

**PREPARATION AND CHARACTERIZATIONS OF LITHIUM
TITANATE ($\text{Li}_4\text{Ti}_5\text{O}_{12}$)**

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**FACULTY OF SCIENCE
UNIVERSITY OF MALAYA
KUALA LUMPUR**

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**PREPARATION AND CHARACTERIZATIONS OF LITHIUM
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KUALA LUMPUR**

2011

DECLARATION

I hereby declare that the work reported in this dissertation is my own unless specified and duly acknowledged by quotation.

1st APRIL 2011

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(NOR AINI ALIAS)

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ABSTRACT

$\text{Li}_4\text{Ti}_5\text{O}_{12}$ is a potential anode material for lithium-ion batteries. This serves as the motivation for the present study. $\text{Li}_4\text{Ti}_5\text{O}_{12}$ compound has been obtained through the sol-gel technique. From thermo gravimetric analysis, $\text{Li}_4\text{Ti}_5\text{O}_{12}$ is thermally stable at temperatures above 700 °C. Organic compounds are observed to decompose at 600 °C and below. From XRD diffractograms peaks due to Li_2TiO_3 and anatase TiO_2 phases observed, $\text{Li}_4\text{Ti}_5\text{O}_{12}$ has a cubic structure. The lattice parameters for all $\text{Li}_4\text{Ti}_5\text{O}_{12}$ are sintered at different temperatures and times are approximately 8.3 Å. The discharge capacity of a cell employing $\text{Li}_4\text{Ti}_5\text{O}_{12}$ as electrode is ~60 mAh g⁻¹ for 20 cycles and the voltage plateau occurs at ~1.5 V corresponding to redox reaction of Ti^{4+} and Ti^{3+} .

In the attempt to produce good dielectric materials particularly for use in capacitor, oxides have been incorporated in polymer host such as poly (vinyl alcohol), PVA. PVA films doped with different amounts of $\text{Li}_4\text{Ti}_5\text{O}_{12}$ have been prepared via the solvent cast technique. The semi-crystallinity of PVA decreases with addition of $\text{Li}_4\text{Ti}_5\text{O}_{12}$ as identified from the XRD diffractogram. C=C stretching band is observed to shift from lower to higher wavenumber i.e. from 1571 cm⁻¹ to 1583 cm⁻¹. From absorption spectroscopy analysis, the edge absorption is observed to shift from 272 nm to 254 nm. Morphology of PVA films doped $\text{Li}_4\text{Ti}_5\text{O}_{12}$ is coarser compared to that of pure PVA film which is homogeneous and smooth. The plot of real modulus versus log f , does not exhibit a relaxation peak. Argand plots clearly show viscoelastic relaxation due its tilted semicircle.

ABSTRAK

Sebatian $\text{Li}_4\text{Ti}_5\text{O}_{12}$ merupakan bahan anod berpotensi untuk bateri lithium-ion. Hakikat ini dijadikan motivasi untuk melangsungkan kajian ini. Sebatian $\text{Li}_4\text{Ti}_5\text{O}_{12}$ berjaya dihasilkan melalui teknik sol-gel. Daripada analisis thermo gravimetri, $\text{Li}_4\text{Ti}_5\text{O}_{12}$ stabil haba pada suhu melebihi $700\text{ }^\circ\text{C}$. Sebatian organik didapati terurai pada suhu $600\text{ }^\circ\text{C}$ dan ke bawah. Daripada difraktogram XRD, puncak fasa Li_2TiO_3 dan anatase TiO_2 dikenalpasti. $\text{Li}_4\text{Ti}_5\text{O}_{12}$ berstruktur kubik. Parameter kekisi untuk semua $\text{Li}_4\text{Ti}_5\text{O}_{12}$ yang dihasilkan pada suhu dan masa pensinteran berlainan menghampiri 8.3 \AA . Kapasiti discas sel yang menggunakan $\text{Li}_4\text{Ti}_5\text{O}_{12}$ sebagai elektrod adalah $\sim 60\text{ mAh g}^{-1}$ selama 20 kitaran dan daripada profile cas-diskas dataran voltan berada pada $\sim 1.5\text{ V}$ yang dikaitkan dengan tindak balas redoks antara Ti^{4+} dan Ti^{3+} .

Dalam usaha untuk menghasilkan bahan dielektrik yang baik khusus untuk penggunaan dalam kapasitor, pelbagai jenis oksida telah digabungkan ke dalam hos polimer seperti poli (vinil alkohol), PVA. Filem-filem PVA terdop dengan jumlah $\text{Li}_4\text{Ti}_5\text{O}_{12}$ berlainan telah disediakan melalui teknik penuangan larutan. Fasa semi-hablur PVA berkurang seiring dengan penambahan $\text{Li}_4\text{Ti}_5\text{O}_{12}$ dan ini dikenalpasti melalui difraktogram XRD. Jalur peregangan C=C didapati berubah dari nombor gelombang rendah ke tinggi dengan penambahan $\text{Li}_4\text{Ti}_5\text{O}_{12}$ iaitu dari 1571 cm^{-1} kepada 1583 cm^{-1} . Analisis spektrum serapan menunjukkan pinggir serapan gelombang berubah daripada 272 nm kepada 254 nm . Morfologi filem PVA terdop $\text{Li}_4\text{Ti}_5\text{O}_{12}$ kasar berbanding morfologi PVA tulen yang licin dan sekata. Plot modulus nyata lawan $\log f$, tidak menunjukkan puncak santaian. Plot Argand, menunjukkan dengan jelas santaian viskoelastik disebabkan bentuk semi-bulatan yang tersenget.

TABLE OF CONTENTS

| | Pages |
|---|--------------|
| DECLARATION | i |
| ACKNOWLEDGEMENT | ii |
| PUBLICATIONS | iii |
| ORIGINAL LITERARY WORK DECLARATION | iv |
| ABSTRACT | v |
| ABSTRAK | vi |
| TABLE OF CONTENTS | vii |
| LIST OF FIGURES | x |
| LIST OF TABLES | xiii |
| LIST OF ABBREVIATIONS | xiv |
| CHAPTER 1: INTRODUCTION TO DISSERTATION | 1 |
| CHAPTER 2: LITERATURE REVIEW | 4 |
| 2.1 Introduction | 4 |
| 2.2 Anode materials for rechargeable lithium ion batteries | 4 |
| 2.2.1 Lithium metal | 5 |
| 2.2.2 Carbonaceous materials | 7 |
| 2.2.2.1 Graphite | 7 |
| 2.2.2.2 Soft carbon | 9 |
| 2.2.2.3 Hard carbon | 11 |
| 2.2.3 $\text{Li}_{4.4}\text{Sn}$ | 13 |
| 2.2.4 $\text{Li}_2\text{Ti}_3\text{O}_7$ | 14 |
| 2.2.5 $\text{Li}_4\text{Ti}_5\text{O}_{12}$ | 14 |
| 2.3 Previous $\text{Li}_4\text{Ti}_5\text{O}_{12}$ synthesis techniques | 17 |

| | | |
|---|---|-----------|
| 2.3.1 | Solid state reaction | 18 |
| 2.3.2 | Hybrid microwave technique | 19 |
| 2.3.3 | Ball-milling technique | 19 |
| 2.3.4 | Sol-gel technique | 20 |
| 2.3.5 | Blend sample preparation | 21 |
| 2.4 | Electrochemical cell characterization | 22 |
| 2.4.1 | Charge-discharge | 22 |
| 2.5 | Polymer composite | 23 |
| 2.5.1 | Poly (vinyl alcohol), PVA | 25 |
| 2.6 | Summary | 26 |
| CHAPTER 3: METHODOLOGY | | 27 |
| 3.1 | Introduction | 27 |
| 3.2 | Sample preparation | 27 |
| 3.2.1 | Powder synthesis | 27 |
| 3.2.2 | Composite film | 28 |
| 3.3 | Sample characterizations | 29 |
| 3.3.1 | Thermo gravimetry mass analysis | 29 |
| 3.3.2 | X-ray diffraction (XRD) | 31 |
| 3.3.3 | Electrochemistry impedance spectroscopy (EIS) | 33 |
| 3.3.4 | Fourier transform infrared (FTIR) spectroscopy | 34 |
| 3.3.5 | Scanning electron microscope (SEM) | 35 |
| 3.3.6 | UV-Vis spectroscopy | 35 |
| 3.4 | Electrochemical cell fabrication and characterization | 36 |
| 3.5 | Summary | 37 |
| CHAPTER 4: STUDIES ON LITHIUM TITANATE | | 38 |

| | | |
|---|---|-----------|
| 4.1 | Introduction | 38 |
| 4.2 | Thermo gravimetric-mass spectroscopy (TGMS) | 38 |
| 4.3 | X-ray diffraction (XRD) | 43 |
| 4.4 | Battery performance | 48 |
| 4.5 | Summary | 50 |
| CHAPTER 5: PVA DOPED WITH $\text{Li}_4\text{Ti}_5\text{O}_{12}$ | | 51 |
| 5.1 | Introduction | 51 |
| 5.2 | X-ray diffraction (XRD) | 52 |
| 5.3 | Fourier transform infrared (FTIR) | 53 |
| 5.4 | UV-Vis spectroscopy | 57 |
| 5.5 | Scanning electron microscopy (SEM) | 60 |
| 5.6 | Dielectric studies | 62 |
| 5.7 | Dielectric relaxation process | 70 |
| 5.8 | Summary | 73 |
| CHAPTER 6: DISCUSSIONS | | 75 |
| CHAPTER 7: CONCLUSIONS AND FUTURE WORKS | | 81 |
| REFERENCES | | 83 |

LIST OF FIGURES

| Figures | | Pages |
|-------------|--|-------|
| Figure 2.1 | Structure of hexagonal graphite showing ABAB stacking and schematic of cross-section of stacking layers considering the thickness of each carbon layer [Azuma <i>et al.</i> , 1999]. | 8 |
| Figure 2.2 | Micrograph of a mesocarbon fiber (MCF) [Takamura, 2002]. | 8 |
| Figure 2.3 | Structure of soft carbon [Azuma <i>et al.</i> , 1999]. | 9 |
| Figure 2.4 | Structure of 1 soft carbon before and after doped with lithium ions [Azuma <i>et al.</i> , 1999]. | 9 |
| Figure 2.5 | Structure of hard carbon before and after doped with lithium ion [Azuma <i>et al.</i> , 1999]. | 12 |
| Figure 2.6 | Intercalation into the $\text{Li}_4\text{Ti}_5\text{O}_{12}$ spinel and de-intercalation out of the LiCoO_2 layered structure form an ideal cell couple [Jansen <i>et al.</i> , 1999]. | 16 |
| Figure 2.7 | SEM images for (a) $\text{Li}_4\text{Ti}_5\text{O}_{12}$ obtained by high temperature solid-state reaction between TiO_2 (anatase) and LiOH and (b) submicron-sized TiO_2 and Li_2CO_3 dispersed in hexane followed by heating [Peramunage and Abraham, 1998]. | 19 |
| Figure 2.8 | SEM micrograph of $\text{Li}_{1.3}\text{Al}_{0.3}\text{Ti}_{1.7}(\text{PO}_4)_3$ sintered pellets prepared by (a) solid-state reaction and (b) sol-gel technique [Wu <i>et al.</i> , 2004]. | 21 |
| Figure 2.9 | SEM images of $\text{Li}_4\text{Ti}_5\text{O}_{12}$ (a) without and (b) with assist ball-milling technique [Yan <i>et al.</i> , 2009]. | 22 |
| Figure 2.10 | Charge and discharge profile of $\text{Li}_4\text{Ti}_5\text{O}_{12}$ samples in 1 M $\text{LiClO}_4/\text{EC} + \text{DEC}$ (1:1 in volume) at 0.1 C rate at various heat treatment (a) 500 °C for three hours, (b) 800 °C in an electric furnace for ten hours and (c) 800 °C in an infrared furnace for one minute [Kanamura <i>et al.</i> , 2006]. | 24 |
| Figure 3.1 | Powder synthesis flowchart. | 28 |
| Figure 3.2 | TG and DSC curves of the codoped precursor solution with $\text{Zn:N:Al}=1:3:0.05$ [Zhang, 2009]. | 30 |
| Figure 3.3 | Mass spectra of HN_3 and H_2O in the resultant gases of the codoped precursor solution [Zhang, 2009]. | 30 |
| Figure 3.4 | XRD patterns of $\text{LiNi}_{0.3}\text{Co}_{0.7}\text{O}_2$ sintered at different temperatures [Hernandez <i>et al.</i> , 2008]. | 32 |
| Figure 3.5 | Complex impedance plot at 300 °C for the $\text{Na}_{1+x}\text{Al}_x\text{Ge}_{2-x}\text{P}_3\text{O}_{12}$ glass-ceramics calcined at 800 °C for 18 hours and the equivalent circuit is shown on the top left corner [Zhang <i>et al.</i> , 2009]. | 33 |
| Figure 3.6 | The variation of the absorption with the wavelength for different concentrations of TiCl_3 [Abdelaziz and Ghannam, 2010]. | 36 |
| Figure 3.7 | The systematic diagram of cell construction. | 37 |
| Figure 4.1 | TG-MS profile for the powder precursors of $\text{Li}_4\text{Ti}_5\text{O}_{12}$ heated in oven at 100 °C for one hour. | 40 |
| Figure 4.2 | Thermal degradation of powder precursors of $\text{Li}_4\text{Ti}_5\text{O}_{12}$ heated in oven at 100 °C for one hour. | 41 |

| | | |
|---------------|---|----|
| Figure 4.3 | XRD profile for the powder precursors of $\text{Li}_4\text{Ti}_5\text{O}_{12}$ heated in oven at (a) 100 °C and (b) 600 °C for one hour. | 43 |
| Figure 4.4 | XRD pattern for $\text{Li}_4\text{Ti}_5\text{O}_{12}$ sintered at (a) 700 °C, (b) 800 °C, (c) 900 °C and (d) 1000 °C for one hour. (*) indicates impurity due to Li_2TiO_3 . | 44 |
| Figure 4.5 | XRD profile of $\text{Li}_4\text{Ti}_5\text{O}_{12}$ sintered at 800 °C for (a) one, (b) two, (c) three, (d) four and (e) five hours. | 45 |
| Figure 4.6 | The charge-discharge profile for the $\text{Li}/\text{Li}_4\text{Ti}_5\text{O}_{12}$ cell. | 49 |
| Figure 4.7 | Discharge capacity of $\text{Li}_4\text{Ti}_5\text{O}_{12}$ at a constant current density of 21.37 mA g ⁻¹ . | 50 |
| Figure 5.1 | PVA film doped with 8 wt. % $\text{Li}_4\text{Ti}_5\text{O}_{12}$. | 51 |
| Figure 5.2 | XRD profile of (a) $\text{Li}_4\text{Ti}_5\text{O}_{12}$, (b) film of PVA dissolved in water, (c) film of PVA dissolved in 1 % acetic acid and doped with (d) 2 wt. % $\text{Li}_4\text{Ti}_5\text{O}_{12}$, (e) 4 wt. % $\text{Li}_4\text{Ti}_5\text{O}_{12}$, (f) 6 wt. % $\text{Li}_4\text{Ti}_5\text{O}_{12}$, (g) 8 wt. % $\text{Li}_4\text{Ti}_5\text{O}_{12}$ and (h) 10 wt. % $\text{Li}_4\text{Ti}_5\text{O}_{12}$. (*) indicates $\text{Li}_4\text{Ti}_5\text{O}_{12}$. | 52 |
| Figure 5.3(a) | FTIR spectrum for film of PVA dissolve in water from 500 cm ⁻¹ to 4000 cm ⁻¹ . | 54 |
| Figure 5.3(b) | FTIR spectrum for film of PVA dissolved in 1 % of acetic acid from 500 cm ⁻¹ to 4000 cm ⁻¹ . | 55 |
| Figure 5.4 | FTIR spectra of PVA film (dissolved in 1 % acetic acid) and doped with (a) 2 wt. % $\text{Li}_4\text{Ti}_5\text{O}_{12}$, (b) 4 wt. % $\text{Li}_4\text{Ti}_5\text{O}_{12}$, (c) 6 wt. % $\text{Li}_4\text{Ti}_5\text{O}_{12}$, (d) 8 wt. % $\text{Li}_4\text{Ti}_5\text{O}_{12}$ and (e) 10 wt. % $\text{Li}_4\text{Ti}_5\text{O}_{12}$. | 56 |
| Figure 5.5 | FTIR spectra of PVA film (dissolved in 1 % acetic acid) and doped with (a) 2 wt. % $\text{Li}_4\text{Ti}_5\text{O}_{12}$, (b) 4 wt. % $\text{Li}_4\text{Ti}_5\text{O}_{12}$, (c) 6 wt. % $\text{Li}_4\text{Ti}_5\text{O}_{12}$, (d) 8 wt. % $\text{Li}_4\text{Ti}_5\text{O}_{12}$ and (e) 10 wt. % $\text{Li}_4\text{Ti}_5\text{O}_{12}$ from 2000 cm ⁻¹ to 4000 cm ⁻¹ . | 57 |
| Figure 5.6 | The UV-Vis absorption spectra of pure PVA film obtained from casting the solution of the polymer in (a) water and (b) acetic acid. | 58 |
| Figure 5.7 | The UV-Vis absorption spectra of PVA films (dissolved in acetic acid) and doped with (a) 2 wt. %, (b) 4 wt. %, (c) 6 wt. %, (d) 8 wt. % and (e) 10 wt. % $\text{Li}_4\text{Ti}_5\text{O}_{12}$. | 59 |
| Figure 5.8(a) | SEM image of PVA film dissolved in water. Circles show white spot. | 60 |
| Figure 5.8(b) | SEM images of PVA film dissolved in acetic acid. | 61 |
| Figure 5.9 | SEM images of PVA film doped with (a) 2 wt. %, (b) 4 wt. %, (c) 6 wt. %, (d) 8 wt. % and (d) 10 wt. % $\text{Li}_4\text{Ti}_5\text{O}_{12}$. | 62 |
| Figure 5.10 | Dielectric constant (ϵ_r) versus log frequency of (a) PVA water, (b) PVA acetic acid, PVA doped with (c) 2 wt. %, (d) 4 wt. %, (e) 6 wt. %, (f) 8 wt. % and (g) 10 wt. % of $\text{Li}_4\text{Ti}_5\text{O}_{12}$. | 63 |
| Figure 5.11 | Real modulus versus log frequency of (a) PVA water, (b) PVA acetic acid, PVA doped with (c) 2 wt. %, (d) 4 wt. %, (e) 6 wt. %, (f) 8 wt. % and (g) 10 wt. % of $\text{Li}_4\text{Ti}_5\text{O}_{12}$. | 64 |
| Figure 5.12 | Imaginary modulus versus log frequency of (a) PVA water, (b) PVA acetic acid, PVA doped with (c) 2 wt. %, (d) 4 wt. %, (e) 6 wt. %, (f) 8 wt. % and (g) 10 wt. % of $\text{Li}_4\text{Ti}_5\text{O}_{12}$. | 64 |
| Figure 5.13 | Dissipation factor ($\tan \delta$) versus log frequency of (a) PVA water, (b) PVA acetic acid, PVA doped with (c) 2 wt. %, (d) | 65 |

| | | |
|-------------|--|-------|
| | 4 wt. %, (e) 6 wt. %, (f) 8 wt. % and (g) 10 wt. % of $\text{Li}_4\text{Ti}_5\text{O}_{12}$. | |
| Figure 5.14 | Tan δ versus $\log f$ (Hz) at various temperatures for film of PVA dissolved in water. | 66 |
| Figure 5.15 | Tan δ versus $\log f$ (Hz) at various temperatures for film of PVA dissolved in acetic acid solvent. | 67 |
| Figure 5.16 | Tan δ versus $\log f$ (Hz) at various temperatures for films of PVA doped with 2 wt. % $\text{Li}_4\text{Ti}_5\text{O}_{12}$. | 67 |
| Figure 5.17 | Tan δ versus $\log f$ (Hz) at various temperatures for films of PVA doped with 4 wt. % $\text{Li}_4\text{Ti}_5\text{O}_{12}$. | 68 |
| Figure 5.18 | Tan δ versus $\log f$ (Hz) at various temperatures for films of PVA doped with 6 wt. % $\text{Li}_4\text{Ti}_5\text{O}_{12}$. | 69 |
| Figure 5.19 | Tan δ versus $\log f$ (Hz) at various temperatures for films of PVA doped with 8 wt. % $\text{Li}_4\text{Ti}_5\text{O}_{12}$. | 69 |
| Figure 5.20 | Tan δ versus $\log f$ (Hz) at various temperatures for films of PVA doped with 10 wt. % $\text{Li}_4\text{Ti}_5\text{O}_{12}$. | 70 |
| Figure 5.21 | Argand plots derived from the conductivity relaxation region for pure PVA based solvent (a) water and (b) acetic acid. | 72 |
| Figure 5.22 | Argand plots PVA doped with different concentrations of $\text{Li}_4\text{Ti}_5\text{O}_{12}$ (a) 2 wt. %, (b) 4 wt. %, (c) 6 wt. %, (d) 8 wt. % and (e) 10 wt. %. | 72-73 |

LIST OF TABLES

| Tables | | Pages |
|---------------|--|--------------|
| Table 2.1 | Lists of components for lithium ion batteries. | 5 |
| Table 2.2 | Characteristics of some anode materials for lithium battery. | 6 |
| Table 2.3 | Physical properties of various coke materials [Tran <i>et al.</i> , 1999; Chen <i>et al.</i> , 1995]. | 11 |
| Table 2.4 | Lattice parameter (a) for spinel-type $\text{Li}_{3.95}\text{M}_{0.15}\text{Ti}_{4.9}\text{O}_{12}$ (M=Al, Ga and Co) and $\text{Li}_{3.9}\text{Mg}_{0.1}\text{Al}_{10.15}\text{Ti}_{4.85}\text{O}_{12}$ materials [Huang <i>et al.</i> , 2007]. | 17 |
| Table 2.5 | Previous works on polymer composites. | 25 |
| Table 2.6 | Properties of poly (vinyl alcohol). | 25 |
| Table 3.1 | Composition of composite film studied in this work. | 29 |
| Table 4.1 | List of weight losses for sample pre-treated at 100 °C. | 42 |
| Table 4.2 | The list of lattice parameter and volume at different sintered temperatures. | 46 |
| Table 4.3 | Density of $\text{Li}_4\text{Ti}_5\text{O}_{12}$ at different sintered temperatures. | 46 |
| Table 4.4 | The list of lattice parameter and volume at different sintered times. | 47 |
| Table 4.5 | Density of $\text{Li}_4\text{Ti}_5\text{O}_{12}$ at different sintered times. | 48 |
| Table 5.1 | Infrared peak assignments of PVA polymer. | 53 |

LIST OF ABBREVIATIONS

| | |
|--------|--|
| AE | Absorption edge |
| DEC | Diethyl carbonate |
| DME | Dimethoxyethane |
| DSC | Differential scanning calorimetry |
| DTA | Differential thermal analysis |
| EC | Ethylene carbonate |
| EGA | Evolved gas analyzer |
| EIS | Electrochemical impedance spectroscopy |
| FTIR | Fourier transform infrared |
| LiPON | Lithium phosphorus oxynitride |
| MCF | Mesocarbon fiber |
| MCMB | Mesocarbon microbeads |
| MS | Mass spectroscopy |
| PC | Propylene carbonate |
| PS | Poly styrene |
| PVA | Poly (vinyl alcohol) |
| PVDF | Poly (vinylidene fluoride) |
| SEI | Solid electrolyte interface |
| SEM | Scanning electron microscopy |
| SHE | Standard hydrogen electrode |
| TAB | Teflon acetylene black |
| TG | Thermo gravimetric |
| TGA | Thermo gravimetry analysis |
| TGA-MS | Thermo gravimetry analysis-mass spectroscopy |
| UV-Vis | Ultraviolet-visible |
| XRD | X-ray diffraction |