Supplementary Data

Nanocubes of Mo₆S₈ Chevrel Phase as Active Electrode Material for Aqueous Lithium-Ion Batteries

Amr Elgendy^{1,2,6}, Athanasios A. Papaderakis^{1,2}, Rongsheng Cai³, Kacper Polus⁴, Sarah J. Haigh^{2,3, 5}, Alex S. Walton^{1,4}, David J. Lewis^{3*}, Robert A. W. Dryfe^{1,2*}

¹Department of Chemistry, University of Manchester, Oxford Road, Manchester, M13 9PL, United Kingdom

² Henry Royce Institute, University of Manchester, Oxford Road, Manchester, M13 9PL, United Kingdom

³ Department of Materials, University of Manchester, Oxford Road, Manchester, M13 9PL, United Kingdom

⁴ Photon Science Institute, University of Manchester, Oxford Road, Manchester, M13 9PL, United Kingdom

⁵ National Graphene Institute, University of Manchester, Oxford Road, Manchester M13 9PL, U.K.

⁶Egyptian Petroleum Research Institute, 11727, Cairo, Egypt.

Corresponding Authors

*E-mail: robert.dryfe@manchester.ac.uk Tel: +44 (0) 161-306-4522. (R.A.W.D).

*E-mail: <u>david.lewis-4@manchester.ac.uk</u> Tel: +44 (0) 161-306-3561. (D.J.L.)

• The technical and experimental details of the ICP-MS protocol used are described right below:

The ICP method is based on British Standards Document BS EN ISO 17294-1 for Trace Metals in waters, using a PerkinElmer NexION 350D ICP-MS. This method uses a 5-point linear calibration with a 99.999% minimum regression, with an internal standard to correct for instrument drift and matrix interferences.

Results are quantified against this curve in mg/L, or ppm, at a limit of detection of 5 ppb, any figures below this should be noted as being below the limit of detection (<LOD); including any quantities below zero, as the instrument below these levels cannot discern peaks from background noise. Each sample is measured three times, and an average of these replicates is calculated by the instrument. The %RSD variance between each replicate is less than 2.5%.



Fig. S1. Comparison of the Raman spectrum of the aged and fresh leached Mo_6S_8



Fig. S2. The survey spectrum of the Mo_6S_8 , contains the expected elements of Mo and S with carbon and oxygen, typical of atmosphere exposed surfaces, in agreement with the EDX spectrum.



Fig. S3. SEM EDX spectraimages of the as-prepared $Cu_2Mo_6S_8$ and Mo_6S_8 . $Cu_2Mo_6S_8$ (a), and Mo_6S_8 (b).



Fig. S4. HAADF-STEM image of the as-prepared $Cu_2Mo_6S_8$ and Mo_6S_8 and their corresponding STEM EDX elemental mapping. $Cu_2Mo_6S_8$ (a-d), and Mo_6S_8 (e-g). Mo (purple), S (red), and Cu (yellow).



Fig. S5. Cyclic voltammetry (CV) of Mo_6S_8 electrode measured in different sodium electrolytes at a scan rate of 3 mV/s. In (b) comparison of the CV profiles with and without NaOAc, shows that the peak response is attributed to the Na⁺ intercalation and not Cs⁺ ions.



Fig. S6. Cyclic voltammetry of Mo_6S_8 *working electrode in* 2*M MgSO4 at* 3 *mV/s.*



Fig. S7. Nyquist plots recorded at open circuit potential (OCP) (determined by monitoring the change in potential, *E*, with time, *t*, and considering a stable potential value for $dE/dt < 1 \mu V$) during the galvanostatic charging/discharging of full cell in the frequency range from 10kHz to 1 mHz.