

**SYNTHESIS AND ELECTROCHEMICAL
PROPERTIES OF LiCoO_2 CATHODE WITH
GRAPHITE OR GRAPHENE ANODE FOR
AQUEOUS RECHARGEABLE LITHIUM
BATTERIES**

NUR AZILINA BINTI ABDUL AZIZ

UNIVERSITI SAINS MALAYSIA

2018

**SYNTHESIS AND ELECTROCHEMICAL PROPERTIES OF
LiCoO₂ CATHODE WITH GRAPHITE OR GRAPHENE ANODE
FOR AQUEOUS RECHARGEABLE LITHIUM BATTERIES**

by

NUR AZILINA BINTI ABDUL AZIZ

**Thesis submitted in fulfilment of the requirements
for the degree of
Doctor of Philosophy**

August 2018

ACKNOWLEDGEMENT

Alhamdulillah, the Most Beneficent and Most Merciful, for giving me the strength to successfully complete this thesis. First and foremost, I would like to express my gratitude and sincere appreciation to my supervisor, Assoc. Prof. Dr. Ahmad Azmin Mohamad for his advice, support, guidance, kindness and time spent throughout this research project. My appreciation also goes to my co-supervisor, Dr. Tuti Katrina Abdullah for her support, advice and knowledge regarding this project.

I further thank the Ministry of Higher Education (MoHE) and Department of Polytechnic for study leave and also Universiti Sains Malaysia (USM) for its financial support through the Postgraduate Research Grant Scheme (PRGS). I express my sincere appreciation to all academic and technical staff of the School of Materials and Mineral Resources Engineering, USM for their contributions to and assistance during my studies. I also thank to our research group for their continuous support, helpful for providing discussions and ideas.

Last but not least, I would like express the deepest appreciation to my beloved husband, Mr. Sukhairi Samsudin, and my kids Syarifah Nur Zahra, Syed Akhtar, Syed Thaqif and Syarifah Nur Raudhah for their prayers, understanding, encouragement, inspiration that enabled me to complete this research. My deepest gratitude to my lovely mother, Mrs Azizah Yusoff and my late farther, Mr. Abdul Aziz Abdullah, who is my greatest supporter. Their advice and guidance will always in my heart forever.

TABLE OF CONTENTS

	Page
ACKNOWLEDGEMENT	ii
TABLE OF CONTENTS	iii
LIST OF TABLES	viii
LIST OF FIGURES	x
LIST OF ABBREVIATIONS	xx
LIST OF SYMBOLS	xxii
ABSTRAK	xxiii
ABSTRACT	xxiv
CHAPTER ONE: INTRODUCTION	
1.1 Background of the study	1
1.2 Problem statement	3
1.3 Objectives of study	6
1.4 Thesis outlines	7
CHAPTER TWO: LITERATURE REVIEW	
2.1 Introduction	8
2.2 Development of Lithium batteries	8
2.3 Lithium-ion batteries	9
2.4 Aqueous rechargeable lithium batteries	10
2.5 Working principle of aqueous rechargeable lithium batteries	11

2.6	Component of aqueous rechargeable lithium batteries	12
2.6.1	Electrolyte	13
2.6.2	Anode	14
2.6.3	Cathode	14
2.7	LiCoO ₂ as a cathode material	15
2.8	Graphite and graphene	16
2.8.1	Graphite	16
2.8.2	Graphene	17
2.9	Synthesis method	18
2.10	Sol-gel method	20
2.11	Thermal properties of LiCoO ₂	21
2.12	Phase and structural properties of LiCoO ₂	24
2.12.1	Phase identification analysis of LiCoO ₂	24
2.12.2	Structural analysis of LiCoO ₂	27
2.13	Morphological properties of LiCoO ₂	29
2.14	Introduction of electrochemical characterization	33
2.15	Cycle behavior properties of LiCoO ₂	34
2.15.1	Redox reaction	34
2.15.2	Diffusion coefficient of Li ⁺	39
2.16	Cycle performance properties of LiCoO ₂	41
2.16.1	Charge-discharge profile	41
2.16.2	Cycle life of LiCoO ₂	45
2.17	Impedance properties of LiCoO ₂	48

CHAPTER THREE: METHODOLOGY

3.1	Introduction	58
3.2	Materials and equipment	60
3.3	Synthesis of LiCoO ₂ via sol-gel method	62
3.4	Fabrication of aqueous rechargeable lithium batteries	65
3.4.1	Preparation of LiCoO ₂ as cathode electrode	65
3.4.2	Preparation of graphite and graphene as anode electrode	66
3.4.3	Preparation of LiNO ₃ electrolyte	67
3.5	Material characterization of LiCoO ₂	68
3.5.1	Thermal analysis	68
3.5.2	Structural analysis	68
3.5.3	Morphological analysis	69
3.6	Electrochemical characterization of LiCoO ₂	70

CHAPTER FOUR: SYNTHESIS OF LiCoO₂ VIA SOL-GEL METHOD FOR AQUEOUS RECHARGEABLE LITHIUM BATTERIES

4.1	Introduction	73
4.2	Synthesis of LiCoO ₂ via sol-gel method	74
4.3	Characterization of synthesized LiCoO ₂ via sol-gel method	76
4.3.1	Thermal analysis	76
4.3.2	Phase identification analysis	80
4.3.3	Reitveld refinement analysis	85
4.3.4	Morphological analysis	90
4.4	Schematic diagram during stirring process for LiCoO ₂ formation	99
4.5	Morphological analysis of anode materials	102

4.6	Electrochemical characterization of LiCoO ₂	104
4.6.1	Cycle behavior analysis	104
4.6.2	Charge-discharge analysis	114
4.6.3	Impedance analysis	119

CHAPTER FIVE – SYNTHESIS OF LiCoO₂ VIA SONICATION SOL-GEL METHOD FOR AQUEOUS RECHARGEABLE LITHIUM BATTERIES

5.1	Introduction	128
5.2	Synthesis of LiCoO ₂ via sonication sol-gel method	128
5.3	Characterization of synthesized LiCoO ₂ via sonication sol-gel method	130
5.3.1	Thermal analysis	131
5.3.2	Phase identification analysis	135
5.3.3	Reitveld refinement analysis	140
5.3.4	Morphological analysis	145
5.4	Schematic diagram during sonication process for LiCoO ₂ formation	156
5.5	Electrochemical characterization of LiCoO ₂	158
5.5.1	Cyclic behavior analysis	158
5.5.2	Charge-discharge analysis	165
5.5.3	Impedance analysis	171

CHAPTER SIX – CONCLUSIONS AND RECOMMENDATIONS

6.1	Conclusions	178
6.2	Recommendations for future work	181

APPENDICES

- Appendix A : Stoichiometric amount of precursors used to synthesis LiCoO_2
- Appendix B : Schematic illustration of synthesized LiCoO_2 via sol-gel process
- Appendix C : Determination of current density for CD rate
- Appendix D – i : TGA-DTG curve of sample stirred at (a) 6, (b) 12, (c) 18, (d) 24, (e) 30, and (f) 36 hours
- Appendix D – ii : TGA-DTG curve of sample sonicated at (a) 15, (b) 30, (c) 60, (d) 90, (e) 120, and (f) 180 minutes
- Appendix E : Standard reference of LiCoO_2
- Appendix F – i : Observed, calculated and difference profiles sample refinement synthesized at different stirring times
- Appendix F – ii : Observed, calculated and difference profiles sample refinement synthesized at different sonication times
- Appendix G : Example of Rietveld refinement report for LiCoO_2 sample stirred at 30 hours
- Appendix H – i : Calculation of crystallite size of LiCoO_2 synthesized via sol-gel method and calcined at $700\text{ }^\circ\text{C}$
- Appendix H – ii : Calculation of crystallite size of LiCoO_2 synthesized via sonication sol-gel method and calcined at $700\text{ }^\circ\text{C}$.
- Appendix I : EIS profile for LiCoO_2 -graphene before CD analysis at 0.1 mV s^{-1}

LIST OF PUBLICATIONS

LIST OF TABLES

	Page	
Table 2.1	Summary of selected papers on material and electrochemical properties to characterize LiCoO ₂ from 2006–2010	51
Table 3.1	Raw materials and chemicals used in this work	60
Table 3.2	Equipment used in this work	61
Table 4.1	Refinement parameter for hexagonal structure of LiCoO ₂ synthesized at different stirring times	89
Table 4.2	Particle size distribution of LiCoO ₂	96
Table 4.3	Average of particle size distribution for anode powder	102
Table 4.4	Comparison of redox peaks for LiCoO ₂ -graphite and LiCoO ₂ -graphene	107
Table 4.5	Diffusion coefficient of Li ⁺ for LiCoO ₂ -graphite and LiCoO ₂ -graphene system	112
Table 4.6	Specific discharge capacity of LiCoO ₂ using different anode	114
Table 4.7	Capacity retention of LiCoO ₂ -graphite and LiCoO ₂ -graphene batteries	118
Table 4.8	R _{ct} before and after three cycles of CV cycling under different scan rates for LiCoO ₂ -graphite and LiCoO ₂ -graphene	123

Table 4.9	Impedance parameters derived using an equivalent circuit model of LiCoO ₂ -graphite and LiCoO ₂ -graphene	125
Table 5.1	Refinement parameter for hexagonal structure of LiCoO ₂ synthesized at different sonication times	144
Table 5.2	Particle size distribution of LiCoO ₂ synthesized via sonication sol-gel method	151
Table 5.3	Average of particle size for synthesized LiCoO ₂ using different sol-gel method	152
Table 5.4	Comparison of redox peaks for LiCoO ₂ -graphite and LiCoO ₂ -graphene	160
Table 5.5	Diffusion coefficient of Li ⁺	165
Table 5.6	Specific discharge capacity of LiCoO ₂ using different anode	166
Table 5.7	Capacity retention of LiCoO ₂ -graphite and LiCoO ₂ -graphene batteries	170
Table 5.8	R _{ct} before and after three cycles of CV cycling under different scan rates for LiCoO ₂ -graphite and LiCoO ₂ -graphene	174
Table 5.9	Impedance parameters derived using an equivalent circuit model of LiCoO ₂ -graphite and LiCoO ₂ -graphene	176
Table 5.10	Overall result of electrochemical system for stirring and sonication process	177

LIST OF FIGURES

		Page
Figure 1.1	Schematic diagram of LiCoO ₂ in ARLB system	5
Figure 2.1	The principle of LiCoO ₂ simulated in lithium-ion batteries [64]	12
Figure 2.2	Structure of LiCoO ₂ , space group R3m (166) (a) 2D view [87], (b) 3D view [88] and (c) Hexagonal structure and position of the ions [85]	16
Figure 2.3	Comparison of cycling behaviors of LiCoO ₂ prepared by a sol-gel method and by the traditional solid state reaction [101]	19
Figure 2.4	Basic principle of the sol-gel process [104]	20
Figure 2.5	TGA/DTA curve of LiCoO ₂ precursors (adapted from Ref. [3])	22
Figure 2.6	TGA/DTA/DTG curves of LiCoO ₂ precursors (adapted from Ref. [31])	23
Figure 2.7	Typical XRD pattern of LiCoO ₂ (adapted from Ref. [19])	24
Figure 2.8	XRD pattern of LiCoO ₂ powder obtained at various calcination temperatures (300-800 °C) for 5 h (adapted from Ref. [114])	25
Figure 2.9	XRD pattern of LiCoO ₂ powders (400 ≤ T ≤ 900 °C) [112]	26

Figure 2.10	LiCoO ₂ phases by Rietveld refinement (adapted from Ref. [110])	28
Figure 2.11	SEM micrographs of LiCoO ₂ powders calcined at various temperatures (a) 600, (b) 700, (c) 800, and (d) 900 °C (adapted from Ref. [3])	29
Figure 2.12	Surface morphology of the samples calcined at different temperatures (a) 350, (b) 450, (c) 550, (d) 650, and (e) 750 °C [107]	30
Figure 2.13	Image of the synthesized LiCoO ₂ (a) SEM showing regular particle with agglomeration, (b) TEM showing individual particle, and (c) particle size distribution histogram corresponds to the TEM image [19]	32
Figure 2.14	Images of LiCoO ₂ powders (a) TEM, and (b) HRTEM (adapted from ref. [37])	33
Figure 2.15	(a) Cyclic voltammogram of the LiCoO ₂ -electrode in saturated Li ₂ SO ₄ solution (adapted from Ref. [16]), (b) Lattice parameter a and c as a function of lithium concentration, x, in Li _x CoO ₂ and phase diagram for Li _x CoO ₂ [119], and (c) Charge-discharge curves Li _x CoO ₂ at C/24 rate in the range 3.6–4.85 V vs. Li/Li ⁺ . The sequence of the several phases is indicated as x varies from 1.0–0.05 [84]	35
Figure 2.16	Cyclic voltammetry of LiCoO ₂ at various calcination temperatures [3]	36

Figure 2.17	Cyclic voltammetry of LiCoO_2 at 0.1 mV s^{-1} in different concentration of LiNO_3 electrolytes (adapted from Ref. [20])	37
Figure 2.18	Cyclic voltammetry curves of LiCoO_2 in $0.5 \text{ M Li}_2\text{SO}_4$ aqueous electrolytes at different scan rates [5])	38
Figure 2.19	(a) Cyclic voltammograms of the LiCoO_2 electrode at different scan rates, (b) Variation of the cathodic peak current with the square root of the scan rate ($v^{1/2}$) of the data in (a) [118], and (c) Cyclic voltammograms of the LiCoO_2 in 1.0 M LiNO_3 solution recorded at different potential sweep rates. Inset dependency of the cathodic and anodic peak currents on the square root of the potential sweep rate [19]	40
Figure 2.20	Typical charge-discharge profile in aqueous solution (adapted from Ref. [18])	42
Figure 2.21	Charge-discharge curves at different C-rate [5]	43
Figure 2.22	Charge-discharge curves at different calcination temperatures (adapted from Ref. [107])	44
Figure 2.23	The relationship of $\text{LiV}_3\text{O}_8/\text{LiCoO}_2$ between discharge capacity and cycle number [18]	45
Figure 2.24	Charge-discharge capacity vs. number of cycles at 1C [14]	46
Figure 2.25	Specific capacity against cycles number at different rates [20]	47

Figure 2.26	Nyquist plots of LiCoO ₂ electrodes after completed at different cycling numbers (adapted from Ref. [123])	49
Figure 3.1	Overall flow chart of the experimental procedure for the synthesis and characterization of LiCoO ₂ in this work	59
Figure 3.2	Two-step heat treatment temperature profile of LiCoO ₂ powder	64
Figure 3.3	Schematic of LiCoO ₂ synthesis via sonication sol-gel method	64
Figure 3.4	Process flow of LiCoO ₂ electrode preparation	66
Figure 3.5	Process flow of graphite electrode preparation	67
Figure 3.6	(a) Schematic diagram, and (b) image of LiCoO ₂ vs. graphite electrode	72
Figure 4.1	Apparent of the mixture (a) before, and (b) after heating at 80 °C for 3 hours	74
Figure 4.2	Image of (a) as-synthesized sample dried in oven (b) dense sample after calcination at 700 °C for 4 hours, and (c) ground powder after calcination at 700 °C for 4 hours	75
Figure 4.3	TGA/DTG curve of LiCoO ₂ synthesized at different stirring times	78
Figure 4.4	TGA/ DTG curve splitting by regions for sample stirred at 30 hours	79

Figure 4.5	XRD patterns of LiCoO_2 synthesized at different stirring times and calcined at 700 °C	81
Figure 4.6	The $I_{(003)}/I_{(104)}$ ratio of the LiCoO_2 powders synthesized at different stirring times	82
Figure 4.7	XRD patterns of synthesized LiCoO_2 stirred at 30 hours (a) before, and (b) after calcined for 700 °C	84
Figure 4.8	Observed, calculated and difference profiles from sample refinement calcined at stirring times of (a) 6, (b) 12, (c) 18, (d) 24, (e) 30, and (f) 36 hours	86
Figure 4.9	Observed, calculated and difference profiles from sample refinement stirred at (a) 6 hours, and (b) 30 hours stirring time	87
Figure 4.10	FESEM images of LiCoO_2 obtained at stirring times (a) 6 hours, and (b) 12 hours	91
Figure 4.11	FESEM images of LiCoO_2 obtained at stirring times (a) 18 hours, and (b) 24 hours	92
Figure 4.12	FESEM images of LiCoO_2 obtained at stirring times (a) 30 hours, and (b) 36 hours	93
Figure 4.13	Histogram of particle size distribution for LiCoO_2 powder synthesized at different stirring times (a) 6, (b) 12, (c) 18, (d) 24, (e) 30, and (f) 36 hours	95
Figure 4.14	Particle size distribution of LiCoO_2 synthesized at stirring times (a) 6, (b) 12, (c) 18, (d) 24, (e) 30, and (f) 36 hours	96

Figure 4.15	<i>Images</i> of LiCoO ₂ for sample stirred at 30 hours (a) <i>TEM</i> , and (b) <i>HRTEM</i>	98
Figure 4.16	Schematic diagram during stirring process for LiCoO ₂ formation	101
Figure 4.17	SEM image of (a) graphite, and (b) graphene powder	103
Figure 4.18	CV of LiCoO ₂ vs. platinum at lower scan rate (0.1 mV s ⁻¹)	105
Figure 4.19	Comparison of CV between (a) LiCoO ₂ -graphite and (b) LiCoO ₂ -graphene at lower scan rate (0.1 mV s ⁻¹)	108
Figure 4.20	CV at different scan rates for (a) LiCoO ₂ -graphite, and (b) LiCoO ₂ -graphene	110
Figure 4.21	Variation of anodic and cathodic peaks current with the square root of scan rate (a) LiCoO ₂ -graphite, and (b) LiCoO ₂ -graphene	111
Figure 4.22	Charge-discharge curves under different C-rates for (a) LiCoO ₂ -graphite, and (b) LiCoO ₂ -graphene	115
Figure 4.23	Comparison of CD curve at 0.5 C rate between (a) LiCoO ₂ -graphite, and (b) LiCoO ₂ -graphene	116
Figure 4.24	Comparison of cycle life at 0.5 C rate between (a) LiCoO ₂ -graphite, and (b) LiCoO ₂ -graphene	118
Figure 4.25	Equivalent circuit of Nyquist plot	120

Figure 4.26	Nyquist plot of LiCoO ₂ at different scan rates and after three cycles of CV (a) LiCoO ₂ -graphite before and (b) LiCoO ₂ -graphite after	121
Figure 4.27	Nyquist plot of LiCoO ₂ at different scan rates and after three cycles of CV (a) LiCoO ₂ -graphene before, and (b) LiCoO ₂ -graphene after	122
Figure 4.28	Nyquist plot before and after three cycles of CD under 0.1 mV s ⁻¹ for (a) LiCoO ₂ -graphite, and (b) LiCoO ₂ -graphene	124
Figure 4.29	(a) Typical Nyquist plots of LiCoO ₂ synthesized at 700 °C, and (b) schematic of diffusion in a cell system	126
Figure 5.1	Apparent of the mixture ((a) before, and (b) after heating at 80 °C for 3 hours	129
Figure 5.2	Image of (a) as-synthesized LiCoO ₂ dried in oven, (b) dense sample after calcination, and (c) ground powder after calcination	130
Figure 5.3	TGA/DTG curve of LiCoO ₂ samples synthesized at different sonication times	132
Figure 5.4	Comparison thermal profile using different synthesize method (a) TGA conventional, (b) DTG conventional, (c) TGA sonication, and (d) DTG sonication sol-gel method	134
Figure 5.5	XRD patterns of LiCoO ₂ synthesized at different sonication times calcined for 700 °C	136
Figure 5.6	The I ₍₀₀₃₎ /I ₍₁₀₄₎ ratio of the LiCoO ₂ powders synthesized at different sonication times	137

Figure 5.7	Comparison between XRD patterns of LiCoO_2 synthesized via (a) conventional, and (b) sonication sol-gel method	139
Figure 5.8	Observed, calculated and difference profiles from sample refinement at sonicating times of (a) 15, (b) 30, (c) 60, (d) 90, (e) 120, and (f) 180 minutes	141
Figure 5.9	Observed, calculated and difference profiles from sample refinement sonicated at (a) 15 min, and (b) 120 min sonication time	142
Figure 5.10	FESEM images of LiCoO_2 obtained at sonication times of (a) 15 minutes, and (b) 30 minutes	146
Figure 5.11	FESEM images of LiCoO_2 obtained at sonication times of (a) 60 minutes, and (b) 90 minutes	147
Figure 5.12	FESEM images of LiCoO_2 obtained at sonication times of (a) 120 minutes, and (b) 180 minutes	148
Figure 5.13	Histogram of particle size distribution for LiCoO_2 powder obtained from sonication times of (a) 15, (b) 30, (c) 60, (d) 90, (e) 120, and (f) 180 minutes	150
Figure 5.14	Particle size distribution of LiCoO_2 synthesized at sonication times of 15, 30, 60, 90, 120, and 180 minutes	151
Figure 5.15	Morphological comparison between LiCoO_2 particle synthesized via (a) stirring process, and (b) sonication process	153

Figure 5.16	<i>Images</i> of LiCoO ₂ for sample sonicated at 120 minutes (a) TEM, and (b) HRTEM	155
Figure 5.17	Schematic diagram of sonication process for LiCoO ₂ formation	157
Figure 5.18	Comparison of CV between (a) LiCoO ₂ -graphite, and (b) LiCoO ₂ -graphene at 0.1 mV s ⁻¹	159
Figure 5.19	CV at different scan rates for (a) LiCoO ₂ -graphite, and (b) LiCoO ₂ -graphene	163
Figure 5.20	Variation of anodic and cathodic peaks current with the square root of scan rate (a) LiCoO ₂ -graphite, and (b) LiCoO ₂ -graphene	164
Figure 5.21	Charge-discharge curves under different C-rates for (a) LiCoO ₂ -graphite, and (b) LiCoO ₂ -graphene	167
Figure 5.22	Comparison of CD curve at C-rate 0.5 C (a) LiCoO ₂ -graphite, and (b) LiCoO ₂ -graphene	168
Figure 5.23	The comparison of cycle life at C-rate 0.5 C between (a) LiCoO ₂ -graphite, and (b) LiCoO ₂ -graphene	169
Figure 5.24	Nyquist plot of LiCoO ₂ at different scan rates and after three cycles of CV (a) LiCoO ₂ -graphite before, and (b) LiCoO ₂ -graphite after	172
Figure 5.25	Nyquist plot of LiCoO ₂ at different scan rates and after three cycles of CV (a) LiCoO ₂ -graphene before, and (b) LiCoO ₂ -graphene after	173

Figure 5.26 Nyquist plot before and after three cycles of CD at lower C-rate (0.5 C) for (a) LiCoO₂-graphite, and (b) LiCoO₂-graphene

175

LIST OF ABBREVIATIONS

ARLB	Aqueous Rechargeable Lithium Batteries
CD	Charge-discharge
CE	Counter Electrode
CV	Cyclic Voltammetry
TGA	Thermogravimetric analysis
DTA	Differential thermal analysis
DTG	Derivative Thermogravimetry
EIS	Electrochemical Impedance Spectroscopy
FESEM	Field-Emission Scanning Electron Microscope
TEM	Transmission Electron Microscope
GoF	Goodness of Fit
R_{wp}	Weighted R Profile
JCPDS	Joint Committee on Powder Diffraction Standards
ICSD	Inorganic Crystal Structure Database
R_{ct}	Charge Transfer Resistance
R_s	Electrolyte Resistance
RE	Reference Electrode
SCE	Saturated Calomel Electrode
WE	Working Electrode
XRD	X-ray Diffraction
EC/DEC	Ethylene carbonate-diethyl carbonate
ED/DMC	Ethylene carbonate-dimethyl carbonate
H ₂	Hydrogen
H ₂ O	Water

$C_2H_3LiO_2 \cdot 2H_2O$	lithium acetate dihydrate
$C_4H_6CoO_4 \cdot 4H_2O$	cobalt acetate tetrahydrate
C_6H_8O	citric acid
$LiCoO_2$	Lithium cobalt oxide
$LiFePO_4$	Lithium iron phosphate
$LiMn_2O_4$	Lithium manganese oxide
$LiNO_3$	Lithium nitrate
$LiOH$	Lithium hydroxide
Li_2SO_4	Lithium sulphate
Li_3PO_4	Trilithium phosphate
LiV_3O_8	Lithium vanadium oxide
N_2	Nitrogen
O_2	Oxygen
Pt	Platinum
PTFE	polytetrafluoroethylene
PVDF	polyvinylidene fluoride
SS mesh	Stainless steel mesh

LIST OF SYMBOLS

%	Percentage
<	Less than
>	More than
°	Degree
Ω	Ohm
λ	Wave length
wt. %	Weight percent
mA h g ⁻¹	Mili-ampere hour per gram
Wh kg ⁻¹	Watt hour per kilogram
°C	Degree Celsius
°C min ⁻¹	Degree Celsius per minute
A	Ampere
C	Current rate
cm	Centimeter
nm	Nanometer
eV	Electron volt
g	Gram
h	Hour
Hz	Hertz
K	Kelvin
Li ⁺	Lithium ions
V	Voltage

SINTESIS DAN PENCIRIAN ELEKTROKIMIA KATOD LiCoO_2 DENGAN GRAFIT ATAU GRAFIN ANOD UNTUK BATERI AKUES LITIUM YANG BOLEH DICAS SEMULA

ABSTRAK

Bateri akues litium yang boleh dicas semula kini mendapat perhatian kerana kos pengeluaran yang lebih rendah dan keselamatan penyimpanan tenaga yang lebih baik. Bahan katod dengan pemilihan parameter sintesis yang sesuai merupakan salah satu faktor yang akan menghasilkan sifat bahan yang baik dan pada masa yang sama turut membantu dalam mencapai prestasi elektrokimia yang lebih baik. Dalam kajian ini, kesan masa pengacauan dan masa sonikasi dikaji dalam mensintesis LiCoO_2 melalui kaedah sol-gel. Keputusan pencirian bahan antara sampel optima pada masa kacau (30 jam) dan sampel optima pada masa sonikasi (120 minit) menunjukkan sampel yang disintesis melalui proses sonikasi adalah lebih baik berbanding proses pengacauan dengan sifat penghabluran yang terbaik dan saiz zarah yang terkecil. Analisis morfologi menunjukkan taburan saiz zarah adalah dalam julat 0.29 - 0.43 μm . Tingkah laku kitaran LiCoO_2 yang disintesis melalui sonikasi dengan grafin sebagai anod mempamerkan kestabilan kitaran yang lebih baik, puncak redoks yang jelas dan beza keupayaan yang kecil (0.13 V). Prestasi bateri LiCoO_2 menunjukkan penambahan kapasiti pelepasan khusus (122.43 mAh g^{-1} pada 0.5 C) dan kebolehulangan dalam elektrolit akues. Peningkatan prestasi elektrokimia disokong oleh analisis impedans. Perbezaan kecil (0.7 Ω) dalam rintangan pemindahan cas sebelum dan selepas analisis cas-discas menunjukkan bahawa ion Li^+ telah tersebar dengan baik semasa proses interkalasi/nyah-interkalasi.