

**Electronic Supplementary Information for:**

**Improved electrode kinetics in lithium manganospinel nanoparticles synthesized by hydrothermal methods: identifying and eliminating oxygen vacancies**

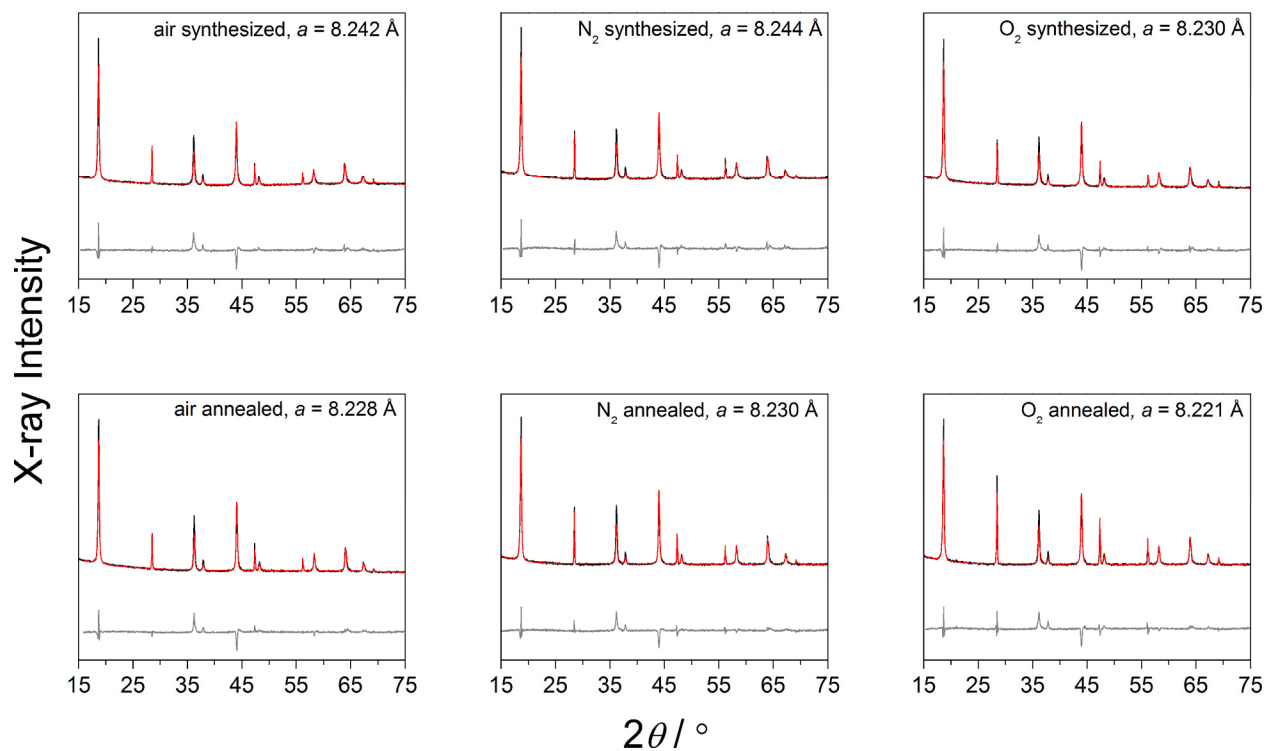
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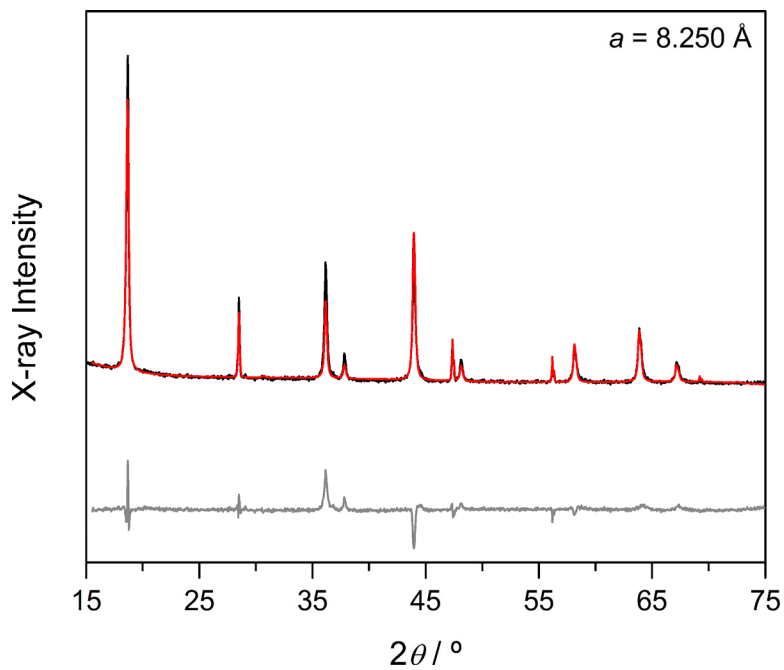
<sup>2</sup>Jülich Centre for Neutron Science (JCNS) outstation at Spallation Neutron Source (SNS)/ORNL

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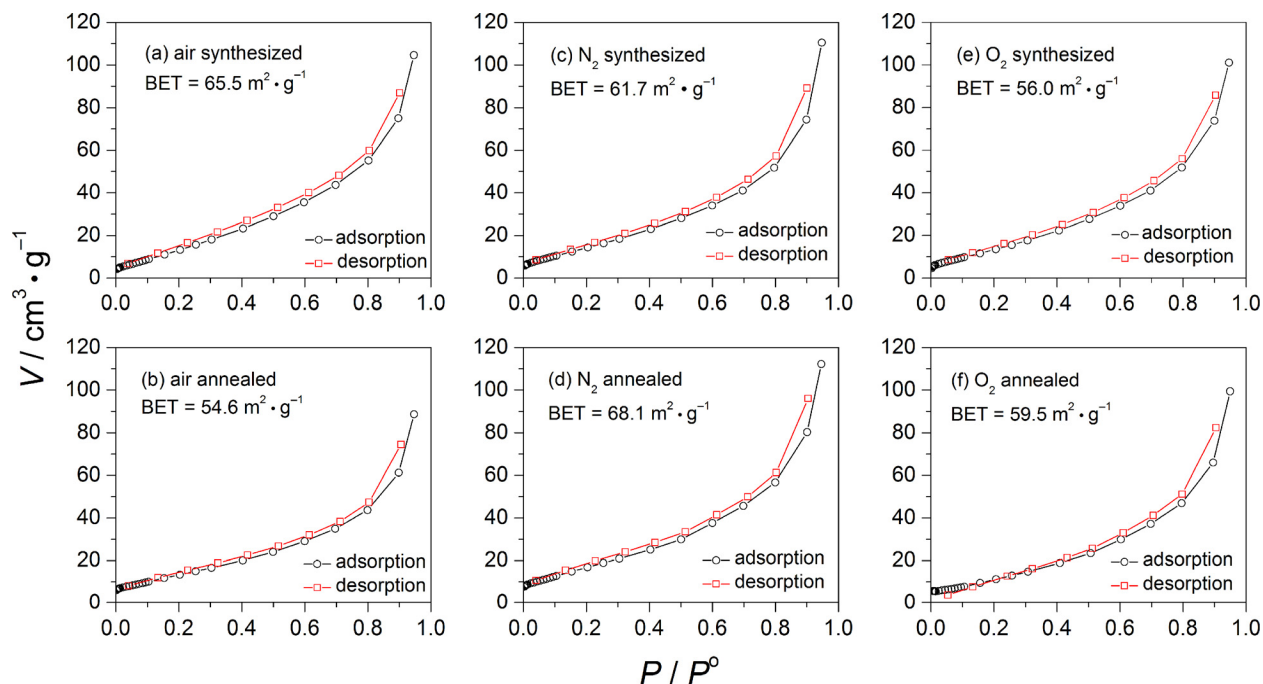
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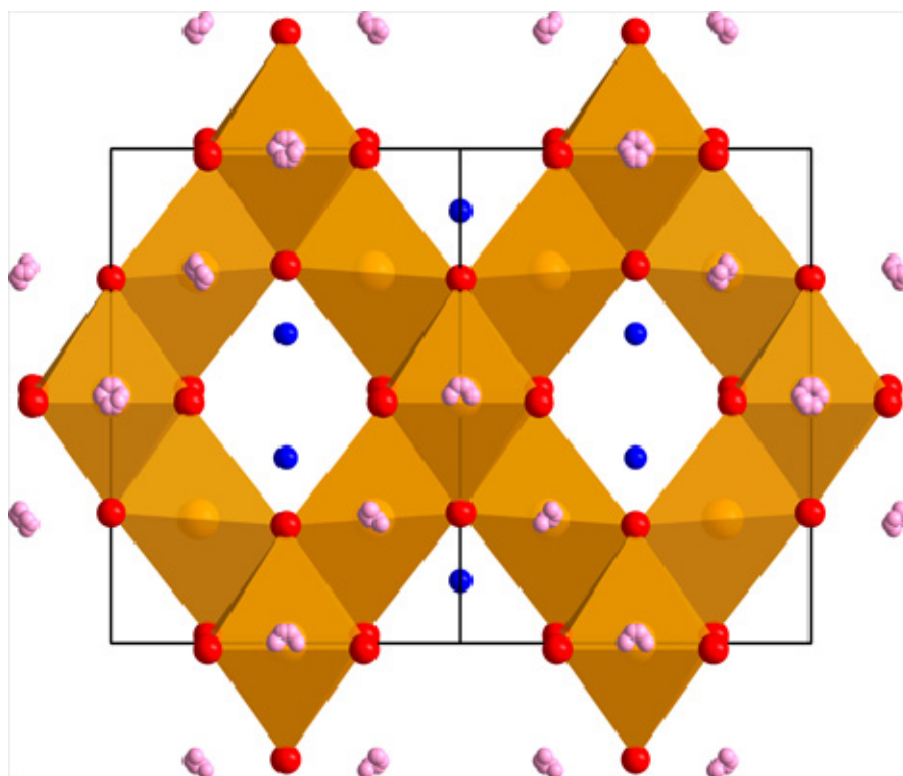
**Figure S1.** XRD patterns of hydrothermally synthesized lithium manganospinels.



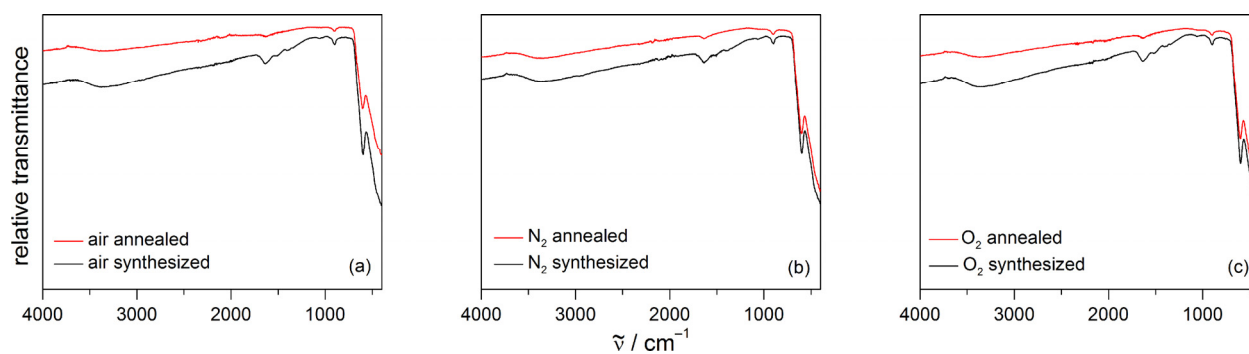
**Figure S2.** XRD pattern of lithium manganospinel synthesized hydrothermal in air followed by annealing under  $\text{N}_2$ .



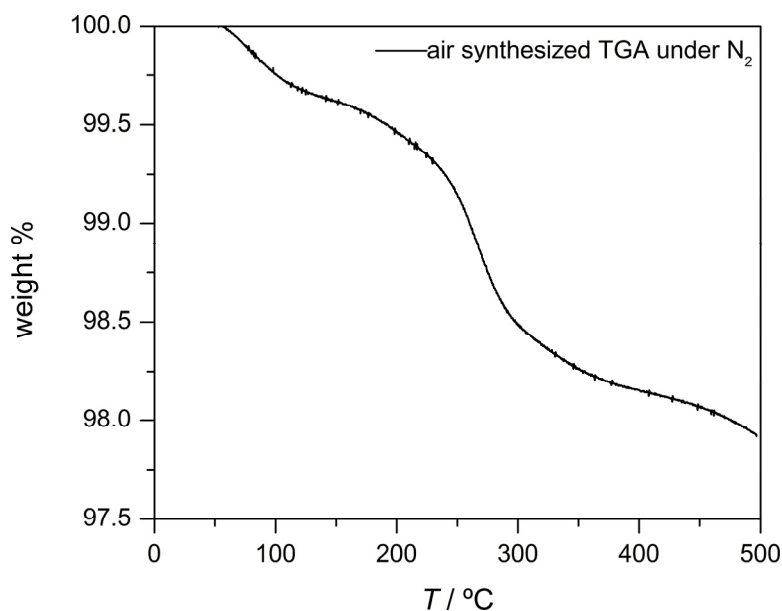
**Figure S3.**  $\text{N}_2$  sorption isotherms for lithium manganospinels prepared hydrothermally. The top panels represent the synthesized samples and the bottom panels represent the annealed samples. All compounds type II isotherms with surface areas of  $\sim 60 \text{ m}^2/\text{g}$  as determined from the Brunauer-Emmett-Teller (BET) method.



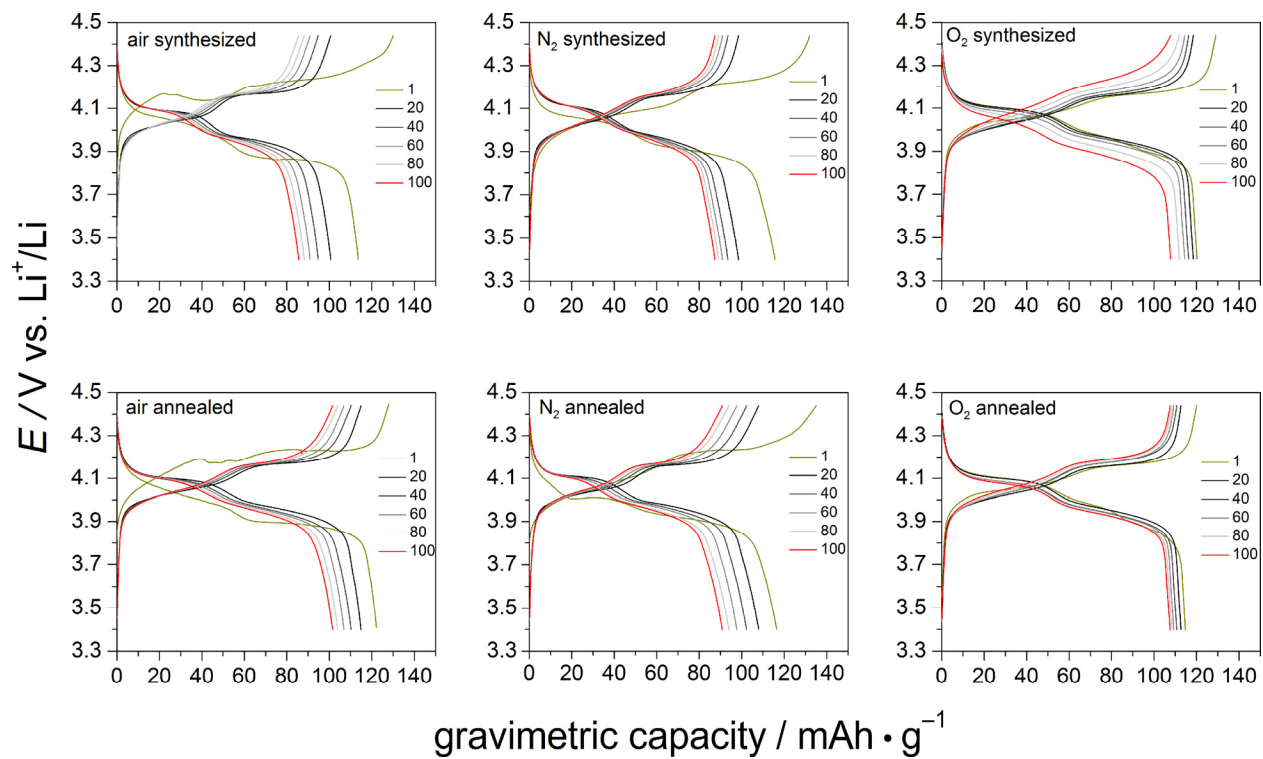
**Figure S4.** Unit cell of cubic  $Fd\bar{3}m$  lithium manganospinel viewed along  $[110]$  highlighting the excess electron density observed in the refined neutron diffraction data at  $(0, 0.26, 0.73)$  shown in pink. Blue, orange, and red spheres represent Li, Mn, and O atoms respectively, with  $MnO_6$  octahedra shaded in.



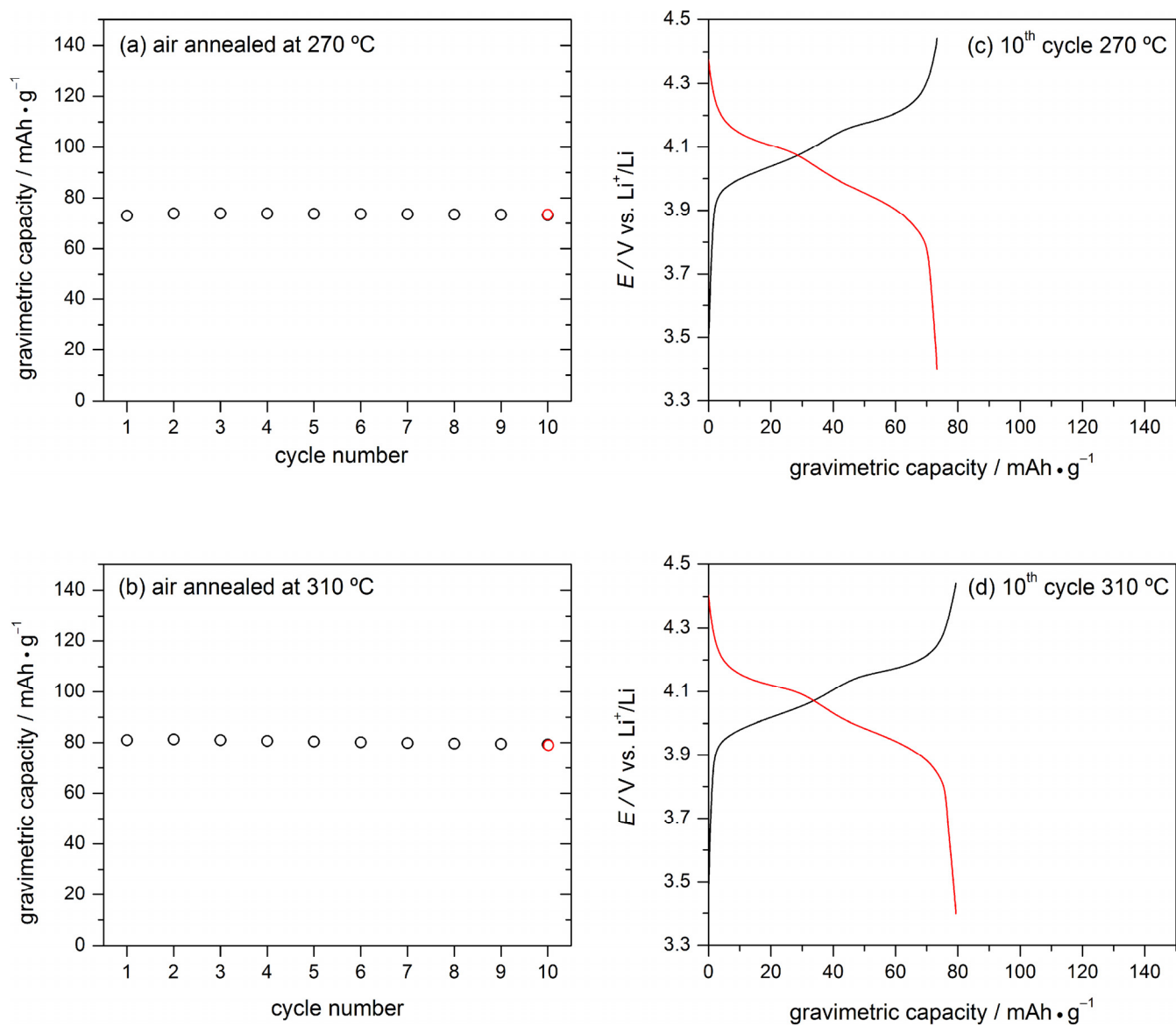
**Figure S5.** FTIR spectra of hydrothermally synthesized lithium manganospinels. Black and red traces represent the synthesized and annealed spectra for compounds prepared in a) air; b) N<sub>2</sub>; c) O<sub>2</sub>, respectively.  $\nu(\text{O-H})$  and  $\delta(\text{H-O-H})$  modes at 3300 and 1630 cm<sup>-1</sup> respectively indicate surface-bound water.



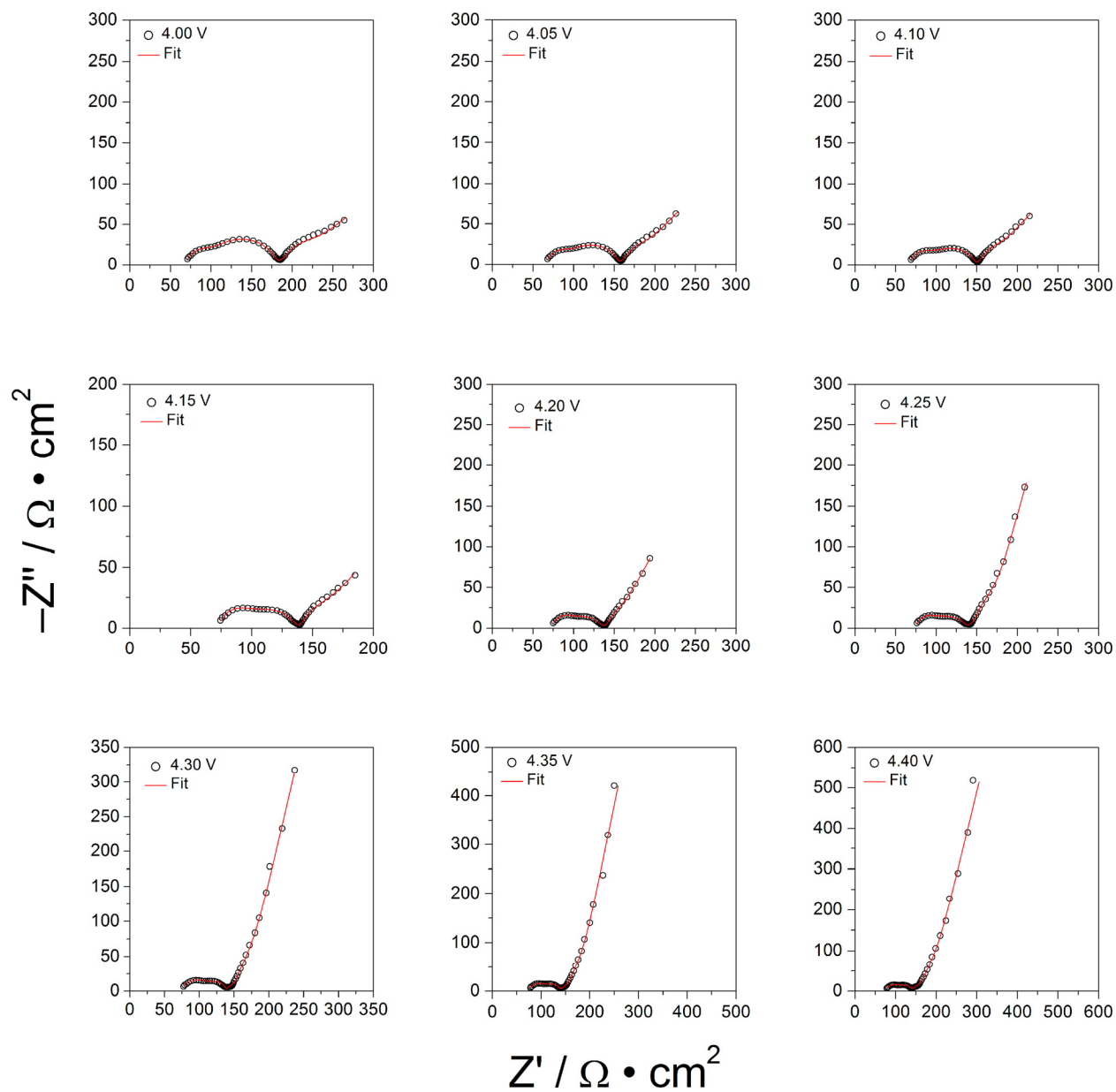
**Figure S6.** TGA of the air synthesized sample performed under N<sub>2</sub> purge. No mass gain associated with oxygen uptake between 250–350 °C is observed.



**Figure S7.** Individual chronopotentiometry profiles for  $C/3$  cycling experiments shown in Figure X. The first charge-discharge cycle is in gold and the 100<sup>th</sup> cycle is in red.



**Figure S8.** Cycling behavior of lithium manganospinel synthesized in air and annealed at (a) 270 °C and (b) 310 °C respectively. Panels (c) and (d) show the charge and discharge curves for the 10<sup>th</sup> cycles of each.



**Figure S9.** Nyquist plots for  $O_2$  annealed sample at varying potential.