Supplementary Information

{Cu₂SiW₁₂O₄₀}@HKUST-1 synthesized by the one-step solution method with
efficient bifunctional activity for supercapacitor and oxygen evolution reaction

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POMOFs constructed of polyoxometalates (POMs) and metal-organic frameworks (MOF) are very desirable for high-efficiency supercapacitor performance and electrocatalytic water oxygen evolution reaction, but it is still challenging. Herein, successfully synthesized the {Cu₂SiW₁₂O₄₀}@HKUST-1 material through a one-step solution method. The synergy between {Cu₂SiW₁₂O₄₀} and HKUST-1 can promote mass/charge transfer and the adsorption/desorption of intermediates on {Cu₂SiW₁₂O₄₀}@HKUST-1. In the three-electrode system, {Cu₂SiW₁₂O₄₀}@HKUST-1 electrode material has a specific capacitance of 5096.5 F g⁻¹ when the current density is 1 A g⁻¹, after 6000 cycles, the retention rate is 92%, which is much higher than reported in the literature. The symmetrical electrode is assembled with foamed nickel as the current collector, in the 1.0 V voltage window, the power density is 510.81 W kg⁻¹, and the energy density is 15.31 Wh kg⁻¹. In 1.0 M KOH aqueous solution, when the scanning speed is 5 mV s⁻¹, the overpotential of electrocatalytic water oxygen evolution reaction (OER) is 340 mV (without iR compensation). Especially at high current density, {Cu₂SiW₁₂O₄₀}@HKUST-1 shows better performance than commercial RuO₂. Further, it has excellent catalytic durability. It provides a basis for the further application of POMOFs.
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1. Experimental Procedures

1.1 Material characterization method

In the Bruker Ver Tex 80 Fourier Transform Infrared Spectrometer (FTIR spectrometer) using KBr particles, the absorption spectrum in the range of 400-4000 cm\(^{-1}\) was recorded. The powder X-ray diffraction (XRD) pattern using Cu-Kα irradiation (λ=1.54056Å) was measured on a Bruker D8 Advance in the 2θ range of 20-80°. Scanning electron microscopy (SEM) images are characterized by Hitachi SU70 SEM coupling with an energy dispersive X-ray (EDX) detector. Transmission electron microscope (TEM) images were obtained from Tecnai G2 F20. X-ray photoelectron spectroscopy (XPS) analysis was performed on the AXIS ULTRA DLD electronic spectrometer by the Mg Ka (1253.6eV) achromatic X-ray source. Brunauer Emmett-Teller (BET) surface area was captured by N\(_2\) adsorption measurement using Nova 2000E at 77.3 K. Brunauer Emmett-Teller (BET) surface area was captured by N\(_2\) adsorption measurement using Nova 2000E at 77.3 K.

1.2 Electrode preparation and electrochemical characterization for SCs

Preparation of glassy carbon electrode(GCE): The composite electrode is made by mixing the active material and conductive carbon black with a mass ratio of 1:4. Weigh 5mg \{Cu\(_2\)SiW\(_{12}\)O\(_{40}\)\}@HKUST-1 and add certain naphthol as dispersant, disperse it ultrasonically for 40min to get a uniform turbid liquid. We used a pipette to remove 5μL drops on a pretreated glassy carbon electrode with a diameter of 3mm. After drying at room temperature, μL of 0.5% Nafion solution was dropped on the surface of the glassy carbon electrode, and dried in vacuum as the working electrode.

Preparation of nickel foam(NF) electrode: The NF was sonicated first with acetone for 30min and subsequently with 3 M HCl for 30 min. Thereafter, it was washed with distilled water and ethanol and then placed in a vacuum oven. Grind and mix the active material and acetylene black in a ratio of 1:1, add a certain amount of ethanol to dissolve ultrasonically, cut the nickel foam into 1×3cm\(^2\) size, and apply 3mg to the nickel foam. Using a tablet press, the nickel foam was pressed into a sheet at 2MPa for 8 seconds.

Assembly of symmetrical button batteries: Cut two foam nickel sheets with a diameter of 1cm, apply the material to the foam nickel and press with a tablet machine. Put a piece into the positive electrode shell of the button electrode, then add a few drops of Na\(_2\)SO\(_4\) (1M) solution. Then add a diaphragm. Put another piece of nickel foam on top, then add one or two drops of Na\(_2\)SO\(_4\) (1M). Put the metal plate shrapnel on it, and finally put the negative electrode.

Preparation of electrocatalytic water electrode: Grind the active material and acetylene black at a ratio of 2:1, weigh 5mg \{Cu\(_2\)SiW\(_{12}\)O\(_{40}\)\}@HKUST-1 and add ethanol as a dispersant to it, and ultrasonically form a uniform solution. We use a pipette to drop 5μL on the pretreated glassy carbon electrode with a diameter of 3mm. At room temperature, 7μL of 0.5% Nafion solution was dropped on the surface of the glassy carbon electrode and dried in a vacuum as a working electrode.

\[ i = av^b \quad \text{Equation(S1)} \]

Where \(i\) is the peak current density, \(v\) is scan rate, and \(a\) and \(b\) are coefficients.

\[ Q = Q_c + Q_d \quad \text{Equation(S2)} \]

The surface treatment controls charge (Qc), the diffusion control charge (Qd)
\[ Q = Q_c + k v^{-1/2} \]  
Equation(S3)

Where \( k \) = variable parameter. \( Q \) = stored charge, \( v \) = scan rate.

\[ C = \frac{2i_m \int V dt}{V^2 |V_f/V_i|} \]  
Equation(S4)

\( C(F\,g^{-1}) \) is the specific capacitance of the constant current charge and discharge curve. \( i_m(A\,g^{-1}) \) represents the current density, \( J V dt \) is the current in the integration region, \( V_i \) and \( V_f \) represent the initial and final values of the potential.

\[ C_s = I \cdot t / (m \cdot \Delta V) \]  
Equation(S5)

Where \( C_s \) represents the specific capacitance, \( I \) is the charge and discharge current(A), \( t \) is the discharge time(s), \( m(g) \) represents the load mass of the active material, and \( \Delta V \) is the voltage difference (V).

2. Results and Discussion

2.1 The characterization diagrams

![Figure S1](image-url) (a,f) SEM images; (b-e) EDX of Si, W, Cu and O of Cu₂SiW₁₂O₄₀.
Figure S2. EDS of Cu$_2$SiW$_{12}$O$_{40}$.

Figure S3. (a,c) SEM images; (b) EDS of HKUST-1. (d-f) EDX of C, Cu and O of HKUST-1.

Figure S4. EDS of {Cu$_2$SiW$_{12}$O$_{40}$}@HKUST-1.
Figure S5. XPS spectra of Cu$_2$SiW$_{12}$O$_{40}$: (a) Cu, (b) O, (c) Si and (d) W.

Figure S6. XPS spectra of HKUST-1: (a) C, (b) Cu and (c) O.

2.2 Supercapacitor test chart of compounds

Figure S7. The CVs at different scan rates: (a) [Cu$_2$SiW$_{12}$O$_{40}$]@HKUST-1, (b) Cu$_2$SiW$_{12}$O$_{40}$, (c) HKUST-1.
Figure S8. Plot of the total charge stored (q) vs. the reciprocal of the square root of the scan rate.

Figure S9. Normalized contribution ratio of the capacitive ($Q_c$) and diffusion-controlled ($Q_d$) charge storage capacities at lower scan rates of (a) HKUST-1, (b) Cu$_2$SiW$_{12}$O$_{40}$.

Figure S10. The GCD of (a)[Cu$_2$SiW$_{12}$O$_{40}$]@HKUST-1, (b)HKUST-1,(c)Cu$_2$SiW$_{12}$O$_{40}$ at different current densities.

2.3 Electrocatalytic water oxygen production diagram of compounds
Figure S11. CVs of (a) HKUST-1; (b) Cu$_2$SiW$_{12}$O$_{40}$ with different rates from 10 to 100 mV s$^{-1}$. Inset: The capacitive current at 0.34V as a function of the scan rate for (a) HKUST-1. (b) Cu$_2$SiW$_{12}$O$_{40}$.

![Graph showing CVs and capacitive current](image)

Figure S12. Time-dependent current density curve of HKUST-1 and Cu$_2$SiW$_{12}$O$_{40}$.

![Graph showing time-dependent current density](image)

Figure S13. (a) SEM image of the {Cu$_2$SiW$_{12}$O$_{40}$}@HKUST-1 catalyst after OER stability test, (b-f) EDX of C, Cu, O, Si and W.

![SEM image and EDX](image)

Figure S14. The W4f XPS spectra of {Cu$_2$SiW$_{12}$O$_{40}$}@HKUST-1 after OER tests.

![W4f XPS spectra](image)
Figure S15. The W4f XPS spectra of Cu$_2$SiW$_{12}$O$_{40}$ after OER tests.

2.4 Comparison of the properties of the Keggin-based materials with published supercapacitors

Table S1 Comparison of the properties of the Keggin-based materials with published supercapacitors

| Compound                        | The capacitances | The cyclic stability | collector                              | Ref   |
|---------------------------------|------------------|----------------------|----------------------------------------|-------|
| 1 PAni/H$_3$PMo$_{12}$O$_{40}$  | 120 F g$^{-1}$   | 70% (1000 cycles)   | Rigid graphite plate                   | [1]   |
| 2 MWCNT/C$_x$PMo$_{12}$O$_{40}$ | 285 F g$^{-1}$ (0.2 A g$^{-1}$) | ----                  | ---                                    | [2]   |
| 3 H$_3$PMo$_{12}$O$_{40}$/MWCNT | 38 F g$^{-1}$ (1 A g$^{-1}$) | -----------          | porous glassy fibrous paper            | [3]   |
| 4 [BMIM]$_4$SiW$_{12}$O$_{40}$  | 172 F g$^{-1}$ (20 mV s$^{-1}$) | 89% (1100 cycles)   | glassy carbon                          | [4]   |
| 5 AC/PMo$_{12}$O$_{40}$         | 140 F g$^{-1}$ (1 A g$^{-1}$) | 91% (8000 cycles)   | glassy carbon                          | [5]   |
| 6 AC/PW$_{12}$O$_{40}$          | 254 F g$^{-1}$ (10 mV s$^{-1}$) | 35% (30000 cycles) | Graphite rods                          | [6]   |
| 7 RGO/PIL/PMo$_{12}$O$_{40}$    | 408 F g$^{-1}$   | 98% (2000 cycles)   | stainless steel foil                   | [7]   |
| 8 rGO/PMo$_{12}$O$_{40}$        | 276 F g$^{-1}$   | 96% (10000 cycles)  | Graphite rods                          | [8]   |
|   | Material                         | Specific Capacity | Cyclic Stability  | Remarks                                      | Reference |
|---|----------------------------------|-------------------|-------------------|----------------------------------------------|-----------|
| 9 | SWCNT/TBA/PMo12V2O40            | 444 F g⁻¹         | 95% (6500 cycles) | glassy carbon rods and graphite papers       | [9]       |
| 10| rGO/ PMo12O40                   | 51.2 F g⁻¹        | 95% (5000 cycles) | commercial flexible carbon cloth              | [10]      |
| 11| AC/PMo12O40                     | 223 F g⁻¹         | 100% (10000 cycles)| Ti foils                                     | [11]      |
| 12| PMo12−XWxO40⁻                    | 140 F g⁻¹ (10 A g⁻¹) | 94.6% (1700cycles) | glassy carbon                                | [12]      |
| 13| [Ag₅(brtmb)₄][VW₁₀V₂O₄₀]        | 206 F g⁻¹ (110 A g⁻¹) | 81.7% (1000 cycles) | glassy carbon                                | [13]      |
| 14| Pinecone AC/PMo12O40           | 361 F g⁻¹         | -----             | titanium foil                                | [14]      |
| 15| AC/PMo12O40                     | 293 F g⁻¹         | -----             | C250 carbon monoliths                        | [15]      |
| 16| [Ag₅(brtmb)₄][VW₁₀V₂O₄₀]        | 206 F g⁻¹ (110 A g⁻¹) | 81.7% (1000 cycles) | glassy carbon                                | [16]      |
| 17| rGO-PMo₁₂                        | 278 mF cm⁻²       | 89% (5000 cycles) | Carbon cloth                                 | [17]      |
| 18| rGO-PMo₁₂∥rGO-PW₁₂              | 110 F cm⁻² (2 mA cm⁻²) | 95% (2000 cycles) | carbon cloth                                 | [17]      |
| 19| [Cu‘(btx)₄][SiW₁₂O₄₀]          | 110.3 F g⁻¹ (3.0 A g⁻¹) | 87% (1000 cycles) | glassy carbon                                | [18]      |
| 20| NENU-5/PPy                      | 779.8 F g⁻¹ (10 mV s⁻¹) | -----             | carbon cloth                                 | [19]      |
| 21| [H(C₁₀H₁₀N₂)Cu₂][PMo₁₂O₄₀]     | 287 F g⁻¹ (1 A g⁻¹) | 81.5% (500 cycles) | glassy carbon                                | [20]      |
| 22| [H(C₁₀H₁₀N₂)Cu₂][PW₁₂O₄₀]      | 153.43 F g⁻¹ (1 A g⁻¹) | 18.2% (500 cycles) | glassy carbon                                | [20]      |
| 23| NiPW₁₂NP/FrGO                   | 437.6 F g⁻¹ (4 A g⁻¹) | 94.3% (5000 cycles) | carbon paper                                 | [21]      |
| 24| mPPy@GO-PMo₁₂                   | 115 mF cm⁻² (1 mV s⁻¹) | 80% (2000 cycles) | glassy carbon                                | [22]      |
| 25| [Ag₅(C₂H₂N₂)₄][H₂SiMo₁₂O₄₀]@15%GO | 230.2 F g⁻¹ (0.5 A g⁻¹) | 92.7% (1000 cycles) | glassy carbon                                | [23]      |
| 26| [CuH₂(C₂H₂N₂)₄][PMo₁₂O₄₀]−[(C₂H₂N)(H₂O)₂] | 249 F g⁻¹ (3 A g⁻¹) | 93.5% (1000 cycles) | glassy carbon                                | [24]      |
| 27| [CuV₁₂(C₂H₂N₂)₄](PMoV₉Mo³O₃₉) | 154.5 F g⁻¹ (3 A g⁻¹) | 91.1% (1000 cycles) | glassy carbon                                | [24]      |
| 28 | [Cu₄H₂(bttx)₃(PMo₁₂O₄₀)₂]·2H₂O | 237 F g⁻¹ (2 A g⁻¹) | 92.5% (1000 cycles) | glassy carbon | [25] |
| 29 | [Cu₄H₂(bttx)₃(PW₁₂O₄₀)₂]·2H₂O | 100 F g⁻¹ (2A g⁻¹) | 90% (1000 cycles) | glassy carbon | [25] |
| 30 | [Cu₄Cu₄(H₂O)₃(bttx)₃(PW₆₀₁₀W₄O₄₀)]·2H₂O | 82.1 F g⁻¹ (2A g⁻¹) | 100% (1000 cycles) | glassy carbon | [25] |
| 31 | [Cu₄Cu₄(bttx)₃(PW₆₀₁₀W₄O₄₀)]·2H₂O | 76.4 F g⁻¹ (2A g⁻¹) | 100% (1000 cycles) | glassy carbon | [25] |
| 32 | [Cu₄Cu₄(bttx)₃(SiMo₃O₁₅M₀O₄₀)]·4H₂O | 138.4 F g⁻¹ (2A g⁻¹) | 97% (1000 cycles) | glassy carbon | [25] |
| 33 | [Ag₃(pz)₃](BW₂O₄₀) | 1058 F g⁻¹ (2.16 A g⁻¹) | 90.3% (1000 cycles) | glassy carbon | [26] |
| 34 | [(Ag₃(pz)₃)(SiW₁₂O₄₀)](OH)·H₂O | 986 F g⁻¹ (2.16A g⁻¹) | 94.5% (1000 cycles) | glassy carbon | [26] |
| 35 | (Hpyr)[(Ag(pz)₂)(PMo₁₂O₄₀)] | 1611 F g⁻¹ (2.16A g⁻¹) | 84.8% (1000 cycles) | glassy carbon | [26] |
| 36 | [Cu₄(pz)₂(phen)₁₂][Cu(phen)]₂ | 825 F g⁻¹ (2.4 A g⁻¹) | 91.4% (3000 cycles) | glassy carbon | [27] |
| 37 | [Ag₃(C₂H₂N₄)₃][H₃SiMo₁₂O₄₀]@15%GO-based electrode | 230.2 F g⁻¹ (0.5A g⁻¹) | 92.7% (1000 cycles) | glassy carbon | [28] |
| 38 | [Ag(bpy)][(Ag(Hbpy))₃(AlW₁₂O₄₀)]·H₂O | 478.41 F g⁻¹ (1 A g⁻¹) | 95.71% (5000 cycles) | carbon cloth | [29] |
| 39 | [H₂en][(Cu(bpy)₃)(AlW₁₂O₄₀)]·3H₂O | 625.99 F g⁻¹ (1 A g⁻¹) | 97.62% (5000 cycles) | carbon cloth | [29] |
| 40 | [Ni(itmb)₄](HPMo₁₂O₄₀)·2H₂O | 477.9 F g⁻¹ (15 A g⁻¹) | ----- | glassy carbon | [30] |
| 41 | [Zn(itmb)₃](H₂O)(HPMo₁₂O₄₀)·4H₂O | 890.2 F g⁻¹ (15 A g⁻¹) | ----- | glassy carbon | [30] |
| 42 | [Mn₃(BTC)₄]₃(H₂O)₆ | 211.0 F g⁻¹ (1 A g⁻¹) | 96.0% (5000 cycles) | nickel foam | [31] |
| 43 | PPD-PMo₁₂@rGO | 790 F g⁻¹ (1 mV s⁻¹) | 90.5% (30 000 cycles) | carbon paper | [32] |
| 44 | PPD-PMo₁₂ | 550 F g⁻¹ (1 mV s⁻¹) | ----- | Carbon paper | [32] |
| 45 | PMo₁₂@GO | 305 F g⁻¹ (1 mV s⁻¹) | ----- | carbon paper | [32] |
| 46 | (imi)₂[(Ag₃(tpb)₃)(H₂O)₂AsW₁₂O₄₀]·6H₂O | 929.7 F g⁻¹ (3.6 A g⁻¹) | 89.6% (5000 cycles) | glassy carbon | [33] |
| 47 | [(Ag₃(bpy)Cl)₃]AsW₆W₄O₄₀ | 986.1 F g⁻¹ (3.6 A g⁻¹) | 91.4% (5000 cycles) | glassy carbon | [33] |
|  | AC/TEAPW$_{12}$ | 82 F g$^{-1}$ (0.5 A g$^{-1}$) | 93% (10000 cycles) | aluminum foil | [34] |
|---|---|---|---|---|---|
| 49 | [HPMo$_{12}$O$_{40}$]@[Cu$_4$($μ_2$-OH)$_2$(C$_6$H$_5$PO$_3$)$_2$(bimb)$_4$] | 267.0 F g$^{-1}$ (5 A g$^{-1}$) | 95.1% (1000 cycles) | glassy carbon | [35] |
| 50 | PMo$_{10}$V$_2$@ZIF-67 | 475 F g$^{-1}$ (2 A g$^{-1}$) | 106.41% (5000 cycles) | Ni foam | [36] |
| 51 | {Cu$_2$SiW$_{12}$O$_{40}$}@HKUST-1 | 5096.5 F g$^{-1}$ (1 A g$^{-1}$) | 92% (6000 cycles) | glassy carbon | This work |
| 52 | H$_4$SiW$_{12}$O$_{40}$ | 604.6 F g$^{-1}$ (1 A g$^{-1}$) | 82% (6000 cycles) | glassy carbon | This work |
| 53 | {Cu$_2$SiW$_{12}$O$_{40}$}@HKUST-1 | 403.7 F g$^{-1}$ (1 A g$^{-1}$) | 91.7% (6000 cycles) | nickel foam | This work |

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