SYNTHESIS AND CHARACTERIZATION OF MXENE-MOS2 HYBRID ELECTRODE FOR SUPERCAPACITOR APPLICATION

This report is submitted in accordance with the requirement of Universiti Teknikal Malaysia Melaka (UTeM) for Bachelor Degree of Manufacturing Engineering (Hons.)

by

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Year 2021

UTeM

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

BORANG PENGESAHAN STATUS LAPORAN PROJEK SARJANA MUDA

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Sesi Pengajian: **2020/2021 Semester 2**

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I hereby, declared this report entitle "Synthesis and Characterization of MXene-MoS2 Hybrid Electrode for Supercapacitor Application" is the result of my own research except as cited in references.

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APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of Universiti Teknikal Malaysia Melaka as a partial fulfillment of the requirements for the Degree of Bachelor of Manufacturing Engineering (Hons.) The member of supervisory committee is as follow:

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ABSTRAK

Bahan 2D telah menarik minat orang ramai dalam bidang sains bahan. Contohnya 2D Molybdenum Disulfide (MoS2) adalah sesuatu bahan yang cerah disebabkan struktur bahan ini memberikan tapak penyerapan dan jugalah laluan difusi yang pendek, tetapi menyusun lembaran MoS2 secara bertambah akan menjejaskan sifat fungsinya serta menurunkan efisiensi. Sesuatu strategi yang dicadangkan adalah menjadikan kedua-dua bahan MoS2 dan MXene sebagai bahan hibrid. 2D MXene boleh menerima banyak kation berlainan antara lapisannya dan jugalah mempunyai kekonduksian elektrik yang bagus. Ciri 2D MXene yang istimewa ini telahpun menarik perhatian dari pelbagai bidang. Dalam kajian ini, bahan dan elektrod yang berhibird MXene/MoS2 telah disintesis dan dibuat pada mulanya dengan menggunakkan teknik pelapisan buburan yang berkonvensional. Diikuti dengan Diffractormetri X-Ray yang menunjukkan kekristalan bahan hibird dan kedua-dua MXene dan MoS2 telahpun berjaya digubah. Selainnya, kajian secara kritikal telahpun dilakukan pada Spektroscopi Raman supaya menberikan hasil jangkaan bahan hibird MXene/MoS2 dalam mempunyai kualiti kristal yang baik dan juga pada Mikroscopi Pengimbasan Elektron yang diharapkan dengan mempunyai luas permukaan yang lebih besar supaya menghasilkan prestasi yang lebih baik. Kemudian, prestasi elektrokimia seperti Ujian Voltametri Siklik dan Caj-Discaj Galvanostatik dalam elektrolit akueus telah dinilaikan. Didapati bahawa kapasiti spesifik yang tertinggi dicapai dalam Ujian Voltametri Siklik dan Caj-Discaj Galvanostatik adalah 15.736Fg^{-1} dan 1.36Fg^{-1} yang dihasilkan daripada komposisi hibird 40:40 MXene/MoS2. Hasil yang terdapat dalam kajian ini telah menunjukkan hibrid 40:40 MXene/MoS2 sebagai sampel yang paling optimum di mana bahan MXene dipercayai sebagai faktor yang berpositif dalam membantu prestasi kapasitif ini dan jugalah menunjukkan potensinya dalam aplikasi penyimpanan tenaga.

ABSTRACT

Two Dimensional (2D) material has attracted much interest in material science field. 2D material such as Molybdenum Disulfide (MoS2) is such a promising material due to its structure provided absorption sites as well as shorter diffusion path, but restacking of MoS2 sheets affect its functional properties which therefore resulting with low efficiency in the device. A strategy has been proposed which is to compose MoS2 with MXene material to form a hybrid material. 2D MXene can host many different cations between its layers and also it has good electrical conductivity. Its special properties have attracted attention from various field. In this study, the MXene/MoS2 hybrid material and electrodes have been synthesized and fabricated at first by applying the conventional slurry coating technique. Followed by the X-ray Diffraction which showed the the hybrid material with crystalline structure and successfully composited between both MXene and MoS2. Furthermore, critical review has been conducted on Raman Spectroscopy which led to an expectation result of MXene/MoS2 hybrid material to have a good crystalline quality as less structural defects and also on Scanning Electron Microscopy which in expectation to have greater surface area that leads to better performance. Later, its electrochemical performances in testing such as Cyclic Voltammetry and Galvanostatic Charge-Discharge in aqueous electrolyte were evaluated. It was found that the highest specific capacitances as achieved in this study were 15.736Fg^{-1} and 1.36Fg^{-1} which attributed to hybrid composition of 40:40 MXene/MoS2 in respecting to cyclic voltammetry and galvanostatic charge-discharge. The results indicated 40:40 MXene/MoS2 hybrid as the most optimal sample at which MXene material is believed as a positive factor in assisting its capacitive performance, thus suggesting its potential in energy storage application.

DEDICATION

To my only

beloved father, Tan Leong Huat

appreciated mother, Ng Soon Lee

adored sisters, Tan Gek Jin and Tan Yee Li

adored brother, Tan Lek Jin

and all my helpful friends

for giving me moral and financial supports, cooperation, encouragement, and also understandings towards of this final year project completion.

Thank You So Much & Love You All Forever

ACKNOWLEDGEMENT

I am appreciated that there are so many people around who shared their supports and provided directly or indirectly guidance throughout the completion of this Final Year Project 1.

First and foremost, I would like to express my greatest gratitude and thank to my respected supervisor, Prof. Ir. Dr. Mohd Asyadi 'Azam Bin Mohd Abid for his given advices, provided guidance, and endless support. With his unending support, helpful advices, and appreciated guidance, it did really help me in motivating myself to contribute throughout this final year project as well as solving the difficulties in any task given.

Furthermore, I would like to give a huge thanks to my family members and friends who gave me much of their cooperation, motivation, as well as mentally and financially supports in completing of this final year project. Without their help, I would be facing hardship in doing this final year project.

Last but not least, I would like to thank everybody who ever assisted me throughout this final year project and also expressing my apology to those have not been mentioned.

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LIST OF ABBREVIATIONS

$(NH_4)_2MoS_4$	-	Ammonium Tetrathiomolybdate
2D	-	Two-Dimensional
AC	-	Activated Carbon
Al	-	Aluminum
ALD	-	Atomic Layer Deposition
CH_3CSNH	-	Thioacetamide
CNT	-	Carbon Nanotubes
$Co(OH)_2$	-	Cobalt (II) Hydroxide
COFs	-	Covalent Organic Frameworks
CP	-	Conductive Polymer
C_{sp}	-	Specific Capacitance
CV	-	Cyclic Voltammetry
CVD	-	Chemical Vapor Deposition
DMSO	-	Dimethyl Sulfoxide
EDLC	-	Electrochemical Double Layer Capacitor
EDX	-	Energy Dispersive X-Ray Spectroscopy
EIS	-	Electrochemical Impedance Spectroscopy
ESR	-	Equivalent Series Resistance

FESEM	-	Field Emission Scanning Electron Microscopy
GCD	-	Galvanostatic Charge-Discharge
GPS	-	Global Positioning System
H_2O	-	Water
H_2S	-	Hydrogen Sulfide
H_2SO_4	-	Sulfuric Acid
HCl	-	Hydrochloric Acid
HF	-	Hydrofluoric Acid
KCl	-	Potassium Chloride
KOH	-	Potassium Hydroxide
LE	-	Liquid Exfoliated
MMX	-	MoS2/Ti_3C_2 Nanohybrids
Mn_3O_4	-	Trimanganese Tetraoxide
Mo	-	Molybdenum
MOFs	-	Metal Organic Frameworks
MoS2	-	Molybdenum Disulfide
MXene	-	Transition Metal Carbides, Nitrides, or Carbonitrides
Na_2MoO_4	-	Sodium Molybdate
Na_2SiO_3	-	Sodium Silicate
Na_2SO_4	-	Sodium Sulfate
$NaNO_3$	-	Sodium Nitrate
NaOH	-	Sodium Hydroxide
NF	-	Nanofiber

Ni_3S_2	-	Trinickle Disulphide
NMP	-	N-methylpyrrolidone
NRs	-	Nanorods
NSs	-	Nanosheets
PAN	-	Polyacrylonitrile
PANI	-	Polyaniline
PVDF	-	Polyvinylidene Fluoride
R_{ct}	-	Charge-Transfer Resistance
R_E	-	Electrolyte Resistance
R_s	-	Intrinsic Resistance
rGO	-	Reduced Graphite Oxide
S	-	Sulphur
SC	-	Supercapacitor
SEM	-	Scanning Electron Microscopy
TMDs	-	Transition Metal Dichalcogenides
wt.%	-	Weight Ratio Percentage
XRD	-	X-Ray Diffraction

LIST OF SYMBOLS

Wh kg^{-1}	-	Watt-hour Per Kilogram
Rpm	-	Revolution Per Minute
ml	-	Milliliter
°C	-	Degree Celsius
h	-	Hour
cm	-	Centimeter
M	-	Molar Mass
ml min^{-1}	-	Milliliter Per Minute
min	-	Minutes
Fg^{-1}	-	Farad Per Gram
ms cm^{-1}	-	Meter-Second Per Centimeter
µm	-	Micrometer
nm	-	Nanometer
mVs^{-1}	-	Milli-Volt Per Second
mA	-	Milli Ampere
Ag^{-1}	-	Ampere Per Gram
sec	-	Second

CHAPTER 1

INTRODUCTION

This chapter will be showing on the research background of Synthesis and Characterization of MXene-MoS2 hybrid electrode for supercapacitor application. Besides, it also includes the problem statement of current environment and energy issue, industry demands, supercapacitor electrode material limitation and their various properties. The objectives, scopes of study, significance of study, and organization of report will be shown and lastly followed by a brief summary.

1.1 Background of Study

Supercapacitors (SCs) is an energy storage device which similar usage as normal capacitors. But unlike normal capacitors which supercapacitors uses charging storage mechanism such as electric double layer capacitance (EDLC), pseudocapacitance, or hybrid mechanism to store and release energy. Supercapacitor is also a device that uses low energy density to produce high power density. While compared to normal batteries, supercapacitors have greater charge-discharge cycle life because supercapacitors used physical ions movement instead of chemical reaction like battery does. Generally, supercapacitors internally constructed with 2 electrodes, 2 current collectors for each positive and negative side, and a separator. Supercapacitors application are usually seen in heavy machineries, elevators, trucks and etc. The device also can be used as voltage support for high energy devices such as fuel cell or batteries.

Molybdenum Disulfide (MoS2) is known as the most typical type of Transition Metal Dichalcogenides (TMDCs). This 2D material has a direct band gap of 1.8eV in its monolayer and which its band structure is dependable on the layers as it can help to overcome the gapless problem of graphene, thus it is important for scientific and industrial area. The generalized formula for TMDCs is MX_2 at which M is the transition metal element (i.e. Ti, Zr, Hf, Mo, and etc.) and X is the chalcogen (i.e. S, Se, or Te). MoS2 is also characterized as a semiconductor and in single film of this material is set up like a sandwich structure in S-Mo-S by covalent bond which interacted and held together by weak Van der Waals force.

For the preparation of MoS2, it is usually by mechanical exfoliation as it can produce better quality of monolayers and it is more favorable in fundamental research, but this method is less appropriate to be use in practical application as it is displayed in low yield and sheet size and the layers are hard to control. Besides, MoS2 can also be prepared by chemical approach which included of ion intercalation and solvent-based exfoliation. Furthermore, Chemical Vapor Deposition (CVD) is a technique which widely used for MoS2 preparation as it can synthesize 2D MoS2 into a wafer-scale that showed high potential to practical application such as large-scaled integrated electronics. In addition, the precursors such as Mo based compound powder, deposited molybdenum based film, and MoS2 powder can also be used to prepare MoS2 film.

MXene is recently a new category of 2D materials. This material is formed by early transition metals (TMs) and carbon/nitrogen (C/N). MXene is a 2D structure which derived from MAX phase and its composition is $M_{n+1}AX_n$ (n=1,2,3), where M is an early transition metal (M= Ti, Sr, Cr, Ta and etc), A represented the elements mainly from group IIIA and IVA, and lastly X is either C or N (carbon or nitrogen) but it could also be both. MAX is the structure which inter-growing with packed A layers and alternatively stacking of MX layers. Using Ti_3AlC_2 (MAX) as an example, by selectively etching of Al on Ti_3AlC_2 in aqueous HF as for us to achieve $Ti_3C_2T_x$ (MXene). The exfoliated carbide or carbonitride nanosheets are then termed as MXene. MXene provides high density, hydrophilic property and also good metallic conductivity. When its 2D layer stacked, more electroactive reaction sites are produced for electrochemical reactions but due to the metal atoms on its surface, oxidation on surface could be easily occurred which is disadvantageous for energy storage application.

2D material such as MoS2 is such promising material because of its short ion diffusion path and absorption site is provided, but restacking of MoS2 sheets showed less efficient in their functional properties. Therefore, composed of MoS2 with another material to form a hybrid could possibly help in solving those problems and acquire the ideal properties. Other than that,