

Abstract Submitted  
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**Synthesis, Structural, and Electrochemical Stability Studies of  
Nanocrystalline 5V Lithiated Oxides for Asymmetric Supercapacitor**

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— For the development of asymmetric (hybrid) supercapacitor, we have synthesized  
nanoscale double substituted  $\text{LiNi}_Y\text{Co}_{1-2Y}\text{Mn}_{1+Y}\text{O}_4$  ( $Y=0.05, 0.1, 0.25$  and  $0.45$ )  
spinel using mechanochemical, hydrothermal, microwave assisted combustion aided  
procedures. The samples have been characterized by XRD, TEM, and XAFS. Lat-  
tice parameter of the spinels increased with nickel content, and decreased from 400 to  
600 °C, at which temperature the particle size is  $\approx 20\text{nm}$ . The sample with compo-  
sition  $\text{LiNi}_{0.45}\text{Co}_{0.1}\text{Mn}_{1.45}\text{O}_4$  has shown the best electrochemical performance, with  
redox potential of 4.6V, capacity of  $129.6\text{mAhg}^{-1}$ , cyclability of 99.6% per cycle,  
and retained the capacity up to 1  $C$  rate. The XANES of Mn and  $M$  as a function  
of  $x$  showed that the high voltage ( $\sim 5$  V) in the cathode materials of an Li secondary  
battery is due to the oxidation of  $M^{3+}$  to  $M^{4+}$  ( $M=\text{Co}$ ) and  $M^{2+}$  to  $M^{4+}$  (in the  
case of  $M=\text{Ni}$ ). The EXAFS analysis revealed that  $\text{Ni}^{2+}$  is oxidized to  $\text{Ni}^{4+}$  via  
the  $\text{Ni}^{3+}$  state with a Jahn–Teller distorted  $\text{Ni}^{3+}\text{-O}$  octahedron. A hybrid device  
employing nanostructured  $\text{LiNi}_Y\text{Co}_{1-2Y}\text{Mn}_{1+Y}\text{O}_4$  /polymer electrolyte/nanoporous  
carbon black (NCB) powders was assembled. grant # W911NF-07-1-0426

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