## **Electronic supplementary information**

## Ultrathin Two-Dimensional Conjugated Metal-Organic Framework Single-Crystalline Nanosheets Enabled by Surfactant-Assisted Synthesis

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## **Materials and methods**

Cu(OAc)<sub>2</sub>, NaOH and sodium dodecyl sulfate (SDS) were purchased from Sigma-Aldrich. Tetrahydroxy-1,4quinone (THQ) was purchased from Fluorochem. Solvents (e.g., ethanol) were purchased from Sigma-Aldrich or TCI with HPLC grade. Water was purified using a Milli-Q purification system (Merck KGaA). All chemical and solvent were used directly without further purification. All the reactions were carried out under an ambient atmosphere. Substrates (e.g., 300 nm SiO<sub>2</sub>/Si wafer, quartz glass, and TEM grids) were obtained from Plano GmbH.

## **General characterization**

Powder X-ray diffraction (XRD) was carried out on Siemens D500 X-ray diffraction using Cu Ka (1.54 Å) radiation at room temperature. Optical microscopy (Zeiss), Atomic force microscopy (AFM) (NT-MDT), transmission electron microscopy (TEM, Zeiss, Libra 200 KV) and scanning electron microscopy (SEM, Zeiss Gemini 500) equipped with EDX were used to investigate the morphology and structure of the samples. The synthetic nanosheets (NSs) were deposited on Si substrate for SEM, and copper grids for TEM characterizations. All optical microscopy and AFM images were recorded on a 300 nm SiO<sub>2</sub>/Si substrate. Aberration-corrected high-resolution transmission electron microscopy (AC-HRTEM) imaging and selected-area electron diffraction (SAED) were conducted on an image-side  $C_s$ -corrected FEI Titan 80-300 microscope operated at 300 kV.

Raman spectra were collected with a Renishaw 2000 model confocal microscopy Raman spectrometer with a CCD detector and a holographic notch filter at ambient conditions. Fourier transforms infrared (FT-IR) spectra were collected using Tensor II (Bruker) with an attenuated total reflection (ATR) unit. Ultraviolet-Visible (UV-vis) spectra were recorded on an Agilent Cary 5000 UV-VIS-NIR spectrophotometer by using 10 mm optical-path quartz cell at room temperature. X-ray photoelectron spectroscopy (XPS) measurements were carried out using an AXIS Ultra DLD system. Both the survey and high-resolution spectra were collected using a beam diameter of 100 µm. All displayed binding energy values are calibrated to the graphitic C1s peak with a value of 284.6 eV. The specific surface area was measured by the Brunauer-Emmett-Teller (BET) method under low-pressure at 77 K. The data of X-ray absorption near edge structure (XANES) spectra and extended X-ray adsorption fine structure (EXAFS) spectra were collected at room temperature in transmission mode at beamline BL14W1 and BL15U1 of the Shanghai Synchrotron Radiation Facility (SSRF, China).

For all electrical measurements, i.e., temperature dependent conductivity, carrier concentration, and Hall mobility are characterized in the *van-der-Pauw*-geometry using a commercial Lakeshore Hall System (9700A). This fully integrated Hall measurement system is capable to detect the resistance ranging from 0.04 m $\Omega$  to 200 G $\Omega$ . Moreover, it is able to set magnetic field up to ±9 T by a superconducting magnet, and the temperature is varied from 1.8 to 400 K by liquid helium cooling. The silver-conductive-glue paste was used to contact the silver-wires to the samples. Standard sample with resistance ranging from 0.04 m $\Omega$  to 200 G $\Omega$  is within the detectable limitation of this fully integrated Hall measurement system. For the electrical conductivity measurements, samples were measured in the temperature range from 243 K to 310 K. After confirming the Ohmic contact at different temperatures, we collected an *I-V* curve by scanning the current from -10 nA to 10 nA and measuring voltage at each step at every certain temperature. For the Hall effect measurements, samples were measured

at certain temperature and a magnetic field perpendicular to the sample plane swept from -4 T to 4 T.

# Synthesis of HHB-Cu NSs

A 500 mL conical flask was used as the reaction container.  $Cu(OAc)_2$  (480 mg, 2.61 mmol) and SDS (150 mg, 0.51 mmol) in 150 mL of water were prepared. Then, a solution of NaOH (150 mg, 3.75 mmol) in 150 mL of water was added to the as-prepared  $Cu(OAc)_2$  and SDS solution, followed by the addition of THQ (300 mg, 1.74 mmol) powder to the mixture. The mixture was sonicated for 30 minutes at 50 °C, then the reaction system was allowed to stay undisturbed for 10 h at 25 °C. The precipitate was collected and washed by water and ethanol in an ultrasonic ice bath for 30 min.<sup>1</sup> After that, the dispersions were kept for 24 h to precipitate large particles. Then, the upper colloidal suspension of HHB-Cu NSs was collected and dried under vacuum for 12 h at 80 °C, while the precipitated large particles were collected for futher sonication. This process continues until the particles are fully exfoliated to afford HHB-Cu NSs in 87.5 % yield. The reaction is scalable by equivalently increasing monomers, SDS and base in the mixture.

## Synthesis of bulk HHB-Cu

A 500 mL conical flask was used as the reaction container.  $Cu(OAc)_2$  (480 mg, 2.61 mmol) in 300 mL NaOH (150 mg, 3.75 mmol) aqueous solution was prepared, followed by the addition of THQ (300 mg, 1.74 mmol) powder to the mixture. The mixture was sonicated for 30 minutes at 50 °C, then the reaction system was allowed to stay undisturbed for 10 h at 25 °C. The precipitate was collected and washed by water and ethanol, and dried under vacuum for 12 h at 80 °C (77.5 % isolated yield) for characterization and electrochemical measurement. The reaction is scalable by equivalently increasing monomers, SDS and base in the mixture.

## Synthesis of HHB-Ni nanosheets (HHB-Ni NSs)

A 500 mL conical flask was used as the reaction container. Ni(OAc)<sub>2</sub> (460 mg, 2.61 mmol) and SDS (150 mg, 0.51 mmol) in 150 mL of water were prepared. Then, a solution of NaOH (150 mg, 3.75 mmol) in 150 mL of water was added to the as-prepared Ni(OAc)<sub>2</sub> and SDS solution, followed by the addition of THQ (300 mg, 1.74 mmol) powder to the mixture. The mixture was sonicated for 30 minutes at 50 °C, then the reaction system was allowed to stay undisturbed for 10 h at 25 °C. The precipitate was collected and washed by water and ethanol in an ultrasonic ice bath for 30 min. After that, the dispersions were kept for 24 h to precipitate large particles. Then, the upper colloidal suspension of HHB-Ni NSs was collected and dried under vacuum for 12 h at 80 °C, while the precipitated large particles were collected for futher sonication. This process continues until the particles are fully exfoliated to afford HHB-Ni NSs in 85.5 % yield. The reaction is scalable by equivalently increasing monomers, SDS and base in the mixture.

## Synthesis of HHTP-Cu nanosheets (HHTP-Cu NSs)

A 500 mL conical flask was used as the reaction container.  $Cu(OAc)_2$  (254 mg, 1.395 mmol) and SDS (150 mg, 0.51 mmol) in 150 mL of water were prepared. Then, a solution of NaOH (150 mg, 3.75 mmol) in 150 mL of water was added to the as-prepared  $Cu(OAc)_2$  and SDS solution, followed by the addition of HHTP (300 mg, 0.93 mmol) powder to the mixture. The mixture was sonicated for 30 minutes at 50 °C, then the reaction system was allowed to stay undisturbed for 10 h at 25 °C. The precipitate was collected and washed by water and ethanol in an ultrasonic ice bath for 30 min. After that, the dispersions were kept for 24 h to precipitate large particles. Then, the upper colloidal suspension of HHTP-Cu NSs was collected and dried under vacuum for 12 h at 80 °C, while the precipitated large particles were collected for futher sonication. This process continues until the particles are fully exfoliated to afford HHTP-Cu NSs in 80.5 % yield. The reaction is scalable by equivalently increasing monomers, SDS and base in the mixture.

## **Preparation of organic cathodes**

To prepare the organic electrodes, the HHB-Cu NSs were blended with super P (conductive additive) and alginate sodium (binder) in a mass ratio of 60:30:10. Water was used as the solvent. After thoroughly grinding in a mortar for 30 min, the highly uniform slurry was cast onto a Cu thin foil and dried at room temperature. The dried electrode was then punched into small discs of 1 cm diameter. The active material areal loading is 1.5-2 mg cm<sup>-2</sup>. Before assembling in coin cells, the electrodes were further dried in a vacuum oven at 80 °C for 8 h.

## **Electrochemical measurement**

The electrochemical performance of the HHB-Cu NSs electrode was evaluated in 2032 coin cells with Li foil as the counter electrode. The electrolyte used here is  $1 \text{ M LiPF}_6$  dissolved in ethylene carbonate/ethyl methyl carbonate (1/1 volume ratio). Celgard 2400 functions as the separator. The coin cells were assembled in an Ar-filled glove box with oxygen and water controlled below 0.1 ppm. The battery performance was collected on the LAND CT2001A battery testing system under various current and voltage at room temperature. The current densities

and specific capacity were calculated based on the mass of active material. The cyclic voltammetry measurements were performed on the CHI650 electrochemical workstation.

## The theoretical capacity of HHB-Cu

The theoretical capacity of HHB-Cu was calculated based on the equation of C=96485× $n/[3600×(M_w/1000)]$ . M<sub>w</sub> is the equivalent molecular weight of unit cells of HHB-Cu. n is the number of electrons involved in the redox reaction of unit cells of HHB-Cu. The molecular weights of repeating benzene ring units (part A, Scheme S1) and CuO<sub>4</sub> units (part B) are 72.06 and 127.54. Considering the number of part A (6×1/3=2) and B (6×1/2=3) in the unit cells, the equivalent molecular weight of HHB-Cu is calculated as 72.06×2+127.54×3=527.74. According to a two-electron redox process (Scheme S3), n is determined as 6 (2×3). So the theoretical capacity of HHB-Cu is 305.3 mAh g<sup>-1</sup>.



Scheme S1. Chemical structure of HHB-Cu.

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**Scheme S2.** The one-electron electrochemical redox mechanism of HHB-Cu.

 $\left[ \left( \begin{array}{c} & 0 & \overline{0} \\ & 0 & \overline{0} \\ & 0 & 0 \end{array} \right)^0 \xrightarrow{+e^-} \left[ \left( \begin{array}{c} & \overline{0} & \overline{0} \\ & 0 & 0 \end{array} \right)^{-1} \xrightarrow{+e^-} \left[ \left( \begin{array}{c} & \overline{0} & \overline{0} \\ & 0 & 0 \end{array} \right)^{-2} \xrightarrow{+e^-} \left[ \left( \begin{array}{c} & \overline{0} & \overline{0} \\ & 0 & 0 \end{array} \right)^{-2} \right]^{-2} \right]^{-2}$ 

Scheme S3. Anticipated two-electron electrochemical redox mechanism of HHB-Cu.<sup>2</sup>



Figure S1. AFM images of HHB-Cu NSs.



**Figure S2.** (A) SEM image of HHB-Cu NSs with flake-like morphology and layered structure. (B) AFM image and the height profile along the marked white line of HHB-Cu NSs.



Figure S3. The EDX mapping of HHB-Cu NSs, showing uniform distribution of C, Cu, O, and Na.



Figure S4. EDX spectrum revealing the composition of HHB-Cu NSs with respect to C, Cu, O, and Na.



**Figure S5.** XPS analysis of HHB-Cu NSs. (A) Energy survey spectrum. High-resolution spectrum in the (B) C 1s region, (C) Cu 2p region and (D) O1s region, respectively.



**Figure S6.** (A) Normalized Cu *K*-edge XANES spectra of HHB-Cu NSs, Cu foil, and CuO. Insert: enlarged pre-edge region in Cu *K*-edge XANES spectra. (B) Fourier transformation EXAFS spectra at Cu *K*-edge of HHB-Cu NSs with Cu foil and CuO as contrast.



Figure S7. Raman spectra of THQ and HHB-Cu NSs.



**Figure S8.** (A) UV/vis absorption spectroscopy of HHB-Cu NSs on a quartz substrate. (B) Tauc plot of HHB-Cu NSs.



Figure S9.  $\mathsf{N}_2$  adsorption-desorption isotherms of HHB-Cu NSs and bulk HHB-Cu.



Figure S10. Synthetic scheme of bulk HHB-Cu without using SDS surfactant.



Figure S11. SEM image of bulk HHB-Cu.



Figure S12. (A) TEM image and SAED pattern of isolated HHB-Cu NSs. (B) HR-TEM image of HHB-Cu NSs and corresponding FFT image.



**Figure S13.** (A) TEM image of HHB-Ni NSs. Inset: SAED pattern of HHB-Ni NSs. (B) AC-HRTEM image of HHB-Ni NSs. Inset: corresponding FFT image. (C) SEM image of HHB-Ni NSs. (D) AFM image of HHB-Ni NSs.



Figure S14. (A) Synthetic scheme of HHTP-Cu NSs using a surfactant-assisted solution synthesis method. (B) AFM image and the height profile along the marked white line of HHTP-Cu NSs. (C) TEM image of HHTP-Cu NSs. Inset: SAED pattern of HHTP-Cu NSs. (D) AC-HRTEM image of HHTP-Cu NSs with the structure model overlaid. (E) Synthetic scheme of bulk HHTP-Cu powders without using surfactant. (F) SEM images of bulk HHTP-Cu powders. (G) Enlarged image of (F) clearly shows rod morphology of HHTP-Cu. Inset: Layer stacked model of HHTP-Cu rod. (H) AC-HRTEM image of isolated HHTP-Cu rod presents a highly ordered linear arrangement with a lattice distance of 1.8 nm. Inset: SAED pattern of HHTP-Cu rod. The nearest reflections were found at 0.56 nm<sup>-1</sup> corresponding to 1.80 nm in real space, matching well with the lattice structure of HHTP-Cu. The diffusive arcs fit nicely into a ring with a radius of 3.0 nm<sup>-1</sup>, i.e., 0.33 nm, which is in agreement with  $\pi$ - $\pi$  stacking distance.



Figure S15. *I-V* curve of HHB-Cu NSs.



**Figure S16.** Electrical conductivity ( $\sigma$ ) of HHB-Cu NSs as a function of temperature ranging from 243 to 310 K. *I-V* curves were recorded to make sure the measurement was performed in the Ohmic region.



**Figure S17.** Magnetic field dependence of the magnetoresistance by measuring the changes of the electrical resistance in an applied field (-4 - 4 T) at 300 K.



**Figure S18.** The measured electrical conductivity of the bulk HHB-Cu through the *van der Pauw* method in its pellet form (~ 0.25 mm in the thickness) from 246 to 310 K



**Figure S19.** Optimization of potential ranges of HHB-Cu NSs electrodes. (A-D) CV curves of HHB-Cu NSs electrodes at 1.3-2.6 V, 1.0-2.6 V, 1.0-3.0 V and 0.1-3.0 V at 1 mV s<sup>-1</sup>. During the optimization of the potential range of HHB-Cu NSs electrode via CV measurements, only one pair of symmetric redox peaks were observed at 1.3-2.6 V (vs Li/Li<sup>+</sup>). At potentials lower than 1.3 V or higher than 2.6 V, the HHB-Cu NSs electrode represents irregular and asymmetric redox bands, indicating poor redox reversibility at these ranges. Therefore, the potential range of 1.3-2.6 V is set as the optimal potential window for the HHB-Cu NSs electrode.



Figure S20. Charge-discharge curves of HHB-Cu NSs and bulk HHB-Cu at 0.1 A g<sup>-1</sup>.



Figure S21. Cycling performance of HHB-Cu NSs at 0.1 A g<sup>-1</sup>.



**Figure S22.** (A) Charge-discharge curves of HHB-Cu NSs, and (B) cycling performance at 0.1 A g<sup>-1</sup> within the potential range of 1.3-3.3 V. (C) Charge-discharge curves. (D) Cycling performance of HHB-Cu NSs electrodes at 1.3-4.0 V (vs Li/Li<sup>+</sup>). The current density is 0.1 A g<sup>-1</sup>. (E) CV curves at a scan rate of 0.5 mV s<sup>-1</sup>.



**Figure S23.** (A) Charge-discharge curves of bulk HHB-Cu at specific current densities. (B) The rate capability of bulk HHB-Cu at different current densities.



Figure S24. CV profiles collected at 1 mV s<sup>-1</sup> for HHB-Cu and HHB-Ni NSs.



Figure S25. Ex-situ FT-IR spectrum spectra recorded for HHB-Cu NSs.

	MOFs NSs	Thickness	Lateral size	Yield	Ref.
		(nm)			
3D MOFs	MON 1	3.0 ± 0.5	0.36 μm²	7 %	3
	MON 2	(1.5-3.0) ± 0.5	0.5 μm²	5 %	-
	NH <sub>2</sub> -MIL-53(AI)	35-45	0.14-0.4 μm	-	4
	Zn <sub>2</sub> (bim) <sub>4</sub>	~1.12	~1.5 μm	15 %	1a
2D MOFs	Cu(HBTC)-1	~ 6	0.7-2 μm	-	5
	Zn-TCPP	7.6 ± 2.6	1.2 ± 0.4 μm		
	Cu-TCPP	4.5 ± 1.2	1.0 ± 0.2 μm		1h
	Cb-TCPP	8.7 ± 2.7	1.2 ± 0.3 μm	-	10
	Co-TCPP	$18.8 \pm 6.4$	0.7 ± 0.1 μm		
	Co-TCPP(Fe)	5.6 ± 1.8			
	Cu-TCPP(Fe)	3.7 ± 1.3	hundreds of nanometers	-	6
	Zn-TCPP(Fe)	$4.8 \pm 1.6$	to few micrometers		
	Cu-TCPP(Co)	3.5 ± 1.2		-	7
	NiCo-UMOFNs	~3.1	-	-	8
	NiFe-MOF	~3.5	>0.1 µm	-	9
	PtSA-MNSs	2.4 ± 0.9	up to several	_	10
			micrometers		
	MOF-74	~2.6	-	-	11
2D <i>c</i> -MOFs	HHB-Cu NSs	4.2 ± 1.1	0.30-0.65 μm²	87.5 %	This
	HHB-Ni NSs	4.5 ± 1.4	0.25-0.56 μm²	85.5 %	work
	HHTP-Cu NSs	5.1 ± 2.6	0.002-0.02 μm²	80.5 %	WOIN

Table S1. Comparison of MOF NSs by solution synthesis.

MOFs	Specific capacity (mA h g <sup>-1</sup> )	Stability	Testing Conditions	Electrolyte	Ref.
Cu(2,7-AQDC)	147	71 %, 50 cycles	1 mV s <sup>-1</sup>	1 M LiPF <sub>6</sub> , EC/DEC	12
NiDI (HIB-Ni)	155	80 %, 300 cycles	0.01 A g <sup>-1</sup>	1 M LiPF <sub>6</sub> EC/DEC	13
MIL-53	75	-	0.025 C	1 M LiPF <sub>6</sub> EC/DMC	14
K <sub>2.5</sub> [(VO) <sub>2</sub> (HPO <sub>4</sub> ) <sub>1.5</sub> (PO <sub>4</sub> ) <sub>0.5</sub> (C <sub>2</sub> O <sub>4</sub> )]	66	87.9%, 60 cycles	0.04 A g <sup>-1</sup>	1 M LiPF <sub>6</sub> EC/DEC	15
Cu-THQ	387	85%, 100 cycles	0.5 A g <sup>-1</sup>	1 M LiPF <sub>6</sub> EC/DEC	16
bulk HHB-Cu	40	65 %, 50 cycles	0.1 A g <sup>-1</sup>	1 M LiPF <sub>6</sub> EC/EMC	This work
HHB-Cu NSs	153	90 %, 1000 cycles	1 A g <sup>-1</sup>	1 M LiPF <sub>6</sub> EC/EMC	This work

Table S2. Comparison of performance of MOFs for Li-ion batteries.

\* DME, dimethoxyethane; DEGDME, diethylene glycol dimethyl ether; EC, ethylene carbonate; DMC, dimethyl carbonate; DEC, di-ethyl carbonates; EMC, ethyl-methyl carbonates.

The cycling stability of HHB-Cu NSs was quite sensitive to the reaction depth of redox centers, which can be well controlled by adopting different voltage windows. Both  $CuO_4$  unit and Cu center are redox active in HHB-Cu NSs.  $CuO_4$  unit can deliver two electrons maximum (two Li<sup>+</sup> uptake/release), while Cu center can transfer one electron with one anion uptake/release. It is found that both multi-electron processes at 1.3-4.0 V (>2 electrons for  $CuO_4^0/CuO_4^{2-}$  and  $Cu^{2+}/Cu^+$ ) and 1.3-3.3 V (2 electrons for  $CuO_4^0/CuO_4^{2-}$ ) showed fast capacity decay, which can be assigned to structure deterioration of HHB-Cu NSs due to the large strain generated during Li<sup>+</sup>/anion (PF<sub>6</sub><sup>-</sup>) uptake. In contrast, the one electron process ( $CuO_4^{-}/CuO_4^{2-}$ ) performed at 1.3-2.6 V exhibited the best cycling performance, which benefits from the least strain caused to HHB-Cu framework. Therefore, under the optimum reaction depth, our HHB-Cu NSs demonstrate the excellent reversibility despite a low porosity.

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