Supporting Information

Amorphous Carbon Nitride/NiO/CoN Based Composite: A Highly Efficient Nonprecious Electrode for Supercapacitor and Oxygen Evolution Reaction

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Experimental section

Pre-treatment of Ni-foam: First, several nickel foams (1 cm \times 2 cm) were put in 0.5 M H₂SO₄ solutions for 5 min to eliminate the surface oxides. Then, they were transferred into 20 mL acetone for 10 min to experience a process of degreasing. Finally, after 30 min ultrasonic cleaning and washing by deionized water, the nickel foams was taken to an oven with 120 °C for 1 h.

Preparation of Ni-Co-CN electrodes: The Ni-Co-CN composite is synthesized by one-step pyrolysis in muffle furnace. In a typically procedure, 2 g urea and 0.5 g cobaltous nitrate hexahydrate (Co(NO₃)₂·6H₂O) were added into 10 mL deionized water under stirring to form a homogeneous and transparent solution. Then, the dried nickel foam is soaked twice in above solution and dried with hair dryer to achieve a uniform load of active substances. Ultimately, the samples were transferred into a boat shape crucible (10 cm \times 3 cm \times 2 cm), and subsequently heated to 350 °C at a ramping rate of 3 °C min⁻¹ and maintained 2 h under air conditions. For the sake of comparison, the same method was applied to fabricate the Ni-Co and Ni-Urea electrodes, accordingly the precursor solutions are changed to 0.5 g cobaltous nitrate hexahydrate $(Co(NO_3)_2 \cdot 6H_2O)$ and 2 g urea aqueous solutions, respectively. Moreover, the effect of calcination temperature on Ni-Co-CN was investigated. To this end, five different temperatures including 200 °C, 300 °C, 350 °C, 400 °C, 500 °C were explored, where the as-fabricated electrodes were denoted as Ni-Co-CN-X, and X is the actual temperature.

Assembly of Asymmetric Supercapacitors: 6 g polyvinyl alcohol (PVA) and 3 g KOH were added into 60 mL deionized water to prepare gel electrolytes for ASC. First, the above solution is heated to 100 °C for 30 min to obtain a homogeneous solution, then the positive electrode materials (Ni-Co-CN, 1 cm²) and negative electrode materials (porous carbon coated nickel foam, 1 cm²) were immersed into the PVA/KOH electrolytes for 1 min. After repeated soaking for three times, the electrodes were transferred to a petri dish and stand for 12 h before being assembled into a button battery. And the specific assembly procedures of button batteries could be found in our previous work.[1]

Characterization methods

A Rigaku Smartlab X-ray diffraction (XRD) equipment was applied to collect the diffraction data of the obtained samples using Cu K α ($\lambda = 0.1544$ nm) radiation. The Fourier transform infrared (FT-IR) spectra were recorded with a Vertex 70 spectrophotometer (Bruker Optik GmbH, Germany). Moreover, a Renishaw Invia Raman spectrometer with a 20 mW Ar⁺ laser source of 532 nm was used to collect the Raman spectra. The surface area and pore size distribution were characterized by the N₂ adsorption isotherms at 77 K with a Micrometrics ASAP 2460 instrument, and the multipoint Brunauer-Emmett-Teller (BET) method was performed to calculate the SSA (specific surface area). The morphologies and structures of samples were investigated by a field emission scanning electron microscope (FESEM) (Sigma 500 Zeiss Germany). In addition, a transmission electron microscopy (TEM, Tecnai F20, FEI, USA) coupled with an energy-dispersive X-ray spectroscopy (EDS) were conducted to detect the microstructure of active materials. The X-ray photoelectron spectra (XPS) were also measured on a Thermo Scientific ECSALab 250Xi X-ray photoelectron spectrometer with an Al Ka X-ray radiation (1486 eV) to investigate the chemical states and surface composition. The contents of transition metal atoms in several samples were measured via Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES PQ9000, Analytik Jena)

Electrochemical measurements

Evaluation of Supercapacitive Performances: All electrochemical tests were based on a CHI 660E electrochemical workstation (CH Instrumental Co, Ltd, Shanghai), and the electrochemical property of individual electrode is studied by a three-electrode electrochemical set-up (Pt foil as the counter electrode, saturated calomel electrode (SCE) as the reference electrode and the self-supported Ni-Co-CN as the working electrode, respectively). In this system, an aqueous solution of 3 M KOH was used as electrolyte, and the stability of electrode was tested under the scan rate of 50 mV s⁻¹. Besides, the range of frequency in the electrochemical impedance spectroscopy (EIS) measurement is from 0.01 HZ to 100 KHz. Meanwhile, the galvanostatic charge-discharge (GCD) tests were carried out from -0.1 V to 0.4 V and the areal specific capacitance (C_s) were calculated by the equation as follows:

$$C_s = It / A\Delta V \tag{1}$$

Where C_s is the areal specific capacitance (mF cm⁻²), I is the discharge current (A), t is the discharge time (s), A represents the area of self-supported electrodes (cm²) and ΔV is the potential window (V).

Besides, the energy density and power density of Ni-Co-CN//porous carbon ASC are calculated by the two equations as follows:

$$E = \frac{1}{2}C_s V^2 \tag{2}$$

$$P = E/t \tag{3}$$

Where *E* represents the energy density (mWh cm⁻²), C_s is the areal specific capacitance (F cm⁻²), *V* means the operating voltage (V), *P* is the power energy (mW cm⁻²), *t* refers to the discharge time (s).

Also, to achieve $q_{+} = q_{-}$, the mass ratio of positive electrode active material to negative electrode active material is determetered from the following equation:

$$m_{+}:m_{-} = \left(\int \frac{idV}{v}\right)_{-} : \left(\int \frac{idV}{v}\right)_{+}$$
(4)

The mass ratio of the positive electrode active material to the negative electrode active materials was determined from the cyclic voltammograms acquired at 10mV/s (**Fig S8a**) for both electrodes.

Evaluation of Electrocatalytic Activity for OER: The catalytic performances of self-supported electrodes were measured in 1 M KOH electrolytes with a standard three-electrode system. During the testing process, the as-fabricated self-supported electrodes used as working electrode directly, a platinum foil as counter electrode and a SCE electrode as the reference electrode, respectively. After successive cyclic voltammetry (CV) scanning for activating, the linear sweep voltammetry (LSV) measurements were performed at a scan rate of 2 mV s⁻¹ without any iR-correction and the overpotential (η) was calculated by the following equation:

$$\eta = E_{vs.} RHE - 1.23 V \tag{5}$$

Where $E_{vs.}RHE$ means all the potentials are recorded relative to the reversible hydrogen electrode (*RHE*), just as shown in the Nernst equation:

$$E_{vs.} RHE = E_{vs.} Hg/Hg_2 Cl_2 + 1.068 + 0.059 pH$$
(6)

In addition, Tafel slope (b, mV decade⁻¹) is calculated based on the equation as follows:

$$\eta = b \log j \tag{7}$$

Where *j* means the current density (mA cm⁻²).

Calculation of C_{dl}

Based on the linear fitting of **Fig S12**, take Ni-Co-CN as an example, we can derive its specific capacitance as follows:

$$C_{dl} = \frac{k}{2} = \frac{33.86}{2} = 16.93 \, mF/cm^2$$
 (8)

where C_{dl} is the specific capacitance of Ni-Co-CN, k is the fitting slope. Asymmetric supercapacitors (ASC)

A solid-state ASC was fabricated to explore the practical application of electrode materials. In the assemble procedure, the as-prepared Ni-Co-CN, porous carbon from our previous work and PVA/KOH gel were chosen as positive electrode, ¹ negative electrode and electrolyte, respectively. As shown in Fig. S8a, the potential windows of Ni-Co-CN//porous carbon ASC were determined through the test of voltage range at a scan rate of 10 mV s⁻¹. As depicted in Fig. S8b and S8c, the CV and GCD tests were conducted at different potential windows from 0.8 V to 1.6 V. No obvious polarization could be observed when the potential window was less than 1.6 V (Fig. **S8b**), suggesting that the operating voltage of ASC could be broadened up to 1.6 V. As displayed in Fig. S8d, a series of redox peaks appeared at the CV plots in different scan rates, indicating that both pseudocapacitance and double-layer capacitance take effect in the process of electrode reaction. And the CV curves of ASC device nearly remain its initial shape, suggesting a good reversibility of electrode materials. Additionally, based on Fig. S8e and Equation 1, the areal specific capacitances of Ni-Co-CN//porous carbon can be calculated as 905, 766, 502, 375 and 270 mF cm⁻² at the current densities of 0.5, 1.0, 2.0, 4.0 and 8.0 mA cm⁻², respectively.

Energy density and power density are two important parameters in evaluating supercapacitive performance and both high values of them means an excellent energy storage material. Herein, the as-obtained Ni-Co-CN//porous carbon ASC device exhibits a superior energy density of 0.29 mWh cm^{-2} when its power energy up to 2 mW cm⁻². And the energy density of ASC devices can still be achieved to 0.19 mWh cm⁻² as the power density up to a high value of 6.4 mW cm⁻², outperforming most of Ni-based asymmetric supercapacitors. The specific values are shown in the Ragone plots (Fig. S9c) and performance comparisons table (Table S6). Furthermore, the values of R_s (~3.06 Ω) and R_{ct} (~2.65 Ω) derived from the EIS measurement in Fig. S9a are used for evaluating the charge transfer process occurred on electrode materials, which demonstrates a good electrochemistry property for Ni-Co-CN//porous carbon ASC. In addition, cycling stability of ASC was also estimated through GCD method at a current density of 1 mA cm⁻². Although there are abnormal fluctuations during testing, the capacitance still remains ca.79.2% of initial values after 5000 cycles (Fig. S9b). Besides, the pattern of "UCAS" that comprises of several LEDs was successfully lighted up by using two ASC devices in series (Fig. S9d). Overall, the Ni-Co-CN//porous carbon ASC devices assembled enable the practical application and display a preferable performance.

Computational datails

First-principles calculations were performed by using the Vienna Ab initio Simulation Package (VASP) ²⁻⁵ to investigate the oxygen evolution reaction (OER) process. The valence-core electrons interactions were treated by Projector Augmented

Wave (PAW) ⁶ potentials and the electron exchange correlation interactions were described by the generalized gradient approximation (GGA) with the Perdew-Burke-Ernzerhof (PBE) functional.⁷ Considered long-range interaction between molecules/intermediates and surface, Van der Waals interactions were considered using DFT-D3 correlation.⁸ Models were built according to growth subsequence and to avoid effects come from another slab, a vacuum of 20 Å was added along z direction. The convergence criterion of geometry relaxation was set to 0.01 eV·Å⁻¹ in force on each atom. The energy cutoff for plane wave-basis was set to 450 eV. The K points were sampled with 3×3×1 by Monkhorst-Pack method.⁹ Free energies of each reaction steps were calculated as $G = E_{DFT} + E_{ZPE} - T\Delta S$, where E_{DFT} is the DFT calculated energy, E_{ZPE} and $T\Delta S$ are calculated by DFT vibration frequency calculations and presented in Table S9. In order to consider the effect of an applied electric potential on the electrode reaction, a value of -neU was added to calculate the free energy of each step, where n is the number of electrons involved in the reaction,

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Fig S1. The photos of obtained Ni-Co-CN, Ni-Co, Ni-Urea and Ni-foam selfsupported electrodes.



Fig S2. (a) The CV, (b) GCD, (c) EIS tests for supercapacitors and (d) LSV plots for OER of Ni-Co-CN-X (X=200°C, 300°C, 350°C, 400°C, 500°C).



Fig S3. XRD patterns of $g-C_3N_4$, Ni-foam, Ni-Co, Ni-Urea and Ni-Co-CN.



Fig S4. Raman spectra of Ni-Urea and Ni-Co-CN electrodes between ~900 cm⁻¹ and ~2200 cm⁻¹.



Fig S5. (**a**, **b**) TEM images of Ni-Urea with different magnifications and locations; (**c**) HRTEM images and (**d**) the corresponding SAED pattern of the Ni-Urea; (**e**) HAADF-STEM image; (**f**) an expansion image in the region of orange rectangle in (e); (**g**) EDS elemental mapping images reveal the homogenous distribution of C, N, O and Ni element of the Ni-Urea composite.



Fig S6. (**a**, **b**) TEM images of Ni-Co with different magnifications and locations; (**c**) HRTEM images and (**d**) the corresponding SAED pattern of the Ni-Co; (**e**) HAADF-STEM image of Ni-Co electrode; (**f**) an expansion image in the region of orange rectangle in (e); (**g**) EDS elemental mapping revealing the homogenous distribution of N, O, Co and Ni element of the Ni-Co composite.



Fig S7. Comparison of CV curves at different cycle numbers for Ni-Co-CN electrode.



Fig S8. (a) CV curves of Ni-Co-CN and porous carbon at a scan rate of 10 mV s⁻¹ in a three-electrode system; (b) CV plots of Ni-Co-CN//porous carbon ASC at different potential windows in a scan rate of 100 mV s⁻¹; (c) GCD curves of Ni-Co-CN//porous carbon ASC at different potential windows from 0.8 to 1.6 V in a current density of 2 mA cm⁻²; (d) CV plots of Ni-Co-CN//porous carbon ASC at various scan rates from 5 to 100 mV s⁻¹; (e) GCD plots of Ni-Co-CN//porous carbon ASC at various current density from 0.5 to 8 mA cm⁻² (f) a function of specific capacitance of Ni-Co-CN//porous carbon ASC to current density.



Fig S9. (a) Nyquist plots of Ni-Co-CN//porous carbon ASC; (b) Capacitance retention of Ni-Co-CN//porous carbon ASC as a function of cycle number (and the inset shows the first five charge-discharge plots). (c) Ragone plots comparison of Ni-Co-CN//porous carbon ASC and other Ni/Co-based devices. (d) Digital photo of yellow-LED powered by the Ni-Co-CN//porous carbon ASC (inset indicates the appearance of assembled ASC).



Fig S10. LSV curves of Ni-foam, Ni-Co, Ni-Urea and Ni-Co-CN samples after iR-correction.



Fig S11. Current-time plot of Ni-Co-CN electrode at 1.65 V vs.RHE.



Fig S12. The CV tests of Ni-foam (a), Ni-urea (b) and Ni-Co-CN (c) electrodes at different scan rates from $10mV s^{-1}$ to $100mV s^{-1}$; (d) Current density at 0.15V of several electrodes as a function of scan rate.



Fig S13. Calculated free energy diagram for the OER on Ni(111), Ni(200) surfaces at zero potential (U=0 V, blank line), equilibrium potential (U=1.23 V, blue line). Ideal reaction process is illustrated by gray dotted line.



Fig S14. Calculated free energy diagram for the OER on Ni-Urea surface at zero potential (U=0 V, blank line), equilibrium potential (U=1.23 V, red line). Ideal reaction process is illustrated by gray dotted line.



Fig S15. Calculated free energy diagram for the OER on Ni-Co surface at zero potential (U=0 V, blank line), equilibrium potential (U=1.23 V, red line). Ideal reaction process is illustrated by gray dotted line.



Fig S16. Density of states profile (DOS) of partial (left) and ensemble (right) for Ni-Urea species.



Fig S17. Density of states profile (DOS) of partial (left) and ensemble (right) for Ni-Co species.

Table S1. The specific performance results of Ni-Co-CN-X (X=200℃, 300℃, 350℃,

Т/°С	200	300	350	400	500
C _S (F cm ⁻²)	2.99	3.75	9.96	1.04	0.14
Overpotential (mV)	251	273	195	456	485

 400° C, 500° C) derived from Fig S2.

Table S2. The contents of each element in Ni-Co-CN species measured by XPS.

Name	Atomic %
C 1s	36.47
N 1s	24.65
O 1s	30.21
Co 2p	1.77
Ni 2p	6.91

Table S3. The contents of transition metal atoms in several samples measured via ICP-OES.

	Elemental content (at %)		
Samples	Со	Ni	
Ni-Co	46.6	23.3	
Ni-Urea	0.2	40.7	
Ni-Co-CN	10.2	65.7	

electrode	electrolyte	Areal specific capacitance/current density (F cm ²)/(mA cm ²)	Reference
NiCo ₂ O ₄ @NiCo ₂ O ₄	2 M KOH	2.2//5	10
NiCo ₂ S ₄ @Co(OH) ₂	2 M KOH	9.6//2	11
NiCo ₂ S ₄ @PPy/NF	3 М КОН	9.781//5	12
Ni(OH) ₂ /RGO/NF	1 М КОН	15.65//7	13
NiCo ₂ O ₄ nanowire@Ni ₃ S ₂	3 М КОН	3//5	14
NiCo2S4@PANI	6 M KOH	4.74//2	15
Co-MOF/NF	2 M KOH	13.6//2	16
Co ₃ O ₄ @NiCo ₂ O ₄	2 M KOH	9.17//2	17
CoS@NiCo ₂ S ₄	2 M KOH	7.62//5	18
CoNi ₂ S ₄	2 M KOH	5.71//20	19
g-C ₃ N ₄ /NiO/CoN	3 M KOH	18.8//2	This work

Table S4. Comparison of Ni/Co-based materials applied to supercapacitors.

Table S5. The values of R_s and R_{ct} for Ni-foam, Ni-Co, Ni-Urea and Ni-Co-CN electrodes.

	Ni-foam	Ni-Co	Ni-Urea	Ni-Co-CN
$R_{s}\left(\Omega ight)$	1.247	1.345	1.189	1.238
$R_{ct}(\Omega)$	36.97	26.44	3.944	2.132

ASC	C _s (mF cm ⁻²)	E (mWh cm ⁻²)	P (mW cm ⁻²)	Reference
Ni@Ni(OH) ₂ /ACC	1910	0.6	0.37	20
Ni ₂ CoS ₄ @NiCo ₂ O ₄ /CFP	1250	0.44	0.16	21
NiCo ₂ O ₄ NG @ CF	25	0.001	2	22
Ni(OH) ₂ Nanowire@OMC	21	0.01	7.3	23
NiO/Ni(OH) ₂ / PEDOT//AC	404	0.01	7.8	24
NiO@MnCo-LDH//Ni/AC	368	0.02	0.38	25
3D- NiMoO4/Ni@CW//rGO/C F	504	0.2	4.1	26
NiO//rGO	248	0.1	0.88	27
NiCo ₂ O ₄ @ Co _x Ni _{1-x} (OH) ₂	888	0.32	0.77	28
NiO//a-Fe ₂ O ₃	229	0.05	0.3	29
Ni-Co-CN// Porous carbon	770	0.3	2	This work

Table S6. Comparison of Ni/Co-based materials assembled to ASC in the field of supercapacitors.

Table S7. The overpotential values for Ni-foam, Ni-Co, Ni-Urea and Ni-Co-CN electrodes under different current densities.

Sample Overpotential Current density	Ni-foam	Ni-Co	Ni-Urea	Ni-Co-CN
10 mA/cm ²	457 mV	367 mV	253 mV	195 mV
40 mA/cm ²	519 mV	417 mV	493 mV	302 mV
80 mA/cm ²	680 mV	629 mV	678 mV	495 mV

catalyst	electrolyte	Overpotential/current density (mV)/(10mA cm ²)	Reference
NiCo ₂ N/NF	1M KOH	290	30
CoN nanowires	1M KOH	290	31
CoN/Ni ₃ N@N-doped carbon	1M KOH	247	32
Co _{5.47} N NP@N-PC	1M KOH	248	33
NiO@CoN PINWs	1M KOH	300	34
Ni-NiO/N-rGO	1M KOH	240	35
Ni _{0.82} Co _{0.18} O@C-NF	1M KOH	190	36
C06.25Fe18.75Ni75Ox	1M KOH	186	37
NiFe-LDH nanoplatelet array	1М КОН	224	38
NiSe ₂ NCs	1М КОН	250	39
g-C ₃ N ₄ /NiO/CoN	1М КОН	195	This work

Table S8. Comparison of Ni/Co-based materials developed for the catalysts of oxygen evolution reaction.

Table S9. Calculated zero-point energies and entropies of different adsorption species, where the * denotes the adsorption site. T was set as 300K.

Adsorption Species	E _{ZPE} (eV)	<i>T∆S</i> (eV)
*OH	0.35	0.08
*0	0.07	0.05
*ООН	0.44	0.12

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