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Review

Co/Ni Oxide electrodes for High Performance Supercapacitors

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Abstract:
The growing demand for efficient and sustainable energy storage systems has stimulated extensive research into advanced supercapacitor electrode materials. Among the various transition metal oxides, those based on cobalt and nickel have emerged as promising candidates due to their rich redox behaviour, multiple oxidation states, and high theoretical capacitance. Notably, mixed Co/Ni oxides have demonstrated superior electrochemical performance compared to individual metal oxides, attributable to the synergistic interaction between cobalt and nickel ions. Recent advancements in nanostructure engineering have significantly enhanced the electrochemical properties of these materials by improving surface area, electrical conductivity, and ion/electron transport pathways. This review offers a comprehensive overview of Co/Ni oxide-based materials for high-performance supercapacitor applications. The fundamental mechanisms of charge storage and key electrochemical parameters are succinctly examined. Various synthesis methods, including hydrothermal, sol–gel, co-precipitation, and electrodeposition techniques, are analysed with a focus on their impact on morphology and electrochemical behaviour. Various nanostructured architectures, such as nanosheets, nanowires, nanoflowers, and hierarchical structures, are analysed with respect to their specific capacitance, rate capability, and cycling stability. Furthermore, the primary challenges related to conductivity, structural degradation, and large-scale fabrication are examined, alongside potential strategies for enhancing performance. Ultimately, future directions for the advancement of durable and efficient Co/Ni oxide supercapacitors are discussed.

Keywords: *Supercapacitors, pseudocapacitor, Co/Ni oxides, Nanostructured electrodes, Electrochemical performance*

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1. Introduction

The continuous growth of modern technologies, including portable electronic devices, electric vehicles, smart grids, and wearable systems, has created a strong demand for efficient and sustainable energy-storage devices.[1] [2]Conventional energy-storage systems such as batteries possess high energy density but often suffer from slow charging rates and limited power delivery. In contrast, conventional capacitors provide rapid power output but store only a small amount of energy. [3] To overcome these limitations, supercapacitors have emerged as an important class of electrochemical energy-storage devices because they combine fast charge–discharge capability, high power density, excellent reversibility, and long cycle life. Based on their charge-storage mechanism, supercapacitors are generally divided into three categories: electric double-layer capacitors (EDLCs), pseudocapacitors, and hybrid supercapacitors. [4], [5] EDLCs mainly store energy through electrostatic charge accumulation at the electrode–electrolyte interface and commonly use carbon-based materials as electrodes. Although they exhibit excellent cycling stability, their energy density is relatively low. Pseudocapacitors store energy through fast and reversible faradaic redox reactions occurring on the surface of electroactive materials such as transition metal oxides and conducting polymers, resulting in higher capacitance and energy density. Hybrid supercapacitors combine the features of EDLCs and pseudocapacitors to achieve improved overall electrochemical performance. [6] [7]Among the various electrode materials investigated for supercapacitor applications, transition metal oxides have attracted significant attention due to their multiple oxidation states, rich redox activity, high theoretical capacitance, and relatively low cost. [8] [9] These materials can participate in reversible electrochemical reactions, making them suitable for high-performance pseudocapacitive energy storage. [10] [11] In particular, cobalt- and nickel-based oxides have been widely explored because of their superior electrochemical properties and excellent redox behaviour. Co/Ni mixed oxides, especially binary oxides such as $NiCo_2O_4$, are considered promising electrode materials for supercapacitors because they exhibit better electrochemical performance than individual cobalt or nickel oxides. [12] [13] The synergistic interaction between cobalt and nickel ions improves electrical conductivity, enhances electrochemical activity, and provides multiple pathways for electron transfer. In addition, Co/Ni oxides can be synthesized in various nanostructured forms, including nanosheets, nanowires, nanoflowers, and hierarchical architectures, which increase the active surface area and facilitate efficient ion diffusion during electrochemical processes. [14] [15] [16]

Despite these advantages, Co/Ni oxides still face challenges such as moderate conductivity, structural degradation during repeated cycling, and limited rate capability at high current densities. [17][18]Therefore, extensive research has focused on improving their electrochemical performance through morphology engineering, composite formation, and optimized synthesis techniques. The present review summarizes recent progress in Co/Ni oxide-based electrode materials for supercapacitor applications. The review discusses the fundamentals of supercapacitors, structural and electrochemical properties of Co/Ni oxides, synthesis approaches, nanostructure design, and electrochemical performance. [19] [20] Furthermore, the major challenges and future prospects associated with Co/Ni oxide supercapacitors are highlighted to provide guidance for the development of advanced energy-storage materials.

2. Fundamentals of Supercapacitors

Supercapacitors, also referred to as electrochemical capacitors or ultracapacitors, represent advanced energy-storage devices that effectively bridge the gap between traditional dielectric capacitors and

rechargeable batteries. [21][22] These devices are characterized by their ability to deliver high power density, rapid charge-discharge rates, and exceptional cycling durability. Due to these attributes, supercapacitors have garnered considerable attention for their potential applications in portable electronics, electric vehicles, backup power systems, and renewable energy technologies. [23][24][25]The performance of a supercapacitor is primarily determined by its charge-storage mechanism and the electrochemical properties of the electrode materials. [26]

2.1 Charge Storage Mechanism

Supercapacitors store energy through two primary mechanisms classified as electric double-layer capacitors (EDLCs) or pseudocapacitors.

Electric Double-Layer Capacitor (EDLC) Mechanism

In electric double-layer capacitors (EDLCs), energy storage is facilitated through the electrostatic accumulation of ions at the interface between the electrode surface and the electrolyte. Upon the application of a potential, positive and negative ions from the electrolyte are drawn toward oppositely charged electrode surfaces, resulting in the formation of an electric double layer. [27], [28]As this process does not involve any chemical reactions, the charge-storage mechanism is highly reversible, thereby offering excellent cycling stability. Carbon-based materials, including activated carbon, graphene, carbon nanotubes, and porous carbon, are extensively utilized as EDLC electrode materials due to their large surface area, superior electrical conductivity, and chemical stability. [29][30]The capacitance in EDLCs is primarily dependent on the accessible surface area of the electrode and the effective interaction between electrolyte ions and the electrode surface. Although EDLCs demonstrate high power density and prolonged cycle life, their energy density is generally lower compared to that of pseudocapacitors. [31], [32]

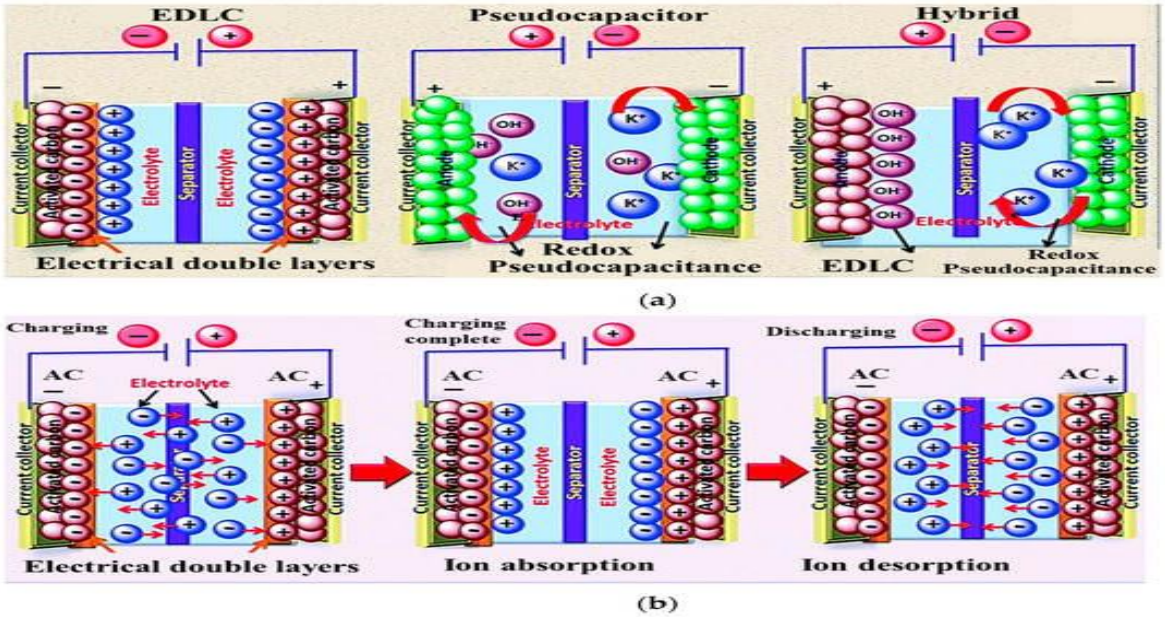

FIG 1:(a) Schematics of various types of capacitors. (b) Charge and discharge energy storage principles of an EDLC where black arrows represent ions in the electrolyte and red arrows reflect the direction of ion movement. [155]

Pseudocapacitive Mechanism

Pseudocapacitors store charge through rapid and reversible faradaic redox reactions occurring at or near the electrode surface. [33]This mechanism involves charge transfer between the electrode material and electrolyte ions, resulting in

significantly higher capacitance compared to electric double-layer capacitors (EDLCs). Transition metal oxides, metal hydroxides, and conducting polymers are frequently employed as pseudocapacitive electrode materials due to their multiple oxidation states and high electrochemical activity. [34][35] Among these materials, transition metal oxides such as cobalt oxide, nickel oxide, and mixed cobalt/nickel oxides have garnered considerable attention because they can undergo rapid oxidation–reduction reactions during electrochemical cycling. These redox processes contribute to enhanced charge-storage capability and improved energy density. [36] However, pseudocapacitive materials may experience structural degradation and reduced cycling stability compared to EDLC materials due to repeated ion insertion and extraction during charge–discharge processes. [37]

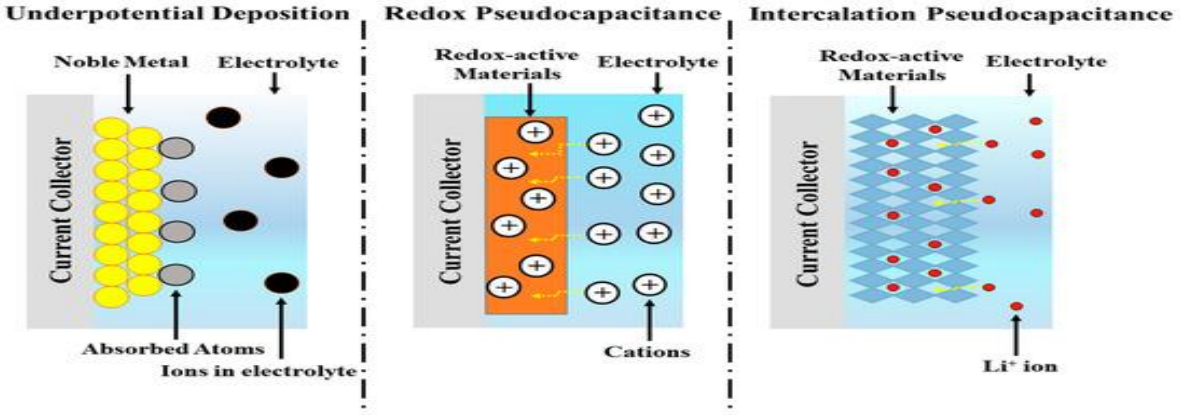

FIG 2 : Working mechanisms of pseudocapacitive electrodes. Red, black, grey and yellow squares represent redox-active materials, ions in electrolyte, absorbed atoms, and noble metal.[155]

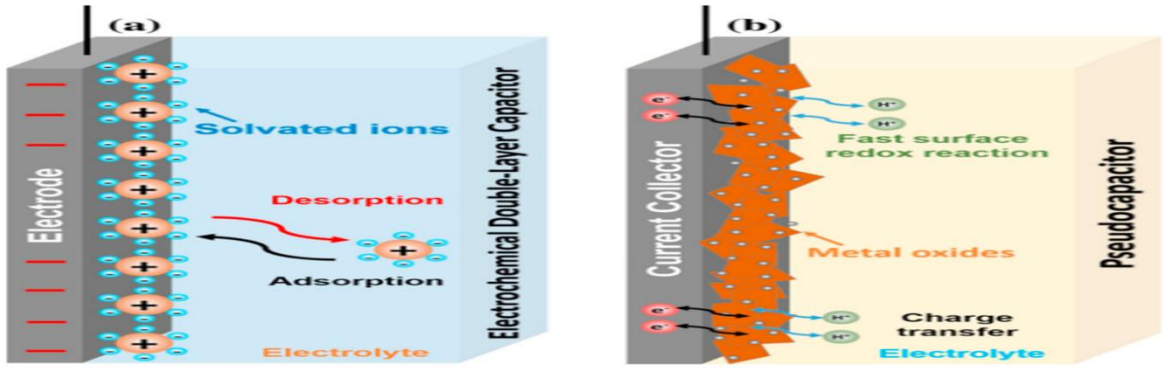

FIG 3 : Comparison of charging in (a) EDLC (carbon) and (b) pseudocapacitor (metal oxides) [156]

2.2 Key Electrochemical Parameters

The electrochemical performance of supercapacitors is assessed through several critical parameters, including specific capacitance, energy density, power density, and cycling stability. These parameters offer insights into the charge-storage capacity, energy-delivery efficiency, and long-term durability of the device. [38][39]

Specific Capacitance:

Specific capacitance is a critical parameter for assessing supercapacitor electrodes, as it quantifies the charge stored per unit mass, area, or volume of the active material. A higher specific capacitance signifies enhanced energy-storage capability of the electrode material. This parameter is typically derived from cyclic voltammetry (CV) or galvanostatic charge–discharge (GCD)

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measurements. The specific capacitance value is significantly affected by factors such as surface area, electrical conductivity, pore structure, morphology, and the electrochemical activity of the electrode material. [40]

Energy Density:

Energy density refers to the quantity of energy stored within a supercapacitor, typically measured in watt-hours per kilogram (Wh kg^{-1}). This parameter is crucial for assessing the practical utility of supercapacitors in energy-storage systems. While supercapacitors generally exhibit lower energy density compared to batteries, the incorporation of pseudocapacitive materials and hybrid systems can markedly enhance their energy-storage capabilities. Common strategies to augment energy density include expanding the operating voltage window and improving the capacitance of electrode materials. [41]

Power Density:

Power density is a critical parameter that quantifies the rate at which a supercapacitor can deliver stored energy, typically measured in watts per kilogram (W kg^{-1}). Supercapacitors are renowned for their high power density, attributable to their ability to charge and discharge rapidly with minimal energy loss. The power density of a supercapacitor is influenced by several factors, including internal resistance, electrical conductivity, ion diffusion rate, and electrode architecture. Materials characterized by high conductivity and porous structures generally demonstrate enhanced power performance. [42], [43]

Cycling Stability:

Cycling stability denotes the capacity of a supercapacitor to maintain its electrochemical performance following numerous charge–discharge cycles. This parameter is crucial for assessing the long-term durability and reliability of electrode materials. Materials based on electric double-layer capacitors (EDLCs) typically demonstrate excellent cycling stability, as their charge-storage process does not entail significant structural alterations. [44]. Conversely, pseudocapacitive materials may experience gradual performance degradation due to repeated redox reactions and structural stress during cycling. Consequently, enhancing the structural stability of electrode materials is imperative for the development of durable, high-performance supercapacitors. [45], [46]

3. Co/Ni Oxides as Electrode Materials

Transition metal oxides have emerged as promising electrode materials for supercapacitor applications due to their rich electrochemical activity and capacity for reversible redox reactions. Among these, cobalt- and nickel-based oxides have garnered significant attention owing to their high theoretical capacitance, multiple oxidation states, and favourable electrochemical reversibility. Notably, mixed Co/Ni oxides demonstrate superior performance compared to individual metal oxides, attributed to the synergistic interaction between cobalt and nickel ions. Their distinctive structural and electronic properties render them suitable candidates for high-performance pseudocapacitors.

3.1 Structural and Electrochemical Properties

Nickel Oxide (NiO)

Nickel oxide (NiO) is a widely investigated electrode material for supercapacitors, attributed to its high theoretical capacitance, environmental stability, and relatively low cost. Typically, NiO exhibits a cubic crystal structure and demonstrates excellent electrochemical behaviour through reversible redox reactions between Ni^{2+} and Ni^{3+} oxidation states in alkaline electrolytes. [47] [48] These reactions significantly contribute to its high pseudocapacitive performance and enhanced charge-storage capability. Nanostructured NiO materials, including nanosheets, nanowires, nanoparticles, and porous architectures, have demonstrated improved electrochemical performance due to their increased surface area and reduced ion-diffusion pathways. Nonetheless, the

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practical application of NiO remains constrained by its relatively poor electrical conductivity and structural instability during prolonged cycling. [49] [50], [51]

Table 1. Recent Structural and Electrochemical Properties of NiO for Supercapacitor Applications.

S. No.	NiO Material / Structure	Synthesis Method	Structural Properties	Morphological Properties	Electrochemical Properties	Specific Capacitance	Cycling Stability	Reference
1	NiO nanoparticles	Sol–gel	Cubic NiO phase, crystallite size ~27.9 nm	Spherical nanoparticles with porous surface	Good redox activity and ion diffusion	168.16 mAh g^{-1} at 6 A g^{-1}	62.51% after 4000 cycles	[118]
2	NiO/CNT nanocomposite	Spray pyrolysis + annealing	Improved crystallinity at 400 °C	CNT-connected porous network	Enhanced conductivity and charge transfer	745 F g^{-1} at 5 A g^{-1}	109% retention after 1000 cycles	[119]
3	NiO–Activated Carbon composite	Co-precipitation	Mixed oxide-carbon structure	Porous interconnected particles	Synergistic EDLC + pseudocapacitance behavior	Enhanced capacitance with AC addition	High cyclic durability	[120]
4	CeO_2/NiO nanocomposite	Hydrothermal	Crystalline mixed oxide phases	Nanograin composite morphology	Reduced charge-transfer resistance	Improved capacitance over pure NiO	Stable cyclic response	[121]
5	Printed NiO electrode	Thermal annealing	Improved crystallinity with annealing	Highly porous continuous structure	Lower resistance and better ion transport	19.54 mF cm^{-2}	96.4% retention after 1000 cycles	[122]
6	NiO–Mn_3O_4 nanocomposite	Electrospinning	Mixed oxide crystalline phases	Ribbon-like nanostructure	Improved electron transport and redox activity	Higher than pure NiO	Good cycling performance	[123]
7	NiO nanotubes	Hydrothermal	Nanocrystalline cubic NiO	Hollow nanotubular morphology	Large electrolyte-accessible area	Up to 2018 F g^{-1}	Nearly 100% after 500 cycles	[124]
8	NiO nanorods	Hydrothermal	Highly crystalline structure	Rod-like aligned arrays	Efficient OH^- ion diffusion	High pseudocapacitance	Excellent cyclic stability	[125]
9	Ni/NiO_x layered electrode	Layered deposition	Layered Ni/NiO_x interface	Thin oxide coating on Ni backbone	Stable cyclic charging/discharging behavior	Good capacitance retention	Long cycle life with minimal degradation	[126]
10	NiO/ZnO heterostructure film	Sol–gel spin coating	Heterostructured oxide layers	Uniform thin-film morphology	Synergistic charge storage behavior	1.627 F g^{-1}	Improved stability	[127]

Cobalt Oxide (Co_3O_4)

Cobalt oxide is a significant transition metal oxide extensively studied for its potential in energy storage applications. Co_3O_4 features a spinel crystal structure comprising both Co^{2+} and Co^{3+} ions, which offer multiple electroactive sites conducive to

reversible redox reactions. [52] [53]This mixed-valence property enhances charge-transfer capabilities and contributes to improved pseudocapacitive behaviour. Co_3O_4 demonstrates high theoretical capacitance and notable electrochemical activity, particularly when synthesized in nanostructured forms such as nanoflowers, hollow spheres, nanorods, and hierarchical architectures. [54], [55]These structures facilitate electrolyte penetration and enhance ion diffusion during electrochemical cycling. Despite these advantages, cobalt oxide is challenged by limited electrical conductivity and structural degradation following repeated charge–discharge cycles. [56] [57]

Table 2. Recent Structural and Electrochemical Properties of Co_3O_4 for Supercapacitor Application.

Sl. No.	Co_3O_4 Material/System	Synthesis Method	Crystal Structure / Phase	Morphology	Surface Area / Porosity	Specific Capacitance	Cycling Stability	Reference
1	Pure Co_3O_4 nanoparticles	Co-precipitation	Cubic spinel Co_3O_4	Spherical nanoparticles	Mesoporous	612 F g^{-1} at 1 A g^{-1}	91% after 5000 cycles	[128]
2	Co_3O_4 nanosheets	Hydrothermal	Spinel cubic phase	Ultrathin nanosheets	High porosity	742 F g^{-1}	89% after 3000 cycles	[129]
3	Co_3O_4 hollow spheres	Template-assisted	Cubic Co_3O_4	Hollow microspheres	Hierarchical pores	915 F g^{-1}	90% after 4000 cycles	[129]
4	Co_3O_4/carbon composite	Sol–gel	Cubic spinel	Carbon-coated nanoparticles	Enhanced ion diffusion	1350 F g^{-1}	96% retention	[129]
5	Co_3O_4/rGO composite	Hydrothermal + reduction	Spinel + graphene	Sheet-like composite	High conductivity network	1280 F g^{-1}	95% after 5000 cycles	[129]
6	Co_3O_4 nanowires	Solvothermal	Cubic spinel	1D nanowires	Large active surface	801 F g^{-1}	93% retention	[130]
7	Co_3O_4/CNT composite	Chemical deposition	Cubic spinel	CNT interconnected nanoparticles	Conductive porous matrix	1105 F g^{-1}	94% retention	[131]
8	Fe-doped Co_3O_4 thin film	Spray pyrolysis	Polycrystalline cubic	Spherical grains	Compact porous film	720 F g^{-1} at 2 mV s^{-1}	Improved stability	[132]
9	Porous Co_3O_4 nanoflowers	Hydrothermal	Spinel cubic phase	Flower-like architecture	High pore volume	980 F g^{-1}	92% after 5000 cycles	[133]
10	Mesoporous Co_3O_4 electrode	Chemical bath deposition	Spinel Co_3O_4	Mesoporous interconnected grains	High surface area	850 F g^{-1}	90% after long cycling	[134]

Nickel Cobalt Oxide ($NiCo_2O_4$)

Among cobalt- and nickel-based oxides, $NiCo_2O_4$ has garnered considerable attention due to its superior electrochemical performance relative to single-component oxides. [58] $NiCo_2O_4$ features a spinel crystal structure, wherein nickel and cobalt ions occupy distinct lattice positions, thereby enhancing electronic conductivity and redox activity. [59] [60] The presence of Ni^{2+}/Ni^{3+} and Co^{2+}/Co^{3+} redox couples offers numerous electrochemically active sites and facilitates rapid electron-transfer processes. [61] Furthermore, $NiCo_2O_4$ typically demonstrates higher electrical conductivity than individual NiO or Co_3O_4, leading to improved specific capacitance and rate capability. Various nanostructured forms of $NiCo_2O_4$, such as nanosheets, nanowires, hollow structures, and porous networks, have been developed to further augment electrochemical performance by increasing the active surface area and promoting ion transport. Owing to these advantages, $NiCo_2O_4$ is regarded as one of the most promising electrode materials for high-performance supercapacitor applications. [62] [63]

Table 3. Recent Structural and Electrochemical Properties of $NiCo_2O_4$for Supercapacitor Applications.

Sl. No.	$NiCo_2O_4$ Material/System	Synthesis Method	Crystal Structure / Phase	Morphology	Surface Area / Porosity	Specific Capacitance	Cycling Stability	Reference
1	Pure $NiCo_2O_4$ nanowires	Hydrothermal	Cubic spinel $NiCo_2O_4$	1D nanowires	Mesoporous structure	1120 F g^{-1} at 1 A g^{-1}	91% after 5000 cycles	[135]
2	$NiCo_2O_4$ nanosheets	Solvothermal	Inverse spinel cubic phase	Ultrathin nanosheets	High active surface area	1358 F g^{-1}	93% retention	[136]
3	Porous $NiCo_2O_4$ nanoflowers	Hydrothermal	Cubic spinel	Flower-like porous architecture	Hierarchical porosity	1480 F g^{-1}	92% after 4000 cycles	[137]
4	$NiCo_2O_4$ hollow spheres	Template-assisted synthesis	Spinel cubic phase	Hollow microspheres	Mesoporous shells	1265 F g^{-1}	90% retention	[138]
5	$NiCo_2O_4$/rGO composite	Hydrothermal + reduction	Spinel + graphene hybrid	Sheet-network composite	Conductive porous matrix	1820 F g^{-1}	95% after 5000 cycles	[139]
6	$NiCo_2O_4$/CNT composite	Chemical deposition	Cubic spinel	CNT interconnected nanoparticles	Enhanced ion pathways	1650 F g^{-1}	94% retention	[140]
7	Zn-doped $NiCo_2O_4$ spinel	Microwave-assisted hydrothermal	Zn-doped cubic spinel	Nanoparticles	Increased BET surface area	1705 F g^{-1}	Excellent long-cycle stability	[141]
8	$NiCo_2O_4$@$NiMoO_4$ heterostructure	Hydrothermal growth	Hierarchical spinel composite	Nanosheet arrays	High electroactive area	3.74 F cm^{-2}	Stable after repeated cycling	[142]
9	Branch-structured $NiCo_2O_4$ on Ni foam	One-step hydrothermal	Spinel $NiCo_2O_4$	Nanoplates@nanowires	Hierarchically porous	1380 F g^{-1} at 5 A g^{-1}	86.7% after 5000 cycles	[143]
10	$NiCo_2O_4$/carbon hybrid electrode	Sol–gel method	Cubic spinel hybrid	Carbon-coated nanoparticles	Improved conductivity and porosity	1900 F g^{-1}	96% capacitance retention	[144]

3.2 Advantages

Multiple Oxidation States

The primary advantage of Co/Ni oxides lies in the presence of multiple oxidation states in cobalt and nickel ions. These varied valence states facilitate rapid and reversible redox reactions during electrochemical cycling, thereby enhancing charge-storage capacity. Furthermore, the coexistence of different oxidation states improves electron-transfer processes and contributes to superior electrochemical reversibility. [64] [65]

Good Redox Activity

The remarkable redox activity of Co/Ni oxides constitutes a significant factor in their enhanced electrochemical performance. The reversible redox reactions occurring at the electrode surface facilitate efficient energy storage and rapid charge-transfer kinetics. [66] Additionally, the nanostructured Co/Ni oxides offer increased contact areas between the electrolyte and the electrode surface, thereby enhancing the electrochemical utilization of active materials. [67]

3.3 Limitations

Poor Conductivity

While Co/Ni oxides demonstrate commendable electrochemical activity, their inherent electrical conductivity remains comparatively low relative to highly conductive carbon materials. This limited conductivity impedes the rapid transport of electrons during charge–discharge processes, thereby adversely impacting rate capability and power performance. [68] [69], [70]

Structural Degradation

The repeated insertion and extraction of ions during electrochemical cycling can induce volume expansion and contraction within the electrode material. These structural alterations may result in cracking, pulverization, and a gradual loss of active material, thereby diminishing cycling stability and capacitance retention over prolonged operation. [71] [72]

4. Synthesis Methods

The electrochemical performance of Co/Ni oxide electrode materials is significantly influenced by their synthesis route. Various preparation methods can modulate the crystal structure, particle size, surface area, porosity, and morphology of the resulting materials, which directly impact ion diffusion and electron transport during electrochemical processes. [73] Consequently, selecting an appropriate synthesis method is crucial for achieving high-performance Co/Ni oxide electrodes for supercapacitor applications. Among the diverse preparation techniques, hydrothermal synthesis, sol–gel processing, co-precipitation, and electrodeposition are the most commonly employed methods due to their simplicity, controllability, and effectiveness in producing nanostructured materials. [74]

4.1 Hydrothermal Method

The hydrothermal method is a prevalent technique for synthesizing Co/Ni oxides with controlled morphology and high crystallinity. In this approach, metal precursors are dissolved in an aqueous solution and subjected to heating in a sealed autoclave under regulated temperature and pressure conditions. [75] The elevated temperature environment facilitates crystal growth and the formation of nanostructured materials. Hydrothermal synthesis is capable of producing a diverse array of morphologies, including nanosheets, nanowires, nanorods, nanoflowers, hollow spheres, and hierarchical architectures. [76] The morphology can be modulated by adjusting reaction temperature, precursor concentration, pH, and reaction time. Materials synthesized through this method typically exhibit high surface area and porous structures, which enhance electrolyte penetration and enable rapid ion transport. Electrochemically, Co/Ni oxides synthesized via

hydrothermal methods often demonstrate enhanced specific capacitance, good rate capability, and improved cycling stability due to their well-defined nanostructures and efficient charge-transfer pathways. However, this method may necessitate relatively long reaction times and specialized high-pressure equipment. [77]

4.2 Sol–Gel Method

The sol–gel method is a versatile chemical synthesis technique employed for the preparation of homogeneous metal oxide materials with controlled composition and particle size. In this process, metal salts or alkoxides are dissolved in a solution to form a colloidal sol, which gradually transitions into a gel through hydrolysis and condensation reactions. The resultant gel is subsequently dried and calcined to yield the final oxide material. [78] This method affords excellent control over chemical composition and uniform distribution of metal ions. Sol–gel synthesis is commonly utilized to produce nanoparticles, porous structures, and interconnected oxide networks characterized by relatively small particle sizes and high purity. [79] The sol–gel method is a versatile chemical synthesis technique employed for the preparation of homogeneous metal oxide materials with controlled composition and particle size. In this process, metal salts or alkoxides are dissolved in a solution to form a colloidal sol, which gradually transitions into a gel through hydrolysis and condensation reactions. The resultant gel is subsequently dried and calcined to yield the final oxide material. [80] This method affords excellent control over chemical composition and uniform distribution of metal ions. Sol–gel synthesis is commonly utilized to produce nanoparticles, porous structures, and interconnected oxide networks characterized by relatively small particle sizes and high purity. [81]

4.4 Electrodeposition

Electrodeposition is an electrochemical synthesis technique wherein active materials are directly deposited onto conductive substrates via controlled electrochemical reactions. In this process, metal ions from an electrolyte solution undergo reduction or oxidation under an applied potential or current, resulting in the formation of thin films or nanostructured oxide coatings on the substrate surface. [82] This technique offers several advantages, including low processing temperature, precise thickness control, rapid synthesis, and direct growth of active materials on current collectors. Electrodeposition can yield various morphologies such as nanosheets, porous films, nanoflakes, and interconnected network structures. [83] [84] Electrodeposited Co/Ni oxides frequently exhibit excellent electrochemical performance due to their strong adhesion to the substrate and reduced contact resistance. Direct deposition on conductive substrates enhances electron transport and improves the utilization of active materials. Furthermore, the porous morphology generated during deposition facilitates electrolyte diffusion and enhances rate performance. Despite these advantages, achieving uniformity and crystallinity during large-scale deposition can sometimes be challenging. [85] [86]Overall, the synthesis method plays a critical role in determining the structural and electrochemical properties of Co/Ni oxide electrode materials. Proper control of morphology, porosity, and particle size through suitable synthesis techniques can significantly enhance specific capacitance, rate capability, and cycling stability, thereby improving the overall performance of supercapacitors. [87]

Synthesis Method	Principle / Process	Typical Precursors	Temperature / Conditions	Morphology Obtained	Advantages	Limitations	Electrochemical Performance Features	References
Hydrothermal Synthesis	Chemical reactions occur in a sealed autoclave under high temperature and pressure to form crystalline nanostructures	Nickel nitrate, cobalt nitrate, urea, ammonium fluoride	120–220 °C for 6–24 h	Nanorods, nanosheets, nanoflowers, hierarchical structures	Excellent crystallinity, controlled morphology, high porosity	Long reaction time, autoclave requirement	High specific capacitance, improved ion diffusion, enhanced cycling stability	[145]
Sol–Gel Method	Formation of a homogeneous colloidal sol followed by gelation and calcination	Metal alkoxides, cobalt acetate, nickel acetate, citric acid	Gelation at room temperature; calcination at 300–600 °C	Porous nanoparticles, interconnected networks	High purity, compositional uniformity, low-temperature processing	Shrinkage and cracking during drying	Large surface area and improved redox activity	[146]
Co-Precipitation	Simultaneous precipitation of metal hydroxides from solution using precipitating agents	Ni(NO3)2, Co(NO3)2, NaOH, NH4OH	Ambient to 90 °C followed by annealing	Nanoparticles and agglomerated clusters	Simple, cost-effective, scalable	Particle agglomeration and lower crystallinity	Good capacitance with economical large-scale production	[147]
Electrodeposition	Electrochemical deposition of active material directly onto conductive substrates	Nickel sulfate, cobalt sulfate, chloride salts	Room temperature under controlled current/voltage	Thin films, porous coatings, nanosheets	Binder-free electrodes, precise thickness control	Limited mass loading, substrate dependent	Low resistance and excellent rate capability	[148]
Chemical Bath Deposition (CBD)	Controlled deposition from aqueous precursor solution via slow release of ions	Metal nitrates, ammonia, thiourea, urea	60–95 °C in aqueous bath	Uniform thin films and nanoflakes	Low cost, easy scale-up, simple equipment	Lower crystallinity without annealing	Good surface accessibility and moderate capacitance	[149]
Combustion Synthesis	Exothermic redox reaction between fuel and oxidizer produces oxide powders rapidly	Metal nitrates with urea or glycine fuel	300–500 °C rapid heating	Porous nanopowders	Fast synthesis, energy efficient	Difficult morphology control	High surface area and rapid charge transfer	[150]
Microwave-Assisted Synthesis	Microwave irradiation accelerates nucleation and crystal growth	Metal nitrates, ethylene glycol, urea	Few minutes to 1 h under microwave heating	Uniform nanoparticles and nanosheets	Rapid synthesis, reduced energy consumption	Specialized equipment required	Enhanced conductivity and fast ion transport	[151]

Table 4: Common Synthesis Methods for Co/Ni Oxide Electrodes for Supercapacitor Applications

5. Nanostructured Co/Ni Oxides

Nanostructure engineering has emerged as an effective approach for enhancing the electrochemical performance of Co/Ni oxide electrode materials in supercapacitor applications. The electrochemical behaviour of transition metal oxides is significantly influenced by their morphology, particle size, surface area, and pore structure. [88] Designing materials at the nanoscale can substantially improve the accessibility of electroactive sites, reduce ion-diffusion pathways, and enhance electron transport during charge–discharge processes. [89] Various nanostructured architectures, including nanosheets, nanowires, nanoflowers, and hollow or hierarchical structures, have been extensively investigated to address the limitations of bulk Co/Ni oxides. [90] This nanostructures offer large active surface areas and

improved contact between the electrolyte and electrode surface, resulting in enhanced specific capacitance, superior rate capability, and improved cycling stability. [91][92]

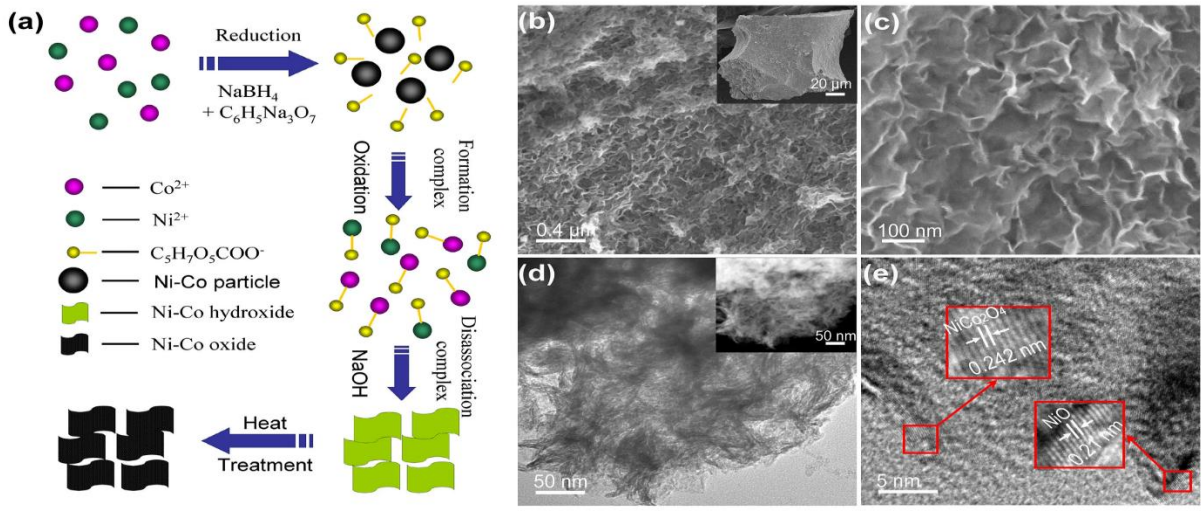

FIG 4 :(a) A schematic show of this facile process of synthesizing Ni-Co oxide nanosheets electrode materials. SEM images (b) and (c), inset image in (b) showing a typical Ni-Co oxide particle. TEM image (d) and the corresponding annular dark field scanning TEM (ADF-STEM) image (inset) and (e) high resolution TEM (HRTEM) image of Ni-Co oxide. The lattice spacing of around 0.242 nm and 0.21 nm are in good agreement of with the data of (311) plane for $NiCo_2O_4$ and (200) plane for NiO, respectively [157]

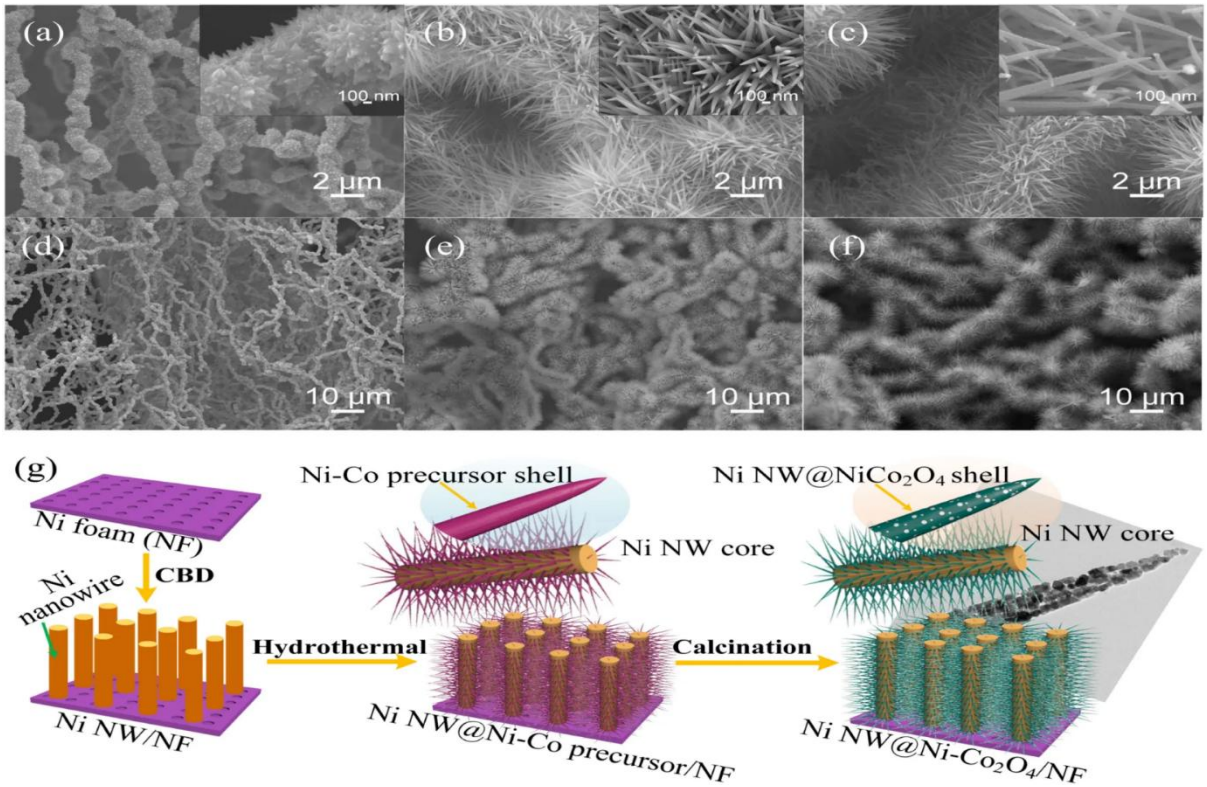

FIG 5: (a,d) Scanning electron microscope (SEM) images of Ni MW/NF. (b,e) SEM images of Ni NW@Ni–Co precursor/NF. (c,f) SEM images of Ni NW@$NiCo_2O_4$/NF. (g) Formation mechanism of the Ni NW@$NiCo_2O_4$/NF.[158]

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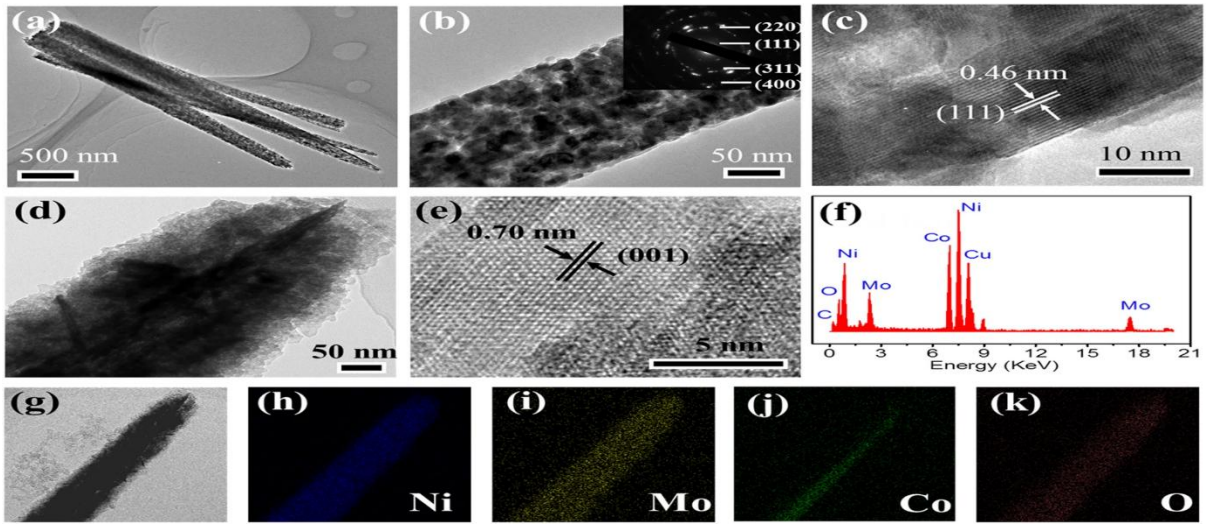

FIG 6: TEM and elemental analysis results of as prepared electrode materials. (a,b) TEM images of $NiCo_2O_4$ naowires. (c) HR-TEM image of a single $NiCo_2O_4$ naowire. (d) TEM image of an individual hierarchical core-shell $NiCo_2O_4$@$NiMoO_4$ nanowire. (e) HR-TEM image of an individual $NiMoO_4$ nanosheet outside. (f–k) EDS and elemental mapping of a single hierarchical core-shell $NiCo_2O_4$@$NiMoO_4$ nanowire under TEM mode.[159]

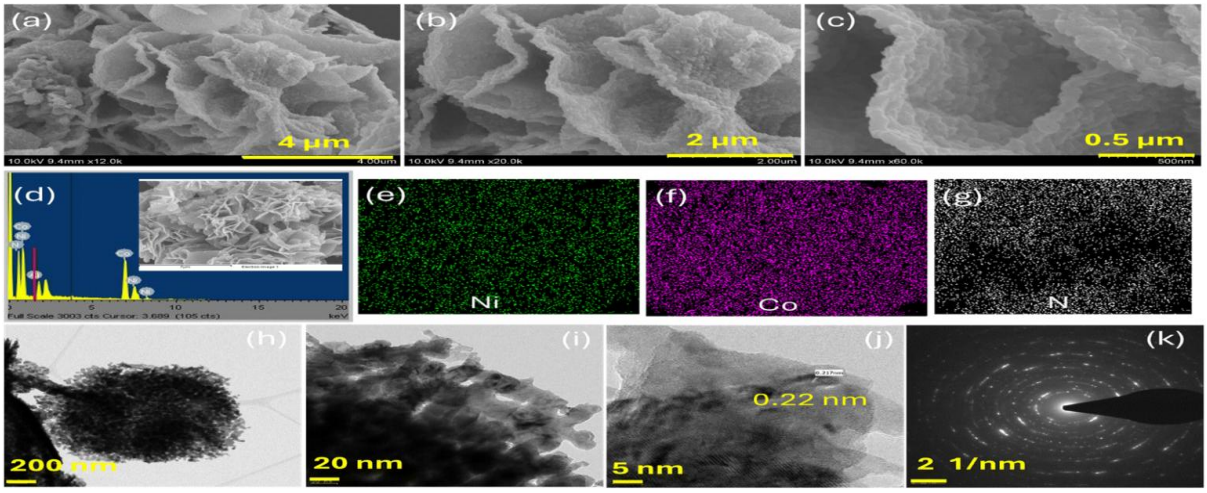

FIG 7 : Fig. 7a–c presents SEM images of the NiCoN heterostructures at different magnifications. The SEM images reveal a nanoporous, flower-like structure composed of highly rough nanosheets. These nanosheets were firmly interconnected, facilitating efficient charge transfer between the active components of NiCoN and the current collector. The loosely arranged nanostructures along with abundant interconnected open spaces, clearly indicate a large electroactive surface area, which is beneficial for enhancing the electrode's electrochemical performance. Additionally, energy-dispersive X-ray spectroscopy (EDS) analysis was performed to investigate the elemental composition and distribution within the NiCoN material. The EDS spectrum, as shown in Fig. 7d, confirms the presence of Ni, Co, and Ni atoms within the synthesized material. EDS mapping images (Fig. 7e–g) further demonstrate the uniform distribution of these elements throughout the NiCoN electrodes, underscoring the homogeneity of the material. Furthermore, the morphological and crystalline

characteristics of the NiCoN electrodes were analyzed using TEM and high-resolution TEM (HR-TEM). The TEM images in Fig. 7h and i depict the hierarchical, flower-like structure composed of porous nanosheets, which aligns with the SEM observations. The measured *d*-spacing of 0.22 nm corresponds to the (002) crystal planes of NiCoN, as confirmed through detailed analysis (Fig. 7j). The selected area electron diffraction (SAED) pattern in Fig. 7k displays a series of well-defined concentric rings, confirming the polycrystalline nature of the NiCoN material.[160]

6. Electrochemical Performance

The electrochemical performance of Co/Ni oxide electrode materials is significantly influenced by their composition, crystal structure, morphology, and synthesis method. Parameters such as specific capacitance, rate capability, cycling stability, and energy–power characteristics are commonly employed to assess the suitability of these materials for supercapacitor applications. In recent years, nanostructured Co/Ni oxides have exhibited remarkable electrochemical properties due to their enhanced surface area, improved conductivity, and efficient ion/electron transport pathways.

Specific Capacitance Comparison

The specific capacitance is a critical parameter for evaluating the charge-storage capacity of supercapacitor electrodes. Co/Ni oxide materials typically demonstrate significantly higher capacitance compared to carbon-based EDLC materials, attributable to their faradaic redox behaviour. Among the various Co/Ni oxides, mixed oxides such as $NiCo_2O_4$ frequently exhibit superior electrochemical performance relative to individual NiO or Co_3O_4, due to the synergistic interaction between cobalt and nickel ions. [93] [94]The capacitance values of Co/Ni oxides are significantly affected by their morphology and synthesis conditions. Nanostructured materials, including nanosheets, nanowires, and hierarchical architectures, offer increased electroactive sites and enhance electrolyte accessibility, thereby improving capacitance. Porous structures also promote efficient ion diffusion and enhance the utilization of active materials during electrochemical cycling. Generally, nanostructures synthesized hydrothermally exhibit higher capacitance due to their well-defined morphology and larger active surface area. Similarly, electrodeposited thin films often show improved electrochemical utilization as a result of direct contact with conductive substrates. [95] [96]

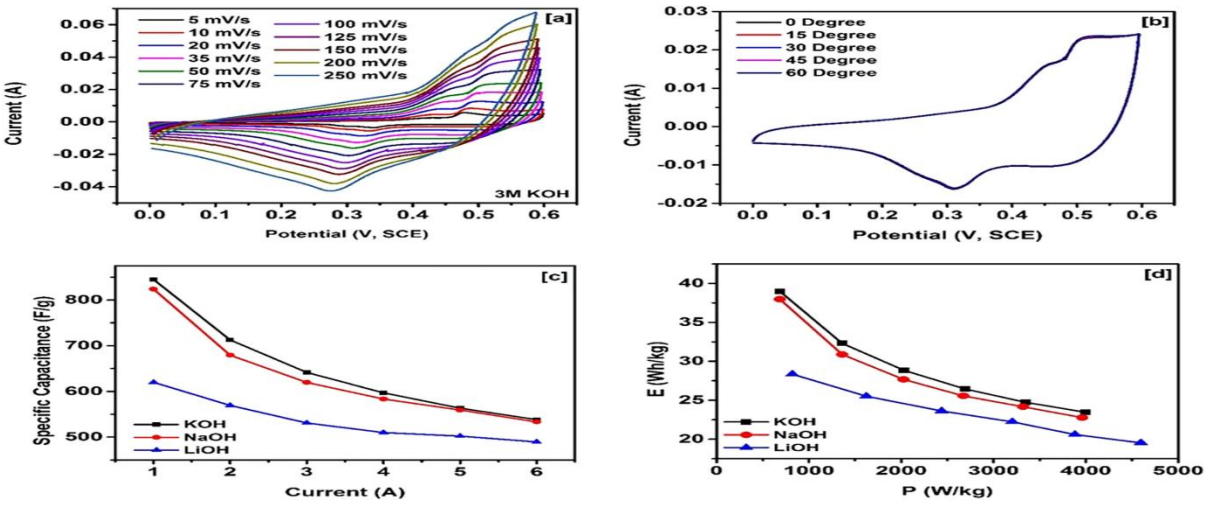

FIG 8: (a) CV curves of $NiCo_2O_4$ at various scan rates in KOH electrolyte, (b) CV curves of $NiCo_2O_4$ at various bending angles in KOH, (c) Charge-discharge characteristics of $NiCo_2O_4$ in various electrolytes and (d) Ragone plots of $NiCo_2O_4$ in various electrolytes.[161]

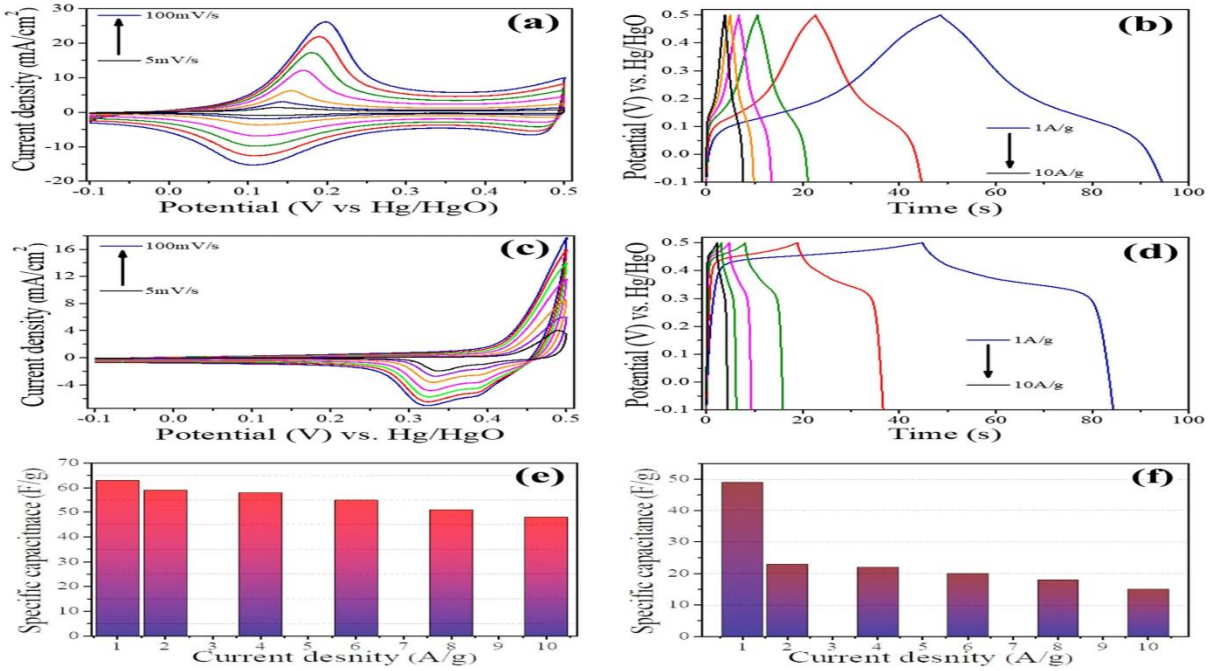

Fig 9 : (a) CV profile of Co sample at the scan rates of 5–100 mV/s. (b) GCD curve of Co sample at current densities of 1–10 A/g. (c) CV profile of Ni sample at the scan rates of 5–100 mV/s. (d) GCD curve of Ni sample at current densities of 1–10 A/g, (e) current density vs specific capacitance plot of Co sample and (f) current density vs specific capacitance plot of Ni electrode. [162]

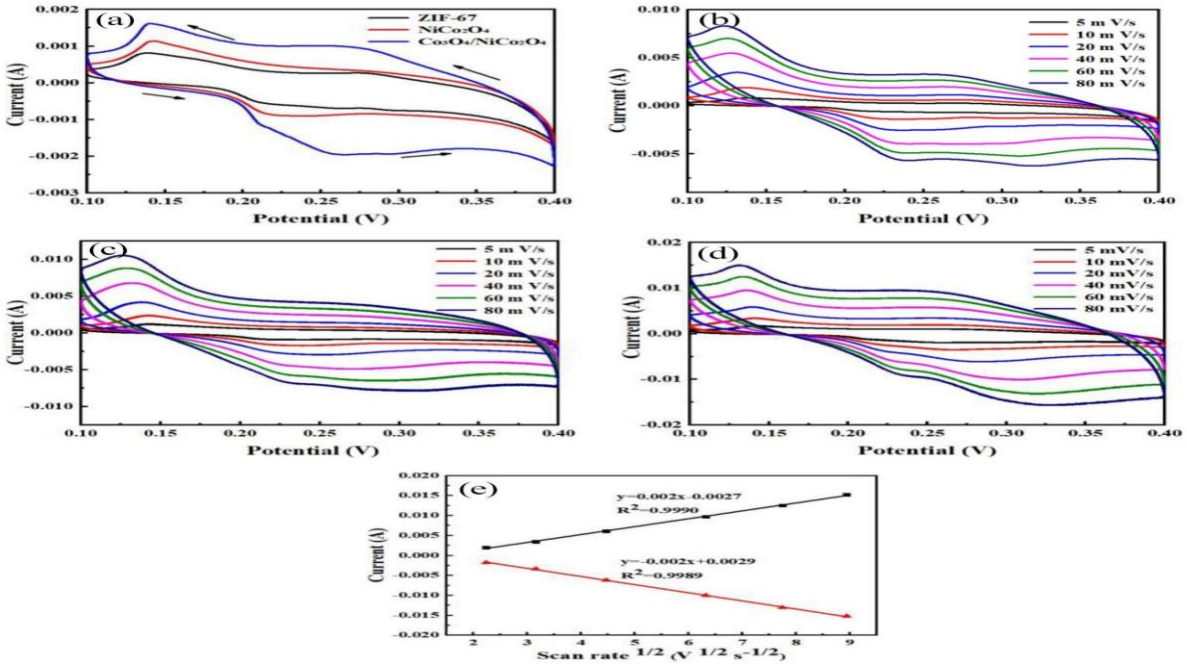

FIG 10: cyclic voltammograms of (a) ZIF-67, $NiCo_2O_4$, and $Co_3O_4/NiCo_2O_4$ at a scan of 5 mV s^{-1}, (b–d) ZIF-67, $NiCo_2O_4$, and $Co_3O_4/NiCo_2O_4$ at different scan rates, and (e) square root of scan rate versus current density for $Co_3O_4/NiCo_2O_4$.[163]

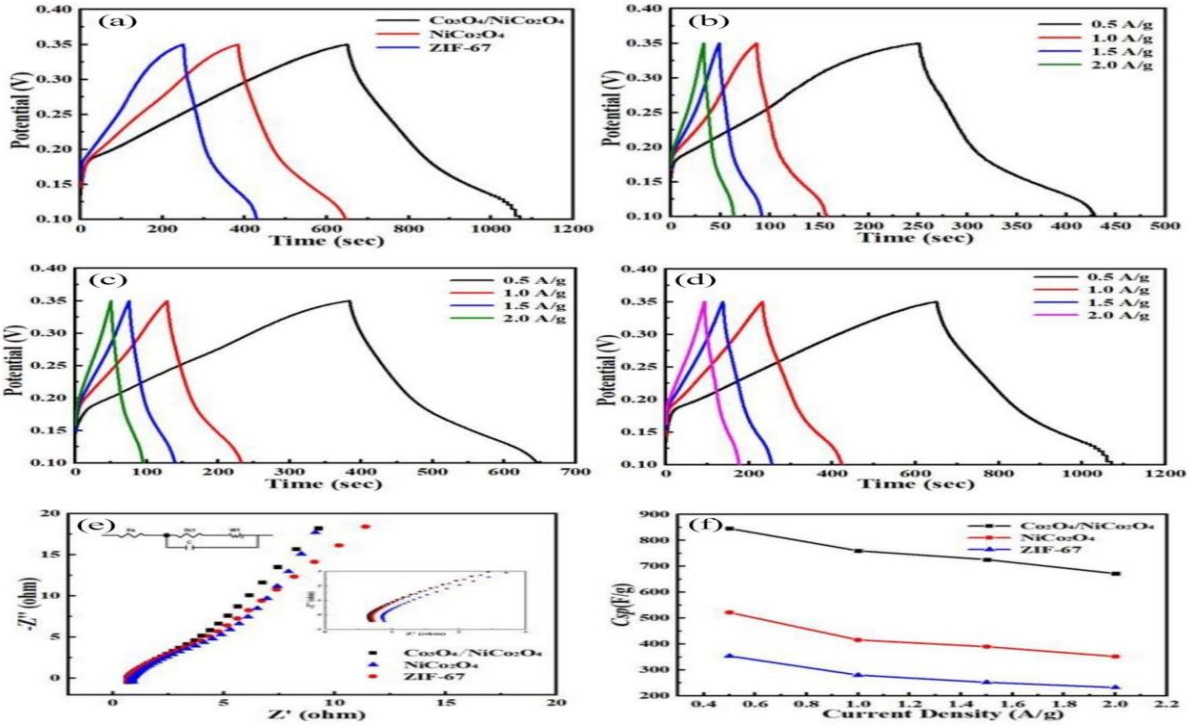

FIG 11: GCD curves (a) several electrodes at a current of 0.5 A g^{-1}; (b–d) ZIF-67, $NiCo_2O_4$, and $Co_3O_4/NiCo_2O_4$ in 6.0 M KOH with different current densities; (e) the electrochemical impedance spectra of ZIF-67, $NiCo_2O_4$, and $Co_3O_4/NiCo_2O_4$; and (f) the specific capacitance of ZIF-67, $NiCo_2O_4$, and $Co_3O_4/NiCo_2O_4$..[163]

7. Challenges and Future Perspectives

Co/Ni oxide-based electrode materials demonstrate considerable promise for high-performance supercapacitor applications, attributed to their extensive redox chemistry, high theoretical capacitance, and adaptable nanostructures. Despite significant progress in recent years, several challenges persist that hinder their practical implementation in commercial energy-storage systems. Addressing these limitations is essential for the advancement of durable, efficient, and economically feasible supercapacitor devices. Therefore, future research should focus on improving conductivity, structural stability, scalable fabrication methods, and advanced device integration. [97] [98]

Conductivity Issues

One of the primary limitations of Co/Ni oxides is their relatively low intrinsic electrical conductivity. This poor conductivity impedes rapid electron transport during charge–discharge processes, leading to diminished rate capability and reduced power performance. Although nanostructure engineering can partially enhance electron-transfer pathways, conductivity remains a significant challenge, particularly at high current densities. [99] To address this limitation, researchers have investigated various strategies, such as integrating Co/Ni oxides with conductive carbon materials, including graphene, carbon nanotubes, and activated carbon. [100]Additionally, conductive polymer coatings and heterostructure formation have demonstrated promising results in enhancing electrical transport properties. [101] [102]

Stability Limitations

One of the significant challenges associated with Co/Ni oxide electrodes is their long-term cycling stability. Repeated charge–discharge cycles can lead

to continuous ion insertion and extraction, resulting in volume expansion, structural deformation, and mechanical stress within the electrode material. These structural alterations may cause cracking, agglomeration, and a gradual loss of electroactive sites, ultimately diminishing capacitance retention. [103] Nanostructured architectures, such as hollow spheres, porous networks, and hierarchical structures, have demonstrated enhanced structural stability due to their ability to better accommodate volume changes during cycling. Nevertheless, maintaining high capacitance over extended periods remains challenging under practical working conditions. Future research should prioritize enhancing mechanical integrity and structural durability through surface engineering, composite formation, and binder-free electrode design. [104], [105]

Scalable Synthesis

Numerous high-performance Co/Ni oxide nanostructures are synthesized through laboratory-scale techniques that may not be amenable to large-scale industrial production. Techniques such as hydrothermal synthesis often necessitate elevated temperatures, prolonged reaction durations, and specialized equipment, which can escalate production costs and constrain commercial viability. [106] For practical applications, synthesis methods must be cost-effective, energy-efficient, environmentally sustainable, and readily scalable. Consistent control over morphology, particle size, and composition is also essential to ensure reproducible electrochemical performance in mass production. Consequently, future research should prioritize the development of straightforward and scalable synthesis strategies capable of producing high-quality Co/Ni oxide materials with controlled nanostructures. [107] [108]

Flexible and Hybrid Devices

The growing demand for wearable electronics and portable energy-storage systems has intensified interest in flexible and lightweight supercapacitor devices. Materials based on Co/Ni oxides exhibit superior electrochemical properties; however, their integration into flexible systems remains challenging due to issues related to mechanical flexibility, adhesion, and structural stability under bending conditions. [109][110] Hybrid supercapacitors, which combine battery-type and capacitive electrodes, have also attracted attention because they can enhance energy density while maintaining high power performance. [111] The incorporation of Co/Ni oxides into asymmetric and hybrid device configurations has shown promising results, yet optimization of electrode balancing, electrolyte compatibility, and device architecture remains necessary. [112] [113]

Future Research Directions

Significant progress has been made in the development of Co/Ni oxide-based supercapacitor electrodes; however, further advancements are necessary to satisfy the growing demands of contemporary energy-storage technologies. [114] Future research should concentrate on several critical areas: the development of multi-component and doped Co/Ni oxides with enhanced conductivity and electrochemical activity; the design of hierarchical and defect-engineered nanostructures to improve ion/electron transport; the exploration of binder-free and self-supported electrode architectures; the integration of Co/Ni oxides with conductive carbon materials and polymers; the investigation of flexible, wearable, and solid-state supercapacitors. [115] The use of advanced characterization techniques to elucidate charge-storage mechanisms and degradation behaviour and the development of environmentally friendly and scalable synthesis methods for industrial applications. Additionally, theoretical modelling and computational studies could aid in predicting material behaviour and guiding the rational design of next-generation electrode materials. [116] The combination of experimental and theoretical approaches will be crucial in expediting the

development of highly efficient and durable Co/Ni oxide supercapacitors. [117] Overall, Co/Ni oxide-based materials continue to be highly promising candidates for advanced supercapacitor applications. Ongoing efforts to enhance conductivity, stability, and large-scale fabrication are anticipated to further augment their practical potential in future energy-storage technologies.

8. Conclusion

Co/Ni oxide-based materials have emerged as highly promising electrode candidates for advanced supercapacitor applications due to their excellent redox activity, multiple oxidation states, and high theoretical capacitance. In recent years, significant progress has been made in enhancing the electrochemical performance of these materials through nanostructure engineering, morphology control, and optimized synthesis techniques. Various architectures, such as nanosheets, nanowires, nanoflowers, and hierarchical porous structures, have demonstrated enhanced surface area, improved ion diffusion, and efficient electron transport, resulting in superior capacitance, good rate capability, and improved cycling stability. Among the different cobalt- and nickel-based oxides, mixed oxides such as $NiCo_2O_4$ have garnered particular attention due to the synergistic interaction between cobalt and nickel ions, which enhances electrical conductivity and electrochemical activity compared to single-component oxides. Furthermore, advanced synthesis methods, including hydrothermal, sol–gel, co-precipitation, and electrodeposition techniques, have enabled the fabrication of well-defined nanostructures with tunble morphology and improved electrochemical behaviour. Despite these advances, several challenges remain for the practical implementation of Co/Ni oxide supercapacitors. Limitations such as poor intrinsic conductivity, structural degradation during long-term cycling, and difficulties in scalable production continue to affect device performance and commercialization potential. Therefore, further research is necessary to develop more stable, conductive, and economically viable electrode materials. Future progress in this field is expected to focus on hybrid material design, defect engineering, flexible electrode architectures, and environmentally friendly synthesis approaches. The integration of Co/Ni oxides with conductive carbon materials, polymers, and advanced nanostructures may further improve electrochemical performance and device durability. Additionally, the development of flexible and solid-state supercapacitors could expand the application of these materials in wearable electronics and portable energy-storage systems. Overall, Co/Ni oxides continue to play an important role in the development of next-generation supercapacitors. With continued advancements in material design, synthesis strategies, and device engineering, these materials are expected to contribute significantly to high-performance and sustainable energy-storage technologies in the future.

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