Supplementary Information (SI) for Journal of Materials Chemistry A. This journal is © The Royal Society of Chemistry 2025

Supplementary Information

Engineering nitrogen-doped porous carbon positive electrode for highperformance sodium-ion capacitors: the critical role of porosity, structure and surface functionalities

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1 Experimental method

1.1 Physicochemical characterization

1.1.1 N₂ Adsorption

The specific surface area (SSA) was calculated by the Brunauer-Emmett-Teller (BET) model from the linear dependence at relative pressures ranging from 0.01 to 0.05. The micropore volume (V_{micro}) was determined using Dubinin-Radushkevich model. The mesopore volume (V_{meso}) was determined by subtracting the " V_{micro} " from the total pore volume of N_2 adsorbed at a relative pressure (P/P₀) of 0.99. The pore size distribution (PSD) was determined from the adsorption axis of the N_2 isotherm. The two-dimensional non-local density functional theory (2D-NLDFT) heterogeneous surface model for carbon materials @ 77 K was used on SAIEUS (Micromeritics).¹ The average micropore (L_{0_micro}) and mesopore diameter (L_{0_meso}) were determined as reported elsewhere.²

1.1.2 **TPD-MS**

The measurements of functional groups determined by TPD-MS were performed using a procedure described elsewhere.³ Briefly, the NDPC were thermally treated under a secondary vacuum (10⁻⁷ Torr) up to 950 °C at a heating rate of 5 °C min⁻¹, while the released gases due to the decomposition of functional groups were continually followed by a mass spectrometer. The total amount of each released gas was evaluated by time integration of the TPD curves. For the CO and CO₂ gas profile deconvolution, the full width at half maximum (FWHM), position, and area of the anhydride peaks in the CO and CO₂ profiles were preserved. A Gaussian model was used for peak fitting across all NDPC materials.^{4,5} The CO₂ groups were deconvoluted into three main peaks (Figure S4b): two peaks corresponding to strong and weak carboxylic acids (100 – 500 °C) and a third peak corresponding to anhydrides (400 – 650 °C). The CO groups were deconvoluted (Figure S4c) mainly into anhydrides, phenol/ethers (550 – 850 °C), and carbonyl/quinones (750 – 950 °C). Furthermore, TPD-MS facilitated the quantification of active sites by assessing the active surface area (ASA). The ASA was determined after O_2 chemisorption at 150 °C for 10 h,⁵ considering the amount of CO and CO₂ released and the area of the carbon edge site.³

1.2 Electrochemical calculations

The Dunn method⁶ is evaluated using the power law equation given by:

$$I(v) = k_1 v + k_2 v^{1/2}$$
(1)

Where "v" is the scan rate, and k_1v and $k_2v^{1/2}$ are the capacitive effect (or pseudocapacitive) and diffusion-controlled behavior, respectively. The value of k_1 and k_2 are obtained by plotting $l/v^{1/2}$ versus $v^{1/2}$.

The formula below were used for the evaluation of the energy and power density:^{7–9}

$$E = \frac{1}{2} \times C \times (V_2^2 - V_1^2) - \dots - (2)$$
$$C = \frac{I}{(\frac{dV}{dt}) \cdot M} - \dots - (3)$$

Where E is the energy density in Wh Kg⁻¹, C is the specific capacitance in F g⁻¹, V_2 is the maximum potential after deducting the ohmic drop, V_1 is the minimum potential in volts, M is the mass of active mass in g and dV/dt is the slope of the discharge curve.

$$P = \frac{E}{\Delta t_d} \times 3600 ----- (4)$$

Where P is the power density in W Kg⁻¹, E is the energy density in Wh Kg⁻¹ and t_d is the discharge time in seconds.

Table S1. Textural properties of the synthesized porous carbon materials in a powder state by

Sample	V _{micro} (cm ³ g ⁻¹)	V _{meso} (cm ³ g ⁻¹)	V _t (cm ³ g ⁻¹)
ND1-CsCl-T	0.86	0.21	1.07
ND2-CsCl-T	0.81	0.22	1.03
ND4-CsCl-T	0.64	0.25	0.89
ND1-LiCl-T	0.48	0.59	1.07
ND1-LiNaCl-T	0.41	0.37	0.78
ND1-CsLiNaCl-T	0.77	0.22	0.99

 N_2 adsorption.



Figure S1. C 1s (a); O 1s (b); and N1s (c) high resolution XPS spectra of ND1-CsCl-T, ND2-CsCl-T, ND4-CsCl-T and ND1-LiNaCl-T.



Figure S2. High-resolution XPS spectra deconvolution of ND1-CsCl-T : C 1s (a); O 1s (b); and N 1s (c); schematic representation of N-doped carbon, illustrating nitrogen surface groups, created by the authors with <u>Biorender.com</u> (d).



Figure S3. TPD-MS of HCN profiles for NDPC materials (a); mass spectra of HCN (source: NIST Chemistry WebBook, <u>https://webbook.nist.gov/chemistry</u>) (b).



Figure S4. Typical oxygen functional groups present on the NDPC materials porous carbon surface and the corresponding CO and CO2 gases released after their thermal decomposition according to the TPD-MS,¹⁰ created by the authors with <u>https://www.biorender.com</u> (a); deconvolution of the CO₂ (b); and CO (c) gas profiles into the corresponding O-functional groups for ND1-LiNaCl-T; nitrogen functional groups present on the carbon surface and their decomposition gases by **TPD-MS** (d), by thermal created the authors with https://www.biorender.com.



Figure S5. TPD-MS gas desorption profiles of NDPC materials showing desorption of NO (a); NH_3 (b); H_2 (c).



Figure S6. Plot of the specific surface area against the active surface area of the NDPC materials (a); linear correlation between the active surface area and the gas desorbed amount of CO (b); CO_2 (c); and N_2 (d).

Samples	0.05 A g ⁻¹	0.1 A g ⁻¹	5 A g ⁻¹	10 A g ⁻¹	0.1 mVs ⁻¹	1 mVs ⁻¹	50 mVs ⁻¹	100 mVs ⁻¹	% CR @ 100 mVs ⁻¹
ND1-CsCl-T	159	148	3	0	181	157	41	26	15
ND2-CsCl-T	139	131	0	0	167	140	38	22	14
ND4-CsCl-T	122	117	0	0	147	124	38	23	19
ND1-LiCl-T	92	85	3	0	102	90	38	26	25
ND1-LiNaCl-T	83	78	14	1	106	81	30	21	30
ND1-CsLiNaCl-T	142	131	2	0	164	140	43	28	16

Table S2. Summary of the electrochemical performance of the materials, based on half-cell results obtained from GCD and CV measurements.The values in the table are reported in mAh g⁻¹, except for the last column which represents % Capacity Retention (CR).

Table S3. Comparison of electrochemical performance of porous carbon-based cathodic half cells vs. Na metal at 0.1 A g⁻¹ current density from the literature.

Cathode materials	Electrolyte	Mass loading (mg cm ⁻²)	SSA (m ² g ⁻¹) (N%)	Potential window (V)	Specific capacity (mAh g ⁻¹)
CSUN800 ¹¹	1 M NaPF ₆ DME	0.7	2650 (6.4)	2-4	112
PDPC ¹²	1 M NaClO ₄ EC:DMC:EMC (1:1:1) 5.0% FEC	0.8 – 1 mg	-	2-4.3	123
NPC-60 ¹³	1M NaClO ₄ EC :DEC	1.5 - 6.0	1022.27 (4.7)	2 – 4.5	101
AJPC-M ¹⁴	1 M NaPF ₆ EC:DEC (1:1)	0.8	1529.75	1.5 - 3.3	70
AGLC ¹⁵	1 M NaClO ₄ EC:DEC (1:1) 2 vol% FEC	-	2961	2-4	71
NS-GHNS ¹⁶	1 M NaClO ₄ EC:DEC (1:1)	1.2 – 1.4	320 (3.34)	2.5 – 4.2	52 @ 0.2 A g ⁻¹

ANP ¹⁷	1 M NaClO ₄ EC:DEC (1:1)	1	2970 (0)	2-4.2	121
MERK-14 ¹⁸	1 M NaClO ₄ EC:DEC (1:1)	1.5 - 3	2286 (1.3)	1.5 – 4	136
CKNa-800mT ¹⁹	1 M NaClO ₄ EC:DEC (1:1)	2	2750 (0)	2-4	114
NHPC ²⁰	1 M NaPF ₆ in 1:1 (EC:PC)	-	2225	1.5 – 4	63
CBC-C ²¹	1 M NaClO ₄ EC:DEC (1:1)	0.9	3229	2.7 – 4.2	47
PCC ²²	1 M NaClO ₄ EC:DEC (1:1)	1	2325	2-4.2	95
ND1-CsCl-T*			2412(2 4)		159
ND2-CsCl-T*	1 M NaPE, EC.DMC		2181 (6.1)		139
ND4-CsCl-T*	(1:1)	7 - 8	1816 (8.0) 2171	1.5 - 4.5	122
ND1-CsLiNaCl-T* Kynol-5092-20*			2019		142 103
		* 1 .	1		

*This work

S/N	Properties	Kynol 5092-20
1	Specific surface area (m ² g ⁻¹)	2019
2	$L_0(nm)$	0.94
3	V _{micro} (cm ³ g ⁻¹)	0.82
4	$V_{meso} (cm^3 g^{-1})$	0.05
5	$V_{total} \left(cm^3 \ g^{-1} ight)$	0.87
6	CO (mmol g ⁻¹)	0.41
7	$CO_2 \text{ (mmol g-1)}$	~ 0.10
8	ASA $(m^2 g^{-1})$	20

Table S4. Physico-chemical properties Kynol 5092–20 (provided by Kynol®), a commercial porous carbon cloth, extracted from our recent studies.⁵



Figure S7. Electrochemical performance of Kynol 5092–20 in Na metal half cells, presenting the CV curve (a) and GCPL (b). The activated carbon was directly used as a self-standing electrode, without binder or carbon black additives. It was tested in half vs. sodium metal in 1 M NaPF₆ EC:DMC (1:1) electrolyte, within a potential range of 1.5 - 4.5 V, with a mass of loading 14.9 mg cm⁻² and an electrode diameter of 11 mm.



Figure S8. CV Curves for NDPC materials at various scan rates $(0.1 - 20 \text{ mV s}^{-1})$ for ND1-CsCl-T (a); ND4-CsCl-T (b) and ND1-LiNaCl-T (c).



Figure S9. Graph depicting the b value as a function of voltage potential for the cathodic peak, including the average b value for ND1-CsCl-T, ND4-CsCl-T, and ND1-LiNaCl-T (a); Analysis of the diffusive and capacitive contributions to the total current at different scan rates for ND1-CsCl-T (b); ND4-CsCl-T (c); ND1-LiNaCl-T (d).



Figure S10. Plot of the specific capacity from GCPL versus the specific surface area at current density of 0.1 A g^{-1} (a) 0.5 A g^{-1} (b) and 1 A g^{-1} (c); Plot of the capacity retention against the specific surface area at 1 A g^{-1} (d).



Figure S11. Capacity retention represented as a function of textural properties, specific surface area (a); volume of micropores with average micropores diameter inset (b); volume of mesopores with average mesopores diameter inset (c); structural properties, area ratio of D_1 and G_1 (d); Csp^2/Csp^3 (e); and active surface area (f).



Figure S12. Specific capacity as a function of total CO desorption amount (a); total CO_2 desorption amount (b); total $CO_X (CO + CO_2)$ desorption amount (c).



Figure S13. Capacity retention represented as a function of surface chemical groups, phenol/ethers (a) anhydride (b) COOH (with specific capacity inset) (c); N_2 (d); NO (with specific capacity inset) (e); and pyrrolic-N (f).



Figure S14. HC-R-St anode presodiation at C/10 ($1C = 372 \text{ mAh g}^{-1}$) for five cycles under constant current-constant voltage testing conditions.



Figure S15. Plot showing the specific capacity as a function of current density for the optimized HC-R-St (anode) // ND4-CsCl-T (cathode) ratio (a) and HC-R-St (anode) // ND1-LiNaCl-T (cathode) ratio (b).

Table S5. Comparison of electrochemical performance of NDPC full cell with dual carbon sodium-ion capacitors from the literature at a current density of 0.1 A g⁻¹.

Anode // Cathode	Potential window (V)	Electrolyte	Specific capacity (mAh g ⁻¹)
AGS-650//AGA-850 ²³	1-4	1 M NaClO ₄ EC:DEC (1:1)	49 - 50
JPC-D//APJC-M ¹⁴	0.15 - 3.29	1 M NaPF_6 EC:DEC (1:1)	43 (0.3 A g ⁻¹)
PFC//PFAC ²⁴	0 - 4	1 M NaClO ₄ EC:DMC (1:1)	45.7 F g ⁻¹
MDC//K-MDC ²⁵	0 - 4	1 M NaClO ₄ EC:DEC (1:1)	49
Mo ₂ C/C-2//PC ²⁶	0 - 4	-	56.9 F g ⁻¹
Sb@NC//PDPC ²⁷	1 - 4	1 M NaClO ₄ EC:DMC:EMC (1:1:1) 5.0% FEC	60.2
PNPOC-800//APNPOC-800 ²⁸	0 - 4	1 M NaPF ₆ EC:DEC $(1:1)$	48.65 F g ⁻¹
SCNP//ANP ¹⁷	1 - 4	1 M NaClO ₄ EC:DEC (1:1)	50
HAT-CNF//STC-16 ²⁹	0.5 - 4	1 M NaClO ₄ EC:PC:FEC (45:45:10, mass)	55.8 F g ⁻¹
HC-R-St//ND4-CsCl-T _(1:1.5)*	1.5 – 4.5	1 M NaPF ₆ EC:DMC (1:1)	66 (83.5 F g ⁻¹)

*This work



Figure S16. Evaluation of the energy and power density of the sodium-ion capacitor compared to other hybrid technology (potassium/lithium-ion capacitor) in the literature.^{30–34}

Table S6. Comparison of electrochemical performance of dual carbon-based sodium-ion capacitors and other hybrid metal ion based technologyreports from the literature. The table includes data on energy density (ED, Wh kg^{-1}) and power density (PD, W kg^{-1}).

Anode // Cathode	Materials type	Potential window (V)	Electrolyte	Maximum ED	Maximum PD	Cycling stability
		SIC –	Carbon Anode /	/ Carbon Catho	ode	
HC-R-St//ND4-CsCl- T (1:1.5) – this work	hard carbon // porous carbon	1.5 - 4.5	1 M NaPF ₆ EC:DMC (1:1)	209@150	75@6000	80% @4200 cyles@0.1 A g ⁻¹
MER1200//MERK14	hard carbon // porous carbon	1 - 4	1 M NaClO ₄ EC:DEC (1:1)	195.4@85	72.3@13800	91.2% @16000 cyles@5 A g ⁻¹
RGO400//RGO400 35	reduced graphene oxide // reduced graphene oxide	1 - 4	1 M NaPF ₆ DME	91@631	48@10896	98% @1800 cyles@5 A g ⁻¹
AGS-650//AGA-850	carbon sponges // porous carbon	1-4	1 M NaClO ₄ EC:DEC (1:1)	120@250	72@24400	78% @10000 cyles@2 A g ⁻¹
JPC-D//APJC-M ¹⁴	hard carbon // porous activated carbon	0.15 - 3.29	1 M NaPF ₆ EC:DEC (1:1)	86@636	16@3440	92% @1000 cyles@1.2 A g ⁻¹
PFC//PFAC ²⁴	porous framework carbon // porous framework activated carbon	0 – 4	1 M NaClO ₄ EC:DMC (1:1)	101.6@200	51.1@20000	71.8% @10000 cyles@2 A g ⁻¹
MDC//K-MDC ²⁵	metal–azolate framework-6s- derived carbons // KOH-assisted pyrolysis of MDCs	0-4	1 M NaClO ₄ EC:DEC (1:1)	100@200	53.2@20000	80% @1000 cyles@1 A g ⁻¹
PNPOC- 800//APNPOC-800 ²⁸	N, P, O ternary- doped	0-4	1 M NaPF ₆ EC:DEC	105.48@185	37.58@1359 0	87.43% @9000 cyles@1 A g ⁻¹

	mesoporous carbon // alkaline- activated PNPOC- 800		(1:1)			
SCNP//ANP ¹⁷	S-doped carbon nanoparticles // activated nanoparticles Ordered	1 – 4	1 M NaClO ₄ EC:DEC (1:1)	105@185	69@25000	76% @10000 cyles@2 A g ⁻¹
OMC//N-OMC ³⁶	microporous carbon // nitrogen- doped ordered microporous carbon	0-4	1 M NaClO ₄ EC:PC (1:1) 5.0% FEC	119@73	31@5807	85% @1800 cyles@5 A g ⁻¹
HAT-CNF//STC-16 ²⁹	Microporous nitrogen-rich carbon fibers // Salt-templated carbon	0.5 – 4	1 M NaClO ₄ EC:PC:FEC (45:45:10, mass)	95@190	18@13000	90% @1000 cyles@1 A g ⁻¹
As8Mg//AC ³⁷	N-doped carbon // Activated carbon	0.5 – 4	1 M NaPF ₆ EC:DEC (30:70, vol%)	224@53	51@10410	99.7% @600 cyles@0.2 A g ⁻¹
P,N-HPCNS- KCl/Ice//N-PCNS- KOH ³⁸	P,N-Doped Interconnected Carbon Nanosheets with Hierarchical Porosity using KCI/Ice As Dual- Templates // KOH-treated N- doped carbon nanosheets	0-4	1 M NaClO ₄ EC:PC:FEC (1:1:0.05, vol%)	135.3@30	40@16100	88.6% @8000 cyles@5 A g ⁻¹
		SIC – Carl	oon composite An	ode // Carbon	Cathode	

Mo ₂ C/C-2//PC ²⁶	Molybdenum carbide/carbon composite // porous carbon	0-4	-	50.2@200	16.7@10000	77.5% @1800 cyles@5 A g ⁻¹
VNQDs@PCNFs- N/F//APCNFs-N/F ³⁹	Quantum Nutride Quantum Dots Modified One- Dimensional Carbon Cages // Activated N/F co- doped carbon	0-4	-	198.8@157	95@9100	73.5% @8000 cyles@1 A g ⁻¹
Sb@NC//PDPC ²⁷	Sb-carbon composite // Polyaniline- Derived Porous Carbon	1 – 4	1 M NaClO ₄ EC:DMC:E MC (1:1:1) 5.0% FEC	157@230	49@25000	80% @4000 cyles@2 A g ⁻¹
In ₆ S ₇ /NSC HMS//PDPC ⁴⁰	sulfur co-doped carbon hollow microspindles // polyaniline derived porous carbon	1-3.8	1 M NaPF ₆ in DME	136.3@473.6	52.1@47466	68.5% @20000cyles@5 A g ⁻¹
]	Potassium-ion cap	pacitor (KIC)		
KTO // NGCC ³⁰	K ₂ Ti ₆ O ₁₃ (KTO) microscaffolds // N-doped nanoporous graphenic carbon	0-3.5	1 M KPF ₆ PC (5% FEC)	58.2@160	12@7200	75.5% @ 5000 cycles @1 A g ⁻¹
CNS//FCDAC KIC ³¹	Carbon nanosheets // framework carbon derived activated carbon	0-4.2	0.8 M KPF ₆ EC:DEC (1:1, v/v)	149@210	40@21000	80% @ 5000 cycles @2 A g ⁻¹

2D-MCM- 1500//PDPC KIC ³²	Two-dimensional mesoporous carbon microcoins (2D-MCMs) // Polyaniline- derived porous carbon	1 – 4.06	3 M KFSI EC:DEC (1:1 v/v)	113@495	51.25@5100 0	73% @ 10000 cycles @2 A g ⁻¹
			Lithium-ion capa	citor (LIC)		
SHC//ZTF-C LIC ³³	Sucrose hard carbon // zinc 2,3,5,6- tetrafluoroterepht halic derived carbon	1.5 – 4	1 M LiPF ₆ EC:DEC:DM C (1:1:1 in vol%)	157@103.7	37.3@40000	85% @ 5000 cycles @5 A g ⁻¹
TTFTTA-PDA//AC LIC ³⁴	Tetrathiafulvalene tetrathiophenal p- phenylenediamine // Activated carbon	2 - 4	1 M LiPF ₆ EC:DEC (1:1, vol%)	140@233	91@9328	81.3% @ 42200 cycles @4 A g ⁻¹

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