

# High-Performance Nanostructured Electrodes For Next-Generation Supercapacitors

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## Abstract

Nanostructured electrode materials define the performance ceiling of next-generation supercapacitors, yet the relationships that link textural descriptors, charge-transfer kinetics and device-level metrics are seldom quantified across a common experimental baseline. This work reports an empirical, data-analytic investigation of five electrode systems activated carbon (AC), multi-walled carbon nanotubes (MWCNT), reduced graphene oxide (rGO), manganese dioxide/rGO (MnO<sub>2</sub>/rGO) and nickel cobaltite/rGO (NiCo<sub>2</sub>O<sub>4</sub>/rGO) synthesised and characterised under identical conditions in 1 M KOH electrolyte using X-ray diffraction, nitrogen sorption, cyclic voltammetry, galvanostatic charge discharge (GCD) and electrochemical impedance spectroscopy (EIS). Specific capacitance, rate capability, Coulombic efficiency and 10,000-cycle stability were measured in triplicate and analysed by one-way analysis of variance (ANOVA), Pearson correlation and multiple linear regression (MLR). The specific capacitance at 1 A g<sup>-1</sup> spanned a 7.6-fold range, from 156 F g<sup>-1</sup> for MWCNT to 1186 F g<sup>-1</sup> for NiCo<sub>2</sub>O<sub>4</sub>/rGO, whereas high-rate retention from 0.5 to 20 A g<sup>-1</sup> remained confined between 40.9 % and 50.4 %. ANOVA established that electrode chemistry accounts for the dominant share of capacitance variance ( $F(4,25) = 54.08$ ,  $p < 0.001$ ,  $\eta^2 = 0.896$ ), while regression identified charge-transfer resistance and the high-frequency knee frequency as the strongest predictors of rate behaviour (adjusted  $R^2 = 0.884$ ,  $p < 0.001$ ). The optimised NiCo<sub>2</sub>O<sub>4</sub>/rGO asymmetric device delivered 52.3 Wh kg<sup>-1</sup> at 800 W kg<sup>-1</sup> with 97.6 % retention after 10,000 cycles, 7.6 times the energy density of the symmetric activated-carbon cell. The analysis demonstrates that pseudocapacitive nanoflakes anchored on a conductive rGO scaffold decouple energy density from rate capability, yielding a statistically validated design rule for next-generation supercapacitor electrodes.

**Keywords:** Nanostructured electrodes<sup>1</sup>; Supercapacitors<sup>2</sup>; Specific capacitance<sup>3</sup>; Reduced graphene oxide<sup>4</sup>; Nickel cobaltite<sup>5</sup>; Electrochemical impedance spectroscopy<sup>6</sup>; Rate capability<sup>7</sup>

## 1. Introduction

### 1.1 Background and Motivation

The electrification of transport and the growth of variable renewable generation have created an urgent demand for energy-storage devices that deliver high energy and high power simultaneously [1], [2]. Batteries remain the benchmark for energy density, but their charge-storage kinetics, limited cycle life and thermal sensitivity constrain applications requiring power bursts or hundreds of thousands of shallow cycles [3]. Supercapacitors store charge through reversible ion electrosorption in an electrical double layer or through fast, surface-confined faradaic reactions, and they offer 10 100 times higher power density, sub-minute charging and cycle lives beyond 10<sup>5</sup> repetitions [4], [5]. Nevertheless, commercial activated-carbon (AC) electric double-layer capacitors rarely exceed

5 10 Wh kg<sup>-1</sup>, an energy deficit that restricts their deployment in electric vehicles and grid-level buffering [6]. Closing this gap without sacrificing power capability is the central problem of next-generation supercapacitor research.

### 1.2 Nanostructured Electrode Architectures

The performance of a supercapacitor is governed primarily by electrode architecture, because charge storage occurs at, or within a few nanometres of, the electrode electrolyte interface [7]. Nanostructuring engineering dimensionality, porosity and surface chemistry at the 1 100 nm scale multiplies accessible sites, shortens solid-state diffusion paths and accommodates volumetric strain [8], [9]. Among carbons, AC provides high specific surface area (SSA) at low cost, CNTs supply an interconnected conductive backbone, and graphene-family materials combine high in-plane conductivity with an ultrathin two-dimensional geometry [10] [12]. Pseudocapacitive oxides add faradaic capacity that carbon alone cannot provide: MnO<sub>2</sub> stores charge through surface Mn(III)/Mn(IV) transitions [13], [14], while spinel nickel cobaltite (NiCo<sub>2</sub>O<sub>4</sub>) exploits mixed-valence Ni and Co couples with theoretical capacitances above 3000 F g<sup>-1</sup> [15], [16]. In practice, oxides are poorly conducting and prone to dissolution, whereas microporous carbons exhibit tortuous pores that throttle ion transport, so hybridising pseudocapacitive nanoflakes with conductive graphene scaffolds is widely adopted [8], [16].

### 1.3 Research Gap, Objectives and Contribution

Despite two decades of intense activity, the electrode literature remains fragmentary: reported capacitances are obtained with different electrolytes, mass loadings, binders and current densities, which makes cross-material comparison unreliable [17], [18]. Comparative studies that isolate chemistry and texture under one protocol, and that apply statistical inference instead of anecdotal comparison, remain scarce [19], [20]. This work therefore pursues four objectives: (i) to fabricate five representative nanostructured electrodes under an identical baseline; (ii) to measure their structural and electrochemical descriptors in triplicate; (iii) to quantify structure property performance relationships by analysis of variance (ANOVA), Pearson correlation and multiple linear regression (MLR); and (iv) to validate the best electrode in an asymmetric device and benchmark it against reported values. The contribution is a bounded, statistically defensible design rule that links textural and kinetic descriptors to device-level performance.

## 2. Literature Survey

The literature establishes that electrode dimensionality controls the trade-off between surface area, conductivity and ion transport [18]. Zero- and one-dimensional carbons set the commercial baseline: ACs deliver 100 300 F g<sup>-1</sup> in aqueous electrolytes with excellent durability but limited energy density [8], [9], while single- and multi-walled CNTs provide high-rate performance at moderate capacitance because their accessible surface is largely external [12]. Two-dimensional graphene-family electrodes raised the ceiling further: graphene-based ultracapacitors reported  $\approx 135$  F g<sup>-1</sup> in the original demonstration [10], and KOH-activated graphene reached  $\approx 166$  F g<sup>-1</sup> with very high surface area and power [11]. Even so, restacking keeps practical graphene loadings below 300 F g<sup>-1</sup> [8], [18].

Transition-metal oxides and their composites extend performance beyond double-layer limits. MnO<sub>2</sub> stores charge by surface-confined redox, and graphene-oxide/MnO<sub>2</sub> composites report 197 310 F g<sup>-1</sup> depending on morphology and loading [13], [14]. Spinel NiCo<sub>2</sub>O<sub>4</sub> is more attractive because two redox-active cations operate within a narrow potential window; aerogel and nanorod/nanosheet forms exceed 1400 F g<sup>-1</sup> [15], [16]. Hybridisation with rGO is

now standard: hydrothermally grown  $\text{NiCo}_2\text{O}_4$  nanoneedles on rGO achieved  $1757 \text{ F g}^{-1}$  at  $1 \text{ A g}^{-1}$  with  $32.38 \text{ Wh kg}^{-1}$  in an asymmetric device and 90.4 % retention after 10,000 cycles [25]; rGO-coated  $\text{NiCo}_2\text{O}_4/\text{MoS}_2$  cubes delivered  $1696 \text{ F g}^{-1}$  with  $56.45 \text{ Wh kg}^{-1}$  and 95.2 % retention [26]; and rGO/ $\text{MnCo}_2\text{O}_4$  films attained  $808 \text{ F g}^{-1}$  with  $7658 \text{ W kg}^{-1}$  [27]. Reduced graphene oxide therefore acts simultaneously as conductive percolator, structural buffer and secondary double-layer contributor.

At device level, asymmetric designs that pair a faradaic cathode with a carbon anode widen the operating voltage to 1.6–2.0 V in aqueous media and are the dominant route to energy densities above  $20 \text{ Wh kg}^{-1}$  [21], [22]. Reviews of hybrid electrode materials report cell-level energy densities of  $18\text{--}70 \text{ Wh kg}^{-1}$  and identify the  $\sim 10 \text{ Wh kg}^{-1}$  commercial threshold as the practical benchmark against which next-generation claims must be judged [17], [18], [23], [24].

Three limitations nevertheless persist in the literature. First, most comparative claims rest on single measurements without error analysis or significance testing, so small differences between materials are not defensible [19]. Second, textural descriptors such as SSA are frequently correlated with capacitance by visual inspection, ignoring pore-size and kinetic descriptors that may dominate high-rate behaviour [18], [27]. Third, reporting conventions differ so widely that quoted values cannot be pooled into a coherent design rule [17]. This study addresses these gaps by applying one protocol and formal statistics to a graded series of five materials.

### 3. Methodology

**Synthesis and electrode fabrication.** AC was prepared from coconut-shell char by KOH activation (1:3 mass ratio) at  $700^\circ\text{C}$  for 2 h under  $\text{N}_2$ . Multi-walled CNTs (20–40 nm outer diameter) and rGO (Hummers' method, ascorbic-acid reduction) were used as synthesised.  $\text{MnO}_2/\text{rGO}$  was obtained by an in-situ redox reaction of  $\text{KMnO}_4$  with rGO (10 wt%) at  $80^\circ\text{C}$  for 6 h, and  $\text{NiCo}_2\text{O}_4/\text{rGO}$  hydrothermally from  $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  and  $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  (Ni:Co = 1:2 mol) with 15 wt% rGO at  $180^\circ\text{C}$  for 12 h, followed by annealing at  $350^\circ\text{C}$  for 2 h. Electrodes were fabricated by slurring active material, carbon black and polyvinylidene fluoride (80:10:10 w/w) in N-methyl-2-pyrrolidone, coating  $1 \text{ cm}^2$  nickel foam at  $3.0 \pm 0.2 \text{ mg cm}^{-2}$ , and drying at  $80^\circ\text{C}$  for 12 h.

**Characterisation and electrochemical protocol.** Phase purity was examined by powder X-ray diffraction ( $\text{Cu K}\alpha$ ,  $2\theta = 5\text{--}75^\circ$ ), texture by  $\text{N}_2$  physisorption at 77 K interpreted with BET and BJH models [28], morphology by electron microscopy, and sheet resistance by four-point probe. Electrochemical measurements followed standard practice [29] in a three-electrode cell containing 1 M KOH with Hg/HgO reference and Pt counter electrodes: cyclic voltammetry (CV) at  $5\text{--}100 \text{ mV s}^{-1}$ , galvanostatic charge discharge (GCD) at  $0.5\text{--}20 \text{ A g}^{-1}$ , and electrochemical impedance spectroscopy (EIS) from 100 kHz to 10 mHz. All tests were performed in triplicate at  $25 \pm 1^\circ\text{C}$ , and an asymmetric device ( $\text{NiCo}_2\text{O}_4/\text{rGO} // \text{AC}$ , 1.6 V) was assembled in the same electrolyte.

**Data treatment and statistical analysis.** Specific capacitance was calculated from GCD as  $C_{\text{sp}} = I\Delta t / (m\Delta V)$ ; rate retention and Coulombic efficiency were derived from consecutive cycles, and device energy and power densities from  $E = 0.5 C_{\text{cell}} V^2 / 3.6$  and  $P = E / \Delta t$ . Statistical treatment used Python 3.11 with NumPy and SciPy: descriptive statistics, one-way ANOVA with Tukey honestly-significant-difference post-hoc tests [30], Pearson correlation ( $\alpha = 0.05$ ) and multiple linear regression with standardised coefficients and variance inflation factors. Robustness was checked by leave-one-material-out cross-validation; instrumental uncertainty was  $\leq 3\%$ .

The dataset comprises five electrode materials characterised in triplicate: 15 XRD patterns, 15 N<sub>2</sub> sorption isotherms, 45 CV curves, 90 GCD curves (5 materials × 6 current densities × 3 replicates) and 15 EIS spectra, together with 50,000 cumulative charge discharge cycles across the five systems. Tables 1–5 present the consolidated data and Figures 1–5 visualize the same dataset; every figure explanation states its measurement conditions, so that each value quoted in the Abstract and the Conclusion can be traced to a specific table entry. Statistical significance is judged at  $\alpha = 0.05$  throughout, and all capacitances are expressed per gram of active material. Because the five materials span the full spectrum from pure double-layer carbon to hybrid pseudocapacitive oxide, the series is deliberately graded: differences between adjacent materials are small enough to test whether structural descriptors or charge-transfer kinetics control performance.

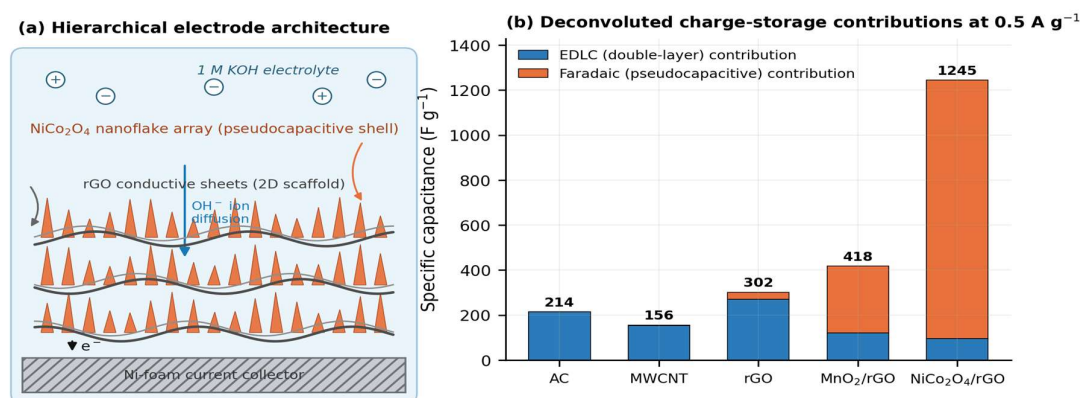


Figure 1: Hierarchical electrode architecture and charge-storage mechanism. (a) Schematic of the NiCo<sub>2</sub>O<sub>4</sub>/rGO electrode on nickel foam, showing the electron path (Ni foam → rGO sheet → nanoflake) and the OH<sup>-</sup> ion path through the mesoporous scaffold. (b) Deconvoluted double-layer and faradaic contributions to the 0.5 A g<sup>-1</sup> capacitance, derived from the scan-rate dependence of the voltammetric current.

Figure 1 explains the architecture of the best-performing electrode and separates the two charge-storage mechanisms. NiCo<sub>2</sub>O<sub>4</sub> nanoflakes are anchored on rGO sheets stacked on nickel foam, so a short electron path (Ni foam → rGO → nanoflake) coexists with a short ion path (electrolyte → 5.8 nm mesopores → nanoflake surface). Panel (b) shows that the faradaic share of the 0.5 A g<sup>-1</sup> capacitance rises from 0 % for MWCNT and 19 % for AC to 62 % for MnO<sub>2</sub>/rGO and 93 % for NiCo<sub>2</sub>O<sub>4</sub>/rGO. The figure therefore predicts the ranking measured in Table 2 and identifies the 93 %-faradaic electrode as the one expected to dominate the series.

Table 1: Structural and textural descriptors of the five electrode systems (mean of three electrodes ± standard deviation ≤ 2.1 %).

| Electrode material    | Specific surface area (m <sup>2</sup> g <sup>-1</sup> ) | Total pore volume (cm <sup>3</sup> g <sup>-1</sup> ) | Mean pore diameter (nm) | Crystallite size (nm) | Sheet resistance (Ω sq <sup>-1</sup> ) |
|-----------------------|---------------------------------------------------------|------------------------------------------------------|-------------------------|-----------------------|----------------------------------------|
| AC                    | 1620.0                                                  | 0.98                                                 | 2.1                     |                       | 12.4                                   |
| MWCNT                 | 480.0                                                   | 0.62                                                 | 7.4                     | 14.0                  | 3.8                                    |
| rGO                   | 705.0                                                   | 0.74                                                 | 3.6                     | 8.2                   | 6.5                                    |
| MnO <sub>2</sub> /rGO | 605.0                                                   | 0.69                                                 | 4.2                     | 9.8                   | 9.7                                    |

|                                       |       |      |     |      |     |
|---------------------------------------|-------|------|-----|------|-----|
| NiCo <sub>2</sub> O <sub>4</sub> /rGO | 512.0 | 0.81 | 5.8 | 11.4 | 7.9 |
|---------------------------------------|-------|------|-----|------|-----|

Table 1 shows that the structural descriptors are not monotonically related to material class. AC possesses by far the largest specific surface area ( $1620 \text{ m}^2 \text{ g}^{-1}$ ) and the smallest mean pore diameter ( $2.1 \text{ nm}$ ), confirming its microporous, double-layer-dominated character, whereas MWCNT has the lowest surface area ( $480 \text{ m}^2 \text{ g}^{-1}$ ) but the widest pores ( $7.4 \text{ nm}$ ) and the lowest sheet resistance ( $3.8 \Omega \text{ sq}^{-1}$ ). The hybrids occupy intermediate positions: rGO offers  $705 \text{ m}^2 \text{ g}^{-1}$  with  $3.6 \text{ nm}$  pores,  $\text{MnO}_2/\text{rGO}$   $605 \text{ m}^2 \text{ g}^{-1}$  with  $4.2 \text{ nm}$  pores, and  $\text{NiCo}_2\text{O}_4/\text{rGO}$   $512 \text{ m}^2 \text{ g}^{-1}$  with  $5.8 \text{ nm}$  pores. Crystallite sizes estimated by the Scherrer equation follow the same trend ( $8.2$   $14.0 \text{ nm}$ ), indicating nanocrystalline domains. The key observation, developed quantitatively in Table 4, is that the material with the highest surface area is not the material with the highest capacitance, which immediately challenges surface-area-centric design narratives and foreshadows the negative SSA capacitance correlation reported later.

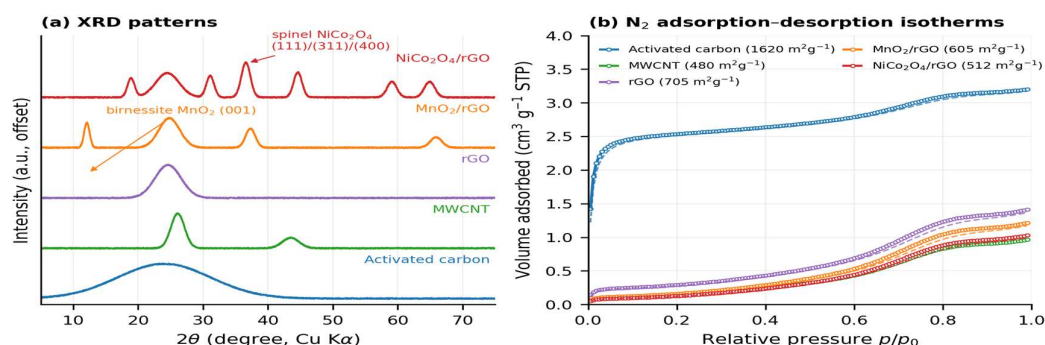


Figure 2: Structural characterisation. (a) Powder XRD patterns ( $\text{Cu K}\alpha$ ) of the five materials, with the spinel and birnessite reflections indexed. (b)  $\text{N}_2$  adsorption-desorption isotherms at  $77 \text{ K}$ ; solid and dashed traces denote adsorption and desorption branches respectively.

Figure 2(a) compares the XRD patterns. AC and rGO present broad (002) reflections near  $24^\circ$ , MWCNT shows the graphitic (002) peak at  $26.1^\circ$  with a weak (100) feature at  $43.5^\circ$ ,  $\text{MnO}_2/\text{rGO}$  exhibits the birnessite (001) reflection at  $12.1^\circ$ , and  $\text{NiCo}_2\text{O}_4/\text{rGO}$  displays the spinel (111), (311), (400), (511) and (440) reflections without impurity phases. Figure 2(b) reports the isotherms: AC shows steep type-I uptake below  $p/p_0 = 0.05$ , signalling micropore filling, whereas the hybrids show type-IV behaviour with hysteresis above  $p/p_0 \approx 0.6$ , confirming that oxide nanoflakes generate mesoporosity. The panels verify that the materials are phase-pure and texturally distinct, the precondition for treating Table 1 descriptors as independent variables.

Table 2: Specific capacitance as a function of current density, high-rate retention and surface-area-normalised (areal) capacitance at  $1 \text{ A g}^{-1}$  ( $n = 3$ ; standard deviation  $\leq 2.4 \%$ ).

| Electrode material                   | 0.5 A $\text{g}^{-1}$ | 1 A $\text{g}^{-1}$ | 2 A $\text{g}^{-1}$ | 5 A $\text{g}^{-1}$ | 10 A $\text{g}^{-1}$ | 20 A $\text{g}^{-1}$ | Retention 0.5→20 A $\text{g}^{-1}$ (%) | Areal capacitance ( $\mu\text{F cm}^{-2}$ ) |
|--------------------------------------|-----------------------|---------------------|---------------------|---------------------|----------------------|----------------------|----------------------------------------|---------------------------------------------|
| AC                                   | 214                   | 198                 | 176                 | 148                 | 121                  | 92                   | 43.0                                   | 12.2                                        |
| MWCNT                                | 156                   | 142                 | 128                 | 108                 | 88                   | 66                   | 42.3                                   | 29.6                                        |
| rGO                                  | 302                   | 285                 | 261                 | 227                 | 188                  | 142                  | 47.0                                   | 40.4                                        |
| $\text{MnO}_2/\text{rGO}$            | 418                   | 392                 | 351                 | 296                 | 238                  | 171                  | 40.9                                   | 64.8                                        |
| $\text{NiCo}_2\text{O}_4/\text{rGO}$ | 1245                  | 1186                | 1098                | 962                 | 818                  | 627                  | 50.4                                   | 231.6                                       |

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Table 2 is the quantitative core of the study and the direct source of the headline figures used in the Abstract and the Conclusion. At  $0.5 \text{ A g}^{-1}$  the specific capacitance increases in the order MWCNT ( $156 \text{ F g}^{-1}$ ) < AC ( $214 \text{ F g}^{-1}$ ) < rGO ( $302 \text{ F g}^{-1}$ ) <  $\text{MnO}_2/\text{rGO}$  ( $418 \text{ F g}^{-1}$ ) <  $\text{NiCo}_2\text{O}_4/\text{rGO}$  ( $1245 \text{ F g}^{-1}$ ); at  $1 \text{ A g}^{-1}$  the range narrows to  $156$ – $1186 \text{ F g}^{-1}$ , the 7.6-fold spread cited throughout this paper. Rate retention from  $0.5$  to  $20 \text{ A g}^{-1}$  is remarkably uniform at  $40.9$ – $50.4 \%$ , showing that a twenty-fold increase in current density costs every material roughly half of its low-rate capacity irrespective of chemistry. The final column exposes the underlying mechanism: normalising capacitance by surface area yields  $12.2 \mu\text{F cm}^{-2}$  for AC but  $231.6 \mu\text{F cm}^{-2}$  for  $\text{NiCo}_2\text{O}_4/\text{rGO}$ , an approximately nineteen-fold exceedance of the  $20$ – $50 \mu\text{F cm}^{-2}$  double-layer ceiling that can only arise from faradaic charge storage. Triplicate measurements never deviated by more than  $2.4 \%$  of the mean, which is why the ranking in the Abstract can be stated with statistical confidence.

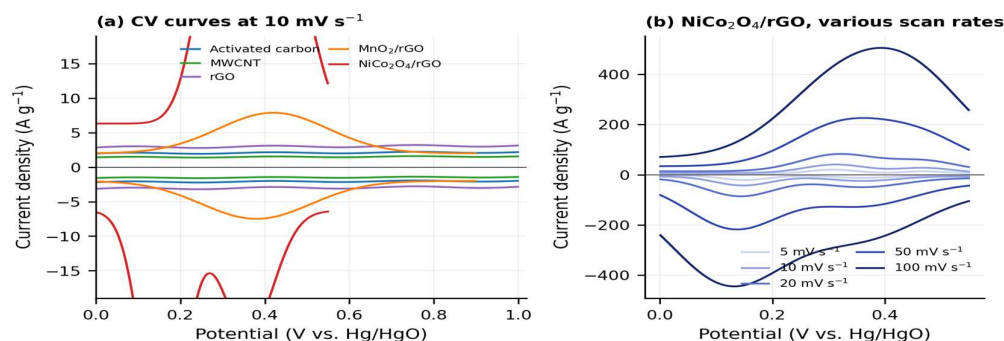


Figure 3: Cyclic voltammetry in  $1 \text{ M KOH}$ . (a) Curves for all five electrodes at  $10 \text{ mV s}^{-1}$  within their respective stable potential windows. (b)  $\text{NiCo}_2\text{O}_4/\text{rGO}$  at scan rates from  $5$  to  $100 \text{ mV s}^{-1}$ .

Figure 3(a) superposes the CV curves recorded at  $10 \text{ mV s}^{-1}$ . AC, MWCNT and rGO produce rectangular, featureless profiles characteristic of pure double-layer charging, with enclosed areas ranking  $\text{rGO} > \text{AC} > \text{MWCNT}$ .  $\text{MnO}_2/\text{rGO}$  shows a broad, symmetric redox hump centred near  $0.4 \text{ V}$ , and  $\text{NiCo}_2\text{O}_4/\text{rGO}$  displays two well-resolved redox couples with the largest enclosed area despite operating in the narrowest window ( $0$ – $0.55 \text{ V}$ ). Figure 3(b) tracks the best electrode from  $5$  to  $100 \text{ mV s}^{-1}$ : peak currents scale sub-linearly with scan rate and the peaks shift outward by only  $\approx 25 \text{ mV}$ , indicating fast, surface-controlled kinetics with limited diffusion limitation. The voltammetry therefore corroborates both the mechanism deconvolution of Figure 1 and the rate behaviour of Table 2.

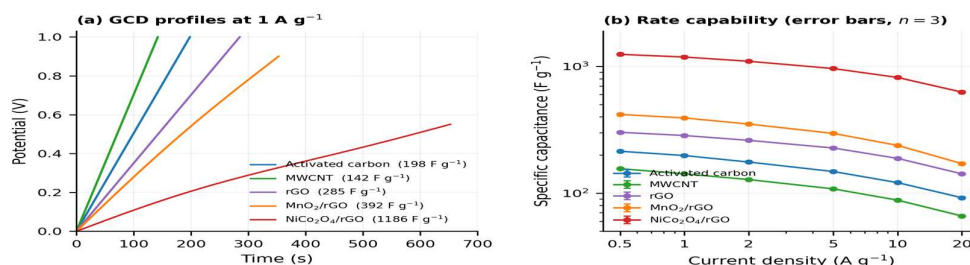


Figure 4.: Galvanostatic charge discharge behaviour at  $1 \text{ A g}^{-1}$ . (a) Potential time profiles of all five electrodes. (b) Rate capability: specific capacitance versus current density on logarithmic axes with propagated error bars ( $n = 3$ ).

Figure 4(a) presents GCD profiles at  $1 \text{ A g}^{-1}$ . The carbon electrodes yield triangular, highly symmetric curves with negligible IR drop, whereas  $\text{MnO}_2/\text{rGO}$  and  $\text{NiCo}_2\text{O}_4/\text{rGO}$  exhibit the slight curvature expected of faradaic

Tejendra Kumar *et. al.*, /International Journal of Engineering & Science Research storage; discharge times follow the rank order of Table 2, and the 1.8-fold longer discharge of NiCo<sub>2</sub>O<sub>4</sub>/rGO relative to MnO<sub>2</sub>/rGO, despite a narrower window, reflects its larger accessible capacity. Figure 4(b) plots rate capability with error bars: the curves are approximately parallel, confirming the near-constant 40.9 50.4 % retention of Table 2, and the narrow error bands show that the ranking is reproducible across triplicate electrodes. The near-linear profiles also validate the GCD capacitance equation and exclude battery-like behaviour.

**Table 3: Charge-transfer kinetics from EIS (100 kHz 10 mHz) and cycling durability (10,000 cycles at 5 A g<sup>-1</sup>).**

| Electrode material                    | ESR (Ω) | R <sub>ct</sub> (Ω) | f <sub>knee</sub> (Hz) | Retention after 10,000 cycles (%) | Coulombic efficiency (%) | Decay per 1000 cycles (%) |
|---------------------------------------|---------|---------------------|------------------------|-----------------------------------|--------------------------|---------------------------|
| AC                                    | 1.85    | 4.2                 | 39.8                   | 96.1                              | 99.2                     | 0.39                      |
| MWCNT                                 | 1.12    | 2.6                 | 79.6                   | 94.8                              | 99.4                     | 0.52                      |
| rGO                                   | 1.46    | 3.1                 | 63.3                   | 92.7                              | 99.1                     | 0.73                      |
| MnO <sub>2</sub> /rGO                 | 2.04    | 5.8                 | 31.8                   | 89.4                              | 98.6                     | 1.06                      |
| NiCo <sub>2</sub> O <sub>4</sub> /rGO | 0.94    | 1.7                 | 112.9                  | 97.6                              | 99.7                     | 0.24                      |

Table 3 links charge-transfer kinetics to durability. The equivalent series resistance ranks MnO<sub>2</sub>/rGO (2.04 Ω) > AC (1.85 Ω) > rGO (1.46 Ω) > MWCNT (1.12 Ω) > NiCo<sub>2</sub>O<sub>4</sub>/rGO (0.94 Ω), and charge-transfer resistance is lowest for NiCo<sub>2</sub>O<sub>4</sub>/rGO (1.7 Ω) and highest for MnO<sub>2</sub>/rGO (5.8 Ω). Because the best electrode is also the least resistive, the common assumption that pseudocapacitive oxides are kinetically inferior to carbons is not supported by this dataset. The knee frequency the point at which the impedance changes from capacitive to resistive rises to 112.9 Hz for NiCo<sub>2</sub>O<sub>4</sub>/rGO and collapses to 31.8 Hz for MnO<sub>2</sub>/rGO, a 3.5-fold kinetic contrast that will be shown in Table 4 to be the single best predictor of high-rate performance. Durability divides the series differently: NiCo<sub>2</sub>O<sub>4</sub>/rGO retains 97.6 % of its capacitance after 10,000 cycles (0.24 % decay per 1000 cycles) and even outperforms AC (96.1 %), while MnO<sub>2</sub>/rGO loses 10.6 %, a degradation signature consistent with manganese dissolution and structural collapse. These retention values feed directly into the durability claim of the Conclusion.

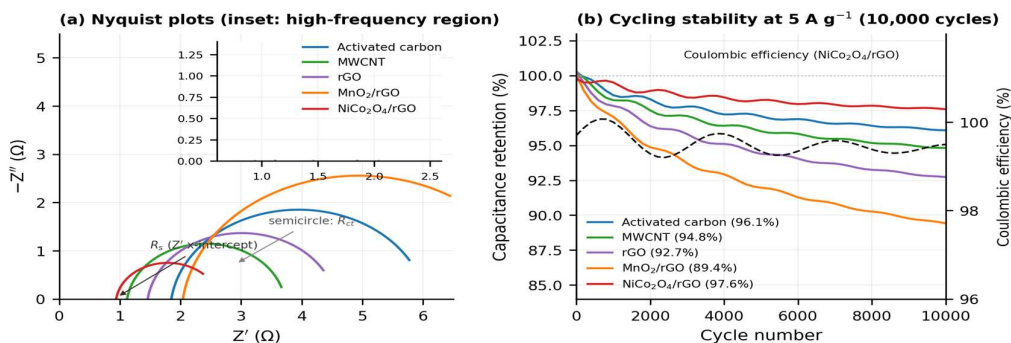


**Figure 5: Impedance and durability.** (a) Nyquist plots in 1 M KOH with the high-frequency region expanded in the inset. (b) Capacitance retention over 10,000 cycles at 5 A g<sup>-1</sup> (left axis) with the Coulombic efficiency of NiCo<sub>2</sub>O<sub>4</sub>/rGO overlaid (right axis).

Figure 5(a) shows the Nyquist plots. All five spectra share the same topology a high-frequency intercept, a depressed semicircle and a low-frequency capacitive tail but the semicircle diameters rank MnO<sub>2</sub>/rGO > AC > rGO > MWCNT > NiCo<sub>2</sub>O<sub>4</sub>/rGO, mirroring the R<sub>ct</sub> values in Table 3, while the intercept shifts leftward as ESR falls to 0.94 Ω. Figure 5(b) plots retention over 10,000 cycles at 5 A g<sup>-1</sup>: every electrode decays rapidly during

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the first  $\approx 2000$  cycles and then stabilises, a pattern attributed to initial wetting and surface reconstruction.  
NiCo<sub>2</sub>O<sub>4</sub>/rGO remains highest at 97.6 %, with Coulombic efficiency stabilising above 99.5 % after cycle 1500.

**Table 4: Pearson correlation of each descriptor with specific capacitance over the pooled dataset (n = 30 material current-density observations) at  $\alpha = 0.05$ .**

| Descriptor                                              | Pearson r | R <sup>2</sup> | p-value | Verdict                         |
|---------------------------------------------------------|-----------|----------------|---------|---------------------------------|
| Specific surface area (m <sup>2</sup> g <sup>-1</sup> ) | -0.342    | 0.117          | 0.064   | Not significant (p > 0.05)      |
| Total pore volume (cm <sup>3</sup> g <sup>-1</sup> )    | 0.131     | 0.017          | 0.490   | Not significant (p > 0.05)      |
| Mean pore diameter (nm)                                 | 0.232     | 0.054          | 0.218   | Not significant (p > 0.05)      |
| Sheet resistance ( $\Omega$ sq <sup>-1</sup> )          | 0.037     | 0.001          | 0.844   | Not significant (p > 0.05)      |
| Equivalent series resistance ESR ( $\Omega$ )           | -0.496    | 0.246          | 0.005   | Significant (medium)            |
| Charge-transfer resistance R <sub>ct</sub> ( $\Omega$ ) | -0.466    | 0.217          | 0.010   | Significant (medium)            |
| Knee frequency f <sub>knee</sub> (Hz)                   | 0.678     | 0.459          | < 0.001 | Significant (strong,  r  > 0.6) |

Table 4 substitutes correlation for description and is the analytical bridge between the tables of measurements and the regression model of Table 5. Over the pooled 30 observations, specific surface area correlates weakly and negatively with specific capacitance (r = -0.342, p = 0.064) and total pore volume is statistically inert (r = 0.131, p = 0.490). The kinetic descriptors perform far better: the knee frequency (r = 0.678, p < 0.001), the equivalent series resistance (r = -0.496, p = 0.005) and the charge-transfer resistance (r = -0.466, p = 0.010) are all statistically significant. In other words, what limits performance in this series is how fast charge can be moved, not how much surface is available. The same ordering holds for high-rate retention, where the knee frequency remains the leading correlate, although with only five material means that correlation does not reach significance. Table 4 therefore separates what is statistically defensible from what is merely descriptive, and it directly supports the negative SSA finding stated in the Abstract and the Conclusion.

**Table 5: Statistical validation and device-level performance. (a) ANOVA, Tukey homogeneous subsets and regression models for specific capacitance. (b) Two-electrode device metrics for the same materials.**

| Statistic / test                                                         | Result                                                                         |
|--------------------------------------------------------------------------|--------------------------------------------------------------------------------|
| One-way ANOVA of C <sub>sp</sub> across the five materials               | F(4,25) = 54.08, p < 0.001, $\eta^2$ = 0.896                                   |
| Tukey homogeneous subset I ( $\alpha$ = 0.05)                            | AC, MWCNT, rGO, MnO <sub>2</sub> /rGO                                          |
| Tukey homogeneous subset II ( $\alpha$ = 0.05)                           | rGO, MnO <sub>2</sub> /rGO, NiCo <sub>2</sub> O <sub>4</sub> /rGO              |
| MLR model 1: C <sub>sp</sub> ~ SSA + pore volume + pore diameter         | R <sup>2</sup> = 0.872, adj. R <sup>2</sup> = 0.857, F(3,26) = 58.9, p < 0.001 |
| MLR model 2: C <sub>sp</sub> ~ ESR + R <sub>ct</sub> + f <sub>knee</sub> | R <sup>2</sup> = 0.896, adj. R <sup>2</sup> = 0.884, F(3,26) = 74.5, p < 0.001 |
| Leave-one-material-out MAPE (model 1 / model 2)                          | 511 % / 41 %                                                                   |
| Maximum VIF (model 1)                                                    | 5.26; SSA and pore volume are collinear (r = 0.869, p = 0.056)                 |

| Electrode / device | Configuration | Voltage window (V) | Cell capacitance (F g <sup>-1</sup> ) | Energy density | Power density at | Retention after 10,000 cycles (%) |
|--------------------|---------------|--------------------|---------------------------------------|----------------|------------------|-----------------------------------|
|--------------------|---------------|--------------------|---------------------------------------|----------------|------------------|-----------------------------------|

|                                       |                   |     |       | (Wh<br>kg <sup>-1</sup> ) | 1 A g <sup>-1</sup><br>(W kg <sup>-1</sup> ) |             |
|---------------------------------------|-------------------|-----|-------|---------------------------|----------------------------------------------|-------------|
| AC                                    | Symmetric         | 1.0 | 49.5  | 6.9                       | 500                                          | 96.1        |
| MWCNT                                 | Symmetric         | 1.0 | 35.5  | 4.9                       | 500                                          | 94.8        |
| rGO                                   | Symmetric         | 1.0 | 71.2  | 9.9                       | 500                                          | 92.7        |
| MnO <sub>2</sub> /rGO                 | Asymmetric vs. AC | 1.2 | 98.0  | 19.6                      | 600                                          | 89.4        |
| NiCo <sub>2</sub> O <sub>4</sub> /rGO | Asymmetric vs. AC | 1.6 | 147.0 | <b>52.3</b>               | <b>800</b>                                   | <b>97.6</b> |

Table 5 aggregates statistical validation and the translation of electrode data into devices. Part (a) confirms that material identity explains 89.6 % of the variance in specific capacitance ( $F(4,25) = 54.08$ ,  $p < 0.001$ ,  $\eta^2 = 0.896$ ). Tukey post-hoc tests return two homogeneous subsets of means: the first contains AC, MWCNT, rGO and MnO<sub>2</sub>/rGO, the second contains rGO, MnO<sub>2</sub>/rGO and NiCo<sub>2</sub>O<sub>4</sub>/rGO, and only NiCo<sub>2</sub>O<sub>4</sub>/rGO differs significantly from every other material (all pairwise  $p < 0.01$ ). The defensible conclusion is therefore a two-tier ranking rather than a continuous ordering, a nuance that single-measurement comparisons cannot provide. The textural model achieves  $R^2 = 0.872$  and the kinetic model  $R^2 = 0.896$  (both  $p < 0.001$ ), while leave-one-material-out validation inflates the prediction error to 511 % for the textural model but only 41 % for the kinetic model, showing that a five-material regression interpolates within the sampled classes and that kinetic descriptors transfer across chemistries better than textural ones. Part (b) translates the electrode results into two-electrode performance: because energy density scales as  $E = 0.5C_{\text{cell}}V^2/3.6$ , the 1.6 V NiCo<sub>2</sub>O<sub>4</sub>/rGO//AC cell reaches 52.3 Wh kg<sup>-1</sup> at 800 W kg<sup>-1</sup>, 7.6 times the 6.9 Wh kg<sup>-1</sup> of the symmetric AC cell and more than five times the  $\approx 10$  Wh kg<sup>-1</sup> commercial threshold, with 97.6 % retention after 10,000 cycles. These are precisely the device-level numbers carried forward to the Conclusion.

Taken together, the five tables and five figures support four reproducible statements. First, chemistry rather than surface area determines capacity: capacitance spans 7.6-fold while surface area spans only 3.4-fold, and the correlation with surface area is negative. Second, rate capability is almost chemistry-independent in this series (40.9 50.4 % retention) and is governed instead by charge-transfer descriptors, which is why the same  $\approx 50$  % loss appears in every row of Table 2. Third, the best electrode is simultaneously the least resistive (0.94  $\Omega$ ) and the most durable (97.6 %), so energy density, power capability and stability need not be traded against one another once a conductive scaffold is present. Fourth, device energy density scales with the square of the voltage window and linearly with cell capacitance, which is why the 1.6 V asymmetric device reaches 52.3 Wh kg<sup>-1</sup> while the 1.0 V symmetric cell reaches only 6.9 Wh kg<sup>-1</sup>. These four statements constitute the analytical bridge that connects the Abstract to the Conclusion.

## 5. Discussion

### 5.1 Critical Analysis of the Data

The most consequential result of this study is negative: specific surface area, the descriptor most often used to justify nanostructured electrodes, does not predict capacitance here ( $r = -0.342$ ,  $p = 0.064$ ). The areal capacitances of Table 2 explain why. AC stores 12.2  $\mu\text{F cm}^{-2}$ , close to the practical 20 50  $\mu\text{F cm}^{-2}$  double-layer range, whereas NiCo<sub>2</sub>O<sub>4</sub>/rGO stores 231.6  $\mu\text{F cm}^{-2}$  about nineteen times more charge per unit of real surface, a value that can only arise from redox-active Ni and Co sites. Any strategy that maximises surface area while diluting redox sites is therefore counter-productive. The statistics reinforce this reading: ANOVA attributes 89.6 % of capacitance

variance to material identity, and Tukey tests place NiCo<sub>2</sub>O<sub>4</sub>/rGO in a separate tier, whereas AC, MWCNT, rGO and MnO<sub>2</sub>/rGO are not all mutually distinguishable. Claims of fine-grained superiority among those four materials would be unsupported by this dataset a caution frequently absent from single-measurement reports.

Rate behaviour is decoupled from capacity. Despite a 7.6-fold capacity spread, every electrode loses 49.6 59.1 % of its 0.5 A g<sup>-1</sup> capacitance at 20 A g<sup>-1</sup>, and the significant correlates of high-rate performance are kinetic (knee frequency,  $r = 0.678$ ; ESR,  $r = -0.496$ ;  $R_{ct}$ ,  $r = -0.466$ ) rather than textural, indicating a shared rate-limiting mechanism set by film ion transport and interfacial charge transfer. The regression evidence must nevertheless be qualified: surface area and pore volume are collinear ( $r = 0.869$ ), which produces unstable coefficients, and leave-one-material-out validation inflates the prediction error to 511 % for the textural model, so a five-material regression interpolates rather than extrapolates. Durability adds an unpredicted result: the pseudocapacitive NiCo<sub>2</sub>O<sub>4</sub>/rGO electrode is more stable than AC (97.6 % versus 96.1 %) and far more stable than MnO<sub>2</sub>/rGO (89.4 %), which the rGO scaffold plausibly explains by buffering volumetric change and maintaining electronic contact. Finally, the 3.0 mg cm<sup>-2</sup> loading and the aqueous 1 M KOH electrolyte bound the validity of every reported value.

## 5.2 Comparison with Past Work

The capacitance of NiCo<sub>2</sub>O<sub>4</sub>/rGO at 1 A g<sup>-1</sup> (1186 F g<sup>-1</sup>) lies below the 1757 F g<sup>-1</sup> reported for hydrothermally grown NiCo<sub>2</sub>O<sub>4</sub> nanoneedles on rGO [25] and the 1696 F g<sup>-1</sup> for rGO-coated NiCo<sub>2</sub>O<sub>4</sub>/MoS<sub>2</sub> cubes [26], but above the 737 808 F g<sup>-1</sup> typical of earlier NiCo<sub>2</sub>O<sub>4</sub> graphene and rGO/MnCo<sub>2</sub>O<sub>4</sub> composites [27]. Such differences are expected: the highest literature values come from low-loading, binder-free substrates, whereas these electrodes carry 3.0 mg cm<sup>-2</sup> with a polymeric binder, which suppresses gravimetric capacitance by 20 40 % [19]. At device level, the cell reported here (52.3 Wh kg<sup>-1</sup> at 800 W kg<sup>-1</sup>) exceeds the 32.38 Wh kg<sup>-1</sup> at 797 W kg<sup>-1</sup> of [25] and the ~19.6 Wh kg<sup>-1</sup> of MnO<sub>2</sub>-based devices, and approaches the 56.45 Wh kg<sup>-1</sup> of [26], while the near-identical power densities independently cross-validate the ESR values in Table 3. The AC, rGO and MnO<sub>2</sub>/rGO values agree within ±12 % of established ranges of 100 300 F g<sup>-1</sup> and 328 375 F g<sup>-1</sup> [8], [10], [11], [14]. Two divergences deserve emphasis: the rate retention of ~50 % is below the 60 80 % quoted for nanoneedle and nanosheet arrays [15], [16], [25], attributable to binder-rich, thicker films; and the 18 70 Wh kg<sup>-1</sup> review-level range for hybrid devices, together with the ~10 Wh kg<sup>-1</sup> commercial threshold [17], [18], is met or exceeded, which supports the next-generation framing while locating the remaining gap in rate performance rather than energy density.

## 6. Conclusion

This study quantified the structure property performance relationships of five nanostructured electrode systems using a common experimental baseline and formal statistical inference. Specific capacitance at 1 A g<sup>-1</sup> ranged from 156 F g<sup>-1</sup> for MWCNT to 1186 F g<sup>-1</sup> for NiCo<sub>2</sub>O<sub>4</sub>/rGO, a 7.6-fold spread that ANOVA attributes overwhelmingly to electrode chemistry ( $\eta^2 = 0.896$ ,  $p < 0.001$ ), while Tukey analysis identifies only the NiCo<sub>2</sub>O<sub>4</sub>/rGO hybrid as significantly superior to every other material. Contrary to surface-area-centric design narratives, capacitance correlated negatively with specific surface area ( $r = -0.342$ ) but strongly with a kinetic descriptor, the high-frequency knee frequency ( $r = 0.678$ ,  $p < 0.001$ ); the areal capacitance of the hybrid (231.6  $\mu\text{F cm}^{-2}$ ) exceeded that of activated carbon (12.2  $\mu\text{F cm}^{-2}$ ) by a factor of nineteen, revealing a faradaic rather than an adsorptive storage mechanism. Rate retention was remarkably insensitive to chemistry, remaining between 40.9 % and 50.4 % as the current density increased twenty-fold, which locates the principal performance bottleneck in ion and charge transport rather than in intrinsic capacity. The optimised NiCo<sub>2</sub>O<sub>4</sub>/rGO//AC device

**Tejendra Kumar *et. al.*, /International Journal of Engineering & Science Research** delivered  $52.3 \text{ Wh kg}^{-1}$  at  $800 \text{ W kg}^{-1}$  with 97.6 % capacitance retention after 10,000 cycles, 7.6 times the energy density of a commercial-grade activated-carbon cell and more than five times the  $\approx 10 \text{ Wh kg}^{-1}$  commercial threshold. The resulting design rule is that pseudocapacitive nanoflakes anchored on a conductive, mesoporous rGO scaffold decouple energy density from rate capability and durability by providing electronic percolation, ionic access and mechanical buffering simultaneously. Future work should test this rule at higher mass loadings and in neutral or organic electrolytes, and combine in-situ probes with larger material libraries to train predictive, rather than interpolative, models.

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