# Electronic Supplementary Information

## Bubble-assisted Fabrication of Hollow $CoMoO_4$ Spheres for Energy Storage

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**Experimental Section**

Synthesis of hollow $CoMoO_4$ spheres.

Mixture of 0.525 g Mo powder, 0.944 g $Co(NO_3)_2 \bullet 6H_2O$ and 0.197 g $CO(NH_2)_2$ was added into a 25-mL Teflon-lined stainless steel autoclave, which is filled with 7 mL 30% $H_2O_2$ solution. Afterwards, the autoclave was screwed at once. Then, hollow $CoMoO_4$ spheres suspension was obtained within 60 s. The suspension was rinsed through centrifugation and resuspension by deionized water for three times. The raw sample was obtained after drying in 60 °C for 12 h in vacuum oven. As for the synthesis of $MnMoO_4$, $MnCl_2 \cdot 4H_2O$ is replaced with all the other conditions the same.

Characterization.

X-ray diffraction (XRD) patterns were determined through an X-ray diffractometer (Empyrean, PANalytical) with a wavelength of 1.54 Å at 40 kV and 40 mA. A field-emission scanning electron microscope (SEM) (JSM-6700F, JEOL) was applied to record the morphological images and energy dispersive X-ray spectroscopy (EDS) information of the obtained sample. High resolution transmission electron microscope (HR-TEM) and bright-field transmission electron microscope (TEM) images were carried out by a transmission electron microscope (JEM-2010 FEF, JEOL). Photoluminescence spectroscopy was achieved with a fluorescence spectrometer (Lambda 35, Perkin Elmer). The $N_2$ adsorption and desorption isotherms of the sample was examined at 77 K (Autosorb-iQASIQ, Quantachrome). Specific surface area was calculated using the Brunauer–Emmett–Teller (BET) equation and the pore size distribution plot was determined via the Barrett–Joyner–Halenda (BJH) theories. X-ray photoelectron microscopy (XPS) data were recorded with an X-ray radiation of 1486.6 eV and assigning the C 1s peak to 284.6 eV (VG Multilab 2000, Thermo Fisher Scientific).

Electrochemical test.

In three-electrode test, the raw sample, acetylene black and poly(tetrafluoro ethylene) was mixed in a mass ratio of 8:1:1 in isopropyl alcohol. Then, the mixture was ground intensely to form a uniform slurry and coated on the surface of nickel foam. After that, the electrode was dried in a vacuum electric oven at 40 °C for 8 h and tableted with a pressure of 10 MPa. A piece of commercial membrane (NKK-MPF30AC-100) served as the separator and the two electrodes were assembled together with the separator sandwiched between them, when fabricating the asymmetric HSCSs//AC device in 2 M KOH electrolyte. Cyclic voltammetry (CV), galvanostatic charge and discharge (GCD), cyclic stability and electrochemical impedance spectroscopy (EIS) measurements were carried out by an electrochemical workstation (CHI 660E). All the electrochemical tests were performed utilizing the sample, Hg/HgO and Pt foil as the working electrode, reference electrode and counter electrode accordingly. The specific capacitance was calculated according to the equation $C=It/m\Delta V$, where C ($F\ g^{-1}$) is specific capacitance, I

(A) is current, V is the work potential, m is the mass of the sample, and t is the discharge time.

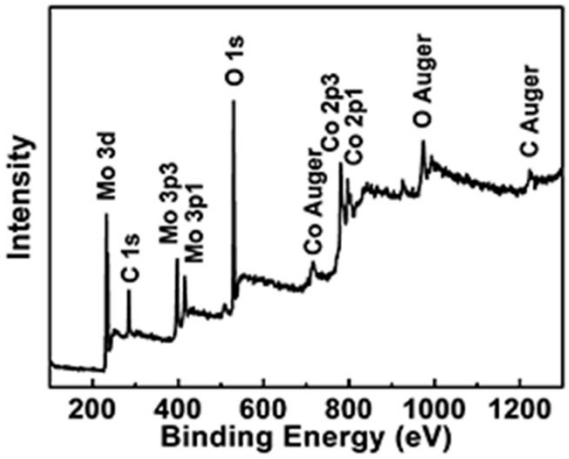


**Fig. S1** XPS spectrum of HSCSs.

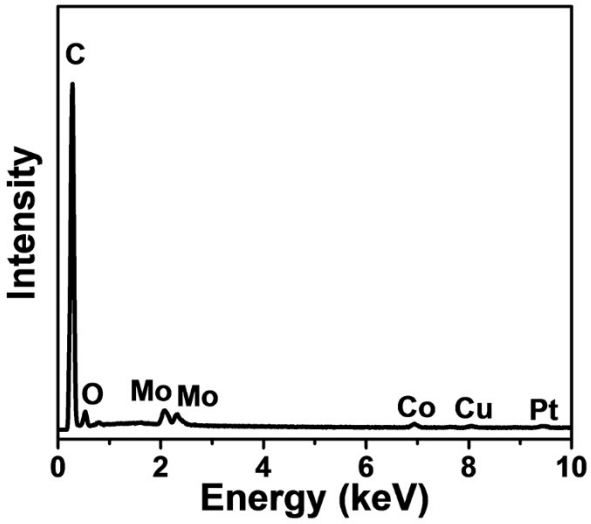


**Fig. S2** EDS spectrum of HSCSs.

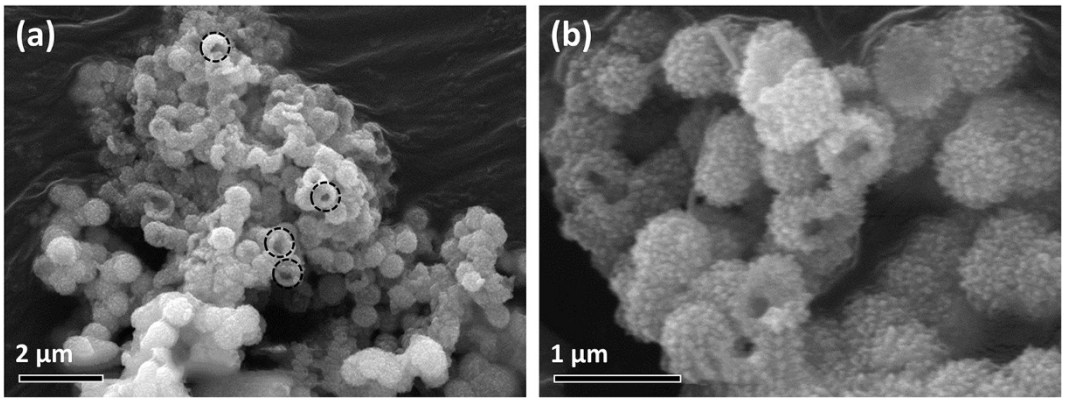

**Fig. S3** (a, b) SEM images of HSCSs at different magnification; black circles in (a) show the cracked HSCSs.

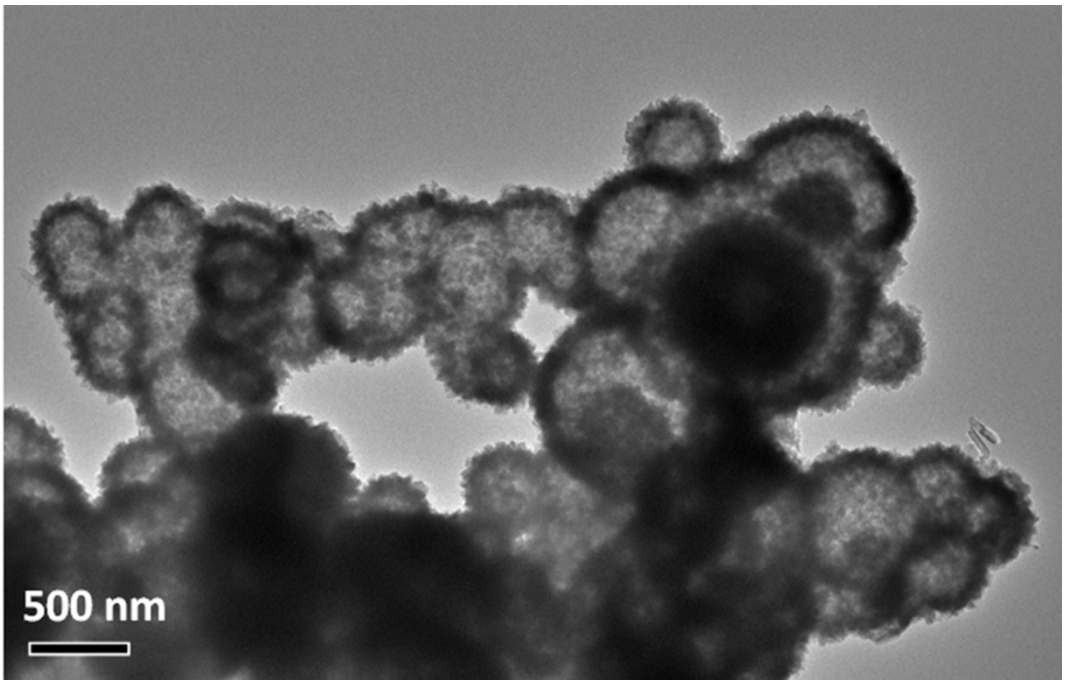


**Fig. S4** TEM image of HSCSs.

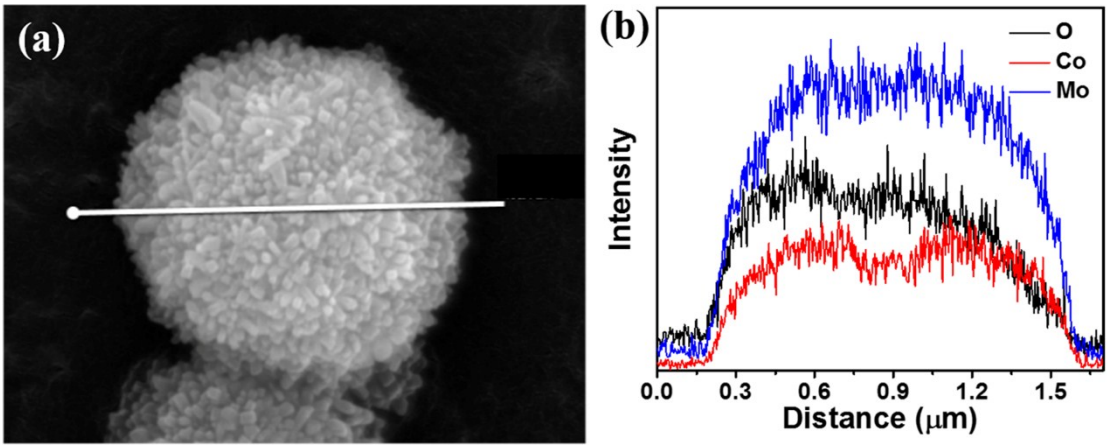


**Fig. S5** (a) SEM image and (b) EDS line scan spectra of O, Co and Mo from HSCSs.

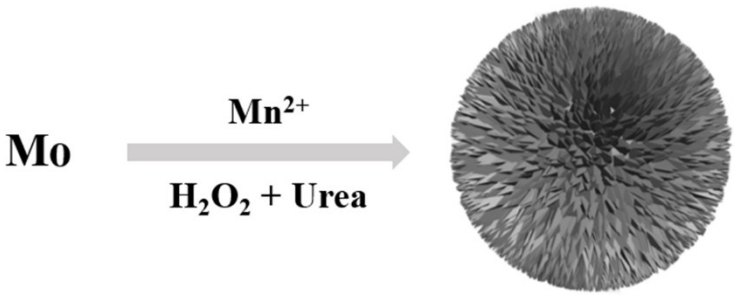


**Fig. S6** Illustration of the synthesis process.

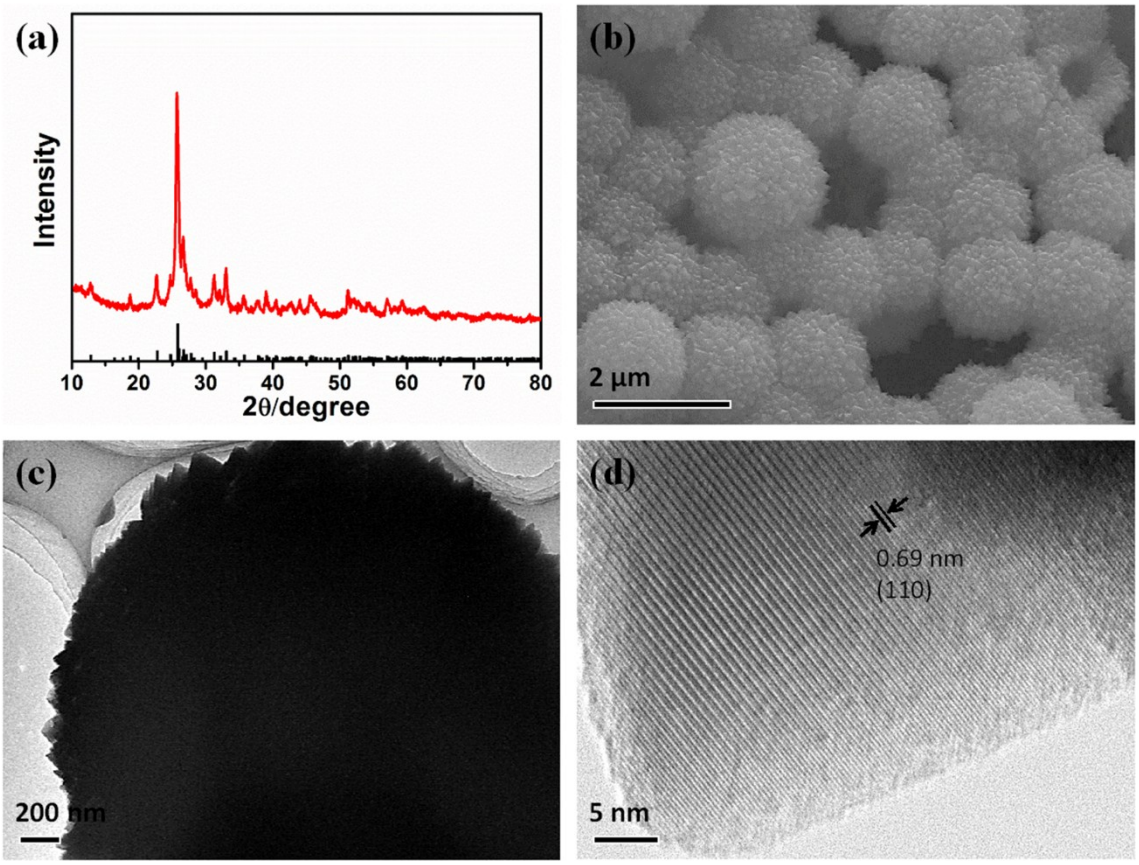


**Fig. S7** (a) XRD pattern, (b) SEM, (c) TEM and (d) HR-TEM images of $MnMoO_4$.

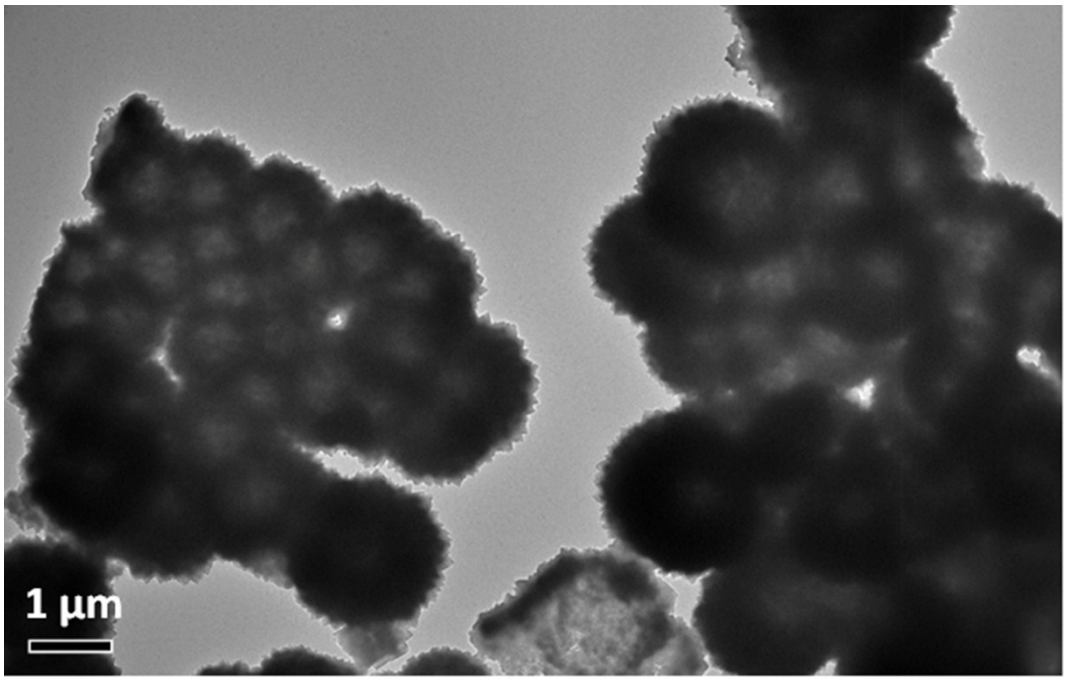


**Fig. S8** TEM image of $MnMoO_4$.

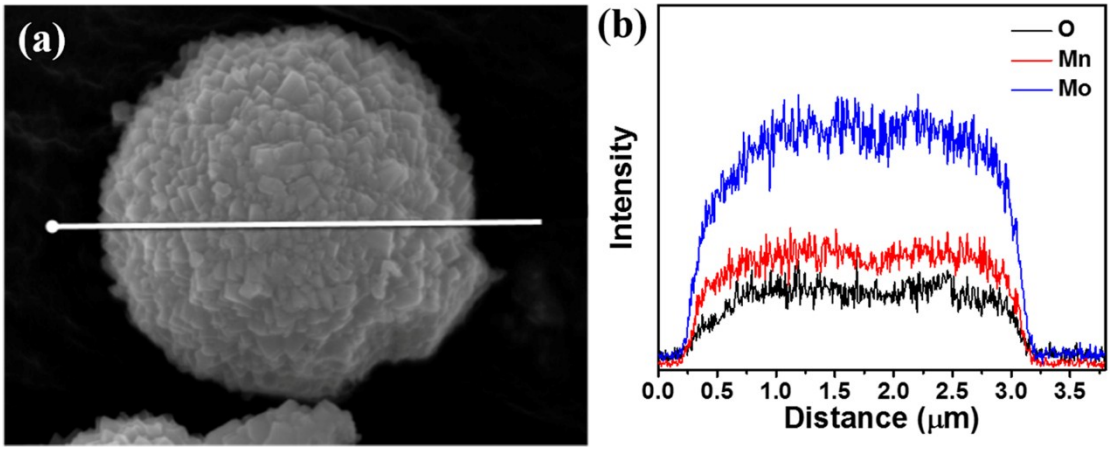


**Fig. S9** (a) SEM image and (b) EDS line scan spectra of O, Mn and Mo from $MnMoO_4$.

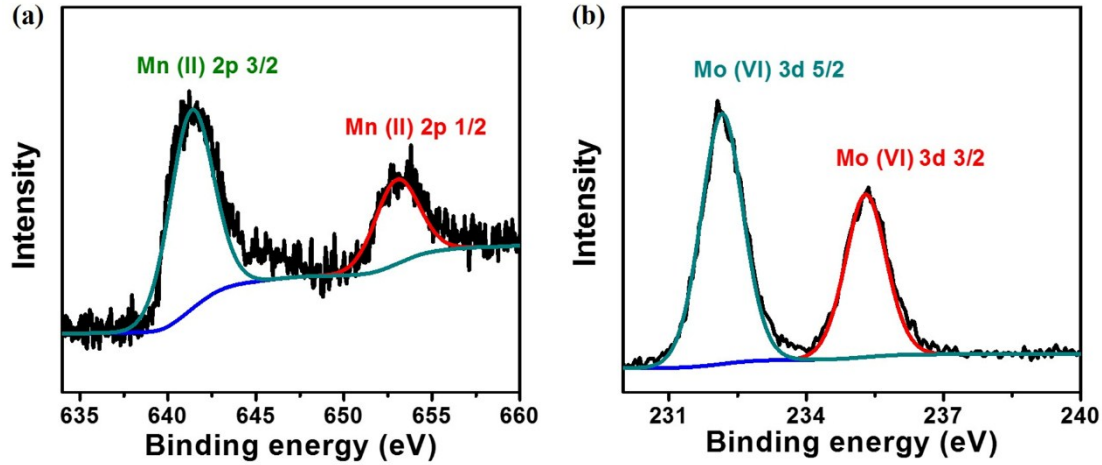


**Fig. S10** High-resolution XPS spectra of (a) Mn 2p and (b) Mo 3d from $MnMoO_4$.

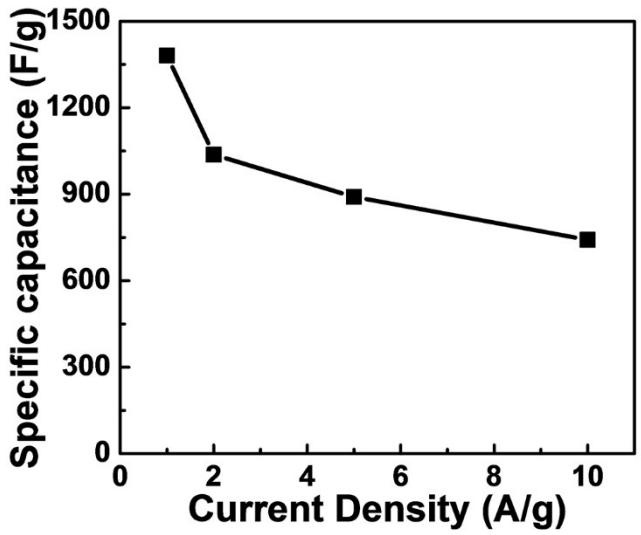


**Fig. S11** Rate capability of HSCSs in three-electrode system.

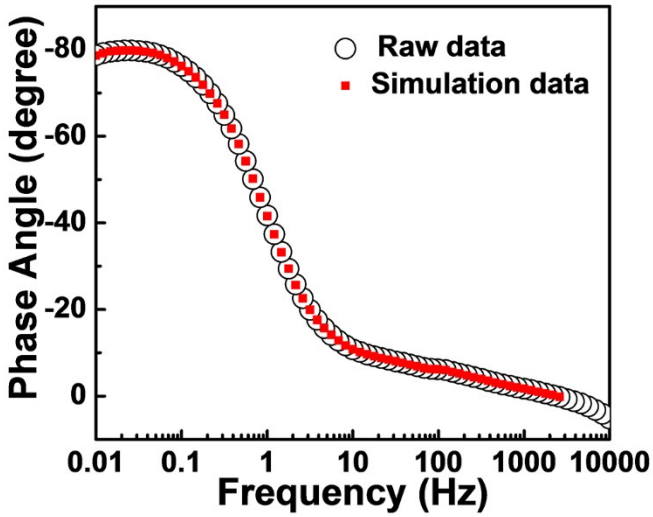


**Fig. S12** Bode plot of HSCSs in three-electrode system.

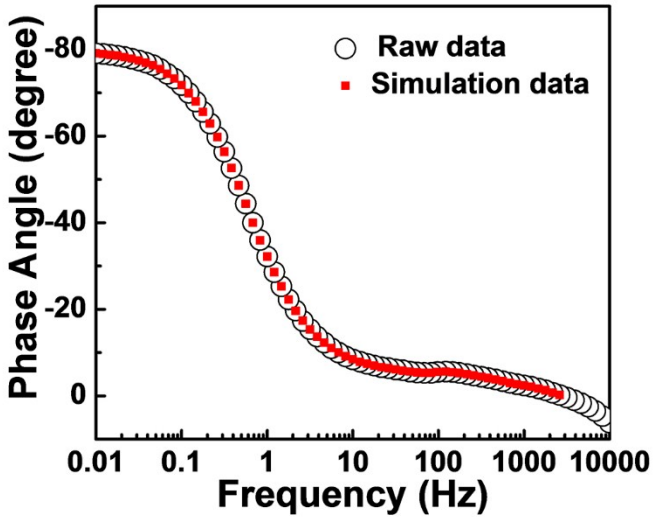


**Fig. S13** Bode plot of HSCSs in two-electrode system.