SYNTHESIS AND CHARACTERIZATION OF MoX_2 (X = O, S, Se, Te) AND THEIR ELECTROCHEMICAL PROPERTIES

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Thesis submitted in fulfillment of the requirements
for the award of the degree of
Bachelor of Applied Science (Hons) Material Technology

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SUPERVISOR'S DECLARATION

I hereby declare that I have checked this thesis and in my opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Bachelor of Applied Science (Hons) Material Technology.

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STUDENT'S DECLARATION

I hereby declare that the work in this thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Malaysia Pahang or any other institutions.

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Dedicated to my family, my friends and my lecturers

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LIST OF SYMBOLS / UNITS

A/g	-	ampere per gram
Å	-	angstrom (10^{-10})
~	-	approximately
atm	-	atmosphere
2θ	-	Bragg angle
$cm^2/V.s$	-	centimeter square per volt per second
X	-	chalcogen
R_{ct}	-	charge transfer resistance
η	-	coulombic efficiency
I	-	current
i	-	current density
°	-	degree
°C	-	degree celcius
°/min	-	degree per minute
R_d	-	diffusive resistance
t_d	-	discharging time
d	-	d-spacing
E_D	-	energy density
R_s	-	equivalent series resistance
$F\ g^{-1}$	-	farad per gram
g	-	gram
g/mol	-	gram per mole
Hf	-	hafnium

h	-	hour
N_2H_2	-	hydrazine hydrate
HCl	-	hydrochloric acid
H_2	-	hydrogen gas
kX	-	kilo magnification
ktoe	-	kilo tonnes of oil
LiCl-PVA	-	lithium chloride-polyvinyl alcohol
LiOH	-	lithium hydroxide
$LiFePO_4$	-	lithium iron phosphate
$LiPF_4$	-	lithium tetrafluorophosphate
X	-	magnification
m	-	mass
m^2g^{-1}	-	meter square per gram
$m^2s^{-1}V^{-1}$	-	meter square per second per volt
mts	-	metre-tonne-second
μ	-	micron (10^{-6})
mA/cm^2	-	milliampere per centimeter square
mA/g	-	milliampere per gram
ml	-	millilitre
mmol	-	millimole
$mS.m^2mol^{-1}$	-	milli-Siemen meter square per mole
mV/s	-	millivolt per second
M	-	molar
MoX_2	-	molybdenum dichalcogenides
MoO_2	-	molybdenum dioxide

$MoSe_2$	-	molybdenum diselenide
MoS_2	-	molybdenum disulfide
$MoTe_2$	-	molybdenum ditelluride
MoO_3	-	molybdenum trioxide
nm	-	nanometer
Nb	-	niobium
Ω	-	ohm
$C_2H_2O_4$	-	oxalic acid
O	-	oxygen
%	-	percent
KCl	-	potassium chloride
KOH	-	potassium hydroxide
P_D	-	power density
rpm	-	revolutions per minute
Se	-	selenium
Ag	-	silver
AgCl	-	silver chloride
NaOH	-	sodium hydroxide
Na_2MoO_4	-	sodium molybdate
Na_2SO_4	-	sodium sulphate
C_s	-	specific capacitance
S	-	sulphur
H_2SO_4	-	sulphuric acid
Ta	-	tantalum
Te	-	tellurium

t	-	time
Ti	-	titanium
W	-	tungsten
V	-	vanadium
V	-	voltage
H_2O	-	water
Wh/kg	-	Watt hour per kilogram
W/kg	-	Watt per kilogram
W/(m.K)	-	Watt per meter per Kelvin
λ	-	wavelength
Zr	-	zirconium

LIST OF ABBREVIATIONS

CNT	-	carbon nanotube
CNT/ZnO	-	carbon nanotube/zinc oxide composite
CVD	-	chemical vapor deposition
CAES	-	compressed air energy storage
CV	-	cyclic voltammetry
EDLC	-	electrochemical double layer capacitor
EIS	-	electrochemical impedance spectroscopy
FESEM	-	field emission scanning electron microscope
FES	-	flywheel energy storage
e.g.	-	for example
LIB	-	lithium ion battery
pH	-	potential of hyrodgen
PVDF	-	polyvinylidene fluoride
PC	-	pseudocapacitor
PHS	-	pumped hydro energy storage
SEM	-	scanning electron microscope
SC	-	supercapacitor
SMES	-	superconducting magnetic coil energy storage
TMDs	-	transition metal dichalcogenides
TOP	-	trioctyl phosphine
2D	-	two dimensional
XRD	-	X-ray diffraction

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ABSTRACT

Supercapacitor (SC) is a type of energy storage systems which has high energy density and power density. Normally, it can divide into two categories, (i) electrical double layer capacitor (EDLC) and (ii) pseudocapacitor (PC). Nowadays, molybdenum dichalcogenides, a type of pseudocapacitive material, has received the attention from researchers due to two-dimensional layered structure morphology which similar to the structure of graphene. This thesis dealt with synthesis and characterization of molybdenum dichalcogenides in their electrochemical properties. The objective of the thesis was to synthesize molybdenum dichalcogenides. Besides, the molybdenum dichalcogenides synthesized were characterized with physico-chemical and electrochemical techniques, using X-ray Diffractometer, Field Emission Scanning Electron Microscope and potentiostat-galvanostat respectively. Molybdenum dichalcogenides were synthesized with hydrothermal method; the precursors were mixed in a sealed Teflon jacket and incubated it with suitable temperature and period. X-ray diffraction (XRD) was used to reveal the information about crystal structures and chemical compositions of molybdenum dichalcogenides; field emission electron scanning microscopy (FESEM) was used to check the morphologies of molybdenum dichalcogenides; electrochemical testing was used to test the electrochemical properties of molybdenum dichalcogenides. There were three types of analyses involved for electrochemical testing, (i) cyclic voltammetry (CV), (ii) charge discharge (CDC) and (iii) electrochemical impedance spectroscopy (EIS). The acquired results showed that molybdenum trioxide (MoO_3), molybdenum disulphide (MoS_2) and molybdenum diselenide ($MoSe_2$) had been produced using hydrothermal method. Each of the molybdenum dichalcogenides had its own structure. MoO_3 possessed nanosheet structure; MoS_2 possessed nanorod structure; $MoSe_2$ possessed nanorod and nanoplate structures. From electrochemical testing, MoS_2 showed better performance in specific capacitance, energy density and coulombic efficiency, with values 117.65 F/g at 5 mV/s, 1.63 Wh/kg at 0.3 A/g and 98.93% respectively.

ABSTRAK

Superkapasitor ialah sejenis peranti penyimpan tenaga yang mempunyai kemampuan penyimpanan muatan dan rapat daya yang besar. Biasanya, superkapasitor boleh dibahagikan kepada dua kategori, (i) double-layer kapasitor (EDLC) dan (ii) pseudokapasitor (PC). Kini, molibdenum dikalkogenida, sejenis bahan pseudokapasitif, telah mendapat perhatian daripada penyelidik kerana struktur berlapis dalam dua dimensi yang lebih kurang sama dengan struktur grafena. Jadi, tesis ini telah membentangkan cara sintesis molibdenum dikalkogenida dan karakterisasinya dalam elektrokimia. Objektif bagi tesis ini adalah menghasilkan molibdenum dikalkogenida. Selain itu, molibdenum dikalkogenida yang dihasilkan boleh dikarakterisasi dengan teknik fiziko-kimia dan elektrokimia; dengan penggunaan *X-ray Diffractometer*, *Field Emission Scanning Electron Microscope* dan *potentiostat-galvanostat*. Sintesis molibdenum dikalkogenida telah dilakukan dengan kaedah hidroterma; pelopor yang terlibat dimasukkan dalam Teflon jaket dan mengeramnya dengan masa dan suhu yang sesuai. Pembelauan sinar-X (XRD) digunakan untuk mendedahkan informasi tentang struktur kristal dan komposisi kimia molibdenum dikalkogenida, fungsi pelepasan bidang electron mikroskopi (FESEM) adalah mengesan morfologi molibdenum dikalkogenida, manakala ujian electokimia digunakan untuk menguji sifat elektrokimia molibdenum dikalkogenida. Tiga jenis analisis yang termasuk dalam ujian elektrokimia ialah (i) kitaran voltammetri (CV), (ii) caj discaj (CDC) and (iii) spektroskopi impedansi elektrokimia (EIS). Keputusan yang diperolehi daripada pembelauan sinar-X (XRD) menunjukkan molibdenum trioksida (MoO_3), molibdenum disulfida (MoS_2) dan molibdenum diselenida ($MoSe_2$) telah dihasilkan. Molibdenum dikalkogenida yang dihasilkan mempunyai struktur tersendiri. MoO_3 mempunyai struktur nano-lapisan; MoS_2 mempunyai struktur *nanorod*; $MoSe_2$ mempunyai struktur *nanarod* dan *nanoplate*. Dari ujian elektrokimia, MoS_2 telah meununjukkan prestasi yang lebih baik dalam kapasitan tertentu (117.65 F/g pada 5 mV/s), kemampuan penyimpanan muatan (1.63 Wh/kg pada 0.3 A/g) and kecekapan coulomb (98.93%).

CHAPTER 1

INTRODUCTION

1.1 Background

Increment of the world's primary energy consumptions by 0.9% per year (corresponded to ~121 mts); which recorded from 2013–2014 revealed a high dependency to sources of energy for daily activities, e.g., construction, education, research and development, business, and transportation (British Petroleum, 2015). Malaysia, as a developing country, possesses a great demand in energy from time to time because sosio-economic development and energy are closely related. Crude oil and natural gas are main primary sources for production of electrical energy in Malaysia; contribute 65.5% (~64 406 ktoe) and 29.1% (~28 576 ktoe) for the total energy production respectively (Suruhanjaya Tenaga Malaysia, 2015).

Natural sources such as crude oil and natural gas will use up at a later time and they also release excess carbon dioxide as waste product when burning which causes greenhouse issue. So renewable energies, such as solar energy, biomass and hydropower are encouraged to be alternative sources. However, renewable sources can be affected by weather condition and cannot work continuously at maximum efficiencies; their performances are intermittent (International Electrotechnical Commission, 2011). Thus researchers are focusing on development of advanced energy storage systems to cope with the energy demand; which the common types are battery and capacitor; which have high energy density–low power density, and high power density–low energy density respectively.

Supercapacitor is explored to overcome drawbacks of both devices, with having high energy density and power density. Supercapacitor could be mainly divided into two categories: (i) electrical double layer capacitor (EDLC), and (ii) pseudocapacitor (PC). The differences of EDLC and PC are electrode materials and working principles. The common electrode materials for EDLC are carbon-based materials, e.g. graphene and graphite and they store up energy by charges adsorption at electrode-electrolyte interfaces (Ke and Wang, 2015). Materials targeted for pseudocapacitor are transition metal oxides and conducting polymers such as polyaniline and polythiophene and the working principles involve faradaic redox reaction, electrosorption and intercalation of ions at the electrode (Zahid et al., 2016). In contrast, there is a trend to create a type of capacitor which combines the characteristics of EDLC and PC, called hybrid capacitor. Hybrid capacitor can store charges in both electrostatic and electrochemical forms.

Electrode material is the main source that affects the performance of supercapacitor. Researcher community always triggers to find electrode materials which can contribute to high energy density-power density and long life cycle. Molybdenum dichalcogenides-based materials, e.g. molybdenum dioxide (MoO_2), molybdenum disulfide (MoS_2), molybdenum diselenide ($MoSe_2$) and molybdenum ditelluride ($MoTe_2$) have attracted the attentions of researchers due to its unique characteristic, which is two-dimensional layered structure. These materials normally will use in various devices. MoO_2 possesses good electric conductivity; which could be incorporated lithium ions in its lattice structure during charge and discharge state for reversible storage applications. Various morphologies of MoO_2 have been studied, such as rod-type and spherical type, to enhance the charge storage properties (Song et al., 2009). MoS_2 is used in transistor because of high electron mobility and low level of noise. The bandgap of multi-layered MoS_2 will be increased as number of layers decrease (Rumyantsev et al., 2015). $MoSe_2$ is a good material for transistor and photodetector because of its high electron mobility and direct band gap. Molybdenum ditelluride ($MoTe_2$) has an energy gap which is equivalent to energy of light in infrared region; useful for infrared detector.

1.2 Problem Statement

Electrochemical properties of two-dimensional layered transition metal dichalcogenides (e.g. MoS_2, and $MoSe_2$) have been studied to deliver high energy and power density for supercapacitor applications. The properties are hypothesized could be enhanced by exchanging transition metal or chalcogen atoms with high atomic weight; however such correlation never been materialized till date.

1.3 Objectives

The objectives of the research are:

1. To synthesize molybdenum dichalcogenides (MoX_2) where X are O, S, Se and Te using hydrothermal method.
2. To characterize the synthesized MoX_2 samples using physico-chemical technique.
3. To evaluate the electrochemical properties of the synthesized samples using potentiostat-galvanostat.

1.4 Research Questions

The research questions are:

1. Would the hydrothermally-synthesized molybdenum dichalcogenides present two-dimensional layered morphology?
2. Would the specific capacitance, power density, and energy density increase with increment of molecular weight of molybdenum dicalcogenides?

1.5 Scopes of Research

1. To synthesize molybdenum dichalcogenides, MoX_2 by hydrothermal synthesis
    a. Mixing chalcogens and transition metal precursors in a sealed Teflon jacket
    b. Incubation of precursors at 150 ℃

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