# SUPPLEMENTARY INFORMATION

#### Yolk-shell-structured microspheres composed of N-doped-carbon-

### coated NiMoO<sub>4</sub> hollow nanospheres as superior performance

## anode material for lithium-ion batteries

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**Fig. S1** Morphologies of yolk-shell-structured NiMoO<sub>4</sub> precursor microspheres prepared by one-pot spray pyrolysis process.



**Fig. S2** XRD patterns of yolk-shell-strucutred NiMoO<sub>4</sub> precursor microspheres (Y-NiMoO<sub>4</sub>), Y-NiSe<sub>2</sub>-MoSe<sub>2</sub> composite via the selenization process of Y-NiMoO<sub>4</sub>, Y-NiMoO<sub>4</sub>-H via the oxidation of Y-NiSe<sub>2</sub>-MoSe<sub>2</sub>, and Y-NiMoO<sub>4</sub>-H@C via carbon coating process of Y-NiMoO<sub>4</sub>-H.



Fig. S3 XPS spectra of yolk-shell-structured NiSe<sub>2</sub>-MoSe<sub>2</sub> composite microsphere: (a) Ni 2p,(b) Mo 3d, and (c) Se 3d.



**Fig. S4** Formation mechanism of the hollow NiMoO<sub>4</sub> nanospheres from NiSe<sub>2</sub>-MoSe<sub>2</sub> composite via nanoscale Kirkendall diffusion.



Fig. S5 TG curve of Y-NiMoO<sub>4</sub>-H measured under an air atmosphere.



**Fig. S6** (a) N<sub>2</sub> gas adsorption and desorption isotherms, and (b) Barrett–Joyner–Halenda (BJH) pore-size distributions of Y-NiMoO<sub>4</sub>-H@C, Y-NiMoO<sub>4</sub>-H, and Y-NiMoO<sub>4</sub>-D.



**Fig. S7** Morphologies of yolk-shell-structured NiO microspheres prepared by one-pot spray pyrolysis process.



Fig. S8 Morphologies of NiSe<sub>2</sub> microspheres prepared by spray pyrolysis and subsequent selenization process.



Fig. S9 Morphologies of MoO<sub>3</sub> microspheres prepared by one-pot spray pyrolysis process.



**Fig. S10** Morphologies of (a,b) Y-NiMoO<sub>4</sub>-D and (c,d) Y-NiMoO<sub>4</sub>-D@C microspheres composed of NiMoO<sub>4</sub> dense nanocrystals.



Fig. S11 XRD patterns of Y-NiMoO<sub>4</sub>-D@C and Y-NiMoO<sub>4</sub>-D microspheres.



Fig. S12 TG curve of Y-NiMoO<sub>4</sub>-D@C microspheres.



Fig. S13 CV curves of (a) Y-NiMoO<sub>4</sub>-H, (b) Y-NiMoO<sub>4</sub>-D@C, and (c) Y-NiMoO<sub>4</sub>-D.



Fig. S14 Redox peaks and the corresponding reactions of yolk-shell-structured NiMoO<sub>4</sub> microspheres.

**Table S1.** Rate performances of various nanostructured NiMoO<sub>4</sub> materials with and without carbon material that were reported in previous literature as anode materials for lithium-ion batteries.

| Various NiMoO4 materials  | Synthesis                                     | Electrochemical properties  | Ref         |
|---|---|---|-------------|
| Honeycomb-like NiMoO <sub>4</sub><br>ultrathin nanosheet arrays   | Electrochemical method                        | The discharge capacities were 870 and 370 mA h g <sup>-1</sup> at current densities of 0.2 and 8.0 A g <sup>-1</sup> , respectively.          | [S1]        |
| Porous NiMoO4<br>nanoarchitectures on 3D<br>graphene foam   | Chemical vapor<br>deposition (CVD)            | The discharge capacities were ~1100 and ~600 mA<br>h g <sup>-1</sup> at current densities of 0.2 and 3.2 A g <sup>-1</sup> ,<br>respectively. | [\$2]       |
| NiMoO4 microspheres with<br>numerous empty nanovoids  | Spray pyrolysis                               | The discharge capacities were 1280 and 413 mA h g <sup>-1</sup> at current densities of 0.5 and 10.0 A g <sup>-1</sup> , respectively.        | [S3]        |
| Porous worm-like NiMoO4<br>coaxially decorated electrospun<br>carbon nanofiber                          | Hydrothermal method                           | The discharge capacities were 1132 and 503 mA h g <sup>-1</sup> at current densities of 0.5 and 2.0 A g <sup>-1</sup> , respectively.         | [S4]        |
| Hierarchical free-standing<br>NiMoO4/reduced graphene oxide<br>membrane                                 | Hydrothermal<br>method & vacuum<br>filtration | The discharge capacities were 1116 and 690 mA h g <sup>-1</sup> at current densities of 0.25 and 4.0 A g <sup>-1</sup> , respectively.        | [85]        |
| Hierarchical NiMoO4 nanowire  | Hydrothermal method                           | The discharge capacities were 1338 and 231 mA h g <sup>-1</sup> at current densities of 0.1 and 1.0 A g <sup>-1</sup> , respectively.         | [S6]        |
| Phase-pure β-NiMoO <sub>4</sub> yolk-shell spheres  | Spray pyrolysis                               | The discharge capacities were 1247 and 612 mA h g <sup>-1</sup> at current densities of 0.5 and 5.0 A g <sup>-1</sup> , respectively.         | [S7]        |
| Yolk-shell-structured<br>microspheres composed of<br>N-doped-carbon-coated<br>NiMoO4 hollow nanospheres | Spray pyrolysis<br>& Kirkendall<br>diffusion  | The discharge capacities were 1267 and 757 mA h $g^{-1}$ at current densities of 0.5 and 10.0 A $g^{-1}$ , respectively.                      | Our<br>work |

#### References

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