DEVELOPMENT OF MESOPOROUS CARBON NANOCOMPOSITES FILM FROM SYNTHETIC PRECURSOR AND NATURAL CELLULOSE INCORPORATED WITH $Mn2O3$ FOR SUPERCAPACITOR APPLICATION

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ITMA 2020 9

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By

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August 2020

Chairman : Professor Zulkarnain Zainal, PhD
Faculty : Institute of Advanced Technology

A good matching between the electrode material, pore size suitable for diffusion of electrolyte ions and the dimensions of ionic species is necessary for an optimal performance of electrode materials. By developing a cost-effective mesoporous carbon (MPC) electrode material with highly developed surface area and high electric conductivity may address the issues to enhance the capacitive performance of the material as well as power density, energy density and its cycle life. Thus, this study aims at developing MPC film from Resorcinol(R) /Formaldehyde(F)/F127 and modifying the carbon precursor using natural cellulose of carboxymethyl cellulose (CMC) before incorporated with manganese oxide (Mn_2O_3). That is purposedly to enhance the capacitance attributed from electric double layer (EDLC) properties in MPC film as well as the pseudocapacitive properties from faradaic redox reactions of Mn_2O_3. All samples were synthesised by using a spin coating self-assembly soft templating method and incipient wetness impregnation followed by calcination. The experimental conditions such as carbonisation temperature, molar ratio, stirring time, concentrations and calcination temperature and time were manipulated to enhance the capacitive performance of the electrode materials. X-ray diffraction (XRD), Fourier transform infrared (FTIR), Raman spectroscopy, X-ray photoelectron spectroscopy (XPS) and field emission scanning electron microscopy (FESEM) analysis was conducted to confirmed the structure and surface morphology of the samples. The electrochemical measurements e.g. cyclic voltammetry (CV) and galvanostatic charge/discharge (GCD) measurement was performed in 1 M potassium chloride (KCl) electrolyte solution in a three-electrode system assembly.

From the results, it was revealed that the specific capacitance of Mn_2O_3/MPC composites film calcined at 300 °C showed 3.5 times higher with 53.59 mF cm^{-2} than optimised MPC film only 15.23 mF cm^{-2}. These are in good agreement with

the impressive results of a low internal resistance value recorded for Mn_2O_3/MPC, which could lead to the interpretation of higher specific capacitance compared to MPC as supported by the CV and GCD results. Mn_2O_3/MPC composite film displayed the highest energy and power density and shows fairly stable capacitance with sample could retain about 71% of its initial capacitance until reaching 1000 cycles. Then the research geared onwards by applying green raw material which is CMCs as a carbon source for the electrode materials. In this study, two types of CMC; bamboo CMC and agro-based CMC commercial were used before Mn_2O_3 incorporation. The enhancement in specific capacitance with 31.98 mF cm^{-2} for Mn_2O_3/CMCPCbam was observed to be 3.3 times higher compared to pure carbon samples. Mn_2O_3/CMCPCbam and Mn_2O_3/CMCPCcom show a relatively stable capacitance of around 65.6% and 68.5% of the initial capacitance after 1000 charge-discharge cycles and showing highest energy and power density.

The incorporation of pseudocapacitance metal oxides with EDLC carbon films is therefore an effective way to increase electrochemical performance in terms of specific capacitance, power density and energy density characteristics of carbon materials. The presence of Mn_2O_3 was strongly proved by the XRD, XPS and FTIR analysis while FESEM-EDX and HRTEM confirmed their existence in the structure. This strategy highlights well-organised mesoporous carbon films from synthetic and natural cellulose precursor with superior electrochemical performance as the promising materials for advanced supercapacitor applications.

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

PEMBANGUNAN FILEM NANOKOMPOSIT KARBON BERLIANG MESO DARIPADA BAHAN SINTETIK DAN SELULOSA SEMULAJADI DIGABUNGKAN DENGAN Mn_2O_3 UNTUK APLIKASI SUPERKAPASITOR

Oleh

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Padanan di antara bahan elektrod, kesesuaian saiz liang untuk penyebaran ion elektrolit dan dimensi spesies ionik adalah diperlukan untuk prestasi optimum bahan elektrod. Pembangunan bahan elektrod karbon berliang meso (MPC) berkos efektif dengan luas permukaan dan kekonduksian elektrik yang tinggi diharap dapat meningkatkan prestasi kapasitans bahan serta ketumpatan kuasa, tenaga dan kitaran hayatnya. Oleh itu, kajian ini tertumpu kepada penghasilan MPC daripada Resorcinol (R)/Formaldehid (F)/F127 dan pengubahsuaian prekursor karbon menggunakan selulosa semulajadi karboksilmetil selulosa (CMC) sebelum digabungkan dengan mangan oksida (Mn_2O_3). Ia bertujuan untuk meningkatkan kapasitans yang disebabkan oleh sifat elektrik dua lapisan (EDLC) dalam filem MPC dan juga sifat pseudokapasitor dari tindak balas redoks faradaik Mn_2O_3. Kesemua sampel disintesis dengan menggunakan kaedah templat enapan berputar pembentukan sendiri dan kaedah serapan basah diikuti dengan proses pengkalsinan. Keadaan eksperimen seperti suhu karbonisasi, nisbah molar, masa pengadukan, kepekatan, suhu dan masa pengkalsinan diubahsuai untuk meningkatkan prestasi kapasitans bahan elektrod. Analisis pembelauan sinar-X (XRD), transformasi inframerah Fourier (FTIR), spektroskopi Raman, spektroskopi fotoelektron sinar-X (XPS) dan mikroskopi pengimbasan elektron pancaran medan (FESEM) dilakukan untuk menunjukkan struktur dan morfologi permukaan sampel. Pengukuran elektrokimia seperti voltammetri berkitar (CV) dan galvanostat cas nyahcas (GCD) dilakukan dalam larutan elektrolit 1 M kalium klorida (KCl) menggunakan sistem sel tiga elektrod.

Berdasarkan keputusan, nilai kapasitans spesifik filem nanokomposit Mn_2O_3/MPC yang dikalsinasi pada suhu 300 °C adalah didapati 3.5 kali ganda lebih tinggi dengan 53.59 mF cm^{-2} berbanding filem MPC optimum dengan hanya 15.23 $mFcm^{-2}$. Ini bertepatan dengan nilai rintangan dalaman yang

rendah yang direkodkan untuk Mn_2O_3/MPC sebagai gambaran kepada kapasitans spesifik yang lebih tinggi berbanding MPC disokong oleh keputusan CV dan GCD. Filem komposit Mn_2O_3/MPC menunjukkan ketumpatan tenaga dan kuasa tertinggi serta kapasitans yang cukup stabil dengan sampel dapat mengekalkan sekitar 71% dari kapasitans awal sehingga mencapai 1000 kitaran. Kemudian, penyelidikan diteruskan dengan mengaplikasikan bahan mentah hijau iaitu CMC sebagai satu sumber karbon untuk bahan elektrod. Dalam kajian ini, CMC buluh dan CMC komersial berasaskan bahan pertanian telah digunakan sebelum penggabungan dengan Mn_2O_3. Peningkatan kapasitans spesifik sebanyak 31.98 mF cm^{-2} untuk Mn_2O_3/CMCPCbam adalah didapati 3.3 kali ganda lebih tinggi berbanding dengan sampel karbon tulen. Mn_2O_3/CMCPCbam dan Mn_2O_3/CMCPCcom menunjukkan kapasitans yang agak stabil iaitu 65.6% dan 68.5% dari kapasitans awal selepas 1000 kitaran cas nyahcas serta menunjukkan ketumpatan tenaga dan kuasa yang tertinggi.

Penggabungan oksida logam pseudokapasitans dengan filem karbon EDLC adalah cara yang berkesan untuk meningkatkan prestasi elektrokimia dari segi kapasitans spesifik, ketumpatan kuasa dan tenaga bahan karbon. Kehadiran Mn_2O_3 telah dibuktikan dengan nyata oleh analisis XRD, XPS dan FTIR sementara analisis FESEM-EDX dan HRTEM mengesahkan kewujudannya dalam struktur bahan tersebut. Strategi ini mengenengahkan filem karbon tersusun berliang meso daripada bahan prekursor sintetik dan selulosa semulajadi sebagai bahan berpotensi yang berprestasi elektrokimia lebih baik untuk tujuan aplikasi superkapasitor termaju.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

MPC	Mesoporous Carbon
R	Resorcinol
F	Formaldehyde
CMC	Carboxymetyl Cellulose
PC	Porous Carbon
FESEM	Field Emission Scanning Electron Microscopy
EDX	Energy Dispersive X-Ray Spectroscopy
XRD	X-ray Diffraction
XPS	X-Ray Photoelectron Microscopy
HRTEM	High Resolution-Transmission Electron Microscopy
SAED	Selected Area Electron Diffraction
Ti	Titanium
AFM	Atomic Force Microscopy
EES	Electrochemical Energy Storage
EDLC	Electrical Double Layer Capacitor
AC	Activated carbon
CA	Carbon Aerogel
CNT	Carbon Nanotube
CMCPC	Carboxymetyl Cellulose Porous Carbon
CV	Cyclic Voltammetry
GCD	Galvanostatic Charge Discharge
EIS	Electrochemical Impedance Spectroscopy
CMCPCbam	Carboxymetyl Cellulose Porous Carbon from bamboo CMC

CMCPCcom	Carboxymetyl Cellulose Porous Carbon from commercial CMC
TGA	Thermogravimetric Analysis
DTG	Differential Thermogravimetry
SDA	Surface Directing Agent
SS	Stainless steel
FTO/ITO	fluorine/indium doped tin oxides
EISA	Evaporation-Induced Self-Assembly
CHNS	Carbon, hydrogen, nitrogen, sulfur
BET	Brunauer-Emmet- Teller
BJH	Barret-Joyner-Halenda
FTIR	Fourier Transform Infrared Spectroscopy
R_s	Cell-Electrolyte Resistance
R_{ct}	Charge Transfer Resistance
ECs	Electrochemical Capacitors
SCs	Supercapacitors
OMC	Ordered Mesoporous Carbon
ESR	Equivalent Series Resistance
ATR	Attenuated Total Reflectance
CAGR	Compound Annual Growth Rate
DI	Deionised water
Mn	Manganese
FWHM	Full width at half-maximum
WITec	Wissenschaftliche Instrumente und Technologie’
D	Crystallite size
λ	Wavelength of Cu Kα radiation (1.54 Å)

β	FWHM of the peak intensity in radian
θ	Bragg's angle corresponding to the peak being analysed
ΔQ	Total amount of charge accumulated over a potential window ΔV
w	Area of active material in an electrode
I	Respondent current
$(\Delta V \mid \Delta t)$	Scan rate of the cyclic voltammetry.
I	Discharge current in amperes
Δt	Discharge time in seconds
A	Area of the active electrode material in cm^2
ΔE	Voltage differences in volts
E	Density of energy in Wh/kg
SC	Specific capacitance of an electrode
V	Potential window
P	Density of power in W/kg
C_{dl}	Doubled- layered capacitance
E	Energy density of the supercapacitor
P	Power density of the supercapacitor
η	Coulombic efficiency
RF	Resorcinol Formaldehyde
G	Band that can be attributed to the in-plane carbon stretching vibrations of ideal graphene sheets
D	Band that can be attributed to structural imperfection of graphene sheets.
IUPAC	International Union of Pure and Applied Chemistry
PAHs	Polycyclic aromatic hydrocarbons
H_2	Hydrogen

PSD	Pore Size Distribution
NC	Nanocellulose
Mn-Co	Manganese-Cobalt oxide
CP	Conducting Polymer
TMO	Transition Metal Oxide
WE	Working electrode
CE	Counter electrode
RE	Reference electrode

CHAPTER 1

INTRODUCTION

1.1 General Introduction

Energy demand is continuously increasing due to the increase in world energy consumption and rapid economic development (Mekhilef et al., 2011). It was predicted that by 2030, Malaysia's global demand for energy will rise at a rate of 1.6 percent which is around 65 percent of the increase will be due to developing countries (Petinrin and Shaaban 2015). The critical issue throughout the world concerning the limited fossil fuel as the main source of energy and other challenges such as global warming and detrimental environmental effects, encourage the policymakers and authorities to focus on renewable energy resources such as plant materials (biomass), wind, solar, waves, small hydro, ocean currents, ocean thermal e.g. temperature differences in the oceans and the energy of the tides (Samsudin et al., 2016). Biomass has been reported to be the world's fourth largest potential energy resource and given that Malaysia is blessed with the tropical and humid climate, we can fully utilise the huge potential of agriculture and tropical forests for the biomass resources. Looking backwards to 1990s till now, the idea of waste-to-wealth has been advocated and well-received where it has driven the transformation from unused waste to value added fuel with the rise in economic efficiency (Mekhilef et al., 2011). Recently, researchers have given significant attention on developing efficient energy generation and storage from renewable resources (Azam et al., 2014) based on advanced design and nanostructured carbon materials synthesised from natural plant and biomass-based due to their low cost, accessibility, environmentally friendliness, and recyclability. Furthermore, their natural hierarchical porous structure facilitates electrolyte penetration and provides generation of extra active sites (Gao et al., 2018).

In a myriad of applications, electrochemical energy storage (EES) devices are important for telecommunications devices such as cell phones, remote communication, walkie-talkies, etc., standby power systems, and hybrid electric vehicles in the form of storage components. This has led to the need for advanced power sources with high power density (Gao et al., 2018; Azam et al., 2014; Conway 1999). Supercapacitors (SCs) also are known by other names such as electrochemical capacitors (ECs) or ultracapacitors or electrochemical double-layer capacitors (EDLCs) or pseudocapacitors; all of which have great potential to produce more power than batteries and store more energy than conventional capacitors with fast charging-discharging time. SCs store energy by means of EDLCs capacitance (ion adsorption) and pseudocapacitane (surface redox reaction). With potential uses in transport, consumer electronics, and energy industries, the SC market's future is promising.

Ever since the launching of SCs to the market, they have drawn worldwide attention. The global market for SCs is projected to hit an estimated $3.1 billion by 2026 and is forecast to grow from 2017 to 2026 at a Compound Annual Growth Rate (CAGR) of 15.5 % (Figure 1.1). SCs provide avenues for researchers to discover all potential materials that can offer improved strength and energy density efficiency, charge-discharge characteristics, power stabilisation, long cycle stability and reversibility (Huang et al., 2019).

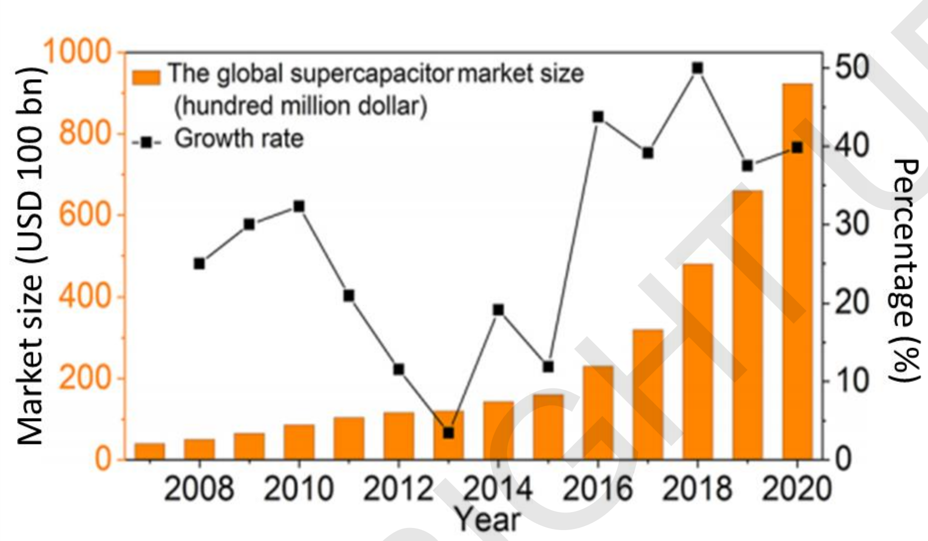

Figure 1.1: The market size and growth rate of global supercapacitor (Huang et al. 2019)

Commercially available SCs mainly use carbon as their electroactive material namely, activated carbon (AC), nanostructured carbon, e.g. carbon nanotubes (CNT), high porosity activated fibers and graphite, nanosised transition metal oxides, conductive polymers, etc. in order to provide good electrical conductivity with the high specific surface area. The electrical capacitance of SCs depends heavily on the number of ions (anions or cations) on the electrode/electrolyte interface, thus the high surface area is essential for SCs to acquire high number of ions to be adsorbed on the electrode surface.

Noteworthy mentioning, it is only the pores that are reachable by the electrolyte ions which will contribute to the capacitance. Porous carbon or AC is commonly recognised for its large surface area and therefore it has high adsorption and high energy storage capabilities. However, due to the narrow pore 'gate', the ion diffusion at the entrance of the pore is limited, thus resulting in the pore not reachable by electrolyte ions. On the other hand, mesoporous carbon with larger pore size contributes towards the efficient electrolyte ions diffusion through the material, which improves the ions mobility thus enhancing the capacitance (Huang et al., 2015). In short, the electrode surface, pore size and ionic species characteristics must perfectly match for the optimum performance of electrode materials.

1.2 Problem Statements

Fossil fuels depletion and environmental pollution in recent years, have drawn the demand and requirement for renewable, cheap and clean energy sources. This includes the development of efficient energy storage or energy conversion devices. Furthermore, such devices are required to satisfy both high-energy and high-power density demands in which usually either one needs to be sacrificed. In energy storage technology, the disadvantage of capacitors is their inability to store big quantities of energy while batteries are incapable of quickly charge/discharge cycles owing to longer times for ion diffusion. This performance contrast is a significant barrier in the electrochemical energy storage, thus SCs were suggested to narrow the gap between capacitors and batteries. Other main features of SCs include ultra-fast charging and discharging as well as incredibly heavy cycling. By developing a porous electrode material with highly developed surface area and high electric conductivity may address this issue to enhance the capacitive performance of the material as well as power density, energy density and its cycle life. There is also a need to develop materials processing methods which are cost-effective as to promote large scale production (Xing et al., 2015).

Ordered mesoporous carbon (OMC) with integrated porous structure materials have been fabricated recently and has gained considerable attention over conventional microporous carbon materials due its unique, chemical and mechanical stability, electronic characteristics and thermal conductivity, thus leading to good mass transport and electrical properties (Mitome et al., 2014; Matsui et al., 2013; Cai et al., 2013; Feng et al., 2011; Chang et al., 2007). In general, OMC can be produced by either hard or soft templating methods. Hard templating method is less desirable due to the high production cost and complex synthesis route, it also involves tedious and environmentally hazardous preparatory process that hinders the large-scale production.

The advancement of carbon materials with high capacitance and minimum equivalent series resistant (ESR) should also be addressed. This calls for the demand for binder-free thin film for electronic devices or free-standing mesoporous carbon thin film. Addressing such issues may resolve problems of interparticle resistance and contact resistance between the carbon electrode and the current collector which eventually contribute towards improvement in many applications such as chemical sensors, separation membranes, membranes of reactors and catalytic membrane electrodes (Zhang et al., 2012).

Concurrently, there are several advantages and disadvantages in the active materials used for SC electrode, for instance, carbon materials suffer from small capacitance even though they possess excellent electrical and mechanical properties with long cycle life. Meanwhile, the cheap and flexible conducting polymer have poor cyclability, and on the other hand the high specific capacitance of metal oxide materials is very costly. Thus, a pseudocapacitor electrode where the energy is stored in the active materials via accelerated reversible faradaic shifts and transitions has been introduced to overcome the issues. As such, this high-quality supercapacitor electrodes fabricated from transition metal oxides are therefore being promising candidates that can enhance the specific capacitance and power density by virtue of pseudocapacitive mechanism (Li et al., 2015; Wang et al., 2011). Due to their abundance, low cost, wide electrochemical windows, rich redox chemistry and high specific theoretical capacitance, manganese oxide (MnO_x) is claimed to be a new class of promising material for supercapacitors (1100–1300 F g^{-1}) (Huang et al., 2015; Prasad et al., 2013; Song et al., 2012; Zhang et al., 2011). Thus, extensive research was carried out for binary and ternary composites to combine and balance the advantages. One of the effective strategies was the utilisation of carbon-inorganic composite of metal oxides and carbon materials (Huang et al., 2015).

The low-cost abundant and renewable biomass of bamboo has indeed been assigned as a precursor of green materials. Based on Kim et al. (2006), owing to good thermal, electrical conductivity and high surface area, good anti-causticity, high stability, fast growing species, low cost and commercially available, bamboo was claimed to be the most suitable candidate in the production of the electrode for supercapacitors. Hence, our local Malaysian bamboo was selected in the study as the starting material because the palm kernel shell and coconut shell become a commodity of shortages. To fix the issue of bamboo residues generated by non-wood industries as by-products e.g. chopstick industries each year, could be processed into value-added products of mesoporous carbon (MPC) from bamboo that convert waste into wealth. Normally, the residue is dumped to self-decompose or open burning. However, this will give rise to greenhouse gases, e.g. contribution of CH_4 and CO_2 to global warming.

To address key issues as highlighted above, there is a need to develop a low cost of mesoporous carbon from biomass sources. To further enhance the electrochemical properties, selection of suitable transition metal oxide is very important to be hybridised with the carbonaceous material as binder-less electrode and ready to be use. The extensive assessments on the relationship between the structure and electrochemical based on the capacitive performance have been performed and discussed. This research should be seen as an innovative work that provides innovative electrochemical solutions for potential energy and power storage devices from MPC materials and the fabricated composites.

1.3 Background of research

The development of high-performance electrode materials has been a topic of major research interest. A good electrode material requires it to be simple and cost-effective, possessing high specific surface area and useful pores, environmentally friendly and good conductivity (Chou et al., 2014; Mitome et al., 2014). In this study, three major strategies were adopted to enhance the electrochemical performance in supercapacitor electrode. First, through the preparation of MPC film using resorcinol/formaldehyde as the carbon source and Pluronic F127 as the structure directing agent. This work is based to the previous comprehensive studies on the electrochemical performance of porous carbons made from biological sugarcane bagasse templates (Chou et al., 2014; Huang and Doong 2012), mesoporous polymer (Wang et al., 2016; Cai et al., 2013; Matsui et al., 2013) and carbon films using mesoporous polymers (Mitome et al., 2016; Xie et al., 2016; Mitome et al., 2014; Feng et al., 2011). The second part is the incorporation of metal oxides, Mn_2O_3 to the porous carbon electrode to form (Mn_2O_3/MPC) nanocomposite film using a simple and facile method, namely, spin coating self-assembly soft templating technique and incipient wetness impregnation which demonstrates excellent capacitance of 3.5 times higher than MPC film. In the last few years, composite electrode materials from manganese oxides-carbon was fabricated such as MnO_2/carbon aerogel (Li et al., 2010), Mn_2O_3 /mesoporous carbon (Wang et al., 2013; Jiang et al., 2011), MnO_2/carbon nanotube, RuO_2/carbon nanotube and NiO/carbon nanotube (Fisher et al., 2013). Due to their charge storage mechanisms with double layer capacitance, the composites demonstrate an improved capacitive behavior (Wang et al., 2011; Zhang and Zhao 2009) and have been produced in recent years.

The last part of the study was carried out by the substitution of synthetic carbon material with plant-based material, namely, carboxymethyl cellulose (CMCs) incorporated with Mn_2O_3 as another potential electrode material. Research and developments on the use of CMC as one of the composite materials in the SC application has been reported by Karaca et al. (2018), Yu et al. (2017) and Xu and Zhang (2015). The target is to develop a greener and eco-friendly product; manganese oxide incorporated CMC porous carbon (CMCPC/Mn_2O_3) composites film by combining both EDLCs and pseudocapacitors (Yang et al., 2015). The research flow and overview of the study is presented in Figure 1.2. CMC has drawn considerable attention owing to its renewability, biocompatibility and environmentally friendliness, (non-toxic and biodegradable) (Akhtar et al., 2018; Esteghlal et al., 2018; Achachlouei and Zahedi 2018). Its properties also can be strengthened due to the presence of carboxyl groups that can be attached to metal ions such as Ca^{2+}, Mg^{2+}, Al^{3+}, Mn^{3+} and Fe^{3+} (Yu et al., 2017).

A comprehensive work has been done towards the electrochemical energy storage potential that utilises plant-based biomass as ‘natural and green chemistry' from a cross-disciplinary perspective that embraces material science for SCs energy storage.

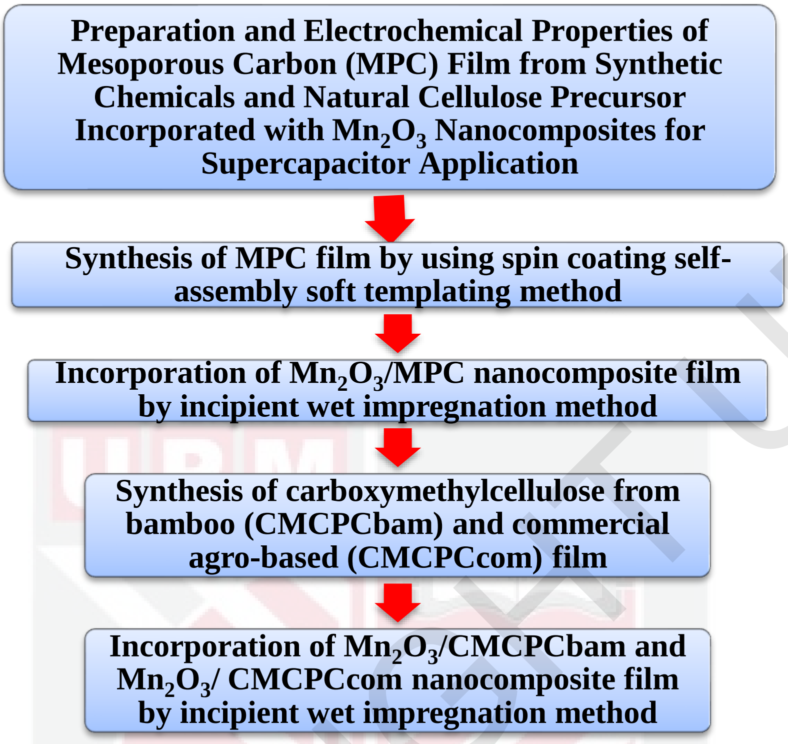

Figure 1.2: Research flow and overview of the study

1.4 Scope of study

The previous study shows that MPCs are suitable to be used as electroactive materials for SC electrode. This research was therefore carried out in order to discover the best fitting between the material of the electrode for optimum electrode material performance and efficiency. The scope of this study is to prepare and characterise the MPC film from resorcinol (R) and formaldehyde (F) as the carbon source and Pluronic F127 as the structure directing agent. The MPC film was prepared by using spin coating self-assembly soft templating method on the Titanium (Ti) substrate. The different molar ratios of F127/R, water/ethanol, carbonisation temperature and stirring time are expected to produce MPC with different electrochemical properties.

Another scope of this study is to incorporate Mn_2O_3 into MPC nanocomposite film by incipient wet impregnation method. The composites are expected to demonstrate an improved capacitive behaviour due to their dual charge storage mechanisms with double layer capacitance enhance the performance of capacitance.

Meanwhile, the potential advantages of using green and natural carboxymethyl cellulose (CMC) as a linker to link Mn_2O_3 particles in order to enhance its properties due to the existence of the carboxyl group. Thus, the synthesis of carboxymethylcellulose from bamboo (CMCPCbam) and commercial agro-based (CMCPCcom) film incorporated with metal oxide, Mn_2O_3/CMCPCbam and Mn_2O_3/ CMCPCcom nanocomposite film was prepared by incipient wet impregnation method and was compared with the results of MPC prepared from chemical synthetic materials for the supercapacitive performance as electrode SC.

1.5 Objectives

The main objective of the project is to develop MPC film from synthetic precursor and natural bamboo-derived cellulose as carbon source-Mn_2O_3 composites for SC electrodes using spin coating self-assembly soft templating and incipient wetness impregnation technique. Below are the specific objectives to be addressed:

1. To synthesise MPC film from synthetic precursor as carbon source incorporated with Mn_2O_3 (Mn_2O_3/MPC) composite film
2. To synthesise the MPC film from natural carboxymethylcellulose precursor i.e. from bamboo (CMCPCbam) and commercial agro-based (CMCPCcom) material.
3. To prepare and characterise CMCPC incorporated Mn_2O_3 (Mn_2O_3/CMCPCbam and Mn_2O_3/CMCPCcom) composites film
4. To optimise study parameter of the MPC, CMCPC and Mn_2O_3 deposition in the composite samples
5. To evaluate the electrochemical performance of all samples produced as electrode material in the SC.

1.6 Structure of thesis

This thesis, which explores the improvement of mesoporous carbon performance, is divided into 6 chapters. Chapter 1 is intended to provide the reader with a general introduction on Malaysia’s overview of energy and technologies also the market perspective of SC industry. It also serves the background of research on SC electrodes and the important factors which govern the performance, green production, problem statements, scope of study and objectives. In addition, this section highlights the importance of this work.

Chapter 2 provides a literature review on the development of mesoporous carbon materials from both the synthetic and natural cellulosed-based materials incorporated with metal oxides for SC applications. It also provides an additional ongoing literature review on the structure and techniques used to prepare the materials, as well as, their focus for use in SCs. This literature review is important for the reader to follow and understand the content discussed in later sections.

Chapter 3 discusses the research methodology which describes the experimental work and procedures completed during this project. This is attempted to produce high capacitance mesoporous carbon film and particularly, the source of the materials and chemicals used. The characterisation and analysis techniques also discussed in this chapter.

To facilitate understanding, the results and discussion were divided into Chapter 4 and Chapter 5, respectively. Chapter 4 exploring on the synthesis of MPC from synthetic precursor followed by the synthesis of Mn_2O_3/MPC nanocomposite film whereby Chapter 5 on the synthesis of CMC porous carbon from lignecellulose material before hybridising with Mn_2O_3/CMCPCbam and Mn_2O_3/CMCPCcom nanocomposite film. Each chapter introduces and discusses the results concerning the parameters that have been studied. The conditions of the experiment, e.g. carbonisation temperature, molar ratio, stirring time, concentrations and calcination temperature and time were varied in the study in order to enhance the capacitive behaviour and electrochemical performance of the electrode materials for supercapacitor. These include characterisation of the morphology, structure and composite.

Chapter 6 provides a summary and conclusion of the results of the projects and a brief discussion on the direction of future work regarding the potential application.

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