Novel Hydrothermal Synthesis of Nano-$LiFePO_4$ via Solution Steering

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Among the various methods developed for the preparation of $LiFePO_4$ (LFP) cathode materials, the hydrothermal synthesis offers a simple solution to the practical and scale-up problems [1-2]. The hydrothermal/solvothermal approach is particularly successful to control the chemical composition and crystallite size. The conventional hydrothermal process involves a reaction time 5-12 h to synthesize LFP with the advantage of synthesis temperature as low as 180°C. It is thus a low energy consuming synthesis process.

In this work, LFP cathode materials were prepared by hydrothermal method assisted by steering that maintained the agitation of the solution during the sample synthesis. Structural identification, surface morphology and electrochemical cycling are reported for two samples, one synthesized by conventional hydrothermal process, the other one assisted by steering at 600 rpm, for comparison. In both cases the same types and ratios of precursors were used, namely: $LiOH{\bullet}H_2O$, $FeSO_4{\bullet}7H_2O$, H_3PO_4 (85 wt.%) and ascorbic acid as carbon source in stoichiometric molar ratios of Li, Fe, P and C (3:1:1:0.2). Annealing was done at 700°C under nitrogen atmosphere as previously described in [2].

The net result of the hydrothermal synthesis of $LiFePO_4$ via solution steering is twofold (i) formation of nano-particles (<50 nm) and (ii) narrow grain size distribution. Fig. 1 shows the SEM images of LFP particles prepared by (a) the conventional and (b) the rotating-assisted hydro-thermal methods.

The electrochemical properties were analyzed in the cut-off voltage range 2-4.0 V vs. Li at room temperature and at 55 °C (Fig. 2). The LFP material synthesized via the rotating method retains more than 98% of the coulombic efficiency even after 20 cycles at C/20 rate. The hydrothermal synthesis of nano-$LiFePO_4$ via solution steering is very effective in producing smaller cathode particles at high yield with very good capacity retention characteristics for use in rechargeable Li-ion batteries.

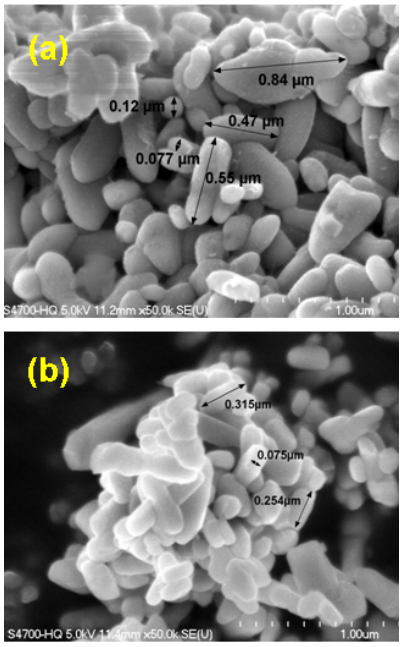

FIG. 1 SEM images of LFP particles prepared by (a) the conventional and (b) the rotating-assisted hydrothermal methods.

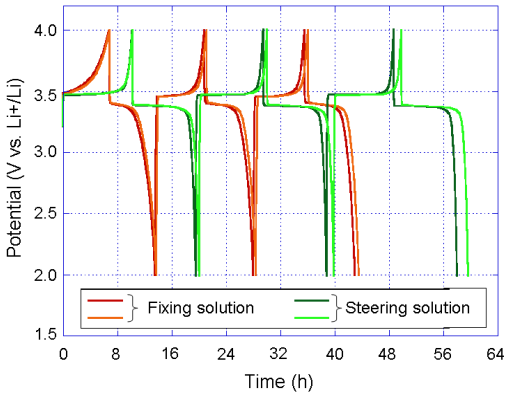

FIG.2. Charge-discharge profiles of Li//LFP cells with cathode materials synthesized by conventional and rotating hydrothermal routes. The electrolyte was 1.0 $mol.L^{-1}$ $LiPF_6$ in a mixture of ethylene carbonate (EC) and diethyl carbonate (DEC) (1:1, v/v).

References

[1] K. Dokko, K. Shiraishi, K. Kanamura, J. Electrochem. Soc. 152 (2005) A2199.
[2] F. Brochu, A. Guerfi, J. Trottier, M. Kopec, A. Mauger, H. Groult, C.M. Julien, K. Zaghib, J Power Sources 214 (2012) 1.