



MILD HYDROTHERMAL SYNTHESIS, CRYSTAL STRUCTURE AND MORPHOLOGY OF LiV_2O_5

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ABSTRACT

Anode materials with high storage capacity and high power density attract considerable interest because of recent advances in Li-ion battery used electronic devices. Vanadium based oxides are the one possible group. LiV_2O_5 was synthesized by hydrothermal method at 3 days (72 hours) and 180 °C. Structural characterization was performed by powder X-ray diffraction, fourier transform infrared spectroscopy. Morphology was scanned by scanning electron microscopy with energy disperse X-ray analysis. The first time synthesized material is crystallized in orthorhombic system with unit cell parameters $a = 11.31$, $b = 9.552$, $c = 3.585$ Å and space group Pmn21 (31).

Indexing terms/Keywords

Hydrothermal synthesis, Lithium vanadium oxide, Anode materials, Electronic devices, LiV_2O_5 .

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Faculty of Science&Art, Chemistry

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1. INTRODUCTION

In past few decades, Li-ion batteries used in electronic devices attract considerable interest because of their anode materials with high storage capacity and high power density [1]. Nevertheless, there are some restrictions on the usage area. First stride is high costs resulting from the trashed battery which is unchargeable. The second one is in comparison of former type of lithium batteries show lower energy density and weaker low temperature performance. However, the restrictions are made in these latest areas, it is another factor to charge lithium batteries in military processes because, 'fast' charge capacity is demanded for many applications [2]. They have been applied in technological tools such as notebook, audiovisual equipments, office automation, and cellular phones. [3] However, they are not expanded sufficiently and are being developed evermore. There is a option to achieving higher capacities in Li-ion batteries is that using electrode materials is able to change oxidation state by two or more. The transition metal oxides having layered structure are the most appropriate materials for these types of batteries [4]. Vanadium based oxides are the one possible group [5]. The lithium ions are intercalated in vanadium oxide layers [3]. It is known that V_2O_5 , as a host, able to intercalate a maximum of 2 Li^+ ions per V_2O_5 reversibly resulting its orthorhombic structure and infinite layers of $[VO_5]$ parallel to (001) [6-8]. The intercalation of Li into V_2O_5 layers leads to the occupation of originally empty conduction band states [9]. The V_2O_5 exhibits higher performance in the electrochemical potential window between 3.6 and 1.5 V [10-13]. Lithium vanadate standouts on a large scale because of its high conductivity of Li^+ ions to obtain high cathode activity resulting good charge-discharge efficiency [14,15]. Also, studying crystalline vanadium based oxide with Li intercalation can be useful to remove properties without any interference from traditional electrode additives in the solid state electrode (SSE) [16].

2. EXPERIMENTAL

2.1. Synthesis of Lithium Vanadium Oxide

Analytical grade lithium chloride ($LiCl$, > 99 %) and vanadium pentaoxide (V_2O_5 , > 99 %) were purchased from Sigma Aldrich. Both chemicals were used without further purification. LiV_2O_5 was synthesized by dissolving lithium chloride and vanadium pentaoxide in ultra high pure water in an appropriate molar ratio. The homogeneous solution put into a stainless-steel Teflon autoclave and heated 3 days (72 hours) at 180 °C. The product was washed by high pure water for removing excessive material, dried at 70 °C for 2 hours and homogenized in an agate mortar.

2.2. Characterization Studies

X-ray powder diffraction analysis (XRD) was performed using PANalytical X'Pert PRO Diffractometer (XRD) with $Cu K\alpha$ (1.5406 Å, 45 kV and 30 mA) radiation. Fourier transform infrared spectroscopy (FTIR) was carried out Perkin Elmer Spectrum 100 FTIR Spectrometer from 4000 to 650 cm^{-1} . Morphological properties and semi-quantitative analyze (SEM/EDX) of the sample were realized by ZEISS Supra 40 VP. The Binder ED 53/E2 furnace and the hydrothermal container of Parr Instrument Company (USA) were used.

3. RESULTS AND DISCUSSION

The XRD of the sample was given in figure 1. The results of comparison of the XRD pattern with the standard ICDD (International Centre for Diffraction Data) correspond to LiV_2O_5 (ICDD = 89-8318). The material is crystallized in orthorhombic system with unit cell parameters $a=11.31$, $b=9.552$, $c=3.585$ Å and space group $Pmn21$ (31).

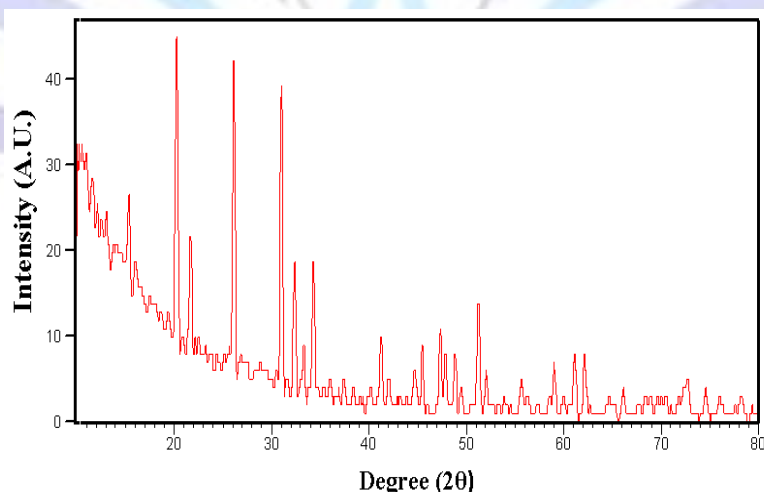


Figure 1. The XRD pattern of the sample. The results shows that $MgTi_2O_5$ (ICSD 157256) was form purely.

FTIR spectra of the product are presented in figure 2. The peaks at 783 and 990 cm^{-1} belong to V-O and V=O vibration frequencies, respectively [17,18].

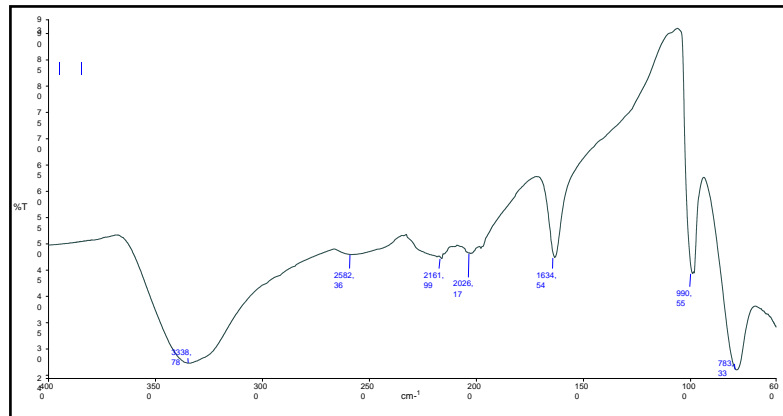


Figure 2. FTIR spectrum of LiV₂O₅.

Reference [18] shows that there is no significant change in 25-800 °C range. After higher degree, lithium vanadate started to decompose. LiV₂O₅ is relatively thermally stable, when we compare to the similar type of compounds.

In figure 3, SEM micrograph of LiV₂O₅ was displayed. The figure supports crystallization of the sample, and particle size measured between 0.5-2 µm.

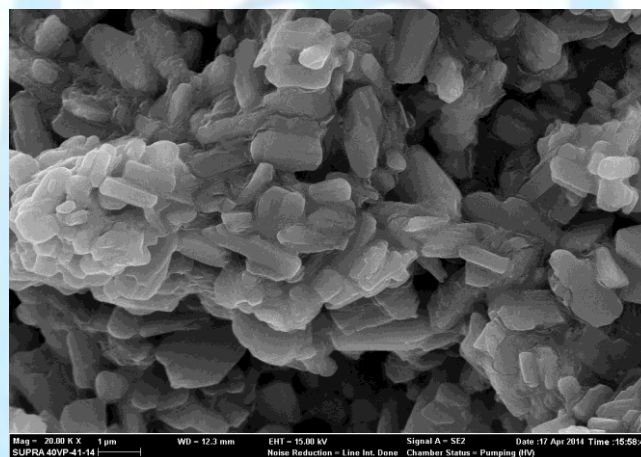


Figure 3. SEM micrograph of LiV₂O₅.

The results of EDX analyze is given in figure 4. Pink and green lines correspond with V and O, respectively. The small peaks near 2 and 5.5 keV become involved with platinum used covering the sample. Detection of lithium is not possible by EDX detector.

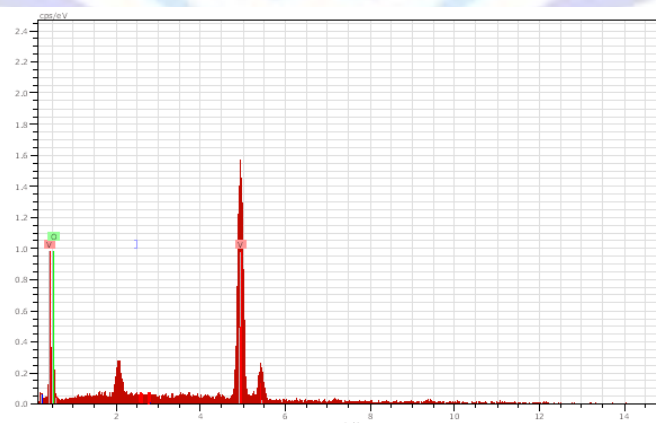


Figure 4. EDX result of LiV₂O₅.

4. CONCLUSION

By this work, we synthesized an electrode additive material in the solid state electrode, LiV₂O₅, by hydrothermal method firstly. Lithium vanadate is crystallized in orthorhombic system with unit cell parameters $a=11.31$, $b=9.552$, $c=3.585$ Å and space group Pmn21(31).



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