

Article

Integrated Effects of NiCo_2O_4 and Reduced Graphene Oxide in High-Performance Supercapacitor Systems

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Abstract

Supercapacitors have attracted significant interest as increased energy storage devices due to their high power density, rapid charge/discharge performance, and long cyclability. In this study, NiO , Co_3O_4 , NCO , and NCO/rGO composite electrodes were prepared and evaluated for high-performance supercapacitor applications. The uniform distribution of elements and the effective incorporation of rGO into the composite were confirmed by structural and morphological characterizations. Among the evaluated materials, the NCO/rGO electrode exhibited high electrochemical performance, delivering a specific capacitance of 998 F g^{-1} in a three-electrode configuration, attributed to the enhanced redox activity of NiCo_2O_4 coupled with the enhanced electrical conductivity of rGO . Additionally, an asymmetric supercapacitor device with activated carbon as the negative electrode and NCO/rGO as the positive electrode showed a power density of 750 W kg^{-1} , an energy density of 29.2 Wh kg^{-1} , and a specific capacitance of 93.7 F g^{-1} . After 5000 charge/discharge cycles, the device maintained 85% of its initial capacitance and a coulombic efficiency of 99%, demonstrating exceptional cyclability. These results highlight the strong potential of the $\text{NiCo}_2\text{O}_4/\text{rGO}$ composite as an advanced electrode material for next-generation energy storage systems.

Keywords: asymmetric supercapacitor; composite electrodes; redox behavior; pseudocapacitance; cyclic stability; energy density



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1. Introduction

With the growing global emphasis on clean and sustainable energy, there is a greater demand for energy storage systems that are efficient and reliable. Supercapacitors have become highly popular because they have a high power density, a high specific capacitance, and a long cycle life [1,2]. This makes them great for quickly storing and delivering energy. However, their wider range of uses is restricted by their comparatively poor energy density in comparison to batteries [3]. To address this constraint, researchers have investigated battery-type electrode materials such as metal oxides [4–7], hydroxides [8–10], and sulfides [11–13], which can store charge via reversible redox reactions that occur

throughout the electrode material. This bulk faradic technique has a substantially larger charge storage capacity than traditional electric double-layer or pseudocapacitive materials.

Among the battery-type materials, mixed transition metal oxides have emerged as especially promising candidates due to their multiple oxidation states, improved electrical conductivity, and enhanced redox activity resulting from the interaction between two or more metal ions. Nickel cobaltite (NiCo_2O_4) has emerged as a promising electrode material for supercapacitors due to its excellent electrochemical properties, versatile synthesis routes, and enhanced performance when combined with complementary materials, offering significant opportunities for advanced energy storage applications [14,15]. Nickel-cobalt oxides, notably NiCo_2O_4 , stand out for their low cost, natural abundance, and good electrochemical characteristics [16,17]. The unique spinel crystal structure of NiCo_2O_4 facilitates fast ion diffusion and electron transport by providing well-organized pathways within the lattice [18]. Furthermore, developments in nanostructure engineering, such as the creation of nanosheets, nanotubes, and hierarchical designs, have greatly improved the specific capacitance and cycling stability of NiCo_2O_4 electrodes. These properties make NiCo_2O_4 a particularly desirable material for next-generation energy storage systems.

To further enhance the performance of NiCo_2O_4 -based electrodes, incorporating conductive carbon materials has become a widely adopted strategy. Specifically, reduced graphene oxide (rGO) provides outstanding mechanical flexibility, a high surface area, and electrical conductivity. When coupled with NiCo_2O_4 , rGO not only increases overall conductivity but also serves as a flexible scaffold, cushioning volume variations during charge–discharge cycles and providing additional active sites for electrochemical reactions [19]. This synergistic combination produces electrodes with higher capacitance, faster charge transfer, and better cycling stability, making NiCo_2O_4 /rGO composites very promising for high-performance supercapacitor applications.

In this work, NiCo_2O_4 was hydrothermally synthesized to produce distinct nanostructures with improved electrochemical characteristics. Similar conditions were also used to create pure NiO and Co_3O_4 for comparison. To determine the benefits of the mixed oxide over the single-component materials, a thorough analysis of their physical and electrochemical properties was conducted. This thorough investigation shows the significant potential of NiCo_2O_4 as an effective and long-lasting electrode material for cutting-edge supercapacitors and offers insightful information on the connection between material structure and energy storage performance.

2. Results and Discussion

The phase composition and crystallinity of the prepared samples were analyzed using X-ray diffraction (XRD) (Figure 1a). The XRD patterns were recorded with a Cu $K\alpha$ radiation source ($\lambda = 1.5406 \text{ \AA}$). The diffraction pattern of NiO shown in Figure 1 displays distinct peaks at 2θ values of 37.2, 43.2, 62.9, and 75.3 corresponding to (003), (012), (110), and (015) planes, confirming the formation of the rhombohedral phase of NiO (JCPDS no: 00-022-1189) [20]. The absence of impurity peaks confirms the formation of pure NiO. The diffraction peaks observed at 2θ values 18.9, 31.2, 36.7, 38.4, 44.7, 55.5 and 65.1 corresponding to the (111), (220), (311), (222), (400), (422), (511) and (440) planes are in excellent agreement with the spinel cubic phase of Co_3O_4 (JCPDS No. 01-080-1533) [21]. The X-ray diffraction (XRD) pattern of the synthesized NiCo_2O_4 (Figure 1a) exhibits well-defined diffraction peaks at 2θ values of 18.9, 31.2, 36.7, 38.4, 44.7, 55.5, and 65.1, which are indexed to the (111), (220), (311), (222), (400), (422), (511), and (440) planes of a cubic spinel structure, respectively, in good agreement with the standard JCPDS card No. 00-042-1467. No additional peaks corresponding to other impurity phases are observed, confirming the formation of a phase-pure NiCo_2O_4 spinel oxide. The sharp and intense reflections indicate

a high degree of crystallinity. The observed diffraction pattern also suggests a well-ordered spinel structure, which is expected to facilitate effective interfacial interaction, beneficial for enhanced charge transfer and redox activity in electrochemical applications. The XRD pattern of the $\text{NiCo}_2\text{O}_4/\text{rGO}$ composite (Figure 1a) shows all characteristic peaks of the cubic spinel NiCo_2O_4 phase. The presence of rGO is indicated by a broad, low-intensity diffraction feature at 26.2° , characteristic of few-layer graphene, which is overlapped by the NiCo_2O_4 reflections due to its low content. The sharp and well-defined NiCo_2O_4 peaks reflect high crystallinity, while the uniformly incorporated rGO sheets are expected to enhance electron transport and structural stability, promoting improved electrochemical performance. The most intense reflections are observed at $2\theta \approx 35.05^\circ$, 56.45° , and 62.01° , which exhibit narrow full-width at half maximum (FWHM) values of 0.2737° , 0.3643° , and 0.3806° , respectively. The sharpness of these peaks indicates good crystallinity of the material.

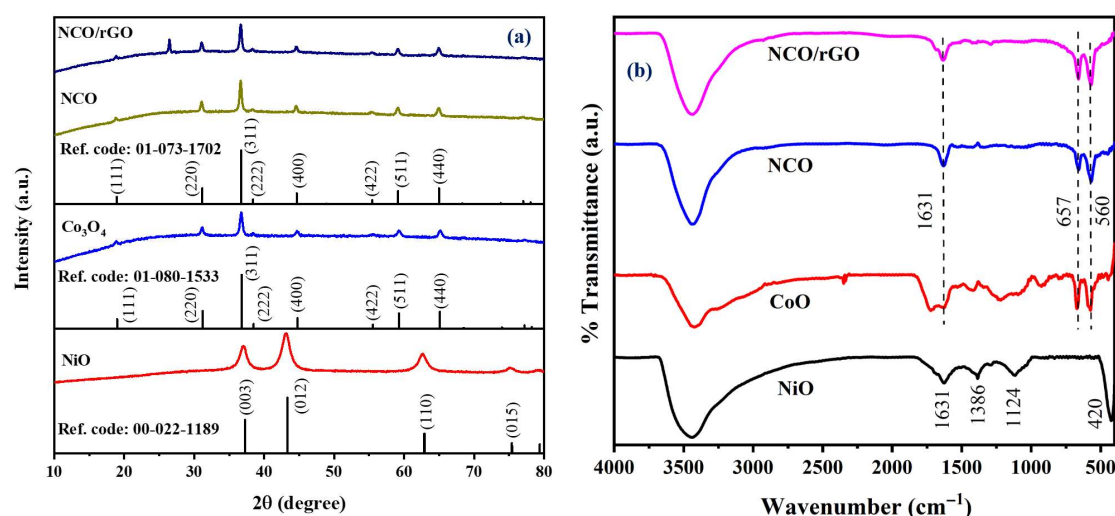


Figure 1. (a) XRD and (b) FTIR spectrum of NiO, Co_3O_4 , NCO, and NCO/rGO.

The average crystallite size calculated using the Scherrer equation for the dominant peaks lies in the range of ~ 30.439 nm, suggesting nanoscale crystallite formation.

$$D = \frac{K\lambda}{\beta \cos\theta} \quad (1)$$

where D is the crystallite size, K is the shape factor, λ is the X-ray wavelength, β indicates the FWHM of the diffraction peak, and θ is the Bragg angle. Broader peaks observed at lower 2θ values (e.g., $\sim 9.07^\circ$ and $\sim 20.46^\circ$) with larger FWHM values indicate the presence of smaller crystallites or structural disorder, which may arise from lattice strain or the contribution of secondary/low-crystallinity components.

Fourier-transform infrared (FTIR) spectra were collected in the $400\text{--}4000\text{ cm}^{-1}$ range using the KBr pellet method. The FTIR spectra of NiO, Co_3O_4 , NiCo_2O_4 (NCO), and NCO/rGO were analyzed after calcination at 350°C . A weak band around 1631 cm^{-1} observed in all samples is attributed to the bending vibration of physically adsorbed moisture from ambient exposure during measurement. For NiO, the prominent band at $\sim 420\text{ cm}^{-1}$ corresponds to the characteristic Ni–O stretching vibration, while the weak features at 1386 cm^{-1} and 1124 cm^{-1} are attributed to surface-related vibrational modes rather than carbonate or nitrate species, which are excluded due to the high calcination temperature. In Co_3O_4 , NCO, and NCO/rGO, the absorption bands at $\sim 657\text{ cm}^{-1}$ and $\sim 560\text{ cm}^{-1}$ are characteristic of spinel-type metal–oxygen vibrations associated with tetrahedral and octahedral sites, arising from overlapping Ni–O and Co–O modes in the NiCo_2O_4

lattice. The similarity of these bands in NCO and NCO/rGO confirms that the incorporation of rGO does not alter the spinel structure, supporting the formation of phase-pure NiCo_2O_4 -based materials.

Using a field-emission scanning electron microscopy (SEM) operated at an accelerating voltage of 3–30 kV, SEM images were taken and energy-dispersive X-ray (EDX) analysis was performed. The scanning electron microscope images of the synthesized samples are shown in Figure 2. Figure 2a–c reveal spherical NiO particles with a relatively rough surface and moderate agglomeration, indicating uniform nucleation. The Co_3O_4 microstructures display a distinct flower-like morphology composed of radially aligned nanorods, suggesting a well-crystallized architecture (Figure 2d–f). The SEM image of NCO is shown in Figure 2g–i, where the formation of densely packed and irregular porous structures indicates strong interfacial interaction between the two oxides. The final composite shown in Figure 2j–l represents the NCO/rGO composite, showing rGO nanosheets uniformly decorated with fine oxide nanoparticles, confirming the successful anchoring of NCO on rGO layers. The elemental mapping of the NCO/rGO composite (Figure 3) further confirms the uniform spatial distribution of Ni, Co, O, and C elements throughout the sample. The homogeneous dispersion of Ni and Co indicates the successful incorporation of both metal oxides within the rGO matrix, while the even carbon signal verifies the continuous presence of rGO sheets. Figure S1 presents the particle size distribution histograms of NiO , Co_3O_4 , and NCO.

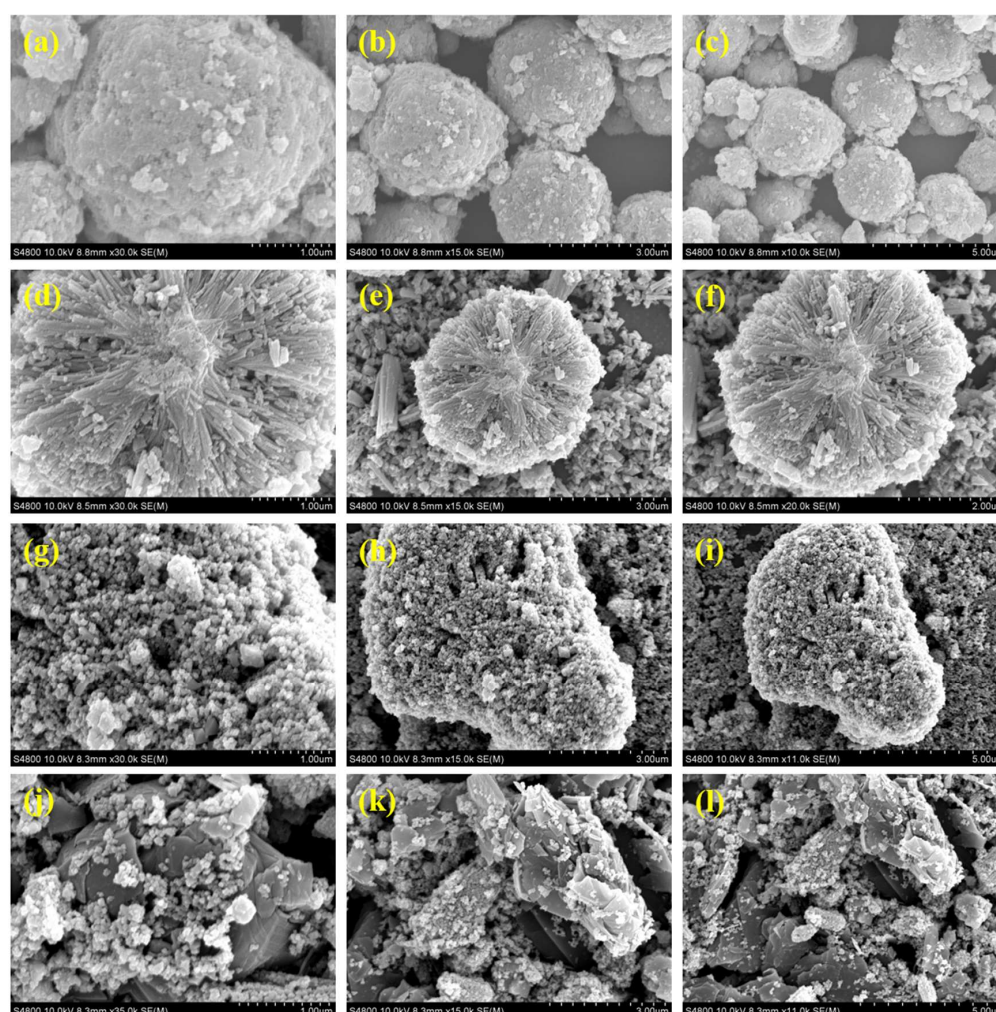


Figure 2. SEM micrographs of NiO (a–c), Co_3O_4 (d–f), NCO (g–i), and NCO/rGO (j–l).

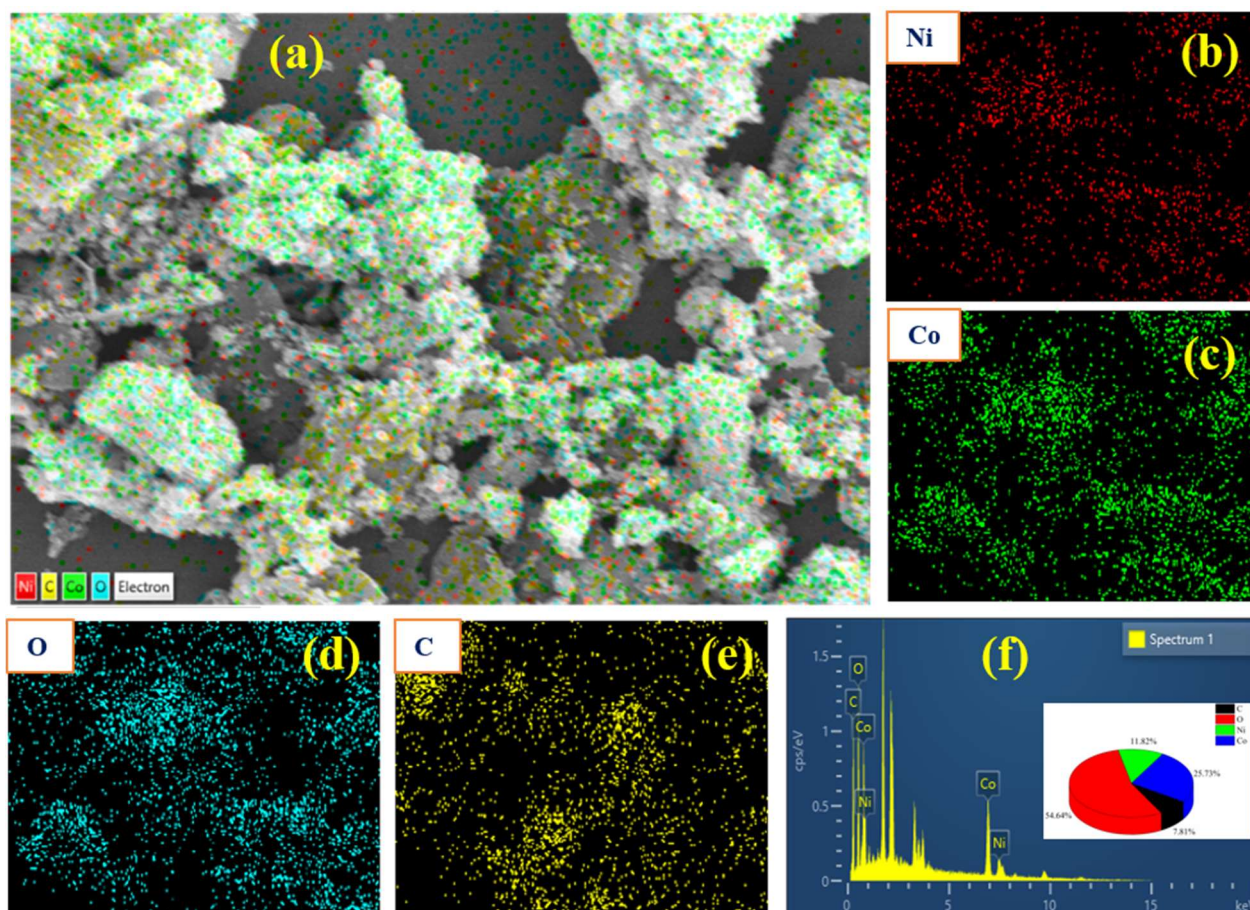


Figure 3. (a–e) Elemental mapping and (f) EDX spectrum of NCO/rGO.

Figure 4a–e illustrate the cyclic voltammetry (CV) curves of the Co_3O_4 , NiO, NCO, and NCO/rGO electrodes, all of which demonstrate distinct pseudocapacitive behavior. The electrode surface undergoes Faradic redox reactions, which are indicated by the presence of distinct redox peaks within the potential window of 0–0.6 V. These reactions are predominantly linked to the reversible redox transitions of nickel and cobalt species, which can be expressed as follows [22]:

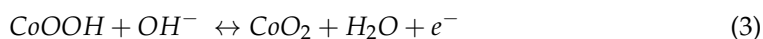


Figure 4a shows that the NCO/rGO electrode has a much higher current response than the Co_3O_4 , NiO, and NCO electrodes. Moreover, as the scan rate increases, the current of the redox peaks also increases, which indicates that the charge transfer kinetics are fast and efficient. The NCO/rGO electrode has a larger CV area and a greater current density, which shows that it has better electrochemical activity and can store more energy. This enhancement is due to the synergistic interaction between NCO and the rGO matrix. The rGO matrix has superior electrical conductivity, more active sites, and a faster ion transport pathway than the other prepared electrodes. Figure 4f illustrates the linear plot of $\log v$ versus $\log I$ based on the power law relationship shown below [23]:

$$i_p = a v^b \quad (4)$$

where i_p is the peak current (A), v is the scan rate (V/s), b is the exponent, and a denotes a constant. The obtained b values (~ 0.5) for both anodic and cathodic peaks confirm that the charge storage process in the sample shows battery-type behavior.

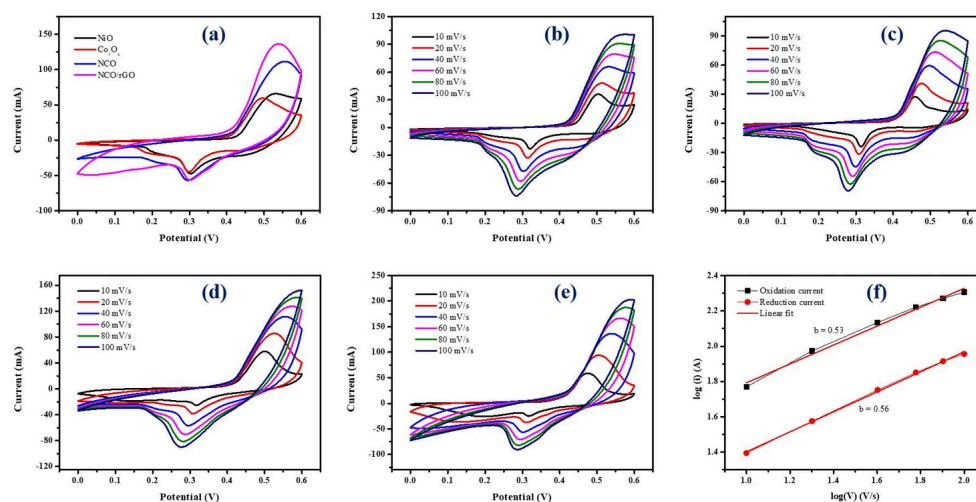


Figure 4. (a) CV curve comparison for all the electrodes at a scan rate of 40 mV/s, (b–e) CV curves of Co₃O₄, NiO, NCO, and NCO/rGO, respectively, at different scan rates, and (f) linear plot of $\log v$ vs. $\log I$.

The GCD plot exhibits a slightly distorted triangular shape, indicating that the charge–discharge process involves both electric double-layer capacitance and faradaic redox reactions (Figure 5a–e). This deviation from the ideal triangular profile confirms the pseudocapacitive behavior of the electrode materials, which arises from reversible redox reactions occurring at the electrode–electrolyte interface [24]. The specific capacitance values obtained from the three-electrode GCD measurements were 378, 298, 742, and 998 F g^{−1} at 1 A g^{−1} for NiO, Co₃O₄, NCO, and NCO/rGO, respectively. The NCO/rGO electrode exhibited the highest capacitance, which can be attributed to multiple redox-active sites and the presence of rGO, which enhances electrical conductivity and facilitates efficient ion/electron transport. The significant improvement in capacitance demonstrates that the incorporation of rGO effectively boosts the electrochemical performance of the composite electrode. Figure 5f presents the comparative specific capacitance values of the electrodes, revealing that the NCO/rGO electrode exhibits the highest performance among all samples. Furthermore, the NCO/rGO electrode delivered higher capacitance compared with the earlier reported results, as shown in Table S1. The EIS plot shown in Figure 5g exhibits a semicircle in the high-frequency region and a nearly vertical line in the low-frequency region, indicating low internal resistance, efficient charge transfer, and near-ideal capacitive behavior of the fabricated ASC device. In general, the high-frequency semicircle corresponds to the charge-transfer resistance (R_{ct}), while the low-frequency straight line is associated with capacitive ion diffusion. Notably, the NCO/rGO electrode shows a significantly smaller semicircle diameter, indicating a lower R_{ct} compared to NiO, Co₃O₄, and NCO, which confirms faster charge-transfer kinetics due to the incorporation of rGO [25]. Moreover, the linear portion of the NCO/rGO spectrum in the low-frequency region shows a steeper inclination toward the x-axis, indicating enhanced ion diffusion and superior conductivity. Figure 5h illustrates the cycling stability of the NCO/rGO electrode over 5000 charge–discharge cycles, retaining 77% of its initial capacitance with a Coulombic efficiency of 99%, confirming its excellent electrochemical stability and reversibility.

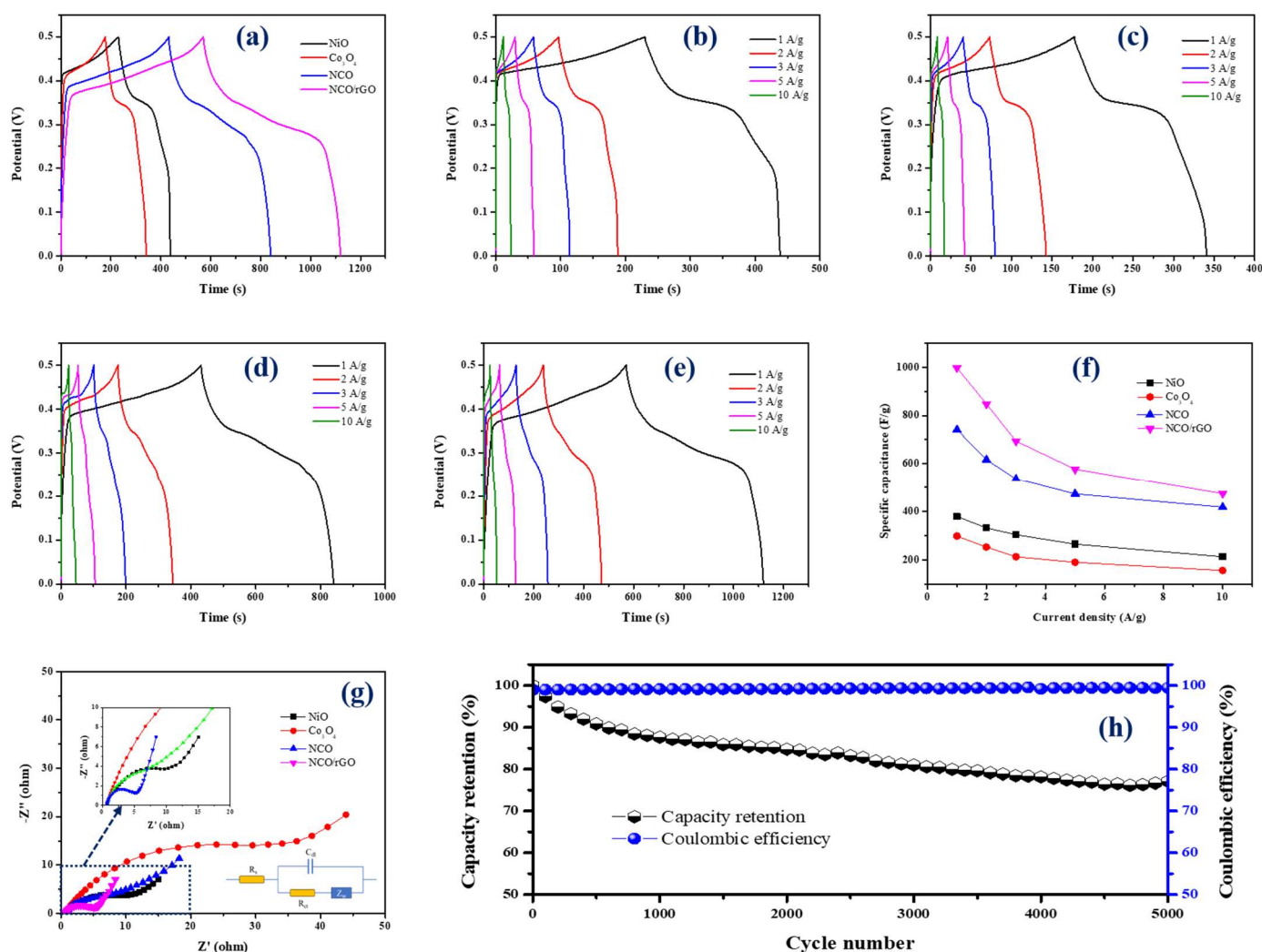


Figure 5. (a) GCD curve comparison for all the electrodes at a current density of 1 A/g, (b–e) GCD curves of Co_3O_4 , NiO, NCO, and NCO/rGO, respectively, at different current densities, (f) comparison profile of current density vs. capacitance, (g) Nyquist plot, and (h) long-term cycling stability of NCO/rGO electrode.

The asymmetric supercapacitor (ASC) device, assembled using NCO/rGO as the positive electrode and activated carbon (AC) as the negative electrode, delivered a specific capacitance of 93.7 F g^{-1} . The device achieved an energy density of 29.2 Wh kg^{-1} at a corresponding power density of 750 W kg^{-1} , demonstrating an excellent balance between energy and power output. The EIS plot shown in Figure 6c exhibits a semicircle in the high-frequency region and a nearly vertical line in the low-frequency region, indicating low internal resistance, efficient charge transfer, and ideal capacitive behavior of the fabricated ASC device. Figure 6d presents the cycling stability of the NCO/rGO-based asymmetric supercapacitor, showing excellent long-term durability. After 5000 charge–discharge cycles, the device retained 85% of its initial capacitance with a Coulombic efficiency of 99%, indicating outstanding reversibility and stable electrochemical performance during continuous operation. Figure 6e illustrates the plot showing the relationship between energy density and power density of the NCO/rGO-based asymmetric supercapacitor. The device maintains a high energy density even at elevated power densities, demonstrating its excellent rate capability and efficient energy delivery, which are essential characteristics for practical energy storage applications.

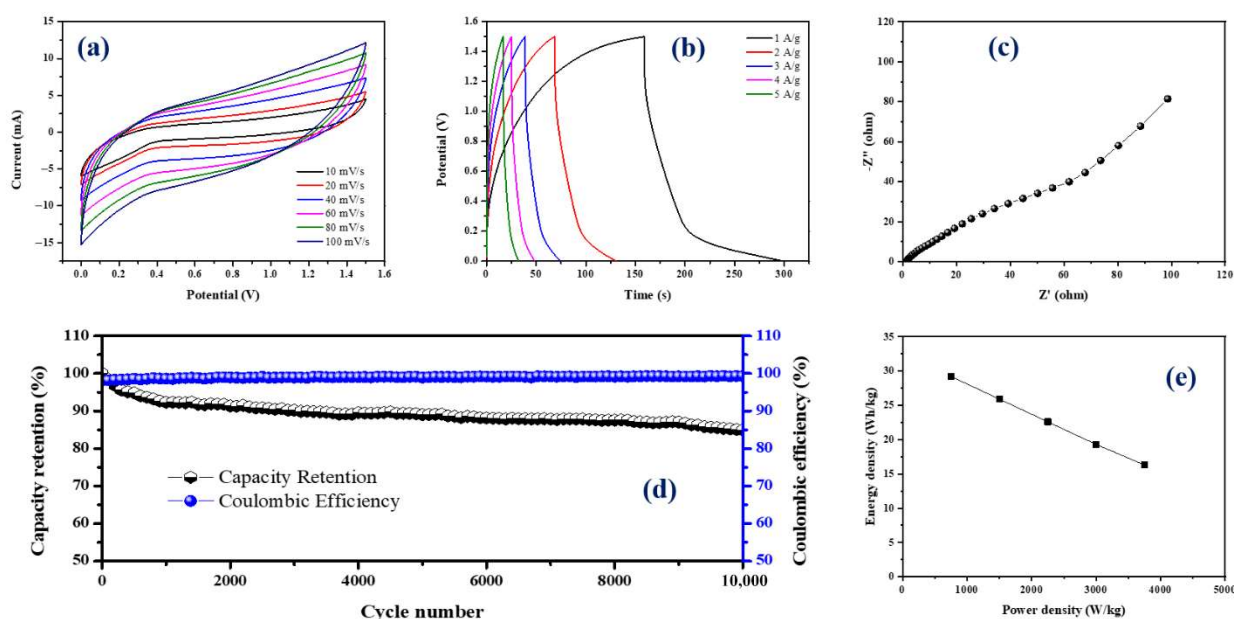


Figure 6. (a) CV curves of NCO/rGO//AC electrode at different scan rates, (b) GCD profile of the NCO/rGO//AC electrode at various current densities, (c) Nyquist plot, (d) long-term cycling stability of NCO/rGO//AC electrode, and (e) Ragone plot of the NCO/rGO//AC electrode.

3. Experimental Work

All chemical reagents used for the synthesis were of analytical grade and were employed without further purification. Nickel chloride ($\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$, $\geq 99\%$ purity) and cobalt chloride ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$, $\geq 99\%$ purity) were procured from Sigma-Aldrich, India. Urea ($\text{CO}(\text{NH}_2)_2$, $\geq 99.5\%$ purity) was obtained from Merck, India. Reduced graphene oxide (rGO, $\geq 98\%$ purity) was purchased from Graphene Supermarket (USA). All reagents were used as received without further purification, and deionized water was used throughout the synthesis.

3.1. Synthesis of $\text{NiCo}_2\text{O}_4/\text{rGO}$ Composite

To prepare NiCo_2O_4 , 0.1 M nickel chloride ($\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$) (solution A) and 0.2 M cobalt chloride ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$) (solution B) were dissolved in deionized water in separate beakers. Then solution B was added gradually to solution A while stirring. Separately, Urea (0.8 M) was dissolved in the deionized water and added dropwise to the above solution under continuous stirring. Urea acts as a slow and homogeneous precipitating agent during the hydrothermal process. Its gradual decomposition releases OH^- and CO_3^{2-} ions, enabling controlled nucleation and growth of nickel–cobalt precursor phases and leading to the formation of phase-pure NiCo_2O_4 . After 1 h of continuous stirring, the resulting solution was transferred to a 100 mL Teflon-lined stainless steel autoclave and heat-treated at 120°C for 8 h. After cooling down to room temperature, the precipitate was collected and washed with ethanol and deionized water several times. Further, the obtained product was dried at 60°C for 12 h. The dry product was calcined at 350°C for 3 h to attain the final product of NiCo_2O_4 .

For the preparation of $\text{NiCo}_2\text{O}_4/\text{rGO}$, the same procedure was followed with the addition of 0.06 g of rGO to the mixture solution prior to the hydrothermal treatment. In comparison, NiO and Co_3O_4 were prepared under similar conditions using only nickel chloride or cobalt chloride, respectively.

3.2. Electrochemical Measurements

The working electrodes (NiO, Co₃O₄, NCO, and NCO/rGO) were prepared as follows: a slurry was obtained by thoroughly mixing 80 wt% of the active material, 10 wt% carbon black (conductive agent), and 10 wt% poly(vinylidene fluoride) (PVdF, binder). N-methyl-2-pyrrolidone (NMP) was added dropwise to the mixture and ground for 15 min to form a homogeneous slurry. The resulting slurry was uniformly coated onto Ni foam and dried in a vacuum oven at 60 °C for 24 h.

Electrochemical measurements were performed in a three-electrode configuration using the material-coated Ni foam as the working electrode, Ag/AgCl as the reference electrode, and a Pt foil as the counter electrode in 3 M KOH electrolyte. For the fabrication of the asymmetric supercapacitor (ASC) device, the NCO/rGO electrode served as the positive electrode and activated carbon (AC) was used as the negative electrode, with 3 M KOH as the electrolyte and Whatman filter paper as the separator. Cyclic voltammetry (CV), galvanostatic charge–discharge (GCD), and electrochemical impedance spectroscopy (EIS) measurements were conducted using a Biologic SP-150e electrochemical workstation (France).

4. Conclusions

In this study, a series of electrode materials (NiO, Co₃O₄, NCO, and NCO/rGO) were successfully synthesized and evaluated for their electrochemical performance. Among them, the NCO/rGO composite exhibited the highest specific capacitance of 998 F g^{−1} in a three-electrode configuration, owing to the enhanced redox activity of NCO and the excellent electrical conductivity of rGO. The EIS analysis confirmed its low charge transfer resistance and superior ion diffusion characteristics. Furthermore, the NCO/rGO-based asymmetric supercapacitor, assembled with activated carbon as the negative electrode, delivered a high specific capacitance of 93.7 F g^{−1}, an energy density of 29.2 Wh kg^{−1}, and a power density of 750 W kg^{−1}, along with 85% capacitance retention after 5000 cycles. These results demonstrate that the NCO/rGO composite is a promising electrode material for high-performance energy storage applications.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/inorganics14020033/s1>, Figure S1: The particle size distribution histogram of (a) NiO, (b) Co₃O₄ and (c) NCO samples; Table S1: Specific capacitance comparison of present work with earlier reported results. References [26–31] are cited in the supplementary materials.

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Conflicts of Interest: The authors declare no conflicts of interest.

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