



UNIVERSITI PUTRA MALAYSIA

**PREPARATION AND CHARACTERIZATION
OF LITHIUM-BASED SOLID STATE BATTERY MATERIALS**

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STATE BATTERY MATERIALS**

By

MUHAMMAD AMIN IDREES

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Faculty: Institute of Advanced Technology

Three different cathodes such as LiMn_2O_4 , LiCoO_2 and $\text{Li}_2\text{Ni}_8\text{O}_{10}$ were synthesized by the sol-gel technique. The said materials at low temperature were achieved through the low temperature technique. The X-ray diffraction study of the compounds confirmed the formation of single-phase compound at higher calcination temperature. At low temperatures the X-ray diffractogram of the samples showed the presence of low intensity diffraction lines with weak impurities indicating the existence of crystallinity but these were not indexed to any kind of impurities of LiMn_2O_4 , LiCoO_2 and $\text{Li}_2\text{Ni}_8\text{O}_{10}$. The formation temperatures of the compounds were analyzed using DTA. The DTA studies showed clearly the lowest formation temperature and this formation temperature depends upon the gelating agent used in the present study. The lowest formation temperatures recorded were 208°C for LiMn_2O_4 , 201°C for LiCoO_2 and 214°C for $\text{Li}_2\text{Ni}_8\text{O}_{10}$.



The thermogravimetric analysis showed that the compounds were stable up to 800 °C. The EDAX analysis was performed for the compounds to identify the purity of the compounds. The EDAX spectrum showed that there was no impurity present in the compounds. It ascertained the formation of single-phase compounds by XRD. Because of low atomic weight lithium could not be detected other than that the EDAX showed the presence of the respective atoms. The particle size distribution of the compounds showed that the particles were distributed in large volume. The particle diameter increased with the increase of calcination temperature. Grinding reduced the large volume distribution and the particle diameter. After grinding by mortar and pestle hand grinder, the particle size was reduced much and the distribution was narrowed down, thereby the surface area of the particle was increased.

The SEM analysis also confirmed the sub-micron size reduction and the distribution was narrowed down, thereby the surface area of the particle was increased. The compounds were used as electrode materials for lithium ion batteries. The battery analysis showed that the capacities of the LiMn_2O_4 , LiCoO_2 and $\text{Li}_2\text{Ni}_8\text{O}_{10}$ were 10 mAh, 24 mAh and 5 mAh respectively.



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Sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

**PENYEDIAAN DAN PENCIRIAN BAHAN-BAHAN BATERI
KEADAN PEPEJAL LITIMUM**

Oleh

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Tiga jenis bahan katod iaitu LiMn_2O_4 , Li CoO_2 dan $\text{Li}_2\text{Ni}_8\text{O}_{10}$ telah disintesis menggunakan kaedah itu 'sol-gel'. Kaedah suhu rendah ini menghasilkan bahan-bahan yang tersebut di atas pada suhu rendah. Kajian belauan sinar-X terhadap sebatian-sebatian ini mengesahkan pembentukan sebatian fasa tunggal pada suhu 'kalsinasi' yang lebih tinggi. Pada suhu rendah difraktogram sinar-X sampel-sampel menunjukkan kewujudan puncak-puncak belauan yang kecil beserta kehadiran sedikit bendasing, menandakan kewujudan sifat kehabluran, tetapi tidak pula merujuk kepada sebarang ketidaktulenan LiMn_2O_4 teroksida. Suhu pembentukan sebatian tersebut dianalisa menggunakan kajian DTA menunjukkan dengan jelas suhu pembentukan terendah ini dan suhu pembentukan ini bergantung kepada 'gelating agent' yang digunakan didalam penyelidikan ini. Suhu pembentukan terendah yang direkodkan adalah 208°C bagi LiMn_2O_4 , 201°C bagi Li CoO_2 dan 214°C bagi $\text{Li}_2\text{Ni}_8\text{O}_{10}$.



Analisa termogravimetrik menunjukkan sebatian-sebatian tersebut adalah stabil sehingga 800°C. Analisa EDAX dilakukan untuk mengenalpasti ketulenan sebatian-sebatian tersebut. Spektrum EDAX menunjukkan tidak terdapat bendasing di dalam sebatian-sebatian tersebut. Fakta ini ditentusahkan lagi dengan kewujudan fasa tunggal seperti ditunjukkan oleh XRD. Disebabkan jisim atom litium yang rendah ia tidak dikesan, melainkan dengan menggunakan EDAX mengesahkan kehadiran atom-atom yang berkenaan. Taburan saiz zarah bagi sebatian-sebatian tersebut menunjukkan bahawa zarah-zarah berkenaan tersebar di dalam isipadu yang besar. Diameter zarah bertambah dengan pertambahan suhu 'kalsinas'. Proses licikan boleh mengurangkan taburan isipadu yang besar dan diameter zarah. Selepas licikan dilakukan dengan mortar dan "pestle hand grinder", saiz zarah menurun dengan ketara dan taburan isipadu dikurangkan.

SEM juga mengesahkan zarah-zarah tersebut adalah bersaiz sub-mikron. Sebatian-sebatian tersebut telah digunakan sebagai bahan katod bagi bateri litium-ion. Analisa ke atas bateri menunjukkan nilai kapasiti bagi LiMn_2O_4 adalah 10 mAh, bagi LiCoO_2 adalah 24 mAh dan bagi $\text{Li}_2\text{Ni}_8\text{O}_{10}$ adalah 5 mAh.

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"Verily never will Allah change the condition of peoples until they change it themselves (with their own souls)" - Al-Quran

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LIST OF SYMBOLS AND ABBREVIATIONS/NOTATION

Ah	ampere-hour
Ah/L	ampere-hours per liter
Å	angstrom
A	ampere
α	alpha
Ah	ampere-hour
Ah/kg	ampere-hours per kilogram
β	beta
Cm	centimeter
C	coulomb
Cr	chromium
Co	cobalt
Cl	chlorine
Cu	copper
$\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	cobaltous(II) nitrate hexahydrate
$\text{C}_6\text{H}_8\text{O}_7 \cdot \text{H}_2\text{O}$	citric acid
$\text{C}_4\text{H}_6\text{O}_4$	succinic acid
$\text{CH}(\text{COOH})(\text{OH})\text{COOH}$	maleic acid
$(\text{CHOH}-\text{COOH})_2$	tartaric acid
DEC	diethyl Carbonate
DTA	differential thermal analysis
DMC	dimethyl carbonate

E_0	standard potential (in volts)
EC	ethylene Carbonate
EPDM	ethylene propylene diene monomer
F	faraday constant (96500 C or 26.8 Ah)
Fe	iron
g	gram
ΔG	Gibbs free energy change
I	current
IR	Internal resistance of a cell
JCPDS	Standards, Joint Committee on Powder Diffraction Standards, Index to the Powder Diffraction File, Swarthmore, Pa.
Kg	kilogram
Li-ion	lithium-ion
LiNO_3	lithium nitrate
Li_2CO_3	lithium carbonate
$\text{Li}_2\text{Ni}_8\text{O}_{10}$	lithium nickel oxide
LiCoO_2	lithium cobalt oxide
LiMn_2O_4	lithium manganese oxide
LiClO_4	lithium perchlorate
LiMeS_2	lithium transition metal sulphide
LiMeS_2	lithium transition metal selenide
LiMeO_2	lithium transition metal oxide
LiMO_2	lithium metal oxide

LiFeO ₂	lithium iron oxide
LiBF ₄	lithium-tetrafluoroborat
LiCF ₃ SO ₃	lithium trifluoromethane-sulfonate
LiTi ₂ O ₄	lithium titanium oxide
LiV ₂ O ₄	lithium vanadateoxide
LiCoVO ₄	lithium cobalt vanadateoxide
LiNiVO ₄	lithium nickel vanadateoxide
mAh	milli-ampere hour
m ²	square meter
m ³	cubic meter
M(mole)	amount of substance
Me	transition metal
mAh/g	milli-ampere hour per gram
Mn	manganese
Mo	molybdenum
MCFC	molten carbonate fuel cells
“n”	no of electrons involved in the electrochemical reaction
N	nitrogen
Ni	nickel
Ni-Cd	nickel cadmium
Ni(NO ₃) ₂ · 6 H ₂ O	nickel (II) nitrate hexahydrate
O	oxygen
PVDF	polyvinylidene fluoride