PREPARATION AND CHARACTERIZATIONS OF LITHIUM TITANATE ($Li_4Ti_5O_{12}$)

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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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PUBLICATIONS

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List of Poster Presentations Presented at Conferences:

1. **N.A. Alias.,** S.R. Majid and A.K. Arof. "Structural and Conductivity Studies of $Li_4Ti_5O_{12}$", presented at the National Workshop on Functional Materials (NWFM), 20-21 June 2009, University of Malaya, Kuala Lumpur.
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ABSTRACT

$Li_4Ti_5O_{12}$ is a potential anode material for lithium-ion batteries. This serves as the motivation for the present study. $Li_4Ti_5O_{12}$ compound has been obtained through the sol-gel technique. From thermo gravimetric analysis, $Li_4Ti_5O_{12}$ is thermally stable at temperatures above 700 ^oC. Organic compounds are observed to decompose at 600 ^oC and below. From XRD diffractograms peaks due to Li_2TiO_3 and anatase TiO_2 phases observed, $Li_4Ti_5O_{12}$ has a cubic structure. The lattice parameters for all $Li_4Ti_5O_{12}$ are sintered at different temperatures and times are approximately 8.3 Å. The discharge capacity of a cell employing $Li_4Ti_5O_{12}$ 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 $Li_4Ti_5O_{12}$ have been prepared via the solvent cast technique. The semi-crystallinity of PVA decreases with addition of $Li_4Ti_5O_{12}$ 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 $Li_4Ti_5O_{12}$ 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 $Li_4Ti_5O_{12}$ merupakan bahan anod berpotensi untuk bateri lithium-ion. Hakikat ini dijadikan motivasi untuk melangsungkan kajian ini. Sebatian $Li_4Ti_5O_{12}$ berjaya dihasilkan melalui teknik sol-gel. Daripada analisis thermo gravimetri, $Li_4Ti_5O_{12}$ stabil haba pada suhu melebihi 700 ^oC. Sebatian organik didapati terurai pada suhu 600 ^oC dan ke bawah. Daripada difraktogram XRD, puncak fasa Li_2TiO_3 dan anatase TiO_2 dikenalpasti. $Li_4Ti_5O_{12}$ berstruktur kubik. Parameter kekisi untuk semua $Li_4Ti_5O_{12}$ yang dihasilkan pada suhu dan masa pensinteran berlainan menghampiri 8.3 Å. Kapasiti discas sel yang menggunakan $Li_4Ti_5O_{12}$ sebagai elektrod adalah ~60 mAh g^{-1} selama 20 kitaran dan daripada profile cas-discas dataran voltan berada pada ~1.5 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 sepeerti poli (vinil alkohol), PVA. Filem-filem PVA terdop dengan jumlah $Li_4Ti_5O_{12}$ berlainan telah disediakan melalui teknik penuangan larutan. Fasa semi-hablur PVA berkurang seiring dengan penambahan $Li_4Ti_5O_{12}$ dan ini dikenalpasti melalui difraktogram XRD. Jalur peregangan C=C didapati berubah dari nombor gelombang rendah ke tinggi dengan penambahan $Li_4Ti_5O_{12}$ iaitu dari 1571 cm^{-1} kepada 1583 cm^{-1}. Analisis spektrum serapan menunjukkan pinggiran serapan gelombang berubah daripada 272 nm kepada 254 nm. Morfologi filem PVA terdop $Li_4Ti_5O_{12}$ 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.

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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