An all Prussian blue analog-based aprotic sodium-ion battery

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Abstract
Sodium-ion batteries have been regarded as ideal alternatives to lithium-ion batteries in large-scale electrochemical energy storage systems because of their low cost and abundant resources. However, the lack of high-performance electrode materials with high energy density hinders the development of sodium-ion batteries. Prussian blue analogs (PBAs) are especially regarded as superior cathodes for sodium-ion batteries due to their high capacity and good electrochemical stability. The species and chemical environment of transition metal ions in PBAs greatly affect the electrochemical properties. In this study, we report a sodium cobalt hexacyanoferrate \( \text{Na}_x\text{Co}[\text{Fe(CN)}_6] \) and potassium iron hexacyanocobaltate \( \text{K}_x\text{Fe}[\text{Co(CN)}_6] \) nanoparticles through the coprecipitation method via changing the coordination structures of Co and Fe. With similar morphologies of well-defined nanocubes and different \( \text{M}_1–\text{CN}–\text{M}_2' \), the initial discharge capacities of \( \text{Na}_x\text{Co}[\text{Fe(CN)}_6] \) cathode and \( \text{K}_x\text{Fe}[\text{Co(CN)}_6] \) anode reach 104.1 and 354.8 mAh g\(^{-1}\), respectively. The sodium-ion full cell assembled using \( \text{Na}_x\text{Co}[\text{Fe(CN)}_6] \) cathode and \( \text{K}_x\text{Fe}[\text{Co(CN)}_6] \) anode achieves a discharge capacity of 141.6 mAh g\(^{-1}\). The outstanding performances of the \( \text{Na}_x\text{Co}[\text{Fe(CN)}_6] \) and \( \text{K}_x\text{Fe}[\text{Co(CN)}_6] \) are attributed to their stable structures, and the facile synthesis of \( \text{Na}_x\text{Co}[\text{Fe(CN)}_6] \) and \( \text{K}_x\text{Fe}[\text{Co(CN)}_6] \) makes them accessible for large-scale applications.

KEYWORDS
potassium iron hexacyanocobaltate, Prussian blue analogs, sodium cobalt hexacyanoferrate, sodium-ion battery

1 INTRODUCTION
In recent years, with the rapid development of electric vehicles and portable electronic devices, secondary battery technology with high energy density, high conversion efficiency, flexibility, and reliability has attracted wide attention both from academia and industry.\(^{1-6}\) However, lithium-ion batteries (LIBs) are predicted to face unsustainability due to the rarity and uneven distribution of lithium resources.\(^{7-9}\) For this reason, sodium-ion batteries (SIBs) have been considered as the promising complementary alternatives to LIBs owing to the utilization of earth-abundant and much cheaper sodium resources.\(^{10-14}\) Electrode material is the key...
factor to dominate the electrochemical performances of SIBs. Prussian blue analogues (PBAs) is one of the promising high-performance cathode materials allowing reversible insertion/extraction of Na⁺ in the structural framework, besides sodium transition metal oxides, polyanion compounds, and organic compounds. PBAs has the advantages of facile preparation, environmental benignity, and low price.

The general composition of PBAs can be expressed as $A_x M_1[\text{M’CN}]_n \cdot y \text{H}_2\text{O}$, the transition metal $M_1$ and $M_2$ are matched with the $N$ and $C$ in the CN ligand to form a three-dimensional open frame structure with $M_1$–$N$–$C$–$M_2$ chain. The electrochemical performances of the material can be adjusted by controlling the types of $M_1$ and $M_2$. For example, the length of the $M_2$–$C$ bond has a great influence on the lattice parameters and channel sizes, affecting the transport behaviors of sodium ions in the PBAs. In addition, the lattice of PBAs may become unstable due to the $[\text{Fe(CN)}_6]^{3−}$ vacancies breaking the $M_1$–$C$–$N$–$M_2$ chain and forming defects during the charge and discharge processes. Moreover, the electron transfer through $M_1$–$C$–$N$–$M_2$ could be destroyed by the existence of $[\text{Fe(CN)}_6]^{3−}$ vacancies, causing an increased Ohmic polarization.

Goodenough’s group developed Na₂MnFe(CN)₆ by removing the interstitial H₂O, which exhibits a capacity of 150 mAh g⁻¹. Goodenough’s group reported that Na₂MnMn(CN)₆ has a high specific capacity of 209 mAh g⁻¹. Cui’s group reported that Na₂MnFe(CN)₆ has a high specific capacity of 209 mAh g⁻¹. However, most Mn-PBAs suffer from low capacity and poor cyclability due to Jahn–Teller distortion of Mn³⁺ and H₂O in the lattice. These problems deteriorate the performances of PBAs. Guo’s group reported a single-iron source route to achieve significant cycling performance. To prepare high-performance PBAs, Han and coworkers reported a NiₓCo₁₋ₓ[Fe(CN)₆] PBAs by a chelating agent/surfactant coassembled crystallization method, which exhibits a capacity of 145 mAh g⁻¹.

In this study, both Na₂Co[Fe(CN)₆] (CF) and KₓFe[Co(CN)₆] (FC) were prepared by a simple coprecipitation method by changing the precursor. The influence of the coordination structure on their electrochemical performance is studied. Both the materials demonstrate good performance in sodium-ion half-cell, as well as the full-cell, using CF cathode coupled with FC anode.

2 | EXPERIMENTAL SECTION

2.1 | Materials

Potassium hexacyanocobaltate (K₃Co(CN)₆, 99%), ferrous chloride tetrahydrate (FeCl₂·4H₂O, 99%; Sinopharm Chemical Reagent Co., Ltd.), sodium hexacyanoferrate decahydrate (Na₄Fe(CN)₆·10H₂O, 99%; Sinopharm Chemical Reagent Co., Ltd.), cobalt chloride hexahydrate (CoCl₂·6H₂O, 99%; Sinopharm Chemical Reagent Co., Ltd.), trisodium citrate dihydrate (Na₃C₆H₅O₇·2H₂O, 98%), Ketjen black (Lion Specialty Chemicals Co., Ltd.), polyvinylidene fluoride (PVDF), N-methylpyrrolidone (NMP, 99.5%; Sinopharm Chemical Reagent Co., Ltd.), glass microfiber (GF/D; Whatman), Al foil (Al/C; Shenzhen Kejing Star Technology Company), Cu foil (Cu/C; Shenzhen Kejing Star Technology Company), sodium (Na, 99.7%), electrolyte (NC-046; Suzhou Duoduo Chemical Technology Co., Ltd.).

2.2 | Materials preparation

Synthesis of KₓFe[Co(CN)₆] sample. First, K₃Co(CN)₆ and FeCl₂·4H₂O were dissolved in 50 ml of deionized water and stirred for 30 min to obtain Solutions A and B with a concentration of 0.1M, respectively. Subsequently, Solution A was mixed with Solution B under stirring for 10 min and the mixture was aged for 24 h. The precipitate was obtained by centrifugation and washed with deionized water and ethanol several times. In the end, the KₓFe[Co(CN)₆] (FC) was dried in a vacuum oven at 120°C for 24 h.

Synthesis of Na₂Co[Fe(CN)₆] sample. First, 0.9681 g Na₂Fe(CN)₆·10H₂O was dissolved in 100 ml of deionized water to obtain Solution A, 2.253 g Na₄Fe(CN)₆·6H₂O and 2 mmol CoCl₂·6H₂O were dissolved in 100 ml of deionized water to obtain Solution B. After stirring for 10 min, Solution A was mixed with Solution B. Then, the mixture was stirred for 10 min and aged for 12 h. The precipitate was obtained by centrifugation and washed with deionized water and ethanol several times. The Na₂Co[Fe(CN)₆] (CF) was dried in a vacuum oven at 120°C for 24 h.

2.3 | Material characterization

Fourier transform infrared spectroscopy (FT-IR; SGE/Agilent 6890/Nicolet 5700) was obtained within 400–4000 cm⁻¹. The morphology of the composites was characterized by scanning electron microscopy (SEM; Hitachi S-4800). The composition and crystal structure were characterized by X-ray diffraction (XRD) in a 2θ range of 10–80°. Thermal gravimetric analysis (TGA) was performed on a Pyris 1 TGA thermal analyzer in the air at a heating rate of 10°C min⁻¹.

2.4 | Electrochemical performance test

The as-synthesized samples, Ketjen black, and PVDF binder were mixed in the mass ratio of 7:2:1 using NMP solvent. The resultant slurry was coated onto Al foil (cathode)
and Cu foil (anode) to prepare the working electrode. Electrochemical tests were carried out in 2025 coin-type cells in which a piece of sodium foil was used as both the counter and reference electrode and 1M NaClO₄ in ethylene carbonate, diethyl carbonate, and propylene carbonate (2:2:1 in a volume ratio) containing 5% fluoroethylene carbonate was used as the electrolyte. The full cell was also assembled using CF as cathode and FC as an anode. The preparation of cathode and anode electrodes of the full cell is the same as that of a half cell. Galvanostatic tests were carried out in the potential range 2.0–4.0 V for half cell and 1.0–4.0 V for full cell by using a battery test system (Neware BTS-5). Cyclic voltammetry (CV) in the potential range of 2.0–4.0 V and electrochemical impedance spectra (EIS) from 1 MHz to 1 Hz were measured by using a CHI660E electrochemical workstation.

3 | RESULTS AND DISCUSSION

3.1 | Morphology and structural analysis

Figure 1 shows the SEM images of Na₅Co[Fe(CN)₆] and K₅Fe[Co(CN)₆]. It can be seen that both PBAs show cubic morphology with a particle size of ~400 nm. While

![SEM images](image1.png)

**FIGURE 1** (A) SEM image of Na₅Co[Fe(CN)₆]; (B) SEM image of K₅Fe[Co(CN)₆]. SEM, scanning electron microscopy

![XRD patterns](image2.png)

**FIGURE 2** (A) XRD patterns of Na₅Co[Fe(CN)₆] and K₅Fe[Co(CN)₆]; (B) The FT-IR spectra of Na₅Co[Fe(CN)₆] and K₅Fe[Co(CN)₆]; TGA curve of Na₅Co[Fe(CN)₆] (C) and K₅Fe[Co(CN)₆] (D) in N₂. FT-IR, Fourier transform infrared spectroscopy; TGA, thermal gravimetric analysis; XRD, X-ray diffraction
K$_x$Fe[Co(CN)$_6$] is composed of irregular cubes, Na$_x$Co[Fe(CN)$_6$], on the other hand, has a smoother surface and presents uniform distribution.

The XRD patterns and FT-IR spectra were used to further characterize the crystal structure of the material. As can be seen from Figure 2A, both Na$_x$Co[Fe(CN)$_6$] and K$_x$Fe[Co(CN)$_6$] can be assigned to face-centered-cubic phases with the Fm-3m space group. Specifically, the XRD diffraction peak of FC shifts to the right, and the lattice constant of CF is larger than that of FC, and the difference of peak intensity indicates that the two samples have different crystal orientation distributions. As shown in Figure 2B, the absorption bands at 3416 and 1620 cm$^{-1}$ are ascribed to the bending vibration of H–O and H–O–H, respectively. For CF, the peaks located at 2180, 596, and 480 cm$^{-1}$ are characteristic peaks of PBAs. The former corresponds to the stretching vibration absorption peak of cyano C≡N, and the latter two correspond to the absorption peaks of M$_1$–CN–M$_2$'. The characteristic peak of C≡N shifts to 2088.2 cm$^{-1}$, indicating that its bond strength is weaker, which is caused by the higher valence states of cobalt and iron in FC. The difference of the M$_1$–CN–M$_2'$ peaks of CF and FC also indicate the different coordination structures: CF is Fe–C≡N–Co and FC is Co–C≡N–Fe. TG analysis was measured in a nitrogen atmosphere, as shown in Figure 2C,D, and the water content of CF and FC are 10.9 and 15.8 wt%, respectively.

3.2 | Electrochemical performance

Figure 3 shows the CV (the potential 2.0–4.0 V vs. Na/Na$^+$) curves of CF and FC. The CV curve of CF displays two pairs of oxidation/redox peaks at 3.10/3.40 and 3.72/3.90 V, upon two reversible Na$^+$ insertions/extractions of the structural framework, which could be attributed to the C-coordinated Fe$^{2+}$/Fe$^{3+}$ and N-coordinated Co$^{2+}$/Co$^{3+}$ redox couples, respectively. However, in the CV curve of FC, as shown in Figure 3B, there is only a pair of oxidation/redox peaks at 3.09/3.45 V, which indicates that Co is electrochemical inactive in FC. Figure 3C,D displays the EIS curves of CF and FC, the charge transfer resistance ($R_{ct}$) of CF is 309 $\Omega$, which is significantly smaller than that of FC (590 $\Omega$), suggesting faster Na$^+$ ions intercalation/deintercalation in the CF electrode.

The corresponding charge/discharge curves of CF at 100 mA g$^{-1}$ in Figure 4A indicate two distinct sets
of plateaus, which is in consistent with the CV results. The first charge–discharge capacity of CF is 140.0/104.1 mAh g\(^{-1}\). Since the initial charge of FC is a deintercalation process of potassium ions, the capacity is only 28.6 mAh g\(^{-1}\) (Figure 4B). FC has only one pair of charge/discharge plateau, its capacity is significantly lower than CF, and the second charge/discharge capacity is 32.5/31.4 mAh g\(^{-1}\). In addition, the transition metal Co and Fe in the FC have changed valence states,\(^{37}\) and there are only one pair of oxidation/redox peaks and charge/discharge plateaus between 2.00 and 4.00 V. FC was investigated as the anode electrode instead to study its electrochemical behavior at the low potential of 0.01–1.60 V.\(^{38}\) As shown in Figure 4C, its first discharge/charge capacity is 1291.5/354.8 mAh g\(^{-1}\), and the corresponding initial Coulombic efficiency is only 27.5%; The capacity degradation is mainly due to the formation of solid-electrolyte interphase and the irreversible reaction between sodium-ions and FC. Then, the Na\(_x\)Co[Fe(CN)\(_6\)]/K\(_x\)Fe[Co(CN)\(_6\)] organic system sodium-ion full cell was assembled by using CF and FC as cathode and anode electrodes, respectively.\(^{28,39,40}\) As shown in Figure 4D, the charge/discharge tests were performed at a current density of 50 mA g\(^{-1}\) in the voltage range of 1.00–4.00 V and the discharge capacity is 141.6 mAh g\(^{-1}\).

4 | CONCLUSION

In summary, Na\(_x\)Co[Fe(CN)\(_6\)] and K\(_x\)Fe[Co(CN)\(_6\)] were successfully prepared through a simple coprecipitation method. The CF electrode delivered a discharge capacity of 104.1 mAh g\(^{-1}\) when used as a cathode for SIBs. The FC is more suitable as an anode electrode, it has an initial charge capacity of 354.8 mAh g\(^{-1}\). The sodium-ion full cell consisting of CF cathode and FC anode delivers a discharge capacity of 141.6 m Ah g\(^{-1}\) with a voltage range of 1.00–4.00 V. The application of CF and FC might shed light on the design of high voltage and high capacity electrode materials for SIBs.

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CONFLICTS OF INTEREST

The authors declare no conflicts of interest.
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DATA AVAILABILITY STATEMENT
The data presented in this study are available on request from the corresponding author.

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