Industrial by-products as precursors for gas-phase nanoparticle synthesis

T. Karhunen¹, A. Lähde¹, T. Torvela¹, J. Jokiniemi^{1,2}

¹Fine Particle and Aerosol Technology Laboratory, University of Eastern Finland, Kuopio, 70211, Finland

²Fine Particles Team, Technical Research Center of Finland, Espoo, 02044 VTT, Finland

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Presenting author email: tommi.karhunen@uef.fi

The structure of global energy supply is changing rapidly towards sustainable energy solutions. However, all forms of sustainable and intermittent energy production (e.g. wind power, photovoltaics) and sustainable transportation (e.g. hybrid and full electric vehicles) require excellent energy storage systems in order to increase their effectiveness (Hall and Bain, 2008).

Li-ion secondary cells are one of the most advanced battery energy storage systems currently available due to their high energy density (Du Pasquier *et al*, 2003). One of the main obstacles in their wide scale adoption, however, is the high cost of such batteries.

This obstacle can be overcome, firstly, by increasing the operational life time of the batteries. LiFePO₄ is a very promising electrode material for achieving this. In proper combination with an conductive additive it can be used to make Li-ion batteries that can undergo at least 1000 charge/discharge cycles without significant degradation in performance (Song *et al*, 2011). Furthermore, LiFePO₄ and its precursors are environmentally friendly.

Secondly, the cost of the precursor materials can be reduced by utilising alternative sources, such as industrial by-products. At least iron and phosphorus are available in the industrial waste streams. Utilising these streams will also reduce the environmental impact of the production.

Lastly, the synthesis process itself can be optimised. Gas-phase processes are highly material and energy efficient routes for synthesis of nanoparticles with controlled composition and particle size. With the proper selection of the process environment the waste streams can also be easily utilised in these processes.

In this paper we present a gas-phase method for the synthesis of LiFePO4 nanoparticles utilising industrial waste stream of FeSO4·7H2O. The precursors were dissolved in water and atomized using an ultrasonic nebuliser. The produced droplets were then carried to the heated zone of the reactor. Experiments were carried out at temperatures between 200 and 1000 °C in atmospheres with varying reduction potentials. The aerosol exiting the heated zone was rapidly cooled with a large volume of dilution gas (air or N2) at ambient temperature.

With increasing temperature the particles became more solid with increasing fraction of LiFePO₄ (Figure 1). Increasing the reducing potential of the synthesis atmosphere increases the ratio of the Pnma-to-Cmcm polymorphs of LiFePO₄ (Figure 2). The effects of temperature, precursor composition and process atmosphere on the product composition will be presented.



Figure 1: TEM images of LiFePO₄ samples synthesised at (a) 800° C and (b) 600° C.



Figure 2: XRD diffractiograms of LiFePO₄ samples synthesised in N2 (red) and 8%H2/N2 (black).

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