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A Quantitative Image Analysis Model for Evaluating Fiber Diameter Distribution & Surface Porosity of Electrospun Fibers

D. Markoglou[1], **N. Bali**[1*], **I. Chronopoulos**[1], **S. Yannopoulos**[1],[2]

[1] Foundation for Research and Technology Hellas, Institute of Chemical Engineering Sciences, 26504 Patras, Greece
[2] Department of Chemistry, University of Patras, 26504 Patras, Greece • contact: nbali@iceht.forth.gr

1 Introduction

Lignin, the most abundant aromatic biopolymer on Earth, is a premier **renewable carbon source** for next-generation energy-storage electrodes. Its high carbon content and intrinsic polyphenolic structure make it an ideal "green-energy" precursor [1].

Through **electrospinning** followed by **CO_2 laser-induced graphitization**, lignin is converted into 3D porous, graphene-like scaffolds — a sustainable alternative to petroleum-based polymers [2].

The central challenge: bridging **synthesis** and **predictive supercapacitor performance**. Here we implement a **computational optimization framework** that quantifies structural descriptors — fiber diameter, porosity, orientation — directly from SEM imagery, enabling high-fidelity correlation between geometric design and electrochemical behaviour.

41.42 %
Global surface porosity

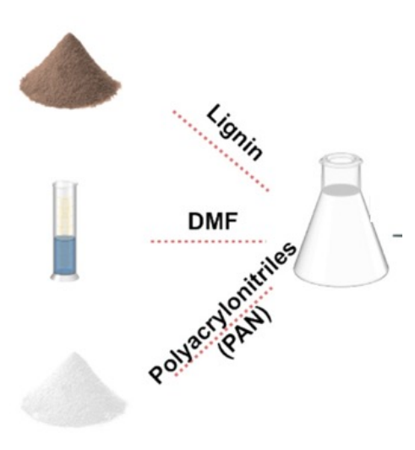
~500
Watershed segments

320.7 nm
Mean fiber diameter

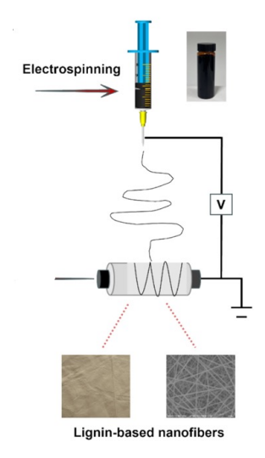
2 Methodology

EXPERIMENTAL — Fiber Production

Step 1
Solution prep



Step 2
Electrospinning



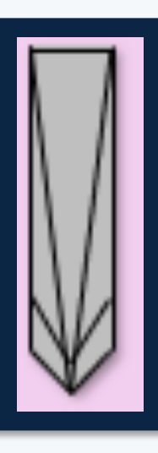
Step 3
Stabilization

Step 4
Laser irradiation

→ *In-situ graphitization via CO_2 laser (10.6 μm)* → *3D porous graphene-like electrode*

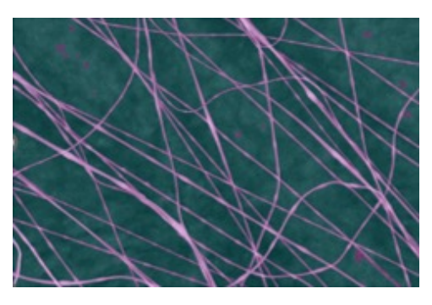
Binder-free Lignin-PAN carbon-fiber electrode

Flexible monolith ready for 3-electrode testing.



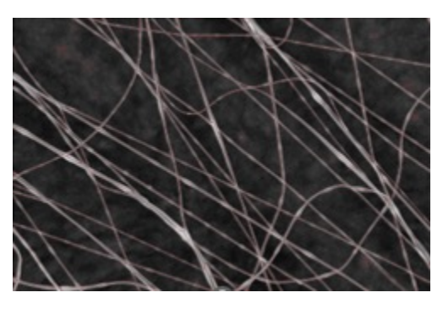
COMPUTATIONAL — Image Framework

Adaptive Thresholding 01



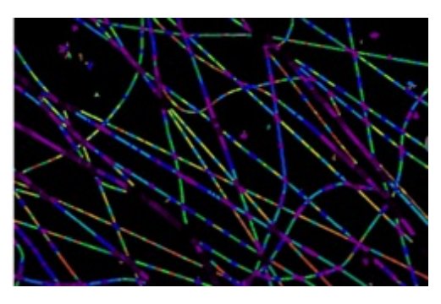
Sauvola local algorithm segments fibers from the background.

Skeletonization 02



Each fiber reduced to a single-pixel centerline.

Watershed Segmentation 03



Resolves overlaps; isolates fiber segments.

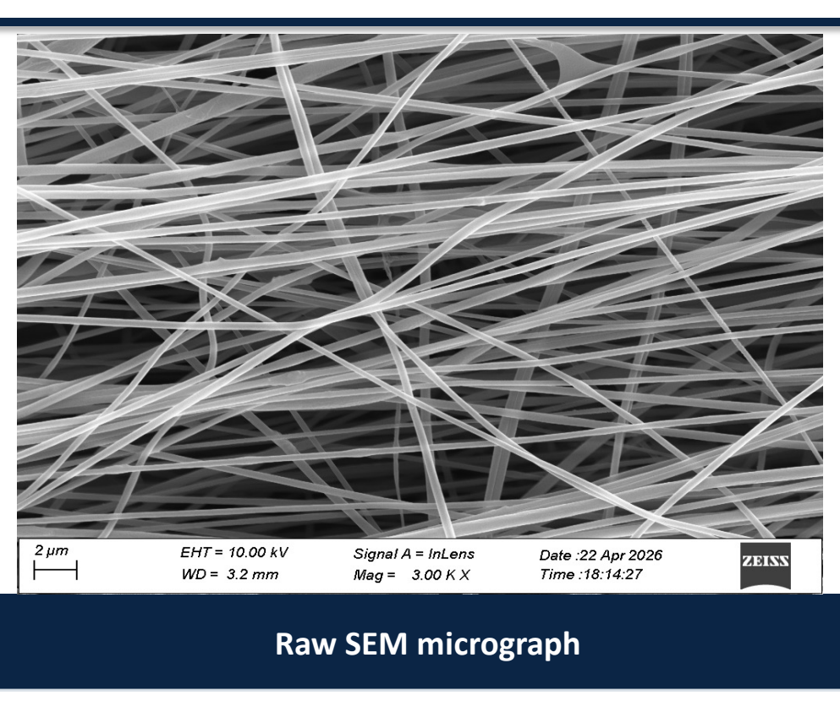
Integrated structural quantification

A custom pipeline couples SEM with adaptive segmentation, skeleton analysis and watershed isolation — yielding high-fidelity descriptors of diameter, porosity & orientation directly linked to electrochemistry.

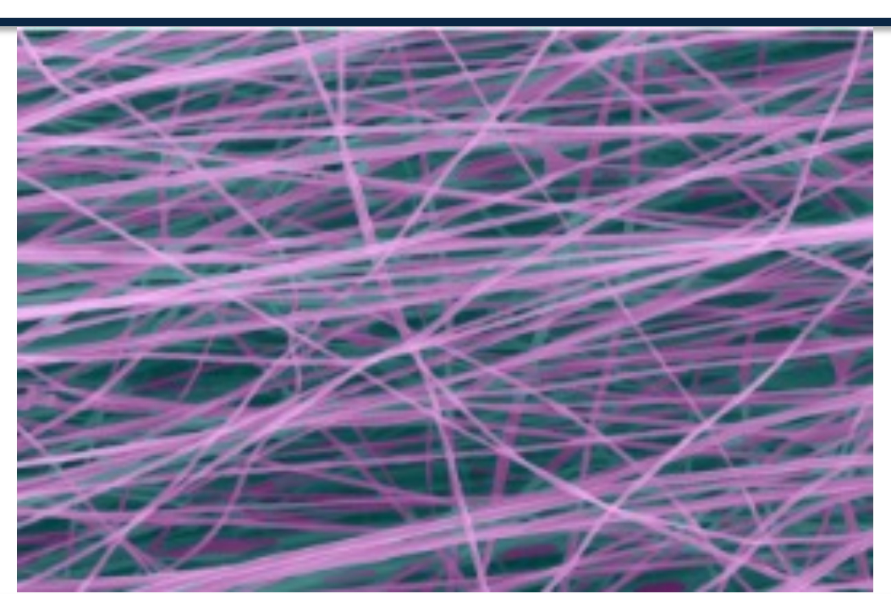
RESULTS

Integrated physicochemical, electrochemical & morphological analysis of Lignin-PAN carbon-fiber electrodes

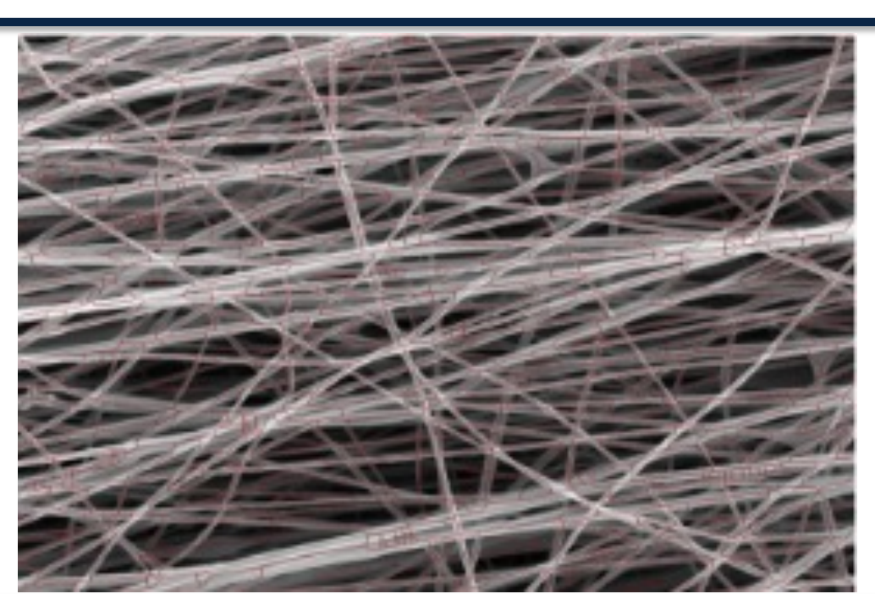
3 Quantitative Morphological Mapping · SEM and Image based Model



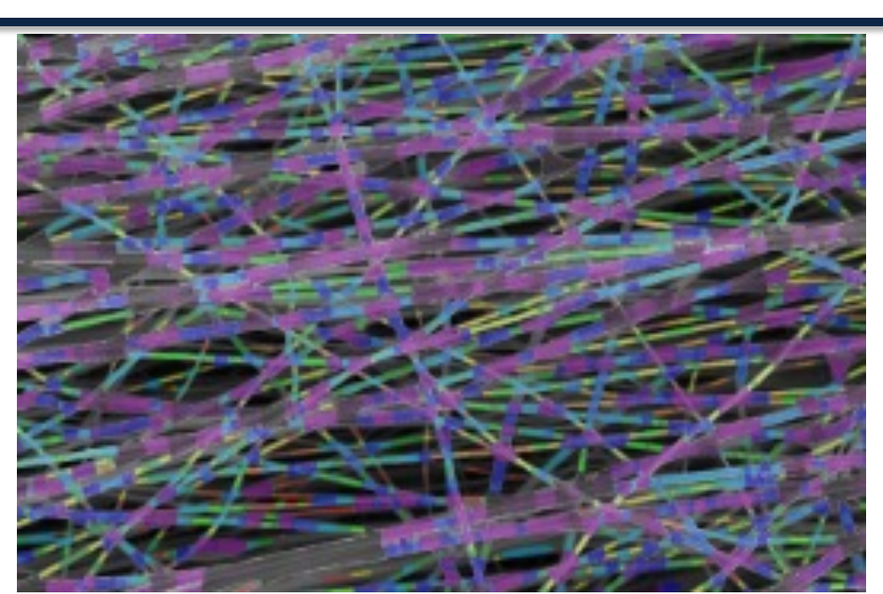
Raw SEM micrograph



Adaptive thresholding



Skeletonization



Watershed segmentation

Fiber diameter distribution

Statistical analysis across 16 independent fibers (N = 10,528 segments)

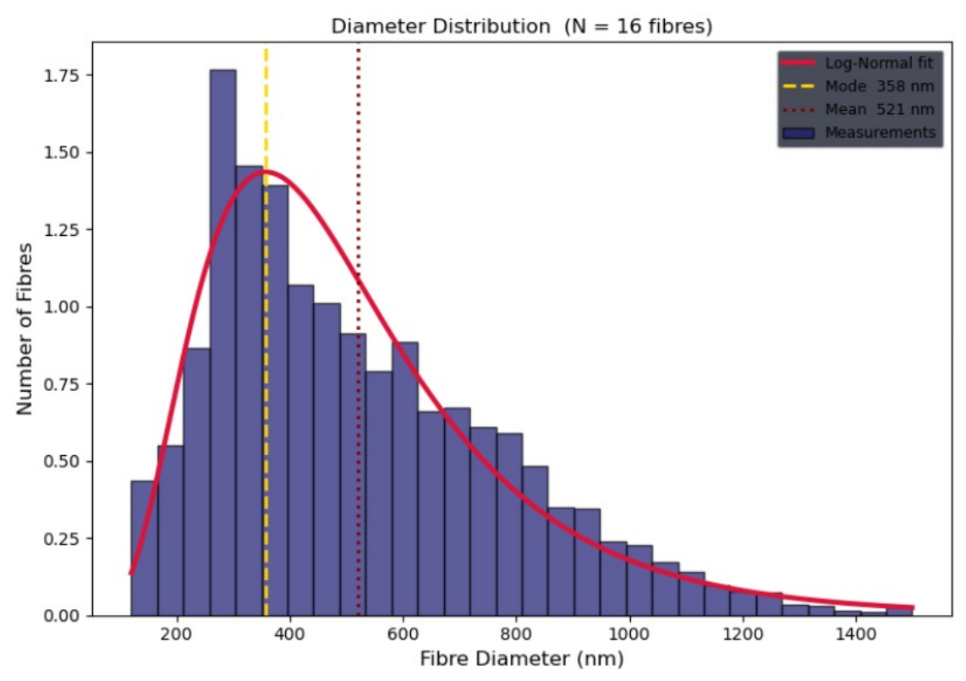


Figure 1 — Diameter histogram & log-normal fit

Fiber orientation analysis

Polar rose plot — randomness index R ≈ 1.000

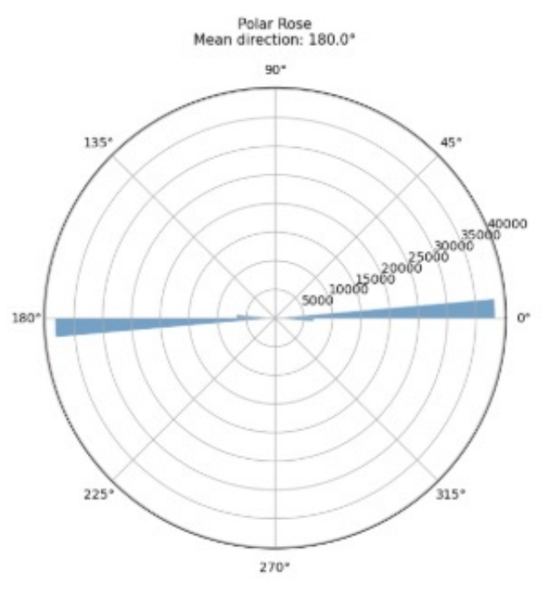


Figure 2 — Orientation rose plot

Key morphological metrics

Watershed segments	10,528
Independent fibers	16
Mean ± SD	520.7 ± 256.7 nm
Mode (Log-Normal)	357.5 nm
Surface porosity	41.42 %
Mean pore ECD	980.9 nm

The computational image analysis pipeline developed in this study was successfully validated on as-spun electrospun Lignin-PAN fibers to establish a baseline for static morphological properties.

Future work will focus on evolving this framework into dynamic modeling simulations to track structural transformations in real-time.

The fiber networks will undergo thermal stabilization and subsequent carbonization processes; the algorithm will be extended to monitor the corresponding evolution of fiber shrinkage, pore closure, and orientation shifts throughout these thermal treatments.

The pipeline will be upgraded to support high-throughput batch processing, enabling the automated, simultaneous evaluation of large image datasets across various processing timeframes to ensure statistical significance.

Surface porosity across SEM samples

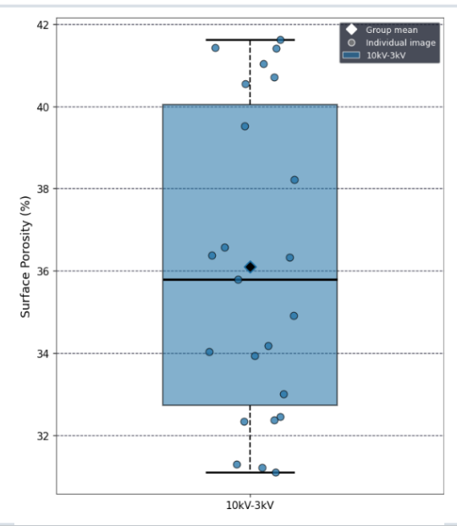


Figure 3 — Surface porosity boxplot

Robust architecture

41.42 % global porosity ensures unhindered electrolyte access and rapid ion diffusion through the electrode.

Statistical reliability

Reported values backed by 10,528 watershed segments across 16 independent skeleton components.

Beyond qualitative SEM

Custom pipeline detects structural bottlenecks invisible to manual SEM screening — a diagnostic tool, not just visualization.

4 Phase Validation & Electrochemical Performance · 3-electrode setup

Raman spectra — Lignin-PAN

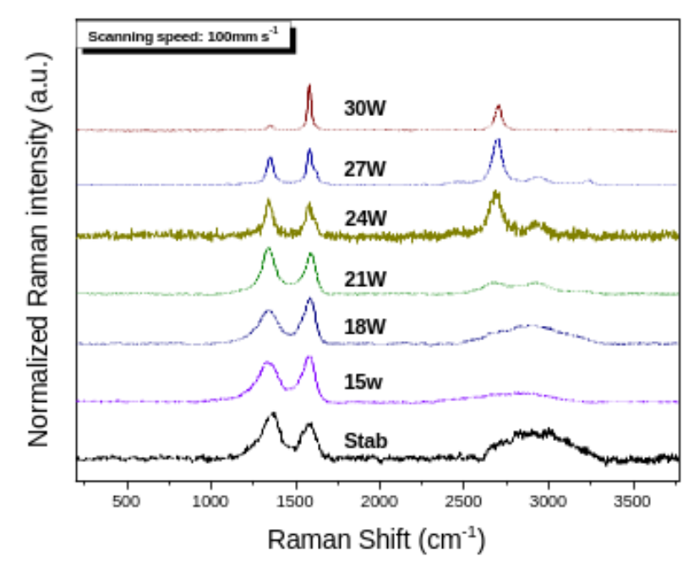


Figure 4 — Raman spectra — Lignin-PAN

Dominant D, G & 2D bands; different laser powers indicate different graphitization levels.

Lorentzian deconvolution

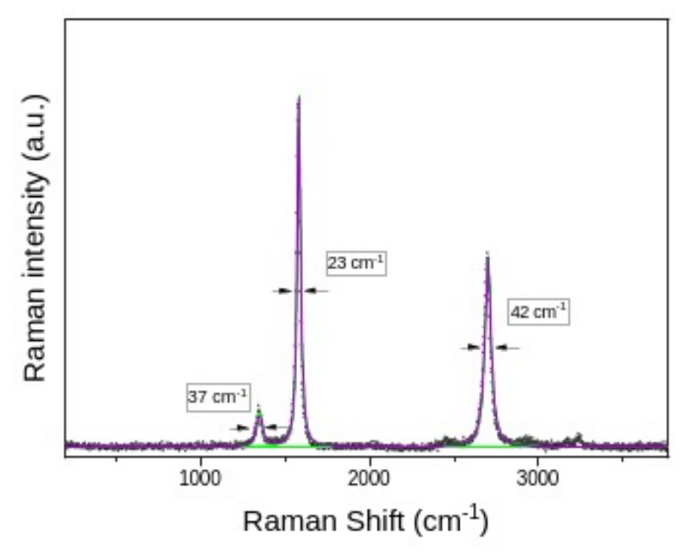


Figure 5 — Lorentzian deconvolution

Single-Lorentzian 2D band → turbostratic arrangement of graphene layers.

Sheet resistance vs. laser power

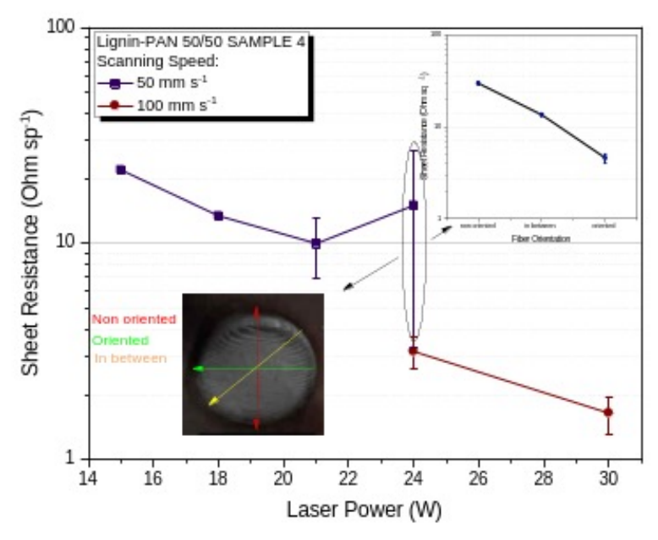


Figure 6 — Sheet resistance vs. laser power

Different laser parameters yield different graphitization & conductivity profiles.

Cyclic voltammetry

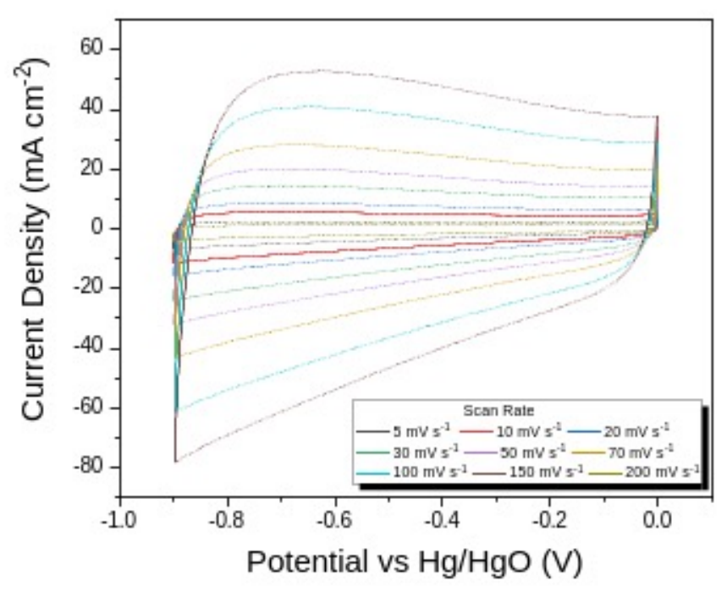


Figure 7 — Cyclic voltammetry

Quasi-rectangular CV → predominant electric double-layer behaviour (EDLC).

Cycling / rate performance

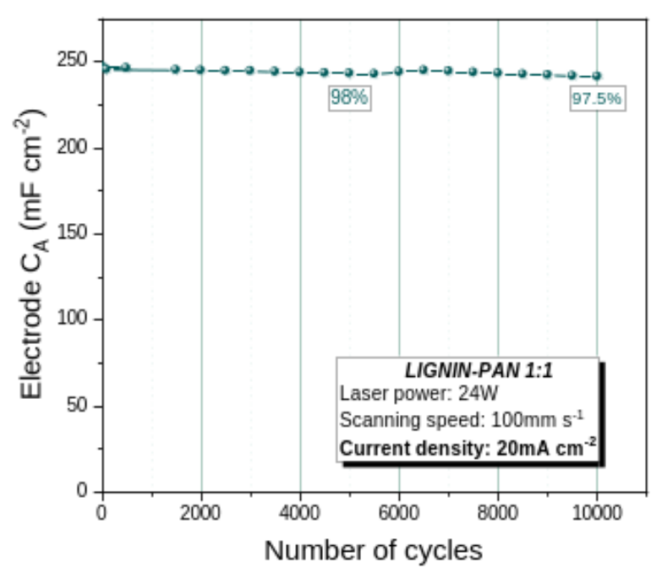


Figure 8 — Cycling / rate performance

Negligible capacitance fade after 10,000 cycles — exceptional long-term stability.

Electrochemical Impedance Spectroscopy

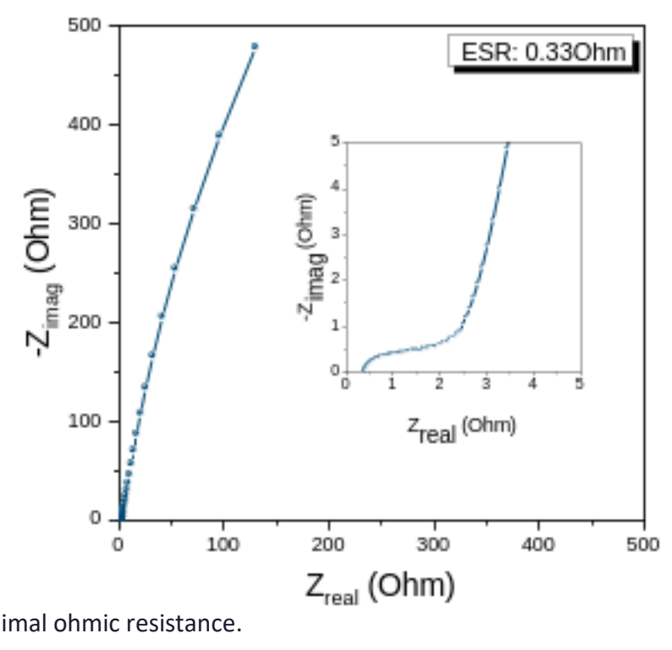


Figure 9 — Electrochemical Impedance Spectroscopy

Very low ESR (0.33 Ω sq^{-1}) — excellent conductivity & minimal ohmic resistance.

310 mF cm^{-2}
Areal Electrode Capacitance at Scan Rate 5 mV s^{-1}

0.33 Ω sq^{-1}
Equivalent Series Resistance

97.5 %
Capacitance Retention after 10 000 cycles

EDLC
dominant charge-storage mechanism

5 Conclusions

1. **Quantitative pipeline**
Custom image-analysis framework reveals structural descriptors beyond qualitative SEM screening.
2. **Optimised architecture**
41.42 % surface porosity → unhindered electrolyte access and rapid ion diffusion.
3. **Statistical robustness**
10,528 watershed segments across 16 independent components confirm representative metrics.
4. **EDLC behaviour**
Areal capacitance vs. scan rate confirms predominant electric double-layer mechanism.
5. **Low ESR**
0.33 Ω sq^{-1} ESR reflects excellent conductivity & minimal ohmic resistance.
6. **Outstanding cycling**
Negligible capacitance fade over 10,000 cycles — strong candidate for sustainable supercapacitors.

6 References

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