Emulsion Detonation Synthesis (EDS): a novel method to produce nanostructured spinel  $LiMn_2O_4$  for Li-ion Batteries

R. Calinas<sup>1,2</sup>, Marisa Rodrigues<sup>1</sup>, João Calado<sup>1</sup>, P.J. Ferreira<sup>2</sup>

<sup>1</sup>Innovnano, Materiais Avançados, SA, 3040-570 Antanhol, Portugal

<sup>2</sup>Materials Science and Engineering Program, University of Texas at Austin, Austin, TX, 78712, USA

Lithium ion batteries are currently an attractive option for efficient transportation and stationary energy storage. However, the success of lithium ion technology requires the development of cathode materials, which exhibit high energy density, concomitant with low cost and high safety. Among the possible material options for the cathode, compounds of the type  $\text{LiMnO}_4$  (M=Mn, Ni,) are of great interest. In particular, the spinel  $\text{LiMn}_2\text{O}_4$  compound offers important advantages, namely low cost, good safety properties, high capacity and good chemical stability.

For the specific case of electric-vehicle (EV) applications it is necessary to have cathode materials of small particle size to provide a high interfacial area between the electrode and the electrolyte, as well as a short path length for Li-ion diffusion within the particle. However, large scale production of nano/submicron size cathode materials is still very challenging for complex metal oxides, which hinders the commercialization of this technology. In this regard, it is critical to develop novel production methods that are low cost and allow highthroughputs.

The emulsion detonation synthesis (EDS) shown herein is an industrial process patented by Innovnano, where the material is subjected to very high pressures (1-10 GPa). This allows novel nano/submicron crystalline structures to be produced with different properties, as well as a perfect mixture of different compounds in the same particle. In this work, we have applied the EDS method to the production of the spinel compound LiMn2O4. Although we have selected this compound due to the fact that it has a relatively simple structure and it is well documented in the literature, the EDS process can be easily adapted to produce other Li-ion battery materials.

To validate the EDS method, X-ray diffraction (XRD) was first used to confirm the structure of the LiMn2O4 compound. Subsequently, transmission electron microscopy (TEM) and scanning electron microscopy (SEM) was used to evaluate the detailed structure of the compound and the particle size. Both XRD and TEM show that the LiMn<sub>2</sub>O<sub>4</sub> compound exhibits a cubic spinel structure, while TEM/SEM analysis revealed an average particle size of 150 nm. In addition, inductively coupled plasma optical emission spectrometry (ICP - OES) showed a compound with the correct Li:Mn stoichiometry. In terms of electrochemical performance, the initial discharge capacity was evaluated as 115 mAh/g for 0.1C rate (1C = 148 mAh/g) and 85 mAh/g for 2C rate. The efficiency after 100 cycles is nearly 75% for all Crates tested.