SYNTHESIS AND CHARACTERIZATION OF MANGANESE OXIDE BASED MATERIALS AS NEGATIVE ELECTRODES FOR LITHIUM ION BATTERIES

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This thesis is written with the aim of exploring and investigating several types of materials with a particular focus on the microstructures, morphology and their electrochemical properties. The concept of integration two types of transition metal oxides (Mn_3O_4, Fe_2O_3) have been implemented due to several advantages which makes it a promising compound to be applied in the lithium (Li) ion battery. Also, with further carbon layering process which was reported could buffer the volume changes during the Li^+ insertion of the Li ion battery. Additionally, the effects of the preparation route, which can lead to small particle size, and excellent morphology, also could automatically fulfill the requirements on the anode materials in this field. Both pure Mn_3O_4 and Mn_3O_4−Fe_2O_3 composite were being synthesized via a simple molten salts method. Mn_3O_4−Fe_2O_3 composite delivered much better electrochemical performance compared to the pure Mn_3O_4 sample where the discharge capacity was calculated to be

538 mAh g^{-1} after 50^{th} cycles, which is higher than the pure sample which was calculated to be 240 mAh g^{-1} at the similar cycle. The excellent electrochemical performance of the Mn_3O_4–Fe_2O_3 sample might be due to addition of another transition metal oxide which could boost the reaction of ions in electrochemical reduction and oxidation processes. Three different weight ratios of Mn_3O_4–Fe_2O_3 composite electrodes which labelled; Sample A (80:10:10), Sample B (75:10:15) and Sample C (70:10:20) were being tested to identify the effect of activated carbon on the electrochemical performance. The sample contained 15% of activated carbon presents much better electrochemical performance compared to the other two samples of 20 and 10%; respectively. The discharge capacity of 15% of activated carbon sample was calculated to be 538 mAh g^{-1}, which is higher than the sample of 20% (405 mAh g^{-1}) and 10% (55 mAh g^{-1}) after 50^{th} cycles. In introducing an amorphous carbon through a carbon layering process, Mn_3O_4–Fe_2O_3 composite was then coated with carbon where malic acid was used as the carbon source. SEM and TEM images confirmed that both uncoated and carbon coated samples consisted of very fine particles in the range of 10-30 nm. The Mn_3O_4–Fe_2O_3–C sample demonstrates a better first discharge capacity of about 549 mAh g^{-1} compared to the uncoated sample (417 mAh g^{-1}) after 50^{th} cycles. All these results concluded that carbon coating on Mn_3O_4–Fe_2O_3 could not only significantly improve the kinetic of the Mn_3O_4–Fe_2O_3–C electrode, but also can effectively buffer the volume expansion and sustain the structural integrity of the composite sample.

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SINTESIS DAN PENCIRIAN TERHADAP BAHAN BERASASKAN MANGAN OKSIDA SEBAGAI ELEKTROD NEGATIF BAGI BATERI ION LITIUM

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Tesis ini ditulis bertujuan untuk meneroka dan menyiasat beberapa jenis bahan dengan tumpuan yang khusus kepada struktur mikro, morfologi dan sifat-sifat elektrokimia bahan tersebut. Konsep penggabungan dua jenis logam oksida peralihan (Mn_3O_4, Fe_2O_3) telah dilaksanakan disebabkan beberapa kelebihan yang menjadikan ia sebatian yang berkeupayaan untuk digunakan di dalam bateri ion litium. Juga, dengan proses pelapisan karbon yang dilaporkan dapat menghalang pengembangan isi padu semasa proses pemasukan Li^+ ion di dalam bateri ion litium. Selain daripada itu, kesan laluan penyediaan, yang boleh membawa zarah yang bersaiz kecil dan morfologi yang sangat baik, secara automatik dapat memenuhi keperluan untuk menjadi bahan anod di dalam bidang ini. Kedua-dua Mn_3O_4 yang tulen dan $Mn_3O_4-Fe_2O_3$ komposit telah disintesis melalui kaedah garam lebur mudah. $Mn_3O_4-Fe_2O_3$ komposit menunjukkan prestasi elektrokimia yang jauh lebih baik berbanding sampel Mn_3O_4 yang tulen di mana kadar kapasiti nyahcas yang ditunjukkan adalah 538 mAh g^{-1} selepas kitaran yang ke-50,

berbanding sampel yang tulen iaitu 240 mAh g^{-1} dalam kitaran yang sama. Mn_3O_4–Fe_2O_3 komposit menunjukkan prestasi elektrokimia yang cemerlang mungkin disebabkan oleh penambahan logam oksida peralihan yang lain yang membantu meningkatkan kadar tindak balas ion dalam proses elektrokimia pengurangan dan pengoksidaan. Tiga elektrod bahan Mn_3O_4–Fe_2O_3 komposit telah dihasilkan dengan nisbah berat bahan yang berbeza yang dilabel sebagai Sampel A (80:10:10), Sampel B (75:10:15) dan Sampel C (70:10:20) telah diuji untuk melihat kesan nisbah karbon aktif terhadap prestasi elektrokimia bahan komposit tersebut. Sampel mengandungi 15% karbon aktif menunjukkan prestasi elektrokimia yang lebih baik berbanding sampel mengandungi 20 dan 10% karbon aktif. Selepas kitaran yang ke 50, bacaan kapasiti nyahcas sampel yang mengandungi 15% karbon aktif adalah 538 mAh g^{-1}, dimana lebih tinggi berbanding sampel mengandungi 20% (405 mAh g^{-1}) dan 10% (55 mAh g^{-1}). Dalam memperkenalkan karbon amorfus melalui proses pelapisan karbon, Mn_3O_4–Fe_2O_3 komposit telah disalut dengan karbon di mana asid malic digunakan sebagai sumber karbon. Melalui imej SEM dan TEM, kedua-dua sampel tidak bersalut karbon dan bersalut karbon terdiri daripada zarah yang sangat halus dalam lingkungan saiz di antara 10-30 nm. Selepas kitaran yang ke 50, sampel Mn_3O_4–Fe_2O_3–C menunjukkan bacaan kapasiti yang lebih baik iaitu 549 mAh g^{-1} berbanding dengan sampel yang tidak bersalut karbon (417 mAh g^{-1}). Kesimpulannya, lapisan karbon pada Mn_3O_4–Fe_2O_3 bukan sahaja dapat meningkatkan tenaga kinetik elektrod Mn_3O_4–Fe_2O_3–C, tetapi juga mampu menghalang pengembangan isi padu, dan mengekalkan integriti struktur di dalam sampel komposit tersebut.