Preparation and characterization of nanoscale $LiVMoO_6$ via soft-mechanochemical synthesis method

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This study is a continuation of our previous synthesis and structural characterization of lithium transitional-metal oxide $LiVMoO_6$, which recently attracted special attention as an electrode active material in rechargeable lithium ion batteries [1, 2]. Several methods for the $LiVMoO_6$ preparation are described in the literature, including solid state reaction and wet chemistry techniques (sol–gel, soft-combustion synthesis and rheological phase reaction). There is no data for the application of mechanochemical synthesis. Especially soft-mechanochemical synthesis method developed by Senna [3] posses some advantageous because highly reactive compounds containing oxygen-hydrogen groups are used as a precursors. A mixture of LiOH. H_2O, V_2O_5 and MoO_3 in 1:1:2 molar ratio of oxides were subjected to intense mechanical treatment in air for different periods of time using a planetary ball mill (Fritsch-Premium line-Pulversette № 7). Powder XRD data indicate the formation of a single phase $LiVMoO_6$ with brannerite-type structure after 30 min.milling time. The IR spectrum contains absorption bands characteristic for the Me_2O_8 (Me=V, Mo) units present in the crystal structure of $LiVMoO_6$. TEM and XRD reveal that the as-obtained $LiVMoO_6$ consists of crystallites mostly in the 25–50 nm size range with spherical shape. XPS analysis shows that $LiVMoO_6$ product contains vanadium and molybdenum ions in a higher oxidation state only - V^{+5} and Mo^{+6}, while the data of the EPR indicate the existence of traces of isolated VO^{2+} species in the as-prepared material.

References

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