



***DEVELOPMENT OF HIGH PERFORMANCE SUPERCAPACITOR USING
MANGANESE OXIDE, CONDUCTING POLYMERS AND CARBON-BASED
FIBER COMPOSITES***

MUHAMMAD AMIRUL AIZAT BIN MOHD ABDAH

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By

MUHAMMAD AMIRUL AIZAT BIN MOHD ABDAL

**Thesis Submitted to the School of Graduate Studies, Universiti
Putra Malaysia, in Fulfilment of the Requirements for the Degree of
Doctor of Philosophy**

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July 2019

Chair : Associate Professor Yusran Sulaiman, PhD
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An efficient energy storage device, supercapacitors have received great attention in achieving high specific capacitance due to its unique characteristics such as rapid charging/discharging rate, high specific power and good long-term cyclability. The fabrication of hybrid supercapacitors has been exploited to enhance the electrocapacitive performance of the electrode. In the present work, manganese oxide (MnO_2), conducting polymers and carbon based fiber composites were rationally designed and fabricated as symmetrical (polyvinyl alcohol (PVA)-graphene oxide (GO)- MnO_2 /poly (3,4-ethylenedioxythiophene) (PEDOT) (PVA-GO- MnO_2 /PEDOT), carbon nanofibers (CNFs)- MnO_2 /polypyrrole (PPy) (CNFs- MnO_2 /PPy) and functionalised carbon nanofibers (*f*-CNFs)/PPy/ MnO_2) and asymmetrical (*f*-CNFs/PEDOT/ MnO_2 //activated carbon (AC) and porous functionalised carbon nanofibers (P-*f*-CNFs)/PEDOT/ MnO_2 //PCNFs) (ASC) supercapacitors. The morphology and chemical properties of all prepared electrodes were examined by means of field emission scanning electron microscopy (FESEM), Fourier transform infrared (FTIR), Raman spectroscopy and X-ray diffraction (XRD). The MnO_2 valence state (Mn^{4+}) was confirmed by the presence of two distinctive peaks of MnO_2 by X-ray photoelectron microscopy (XPS). Porous functionalised carbon nanofibers (P-*f*-CNFs) was initially optimised using different polyacrylonitrile /polytetrafluoroethylene (PAN/PTFE) blend ratio (1:2, 2:1, 2:3 and 3:2) in order to obtain mesoporous structure. Using PAN/PTFE (2:3), the capacitance of P-*f*-CNFs displayed the highest (176.6 F/g) with a specific surface area of 281 m^2/g . The electrochemical performances of MnO_2 based fiber composites were studied using two-electrode configuration in 1 M KCl electrolyte. Interestingly, the assembled ASC P-*f*-CNFs/PEDOT/ MnO_2 //PCNFs showed an excellent specific capacitance of 719.8 F/g compared to *f*-CNFs/PPy/ MnO_2 (409.88 F/g), *f*-CNFs/PEDOT/ MnO_2 //AC (354 F/g), CNFs-

MnO₂/PPy (315.80 F/g) and PVA-GO-MnO₂/PEDOT (144.66 F/g) at 25 mV/s. A good synergistic effect contributed by each material in P-*f*-CNFs/PEDOT/MnO₂/PCNFs possessed remarkable specific energy of 60.5 Wh/kg and specific power of 555.3 W/kg at 0.6 A/g, indicating excellent electrochemical capacity. In addition, the enhancement of surface wettability and good mechanical strength of P-*f*-CNFs/PEDOT/MnO₂/PCNFs ASC demonstrated better cycle life with 104.6% initial capacitance over 5000 cycles compared to other electrodes. Three assembled ASC devices could successful light up 25 red light emitting diodes (LEDs), implying the capability of this material to be used in the practical supercapacitor application. Therefore, P-*f*-CNFs/PEDOT/MnO₂/PCNFs could be considered as a prospective candidate for high performance supercapacitor due to its remarkable supercapacitive performance.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia
sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

**PEMBANGUNAN SUPERKAPASITOR BERPRESTASI TINGGI
MENGUNAKAN MANGAN OKSIDA, POLIMER KONDUKSI DAN KARBON
BERASASKAN FIBER KOMPOSIT**

Oleh

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Satu alat penyimpanan tenaga baru yang cekap, superkapasitor telah mendapat perhatian yang tinggi dalam mencapai kapasitan spesifik yang tinggi disebabkan oleh ciri-cirinya yang unik seperti kadar caj/discaj yang pantas, kuasa spesifik yang tinggi dan jangka hayat kitaran yang baik. Pembuatan superkapasitor hibrid telah dieksploitasi untuk meningkatkan prestasi elektrokapasitif elektrod. Dalam penyelidikan ini, komposit fiber berasaskan mangan oksida (MnO_2), polimer konduksi dan karbon direka bentuk secara rasional dan dihasilkan sebagai simetri (poli(vinil alkohol)-grafen oksida- MnO_2 -poli(3,4-etilenadioksitiofena) (PVA-GO- MnO_2 /PEDOT), nanofiber karbon- mangan oksida/polipirol (CNFs- MnO_2 /PPy) dan nanofiber karbon berfungsi/polipirol/mangan oksida (f -CNFs/PPy/ MnO_2) dan asimetri nanofiber karbon berfungsi/poli(3,4-etilenadioksitiofena)/mangan oksida//karbon aktifan (f -CNFs/PEDOT/ MnO_2 //AC) dan nanofiber karbon berfungsi yang berliang/poli(3,4-etilenadioksitiofena)/mangan oksida//nanofiber karbon yang berliang (P- f -CNFs/PEDOT/ MnO_2 //PCNFs) superkapasitor asimetri (ASC). Morfologi dan ciri-ciri kimia bagi semua elektrod yang disediakan telah diperiksa dengan menggunakan FESEM, spektroskopi Fourier inframerah (FTIR), spektroskopi Raman dan belauan sinar-X (XRD). Keadaan valens MnO_2 (Mn^{4+}) telah disahkan dengan kehadiran dua puncak tersendiri bagi MnO_2 oleh spektroskopi fotoelektron sinar-X (XPS). Pada awalnya, nanofiber karbon berfungsi yang berporos tinggi (P- f -CNFs) dioptimumkan dengan menggunakan campuran poliakrilonitril/politetrafluoroetilena (PTFE) (PAN/PTFE) yang berbeza (1:2, 2:1, 2:3 and 3:2) untuk mendapatkan struktur liang meso. Dengan menggunakan PAN/PTFE (2:3), P- f -CNFs menunjukkan kapasitan yang paling tinggi (176.6 F/g) dengan luas permukaan spesifik sebanyak $281 \text{ m}^2/\text{g}$. Prestasi elektrokimia bagi komposit fiber berasaskan MnO_2 dikaji menggunakan konfigurasi dua elektrod dalam elektrolit 1 M KCl. Menariknya, ASC P- f -

CNFs/PEDOT/MnO₂/PCNFs menunjukkan kapasitan spesifik tertinggi 719.8 F/g berbanding *f*-CNFs/PPy/MnO₂ (409.88 F/g), *f*-CNFs/PEDOT/MnO₂/AC (354 F/g), CNFs-MnO₂/PPy (315.80 F/g) dan PVA-GO-MnO₂/PEDOT (144.66 F/g) pada kadar imbas 25 mV/s. Kesan sinergistik yang baik yang disumbangkan oleh setiap bahan dalam P-*f*-CNFs/PEDOT/MnO₂/PCNFs mempunyai tenaga spesifik yang luar biasa 60.5 Wh/kg dan kuasa spesifik 555.3 W/kg pada 0.6 A/g, menunjukkan kapasiti electrokimia yang tinggi. Sebagai tambahan, peningkatan kelembapan permukaan dan kekuatan mekanikal yang baik oleh P-*f*-CNFs/PEDOT/MnO₂/PCNFs ASC menunjukkan jangka hayat kitaran yang lebih panjang dengan mengekalkan kapasitan spesifik 104.6% selepas kitaran yang ke 5000 berbanding dengan elektrod yang lain. Tiga peranti ASC terhimpun berjaya menyalakan 25 lampu merah diod pancaran cahaya (LED), menunjukkan kemampuan bahan ini untuk digunakan dalam aplikasi superkapasitor yang praktikal. Oleh itu, P-*f*-CNFs/PEDOT/MnO₂/PCNFs boleh dianggap sebagai calon prospektif untuk superkapasitor yang berprestasi tinggi kerana prestasi superkapasitifnya yang luar biasa.

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I certify that a Thesis Examination Committee has met on 29 July 2019 to conduct the final examination of Muhammad Amirul Aizat Bin Mohd Abdah on his thesis entitled “Development of high performance supercapacitors using manganese oxide, conducting polymer and carbon-based fiber composites” in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the (insert the name of relevant degree).

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LIST OF SYMBOLS

Symbol	Meaning	Unit
C_{sp}	Specific capacitance	F/g
E	Specific energy	Wh/kg
P	Specific power	W/kg
R_{ct}	Charge transfer resistance	Ω
ESR	Equivalent series resistance	Ω
m	Mass of active materials	g
v	Scan rate	mV/s
I	Applied current	A
V	Potential	V
$\Delta V/\Delta E$	Potential window/operating voltage	V
Δt	Discharging time	s
Z'	Real impedance	Ω
Z''	Imaginary impedance	Ω
Cdl	Electrical double layer capacitance	-
CPE	Constant phase element	-
W	Warburg diffusion resistance	-
χ^2	Chi square	-

LIST OF ABBREVIATIONS

AC	Activated carbon
ACNFs	Activated carbon nanofibers
APD	Average pore distribution
ASCs	Asymmetric supercapacitors
BA	Boric acid
BET	Brunauer-Emmett-Teller
CB	Carbon black
CFC	Carbon fiber cloth
CFP	Carbon fiber paper
CFS	Carbon fibers sheet
CNF _{Mn}	Electrosprayed MnO ₂ particles
CNF _{ox}	Oxidised carbon nanofibers
CNFs	Carbon nanofibers
CNT	Carbon nanotube
Co ₃ O ₄	Cobalt oxide
CPs	Conducting polymers
CV	Cyclic voltammetry
CVD	Chemical vapour deposition
DMF	N,N-dimethylformamide
ECs	Electrochemical capacitors
EDLCs	Electrical double layer capacitors
EDOT	3,4-ethylenedioxythiophene

EIS	Electrochemical impedance spectroscopy
ES	Electrospinning
<i>f</i> -CNFs	Functionalised carbon nanofibers
Fe ₃ O ₄	Iron oxide
FeOOH	Porous iron oxide-hydroxide
FESEM	Field emission scanning electron spectroscopy
FSCs	Flexible supercapacitors
FTIR	Field transform infrared spectroscopy
G	Graphene
GCD	Galvanostatic charge-discharge
GNFs	Graphitic nanofibers
GO	Graphene oxide
GQD	Graphene quantum dot
H ₃ BTC	Trimesic acid
HCS	Hybrid supercapacitors
HRTEM	High-resolution transmission electron microscopy
ITO	Indium tin oxide
KMnO ₄	Potassium permanganate
LED	Light-emitting diode
LIBs	Lithium-ion batteries
Mn(NO ₃) ₂	Manganese nitrate
MnCO ₃	Manganese carbonate
MnO ₂	Manganese oxide
MOF	Metal-organic framework

MWs	Metal wires
NFs	Nanofibers
NiO	Nickel oxide
NPs	Nanoparticles
PAN	Polyacrylonitrile
PANi	Polyaniline
PBI	Poly(benzimidazole
PCNFs	Porous carbon nanofibers
PCs	Pseudocapacitors
PEDOT	Poly(3,4-ethylenedioxythiophene)
PEDOT:PSS	Poly(3,4-ethylenedioxythiophene)- poly(styrenesulfonate)
PEO	Polyethylene oxide
PET	Polyethylene terephthalate
PLA	Poly(lactic acid)
PMMA	Polymethacrylate
PPy	Polypyrrole
PTFE	Polytetrafluoroethylene
PVA	Polyvinyl alcohol
PVAc	Poly(vinyl acetate)
PVC	Polyvinylchloride
PVDF	Poly(vinylidene fluoride)
PVP	Poly(vinyl pyrrolidone)
RGO	Reduced graphene oxide
RuO ₂	Ruthenium oxide

S_{BET}	BET specific surface area
SSA	Specific surface area
SWCNTs	Single walled carbon nanotubes
TEM	Transmission electron microscopy
TMOs	Transition metal oxides
V_2O_5	Vanadium oxide
VACNFs	Vertically aligned carbon nanofibers
VGCNFs	Vapour grown carbon nanofibers
VMCNFs	Amorphous V_2O_5 doped with multichannel CNFs
V_t	Total pore volume
XPS	X-ray photoelectron spectroscopy
XRD	X-ray diffraction
ZnO	Zinc oxide

CHAPTER 1

INTRODUCTION

1.1 Background of research

In the past decades, the energy crisis has become a worldwide concern issue due to the rapid growth of human population, along with the accelerated global economy. The surveys on total world energy consumption provided by Energy Information Administration (EIA) show that fossil fuels (petroleum, coal, natural gas, etc.) play a major contribution in fulfilling the demand for sustainable energy. However, the dependence on energy based on fossil fuels could lead to excessive depletion and aggravating environmental pollution. Due to this environmental concern, the need for renewable energies such as solar, wind, hydro, geothermal, biogas and wave can be served as alternative sustainable energy resources owing to their abundance in nature and cost-effective. It has been reported that wind and solar energy could supply about 2.3% and 0.98% of the energy consumption, respectively, leading to the development of clean and environmental-friendly energy sources (Energy, 2016). Although tremendous efforts have been made to fully utilised these recyclable resources, climate change and environmental destruction have affected the requirements for the future energy system. These challenges have evolved the researcher's interest in developing advanced energy storage/conversion technologies with multifunctional applications. In the meantime, fuel cells, batteries, conventional capacitors and supercapacitors have taken center stage as the efficient energy storage devices to replace the existing energy storage sources (Sinprachim *et al.*, 2016). For fuel cells and batteries, despite their fascinating specific energy properties, but yet they may suffer from a drawback of poor specific power. To address this discrepancy, supercapacitors, also called as ultracapacitors have received great attention and emerged as promising energy storage candidates which fill the gap between batteries and traditional capacitors (Shown *et al.*, 2015). Supercapacitors are believed to meet the requirement for a future practical application which holds long cycle lifespan, high specific power and fast charging-discharging rate and relatively low cost. Additionally, supercapacitors have been employed in a wide range of applications include hybrid transportation, portable electronics, memory backup power as well in large industrial scale production (Yang *et al.*, 2016). In general, the enhancement of the overall performance of supercapacitors is dependent on the reasonable design of electro-active materials, therefore, supercapacitors should have a large specific surface area, excellent intrinsic conductivity, good mechanical stability and high specific energy and specific power. To date, the exploration of hybrid supercapacitors has been intensively studied as the demand for

advanced electronic devices increase, which brings a new strategy in developing flexible and stretchable electronic devices in future applications.

1.2 Problem statement

As it is well known, supercapacitors have outperformed other energy storage devices at this current stage of technology due to their superior capacitive features such as fast charge/discharge rate, long cyclic life and high specific power. The ongoing technological advances such as consumer electronics, hybrid vehicles, wireless sensor networks and energy harvesting have attracted extensive concern from the researchers to design and develop high-performance electrode materials for supercapacitors. Therefore, the selection of electrode materials plays an important role in determining the electrochemical performance of the supercapacitor.

The fabrication of one-dimensional (1D) nanofibers (NFs) has drawn considerable attention and shown great potential as an active nanomaterial for supercapacitor. A continuous electrospun NFs derived from biodegradable polymer such as polyvinyl alcohol (PVA) is widely used as a template to enlarge the surface area for charge accumulation. However, PVA nanofibers are electronically non-conductive and suffer from low mechanical strength, which severely limits their applications in supercapacitors. In order to address the drawback, the contribution of electrochemical double layer capacitors (EDLCs) such as graphene oxide (GO), carbon fibers (denoted as CNFs) and activated carbon (AC) are the best solution to improve the long-term stability performance of the fiber composite as they exhibit good electrical conductivity, an excellent mechanical strength with large accessible specific surface area but yet suffering from poor specific capacitance. Therefore, hybrid configuration of carbon materials (CNFs) with pseudocapacitive materials such as transition metal oxides (TMOs) and conducting polymers (CPs) could significantly enhance their supercapacitive properties.

Among various TMOs, manganese oxide (MnO_2) is one of the promising candidates which possesses high theoretical specific capacitance, environmentally friendly and low-cost. Unfortunately, the inferior conductivity and densely packed structure of MnO_2 can restrict its actual specific capacitance at the same time giving poor rate capability. To overcome these constraints, conducting polymers such as poly(3,4 ethylenedioxythiophene) (PEDOT) and polypyrrole (PPy) have been introduced due to their high electrical conductivity and good chemical stability. However, such materials usually suffer from poor long-term stability due to volumetric change and subsequent mechanical degradation during the charging-discharging process. Thus, development of new supercapacitor electrodes consisting of MnO_2 , CPs and carbon based fiber composites are much needed as they are believed to deliver significant

improvement towards their capacitive performances such as specific capacitance, specific energy and cycling stability for supercapacitor applications.

1.3 Objectives

The aim of this research is to prepare and investigate the supercapacitive performances of MnO₂-based fiber composites for supercapacitors. The objectives of this research are:

1. To prepare MnO₂, CP and carbon based fiber composites as symmetrical and asymmetrical electrodes for supercapacitors.
2. To study the effect of surface wettability of MnO₂ based fiber composite towards its specific energy performance via electrochemical functionalisation.
3. To study the effect of porogen at different concentration on the supercapacitive performance of the P-*f*-CNFs/PEDOT/MnO₂ fiber composite.
4. To evaluate the supercapacitive performance of MnO₂, CP and carbon based fiber composites.

1.4 Scope of study

This study focuses on developing high performance supercapacitor using MnO₂, CP and carbon based fiber composites. The first supercapacitor electrode of PVA-GO-MnO₂/PEDOT was fabricated using electrospinning and electrochemical technique. Then, the fibers which derived from PAN-MnO_x precursor was carbonised into CNFs-MnO₂ to enhance the conductivity of the fibers, followed by *in-situ* polymerisation of PPy (CNFs-MnO₂/PPy). The improvement of specific energy has been achieved by modifying the surface wettability of CNFs via electrochemical functionalisation. In addition, the deposition of PPy and MnO₂ using chemical and electrochemical methods were performed on the functionalised carbon fibers (*f*-CNFs) to form *f*-CNFs/PPy/MnO₂. These three fabricated electrodes were assembled in symmetrical form and their electrochemical properties were studied using cyclic voltammetry (CV), galvanostatic charge-discharge (GCD) and electrochemical impedance spectroscopy (EIS) measurements. In order to achieve wide potential windows, the asymmetrical supercapacitors were introduced consisting of *f*-CNFs/PEDOT/MnO₂ (positive electrode) and AC (negative electrode). By using the same assembly, the porogen (PTFE) was included in CNFs to form porous CNFs (PCNFs). The ratios between PAN:PTFE were varied to obtain the maximum specific surface area. Thereafter, P-*f*-CNFs were electrochemically incorporated with PEDOT and MnO₂ to form hybrid P-*f*-CNFs/PEDOT/MnO₂. The ASC device of P-*f*-CNFs/PEDOT/MnO₂/PCNFs was fabricated and its electrochemical performance was further evaluated using the same characterisations.

1.5 Organisation of chapters

This thesis consists of 8 chapters and organised as follows. Chapter 1 is the introduction of the thesis which comprises of research background, problem statement and research objectives. Recent progress on TMO-based fibers, TMO-based fibers, CP-based fibers and TMO/CP-based fibers in term of their materials design and synthesis, cell configuration and electrochemical properties are reviewed in Chapter 2. Chapter 3 discusses the electrochemical performances of the as-prepared PVA-GO-MnO₂/PEDOT. Chapter 4 elaborates the advantages of fabricated CNFs-MnO₂/PPy as symmetrical fiber composite in comparison with pure CNFs, CNFs/MnO₂ and CNFs/PPy. The enhancement of specific energy derived from the *f*-CNFs/PPy/MnO₂ electrodes using a similar approach in Chapter 4 and the benefits for the formation of *f*-CNFs via electrochemical functionalisation are explained in Chapter 5. Chapter 6 discusses the electrochemical behaviors of ASC based on *f*-CNFs/PEDOT/MnO₂ (positive) and AC (negative) using both three- and two-electrode configurations. Chapter 7 elaborates the effect of different PAN:PTFE ratios towards porosity and specific surface area of the as-prepared P-*f*-CNFs. The supercapacitive performances of assembled P-*f*-CNFs/PEDOT/MnO₂//PCNFs ASC were also studied in the same chapter. Finally, Chapter 8 states the conclusion drawn from the works, significant findings and provide the directions for future work.

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