

Supporting Information

**Oxygen Defect-mediated NiCo₂O₄ Nanosheets as Electrode
for Pseudocapacitor with Improved Rate Capability**

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Experimental Section

Synthesis of $r\text{-NiCo}_2\text{O}_4$ nanosheets: In a typical synthesis, 1.1 mmol $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, 2.2 mmol $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and 13.2 mmol hexamethylenetetramine (HMT) are dissolved into 80 mL mixed solution of ethylene glycol/water (v/v=7/1) to form the clear pink solution after violent stirring for 30 min. Then the clear pink solution is transferred into a 100 mL Teflon lined stainless autoclave. The autoclave is sealed and maintained at 120°C for 4 hours and then cooled down to room temperature. The precipitates are collected by centrifugation and rinsed for three times with abundant amount of deionized water and ethanol, respectively. Then, the green solid product is dried at -40°C in freezing dryer for 36 h. The dried samples are further calcined at 300°C for 2 hours in the air to convert the hydroxide into the NiCo_2O_4 . For oxygen defect-mediated NiCo_2O_4 , 0.1 g original NiCo_2O_4 NSs are immersed in 100 mL 1.0 M NaBH_4 solution for 1 h at room temperature. After reduction, the samples are filtered, thoroughly washed with deionized water and ethanol for several times and dried in a vacuum oven at 60°C for 12 h. The synthesis process of the irregular NiCo_2O_4 NPs is similar to the aforementioned method, while the solvent is all deionized water and the reaction time is 12 hours.

Characterization: The samples are characterized by field emission scanning electron microscopy (FESEM, Hitachi, S-4800), transmission electron microscopy (TEM, JEOL, JEM-1101), high-resolution transmission electron microscopy (HRTEM, JEOL, JEM-2100) and X-ray powder diffractometry

(XRD, Bruker D8 Advance X-ray diffractometer system with Co K α radiation of 1.7902 Å.). The specific surface areas of the products are evaluated by the ASAP 2020 instrument. An X-ray photoelectron spectroscopy (XPS) survey is conducted using a PHI 5000 VersaProbe with an Al K α excitation source. The Raman spectra are obtained using a Renishaw in Via Reflex Raman system with the excitation laser of 532 nm. The electrical conductivity is measured by a four-wire method using a source measure unit (SMU, Keithley 6430). Thermal gravimetry analysis (TGA) is recorded on Netzsch STA 449C under air and nitrogen atmosphere with the heating rate of 5 °C/min.

Electrochemical Measurements: The working electrode is prepared by mixing 80 wt.% as-made products, 10 wt.% acetylene black, and 10 wt.% polytetrafluoroethylene (PTFE) in small amount of ethanol to obtain the slurry mixture. The obtained slurry is coated on the single surface of nickel foams with a size of 1 cm \times 1 cm and dried in vacuum at 80°C for 12 h. Then, the nickel foams with the electroactive materials are pressed under 10 MPa with the thickness of 0.11 mm. The mass loading of active material is about 1.8 mg cm⁻². The electrochemical tests of all samples are conducted in a three-electrode system with a Pt counter-electrode and an Hg/HgO reference electrode in 6.0 M KOH solution. The electrochemical AC impedance measurements are carried out to assess the electrical properties at an open circuit voltage with frequency from 0.01 Hz to 100 kHz and an amplitude of 5 mV. All the electrochemical tests are carried out on the electrochemical working station (CHI660E, Shanghai, China).

The specific capacitance of the supercapacitor can be calculated by galvanostatic discharge-charge (GCD) test as following equation:

$$C_m = \frac{I}{m} \times \frac{\Delta t}{\Delta V}$$

Where C_m is the specific capacitance of the capacitor (F g^{-1}); I is the current of the charge/discharge process; Δt is the discharging time period in seconds for the potential change ΔV , in volts; m is the mass loading of the active material. All the electrochemical measurements are carried out at room temperature.

Supplementary Figures and Tables

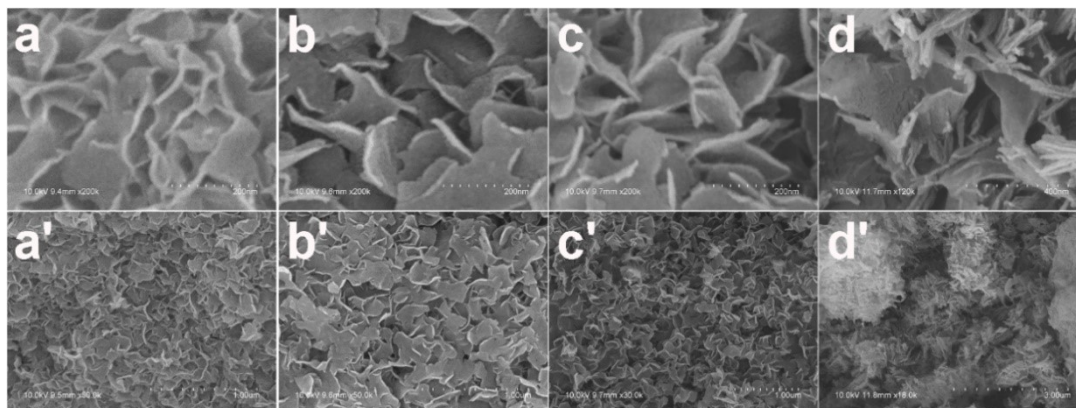


Figure S1. SEM images of nickel-cobalt hydroxides nanosheets obtained at various solvothermal reaction times: (a) and (a') for 1 h; (b) and (b') for 2 h; (c) and (c') for 4 h; (d) and (d') for 6 h.

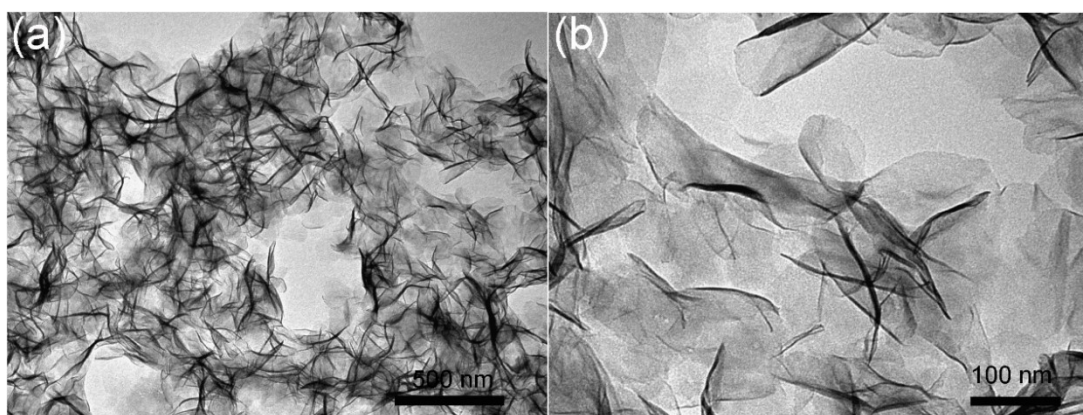


Figure S2. TEM images of optimized nickel-cobalt hydroxide nanosheets at 120 °C for 4 h.

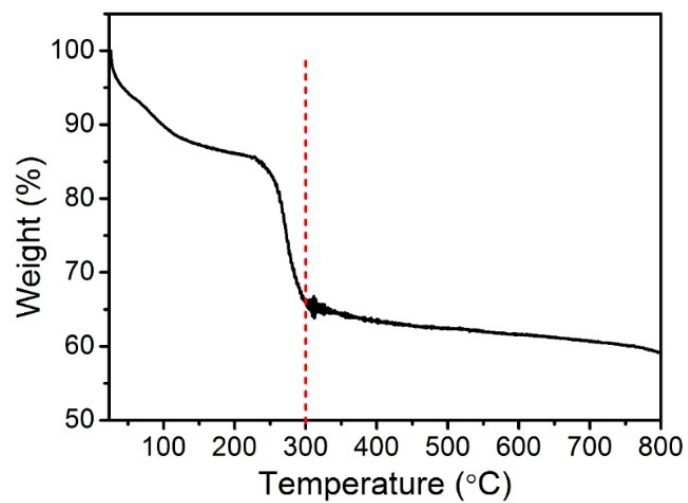


Figure S3. TGA curve of optimized nickel-cobalt hydroxide nanosheets at 120 °C for 4 h.

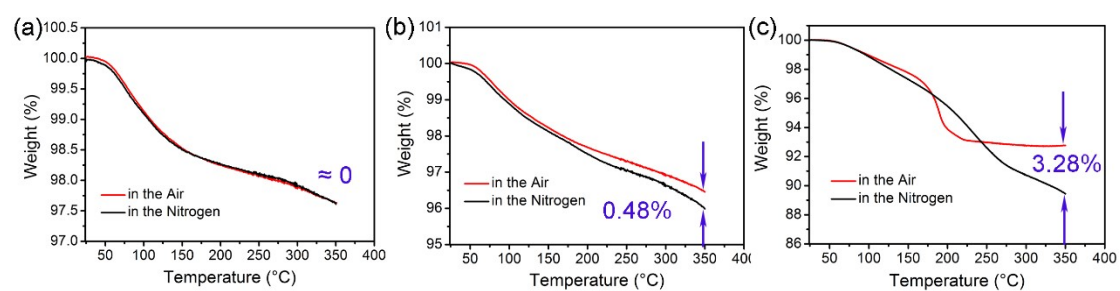


Figure S4. TGA analysis of (a) NiCo_2O_4 NPs, (b) original NiCo_2O_4 NSs and (c) $\text{r-NiCo}_2\text{O}_4$ NSs in air and argon atmosphere. (Flow of 20 mL min^{-1} and a ramping rate of 5°C/min). The concentrations of oxygen defects are calculated from the difference in weight decrease between the two TGA curves.

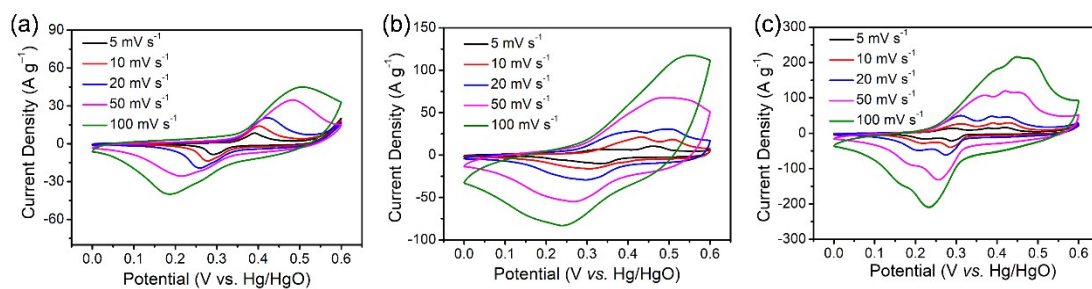


Figure S5. CV curves of NiCo_2O_4 NPs, original NiCo_2O_4 and r- NiCo_2O_4 NSs at various scan rates.

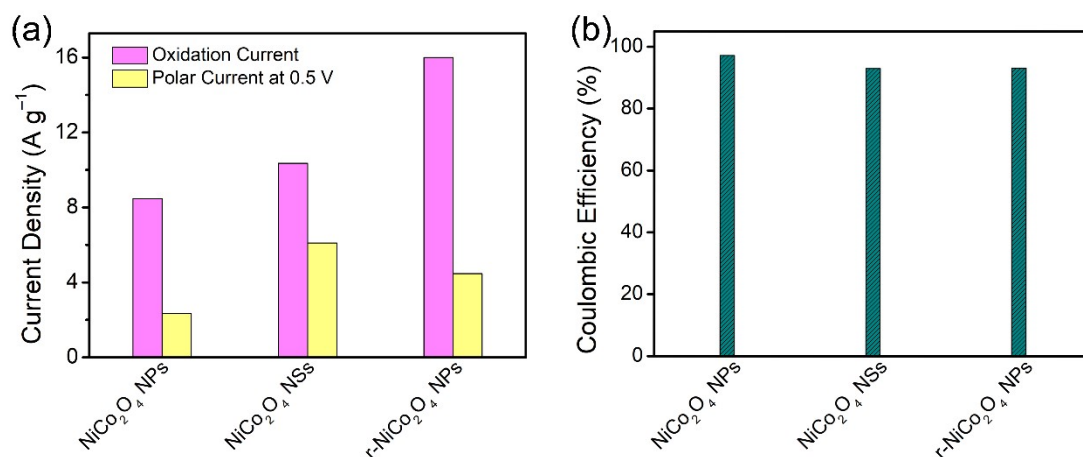


Figure S6. (a) The polarization currents at 0.5 V and the oxidation currents of NiCo_2O_4 NPs, NiCo_2O_4 NSs, and r- NiCo_2O_4 NSs electrodes. (b) Coulombic efficiencies of NiCo_2O_4 NPs, NiCo_2O_4 NSs, and r- NiCo_2O_4 NSs at the current density of 20 A g^{-1} .

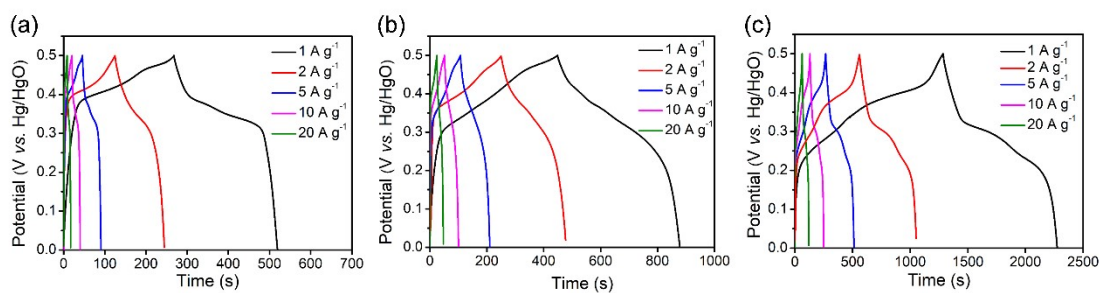


Figure S7. (a-c) Galvanostatic charge and discharge voltage profiles of NiCo₂O₄ NPs, original NiCo₂O₄ and r-NiCo₂O₄ NSs at various current densities.

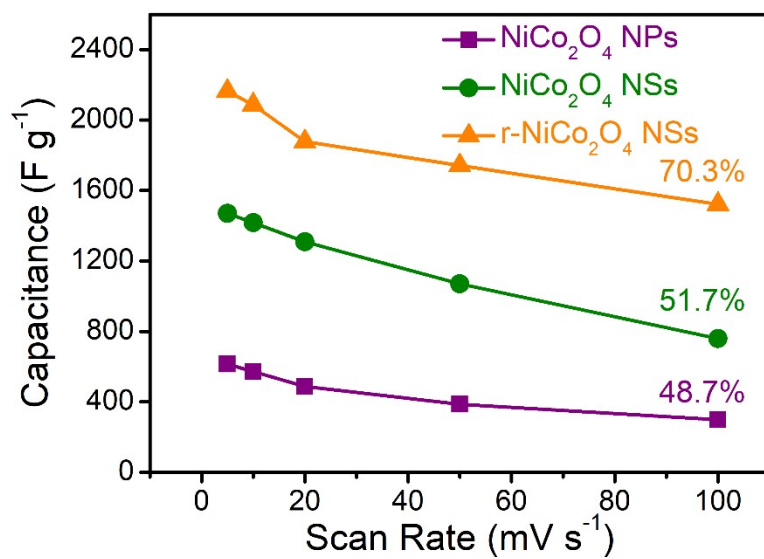


Figure S8. Plots of specific capacitance as a function of scan rate of NiCo₂O₄ NPs, original NiCo₂O₄ NSs, and r-NiCo₂O₄ NSs.

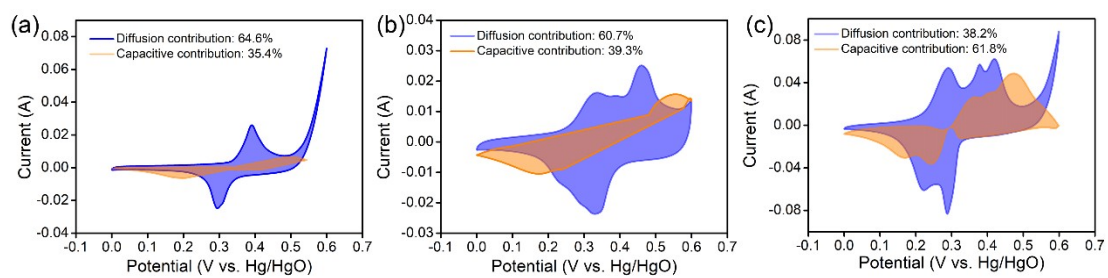


Figure S9. Diffusion and capacitive-controlled current contributions of NiCo_2O_4 NPs, original NiCo_2O_4 NSs, and r- NiCo_2O_4 NSs calculated from CV curves at the scan rate of 5 mV s^{-1} .

Table S1. Comparison of the electrochemical performances of r-NiCo₂O₄ electrode in this work with NiCo₂O₄ and carbon-based electrodes in the literature

NiCo ₂ O ₄ -based electrode	Specific capacitance		Rate capability	Ref.
	C _{min}	C _{max}		
Mesoporous NiCo ₂ O ₄	956 F g ⁻¹ @ 1 A g ⁻¹	828 F g ⁻¹ @ 10 A g ⁻¹	86.6% (1-10 A g ⁻¹)	1
rGO wrapped porous NiCo ₂ O ₄	1176 F g ⁻¹ @ 2 A g ⁻¹	1020 F g ⁻¹ @ 8 A g ⁻¹	86.7% (2-8 A g ⁻¹)	2
NiCo ₂ O ₄ -decorated porous carbon	596.8 F g ⁻¹ @ 2 A g ⁻¹	185 F g ⁻¹ @ 20 A g ⁻¹	30.9% (2-20 A g ⁻¹)	3
NiCo ₂ O ₄ tetragonal microtubes	1388 F g ⁻¹ @ 2 A g ⁻¹	863 F g ⁻¹ @ 30 A g ⁻¹	62.2% (2-30 A g ⁻¹)	4
nickel cobalt oxide/graphene oxide	1211 F g ⁻¹ @ 1 A g ⁻¹	687 F g ⁻¹ @ 10 A g ⁻¹	56.7% (1-10 A g ⁻¹)	5
NiCo ₂ O ₄ hollow spheres	1204 F g ⁻¹ @ 2 A g ⁻¹	822 F g ⁻¹ @ 20 A g ⁻¹	68.3% (1-20 A g ⁻¹)	6
NiCo ₂ O ₄ microspheres	1822.3 F g ⁻¹ @ 2 A g ⁻¹	1250.9 F g ⁻¹ @ 20 A g ⁻¹	68.6% (1-20 A g ⁻¹)	7
Ni/Co porous oxide nanosheets	830 F g ⁻¹ @ 1 A g ⁻¹	710 F g ⁻¹ @ 20 A g ⁻¹	85.5% (1-20 A g ⁻¹)	8
Mesoporous NiCo ₂ O ₄	655 F g ⁻¹ @ 1 A g ⁻¹	430 F g ⁻¹ @ 10 A g ⁻¹	65.6% (1-10 A g ⁻¹)	9
hollow NiCo ₂ O ₄ nanospheres	1229 F g ⁻¹ @ 1 A g ⁻¹	1027 F g ⁻¹ @ 25 A g ⁻¹	83.6% (1-25 A g ⁻¹)	10
Mesoporous NiCo ₂ O ₄ /NF	708 F g ⁻¹ @ 1 A g ⁻¹	628 F g ⁻¹ @ 10 A g ⁻¹	88.7% (1-10 A g ⁻¹)	11
N-doped graphene framework (NCO/NGF)	1198 F g ⁻¹ @ 1 A g ⁻¹	634 F g ⁻¹ @ 20 A g ⁻¹	52.9% (1-20 A g ⁻¹)	12
rGO/NiCo ₂ O ₄	1003 F g ⁻¹ @ 1 A g ⁻¹	893 F g ⁻¹ @ 10 A g ⁻¹	89.0% (1-10 A g ⁻¹)	13
NiCo ₂ O ₄ /CuO-x	1670 F g ⁻¹ @ 1 A g ⁻¹	692 F g ⁻¹ @ 10 A g ⁻¹	41.4% (1-10 A g ⁻¹)	14
rHGO/ NiCo ₂ O ₄ @CF	1178 F g ⁻¹ @ 1 A g ⁻¹	1100.4 F g ⁻¹ @ 10 A g ⁻¹	93.4% (1-10 A g ⁻¹)	15

Co–Ni–O/3DG	1586 F g ⁻¹ @ 1 A g ⁻¹	~ 1100 F g ⁻¹ @ 10 A g ⁻¹	69.4% (1-10 A g ⁻¹)	16
NiCo ₂ O ₄ @GQDs	1382 F g ⁻¹ @ 1 A g ⁻¹	1278 F g ⁻¹ @ 20 A g ⁻¹	92.5% (1-20 A g ⁻¹)	17
hexagonal NiCo ₂ O ₄	1525 F g ⁻¹ @ 1 A g ⁻¹	610 F g ⁻¹ @ 10 A g ⁻¹	40.0% (1-10 A g ⁻¹)	18
NiCo ₂ O ₄ Nanoparticle	1084 F g ⁻¹ @ 2 A g ⁻¹	300 F g ⁻¹ @ 10 A g ⁻¹	27.7% (2-10 A g ⁻¹)	19
P- NiCo ₂ O ₄	1642 F g ⁻¹ @ 2 A g ⁻¹	1335 F g ⁻¹ @ 20 A g ⁻¹	81.3% (2-20 A g ⁻¹)	20
NiCo ₂ O ₄ @AMCRs	1691 F g ⁻¹ @ 3 A g ⁻¹	1275 F g ⁻¹ @ 20 A g ⁻¹	75.4% (3-20 A g ⁻¹)	21
NiCo ₂ O ₄ /PANI/MF	1540.1 F g ⁻¹ @ 1 A g ⁻¹	1220 F g ⁻¹ @ 20 A g ⁻¹	79.2% (1-20 A g ⁻¹)	22
Mesoporous NiCo ₂ O ₄ flower	122.5 C g ⁻¹ @ 1 A g ⁻¹	82.5 C g ⁻¹ @ 10 A g ⁻¹	67.3% (1-10 A g ⁻¹)	23
NiCo ₂ O ₄ /carbon	1480.9 F g ⁻¹ @ 1 A g ⁻¹	995.2 F g ⁻¹ @ 20 A g ⁻¹	67.2% (1-20 A g ⁻¹)	24
NiCo ₂ O ₄ /Carbonized melamine foam	1541 F g ⁻¹ @ 1 A g ⁻¹	1120 F g ⁻¹ @ 20 A g ⁻¹	72.7% (1-20 A g ⁻¹)	25
Hierarchically porous carbon	218.6 F g ⁻¹ @ 1 A g ⁻¹	164.5 F g ⁻¹ @ 20 A g ⁻¹	75.3% (1-20 A g ⁻¹)	26
O-N-S co-doped hierarchical porous carbon	576 F g ⁻¹ @ 1 A g ⁻¹	267 F g ⁻¹ @ 20 A g ⁻¹	43.4% (1-20 A g ⁻¹)	27
Porous carbon sheets	407 F g ⁻¹ @ 1 A g ⁻¹	224 F g ⁻¹ @ 20 A g ⁻¹	55.0% (1-20 A g ⁻¹)	28
Carbon spheres@ porous carbon	337 F g ⁻¹ @ 1 A g ⁻¹	280 F g ⁻¹ @ 20 A g ⁻¹	83.1% (1-20 A g ⁻¹)	29
N-doped porous graphene	390 F g ⁻¹ @ 1 A g ⁻¹	238 F g ⁻¹ @ 20 A g ⁻¹	61.0% (1-20 A g ⁻¹)	30
Ultrathin NiCo ₂ O ₄ Nanosheets/NF	2010 F g ⁻¹ @ 2 A g ⁻¹	1450 F g ⁻¹ @ 20 A g ⁻¹	72.1% (2-20 A g ⁻¹)	31
Ultrathin NiCo ₂ O ₄ Nanosheets/CF	1002 F g ⁻¹ @ 1 A g ⁻¹	520 F g ⁻¹ @ 1 A g ⁻¹	48.8% (1-20 A g ⁻¹)	32
Porous NiCo ₂ O ₄ Nanosheet/Carbon Fabric	2658 F g ⁻¹ @ 2 A g ⁻¹	1866 F g ⁻¹ @ 20 A g ⁻¹	70.2% (2-20 A g ⁻¹)	33
Pd-NiCo ₂ O ₄ /NF	2484.4 F g ⁻¹ @ 2 A g ⁻¹	2011 F g ⁻¹ @ 2 A g ⁻¹	80.9% (2-20 A g ⁻¹)	34
Mesoporous NiCo ₂ O ₄ Nanosheets	560 F g ⁻¹ @ 2 A g ⁻¹	400 F g ⁻¹ @ 2 A g ⁻¹	71.4% (2-20 A g ⁻¹)	35
r-NiCo₂O₄ nanosheets	1980 F g⁻¹ @ 1 A g⁻¹	1897 F g⁻¹ @ 10 A g⁻¹	95.8% (1-10 A g⁻¹)	This work
	1968 F g⁻¹ @ 2 A g⁻¹	1812 F g⁻¹ @ 20 A g⁻¹	91.5% (1-20 A g⁻¹)	

			92/1% (2-20 A g ⁻¹)	
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