

Supporting Information for Hill *et al.*

Periodic Mesoporous $\text{Li}_x(\text{Mn}_{1/3}\text{Ni}_{1/3}\text{Co}_{1/3})\text{O}_2$ Spinel

Experimental Details

Nitrogen adsorption experiments were performed on a Quantachrome Autosorb 1 at -196 °C over the partial pressure range $0.005 \leq P/P_0 \leq 0.975$. Prior to analysis, samples were degassed at 200°C and 5×10^{-3} Torr for 24 hours. Parameters including BET surface area, total pore volume, and t-plot micropore analysis were determined using the 'Autosorb 1 for Windows' ver 1.55 Software, © 2001. BdB-BHH pore size distributions were determined following the procedure described by Lukens *et al.*^[1]

X-ray diffraction was performed on the powder diffraction beamline at the Australian Synchrotron with an incident wavelength of 1.00 Å. Each sample was placed in a 0.3 mm diameter quartz capillary and examined over the range of $2 < 2\theta < 82$. Samples were also examined on a Bruker D8 Advance X-ray Diffractometer with $\text{CuK}\alpha$ radiation (40kV, 40mA) monochromatised with a graphite sample monochromator was employed to determine the X-ray diffraction (XRD) patterns. Each sample was scanned over the 2-theta range 5 to 85° with a step size of 0.02° and a count time of 4 seconds per step. Analyses were performed on the collected XRD data for each sample using the Bruker XRD search match program EVA™. Crystalline phases were identified using the ICDD-JCPDS powder diffraction database. Structure calculations were performed using the freely available Crysfire software.

Transmission Electron Microscopy (TEM). The samples were first dispersed in Milli-Q water and deposited on 200 mesh carbon coated copper grids. Microscopy was then performed on a Philips CM30 Transmission Electron Microscope, operating at an accelerating voltage of 200kV. Selected area electron diffraction was recorded on a JEOL 2100F operated at 200kV. The material was prepared for TEM observation simply by dispersing its powder form in high-purity ethanol and depositing a drop of the suspension onto a copper grid coated with holey-carbon film.

Preparation of KIT-6 silica

This material was prepared according to a previously reported procedure.^[2] 24.00 g of Pluronic P123 dissolved at 37 °C in a polypropylene flask containing a mixture of 864 mL water and 47.4 mL of 35 % HCl. After complete dissolution 24.00 g of *n*-BuOH was added and the mixture stirred at 37 °C for a further 60 minutes. Subsequently tetraethyl *ortho*-silicate (51.60 g, 0.248 mol) was added and the mixture stirred for a further 20 hours. Stirring was ceased and the mixture was aged at 100 °C for 24 hours. The mixture was filtered while hot, dried under flowing air, washed with a small amount of 10 % HCl-EtOH, dried once more, and then calcined at 550 °C for 6 hours, the temperature was ramped at a rate of 1 °C/min. Yield 14.59 g, 98 %. BET Surface area^a 823 m²g⁻¹, total pore volume^b 1.050 cm³g⁻¹, micropore volume^c 0.114 m²g⁻¹. ^a BET surface area calculated from the nitrogen adsorption isotherm in the range 0.05 ≤ P/Po ≤ 0.30. ^b Calculated from a single point on the BET adsorption isotherm at P/Po=0.95. ^c Calculated

Results and Discussion

Structure solution of the metal oxide phase

Samples were examined using synchrotron radiation to better isolate the peak positions in comparison to that obtained by laboratory means, which delivered extremely broad peaks (see Figure 2 of main article) due to the small crystallite size of the mesoporous material.

Table S1. Calculated and observed peaks assigned to monoclinic lithium manganese nickel cobalt oxide with unit cell parameters $a= 8.148 \text{ \AA}$ $b= 2.884 \text{ \AA}$ $c= 5.780 \text{ \AA}$,
 $\beta= 90.59^\circ$, $V= 135.83 \text{ \AA}^3$.

hkl	d_{obs} (XRD)	D_{obs} (SAED)	d_{calc}
1 0 1	4.693	4.68 (2)	4.688
2 0 0	2.886	2.86 (2)	2.887
1 0 -3	2.467	2.45 (2)	2.467
2 0 -2	2.369	2.35 (3)	2.367
2 2 0	2.042	2.06 (2)	2.041
2 2 -2	1.831	1.81 (3)	1.831
3 2 -1	1.574	1.58 (1)	1.574
2 2 -4	1.447	1.44 (1)	1.447

Elemental analysis

Table S2. Results of ICP analysis.

Calcination Temperature/°C	Stoichiometric Composition	Crystal Size/nm	1 st Charge Capacity/mAhg ⁻¹	1 st Discharge Capacity/mAhg ⁻¹
500	Li _{0.33} Mn _{0.3} Ni _{0.3} Co _{0.3} O ₂	7.21	180	168
600	Li _{0.35} Mn _{0.3} Ni _{0.3} Co _{0.3} O ₂	9.31	109	102
700	Li _{0.40} Mn _{0.3} Ni _{0.3} Co _{0.3} O ₂	9.92	80	93
1000		83.75		

Prior to elemental Analysis all samples were digested in HCl/HNO₃ acid mixture with elemental ratios determined by an Inductively Coupled Plasma – Atomic Emission Spectrometer.

References

1. Lukens, W. W.; Schmidt-Winkel, P.; Zhao, D. Y.; Feng, J. L.; Stucky, G. D., *Langmuir* **1999**, 15(16), 5403.