Pengaruh penambahan karbon dan suhu kalsinasi pada sintesis hidrotermal lifepo4 sebagai material katoda baterai lithium ion = The effect of carbon addition and calcination temperature on hydrothermal synthesis of lifepo4 used as lithium ion battery cathode material

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Abstrak

Telah dilakukan sintesis LiFePO4/C sebagai material katoda baterai lithium ion dengan menggunakan metode hidrotermal dari bahan LiOH, NH4H2PO4, FeSO4.7H2O, carbon black dan sukrosa. Proses hidrotermal dilakukan pada suhu reaktor 180C dengan lama waktu penahanan 20 jam. Penambahan karbon dilakukan dengan 2 cara. Pertama menggunakan sukrosa sebagai sumber karbon yang dilarutkan bersama prekusor dan kedua menggunakan carbon black yang ditambahkan setelah proses hidrotermal sebelum proses kalsinasi. Temperatur kalsinasi divariasikan pada 500, 600 dan 750C selama 5 jam. Proses dekomposisi termal dianalisis menggunakan DTA-TGA analyzer, karakterisasi fasa dilakukan dengan XRD, morfologi dengan SEM/EDX, nilai konduktifitas dan kapasitansi material dengan LCR-EIS, dan performa baterai dengan pengujian charge-discharge menggunakan baterai analyzer. Hasil LiFePO4/C yang murni berbentuk flake berhasil disintesis dengan penambahan carbon black 5 wt%, sedangkan untuk penambahan karbon melalui pelarutan sukrosa masih terdapat pengotor Fe3(PO4)2 pada hasil kalsinasi. Temperatur kalsinasi optimal adalah 750C dengan ukuran kristalit 39,7 nm, tebal butiran flake 80 nm dan besar butiran rata-rata 427 nm. Konduktifitas LiFePO4 murni terukur 5 x 10-7 S/cm dan konduktifitas LiFePO4/C adalah 2,23 x 10-4 S/cm yang dihasilkan dari sampel dengan tambahan carbon black 5wt% kalsinasi 750C. Dari pengujian charge/discharge didapatkan siklus terbaik dihasilkan oleh sampel LiFePO4/C yang dikalsinasi 750C yang stabil dengan tegangan 3,3-3,4 V, kapasitas spesifik dihasilkan pada 0,1 C = 11,6 mAh/g ; 0,3C = 10,78 mAh./g dan 0,5 C = 9,45 mAh/g.

.....LiFePO4/C has been succesfully synthesized through hydrothermal method from LiOH, NH4H2PO4, and FeSO4.7H2O as starting materials and either carbon black or sucrose as carbon source used as cathode material for lithium ion batteries. In this work, hydrothermal reaction temperature was at 180C for 20 hours.Carbon sources were added in two routes. Firstly, sucrose solution was mixed with precursor solution before hydrothermal reaction. Secondly carbon black was added after hydrothermal reaction before calcination process. Calcination temperatures were performed at 500, 600, and 750C each for 5 hours. Thermal decomposition process was analyzed using DTA-TGA analyzer, phases and morphological were characterized by using XRD and SEM/EDX measurement, conductivity and electrical capacity were characterized by EIS measurement, and batteries performance were tested with charge discharge testing by battery analyzer. Pure LiFePO4/C flake shaped was successfully synthesized with the addition of 5 wt% carbon black, while the addition of carbon through the dissolution of sucrose still contained impurity from Fe3(PO4)2 in calcination product. Optimal calcination temperature was obtained at 750C with crytallite size of 39.7 nm, flake particles diameter of 80 nm with particles average length of 427 nm. Pure LiFePO4 conductivity was measured to be 5 x 10-7 S/cm and conductivity LiFePO4/C was 2.23 x 10-4 S/cm produced from samples with carbon black addition of 5 wt% and calcined at 750C. Charge/discharge cycles test showed that best battery performance was obtained from the sample with carbon black of 5wt% calcined

at 750C, with a stable voltage 3.3 to 3.4 V, specific capacity of 0.1 C = 11.6 mAh/g ; 0.3C = 10.78 mAh./g dan 0.5 C = 9.45 mAh/g.