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Making and Characterization of Limnpo4 Using Solid State Reaction Method for Lithium Ion Battery Cathodes

Adelyna Oktavia, Kurnia Sembiring, Slamet Priyono
Department of Physics, University of Sumatera Utara, Medan, Sumatera Utara, Indonesia

ABSTRACT

Hospho-material of olivine, $LiMnPO_4$ identified as promising for cathode material generation next Lithium-ion battery and has been successfully synthesized by solid-state method with Li_2Co_3, $2MnO_2$, 2NH4H2PO4 as raw material. The influence of initial concentration of precursors at kalsinasi temperatures (400-800 ° C) flows with nitrogen. The purity and composition phase verified by x-ray diffraction analysis (XRD), scanning electron microscopy (SEM), spectroscopy, energy Dispersive x-ray Analysis (EDS), Raman spectra. General investigation shows that there is a correlation between the concentration of precursors, the temperature and the temperature of sintering kalsinasi that can be exploited to design lithium-ion next generation.

Keywords: Lithium-Ion, Synthesis, Olivine, Cathode.

I. INTRODUCTION

Technological developments, especially in the field of electrical energy storage, in the form of batteries. Electronic devices require batteries as a driving source, so the prospect of batteries is a strategic and economical source of energy. Batteries are devices that can generate electric current, involving the transfer of electrons through the negative electrode (anode) to the positive electrode (cathode) and because of the potential difference. Rechargeable batteries or rechargeable batteries now replace primary batteries because they save resources and are environmentally friendly. Examples of secondary battery types include Pb-acid, Ni-Cd and Li-ion. Among the examples of secondary batteries that are most developed at this time are lithium ion batteries.

The first cathode material used in lithium ion batteries is $LiCoO_2$. Then other cathode materials appear $LiNiO_2$, $LiMnO_4$, LiNi1 / 3Co1 / 3Mn1 / 3O2, and $LiFePO_4$. $LiFePO_4$ is extensively developed as a cathode material because of its moderate theoretical capacity (170 mAh / g), stable, inexpensive and environmentally friendly.

In making $LiMnPO_4$ powder using a solid state reaction reaction method where solid state reaction is a conventional synthesis method, which usually requires a two-step heating treatment, including first heating at a temperature of 300˚C - 400˚C and then temperature 600˚C - 800˚C. The solid state reaction method has become a technology developed and often used because it is easily synthesized and easily mass produced. The need for high temperatures unfortunately upgrades costs; What's more, the size of the product must always not be small. So, other actions are added to avoid the problem in processing olivine.

II. METHODS AND MATERIAL

A. Appliance and Materials Research

The composition of making $LiMnPO_4$ powder in this study was 15 grams. So that the mass of the main material needed is as shown in the following table.

Table 1. Mass of $LiMnPO_4$ raw materials

The main ingredient	Mass (gram)
Li_2CO_3	3,48
MnO_2	8,27
$NH_4H_2PO_4$	10,92

Preparation of $LiMnPO_4$ initial powder with a mixture of Li_2CO_3, MnO_2 and $NH_4H_2PO_4$ with a mass of 3.48 grams of Li_2CO_3, 8.27 grams of MnO_2 and 10.92 grams of $NH_4H_2PO_4$ into 20grams of powder which will be used to make $LiMnPO_4$ sheets the characteristics of $LiMnPO_4$ using the solid state reaction method with the testing stages namely DTA, CV, CD, SEM-EDX.

III. RESULTS AND DISCUSSION

A. Differential Thermal Analysis

On the graph of the DTA in the temperature range 50^{0}C-150^{o}C be curved down charts with peak disuhu around 150^{o}C which is the release of organic elements such as CO_2 and water vapor contained in the raw material, in the temperature range 150^{0}C-200^{0}C which is an endothermic reaction (requiring heat) Peak this is the process of decomposition of material due to the melting point of $NH_4H_2PO_4$, $NH_4H_2PO_4$ seen from material safety data that the melting point of the material is 190^{0}C, in the temperature range 300^{0}C-900^{o}C occurrence crystallization of LiMnPO4 while in the temperature range 900^{0}C-1000^{0}C suspected of such raw materials at a temperature of material already melted melting titih from each of which "the raw material is very small like Li_2CO_3:1300^{0}C/700^{0}C, $NH_4H_2PO_4$ MnO_2:500^{0}C and: 190^{0}C.

On the graph of the TGA range temperature 100^{0}C-occurring as much mass loss 400^{o}C-20.0735% dikarnakan the existence of a mass release of water vapor and decomposition of material strung on each subsequent temperature $NH_4H_2PO_4$, between 400^{0}C-550^{0}C the existence of a new phase of the formation occurs i.e. phases, While in the temperature range 550^{0}C – 900^{0}C the occurrence of crystallization LiMnPO4.

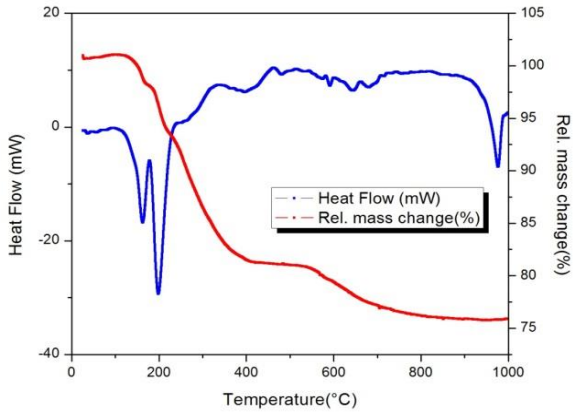

Figure 1. Graph of TTA from LiMnPO4 precursor powder

The derivatives curve shows the DTA LiMnPO4 precursors. The curve of the derivatives used to confirm changes phase motors. In the temperature range 100-and 200-200^{o}c 300^{o}c there are peaks which indicated changes phasa of raw materials caused by the loss of the organic elements. In the range 400-900°c there is no peak indicate the process of crystallization of $LiMnPO_4$. And the range of 900 to 1000^{o}c peak which indicates there has occurred a change of phase Motors $LiMnPO_4$ into another phase motors. Therefore in this study will be conducted at a temperature of 800^{o}C sinter.

B. XRD LiMnPO4 Analysis

Powder resulting from the synthesis of $LiMnPO_4$ show greyish black color. Identification of the establishment phase of the Lithium manganese posphat ($LiMnPO_4$) XRD analysis conducted on a sample test that has sintered at a temperature of 800^{0}C – 8 hours. Identification phase formed is done by comparing each angle θ 2 test results at a price of 2 θ JPDS of standards. With angle measurements from 10-800 XRD Analysis Results more information can be found in the Appendix.

Figure 2 shows the XRD patterns of the synthesis of $LiMnPO_4$ $LiMnPO_4$ compared to commercial and matched the pattern of the ICDD XRD $LiMnPO_4$ spectrum. The observations indicate that the results of XRD patterns of synthesis has the same pattern with peaks corresponding corner 2-theta the same and both are identic with the ICDD XRD patterns of $LiMnPO_4$. These peaks indicates system orthorhombic with space group pmnb. XRD peaks higher than synthesis of $LiMnPO_4$ commercial indicating that the material has a higher degree of crystallinity. This high level of crystallinity can be obtained due to a long sinter time up to 8 hours. But the longer the sintered will cause the growth of grain so that the powder synthesis of LiMnPo4 has a large particle size.

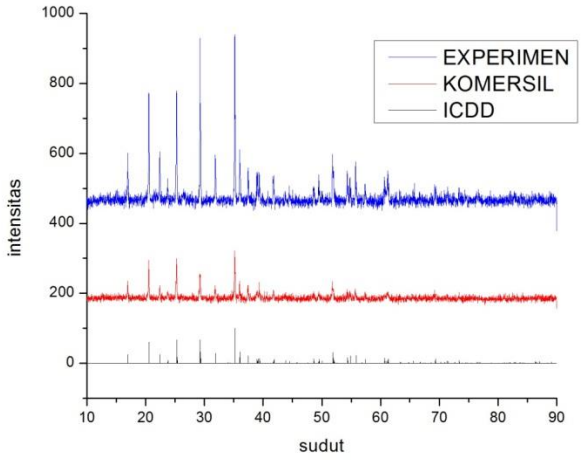

Figure 2. Results of comparison of Analysis of Commercial $LiMnPO_4$ powder and experimental $LiMnPO_4$.

C. SEM and EDX LiMnPO4 Analysis

The microstructure of $LiMnPO_4$ with sintered temperature 400^{0}c was nitrogen-fed for 1 hour and 4000c was nitrogen-fed for 4 hours observed with SEM. Figure 3 shows photos of SEM results obtained with variations in sintered 400^{0}c temperature flowed with nitrogen for 1 hour and 400^{0}c with nitrogen for 4 hours. Observation of enlargement is carried out at 200 to 500.

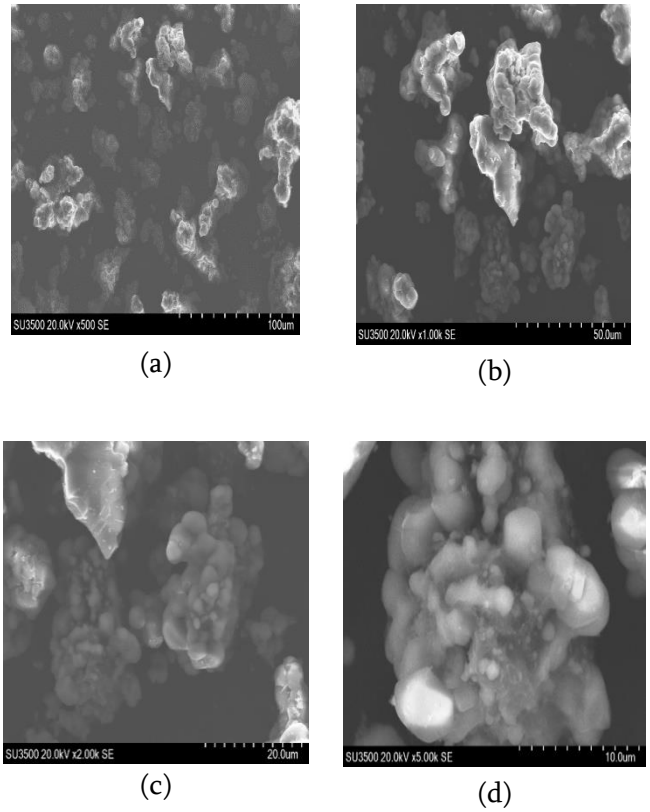

Figure 3. Photos SEM SE method LiMnPO4 enlargement done at 500, 1000, 2000 and 5000x.

Figure 4 shows a SEM/EDX $LiMnPO_4$ each elemental mapping mode. Li element can not be dimapping because their atomic weight that is very light. It appears that the elements Mn more evenly to all parts of the material because it has the highest atomic weight. While the elements oxygen and phosfat are present in some parts of the material doesn't look (there is a section of the poor oxygen and phosfat) this can be caused because the results of the miliing less

evenly. Overall that the distribution of each element (Mn, P, and O) are pretty evenly distributed and can be said to be homogeneous.

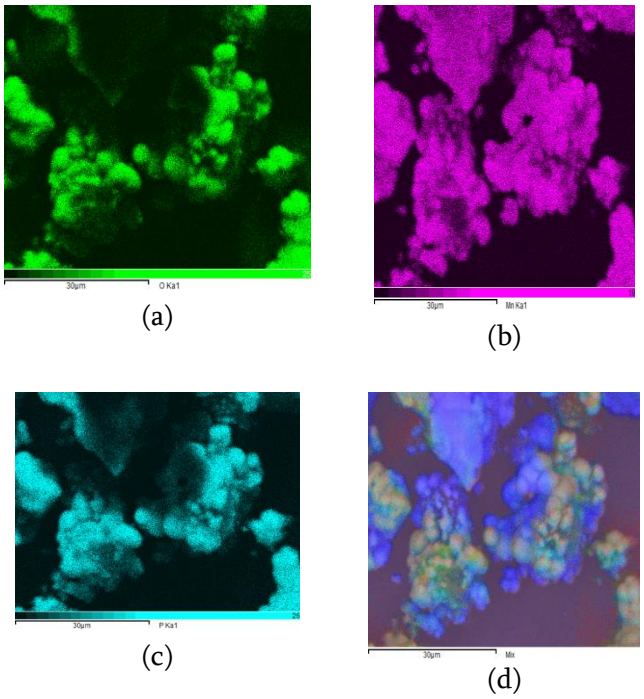

Figure 4. Photo SEMEDX $LiMnPO_4$ with the mapping element (a) oxygen (b) Manganese Phosfat, (c) and (d) Mix element

D. Cyclic Voltammetry (CV)

The results of Cyclic voltametry in the form voltamogram, i.e., the graph of the relationship between the voltage against the flow. Given the voltage on the battery cell range, and then measured the magnitude of the current case. Based on voltamagram can be known the existence of the peak peak reduction and oxidation. The battery cells showed a peak of oxidation and reduction then stated that the material is able to undergo the process of charge and discharge, this is a basic requirement as a secondary battery (recheargable). Voltamogram Cyclic Voltametry test results for samples of cathode.

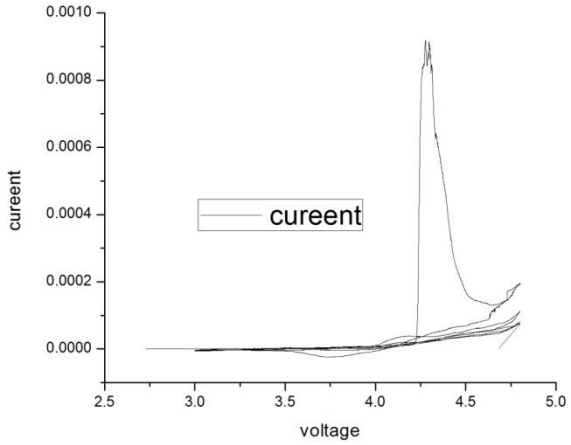

Figure 5. Cyclic Voltammetry (CV) LiMnPO4 test results

Based on the image can be seen the 5 voltamagram that the above samples are experiencing peak oxidation. Oxidation reactions can be seen on the graph, i.e. with an increase in positive flow. While the peak reduction of the sample above is not so noticeable, but that there remains only the peak value is too small. The existence of this reduction process can be seen on the graph, that is indicated by the presence of the negative currents. Peak reduction are low on the sample above shows that the Li + ions migrated to the anode during the charging process, not all of it back to the anode when discharging process. Based on the graph of cyclic voltammetry is can be said that the cathode LiMnPO4 capable of undergoing oxidation and reduction, although the peak value reduksinya is still very small. The small value of the peak reduction of this possibility because the cathode produced somewhat less pollutant than chemical structure used or wet (binds oxygen). This relates to the use of the preparation of cell battery, the electrolyte liquid electrolyte is used i.e. LiPF6, which can make the cathode LiMnPO4 became increasingly wet.

Cathode LiMnPO4 successfully undergoes a process of reduction and oxidation, to further assure that the cathode is capable of being used as a secondary

battery cells need further testing i.e. discharge-charge testing.

E. Charge – Discharge (CD)

Based on the graph of the test charge/discharge voltage value can be obtained for a sample of LMP (composition of MnO2 0%) amounting to 2.93 V,. Time of charge/discharge of the sample can also be known, namely the time charge and discharge for sample LMP was 8 minutes and 12 seconds. Graph testing charge/discharge can also show the value of the capacity of each sample LiMnPO4 glass cathode. Sample composition of MnO2 0% indicates the value of the capacity of 16 μAHr. Cathode glass LiMnPO4 made successful exhibit as a secondary battery for being able to experience the process of charge and discharge, however the cathode is not successfully demonstrate a good capacity value.

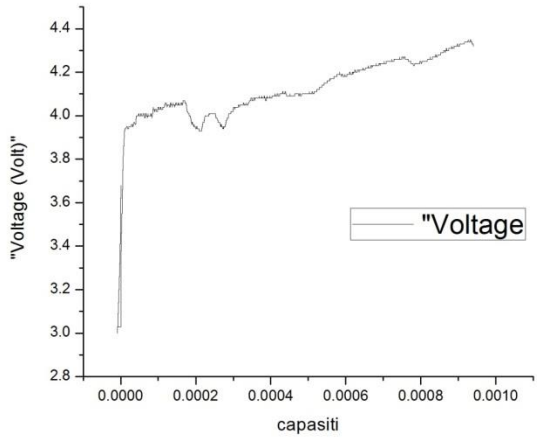

Figure 6. $LiMnPO_4$ Charge - Discharge (CD) test results

IV. CONCLUSION

From the research that has been done, the following conclusions can be drawn:

The LiMnPO4 cathode was successfully made using the method of solid state reaction with the percentage of material composition 3 Li2CO3: 3.478 grams, MnO2: 8.265 grams, NH4H2PO4: 10.925 grams. Cathode Structure LiMnPO4 is amorphous, but there is an impurity peak in the sample, which is thought to be the LiMnO4 phase. The optimum value of conductivity in the composition of 1.5% MnO2. The highest conductivity value is 7.25x10-4 S / cm, which is in the cathode sample with a composition of 1.5% MnO2. LiMnPO4 cathodes fulfill the requirements as a cathode in secondary batteries, which are characterized by peak oxidation and peak in cyclic voltammetry testing and the process of charge and discharge.

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