UNIVERSITI PUTRA MALAYSIA

SPINEL $LiMn_2O_4$ CATHODE AND CARBONACEOUS ANODE MATERIAL FOR ELECTROCHEMICAL ENERGY STORAGE LITHIUM-ION BATTERY

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FS 2022 30

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By

AHMED ZAHOOR

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Doctor of Philosophy

January 2021

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Doctor of Philosophy

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Chairman : Professor Zainal Abidin Talib, PhD
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Battery technology is one of the most promising technology for next-generation portable electronics, electric vehicle (EV), hybrid electric vehicles (HEV) and stationary energy storage systems. In this aspect, Li-ion batteries are the most attractive power-source candidate due to their superior high energy density as a comparison to the available rechargeable batteries. High energy density mainly depends on a high voltage and high specific capacity. Novel electrode materials research, specifically cathode plays an essential role in the development of advanced lithium-ion batteries. The electrochemical performance of active electrode material mainly relies on the crystal size and morphology. In the present study, various nanostructured cathode materials were synthesized to improve the energy density by using sol-gel assisted pechini, coating and ball milling techniques. Research reveals that nanomaterial-based cathode materials are preferable due to various advantages such as dimension reduction, faster ionic (Li+) and electronic (e-) transport and mechanical stability as compared to traditional solid-state synthesis-based materials.

First, spinel-based cathode $LiMn_2O_4$ and carbon composite were studied in Li-ion battery application due to eco-environment, natural abundance, low cost with high operating potential, theoretical capacity and power density properties. In this work, step potential electrochemical spectroscopy (SPECS)/galvanostatic intermittent titration technique (GITT) is conducted on three-electrode systems, including spinel prepared by solid-state and sol-gel methods. SPECS experimental data has been fitted with planar, double planar, spherical, and Cottrell diffusional model, to elucidate the diffusional mechanisms and obtain accurate diffusivities for these materials. Theoretical models clearly illustrate that both spherical and double plane diffusion models are in excellent agreement with experimental data. LMOSS exhibits better diffusivity, with an average diffusivity closer to 10^{-13} -10^{-11} cm^2/s as compared to 10^{-13}-10^{-9} cm^2/s for

LMOSG. This work aims to develop a more comprehensive analysis technique for future work. The crystal structure, materials morphology and elemental composition were characterized by x-ray diffraction (XRD), field emission scanning electron microscopy (FE-SEM), energy dispersive x-ray spectroscopy (EDS), and high-resolution transmission electron microscopy (HR-TEM). These experiments reveal the potential benefits of understanding Li-ion diffusion of spinel $LiMn_2O_4$ for high-power lithium-ion batteries (LIBs) storage performance.

Secondly, anion substitution of fluorine into spinel cathode material was designed by using the sol-gel assisted pechini method. Fluorinated based spinels show better electrochemical performance as compared to pristine spinel. The fluorine doped spinels $LiMn_2O_{3.8}F_{0.2}$ and $LiMn_2O_{3.9}F_{0.10}$ showed improved capacity retention of around 94% and 90% respectively as compared to 90% for the pristine $LiMn_2O_4$ at 0.1C. Finally, Electrolytic reduction of molten carbonates (Li/K/Na) has been suggested as a practical approach improving the performance of lithium-ion batteries as anode material. Herein, novel carbonaceous materials were synthesized by using molten carbonates as an exciting new method of producing tuned carbons for battery applications. The electrodeposited carbon anode displays the highest specific capacity with 334 mAh g^{-1} at 0.1 C with coulombic efficiency of 95.70% and 255 mAh g^{-1} at 1C with a capacity retention (coulombic efficiency) of 85.8 % (100%) after 100 cycles in the potential window of 0.01-2V (vs Li/Li^+). The electrochemical properties as measured using Galvanostatic charge-discharge, cycle ability, rate performance, cyclic voltammetry and electrochemical impedance spectroscopy were observed to be greatly enhanced by using the carbonate-derived anode as compared to the commercial graphite

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

SPINEL $LiMn_2O_4$ CATHODE DAN KARBONACEOUS ANODE MATERIAL UNTUK PENYIMPANAN ELEKTROCHEMICAL ENERGY LITHIUM-ION BATERI

Oleh

AHMED ZAHOOR

Januari 2021

Pengerusi : Profesor Zainal Abidin Talib, PhD
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Teknologi bateri adalah salah satu teknologi yang paling menjanjikan untuk elektronik mudah alih generasi berikutnya, kenderaan elektrik (EV), kenderaan elektrik hibrid (HEV) dan sistem penyimpanan tenaga pegun. Dalam aspek ini, bateri Li-ion adalah calon sumber tenaga yang paling menarik kerana ketumpatan tenaga tinggi yang unggul sebagai perbandingan dengan bateri boleh dicas semula yang sediaada. Ketumpatan tenaga tinggi bergantung terutamanya pada voltan tinggi dan kapasiti spesifik tinggi. Penyelidikan bahan elektrod baru, khususnya katod memainkan peranan penting dalam pengembangan bateri lithium-ion canggih. Prestasi elektrokimia bahan elektrod aktif bergantung pada ukuran kristal dan morfologi. Dalam kajian ini, pelbagai bahan katod berstruktur nano disintesis untuk meningkatkan ketumpatan tenaga dengan menggunakan teknik pechini yang dibantu oleh sol-gel, salutan dan penggilingan bola. Penyelidikan menunjukkan bahawa bahan katod berasaskan nanomaterial lebih disukai kerana pelbagai kelebihan seperti pengurangan dimensi, pengangkutan ionik (Li +) dan elektronik (e-) lebih cepat dan kestabilan mekanikal berbanding dengan bahan sintesis berasaskan keadaan pepejal tradisional.

Pertama, katod berasaskan spinel $LiMn_2O_{4\ 0}$ dan komposit karbon dikaji dalam aplikasi bateri Li-ion kerana persekitaran eko, kelimpahan semula jadi, kos rendah dengan potensi operasi yang tinggi, keupayaan teori dan sifat ketumpatan kuasa. Dalam projek ini, langkah spektroskopi elektrokimia berpotensi (SPECS) / teknik titrasi intermiten galvanostatik (GITT) dilakukan pada sistem tiga elektrod, termasuk spinel yang disediakan dengan kaedah keadaan pepejal dan sol-gel. Data eksperimen SPECS telah dilengkapi dengan model diffusional planar, double planar, spherical, dan Cottrell, untuk menjelaskan mekanisme penyebaran dan mendapatkan nilai penyebaran yang

tepat untuk bahan-bahan ini. Model teori dengan jelasnya menunjukkan bahawa kedua-dua model penyebaran satah sfera dan satah ganda sangat berpadanan dengan data eksperimen. LMOSS menunjukkan penyebaran yang lebih baik, dengan purata penyebaran mendekati 10^{-13}-10^{-11} cm^2/s berbanding dengan 10^{-13}-10^{-9} cm^2/s untuk LMOSG. Projek ini bertujuan untuk mengembangkan teknik analisis yang lebih komprehensif untuk kerja masa depan. Struktur kristal, morfologi bahan dan komposisi unsur dicirikan oleh pembelauan sinar-x (XRD), mikroskop elektron pengimbas pelepasan medan (FE-SEM), spektroskopi sinar-x dispersi tenaga (EDS), dan mikroskop elektron transmisi resolusi tinggi (HR) - TEM). Eksperimen ini mendedahkan potensi manfaat untuk memahami penyebaran Li-ion spinel $LiMn_2O_4$ untuk prestasi penyimpanan bateri lithium-ion berkuasa tinggi (LIB).

Kedua, penggantian anion fluorin menjadi bahan katod spinel dirancang dengan menggunakan kaedah pechini yang dibantu sol-gel. Spinel berasaskan fluorinasi menunjukkan prestasi elektrokimia yang lebih baik berbanding dengan spinel yang asli. Spinel dop fluorin $LiMn_2O_{3.8}F_{0.2}$ dan $LiMn_2O_{3.9}F_{0.10}$ menunjukkan peningkatan pengekalan kapasiti sekitar 94% dan 90% masing-masing berbanding dengan 90% untuk $LiMn_2O_4$ asal pada 0.1C. Akhirnya, pengurangan elektrolit karbonat lebur (Li/K/Na) telah dicadangkan sebagai pendekatan praktikal untuk meningkatkan prestasi bateri lithium-ion sebagai bahan anod. Di sini, bahan karbonat baru disintesis dengan menggunakan karbonat lebur sebagai kaedah baru yang menarik untuk menghasilkan karbon untuk aplikasi bateri. Anod karbon yang dimendap elektro menunjukkan kapasiti spesifik tertinggi dengan 334 mAh g^{-1} pada 0.1 C dengan kecekapan coulombic 95.70% dan 255 mAh g^{-1} pada 1C dengan pengekalan kapasiti (kecekapan coulombic) 85.8% (100%) selepas 100 kitaran dalam tetingkap berpotensi 0,01-2V (vs Li/Li^+). Sifat elektrokimia yang diukur menggunakan pelepasan muatan Galvanostatik, kemampuan kitaran, prestasi kadar, voltammetri siklik dan spektroskopi impedans elektrokimia dapat dipertingkatkan dengan menggunakan anoda yang dihasilkan dari karbonat berbanding dengan grafit komersial.

ACKNOWLEDGEMENTS

First and foremost, thanks to Almighty Allah for his marvellous blessing and mercifulness in allowing me to complete my thesis. I want to express my very great appreciation to my joint PhD supervisors Prof Scott Donne, Prof Zainal Abidin, Dr Vicki, Dr Josephine, Dr Jumiah Binti Hassan and Dr Abdul Shakoor for their valuable and constructive suggestions during the planning and development of this research work.

I would also like to thank University of Newcastle International Postgraduate and Central Scholarship (UNIPRS, UNRSC), NSW, Australia, for providing generous research funding. Also, I am grateful to University Putra Malaysia international office Mr Saiful for his continuous support to attain joint degree program.

Finally, but by no means least, thanks go to my beloved wife Zainab Awan for almost unbelievable support, my kids Mustafa Ahmed and Fatimah Ahmed. I am dedicating my thesis to my spouse, and kids.

This thesis was submitted to the Senate of the Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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Date: 14 April 2022

Declaration by Members of Supervisory Committee

This is to confirm that:

- the research conducted and the writing of this thesis was under our supervision;
- supervision responsibilities as stated in the Universiti Putra Malaysia (Graduate Studies) Rules 2003 (Revision 2012-2013) are adhered to.

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LIST OF ACRONYMS

CV	Cyclic Voltammetry
RC	Rate Capability
CA	Cycle Ability
EIS	Electrochemical Impedance Spectroscopy
CNT	Carbon nanotube
XRD	X-ray Diffraction
XPS	X-ray Photoelectron Spectroscopy
LIBs	Lithium-Ion/Li-ion Batteries
LMOSG	Lithium manganese oxide by sol-gel
LOMSS	Lithium manganese oxide by solid-state
LMOF10, LMOF20	F doped Lithium manganese oxide

CHAPTER 1

INTRODUCTION

1.1 General Introduction to Energy

Global warming, CO_2 emission, climate change and depletion of fossil fuels are the major problems due to increasing demands for energy consumption [1, 2]. The gradual increase in global energy demand in various types of energy such as coal, natural gas, petroleum, other renewables, biomass, hydropower and nuclear from 2011-2040 as shown in Figure.1.1. As a consequence, to protect our planet from greenhouse gas emission, it is important to consider novel renewable energy and clean energy power systems such as solar photovoltaic cells, fuel cells, battery, and supercapacitors. There are different kinds of energy storage systems such as thermal energy [4], nuclear energy [5], solar energy [6] and electrochemical energy systems [7]. Electrochemical energy sources (rechargeable batteries) are the most efficient form of energy conversion directly from chemical to electrical energy [1]. There are several advantages by using electrochemical energy storage systems such as (1) Pollution free (2) high energy density (3) commercial applications such as electric vehicles (EV's) and hybrid electrical vehicles (HEV's) (4) compact and robust. There is still a need for optimization of current battery technology such as (1) power density for vehicles industry (2) energy density for cell phones (3) micro battery for medical applications. The electrochemical characteristics of lithium ion batteries (LIBs) depend on chemical and structural properties of electrodes materials.

1.2 Energy Storage Devices

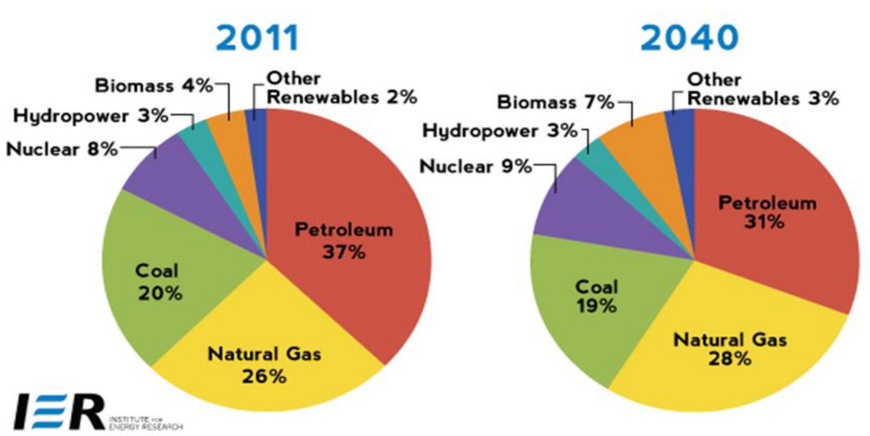

Figure 1.1 : Global energy demand between 2011 till 2040

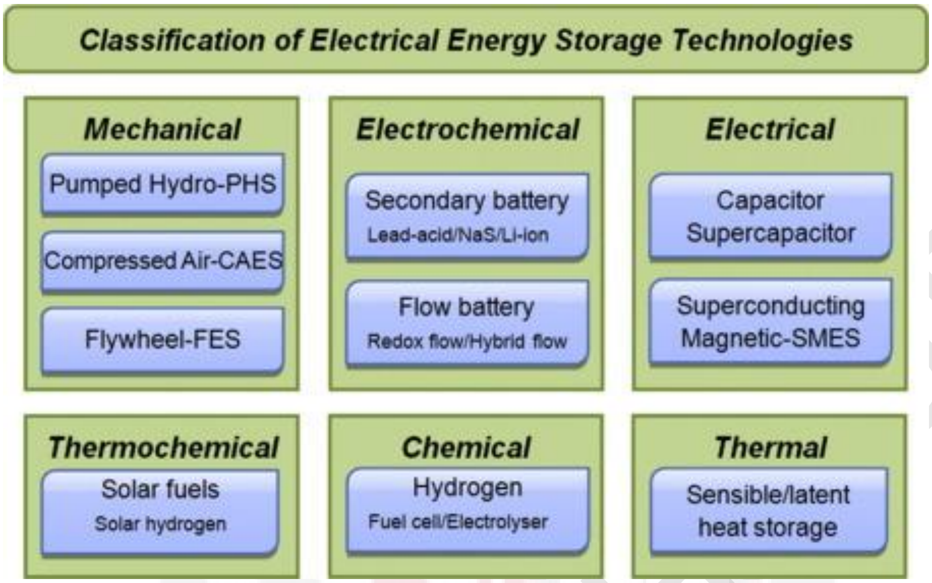

Figure 1.2 : Classification of various electrical energy storage technologies

Classification has been done on several electrical energy storage technologies in term of response time, functioning and, energy storage time [8]. The widely used electrical energy storage devices classified into mechanical, electrochemical, electrical, thermochemical, chemical and thermal energy storage as shown in Figure.1.2. Mechanical energy storages such as pumped hydroelectric, compressed air energy storage and flywheels; Thermal energy storages such as solar thermal storage integration and thermal storage for heating, ventilation, and air conditioning (HVAC); Chemical energy storages such as using hydrogen or other chemicals as an energy storage systems; Electrical and electrochemical energy storage systems such as capacitors, supercapacitors, superconducting magnetic, secondary batteries, flow battery and fuel cells.

The main purpose of current work is based on electrochemical energy systems. A Ragone plot for various electrochemical energy storage systems such as fuel cells, batteries, capacitors, and supercapacitors is shown in Figure.1.3. Batteries, fuel cells, and capacitors will perform different functions within any system requiring both power and energy, with capacitors providing burst power or power smoothing, and batteries/fuel cells providing long-term power[11].

The demand for batteries technologies is increasing rapidly globally and specifically Li-ion batteries. The main reason behind for Li-ion batteries demand globally is its application in next-generation electric vehicle systems[12]. Li-ion batteries production demand for electric vehicles batteries trend from 2008-2020 is shown in Figure.1.4. The expectation is nearly 100 GW of Li-ion batteries to overcome the need of consumer as well as electric vehicles powered technology[14]. Moreover, Li-ion batteries will be used to smooth the energy available on the grid. These fluctuations arise, in general, from changes in energy demand and the variable production capacity of renewables. That is, excess energy is stored when there is an abundance of energy on the grid, and utilized when there is a deficit. It is important that the energy storage technologies such as batteries and capacitors utilized to couple both high power density and high energy density. Large-scale application of Li-ion batteries such as grid will require the low-cost production of batteries.

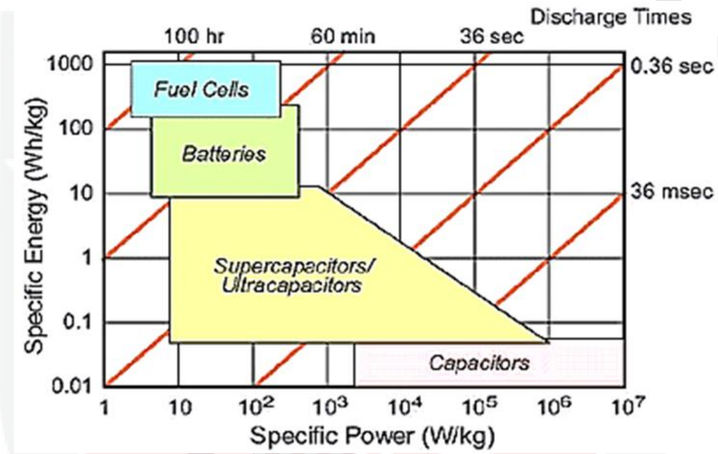

Figure 1.3 : Ragone plot showing various energy storage systems, where red line indicates the complete discharge time for the energy storage devices

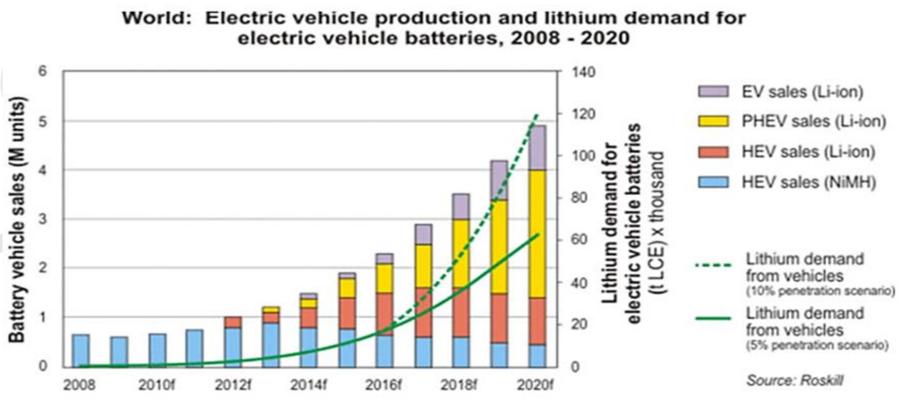

Figure 1.4 : Demand for Li-ion batteries for electric vehicles batteries technologies trend 2008-2020

The amount of energy stored by batteries depend on discharge current $\mathbf{I_{(dis)}}$; it can be achieved by measuring time t given by[15]:

$$I_{(\mathrm{dis})} = \frac{dq}{dt} \quad \text{(Eq.1.1)}$$

where dq represents the state of charge/discharge. The energy is expressed as the product of $I_{(dis)}$ and voltage V summing over the charge/discharge time[15] i.e:

$$\mathrm{Energy} = \int_0^t \mathrm{V} I_{(\mathrm{dis})} dt \quad \text{(Eq.1.2)}$$

where the total charge Q per unit weight (Ah/g) depends on the current transferred by discharge state given by[15]:

$$\mathrm{Q} = \int_0^t \mathrm{I}_{(\mathrm{dis})} \mathrm{dt} \quad \text{(Eq.1.3)}$$

Q is also known as capacity (mAh g^{-1}); Q is a function of I and the reason is that it mainly depends on the amount of ion transferred across electrode/electrolyte interface which becomes diffusion limited at high current density[15].

1.3 Current Status and Future Challenges in Li-ion Batteries Research

Li-ion batteries witnessed great contribution in last two decades to consumable electronics based on reliable energy storage systems such as computers[16], smart phones[17], grid stabilization[18], electric vehicles and plug-in hybrid electric vehicles[19]. Next generation batteries will require lightweight batteries, long cycle life, and high energy density as well power density. In general, batteries are facing challenges like high energy density with long cyclic efficiency, fast charge/discharge rate, eco-environmentally, aging effect, safety risk and material cost for high production of batteries for 100% electric vehicle technology[20, 21]. Safety and cost aspect of Li-ion batteries research by increasing voltage vs energy density by keeping safety at the expense of cost is shown in Figure 1.5. Furthermore, fabricating low voltage 2V-3V systems like $LiBOB/Li_4Ti_5O_{12}/LiFePO_4$[22] means reducing cost and safety simultaneously, but the other drawback of this system is low energy density. Electrode-electrolyte research should be focussed if we move to high specific energy having high energy and high voltage (5V) nanomaterial either cathode/anode. Future generation of solid-state Li-ion batteries which relying on dry polymers/inorganic electrolytes material will bring to zero risk[22].

1.4 Research Importance

The primary focus of this Ph.D. thesis revolves around existing and advanced lithium-ion batteries (LIBs) fabrication based on nanomaterial. Nanostructures are a vital ingredient for the development of many novel advanced materials and electronic devices. The overall battery performance relies on several factors such as synthesis route, chemical composition and surface area of the electrode.

1.4.1 Research Gap

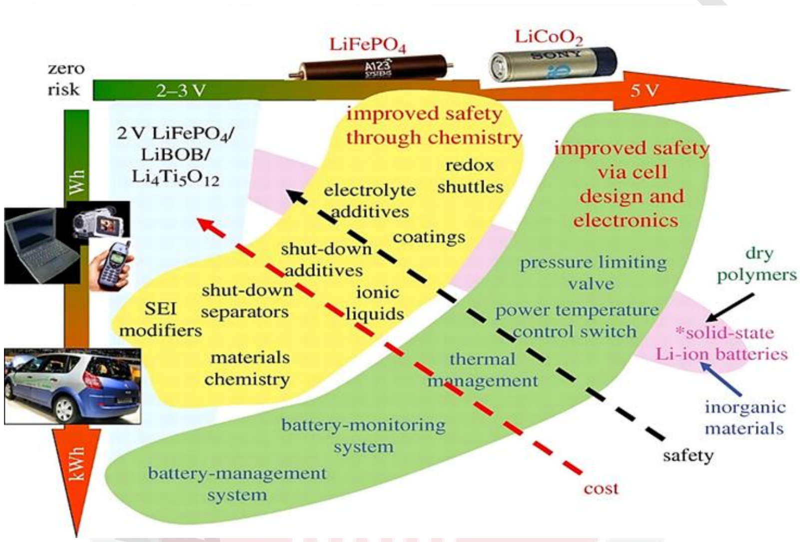

Figure 1.5 : Safety and cost aspect of Li-ion batteries research

Following are the gaps identified based on the literature discussed in section 2.3 of chapter 2 :

- Research gap to combine various synthesis routes both wet and dry chemistry,
- Gap in material characterization like electronic conductivity, and titration techniques,
- Gap in research combining basic battery performance and advance electroanalytical models for lithium-ion diffusion studies,
- Gap in combining lithium-ion battery application by using three electrode reference electrochemical cell.

1.4.2 Contribution

Following are the contribution of doctoral research based on the literature gap identified:

- To fabricate nanomaterials such as cathode spinels $LiMn_2O_4$, fluorine doped spinels $LiMn_2O_{4-x}F_x$ ($0 \leq x \leq 0.2$), by combining various experimental techniques such as sol-gel, sol-gel assisted pechini method, solid-state synthesis,
- To fabricate novel carbonaceous anode nanomaterials synthesized by molten carbonates as exciting new method of producing tuned carbons for lithium ion batteries,
- To optimize the discharge capacity (cycle life) as well as to improve the working voltage of the cathode spinels and anode carbonaceous electrode materials,
- To combine battery performance with advanced electrochemical diffusion properties using galvanostatic intermittent titration technique (GITT) and step potential electrochemical spectroscopy (SPECS),
- To compare various lithium-ion diffusion electroanalytical models based on SPECS and GITT,
- To construct two-electrode coin cell CR2032 for battery performance,
- To construct three-electrode Swagelok type electrochemical cell with both counter and reference electrode as lithium metal for studying kinetic as well as diffusion properties.

1.5 Research Objective and Structure

The main objective of the current thesis is to optimize the energy density, operating voltage, rate capability and cycle life as well as the power density of existing cathode and anode material for lithium-ion batteries (LIBs). The overall goal is to fabricate novel nanomaterials for lithium-ion batteries by using various experimental techniques such as sol-gel, sol-gel assisted pechini method solid-state synthesis. The physical characterization followed by electrochemical energy storage lithium-ion battery application. Further investigation has been conducted on conductivity and lithium-ion diffusion in above-developed materials using electrochemical impedance spectroscopy (EIS) Nyquist plots. The main scope of the current research was as follows:

- Literature review of the state-of-the-art nanostructured Li-ion batteries and mechanistic properties by understanding various diffusion models (CH: 2),
- To compare spinel $LiMn_2O_4$ prepared by solid state and sol-gel methods with commercially available cathode spinel $LiMn_2O_4$ material. The

crystal structure, materials morphology and elemental composition were characterized by x-ray diffraction (XRD), field emission scanning electron microscopy (FE-SEM), energy dispersive x-ray spectroscopy (EDS), and high-resolution transmission electron microscopy (HR-TEM). By using electrochemical characterization techniques for battery performance such as cyclic voltammetry (CV), galvanostatic charge-discharge (GCD), cycle ability (CA), rate capability (RC). Also, novelty is in diffusion properties were studied by step potential electrochemical spectroscopy (SPECS) and via glvanostatic intermittent titration technique (GITT) (CH: 3)

- To synthesize anion doped nanocomposite spinels $LiMn_2O_4$ using sol-gel synthesis route. To investigate the effect of Fluorine with different concentrations in spinel material $LiMn_2O_{4-x}F_x$ ($0 \leq x \leq 0.2$) with (x=0.05, 0.1, 0.2), in order to understand the physical, structure morphology, conductivity, and electrochemical properties of the developed cathode materials (CH: 4),
- Novel carbonaceous materials were synthesized by using molten carbonates as exciting new method of producing tuned carbons as anode for Li-ion battery applications. Electrodeposited carbonaceous materials morphology and elemental composition were characterized by x-ray diffraction (XRD), field emission scanning electron microscopy (FE-SEM), energy dispersive x-ray spectroscopy (EDS). The electrochemical properties were measured using Galvanostatic charge-discharge (GCD), cycle ability (CA), rate performance, cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS) (CH: 5),
- Finally, a summary of the doctoral project and the future recommendation were made for improvement as well as enhancement of the cathode/anode material for Li-ion battery applications. (CH: 6).

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