UNIVERSITY TEKNOLOGI MARA

SYNTHESIS AND CHARACTERIZATION OF LiMn_{(2-x)}Fe_{x}O_{4} CATHODIC NANO MATERIAL FOR ADVANCED LITHIUM-ION BATTERIES

AIDA FAZLIZA MAT FADZIL

Thesis submitted in fulfillment of the requirements for the degree of Master of Science

Faculty of Applied Sciences

April 2007
Candidate's Declaration

I declare that work in this thesis was carried out in accordance with the regulations of University Teknologi MARA. It is original and is the results of my work, unless otherwise indicated or acknowledged as referenced work. This thesis has not been submitted to any other academic institution or non-academic institution for any other degree or qualification.

In the event that my thesis be found to violate the conditions mentioned above, I voluntarily waive the right of conferment of my degree and agree to be subjected to the disciplinary rules and regulations of University Teknologi MARA.

Name of Candidate          Aida Fazliza Mat Fadzil
Candidate’s ID No.         2004188448
Programme                  Advanced Material
Faculty                    Faculty of Applied Sciences
Thesis Title               Synthesis and Characterization of \( \text{LiMn}_{2-x}\text{Fe}_x\text{O}_4 \) Cathodic Nano Material for Lithium-ion Batteries

Signature of Candidate
Date                        29 April 2007
Abstract

New lithium transition metal oxides, LiMn_{(2-x)}Fe_xO_4 where 0 < x ≤ 0.5 are first synthesized using the sol-gel method. Stoichiometric samples are prepared. Thermogravimetric analysis (TGA) are carried out on the precursors to study the thermal properties and decomposition of the samples. The precursors are later annealed at 850°C for 24 hours. X-Ray diffractions (XRD) of the samples were taken to examine the crystallinity of the materials and to check the purity of the products. Field Emission Scanning Electron Microscope (FESEM)/Energy Dispersive X-Ray Analysis (EDX) are carried out to observe the changes in surface morphology and to check the stoichiometry. Cyclic Voltammetry (CV) is carried out to determine the characteristics of the electrical potential of the materials. Fabrication of the cathodes and testing of the batteries are later done. The best two materials are then processed to become nano size and were characterized using XRD, FESEM/EDX, particle size analyzer and CV. Fabrication of the batteries are carried out in an argon filled glove box. Batteries fabricated were then characterized for their electrochemical characteristics using the Multistat Solartron 1480 and the results obtained from both bulk and nano materials were compared.
Acknowledgement

I would firstly like to express my sincere appreciation to my research supervisor, Assoc. Prof. Dr Norlida Kamarulzaman. I wish to express my deep gratitude to my co-supervisor, Assoc. Prof. Dr Ri Hanum Yahaya Subban for her great assistance, academic guidance and constant encouragement during my studies and throughout the project.

Special thanks are given to Dr Mohamad Azni Bustam, University Technology Petronas for providing enormous help and guidance especially in the technical parts. Also particularly to Dr Mohd Zu Azhan for allowing me to use the glove box. My thanks also go to all the staff members at the Institute of Science and to all the technicians at the Faculty of Applied Sciences and Combicat, UM for utilizing the facilities and instruments. Lastly, to my lab mates, Sha and Ila for helping me through the hard and bad time to finish this thesis.

Finally, I wish to express my deepest appreciation to my parents, Tuan Haji Mat Fadzil b Ahmad and Hajjah Siti Aminah bt Mohd Noor and gratitude to my sisters, Ida, Ina and Anita, brother, Adi my Ateh, and my bestfriends, Azrul Firdaus, Farah and Ameir for their encouragement, understanding, patience and endless love during my studies.

Aida Fazliza Binti Mat Fadzil
University Technology MARA
January 2007
# TABLE OF CONTENTS

<table>
<thead>
<tr>
<th>Section</th>
</tr>
</thead>
<tbody>
<tr>
<td>Title page</td>
</tr>
<tr>
<td>Abstract</td>
</tr>
<tr>
<td>Acknowledgements</td>
</tr>
<tr>
<td>Table of Contents</td>
</tr>
<tr>
<td>List of Tables.</td>
</tr>
<tr>
<td>List of Figures.</td>
</tr>
</tbody>
</table>

## Chapter 1  Introduction

1.1 Introduction To Batteries.                                           1
1.2 Market Review of Lithium-ion Batteries.                                3
1.3 Objectives of Research.                                               6

## Chapter 2  Literature Review

2.1 Introduction to Lithium-Ion Batteries.                                 7
2.2 The Historical Development of Batteries.                              10
2.3 The Principle of Cells.                                                14
2.4 Electrode materials for Lithium-Ion Batteries.                         16
   2.4.1 Cathode Materials                                                16
       (i) Li-Ni / Co-O compounds                                          16
       (ii) Li-Mn-O spinels                                               20
   2.4.2 Anode Materials                                                 23
2.5 Electrolyte for Lithium-Ion Batteries                                  24
2.6 Sol-Gel Technique.                                                    28
2.7 Development of Nanotechnologies.                                      31