at $\sim 12.1^\circ$, 18.8° , 23.6° , 27.1° , 29.6° , 32.1° , 34.6° , and 54.8° (JCPDS card no. 38-0715) as described by Zhang *et al.* [12]. This result confirms that the electrodeposition process successfully yields $\text{Ni(OH)}_2/\text{NiMoO}_4$ composites on the rGO/NF template.

The functional groups present in the $\text{Ni(OH)}_2/\text{NiMoO}_4/\text{rGO}/\text{NF}$ electrode were evaluated from the FTIR spectrum, as displayed in Figure 3. The electrode has functional groups of Mo-O-Ni at 708.66 cm^{-1} and Mo=O at 884.78 cm^{-1} , which indicates NiMoO_4 [21]. C-O and C=C as characteristic bonds of rGO are observed at 1380.61 cm^{-1} and 1617.16 cm^{-1} , respectively. This is in accordance with the FTIR spectrum of pristine rGO as shown in the inset of Figure 3. Moreover, the asymmetric C-H at 2924.02 cm^{-1} , symmetric C-H at 2849.29 cm^{-1} , and O-H at 3470.86 cm^{-1} are indications of the presence of Ni(OH)_2 in the $\text{Ni(OH)}_2/\text{NiMoO}_4$ composite [22]. While the FTIR analysis was focused on the final composite to confirm the coexistence of Ni(OH)_2 , NiMoO_4 , and rGO functional groups, the spectra of the resulting sample in each stage were not included here, as their structural identities were verified by XRD and SEM/EDX analyses.

The surface morphologies of rGO and $\text{Ni(OH)}_2/\text{NiMoO}_4/\text{rGO}/\text{NF}$ are presented in Figure 4(a-b), respectively. rGO shows wrinkle stacking layers having some parts that are more transparent and thinner compared with others, which confirms the existence of multi-layer rGO. This is a typical rGO structure obtained from both graphite [18] and biomass [7,23]. The thin layer-like nature of rGO remains in the $\text{Ni(OH)}_2/\text{NiMoO}_4/\text{rGO}/\text{NF}$ electrode, and it is visible on a surface that appears more indented. Urchin-like and nanosphere-like particles were also observed, and they spread all over the surface. It has been previously reported that NiMoO_4 has a structure resembling that of sea urchins [24], while Ni(OH)_2 looks like a nano-sized sphere [12]. The SEM image hence verifies the presence of the $\text{Ni(OH)}_2/\text{NiMoO}_4$ composite on the rGO/NF template, which is consistent with the XRD data.

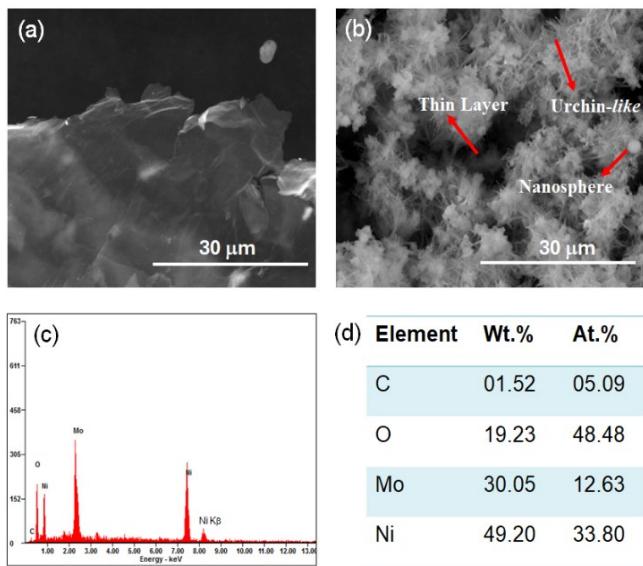


Figure 4. SEM images of (a) rGO and (b) the $\text{Ni(OH)}_2/\text{NiMoO}_4/\text{rGO/NF}$ electrode, (c) its EDX spectrum, and (d) the relative weight (wt%) and atomic (at%) percentages of the observed K emission of each element.

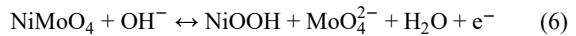
The EDX spectrum of the $\text{Ni(OH)}_2/\text{NiMoO}_4/\text{rGO/NF}$ electrode is presented in Figure 4(c), showing the presence of Ni, Mo, C, and O peaks. Three characteristic Ni emissions are identified: (i) Ni-L at ~ 1 keV, (ii) Ni-K α at ~ 7.5 keV, and (iii) Ni-K β at ~ 8.2 keV. Similar Ni emissions have also been reported in previous studies [25,26]. The relative weight (wt%) and atomic (at%) percentages of each element, calculated from the K emissions, are summarized in Figure 4(d). Ni, Mo, and O are relatively dominant, consistent with their presence in the $\text{Ni(OH)}_2/\text{NiMoO}_4$ composite on the surface of the electrode.

3.2 Electrochemical properties

Figure 5(a) displays the current-potential (I-V) curves of the NF, rGO/NF, $\text{NiMoO}_4/\text{rGO/NF}$, and $\text{Ni(OH)}_2/\text{NiMoO}_4/\text{rGO/NF}$ electrodes obtained from each synthesis step at a scan rate of $5 \text{ mV}\cdot\text{s}^{-1}$. The bare NF exhibited a relatively low current response, indicating limited electrochemical activities with relatively small charge storage [19]. However, a closed view of the curve shows a small redox peak at 0.2 V to 0.3 V , which can be associated with the electrochemical behavior of Ni in the KOH electrolyte. When Ni is oxidized in the electrolyte, it forms Ni(OH)_2 or NiO(OH) , while during reduction, NiO(OH) is reduced back to Ni(OH)_2 [14]; thus, the redox process is reversible. This behavior, along with its high surface area, makes NF a robust electrode template for rechargeable battery systems. When NF is coated with rGO, the current is slightly enhanced, showing that rGO provides better conductivity but does not significantly contribute to the redox activity.

A noticeable increase in current was observed when NiMoO_4 was deposited on the rGO/NF substrate. The redox peak became more prominent, which is likely due to the catalytic activity of NiMoO_4 . In addition to its good structural and thermal stability, NiMoO_4 exhibits multiple reversible redox reactions associated with $\text{Ni}^{2+}/\text{Ni}^{3+}$ and $\text{Mo}^{6+}/\text{Mo}^{5+}$, which contribute to additional redox-active sites and enhance the overall charge storage capacity [27]. The $\text{Ni(OH)}_2/\text{NiMoO}_4/\text{rGO/NF}$ showed the highest current response, indicating a significant

enhancement of the redox reactions, likely involving $\text{Ni(OH)}_2/\text{NiOOH}$ and NiMoO_4 , as indicated in Equation (5) and Equation (6) [28,29].



The largest I-V curve area also confirms that this electrode exhibits the highest specific capacitance at $5 \text{ mV}\cdot\text{s}^{-1}$ compared to the others. Moreover, all curves display typical pseudocapacitive behavior rather than electric double-layer capacitor (EDLC) [30]. The incorporation of Ni(OH)_2 onto $\text{NiMoO}_4/\text{rGO/NF}$ creates a synergistic effect, providing more accessible active sites, enhancing electron transport through rGO, and improving stability and ionic diffusion due to the hierarchical structure of the electrode. All electrodes show a continuous and rapid increase in current without a distinct peak as the potential exceeds $\sim 0.5 \text{ V}$. This increase is characteristic of non-Faradaic or pseudocapacitive behavior, in which redox reactions occur rapidly at or near the surface [30]. Another possibility is that water oxidation to oxygen (OER) [31] begins to dominate at $\sim 0.5 \text{ V}$. Therefore, the observed current increase may result from a combination of pseudocapacitive behavior and the onset of OER.

The I-V curves of the $\text{Ni(OH)}_2/\text{NiMoO}_4/\text{rGO/NF}$ electrode at various scan rates are shown in Figure 5(b). Redox peaks are prominent at low scan rate but become less noticeable as the scan rate increases. The C_s , E , and P values of each electrode at varying scan rate were estimated by applying Equation (1) to Equation (3), and the results are plotted in Figure 5(c). All exhibit a decreasing trend in the C_s value as the scan rate increases. At the $5 \text{ mV}\cdot\text{s}^{-1}$ scan rate, the C_s is higher because ions have sufficient time to access the electrode surface and pores, while a decrease in the C_s as the scan rate increases is due to limited ion diffusion at higher rates [14,32]. It is clearly shown that $\text{Ni(OH)}_2/\text{NiMoO}_4/\text{rGO/NF}$ has the highest C_s at low scan rate, but it drops the most. By comparing to the initial C_s at $5 \text{ mV}\cdot\text{s}^{-1}$, it has $\sim 21\%$ retention at $100 \text{ mV}\cdot\text{s}^{-1}$. On the other hand, rGO/NF maintained better retention at high scan rates, although it had the lowest C_s at low scan rates. rGO/NF is more like surface-based storage (EDLC behavior), while in $\text{Ni(OH)}_2/\text{NiMoO}_4/\text{rGO/NF}$, ion diffusion at high scan rates is limited because of the slower kinetics of redox processes and that ions can not access deeper redox sites effectively [14]. The Ragone plot in the inset of Figure 5(c) emphasizes that $\text{Ni(OH)}_2/\text{NiMoO}_4/\text{rGO/NF}$ has the highest energy density and maintains good power density, making it suitable for high-performance energy storage applications.

Figure 5(d) presents the Nyquist plot of electrodes. The data is fitted using an equivalent circuit consisting of R_s , R_{ct} , CPE, and Z_w , which represent the solution resistance, charge transfer resistance, constant phase element, and Warburg impedance, respectively. The Z_w is associated with the straight line at low frequency and is related to ion diffusion [32]. Among the presented plots, $\text{Ni(OH)}_2/\text{NiMoO}_4/\text{rGO/NF}$ has the smallest semicircle at high frequencies, indicating the lowest R_{ct} , and a steeper slope, implying better capacitance. At the constant frequency of 0.01 kHz , the electrical conductivity was enhanced in the order of NF ($0.35 \text{ S}\cdot\text{m}^{-1}$), rGO/NF ($0.92 \text{ S}\cdot\text{m}^{-1}$), $\text{NiMoO}_4/\text{rGO/NF}$ ($1.07 \text{ S}\cdot\text{m}^{-1}$), and $\text{Ni(OH)}_2/\text{NiMoO}_4/\text{rGO/NF}$ ($1.30 \text{ S}\cdot\text{m}^{-1}$).

The GCD curves of $\text{Ni(OH)}_2/\text{NiMoO}_4/\text{rGO/NF}$ at various scan rates (Figure 5(e)) exhibit quasi-triangular shapes with slight deviations from linearity, indicating a combination of EDLC and pseudocapacitance

contributions. As the current density increases, the discharge time decreases significantly, reflecting the limited ion diffusion and shorter interaction time with active sites at higher rates. The longest discharge duration at $0.25 \text{ A}\cdot\text{g}^{-1}$ demonstrates the highest $C_{\text{S-dis}}$ under low-rate conditions, whereas higher current densities yield lower $C_{\text{S-dis}}$ but better rate capability, confirming the electrode's suitability for high-power applications.

The GCD responses of the electrode at of $0.25 \text{ A}\cdot\text{g}^{-1}$ are shown in Figure 5(f). The non-linear (pseudocapacitive) profile becomes more

pronounced in $\text{NiMoO}_4/\text{rGO/NF}$ and $\text{Ni(OH)}_2/\text{NiMoO}_4/\text{rGO/NF}$, indicating a contribution from Faradaic redox processes. Among the samples, $\text{Ni(OH)}_2/\text{NiMoO}_4/\text{rGO/NF}$ exhibits the longest discharge time, reflecting its highest specific capacitance and superior energy storage capability. The nearly triangular shapes of the curves suggest good electrochemical reversibility and minimal internal resistance [32]. The inset is the $C_{\text{S-dis}}$ values of $\text{Ni(OH)}_2/\text{NiMoO}_4/\text{rGO/NF}$ at $0.25 \text{ A}\cdot\text{g}^{-1}$ over 50 cycles, demonstrating a retention rate of $\sim 86\%$, indicative of fairly stable cycling performance.

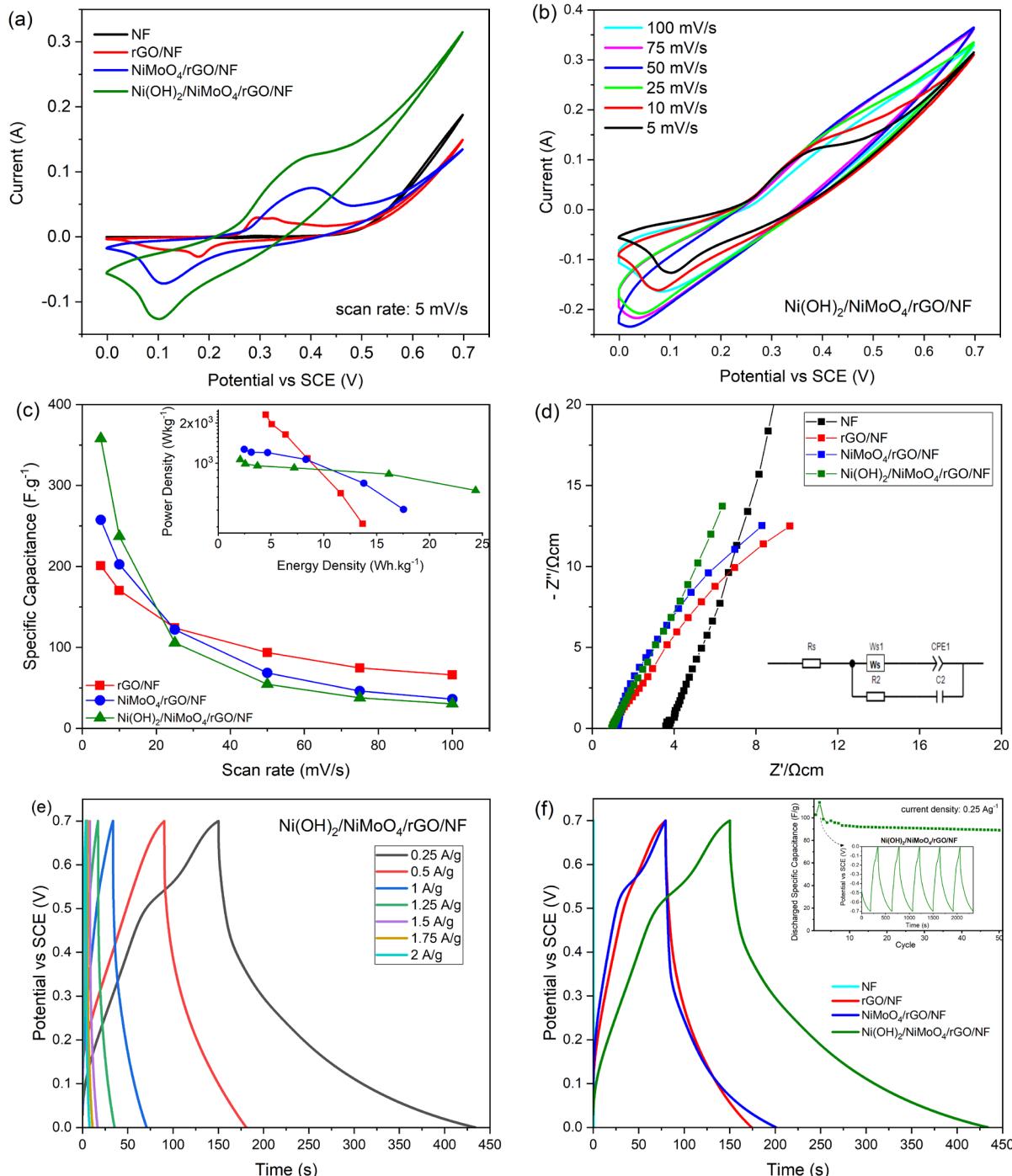


Figure 5. (a) I-V curves of NF, rGO/NF, $\text{NiMoO}_4/\text{rGO/NF}$, and $\text{Ni(OH)}_2/\text{NiMoO}_4/\text{rGO/NF}$ electrodes at $5 \text{ mV}\cdot\text{s}^{-1}$, (b) I-V curves of $\text{Ni(OH)}_2/\text{NiMoO}_4/\text{rGO/NF}$ at various scan rates, (c) The C_{S} vs. scan rate for all electrodes; inset is power density vs. energy density, (d) Nyquist plots with the equivalent circuit, (e) GCD curves of $\text{Ni(OH)}_2/\text{NiMoO}_4/\text{rGO/NF}$ at various current densities, and (f) GCD curves of the electrodes at $0.25 \text{ A}\cdot\text{g}^{-1}$. Inset is the discharged C_{S} over 50 cycles with 86% retention.

Table 1. Comparison of Cs values measured in a three-electrode setup of this study with previous reports.

Material composition	Preparation method	Electrolyte	Current density/scan rate	Specific capacitance	Ref.
Ni(OH) ₂ /NiMoO ₄ on rGO-coated NF	Hydrothermal and electrodeposition	2 M KOH	5 mV·s ⁻¹	358 F·g ⁻¹	This study
Ni(OH) ₂ /rGO nanorods on NF	Hydrothermal	6 M KOH	1 A·g ⁻¹	1143 F·g ⁻¹	[33]
Ni(OH) ₂ /g-C ₃ N ₄ /rGO on NF	Hydrothermal	6 M KOH	1 A·g ⁻¹	543.8 F·g ⁻¹	[34]
NiMoO ₄ on NF	Hydrothermal	3 M KOH	7 A·g ⁻¹	1740 F·g ⁻¹	[35]
NiMoO ₄ @Ni(OH) ₂ on NF	Hydrothermal and electrodeposition	2 M KOH	4 mA·cm ⁻²	7.43 F·cm ⁻²	[36]
Ni(OH) ₂ @CoMoO ₄ on NF	Hydrothermal and electrodeposition	2 M NaOH	8 mA·cm ⁻²	5.23 F·cm ⁻²	[37]
NiMoO ₄ /WO ₃ on NF	Hydrothermal	3 M KOH	1 A·g ⁻¹	429.46 F·g ⁻¹	[38]
Mo ₄ V ₆ O ₂₅ on Ni foil	Wet chemical	1 M KOH	1 A·g ⁻¹	203 C·g ⁻¹	[39]
Ag ₂ Mo ₂ O ₇ on Ni foil	Wet chemical	1 M KOH	1 A·g ⁻¹	430 C·g ⁻¹	[40]
ZrMo ₂ O ₈ on Ni foil	Wet chemical	1 M KOH	1 A·g ⁻¹	288 C·g ⁻¹	[41]
SrMoO ₄ on Ni foil	Wet chemical	1 M KOH	5 mV·s ⁻¹	384 C·g ⁻¹	[42]

A summary of the electrochemical performance of this work compared with prior related materials is presented in Table 1. The comparison includes electrolyte type, current density or scan rate, preparation method, and electrode configuration. Although our specific capacitance is lower than that of some high-performing systems, this study provides valuable proof-of-concept evidence for the integration of Ni(OH)₂/NiMoO₄ composites with rGO on NF, highlighting a balanced approach to enhancing energy density, conductivity, and cycling stability. This hybrid electrode design provides a promising foundation for future optimization toward high-performance supercapacitor applications.

4. Conclusions

In this study, a hierarchical Ni(OH)₂/NiMoO₄ composite was successfully synthesized on an rGO-coated NF (rGO/NF) template through a combination of hydrothermal growth and electrodeposition. Structural analyses confirmed the integration of Ni(OH)₂ nanospheres and NiMoO₄ urchin-like structures on rGO/NF, providing abundant redox-active sites and enhanced electrical conductivity. Electrochemical evaluation demonstrated that the Ni(OH)₂/NiMoO₄/rGO/NF electrode achieved a high specific capacitance of \sim 358 F·g⁻¹ at 5 mV·s⁻¹, an energy density of \sim 24 Wh·kg⁻¹, a power density of \sim 626 W·kg⁻¹, and conductivity of 1.30 S·m⁻¹ at 0.01 kHz. The electrode also retained \sim 86% of its capacitance after 50 charge-discharge cycles, indicating fair cycling stability. These results highlight the synergistic contribution of NF's structural support, rGO's conductivity, and the pseudocapacitive activity of Ni(OH)₂/NiMoO₄, making this hybrid architecture a promising candidate for next-generation high-performance hybrid battery-supercapacitor devices. For future studies, long-term cycling stability over higher cycle numbers and electrochemical measurements in a two-electrode (coin cell) configuration should be conducted to further assess the electrode's durability and practical applicability.

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