

## **DELHI TECHNOLOGICAL UNIVERSITY**

### **CERTIFICATE**

This is to certify that Abhishek Kumar Singh (Roll No. 2K13/NST/03) has successfully completed project work on "Synthesis and Characterization of Alternative Anode Material, Li<sub>4</sub>Ti<sub>5</sub>O<sub>12</sub> by Solid State Route" in partial fulfillment of the requirements of the M.Tech in Nano Science & Technology of Delhi Technological University under the supervision and guidance of Dr. Amrish K. Panwar, Assistant Professor, Department of Applied Physics, DTU.

.....

**Dr. Amrish K. Panwar** Assistant professor Dept. of Applied Physics Delhi Technological University, Delhi - 110042 .....

**Prof. S. C. Sharma** HOD, Dept. of Applied Physics. Delhi Technological University, Delhi - 110042

### DECLARATION

I hereby declare that the work presented in this dissertation entitled "Synthesis and Characterization of Alternative Anode Material, Li<sub>4</sub>Ti<sub>5</sub>O<sub>12</sub> by Solid State Route", has been carried out by me under the guidance of Dr. Amrish K. Panwar, Assistant Professor and hereby submitted for the partial fulfillment for the award of degree of Master of Technology in Nanoscience and Technology at Applied Physics Department, Delhi Technological University (Formerly Delhi College of Engineering), New Delhi.

I further undertake that information and data enclosed in this dissertation is original and has not been submitted to any University/Institute for the award of any other degree.

#### Abhishek Kumar Singh

(2K13/NST/03)

M.Tech. Nanoscience & Technology

Delhi Technological University

Delhi.

### **ACKNOWLEDGEMENT**

I gratefully acknowledge my indebtedness to my project guide **Dr. Amrish K. Panwar** and my M.Tech coordinator **Dr. Pawan K. Tyagi** and our HOD **Prof. Suresh C. Sharma** research scholar **Mr.Aditya Jain** and **Mr. Rakesh Saroha** and my classmate **Ms. Divya singh** for their cooperation and suggestions during the preparation of this project work. Our teachers have been a great source of inspiration for us who has given the ideas related to our project topic and most of their ideas and suggestions have been incorporated in this report. Some of our classmates proved to be a great source of information in finalizing the project topic from concept to finishing of the project. We express our deep gratitude to the friends and teachers for their cooperation, suggestions and comments.

#### Abhishek Kumar Singh

(Roll. No. 2K13/NST/03)M.Tech (Nano Science & Technology)Department of Applied Physics

# TABLE OF CONTENTS

CERTIFICATE	Ι
DECLARATION	II
ACKNOWLEDGEMENT	III
LIST OF FIGURES	VI
LIST OF TABLES	VII
ABSTRACT	VIII
CHAPTER-1-INTRODUCTION	
1.1. Introduction to Batteries	1
1.2. Working of Li-ion batteries	2
1.3. Battery safety	5
1.3.1. Overcharging	5
1.3.2. Over temperature	6
1.3.3. Mechanical abuse	6
1.4. Scope of thesis	7
CHAPTER-2-LITRATURE REVIEW	
2.1. Introduction	8
2.2. Anode material for Li-ion batteries	8
2.2.1. Lithium metal as Anode	8
2.2.2. Ordinate of Lithium metal as anode	8
2.3. Introduction to $Li_4Ti_5O_{12}$	10
2.4 Key features of $Li_4Ti_5O_{12}$ as potential negative electrode	10
2.5. Structure of Li <sub>4</sub> Ti <sub>5</sub> O <sub>12</sub>	11
2.6. Structure during cell charging and discharging	12
2.7. Synthesis methods	16
2.7.1. Solid State method	16

2.7.2. Hydrothermal method	16
2.7.3. Sol-Gel method	16
2.7.4. Other methods	17
CHAPTER-3-SYNTHESIS AND EXPERIMENTAL WORK	
3.1. Introduction	18
3.2. Experimental work	18
3.2.1. Synthesis procedure	18
3.2.2. Solid state reaction method	19
3.3 characterization methods	20
3.3.1 XRD	20
3.3.2 SEM	23
3.3.3 EDS	23
3.3.4 Conductivity Measurement	24
3.3.5 I-V characteristics	25
3.3.6 Activation Energy Calculation	26
CHAPTER-4-RESULTS AND DISCUSSION	
4.1. XRD Results	27
4.2. SEM images of pure LTO and C <sub>2</sub> H <sub>2</sub> treated LTO	28
4.4. EDS Results	29
4.5. Conductivity measurement	31
4.6. I-V Characteristics	32
4.7. Activation Energy	33
CHAPTER-5- SUMMARY AND CONCLUSION	34
ACKNOWLEGEMENT	35
REFERENCES	36
	50

# **LIST OF FIGURES**

Figure 1.1. Working and charging/ discharging process of Li-ion battery with graphite as an	node
and LiCoO <sub>2</sub> as cathode	3
Figure 1.2. Thermal reactions in Li-ion batteries with metal-oxide poselectrode	
Fig 2.1. Structure of Li <sub>4</sub> Ti <sub>5</sub> O <sub>12</sub>	12
Fig 2.2. Change in structure during charging and discharging process	13
<b>Fig 2.3.</b> Structure of Spinal type of Li <sub>4</sub> Ti <sub>5</sub> O <sub>12</sub>	14
<b>Fig 2.4</b> Structure of Rock salt type of Li <sub>7</sub> Ti <sub>5</sub> O <sub>12</sub>	15
Fig 3.1. Block diagram representation of solid state approach of synthesis	20
Fig 3.2. Theoretical diagram of XRD	21
Fig 3.3. Set up used to measure XRD patterns of LTO	22
Fig.3.4. Schematic diagram for the method of EDS analysis	24
Fig 4.1. XRD pattern for LTO and Carbon treated LTO	27
Fig 4.2. SEM images of Li <sub>4</sub> Ti <sub>5</sub> O <sub>12</sub>	28
<b>Fig 4.3.</b> SEM images of carbon treated Li <sub>4</sub> Ti <sub>5</sub> O <sub>12</sub>	29
Fig 4.4. EDS graph of LTO	30
Fig 4.5. Nyquist plot	31
Fig 4.6. I-V curve	32
Fig 4.7. Graph for the Activation Energy calculation	33

# **LIST OF TABLES**

<b>Table 1.1.</b> Characteristics and comparison of widely used rechargeable batteries	2
<b>Table 2.1.</b> List of components for different Li-ion batteries.	9
<b>Table 2.2.</b> List of the specific and volumetric capacities for the different lithium ion anode materials.	2
<b>Table: 4.1</b> Quantitative data of elements in wt. % of LTO observed in EDS	30

### **ABSTRACT**

As the demand for the better, lighter and more efficient microelectronic portable devices increases, the development of lithium ion batteries as power sources with high performance becomes essential. Within this frame work alternative Li<sub>4</sub>Ti<sub>5</sub>O<sub>12</sub> (LTO) anode material has been prepared and characterized to evaluate its electrical properties.

Synthesis of LTO through solid state route is considered as economical, efficient and easy way for the mass production. Therefore, spinel type LTO is prepared using solid state reaction route. Further, the different physio-chemical characterization such as SEM, EDS, XRD, Conductivity measurement, I-V characteristics and activation energy calculation for LTO was performed. To optimize performance of lithium ion batteries in terms of good cycle ability, capacity and power density during the electrochemical analysis of batteries. These characterizations showed that LTO has low conductivity and have high activation energy. Hence to improve the conductivity, C<sub>2</sub>H<sub>2</sub> treatment was performed on LTO. Which showed improvement in its characteristics by lowering the activation energy and increasing the conductivity due to carbon deposition.