Synthesis of Nano FePO₄ in Novel Rotating Packed Bed Reactor

Weihua Pu, Yulin Wu, Jianguo Ren, Xiangming He
Institute of Nuclear & New Energy Technology, Tsinghua University, Beijing 100084, PR China.

Introduction

Recently, some research results have shown the nano-sized LiFePO₄ materials have good C-rate performance and low temperature performance. The preparation methods which are easily controlling size and morphology, large scale and lower cost for the nano-sized precursory such as FePO₄, Li₂CO₃, and LiFePO₄ materials become the research issues. These are also the bases of nano LiFePO₄ materials vast application.

Experimental

High gravity technology, also called Higee technology, carried out in a rotating packed bed (RPB) reactor, was originally invented in 1979 for the separation processes. The technology was also employed to synthesize various kinds of nano materials in liquid-liquid, gas-liquid and gas-liquid-solid reactant systems. Compared with the conventional precipitation process which carried out in stirred tanks or column reactors, the High gravity technology can guarantee the quality of the products and control the morphology, size and size distribution of the particles due to the presence of a sharp supersaturation interface accompanied by the very short liquid residence time in the RPB. In this paper, nano-sized FePO₄ particles were synthesized in the RPB reactor as Fig.1. The preparation reactions can be written as:

\[
\text{Fe(NO}_3\text{)}_3(l) + \text{H}_3\text{PO}_4(l) \rightarrow \text{FePO}_4(s) + 3\text{HNO}_3(l)
\]

Fe(NO₃)₃ and H₃PO₄ liquid reactants are pumped into RPB simultaneously. Meanwhile, NH₃·H₂O was also fed into the reactor to adjust the pH of the reaction. Both of solutions enter the packed section of RPB through two separate liquid distributors, vigorously mixing and reacting in the packing to form a mixing solution with FePO₄·2H₂O particles via a outlet into the products tank. The result production was then separated, washed and dried to obtain the spherical FePO₄·2H₂O powders. Nano FePO₄ was prepared by heating FePO₄·2H₂O at 400°C for 6h in air.

Results and Discussion

The particle size of FePO₄ materials is range from 5nm to 60nm by controlling the appropriate reaction parameters. Typical TEM micrographs of nano-sized FePO₄ are illustrated in Fig.2. These nanoparticles exhibit uniform particle size distribution and good dispersion. The performance of nano LiFePO₄ prepared the nano FePO₄ are investigating.

Fig.2 Typical TEM micrographs of nano FePO₄

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Reference