Electronic Supplementary Material (ESI) for CrystEngComm.

Supporting Information

Controllable

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Experiment

Preparation of NiCoMo-LDH

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0.03 M of Ni $(NO_3)_2 \cdot 6H_2O$, 0.015M of $Co(NO_3)_2$.6H2O, and 0.015 M of $Na_2MoO_4 \cdot 2H_2O$ were dissolved in deionized water (100 mL) containing 0.072 M urea. The pre-treated nickel foam and precursor solution were then transferred to a stainless steel autoclave lined with Teflon and subjected to a hydrothermal reaction at 110℃ for 10 hours. The sample was taken out and cooled to room temperature, and then subjected to ultrasonic treatment in deionized water and dried at 60℃ for 8 hours. The obtained sample was named NCM-LDH.

Preparation of NiCoMo-LDH@NiCoMoS

The sample NCM-LDH was placed in an 80mL Teflon-lined stainless steel reactor containing 0.1M $Na_2S \cdot 9H_2O$ solution and the reaction time was set to 2 h, 5 h, 8 h and 12 h. After cooling to room temperature, the samples obtained by ultrasound for 5 min in deionized water and subsequently dried in an oven at 60°C for 8 h. The resulting samples were named NCMS-2, NCMS-5, NCMS-8 and NCMS-12. The loadings of NiCoMo-LDH, NiCoMoS-2, NiCoMoS-5, NiCoMoS-8 and NiCoMoS-12 electrodes were 1.9, 3.0, 3.7, 4.3 and 5.0 mg /cm^2, respectively.

Material characterization

The phase, morphology and microstructure of the synthesized samples were characterized by an X-ray diffractometer (XRD, Bruker, Germany), field emission scanning electron microscope (FESEM, NovaNano-450 FEI) and transmission electron microscope (TEM, JEM-2100). The surface characteristics of the samples were analyzed by X-ray photoelectron spectroscopy (XPS, Escalab250). The N_2 adsorption-

desorption isotherm was determined by Brunauer-Emmett-Teller (BET) theory with the surface area analyzer (BSD 3H-2000PS1, BeiShiDe Instrument).

Electrochemical measurements

Electrochemical tests of the samples were performed using electrochemical workstation (PGSTAT302N, AUTOLAB) in a 2 M KOH electrolyte. In the three electrode test, the samples was used as the working electrode, Hg/HgO as the reference electrode, Pt plate as the counter electrode. In the ASC measurement, NCM-LDH@NCMS was used as the positive electrode and AC as the negative electrode. Cyclic voltammetry (CV) and Galvanostatic charge / discharge (GCD) curves of NCM-LDH@NCMS were recorded in the potential window -0.2V to 0.8V at ranging from 10 to 35 mV and 0V to 0.5V at ranging from 10 to 50 mA cm^{-2}, respectively. Electrochemical impedance spectroscopy (EIS) was performed in the frequency range of 100 kHz to 0.01 Hz. The capacitance of the electrode can be calculated from the GCD curve according to equations (S1):

$$C_A = \frac{I \times \Delta t}{S \times \Delta V} \qquad \text{(S1)}$$

Where C_A (F cm^{-2}) represents the areal capacitance of NCM-LDH@NCMS, I (A) refers to the charge and discharge current of GCD, Δt (s) is the discharge time of the GCD curve, S (cm^2) is the geometrical area of NCM-LDH@NCMS , m (g) represents the mass loading of the active material, ΔV (V) represents the potential.

In ASC system, NCM-LDH@NCMS-2, AC and 2 M KOH were used as positive electrode, negative electrode and electrolyte, respectively. The charge balance between the two electrodes obeys the relationship $q^+ = q^-$ and $q = C_A \times S \times \Delta V$. The mass loading of the two-electrode system (ASC) was about 8.6 mg. The energy density (E, mWh cm^{-2}) and power density (P, mW cm^{-2}) of the ASC devices were calculated based on GCD curves as followed according to equations (S2) and (S3):

$$E = \frac{C \times \Delta V^2}{2 \times 3.6} \qquad \text{(S2)}$$

$$P = \frac{3600 \times E}{\Delta t} \qquad \text{(S3)}$$

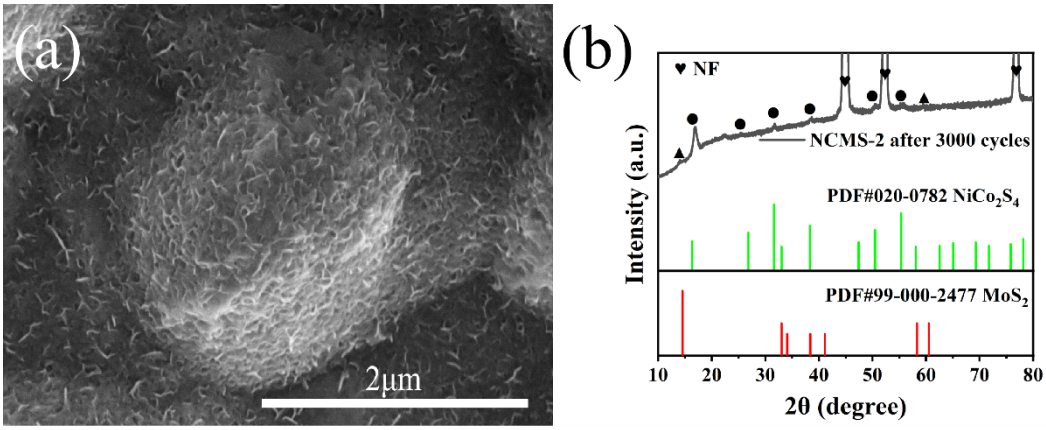

Fig. S1 FESEM image (a) and XRD patterns (b) of NCMS-2 after 3000 cycles.