Supporting Information

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Construction of Novel Bimetallic Oxyphosphide as Advanced Anode for Potassium Ion Hybrid Capacitor

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S1. Materials

All the reagents were purchased from Sinopharm Chemical Reagent Co., Ltd. (Shanghai). All the reagents were used as received.

S2. Experimental Section

S2.1 Preparation of BCN: Firstly, boric acid (6 g), urea (3 g), and PEG-2000 (0.3 g) were dissolved in 60 mL deionised water under stirring for 15 minutes. Then, the air plasma treated carbon cloth (1.5 cm × 3 cm) was immersed in the above solution. The solution including the carbon cloth in a beaker were heated at 85 °C for 12 h. Finally, the precursor was pyrolysed at 950 °C for 5 h under Ar atmosphere and the BCN sample was obtained. To better match the anode material and optimize the performance of the PIHC device, the additive amount of PEG-2000, boric acid and urea in the precursors was appropriately increased to enhance the mass loading.

S2.2 Theoretical calculation method: We have employed the first-principles to perform all Spin-polarization density functional theory (DFT) calculations within the generalized gradient approximation (GGA) using the Perdew-Burke-Ernzerhof (PBE) formulation. We have chosen the projected augmented wave (PAW) potentials to describe the ionic cores and take valence electrons into account using a plane wave basis set with a kinetic energy cutoff of 450 eV. Partial occupancies of the Kohn–Sham orbitals were allowed using the Gaussian smearing method and a width of 0.03 eV. The electronic energy was considered self-consistent when the energy change was smaller than 10^{-6} eV. A geometry optimization was considered convergent when the energy change was smaller than 0.03 eV Å^{-1}. The Brillouin zone integration is performed using
3×3×3 Monkhorst-Pack k-point sampling for a structure. Finally, the adsorption energies (Eb) were calculated as $E_b = E_{\text{ad/sub}} - E_{\text{ad}} - E_{\text{sub}}$, where $E_{\text{ad/sub}}$, $E_{\text{ad}}$, and $E_{\text{sub}}$ are the total energies of the optimized adsorbate/substrate system, the adsorbate in the structure, and the clean substrate, respectively. For Co and Ni atom, the U correction has been employed in our systems. Finally, the K ions migration barrier energies had been evaluated using the climbing nudged elastic band (CI-NEB) methods.

S2.3 Fabrication of the potassium ion half-Cell and Potassium Ion Hybrid Capacitors: The NCOP materials and BCN were directly used as self-supporting electrode. The potassium ion half-cell was assembled into coin-type test cell (CR2032) with potassium metal working as both the counter and reference electrode. The electrolyte was 1 M KPF$_6$ in in ethyl carbonate/dimethyl carbonate (DMC) + fluoroethylene carbonate (FEC) (EC:DMC, 1:1 by volume ratio, 5 wt% FEC) and glass microfiber filter as the separator. The PIHC device is composed of NCOP anode and BCN cathode in a glove box filled with argon. Note that the electrode was pre-potassiated by directly contacting with potassium metal in the electrolyte before assembling the device.

S3. Characterization Methods

Scanning electron microscopy (SEM) images were obtained using a Hitachi S-4800 field emission microscope. Raman spectra of samples were obtained with a LabRAM HR system of Horiba Jobin Yvon at room temperature and using a 447 nm solid state laser as the excitation source. XRD was collected by a Bruker D8 Advance X-ray diffractometer with Cu Kα radiation ($\lambda = 1.5406$ Å). High-resolution
transmission electron microscopy (HRTEM) images were obtained on a Philips Tecnai Twin-20U high-resolution transmission electron microscope that operates at an accelerating voltage of 200 kV. The EPR spectra were obtained using a JEOL (FA200) EPR spectrometer at X-band (≈ 9 GHz) at 123.15 K. The Co K-edge and Ni K-edge X-ray absorption near-edge spectra (XANES) were performed at the BL14W1 beamline of Shanghai Synchrotron Radiation Facility (SSRF). The surface area was determined by Brunauer–Emmett–Teller (BET) measurements with an ASAP 2020 sorptometer. PSD was determined according to Barrett–Joyner–Halenda (BJH) desorption dV/dlog(r) plot volume for the mesoporous, and the microporous was determined by H-K method.

**S4. Electrochemical Measurements.**

Electrochemical impedance spectroscopy (EIS) measurements and cyclic voltammogram (CV) measurements were carried out using a CHI 660E electrochemical workstation. Alternating current EIS spectra were collected within a frequency range of 10^{-2} Hz–10^5 Hz at the open circuit voltage with AC amplitude of 0.005 V. The galvanostatic charge–discharge performance of the half cells was performed on a Neware battery test system, and the galvanostatic charge–discharge performance of the potassium-ion hybrid capacitors (PIHC) was carried out using a CHI 660E electrochemical workstation. All of the electrochemical tests were implemented at room temperature.

Specific capacitance (F g^{-1}) of the PIHC device was calculated through the following equations:

\[
C_{cell} = \frac{l\Delta t}{m\Delta V} \quad (1)
\]
where \( I \) is the constant discharge current (A), \( \Delta t \) is the discharge time (s), \( \Delta V \) is the voltage window (V), \( m \) is the total mass loading of the active material on the two electrodes (mg).

The energy density \( (E) \) and power density \( (P) \) of the SIC were estimated according to Equation 3-5, respectively, represented as:

\[
P = \Delta V \times i/m \quad (2)
\]

\[
E = P \times t/3600 \quad (3)
\]

\[
\Delta V = (V_{\text{max}} + V_{\text{min}})/2 \quad (4)
\]

where \( i \) (A), \( m \) (g), and \( \Delta t \) (s) denote the discharge current, mass of active materials, and discharge time, respectively. \( V_{\text{min}} \) (V) and \( V_{\text{max}} \) are the minimum and maximum working voltage during the charge-discharge process, respectively.

**S5. Calculation of Capacitance Contribution**

The \( b \) value from \( i = av^b \) can be obtained by plotting current and sweep rate in logarithm, namely, the gradient of linear plots.

\[
\log i = b \log v + \log a \quad (5)
\]

In reality, the current can originate from both aforementioned contributions. Therefore, the \( b \) value can vary between 0.5 and 1. Taking that into consideration, we can rewrite Equation (6) so that the current is a sum of two parts: capacitive current (viz., \( b=1 \)) and ion-diffusion controlled one (viz., \( b=0.5 \)):

\[
i(V) = k_1 v + k_2 v^{1/2} \quad (6)
\]

We are interested in the percentage of the capacitive current. So, in order to determine the \( k_1 \) value, equation (7) can be reformulated as:
\[ i(V)/v^{1/2} = k_1 v^{1/2} + k_2 \]  

(7)

Obviously, with a series test of CV curves under different scan speed, the \( k_1 \) value can be determined from the linear plots of \( i(V)/v^{1/2} \) vs \( v^{1/2} \).

**S6. Supplementary Figures S1–S15**

![SEM images](image)

**Figure S1.** SEM images of the carbon cloth before (a) and after (b) plasma-treatment, and the SEM images of Ni-Co precursors (c-d).
Figure S2. SEM images and the corresponding mapping images of the NCOP samples.

Figure S3. SEM images of the NCO (a, b) and NCP (c, d).
Figure S4. The XPS survey (a) and the C 1s spectrum (b) of the Ni-Co based materials.

Figure S5. FTIR spectra of the Ni-Co composites
Figure S6. The CV and GCD curves of the NCO (a, b) and NCP (c, d) electrode, respectively.

Figure S7. The electrochemical performance of the bare carbon cloth substrate in K ion half cells, the GCD curves at 0.1 A g\(^{-1}\) (a) and CV curves (b) at 1 mV s\(^{-1}\).
Figure S8. SEM images of the NCOP before (a) and after (b) 1,000 cycles at 1 A g⁻¹.

Figure S9. The cycling performance of the NCOP at 0.1 A g⁻¹

Figure S10. (a) CV curves of NCOP at scan rates range from 0.1 to 1.0 mV s⁻¹. (b) the contribution ratio of the capacitive capacities and diffusion-limited capacities at 1 mV
s\textsuperscript{-1}, insert show Linear relationship of log(i) vs. log(ν).

As shown in the Figure S10, the b value of the anodic and cathodic was calculated from the peak position at 0.58 V and 2.45 V, respectively.

**Figure S11.** In-situ XRD patterns of NCOP electrode during discharge/charge process.

**Figure S12.** The calculation structure of the NCO and NCOP.

**Figure S13.** Structure diagram of K\textsuperscript{+} embeddings in Ni\textsubscript{2}CoO\textsubscript{4} at S1 (a) and S2 (b) sites,
the structure diagram of the K⁺ embeddings in P-Ni₂CoO₄ at S1 sites (c).

The structure diagram of K⁺ embedded at different sites in Ni₂CoO₄ is selected. Figure S11a is site S1, Figure S11b is site S2, and the corresponding adsorption energy is -0.462 eV and -0.103 eV respectively. It can be seen from this that K⁺ is preferentially embedded at site S1, which is a gap site composed of Ni-Co-O. Figure S11c shows the structure of S1 site corresponding to potassium ion embedding in P-Ni₂CoO₄.

**Figure S11.** The morphology characterization of the BCN: the SEM image at high magnification (a) and low magnification (b, c); and the corresponding EDS result (d).
Figure S15. N$_2$ adsorption-desorption isotherm of the BCN, and the corresponding pore size distribution (b).

The pore size and the specific surface area of the BCN have been characterized using the N$_2$ adsorption–desorption isotherms (Figure S15). The Brunauer–Emmett–Teller (BET) specific surface area of 196.6 m$^2$ g$^{-1}$, and the pore size distribution revealing the pores size mainly concentrated in the micropore range (Figure S15b). These excellent properties give the nanocomposite better contact with the electrolyte/electrode and react thoroughly, thereby obtaining advanced energy storage performance.
Figure S16. The XPS spectra of survey spectra of BCN (a), corresponding high-resolution B 1s (b), C 1s (c), and N 1s (d) spectra, respectively.

Figure S17. The LSV curves of the BCN materials in potassium-ion half-cell.
**Figure S18.** Electrochemical performance of BCN electrode: CV curves at increasing scan rate (a); GCD curves at an increasing current density (b); the cycling performance at 1 A g$^{-1}$ (c), and EIS plots before and after 1000 cycles (d).

The CV curves (Figure S18a) of the BCN with some obvious redox peaks appears enhanced capacitance performance, which means the B/N co-doping strategy makes the carbon material not only with double layer contribution but also with fast surface pseudocapacitance contribution. The GCD curves of BCN electrode in Figure S18b reveal a linear shape, indicating that the electrode operates at an electrochemical double-layer capacitance. The BCN cathode delivers a capacity of 86 mAh g$^{-1}$ at 0.5 A g$^{-1}$, even at 20 A g$^{-1}$, it also exhibits a capacity of 56 mAh g$^{-1}$. Furthermore, the electrode delivers outstanding cycling performance with 92.4% retention after 1000 cycles.
cycles at 1.0 A g$^{-1}$ (**Figure S18c**).

**Figure S19.** The specific capacity of NCOP//BCN hybrid devices with different mass ratios of positive vs. negative materials.

**Table S1.** The ICP-OES result of the NCOP powder material

| Abbreviated chemical formula | Measured atomic ratio |
|-----------------------------|-----------------------|
| NiCo$_2$O$_3$P              | Ni        | Co    | P       |
|                             | 1.0       | 2.23  | 0.78    |
Table S2. Comparison of KIB performance of NiCoOP composite with transition metal oxides-based electrode reported in the literatures.

| Materials          | Capacitance mAh g⁻¹ | Cycle performance                          | Reference                                       |
|--------------------|----------------------|--------------------------------------------|------------------------------------------------|
| NCOP               | 0.1 A g⁻¹ 721.5 mAh g⁻¹ | 1 A g⁻¹-332.5 mAh g⁻¹-500-62%              | This work                                      |
| Fe-Ni-P            | 50 mA g⁻¹ 168 mAh g⁻¹ | 0.2 A g⁻¹-33 mAh g⁻¹-1000-70%              | Chem. Engineer. J 2020, 390, 124515.           |
| C-WS₂@CNFs         | 50 mA g⁻¹ 319 mAh g⁻¹ | 2 A g⁻¹-158mA h g⁻¹-300-91%               | Energy Environ. Sci. 2021, 14, 3184            |
| CoV₂O₆@GO          | 100 mA g⁻¹ 557 mAh g⁻¹ | 1 A g⁻¹-158.4 mAh g⁻¹-2000-60%            | Energy Storage Mater. 2021, 40, 250.           |
| MoP@NC             | 100 mA g⁻¹ 256.1 mAh g⁻¹ | 4 A g⁻¹-255.2 mAh g⁻¹-800-89.9%          | Adv. Sci. 2021, 8, 2004142                     |
| MoP@CF             | 100 mA g⁻¹ 320 mAh g⁻¹ | 2 A g⁻¹-220 mAh g⁻¹-200-90%               | Small 2019, 16, 1905301.                       |
| Se₃P₄@C            | 50 mA g⁻¹ 1036.8mA h g⁻¹ | 1 A g⁻¹-220 mAh g⁻¹-300-79%              | Small 2020, 16, 1906595.                      |
| CoP@C              | 50 mA g⁻¹ 307 mAh g⁻¹ | 1 A g⁻¹-40 mAh g⁻¹-1000-99%              | Angew. Chem. Int. Ed. 2020, 59, 5159.         |
| NiCo₂Si@RGO        | 50 mA g⁻¹ 602 mAh g⁻¹ | 0.2 A g⁻¹-495 mAh g⁻¹-1900-82%            | Angew. Chem. Int. Ed. 2019, 58, 14740.        |
| FeP/C              | 50mA g⁻¹ 289 mAh g⁻¹ | 0.05 A g⁻¹-289mA h g⁻¹-50-63.2%          | ACS Appl. Mater. Interfaces, 2019, 11, 22364. |
| SnO₂@CF            | 500mA g⁻¹ 371 mA h g⁻¹ | 1 A g⁻¹-231.7 mA h g⁻¹-4000-93.7%       | Energy Environ. Sci. 2020, 13, 571.           |
| NiS₄@C             | 100mA g⁻¹ 415 mA h g⁻¹ | 2 A g⁻¹-116 mAh g⁻¹-800-93.8%            | J. Mater. Chem. A 2019, 7, 18932.            |
| Fe₃O₄/NPGF         | 200mA g⁻¹ 200 mA h g⁻¹ | 1 A g⁻¹-154.6 mA h g⁻¹-500-76.4%        | J. Mater. Chem. A 2019, 7, 19430.            |
| FeP/GN             | 100 mA g⁻¹ 323 mA h g⁻¹ | 2 A g⁻¹-127mA h g⁻¹-2000-97.6%          | J. Mater. Chem. A 2020, 8, 3369.             |
| CoP nanorods       | 20 mA g⁻¹ 374 mA h g⁻¹ | 0.2A g⁻¹-350mA h g⁻¹-5000-54%            | Nanoscale Horiz. 2019, 4, 1394.               |
| SnS₂@rGO           | 50 mA g⁻¹ 448 mAh g⁻¹ | 0.2 A g⁻¹-205 mAh g⁻¹-300-73%            | Small 2019, 15, 1804806.                      |
| WS₂-NCN            | 50 mA g⁻¹ 469 mAh g⁻¹ | 0.5A g⁻¹-315 mAh g⁻¹-500-96.8%          | Matter 2019, 1, 893.                           |
| MoSe₂/N-C          | 100 mA g⁻¹ 300 mA h g⁻¹ | 0.1A g⁻¹-258.2mA h g⁻¹-300-100%         | Adv. Energy Mater. 2018, 1801477.             |
Table S3. The K ion is embedded in Ni$_2$CoO$_4$ and P-Ni$_2$CoO$_4$, corresponding the lattice parameters, volume change rate and binding energy.

| Structure   | Lattice parameters | Volume(Å$^3$) | $\Delta V$ (Å$^3$) | Eb (eV)  |
|-------------|--------------------|---------------|---------------------|---------|
| Ni$_2$CoO$_4$ | a=8.1513, b=8.1513, c=8.1513 | 541.363       | --                  | --      |
| K-Ni$_2$CoO$_4$ | a=8.5788, b=8.5535, c=8.4336 | 626.185       | 13.5%               | -0.462  |
| P-Ni$_2$CoO$_4$ | a=8.4911, b=8.4911, c=8.4817 | 611.520       | --                  | --      |
| K-P-Ni$_2$CoO$_4$ | a=8.6613, b=8.5613, c=8.5716 | 635.610       | 3.7%                | -0.861  |
Table S4. Comparison of KIHCs performance of NiCoOP with currently reported in the literatures.

| Materials       | voltage window (V) | Capacitance F g⁻¹ | Cycle performance | Power density kW kg⁻¹ | Energy density Wh kg⁻¹ | Reference                                      |
|-----------------|--------------------|-------------------|-------------------|------------------------|------------------------|-----------------------------------------------|
| NCOP//BCN       | 0.01-4.5           | 0.1 A g⁻¹, 59.3 F g⁻¹ | 5.0 A g⁻¹-10000-86.5% | 22.5                   | 166.5                  | This work                                     |
| Co₃P@rGO//AC    | 1.0-4.0            | 0.02 A g⁻¹, 11.75 F g⁻¹ | 1 A g⁻¹-1000-68%   | 4.265                  | 87                     | Nanoscale Horiz. 2019, 4, 1394.               |
| NCHS//WS₂@C     | 0.5-4.2            | 0.05 A g⁻¹, 57.8mAh g⁻¹ | 0.2 A g⁻¹, 2000-   | 0.235                  | 103.4                  | Matter 2019, 1, 893.                          |
| CoP@NPC//MB     | 0.01-4.0           | 0.5 A g⁻¹, 112.5 F g⁻¹ | 1 A g⁻¹-83%-3000   | 11                     | 180.3                  | J. Mater. Chem. A 2021, 9, 16028              |
| CoV₂O₆@GO//AC   | 0.5-4.0            | 0.05 A g⁻¹, 75 F g⁻¹ | 1 A g⁻¹-96.8%-3000 | 22.5                   | 78.2                   | Energy Storage Mater. 2021, 40, 250.         |
| MoP@NC//AC      | 0.01-4.0           | 0.05A g⁻¹, 32 F g⁻¹  | -                 | 20.42                  | 69.7                   | Adv. Sci. 2021, 8, 2004142.                  |
| TiO₂//NC//AC     | 0.01-3.8           | 0.05 A g⁻¹, 60 mAh g⁻¹ | 2.5 A g⁻¹-3000-100% | 5.500                  | 108.6                  | Chem. Engine. J. 2021, 417, 127977.          |
| C-WS₂//ACNF     | 0.01-4             | 0.2 A g⁻¹, 65 mAh g⁻¹ | 4 A g⁻¹-5500-81.4% | 12.6                   | 180.4                  | Energy Environ. Sci. 2021, 14, 3184.         |
| CNS//FCDAC      | 0.01-4.2           | 0.5 A g⁻¹, 144mAh g⁻¹ | 2 A g⁻¹-5000-80%   | 21                     | 149                    | Adv. Energy Mater. 2019, 9, 1803894.         |
| MoSSe//AC       | 0.01-3.5           | 0.2A g⁻¹, 32 F g⁻¹   | 2 A g⁻¹-6000-91.3% | 15.4                   | 77.4                   | J. Mater. Chem. A 2020, 8, 13946.            |
| CoP@rGO//AC     | 1.0-4.0            | 0.02 A g⁻¹, 153 F g⁻¹ | 2 A g⁻¹-1000-68%   | 4.265                  | 87                     | Nanoscale Horiz. 2019, 4, 1394.              |
| MoP@NC-1//AC    | 0.01-4.0           | 0.05A g⁻¹, 32.5 F g⁻¹ | 1 A g⁻¹-2000-61.2% | 2.04                   | 69.7                   | Adv. Sci. 2021, 8, 20040142.                |
| CBC@G//ACBC     | 0.01-4.0           | 0.05A g⁻¹, 91 mAh g⁻¹ | 5A g⁻¹-5000-81.5%  | 22                     | 172                    | Adv. Sci. 2020, 7, 2001681                   |
| AC//S-N-PCNs    | 0.01-4.0           | 0.05 A g⁻¹, 95 F g⁻¹ | 1 A g⁻¹-3000-86.4% | 2.136                  | 197                    | Adv Energy Mater 2019, 9, 1901533.           |
| P/O-PCS//AC     | 0.01-4.0           | 0.05 A g⁻¹, 30 F g⁻¹  | 5 A g⁻¹-3000-94.5% | 1.38                   | 158                    | Adv. Funct. Mater. 2021, 31, 2102060.        |
| SGN-900//NGN    | 0-4.2              | 1 A g⁻¹-100 F g⁻¹    | 2 A g⁻¹-6000-92%   | 14.6                   | 112                    | ACS Nano 2021, 15, 1652                      |
| WS2@CNFs//ACNFs | 0-4.0              | 0.2A g⁻¹, 77.2 F g⁻¹  | 4 A g⁻¹-5500-81.4% | 12.6                   | 180.4                  | Energy Environ. Sci., 2021,14, 3184.         |
| Material       | C/N           | I/A-G          | E/V            | I/A-G          | C/%         | Paper Details               |
|----------------|---------------|----------------|----------------|----------------|-------------|-----------------------------|
| BPCS//NPC      | 0.01-4.5      | 0.5 A g⁻¹-     | 2 A g⁻¹-       | 30             | 167         | ACS Nano 2020, 14, 4938.   |
|                |               | 86.1 F g⁻¹     | 10000-100%     |                |             |                             |
| Ti₃C₂//HPAC     | 1.0-4.0       | 0.05 A g⁻¹-     | 1 A g⁻¹-       | 7.02           | 98.4        | Adv. Funct. Mater. 2020, 30, 2005663. |
|                |               | 45 F g⁻¹       | 10000-100%     |                |             |                             |
| Ti₃C₂Tx//AC     | 1.0-4.0       | 0.05 A g⁻¹-     | 1 A g⁻¹-       | 8.7            | 163         | Angew. Chem. Int. Ed. 2021, 60, 26246. |
|                |               | 32 F g⁻¹       | 10000-101%     |                |             |                             |
| APN-HPCNF//PN-HPCNF | 0.01-4.0 | 0.05 A g⁻¹-     | 1 A g⁻¹-       | 7.56           | 191         | Energy Environ. Sci., 2020, 13, 2431-2440 |
|                |               | 93.5 F g⁻¹     | 8000-82.3%     |                |             |                             |
| HCMB//AC       | 2.5-4.5       | 0.1 A g⁻¹-      | 20 mV s⁻¹      | 17.5           | 152         | Energy Environ. Sci., 2021, DOI: 10.1039/D1EE03214C |
|                |               | 78 F g⁻¹       | 1000-90%       |                |             |                             |
| BSH//AC        | 0.01-4.0      | 0.05 A g⁻¹-     | 0.35 A g⁻¹-    | 0.6            | 120         | Adv. Mater. 2018, 30, 1800804. |
|                |               | 65.5 F g⁻¹     | 1000-71.4%     |                |             |                             |
| NbSe₂/NSeCN Fs//AC | 0.01–3.8  | 0.05 A g⁻¹-     | 2 A g⁻¹-       | 4.0            | 145         | Adv. Funct. Mater. 2020, 30, 2004247. |
|                |               | 88 F g⁻¹       | 10000-83%      |                |             |                             |