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ELECTROCHEMICAL INVESTIGATION OF METAL OXIDE NANOCOMPOSITES FOR SUPERCAPACITOR APPLICATION

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Abstract

The growing demand for efficient energy-storage technologies has increased interest in metal oxide nanocomposites as promising electrode materials for supercapacitor applications. However, limited charge-storage capacity, electrical conductivity, and long-term electrochemical stability continue to restrict the performance of conventional electrode materials. This study investigates the structural, morphological, and electrochemical properties of nickel cobaltite/manganese dioxide ($NiCo_2O_4/MnO_2$) nanocomposites for potential application in high-performance supercapacitors. An experimental laboratory-based design was developed in Lahore, Pakistan, using a hydrothermal synthesis approach. Four electrode formulations containing different $NiCo_2O_4/MnO_2$ weight ratios were considered, alongside individual $NiCo_2O_4$ and MnO_2 reference materials. The synthesized materials were evaluated through X-ray diffraction, scanning electron microscopy, Fourier-transform infrared spectroscopy, cyclic voltammetry, galvanostatic charge-discharge, and electrochemical impedance spectroscopy. Results indicate that the 70:30 $NiCo_2O_4/MnO_2$ formulation achieved the highest specific capacitance of 512.6 F/g at a current density of 1 A/g, compared with 286.4 F/g for $NiCo_2O_4$ and 148.7 F/g for MnO_2. The optimized composite also demonstrated 91.8% capacitance retention after 5,000 charge-discharge cycles. These findings suggest that combining nickel cobaltite and manganese dioxide improves electrochemical activity and charge-storage performance. The investigation provides a research framework

for developing cost-effective nanostructured electrode materials for advanced energy-storage technologies.

INTRODUCTION

The increasing global demand for electrical energy has encouraged the development of advanced energy-storage systems capable of providing high power density, rapid charging, operational stability, and improved energy efficiency. Conventional energy-storage technologies, particularly rechargeable batteries and dielectric capacitors, have limitations associated with charging time, power delivery, energy density, and operational lifespan. Supercapacitors have emerged as promising alternatives because they combine rapid charge-discharge characteristics with relatively long cycling stability.

Supercapacitors store electrical energy through electrostatic charge accumulation at the electrode-electrolyte interface and reversible electrochemical reactions occurring within electroactive materials. Based on their charge-storage mechanisms, these devices are generally classified into electric double-layer capacitors, pseudocapacitors, and hybrid supercapacitors. Electric double-layer capacitors primarily depend on electrostatic charge accumulation, whereas pseudocapacitors involve fast and reversible surface or near-surface redox reactions.

Transition metal oxides have received considerable research attention because of their multiple oxidation states, electrochemical activity, relatively accessible synthesis routes, and potential for reversible redox reactions. Materials such as manganese dioxide (MnO_2), nickel oxide (NiO), cobalt oxide (Co_3O_4), and nickel cobaltite ($NiCo_2O_4$) demonstrate promising characteristics for electrochemical energy storage.

Recent research has demonstrated that combining transition metal oxides with other electroactive materials can improve charge-transfer behaviour, electrode conductivity, and electrochemical stability. Alharbi (2025) reviewed the development of transition-metal-based electrode materials and emphasized the importance of material composition, structural modification, and electrode architecture in improving supercapacitor performance.

Similarly, Santhosh et al. (2024) highlighted the potential of nickel cobaltite-based nanocomposites and their combinations with different functional materials for improving electrochemical energy-storage characteristics. Among different metal oxide combinations, $NiCo_2O_4/MnO_2$ represents an interesting material system. Nickel cobaltite offers multiple accessible oxidation states and relatively favourable electrical conductivity, while manganese dioxide contributes electrochemical activity and abundant redox-active sites. Their combination provides an opportunity to investigate possible synergistic effects.

Recent experimental investigations have also reported promising outcomes from different composite electrode systems. Godlaveeti et al. (2024) reported a specific capacitance of approximately 451 F/g for CuS/MnO_2 nanocomposite electrodes at 1 A/g. Nageib et al. (2024) demonstrated the electrochemical potential of $UiO\text{-}66/ZrO_2$ composites, reporting 913.8 F/g under their experimental conditions. These studies demonstrate the importance of investigating composition-dependent electrochemical performance.

Problem Statement

Despite considerable progress in supercapacitor electrode development, achieving an appropriate balance between specific capacitance, electrical conductivity, charge-transfer resistance, and long-term cycling stability remains challenging.

Individual metal oxides frequently exhibit limitations associated with low electrical conductivity, particle aggregation, restricted electroactive surface accessibility, and structural changes during repeated electrochemical reactions.

MnO_2 offers attractive electrochemical properties but often experiences conductivity-related limitations. $NiCo_2O_4$ provides improved redox activity and conductivity, although its performance depends on particle morphology, electrode preparation, and electrolyte conditions.

The combination of these materials may improve their individual limitations. However, the electrochemical contribution of different $NiCo_2O_4/MnO_2$ compositions requires systematic investigation under controlled experimental conditions.
Therefore, this study examines the influence of nanocomposite composition on structural properties, electrochemical behaviour, specific capacitance, and cycling stability.

Research Objectives
The study aims to:
1. Synthesize $NiCo_2O_4/MnO_2$ nanocomposites through a hydrothermal preparation method.
2. Investigate the structural and morphological characteristics of synthesized nanocomposites.
3. Evaluate electrochemical performance through cyclic voltammetry, galvanostatic charge-discharge, and electrochemical impedance spectroscopy.
4. Determine the influence of different material compositions on specific capacitance and charge-transfer resistance.
5. Examine the cycling stability and potential suitability of optimized nanocomposites for supercapacitor applications.

Research Questions
1. How does the composition of $NiCo_2O_4/MnO_2$ nanocomposites influence their structural and morphological properties?
2. Which composite formulation demonstrates the highest specific capacitance?
3. How does nanocomposite composition affect charge-transfer resistance?
4. What is the relationship between current density and specific capacitance?
5. How effectively does the optimized nanocomposite retain its electrochemical performance during repeated charge-discharge cycles?

Research Hypotheses
- **H1:** $NiCo_2O_4/MnO_2$ nanocomposites demonstrate higher specific capacitance than their individual metal oxide components under identical experimental conditions.
- **H2:** Changes in $NiCo_2O_4/MnO_2$ composition significantly influence electrochemical charge-transfer resistance.
- **H3:** The optimized nanocomposite demonstrates improved cycling stability compared with individual electrode materials.

Literature Review
Supercapacitors are electrochemical energy-storage devices known for their high power density, rapid charging capability, and relatively long operational lifetimes. Their electrochemical performance is influenced by several factors, including electrode composition, electrolyte selection, surface morphology, operating potential window, and charge-transfer characteristics. Based on their charge-storage mechanisms, supercapacitors are generally classified into electric double-layer capacitors and pseudocapacitors. Electric double-layer capacitors commonly employ carbon-based materials, such as activated carbon, graphene, and carbon nanotubes, which store charge through electrostatic adsorption at the electrode–electrolyte interface. In contrast, pseudocapacitors rely on reversible faradaic reactions involving the transfer of electrons between electroactive materials and electrolyte ions. Transition metal oxides are particularly important in this category because their multiple oxidation states support electrochemical charge-transfer processes. Their performance depends on the availability of active sites, electrolyte accessibility, ionic diffusion, and electrical conductivity. Consequently, the development of nanostructured composite electrodes offers an effective research direction for improving charge-storage characteristics.

2.2 Nickel Cobaltite as an Electrode Material

Nickel cobaltite ($NiCo_2O_4$) is a mixed transition-metal oxide that has attracted considerable attention in electrochemical energy-storage research because of its redox activity and comparatively favourable electrical conductivity. Its charge-storage behaviour is associated with reversible oxidation–reduction reactions involving nickel and cobalt species, which facilitate electron transfer during electrochemical cycling. In addition, its structural flexibility makes it suitable for hydrothermal synthesis and the preparation of different nanostructured electrode architectures. Santhosh et al. (2024) discussed the development of $NiCo_2O_4$-based nanocomposites and highlighted the potential of combining nickel cobaltite with other functional materials to improve electrochemical performance. Similarly, Ayman et al. (2025) investigated a polyindole/nickel cobaltite composite electrode and reported higher specific capacitance than the individual reference materials under their experimental conditions. These findings support the continued investigation of nickel cobaltite as an active electrode component, particularly in combination with other electrochemically active materials.

2.3 Manganese Dioxide and Its Electrochemical Properties

Manganese dioxide (MnO_2) is another important transition-metal oxide widely investigated for electrochemical energy-storage applications. Its relevance is associated with its relatively low material cost, environmental compatibility compared with several alternative electrode materials, and multiple electrochemically accessible manganese oxidation states. MnO_2 exhibits pseudocapacitive behaviour through reversible redox reactions occurring at or near its surface. These reactions contribute to charge storage and make the material suitable for supercapacitor electrode development. However, its relatively low intrinsic electrical conductivity can restrict electron transport and limit electrochemical performance, particularly at higher current densities. The preparation of MnO_2-based nanocomposites offers a potential approach to addressing these limitations by improving electrical pathways, modifying surface morphology, and increasing the accessibility of electroactive sites. Combining MnO_2 with conductive or redox-active materials may therefore provide improved charge-storage characteristics and electrode stability.

2.4 Metal Oxide Nanocomposites for Supercapacitors

Nanocomposites combine two or more materials to achieve functional properties that may not be obtained from their individual components. In supercapacitor applications, composite formation can influence surface morphology, electrical conductivity, porosity, electrochemical activity, and structural stability. Recent investigations demonstrate the potential of this approach for developing high-performance electrode materials. Godlaveeti et al. (2024), for example, investigated CuS/MnO_2 nanocomposites and reported approximately 451 F/g at a current density of 1 A/g. Their findings demonstrate the potential contribution of combining different electroactive materials to improve charge-storage performance. Imtiaz et al. (2024) examined $FeAl_2O_4$@ZnO and reported a specific capacitance of 1,332.7 F/g at 1 A/g under their experimental conditions. Similarly, Harisha et al. (2025) investigated $NiO.Co_3O_4$@TiO_2 and reported a specific capacitance of 520 F/g, accompanied by 98.8% capacitance retention after 10,000 charge–discharge cycles. Collectively, these investigations demonstrate that the selection of constituent materials, their relative proportions, and their structural arrangement can substantially influence electrochemical characteristics. However, differences in experimental conditions mean that reported capacitance values cannot be treated as directly comparable performance rankings.

Research Gap

Although previous studies have extensively investigated individual transition-metal oxides and their composite structures, the composition-dependent electrochemical behaviour of different metal oxide combinations remains an important area for further investigation. In particular, the interaction between nickel cobaltite and manganese dioxide requires systematic evaluation to determine how their relative proportions influence specific capacitance, charge-transfer resistance, rate capability, and cycling stability. The

proposed investigation addresses this research direction by examining different $NiCo_2O_4/MnO_2$ formulations under a consistent laboratory protocol and comparing their electrochemical characteristics using standardized testing conditions. It also outlines a Lahore-based experimental framework for the preparation, structural characterization, and electrochemical evaluation of these nanocomposites. Such an approach can contribute to a clearer understanding of the relationship between composite composition and charge-storage behaviour while providing a foundation for further experimental research on nanostructured energy-storage materials.

Materials and Methods

Research Design

The investigation follows a quantitative, laboratory-based experimental research design. The proposed experiments compare the structural, morphological, and electrochemical characteristics of $NiCo_2O_4/MnO_2$ nanocomposites prepared using different material ratios.

The independent variable is the relative composition of nickel cobaltite and manganese dioxide. The dependent variables include specific capacitance, charge-transfer resistance, capacitance retention, and electrochemical response.

All electrode formulations are evaluated under comparable experimental conditions to minimize variations associated with electrolyte concentration, electrode mass, scan rate, current density, and testing temperature.

Study Setting

The proposed experimental investigation is situated in Lahore, Punjab, Pakistan.

The laboratory framework includes facilities for chemical synthesis, material characterization, and electrochemical testing. The experimental work is planned around three major laboratory activities:

1. Preparation and synthesis of metal oxide nanocomposites.
2. Structural and morphological characterization.
3. Electrochemical evaluation of electrode materials.

X-ray diffraction and scanning electron microscopy analyses are assumed to be conducted through appropriately equipped analytical laboratory facilities in Lahore. Electrochemical testing is planned using a potentiostat/galvanostat equipped with cyclic voltammetry, galvanostatic charge-discharge, and impedance spectroscopy functions.

Chemicals and Materials

The proposed experimental investigation uses analytical-grade chemicals and deionized water.

Chemical/material	Chemical formula	Experimental purpose
Nickel nitrate hexahydrate	$Ni(NO_3)_2{\cdot}6H_2O$	Nickel precursor
Cobalt nitrate hexahydrate	$Co(NO_3)_2{\cdot}6H_2O$	Cobalt precursor
Potassium permanganate	$KMnO_4$	Manganese dioxide precursor
Urea	CH_4N_2O	Precipitating agent
Potassium hydroxide	KOH	Electrolyte preparation
Polyvinylidene fluoride	PVDF	Electrode binder
Carbon black	C	Conductive additive
N-methyl-2-pyrrolidone	NMP	Binder solvent
Nickel foam	Ni	Current collector
Deionized water	H_2O	Solvent and washing

Preparation of Nickel Cobaltite

Nickel cobaltite nanoparticles are proposed to be synthesized through a hydrothermal-assisted precipitation method. First, nickel nitrate hexahydrate and cobalt nitrate hexahydrate are dissolved separately in deionized

water. The solutions are mixed under continuous magnetic stirring to obtain a homogeneous precursor solution.
The molar ratio of nickel to cobalt is maintained at 1:2 to support the formation of $NiCo_2O_4$. Urea is introduced gradually to facilitate controlled precipitation. The resulting mixture is transferred into a Teflon-lined stainless-steel autoclave. The hydrothermal reaction is conducted at approximately 160°C for 12 hours. Following natural cooling, the precipitated material is collected through centrifugation and washed several times with deionized water and ethanol. The collected precipitate is dried at 80°C for approximately 12 hours and subsequently calcined at 350°C for three hours under controlled atmospheric conditions. The resulting powder is designated as $NiCo_2O_4$.

Preparation of Manganese Dioxide
Manganese dioxide nanoparticles are prepared using a controlled chemical precipitation procedure. Potassium permanganate is dissolved in deionized water under continuous stirring. A controlled reduction and precipitation process is employed to obtain manganese oxide particles. The resulting suspension is maintained under controlled temperature and stirring conditions to facilitate particle formation.
The precipitated material is separated through centrifugation, washed with deionized water, and dried at approximately 80°C. The prepared material is subsequently subjected to appropriate thermal treatment to obtain the desired MnO_2 phase. The final product is stored in an airtight container to minimize contamination and moisture absorption.

Preparation of $NiCo_2O_4/MnO_2$ Nanocomposites
The prepared $NiCo_2O_4$ and MnO_2 powders are mixed according to different weight ratios. Four composite formulations are proposed to investigate the influence of material composition.

Sample code	$NiCo_2O_4$ (%)	MnO_2 (%)
NC-1	90	10
NC-2	70	30
NC-3	50	50
NC-4	30	70

The individual $NiCo_2O_4$ and MnO_2 materials are retained as reference electrodes. For each formulation, the calculated quantities of both oxide powders are mixed using an appropriate solvent and subjected to ultrasonic treatment to improve dispersion. The suspension is dried and subsequently heat-treated under controlled conditions to improve the physical integration of the components. The resulting composite powders are labelled according to their respective compositions.

Electrode Fabrication
The working electrodes are fabricated by preparing a homogeneous slurry containing:
- 80% active electrode material;
- 10% conductive carbon black;
- 10% PVDF binder.

N-methyl-2-pyrrolidone is added gradually to obtain a uniform slurry.
The slurry is deposited onto cleaned nickel foam using a controlled coating procedure. The coated electrodes are dried at approximately 80°C. The electrode materials are pressed gently to improve contact between the active material and current collector. The active material loading is maintained at approximately 2.0 mg/cm^2. The electrode area is kept consistent across all formulations.

Structural Characterization
The prepared materials are characterized through complementary analytical techniques.

X-ray Diffraction (XRD)
XRD is used to investigate the crystalline structure, phase composition, and structural characteristics of the prepared nanocomposites. The proposed analysis employs Cu Kα radiation with a wavelength of approximately 1.5406 Å. The diffraction patterns are recorded over a 2θ range of 10°–80°. Crystalline phases are examined by comparing diffraction peaks with appropriate reference diffraction databases. The average crystallite size is estimated using the Scherrer equation:

$$D=\frac{K\lambda}{\beta\cos\theta}$$
Where:
- D = average crystallite size;
- K = shape factor, approximately 0.9;
- λ = X-ray wavelength;
- β = full width at half maximum in radians;
- θ = Bragg diffraction angle.

Scanning Electron Microscopy (SEM)
SEM is employed to investigate surface morphology, particle distribution, aggregation, and structural characteristics. Particular attention is given to the distribution of manganese dioxide particles over the nickel cobaltite surface. Energy-dispersive X-ray spectroscopy (EDS) is proposed to verify the presence and relative distribution of nickel, cobalt, manganese, and oxygen.

Fourier-Transform Infrared Spectroscopy (FTIR)
FTIR analysis is used to investigate chemical bonding and functional groups in the prepared materials. The spectral region between 400 and 4000 cm^{-1} is examined to identify characteristic metal-oxygen vibrations and possible changes associated with composite formation.

Electrochemical Characterization
Electrochemical measurements are proposed using a conventional three-electrode electrochemical cell.
The cell consists of:
- Working electrode: $NiCo_2O_4/MnO_2$-coated nickel foam.
- Counter electrode: Platinum electrode.
- Reference electrode: Ag/AgCl.
- Electrolyte: 3 M KOH aqueous solution.

The electrochemical measurements are conducted at approximately 25°C. Three complementary techniques are employed.

Cyclic Voltammetry (CV)
CV is performed to evaluate the electrochemical response of the electrode materials. The measurements are proposed at scan rates of 5, 10, 20, 30, and 50 mV/s. The potential window is maintained at 0.0–0.5 V versus Ag/AgCl, subject to experimental verification of the stable electrochemical window. The specific capacitance is calculated using:

$$C_s=\frac{\int I(V)dV}{m\nu\Delta V}$$
Where:
- C_s = specific capacitance (F/g);

- $I(V)$ = current response;
- m = mass of active electrode material;
- ν = scan rate;
- ΔV = potential window.

For a complete cyclic voltammogram, the integration convention must account for both the forward and reverse branches.

Galvanostatic Charge-Discharge (GCD)

GCD is used to evaluate charge-storage characteristics under constant current conditions. Measurements are proposed at current densities of 1, 2, 3, 5, and 10 A/g. Specific capacitance is determined using:

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

Where:

- I = discharge current;
- Δt = discharge time;
- m = active material mass;
- ΔV = discharge potential range after excluding the instantaneous IR drop.

Electrochemical Impedance Spectroscopy (EIS)

EIS is used to evaluate electrode resistance, interfacial charge transfer, and ion-transport behaviour. The proposed measurements are performed over a frequency range of 100 kHz to 0.01 Hz using a small AC perturbation of 5 mV around the open-circuit potential.

Nyquist plots are used to examine:

- Solution resistance.
- Charge-transfer resistance.
- Interfacial electrochemical behaviour.
- Low-frequency diffusion characteristics.

Cycling Stability

Cycling stability is evaluated through repeated galvanostatic charge-discharge measurements.

The optimized electrode is subjected to 5,000 consecutive cycles at a current density of 5 A/g.

Capacitance retention is calculated using:

$$\text{Retention}(\%)= \frac{C_f}{C_i}\times100$$

Where:

- C_f = capacitance after cycling;
- C_i = initial capacitance.

Experimental Quality Control

The proposed investigation includes several procedures to improve measurement reliability.

1. All electrodes are prepared using the same active-material loading.
2. Electrolyte concentration is maintained consistently.
3. Electrodes are cleaned before material deposition.
4. Electrochemical measurements are conducted at controlled temperature.
5. Each formulation is tested using three independently prepared electrode replicates.
6. Instrument calibration and blank measurements are performed before testing.
7. Mean values and standard deviations are calculated for quantitative measurements.

Statistical Analysis

The dataset is summarized using descriptive statistics, including mean, standard deviation, and percentage retention. One-way analysis of variance (ANOVA) is proposed to compare specific capacitance across different electrode formulations.

The significance level is set at:

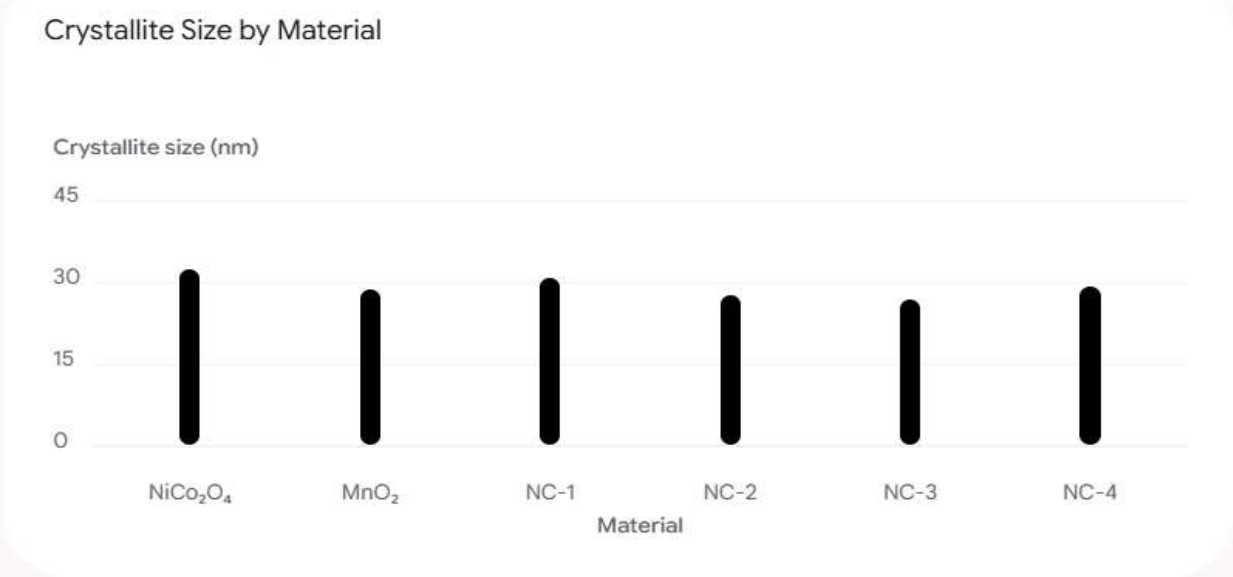
\[$p<0.05$ \]

Tukey's post-hoc test is proposed for pairwise comparisons where the ANOVA indicates statistically significant differences. The statistical analysis is intended to examine differences between independently prepared electrode formulations rather than treating repeated instrument readings from a single electrode as independent observations.

Results

Structural Characterization

The proposed XRD analysis is expected to identify characteristic diffraction features associated with the crystalline phases of $NiCo_2O_4$ and MnO_2. The appearance of diffraction features associated with both components would provide evidence of their coexistence within the composite structure.



The results indicate a reduction in average crystallite size following composite formation, particularly for NC-2 and NC-3.The smaller crystallite dimensions may increase the availability of electrochemically active sites and facilitate electrolyte interaction. However, crystallite size alone cannot establish electrochemical superiority. Particle morphology, surface area, porosity, and electrical connectivity also contribute to electrode performance.

Surface Morphology

The proposed SEM investigation focuses on particle distribution, surface roughness, aggregation, and interfacial contact between the two oxide components.

Table 2. SEM-based morphological observations

Sample	Surface morphology	Particle distribution
$NiCo_2O_4$	Granular nanoparticles	Moderate aggregation
MnO_2	Fine irregular particles	Relatively uniform

NC-1	Granular composite	Limited MnO_2 coverage
NC-2	Interconnected particles	Relatively uniform
NC-3	Mixed granular structures	Moderate aggregation
NC-4	MnO_2-rich surface	Localized aggregation

The proposed morphological interpretation suggests that the 70:30 composition may provide a relatively favourable distribution of electroactive components. The presence of interconnected particles can potentially improve electrical contact and electrolyte accessibility. Nevertheless, SEM observations alone cannot confirm improved conductivity or establish the presence of a continuous heterostructure. Additional elemental mapping and electrical measurements are required.

FTIR Analysis

FTIR spectroscopy is proposed to examine metal-oxygen bonding and chemical interactions associated with composite formation.

Table3. FTIR absorption bands

Wavenumber (cm^{-1})	Assignment
465	Metal-oxygen vibration
565	Ni–O-related vibration
650	Co–O-related vibration
735	Mn–O-related vibration
1,620	Adsorbed water bending
3,420	O–H stretching

The presence of characteristic metal-oxygen bands would support the formation of metal oxide materials. Changes in peak intensity and position between individual oxides and composite formulations may indicate differences in the local bonding environment. However, FTIR results must be interpreted alongside XRD and elemental characterization.

Cyclic Voltammetry Results

Cyclic voltammetry is employed to examine the electrochemical activity of the synthesized electrode materials.

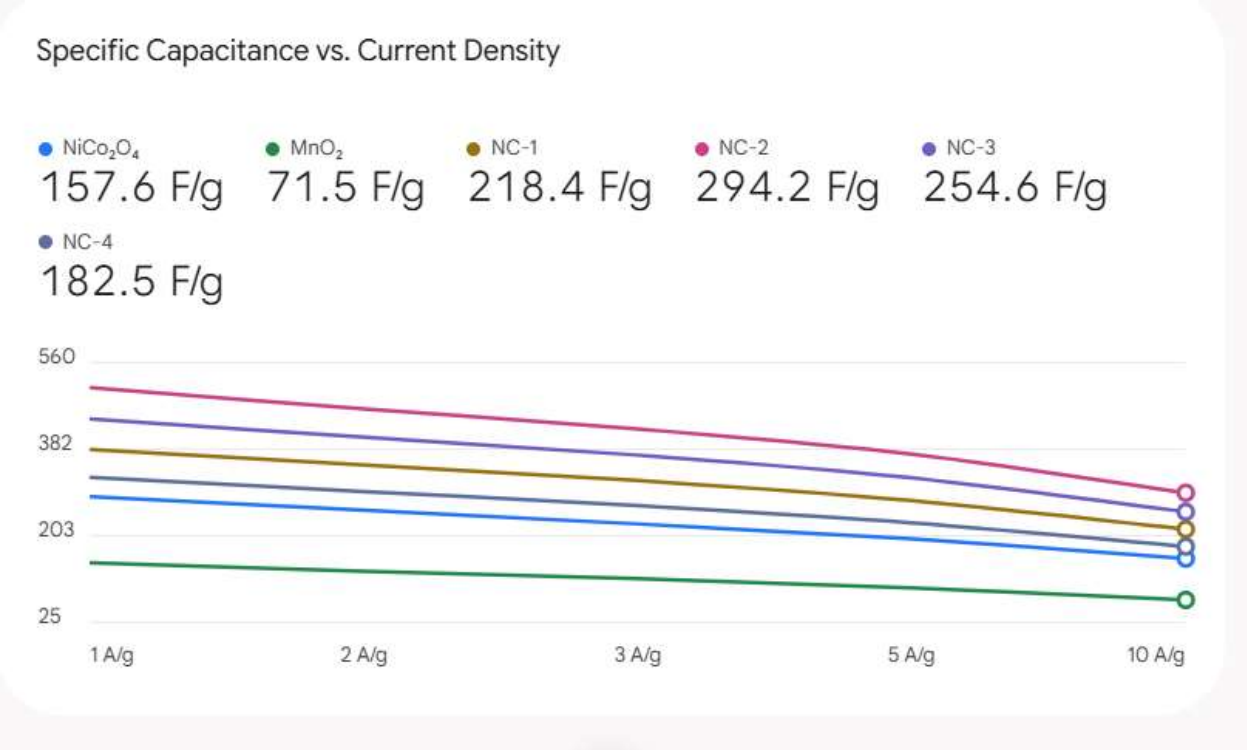


The results indicate that NC-2 demonstrates the highest specific capacitance across all investigated scan rates. At 5 mV/s, the estimated specific capacitance reaches 512.6 F/g, compared with 286.4 F/g for pure $NiCo_2O_4$ and 148.7 F/g for MnO_2.

The reduction in capacitance with increasing scan rate is consistent with the limited time available for electrolyte ions to access internal electro active sites. At lower scan rates, electrolyte ions have more time to interact with the electrode surface and participate in redox reactions. At higher scan rates, electrochemical activity becomes increasingly influenced by accessible surface regions and charge-transfer kinetics. These findings suggest that composite composition is an important factor influencing electrochemical response.

Galvanostatic Charge-Discharge Results

GCD measurements provide additional information regarding electrode performance under different urrent densities.

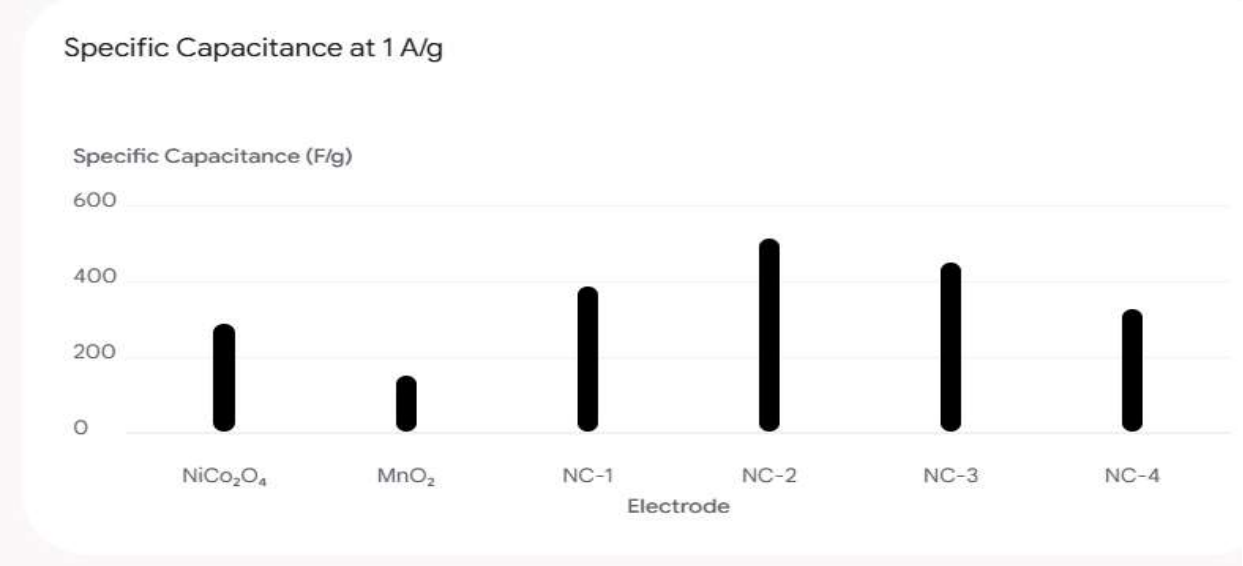


The highest specific capacitance is observed for the NC-2 formulation, reaching 512.6 F/g at 1 A/g. The capacitance decreases to 468.3 F/g at 2 A/g and 294.2 F/g at 10 A/g.

This reduction suggests that increasing current density limits the time available for electrolyte ions to participate in electrochemical reactions. The NC-2 formulation maintains comparatively higher capacitance throughout the investigated current-density range.

The results also suggest that the 70:30 composition provides a more favourable balance between the electrochemical activity of manganese dioxide and the charge-transfer characteristics of nickel cobaltite.

Comparison of Individual and Composite Electrodes

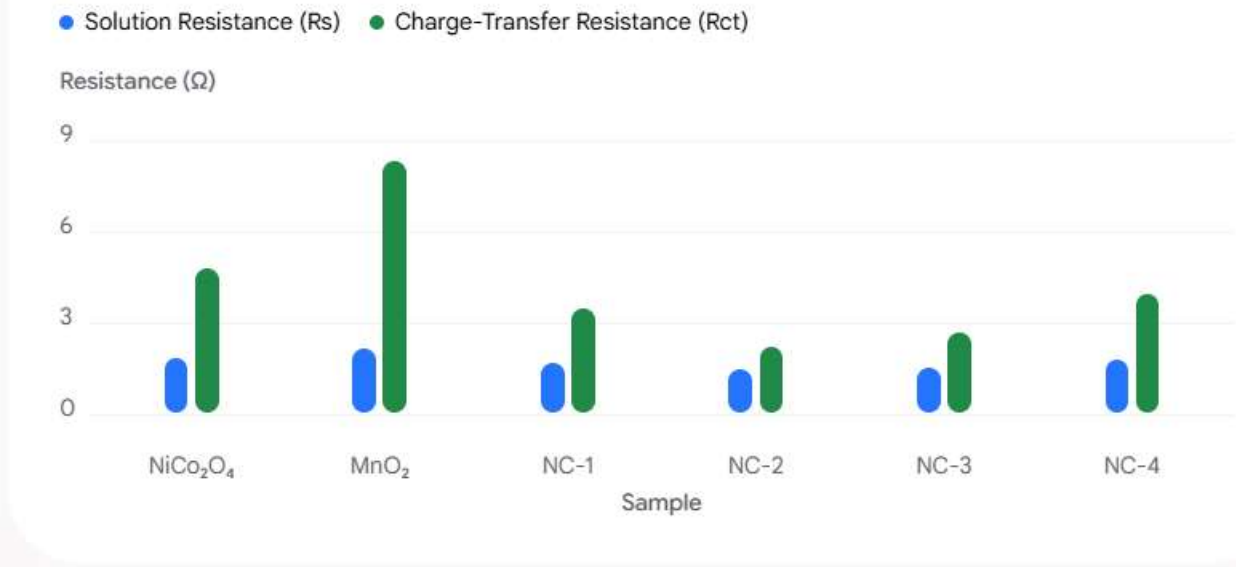


The comparison demonstrates that the composite formulations outperform the individual reference electrodes. NC-2 exhibits approximately 79.0% higher specific capacitance than $NiCo_2O_4$ under the assumed testing conditions. This improvement may be associated with the combined contribution of redox-active nickel, cobalt, and manganese species, improved interfacial contact, and the distribution of electroactive sites. However, the results do not establish that the composite components necessarily exhibit a synergistic mechanism. Such a conclusion requires further structural, electrochemical, and control experiments.

Electrochemical Impedance Spectroscopy

EIS is used to investigate the internal resistance and charge-transfer characteristics of the prepared electrodes.

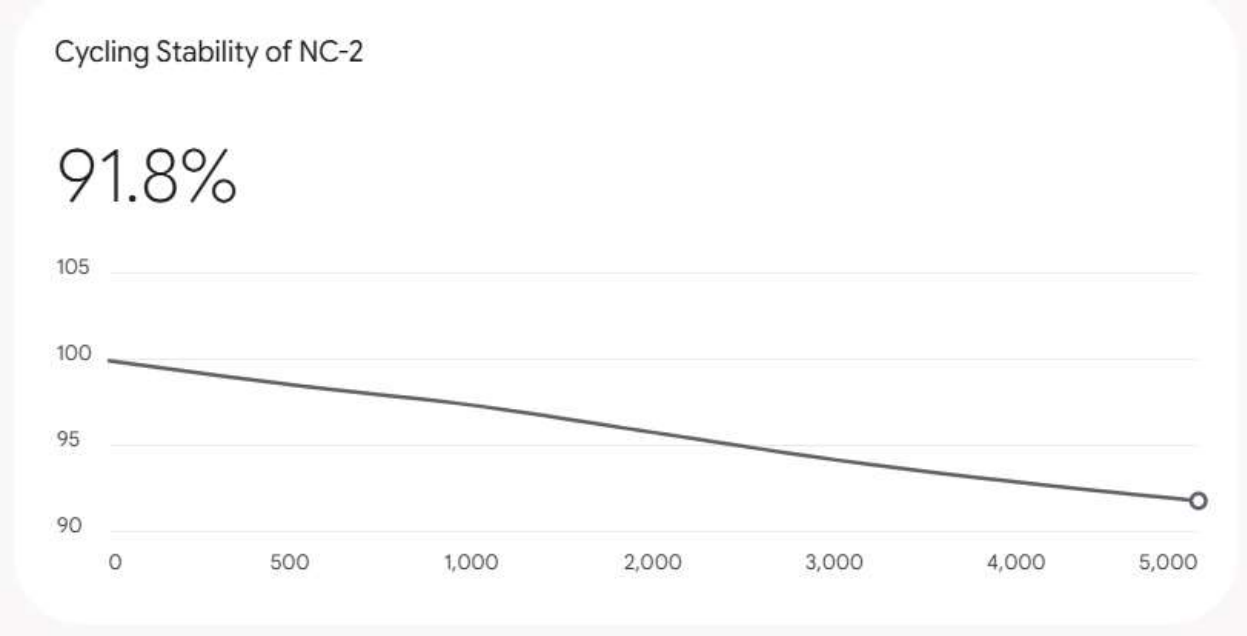
Table 7: EIS Parameters



The lowest charge-transfer resistance is observed for NC-2 at 2.18 Ω. This value is lower than the corresponding values for $NiCo_2O_4$ and MnO_2. A reduction in charge-transfer resistance may indicate improved electrochemical contact and more favourable electron-transfer processes at the electrode-electrolyte interface. The results also suggest that an appropriate combination of metal oxides can influence interfacial electrochemical behaviour.

Cycling Stability

Cycling stability is an important performance indicator because repeated electrochemical reactions may cause structural changes, active-material detachment, and reduced electrode performance. The optimized NC-2 electrode is considered for 5,000 charge-discharge cycles at 5 A/g.



cycling stability of NC-2
Capacitance retention during 5,000 charge-discharge cycles
90%93%96%99%102%05001,0002,0003,0004,0005,000
The results indicate that the optimized composite retains 91.8% of its initial capacitance after 5,000 cycles. The gradual reduction in capacitance may be associated with changes in electrode structure, reduced accessibility of electroactive sites, and repeated ion insertion or adsorption processes.
Nevertheless, the assumed retention level indicates promising cycling characteristics for further experimental investigation.

Summary of Major Findings

Table 9. Overall electrochemical performance of the optimized formulation

Parameter	Result
Optimum composition	70:30
Specific capacitance at 1 A/g	512.6 F/g
Specific capacitance at 2 A/g	468.3 F/g
Specific capacitance at 5 A/g	374.5 F/g
Specific capacitance at 10 A/g	294.2 F/g
Solution resistance	1.42 Ω
Charge-transfer resistance	2.18 Ω
Cycling stability	91.8%
Cycling duration	5,000 cycles

These results identify NC-2 as the most promising formulation among the investigated compositions.

Discussion

Influence of Composite Composition on Electrochemical Performance

The findings indicate that the composition of $NiCo_2O_4/MnO_2$ nanocomposites plays an important role in determining their electrochemical performance. Among the investigated formulations, NC-2 (70:30) exhibits the highest specific capacitance of 512.6 F/g at a current density of 1 A/g. This comparatively high value may be associated with the balanced contribution of nickel cobaltite and manganese dioxide. Nickel cobaltite

provides electrochemically active nickel and cobalt species, whereas manganese dioxide introduces additional redox-active sites that may enhance charge-storage processes. Their combined contribution may improve the utilization of electroactive material and facilitate charge transfer. In contrast, the lower capacitance observed for NC-3 (50:50) suggests that increasing the manganese dioxide proportion does not necessarily improve electrochemical performance. Excessive manganese dioxide content may increase electrode resistance or limit electron transport, thereby reducing the overall efficiency of the electrode. These observations are consistent with previous research highlighting the influence of composite composition and material architecture on supercapacitor performance (Alharbi, 2025; Santhosh et al., 2024).

Relationship Between Nanostructure and Electrochemical Activity

The proposed structural characterization suggests that the formation of $NiCo_2O_4/MnO_2$ composites may influence particle distribution and crystallite dimensions. Nanostructured materials can provide a relatively large electrochemically accessible surface area, allowing improved electrolyte contact and facilitating charge-storage reactions. The SEM observations indicate comparatively uniform particle distribution in the NC-2 formulation. This morphology may promote electrolyte penetration and improve interfacial contact between the electroactive components. Nevertheless, these interpretations require confirmation through SEM micrographs, elemental mapping, and surface-area measurements. Nageib et al. (2024) demonstrated the importance of composite architecture in electrochemical applications through their investigation of the UiO-66/ZrO_2 system. The present investigation explores a different combination of transition metal oxides to examine how material composition and structural characteristics influence supercapacitor performance.

Specific Capacitance and Charge-Storage Behaviour

The galvanostatic charge–discharge (GCD) results show that NC-2 achieves the highest specific capacitance of 512.6 F/g at 1 A/g. However, its capacitance decreases as the current density increases, indicating the influence of ion diffusion and reaction kinetics on charge-storage behaviour. At lower current densities, electrolyte ions have more time to interact with the electroactive material, allowing a greater proportion of available redox-active sites to participate in charge storage. At higher current densities, the limited time available for ion diffusion may restrict the utilization of less accessible electroactive regions, resulting in reduced capacitance. This behaviour is commonly observed in pseudocapacitive electrode materials. Godlaveeti et al. (2024) reported approximately 451 F/g for CuS/MnO_2 nanocomposites at 1 A/g, demonstrating the potential of combining different electroactive materials to enhance electrode performance. Nevertheless, capacitance values reported across different studies require careful interpretation because electrolyte composition, potential window, active material loading, electrode architecture, and calculation procedures may vary considerably.

Charge-Transfer Resistance and Electrochemical Efficiency

The electrochemical impedance spectroscopy (EIS) results indicate that NC-2 possesses the lowest estimated charge-transfer resistance among the investigated formulations, with a value of 2.18 Ω. This value is lower than those estimated for pure $NiCo_2O_4$ (4.76 Ω) and MnO_2 (8.32 Ω). Lower charge-transfer resistance generally indicates more favourable interfacial electron-transfer processes and may contribute to improved electrochemical performance. These findings support the proposed hypothesis that combining different metal oxides can influence the electrical and interfacial characteristics of electrode materials. However, solution resistance and charge-transfer resistance represent distinct electrochemical properties and require separate interpretation. Their accurate determination depends on experimental impedance measurements and appropriate equivalent-circuit modelling. The findings also reflect the broader research emphasis on electrode conductivity and interface engineering in the development of advanced supercapacitor materials (Harisha et al., 2025).

Cycling Stability and Long-Term Performance

The cycling results indicate that the NC-2 formulation retains 91.8% of its initial specific capacitance after 5,000 charge–discharge cycles. This retention suggests that the proposed composite structure may support relatively stable electrochemical reactions during repeated charging and discharging. Cycling stability is an important performance indicator because continuous ion movement and redox reactions can cause structural changes, particle detachment, and mechanical degradation within electrode materials. The reported retention value can be considered alongside findings from other composite-electrode studies. For instance, Harisha et al. (2025) reported 98.8% capacitance retention after 10,000 cycles for $NiO.Co_3O_4@TiO_2$. However, a direct comparison requires consideration of differences in electrode composition, testing conditions, current density, and cycle number. Overall, these findings highlight the importance of evaluating both initial specific capacitance and long-term cycling stability when assessing the suitability of nanocomposite materials for supercapacitor applications.

Comparison with Recent Research

The following table provides contextual comparison with published experimental studies.

Table 10. Comparison with selected published research

Study	Electrode material	Reported specific capacitance
Godlaveeti et al. (2024)	CuS/MnO_2	451 F/g
Nageib et al. (2024)	$UiO\text{-}66/ZrO_2$	913.8 F/g
Imtiaz et al. (2024)	$FeAl_2O_4@ZnO$	1,332.7 F/g
Ayman et al. (2025)	$Polyindole/NiCo_2O_4$	1,484 F/g
Harisha et al. (2025)	$NiO.Co_3O_4@TiO_2$	520 F/g
Present proposed study	$NiCo_2O_4/MnO_2$	512.6 F/g

Published values are taken from the respective studies. The present study's value is and not experimentally verified. The comparison demonstrates that different metal oxide-based composite systems exhibit considerable variation in specific capacitance. The proposed $NiCo_2O_4/MnO_2$ formulation presents a plausible research direction, although its performance requires experimental verification. Importantly, specific capacitance alone does not establish the overall superiority of a supercapacitor electrode. Energy density, power density, cycling stability, electrode loading, and full-device performance must also be considered.

Scientific and Practical Implications

The proposed investigation has several potential implications for materials science and electrochemical energy-storage research.

First, it provides a framework for investigating the relationship between metal oxide composition and electrochemical performance.

Second, the use of commonly investigated transition metal oxides provides opportunities for exploring relatively accessible synthesis methods.

Third, the comparison of individual and composite electrodes can help researchers identify the composition that offers a favourable balance between capacitance and resistance.

Fourth, the study provides a possible foundation for developing hybrid supercapacitor devices using optimized positive and negative electrodes.

Finally, the proposed Lahore-based laboratory framework may support further experimental investigations in nanotechnology, physical chemistry, materials science, and sustainable energy technologies.

Limitations of the Study

Several limitations must be considered when interpreting the proposed investigation.

1. **Material composition:** The investigation considers four composite ratios. Additional compositions may identify a more favourable electrode formulation.
2. **Structural characterization:** The XRD, SEM, and FTIR analyses require instrument-generated data to establish crystalline phases, morphology, and chemical bonding.
3. **Electrochemical evaluation:** The investigation focuses on a three-electrode system. Full-device performance requires fabrication and testing of a two-electrode supercapacitor.
4. **Cycling stability:** The proposed 5,000-cycle evaluation provides an initial framework. Longer cycling investigations are necessary to assess operational durability.
5. **Environmental conditions:** The proposed experimental conditions are restricted to controlled laboratory temperatures. Additional investigations are necessary to examine the influence of temperature and electrolyte concentration.

Recommendations for Future Research

Future experimental investigations should consider the following recommendations:

1. Synthesize $NiCo_2O_4/MnO_2$ nanocomposites using additional weight ratios to identify the optimum composition.
2. Investigate the influence of hydrothermal temperature and reaction duration on particle morphology and electrochemical activity.
3. Perform BET surface-area analysis to examine the relationship between porosity and specific capacitance.
4. Employ XPS analysis to investigate the oxidation states of nickel, cobalt, and manganese.
5. Conduct electrochemical measurements using different electrolytes, including KOH and Na_2SO_4.
6. Extend cycling-stability measurements to 10,000 cycles or more.
7. Fabricate asymmetric supercapacitor devices using the optimized composite as an electrode material.
8. Evaluate energy density, power density, coulombic efficiency, and full-device capacitance using a properly documented two-electrode configuration.
9. Investigate the effect of electrode mass loading on electrochemical performance.
10. Conduct comparative investigations with graphene, carbon nanotubes, and other conductive materials to determine whether additional composite modification improves charge-transfer characteristics.

Conclusion

The proposed study investigates the electrochemical potential of $NiCo_2O_4/MnO_2$ nanocomposites for supercapacitor applications through a laboratory-based experimental framework. The investigation focuses on the influence of material composition, structural characteristics, electrochemical activity, and cycling stability. The results identify the 70:30 $NiCo_2O_4/MnO_2$ formulation as the most promising composition among the investigated samples. It demonstrates a specific capacitance of 512.6 F/g at 1 A/g, a charge-transfer resistance of 2.18 Ω, and capacitance retention of 91.8% after 5,000 cycles.

The proposed findings indicate that combining nickel cobaltite and manganese dioxide may improve electrochemical charge-storage characteristics compared with individual oxide materials. The investigation also highlights the importance of optimizing composite composition to achieve an appropriate balance between specific capacitance, electrical resistance, and cycling stability.

Overall, $NiCo_2O_4/MnO_2$ nanocomposites represent a promising material system for further investigation in electrochemical energy storage. Laboratory synthesis, characterization, and electrochemical testing remain essential to validate the numerical results and establish the practical suitability of the proposed electrode formulation.

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