SUPPORTING INFORMATION

Electrochemical Synthesis of Polyaniline-Ni-Co Hybrid Structures on Titanium Substrates for Ultrahigh-Capacitance Supercapacitor Electrodes

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S1. Equivalent thickness estimation from gravimetric mass loading

The coating thickness was estimated as an *equivalent average thickness* using the gravimetric areal mass loading (Γ) and an assumed film density (ρ), according to:

$$t_{eq} = \frac{\Gamma}{\rho} \quad (1)$$

where t_{eq} is the equivalent thickness (cm), Γ is the areal mass loading g cm^{-2}, and ρ is the density mg cm^{-3}. Using the experimentally determined values $\Gamma_{Ni–Co}$=0.102 mg cm^{-2} and Γ_{PANI}=0.128 mg cm^{-2}, the corresponding equivalent thicknesses were calculated as:

$$t_{eq,Ni-Co} = \frac{0.102 \times 10^{-3}}{\rho_{Ni-Co}}$$

$$t_{eq,PANI} = \frac{0.128 \times 10^{-3}}{\rho_{PANI}}$$

The Ni-Co alloy density was estimated as $\rho_{Ni\text{-}Co}$ ~8.9 g cm^{-3} [1] and the PANI density was estimated as ρ_{PANI} ~ 1.33 gcm^{-3} [2]. Based on these values, the thickness of the layers was 0.12 μm and 0.96 μm, respectively. However, these values represent equivalent thicknesses; the true physical thickness can differ due to film porosity/roughness.

Table S1: XRD peak integrated areas for the PANI–Ni–Co/Ti sheet, including the PANI amorphous halo reflection (Ti substrate and Ni–Co underlayer peaks identified for comparison from PANI crystallinity analysis).

2θ	FWHM	Integrated Area	Peak Type	Peak Assignment
25.79	1.69328	892.9937	Broad	PANI Layer
35.39	0.52486	1204.06152	Sharp	Ti Substrate
38.76	0.42208	3684.73521	Sharp	Ti Substrate
40.48	0.42813	5238.63495	Sharp	Ti Substrate
43.18	0.2121	605.30439	Sharp	Ni-Co Layer
53.27	0.49147	1230.95516	Sharp	Ti Substrate
63.25	0.62423	556.26526	Sharp	Ti Substrate
70.91	0.55859	1877.0921	Sharp	Ti Substrate
76.49	0.58853	837.4645	Sharp	Ti Substrate
77.64	0.82764	434.21259	Sharp	Ti Substrate

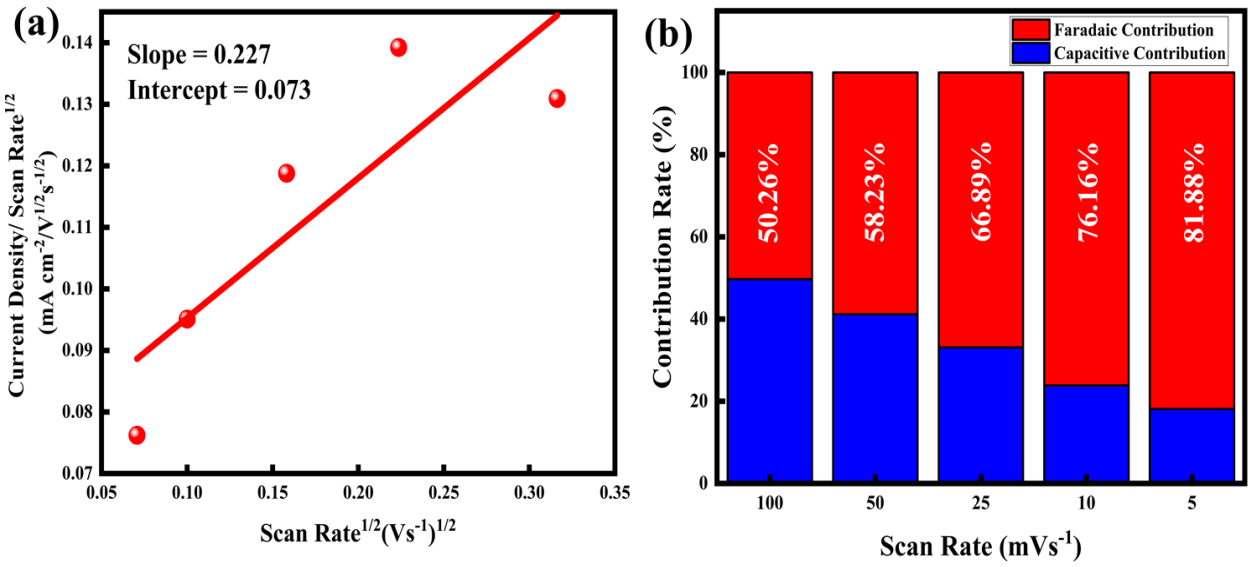

Figure S1: Quantification of capacitive and faradaic (diffusion-controlled) charge-storage contributions of the PANI-Ni-Co/Ti electrode; (a) linear fit of $i/\upsilon^{1/2}$ versus $\upsilon^{1/2}$(Dunn method) at a fixed potential (0.6V); (b) comparison of capacitive and faradaic contribution percentages at scan rates of 5–100 mV s^{-1}.

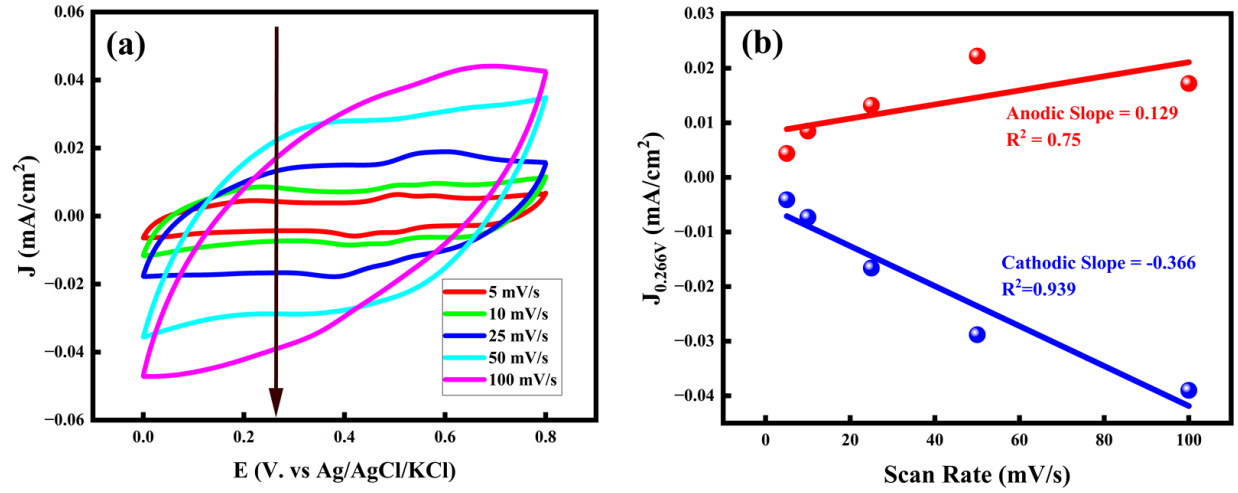

Figure S2: (a) CVs of the PANI–Ni–Co/Ti electrode recorded in a non-faradaic potential region at different scan rates; (b) Corresponding double layer capacitance (C_{dl}) derived from CV curves.

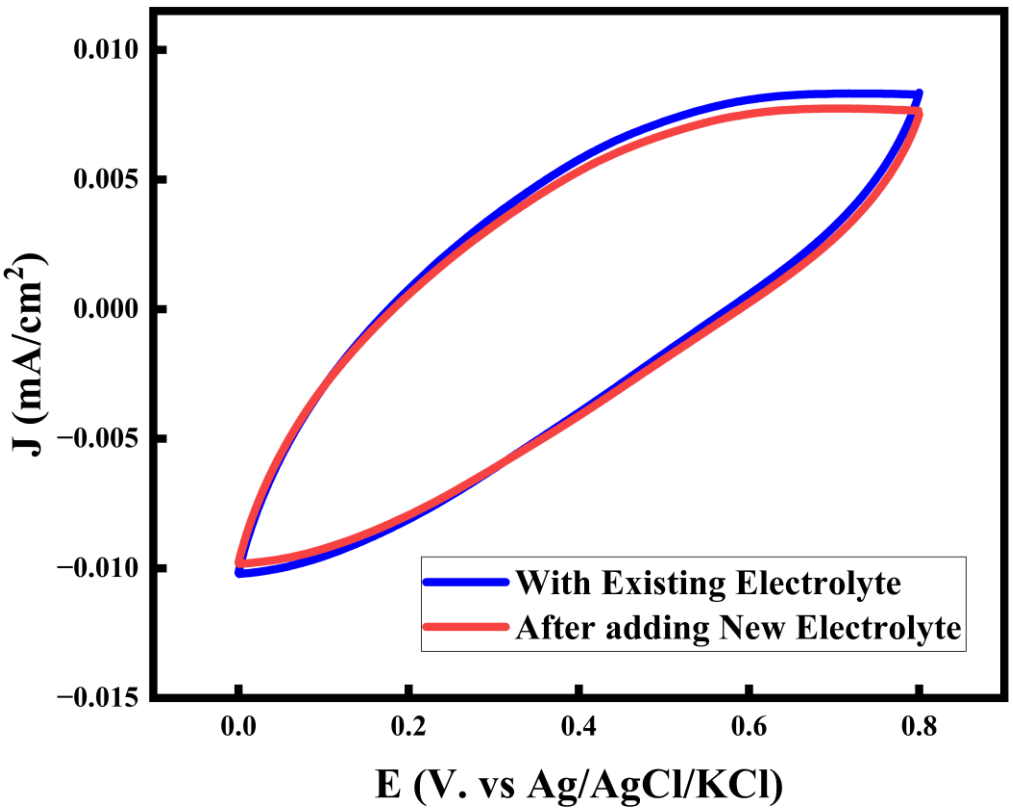

Figure S3: Post-cycling CV curves of the PANI-Ni-Co/Ti electrode recorded at 50 mV s^{-1} in 1.0 M H_2SO_4 (0.0–0.8 V vs Ag/AgCl) using the existing electrolyte and after replacement with fresh electrolyte, showing largely preserved CV profiles after the 1000-cycle stability test.

References

1 P. Barbu, M. M. Codescu, M. Iordoc, V. Marinescu, E. Manta, C. Ilie and M. Popa, *Rev. Chim.*, 2018, **69**, 1355–1362.

2 J.-W. Jeon, S. R. Kwon and J. L. Lutkenhaus, *J. Mater. Chem. A*, 2015, **3**, 3757–3767.