Electronic supplementary information for

Bismuth oxychloride nanoflake assemblies as a new anode for potassium ion batteries

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Experimental details

Materials preparation: BiOCl NFAs were synthesized through a solvothermal reaction. In a typical procedure, polyvinylpyrrolidone (PVP, 1.5 g) was dissolved in ethylene glycol (EG, 40 mL). Bi(NO₃)₃·5H₂O (0.97 g) was added to the solution, followed by adding NH₄Cl (0.268 g) while vigorously stirring the solution for 30 min to ensure all reagents were dispersed homogeneously. The solution was transferred into a Teflon-lined stainless-steel autoclave with a capacity of 50 mL, sealed and heated at 170°C for 20 h. The mixture cooled to room temperature naturally. The powders were collected and rinsed with deionized water and ethanol repeatedly to remove the residual trace of reactants. Finally, the powders were dried in an oven at 60 °C under a vacuum.

Materials characterizations: X-ray diffraction (XRD) analysis was performed on an 18 KW D/MAX2500V+/PC diffractometer using Cu K α (λ =1.54 Å) radiation at a scanning rate of 2°/min. Scanning electron microscopy (SEM) analysis was conducted using a JEOL JSM-7500F field-emission scanning microscopy. Transmission electron microscopy (TEM) analysis was performed on a JEOL JEM-2100F transmission electron microscope. X-ray photoelectron spectroscopy (XPS) was acquired on a ThermoFisher ESCALAB 250 Xi with Al K α (h ν =1486.6 eV) as the excitation source. The binding energies obtained in the XPS spectra analysis were corrected for specimen charging by referencing C 1s to 284.8 eV.

Electrochemical measurements: working electrodes were fabricated by mixing BiOCl NFAs, Super P and carboxymethyl cellulose sodium (CMC) with a weight ratio of 7:2:1. The mixture was coated (doctor-blade) uniformly on a copper foil with a mass loading of 1-2 mg cm⁻². It was then dried at 110°C under a vacuum for 12 h. Electrochemical measurements were conducted using coin cells, CR2032, and the cells were assembled in an argon-filled glovebox with oxygen and moisture concentrations below 0.1 ppm. K as the counter electrode was separated from the working electrode by a piece of glass microfiber filter (Whatman, Grade GF/B). The electrolyte was 1.0 M potassium bis(fluorosulfonyl)imide (KFSI) in a mixture of ethylene carbonate (EC) and diethyl carbonate (DEC) with a volumetric ratio of 1:1. Cyclic voltammetry was carried out on a VSP electrochemical workstation (Bio-Logic, France) in a scan rate range of 0.1 mV s⁻¹. Galvanostatic charge/discharge was performed on a Land CT 2001A battery testing system (Land, China) in a voltage window of 0.01-2.0 V at the rates of 0.05-1 A g⁻¹.



Fig. S1 XPS survey spectrum of BiOCl NFAs.



Fig. S2 TEM image of a single BiOCl NFA, showing the assembling structure and a thickness of the nanoflakes (40 nm).



Fig. S3 SEM image of BiOCl NFAs after ultrasonic treatment for 1 h.



Fig. S4 HRTEM of BiOCl nanoflake showing the lattice fringes along the edges and demonstrating the lateral surfaces are {110} facets.



Fig. S5 O 1s XPS spectra of the BiOCl electrodes at pristine state, after discharge and charge states.



Fig. S6 Cl 2p XPS spectra of the BiOCl electrodes at pristine state, after discharge and charge states.

$$BiOCI \xrightarrow{+K^{+}} Bi + K_{2}O + KCI (dissolved) \xrightarrow{+K^{+}} KBi + K_{2}O + K^{+} + K^{+}$$

Fig. S7 Scheme of the overall reaction pathways of the K⁺ storage in BiOCl.



Fig. S8 SEM images of BiOCl NFAs after 50 cycles, showing the preservation of the hierarchical structure and the nanoflakes.