Supporting Information

MOF-derived Carbon-Coated Cuprous Phosphide Nanosheets for Electrocatalytic Glucose Oxidation

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2. Experimental part

2.1. Materials

All chemicals are used as is, without purification. Copper nitrate hydrate (Cu(NO₃)₂·3H2O, AR, 99.5%), 1, 3, 5-benzenetricarboxylic acid (H₃BTC, AR, 99%). Carbon cloth (CC, WIS1009, 1 cm²), acetylene carbon black, distilled water, ethanol (EtOH, moisture content $\leq 0.3\%$ from Aladdin), uric acid (UA, 99%), glucose (AR), D-fructose (99%), Sucrose (AR, 98%).

2.2. Preparation of Cu-BTC

Take 0.59 g Cu(NO₃)₂· $3H_2O$ and 0.34 g 1, 3, 5-benzenetricarboxylic acid and dissolve it in 5 mL of deionized water, ethanol, DMF, transfer the above mixed solution to the reaction flask and sonicate for 5 minutes to form uniform blue solution. And react in an oven at 85°C for 20 hours. After cooling to room temperature, wash with absolute ethanol and centrifuge several times. Finally, put the Cu-BTC particles obtained by centrifugation into an oven at 85°C to dry for 5 hours. The yield of crystalline Cu-BTC is calculated to be ~60% based on the added organic ligands.

2.3. Preparation of Cu-BTC-C and CuBTC-CP

The prepared Cu-BTC blue powders are heated to 800 °C in an

argon atmosphere for carbonization, and the heating rate is 5 °C min⁻¹ to obtain Cu-BTC-C. The Cu-BTC-C produced by the carbonization of the precursor Cu-BTC is about 40%. Then weigh a certain amount of sodium hypophosphite and Cu-BTC-C powder (mass ratio = 10:1) and put them into the two ends of the sleeve. Sodium hypophosphite is placed upstream of the tube furnace. Through a typical phosphating reaction, Cu-BTC-C powder is phosphatized at 300 °C for 2 hours under the protection of argon at a heating rate of 2.5 °C min⁻¹ to obtain black Cu-BTC-CP powder. The yield of phosphide Cu-BTC-CP produced by phosphating of Cu-BTC-C is about 50%.

2.4. Physical characteristics

The SEM image was passed through the JSM-6700F field emission scanning electron microscope (SEM). The samples are made on clean silicon wafers. Some samples have poor conductivity, so spray gold for 30 seconds. The samples of (HR-TEM) images and energy dispersive X-ray spectroscopy (EDS) images are ultrasonically dispersed in ethanol and spotted on a microgrid. After drying under an infrared lamp, it was observed with a 200 kV JEOLJEM-2100F high-resolution transmission electron microscope. The N₂ adsorption test is to obtain adsorption isotherms, BET surface area and pore size distribution (PSD) in the Micromeritics ASAP 2020 analyzer. Simultaneous thermogravimetric analysis (TGA) under flowing N_2 .

2.5.Electrochemical analysis

All electrochemical tests are performed in a universal 3-electrode unit, using a CHI-760E workstation (Shanghai Chenhua, 25 °C). The electrochemical test and electrocatalytic performance of the prepared material were measured at room temperature. In all experiments, NaOH (0.1 M) was used as the electrolyte. Electrochemical impedance spectroscopy (EIS) is performed at a frequency of 0.01 to 100 kHz. In addition, it is necessary to perform chronoamperometry at 0.58 V (vs Ag/AgCl) for 20 minutes to obtain a stable electrochemical state before detection. For the current response test, glucose is added to the electrolyte under continuous stirring to test the current response.

Items	Cu-BTC	
empirical formula	C ₁₄₄ H ₄₈ O ₁₂₀ Cu ₂₄	
formula weight	5266.70	
crystal system	Cubic	
space group	Fm-3m	
<i>a</i> (Å)	26.536(5)Å	
<i>b</i> (Å)	26.536(5)Å	
c (Å)	26.536(5)Å	
α (deg)	90°	
β (deg)	90°	
γ (deg)	90°	
V (Å ³)	18686(10)Å ³	
Z	2	
$\rho_{\text{calc}} (\text{g cm}^{-3})$	0.936 Mg/m ³	
μ (mm ⁻¹)	1.565 mm ⁻¹	
N _{ref}	41847	
F(000)	5184	
Goodness-of-fit on F ²	1.077	
R1, wR ₂ [I > $2\sigma(I)$]	R1=0.0395, wR2=0.1229	
R1, wR ₂ (all data)	R1=0.0439, wR2=0.1302	

Table S1 Single crystal XRD data of Cu-BTC.

The above single crystal data can be further indexed in J. Phys. Chem. A, 2011, 115, 11519-11524; Journal of Power Sources, 2021, 499, 229947, Journal of Colloid and Interface Science, 2022, 606, 1833-1841.

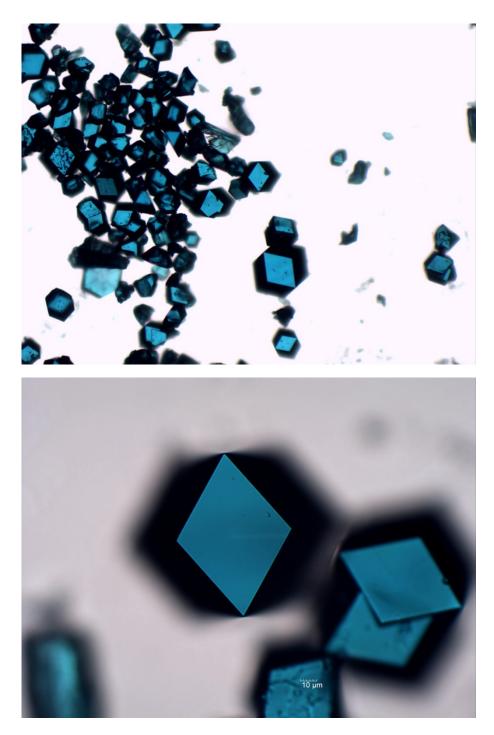


Figure S1. Optical images of Cu-BTC

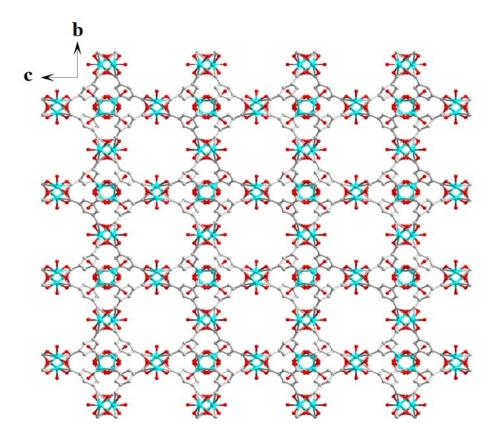


Figure S2. The frame viewed along *a* axis.

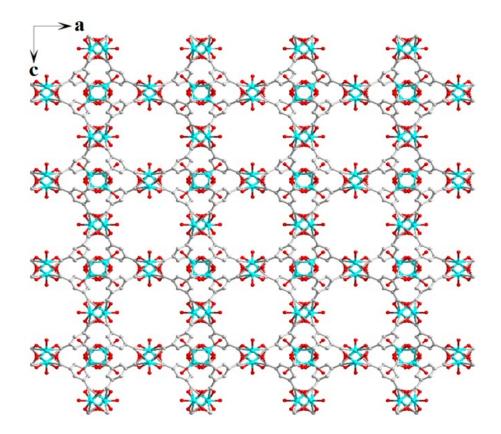


Figure S3. The frame viewed along *b* axis.

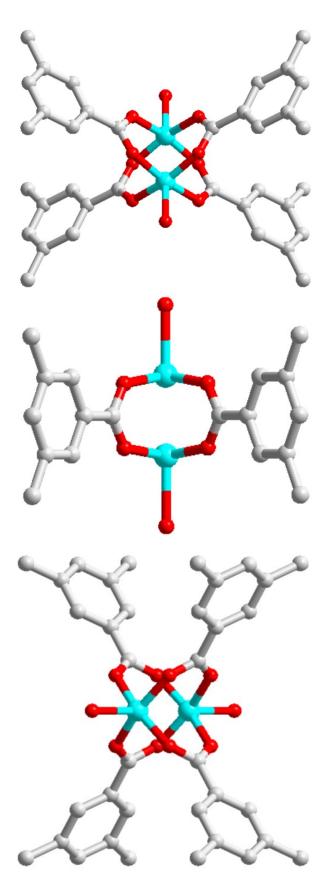


Figure S4. The coordination environment of inorganic SBU.

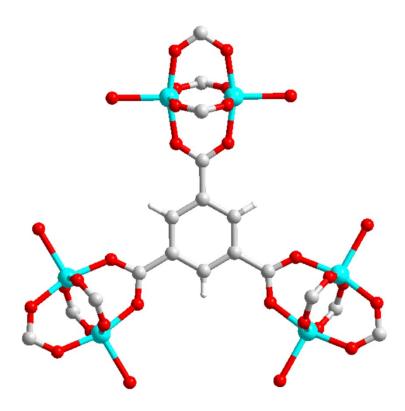


Figure S5. The coordination environment of organic BTC³⁻ linker.

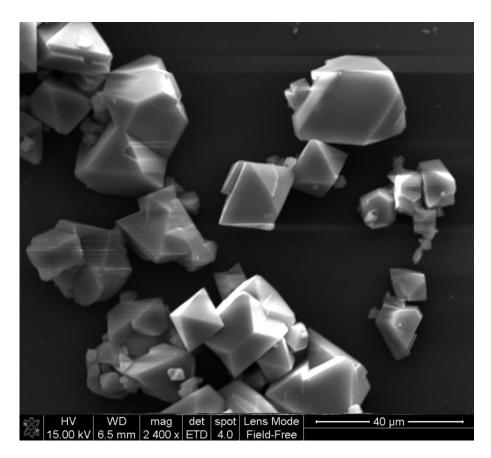


Figure S6. SEM image of Cu-BTC.

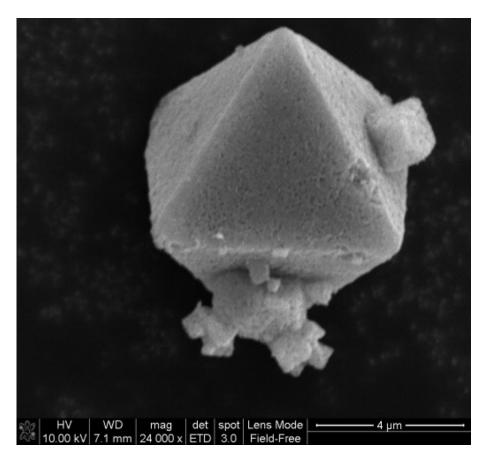


Figure S7. SEM image of Cu-BTC-C.

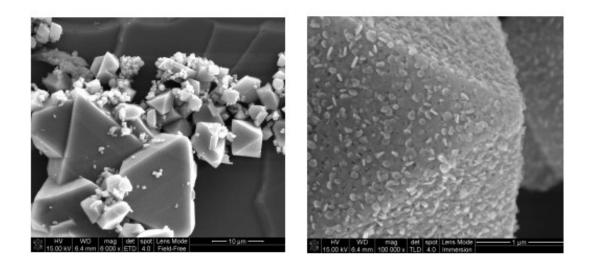


Figure S8. SEM image of Cu-BTC-CP.

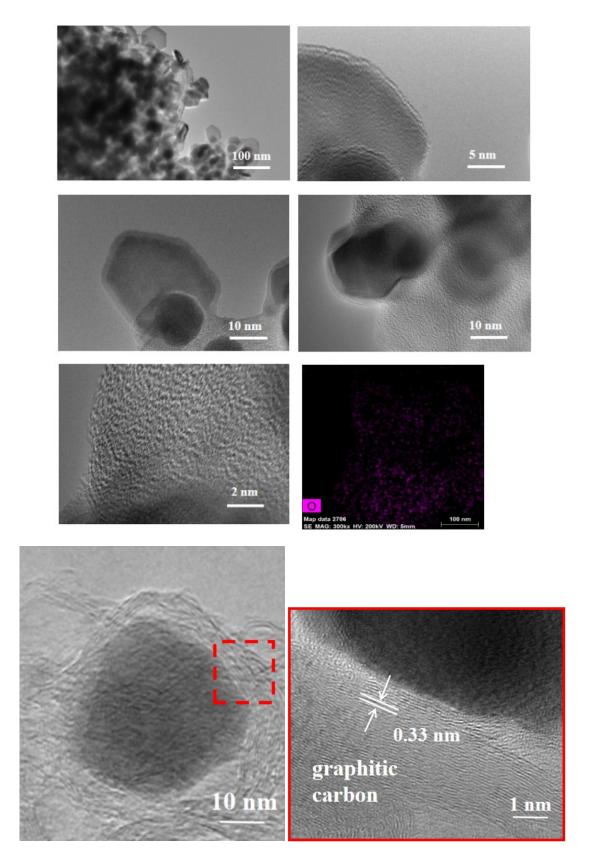


Figure S9. TEM and O mapping images of Cu-BTC-CP.

items	Cu-BTC-C	Cu-BTC-CP
Specific surface area	51.0894	50.9146
(m^2/g)		
Micropore volume	0.0155	0.0079
(m^{3}/g)		
Total pore volume	0.3123	0.2438
(m^{3}/g)		
Average hole diameter	24.4513	19.1536
(nm)		

Table S2 Porous characteristics of Cu-BTC-C and Cu-BTC-CP

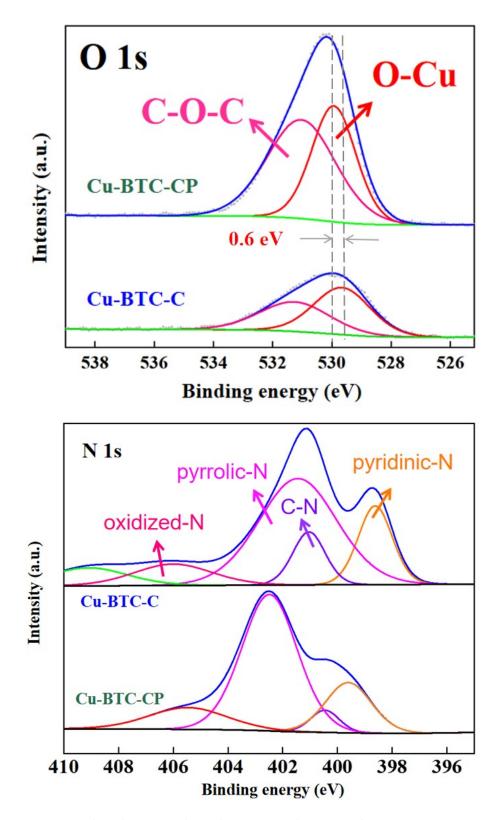


Figure S10. The deconvoluted O 1s and N 1s of Cu-BTC-CP.

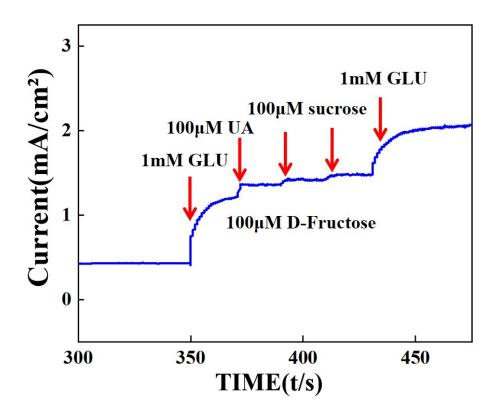


Figure S11. The Amperometric responses with different interferents for Cu-BTC-C.

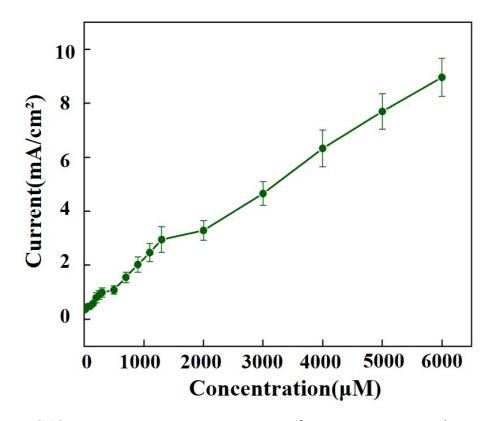


Figure S12. Average current response of Cu-BTC-CP to glucose.

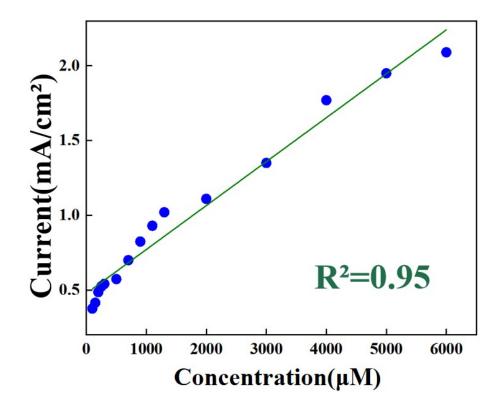


Figure S13. Calibration curve of Cu-BTC-C.