## ABSTRACT

The synthesis by direct precipitation in aqueous medium has been adapted for the preparation of powders with theoretical composition  $\text{LiFe}_{1-y}\text{Mn}_y\text{PO}_4$  ( $0 \le y \le 1$ ), with a significant amount of structural defects. The as-made powders have been annealed in air at moderate temperature (~300 °C) in order to oxidize the iron present in the material, and thus create vacancies in the structure and unveil new structural and electrochemical properties.

The combination of ex situ and in situ techniques on the different  $LiFe_{1-y}Mn_yPO_4$   $(0 \le y \le 1)$  powders led to the elaboration of a structural model of the pristine and annealed materials. The different structural defects has been identified, and linked in particular to the low unit-cell volume observed.

The electrochemical signature of the pristine defective powders significantly differs from the reported one. The presence of a high amount of trivalent iron gives rise to the appearance of a new low-potential contribution. A model during the electrochemical charge and discharge of the different materials has been proposed.