Solvothermal Synthesis of Lithium Iron Phosphate from a High Concentration Precursor

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Abstract—LiFePO 4 cathode material was synthesized using a solvothermal method at various relatively high concentration precursors. The samples were prepared using LiOH·H 2 O, FeSO 4 ·7H 2 O, H 3 PO 4, and citric acid which are dissolved in ethylene glycol and water mixture solvent with various concentration of LiOH from 0.3 to 1.8 M while the Li:Fe:P molar ratio was fixed at 3:1:1. The precursors were heated in an autoclave at fixed temperature of 180°C for 8 hours. The prepared samples were characterized using an X-Ray Diffraction and Scanning Electron Microscopy to study the crystal structures and morphology of the prepared particles, respectively. It was found that LiFePO 4 structures were observed at all various concentrations. However, a small part of crystal structure impurity was detected for the samples that fabricated at concentration 0.3 M and some coagulated part of sample from 1.5 M and 1.8 M. We found that the optimum precursor concentration for the present method and preparation condition was at 1.2 M of LiOH precursor.

Keywords—Cathode material, LiFePO 4, Lithium ion battery, Solvothermal

I. INTRODUCTION

Lithium Iron Phosphate (LiFePO 4) is a cathode material of lithium-ion battery with high theoretical capacity at 170 mAh/g. LiFePO 4 has advantages such as stable voltage, affordable price since the basic material (iron) is abundant in nature, no memory effect, and environment friendly. However, the material has low electronic conductivity and ion diffusion coefficients. There are various techniques to improve the performance and to increase the electronic conductivity such as carbon coating, metal coating, ion doping, and particle size optimization [1].

Various methods have been reported in the preparation of LiFePO 4 such as solid state [2], sol-gel [3], spray pyrolysis [4], microwave [5], carbothermal [6], hydrothermal [7], solvothermal [8], etc. Solvothermal is an interesting method because it produces a high purity material with a simple process. Moreover, by using the method it is easy to control the reaction process such as variation of heating temperature, pH, and heating time for improving materials performance.

The previous studies reported on the effect of temperature variation, heating time and pH of the precursor solution in solvothermal method [8]. However, the effects of precursor with high concentration and increasing the concentration of precursor have not been reported in detail. By increasing the concentration of precursor solution, a large quantity of material will be obtained, thus, will increase the efficiency and conserve the energy. In this study, a solvothermal synthesis of lithium iron phosphate in high concentration precursor using the solvothermal method is explored further.

II. EXPERIMENTAL

The precursor solutions were prepared by dissolving of LiOH·H 2 O, FeSO 4 ·7H 2 O, H 3 PO 4 and citric acid at molar ratio of 3:1:0.5 in 100 ml solvent consisting of ethylene glycol and water with a volume ratio of 3:2. Citric acid was used to minimize the oxidation of Fe 2+ to Fe 3+. The precursors were put into autoclave and heated at 180°C for 8 hours with stirring process. Furthermore, the solution was naturally cooled and centrifuged for 30 min at 2500 rpm. The precipitate was then washed using a distilled water, filtered, and dried at 70°C for 30 min. The schematic of synthesis LiFePO 4 is showed in Fig 1. In this experiment, the precursor concentration was varied as given in Table 1.

![Fig. 1 Flowchart of LiFePO 4 synthesis process.](image-url)