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Synthesis of $LiM_xMn_{2-x}O_4$ Doped Co, Ni, Cr and Its Characterization as Lithium Battery Cathode

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Abstract : Manganese dioxide (MnO_2) and its derivatives are among the most widely used materials for the positive electrode in both primary and rechargeable lithium batteries. The MnO_2 derivative compound of $LiM_xMn_{2-x}O_4$ (M: Co, Ni, Cr) is one of the leading candidates for positive electrode materials in lithium batteries as it is abundant, low cost and environmentally friendly. Over the years, synthesis of $LiM_xMn_{2-x}O_4$ (M: Co, Ni, Cr) has been carried out using various methods including sol-gel, gas condensation, spray pyrolysis, and ceramics. Problems with these various methods persist including high cost (so commercially inapplicable) and must be done at high temperature (environmentally unfriendly). This research aims to: (1) synthesize $LiM_xMn_{2-x}O_4$ (M: Co, Ni, Cr) by reflux technique; (2) develop microstructure analysis method from XRD Powder $LiM_xMn_{2-x}O_4$ data with the two-stage method; (3) study the electrical conductivity of $LiM_xMn_{2-x}O_4$. This research developed the synthesis of $LiM_xMn_{2-x}O_4$ (M: Co, Ni, Cr) with reflux. The materials consisting of $Mn(CH_3COOH)_2$. $4H_2O$ and $Na_2S_2O_8$ were refluxed for 10 hours at 120°C to form β-MnO_2. The doping of Co, Ni and Cr were carried out using solid-state method with LiOH to form $LiM_xMn_{2-x}O_4$. The instruments used included XRD, SEM-EDX, XPS, TEM, SAA, TG/DTA, FTIR, LCR meter and eight-channel battery analyzer. Microstructure analysis of $LiM_xMn_{2-x}O_4$ was carried out on XRD powder data by two-stage method using FullProf program integrated into WinPlotR and Oscail Program as well as on binding energy data from XPS. The morphology of $LiM_xMn_{2-x}O_4$ was studied with SEM-EDX, TEM, and SAA. The thermal stability test was performed with TG/DTA, the electrical conductivity was studied from the LCR meter data. The specific capacity of $LiM_xMn_{2-x}O_4$ as lithium battery cathode was tested using an eight-channel battery analyzer. The results showed that the synthesis of $LiM_xMn_{2-x}O_4$ (M: Co, Ni, Cr) was successfully carried out by reflux. The optimal temperature of calcination is 750°C. XRD characterization shows that $LiMn_2O_4$ has a cubic crystal structure with Fd3m space group. By using the CheckCell in the WinPlotr, the increase of Li/Mn mole ratio does not result in changes in the $LiMn_2O_4$ crystal structure. The doping of Co, Ni and Cr on $LiM_xMn_{2-x}O_4$ (x = 0.02; 0.04; 0; 0.6; 0.08; 0.10) does not change the cubic crystal structure of Fd3m. All the formed crystals are polycrystals with the size of 100-450 nm. Characterization of $LiM_xMn_{2-x}O_4$ (M: Co, Ni, Cr) microstructure by two-stage method shows the shrinkage of lattice parameter and cell volume. Based on its range of capacitance, the conductivity obtained at $LiM_xMn_{2-x}O_4$ (M: Co, Ni, Cr) is an ionic conductivity with varying capacitance. The specific battery capacity at a voltage of 4799.7 mV for $LiMn_2O_4$; $Li_{1.08}Mn_{1.92}O_4$; $LiCo_{0.1}Mn_{1.9}O_4$; $LiNi_{0.1}Mn_{1.9}O_4$ and $LiCr_{0.1}Mn_{1.9}O_4$ are 88.62 mAh/g; 2.73 mAh/g; 89.39 mAh/g; 85.15 mAh/g; and 1.48 mAh/g respectively.

Keywords : $LiM_xMn_{2-x}O_4$, solid-state, reflux, two-stage method, ionic conductivity, specific capacity
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