Electronic Supplementary Material (ESI) for RSC Advances


# Supporting Information

## High voltage $LiMnPO_4$-$LiMn_2O_4$ nanocomposite cathode by one-pot pyro synthesis for Li-ion batteries

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# Experimental Section

***Material Preparation***: LMP-LMO powders were obtained by a polyol-assited pyro-synthetic method followed by mild annealing. The starting materials used for the present synthesis was lithium acetate ($C_2H_3LiO_2$, ≥99% - JUNSEI), manganese acetate ($Mn(CH_3COO)_2$, 97% - ADRICH), and phosphoric acid ($H_3PO_4$, ≥85% - DAE JUNG).

In brief, the starting precursors weighed out to the molar stoichiometric ratio of 2:3:1 (Li:Mn:P) corresponding to the combined stoichiometry of $LiMnPO_4$ and $LiMn_2O_4$ were introduced to 80 mL of tetraethylene glycol ($C_8H_{18}O_5$, 99.5% - DAEJUNG) and stirred for 24 hours at room temperature to obtain a homogenous solution. The polyol-fuel solution is then ignited by a torch to induce a self-extinguishable combustion process that facilitates the formation of a carbon-saturated amorphous LMP-LMO precursor. Subsequently, the as-prepared powder was annealed at 450 °C for 5 h in air to obtain the final LMP-LMO nanocomposite.

Commercially available pure $LiMn_2O_4$ sample purchased from Daejung, Korea and pure $LiMnPO_4$ prepared by the pyro-synthetic purposes using the same abovementioned precursors, which was used to make the present composite, weighed out according to the molar stoichiometric ratio of 1:1:1 for Li:Mn:P respectively was used for comparison purposes.

***Structural Chracterizations***: Powder X-ray diffraction data of the obtained samples were recorded using a Shimadzu X-ray diffractometer with Ni-filtered Cu Kα radiation (λ=1.5406 Å) operating at 40 kV and 30 mA within the scanning angle, 2θ, range of 10 - 80° in steps of 0.01°. The particle morphologies and sizes were determined by field emission–scanning electron microscopy (FE-SEM) using S-4700 equipment from HITACHI.

In order to investigate the thermal safety of the prepared sample, ex–situ differential scanning calorimeter (DSC) measurement was performed until 400 °C at a heating rate of 5 °C/min in a nitrogen atmosphere using a Q1000 (TA instruments, USA) at the Korea Electro-technology Research Institute (KERI). First the LMP-LMO electrode was charged at a current density of 0.02 mAh $g^{-1}$ and maintained at 4.5 V for 12 h during the 1st charge cycle. After the electrochemical reaction, the electrode was rinsed with dimethyl carbonate (DMC) in order to remove the remaining electrolyte and then dried in an Ar filled glove box at ambient temperature. The electrode was then separated from the stainless steel mesh current collectors and sealed in a vial in a glove box.

***Electrochemical characterization***: The electrochemical properties of the LMP-LMO samples were evaluated using lithium metal as the reference electrode. The prepared sample was mixed with 25 wt% of conducting carbon and polytetrafluoroethylene (PTFE) was used as binder. This mixture was pressed onto a stainless steel mesh and dried under vacuum at 150℃ for 10 h to form the cathode. The cathode was separated from the lithium metal anode by a glass fiber that contained propylene carbonate (EC) containing 1 M $LiPF_6$ as the electrolyte.

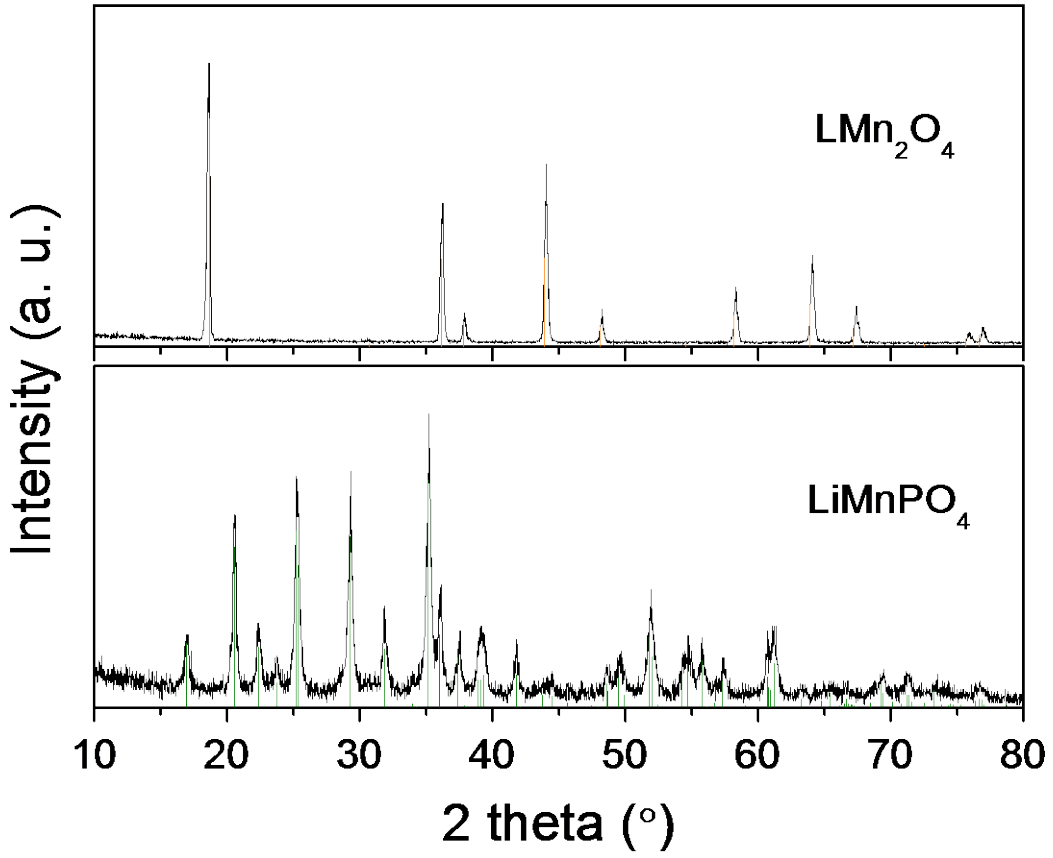


**Fig S1.** The XRD patterns of commercial $LiMn_2O_4$ (Daejung) and pure $LiMnPO_4$ prepared by pyro-synthesis.

For comparison, the XRD patterns of commercial $LiMn_2O_4$ (Daejung) and pure $LiMnPO_4$ prepared by pyro-synthesis are also provided in Figure S1. As anticipated, all the diffraction peaks of both the samples match well with their respective standard patterns.