

Pengaruh penambahan karbon dan suhu kalsinasi pada sintesis hidrotermal LiFePO_4 sebagai material katoda baterai lithium ion = The effect of carbon addition and calcination temperature on hydrothermal synthesis of LiFePO_4 used as lithium ion battery cathode material

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Abstrak

Telah dilakukan sintesis LiFePO_4/C sebagai material katoda baterai lithium ion dengan menggunakan metode hidrotermal dari bahan LiOH , $\text{NH}_4\text{H}_2\text{PO}_4$, $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, carbon black dan sukrosa. Proses hidrotermal dilakukan pada suhu reaktor 180°C dengan lama waktu penahanan 20 jam. Penambahan karbon dilakukan dengan 2 cara. Pertama menggunakan sukrosa sebagai sumber karbon yang dilarutkan bersama prekursor dan kedua menggunakan carbon black yang ditambahkan setelah proses hidrotermal sebelum proses kalsinasi. Temperatur kalsinasi divariasikan pada 500 , 600 dan 750°C 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 LiFePO_4/C yang murni berbentuk flake berhasil disintesis dengan penambahan carbon black 5 wt%, sedangkan untuk penambahan karbon melalui pelarutan sukrosa masih terdapat pengotor $\text{Fe}_3(\text{PO}_4)_2$ pada hasil kalsinasi. Temperatur kalsinasi optimal adalah 750°C dengan ukuran kristalit 39,7 nm, tebal butiran flake 80 nm dan besar butiran rata-rata 427 nm. Konduktifitas LiFePO_4 murni terukur $5 \times 10^{-7} \text{ S/cm}$ dan konduktifitas LiFePO_4/C adalah $2,23 \times 10^{-4} \text{ S/cm}$ yang dihasilkan dari sampel dengan tambahan carbon black 5wt% kalsinasi 750°C . Dari pengujian charge/discharge didapatkan siklus terbaik dihasilkan oleh sampel LiFePO_4/C yang dikalsinasi 750°C 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.

..... LiFePO_4/C has been succesfully synthesized through hydrothermal method from LiOH , $\text{NH}_4\text{H}_2\text{PO}_4$, and $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ 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 180°C 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 750°C 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 LiFePO_4/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 $\text{Fe}_3(\text{PO}_4)_2$ in calcination product. Optimal calcination temperature was obtained at 750°C with crytallite size of 39.7 nm, flake particles diameter of 80 nm with particles average length of 427 nm. Pure LiFePO_4 conductivity was measured to be $5 \times 10^{-7} \text{ S/cm}$ and conductivity LiFePO_4/C was $2.23 \times 10^{-4} \text{ S/cm}$ produced from samples with carbon black addition of 5 wt% and calcined at 750°C . 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.