

TERNARY MAGNETIC BIOCHAR COMPOSITE FROM *CITRULLUS LANATUS*
RIND FOR SUPERCAPACITOR'S ELECTRODE

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DEDICATION

This thesis is dedicated to my Mama and Abah, who always believed in my potential and taught me that everything happens for a reason; thus, keep faith in Allah, He is the best planner. It is also dedicated to my husband and kids, who always stand by my side in every thick and thin. And not to forget to my late mother-in-law, who patiently waiting for me to complete my Doctorate Degree, but Allah loves her more.

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ABSTRACT

Metal oxides and conducting polymers are renowned as some of the promising materials for electrochemical energy storage (EES) devices, which include batteries, supercapacitors, and hybrid EES devices. This is due to their unique redox properties, significant theoretical capacitance, and environmentally benign nature. However, unsupported metal oxides and conducting polymer nanostructures suffer from particle aggregation, which decreases their electrochemical surface area. In recent years, the preparation of EES from renewable biomass has been developed taking into consideration the economic and environmental feasibility. Biochar is one of the major products of the thermochemical conversion of biomass. Applications of biochar for agricultural and environmental areas have been studied and reviewed extensively but biochar for energy storage materials has not been widely explored and examined. Therefore, the aim of this study is to convert the watermelon rind (WR) into magnetic biochar through a single-route self-purging pyrolysis method. Binary metal oxides (BMOs), such as nickel ferrite (NiFe_2O_4) and cobalt ferrite (CoFe_2O_4), were impregnated in dried watermelon rind to incorporate metal ions into the magnetic biochar. Response surface methodology (RSM) was employed to determine the magnetic watermelon rind biochar (MWRB) synthesis conditions (pyrolysis temperature, pyrolysis time, and WR: BMO ratio). The optimised magnetic biochar was combined with polyaniline (PANI) to produce a ternary magnetic biochar composite with PANI (TC-MWRB/PANI) via in-situ polymerisation to further enhance its electrochemical performance. RSM was also implemented to determine the TC-MWRB/PANI synthesis conditions (PANI concentration, sonication time, and sonication amplitude). Characterisations were done through field emission scanning electron microscopy (FESEM), energy dispersive X-ray spectroscopy (EDX), X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy, Brunauer-Emmett-Teller (BET) surface area, and vibrating sample magnetometer (VSM). Electrochemical evaluations were performed through cyclic voltammetry (CV), galvanostatic charge-discharge (GCD), and electrochemical impedance spectroscopy (EIS). The XRD and FTIR results confirmed the successful formation of MWRB and TC-MWRB/PANI. The FESEM images revealed the porous structure of MWRB and fibrous-look PANI embedded on the surface of TC-MWRB/PANI, while the EDX results showed their associated elemental composition. The electrochemical investigations revealed excellent electrochemical performance of the MWRB and TC-MWRB/PANI for energy storage applications. Based on the electrode specific capacity, the regressed model and experimental results for the fabricated $\text{MWRB}_{\text{NiFe}_2\text{O}_4}$ and $\text{MWRB}_{\text{CoFe}_2\text{O}_4}$ were determined to be 191 C g^{-1} and 187 C g^{-1} and 200.05 C g^{-1} and 200.96 C g^{-1} , at 5 mV s^{-1} , respectively. In addition, the electrode specific capacity based on the regressed model and experimental results for the fabricated TC-MWRB $_{\text{CoFe}_2\text{O}_4}$ /PANI were determined to be 488.22 C g^{-1} and 491.29 C g^{-1} . A two-electrode configuration with TC-MWRB $_{\text{CoFe}_2\text{O}_4}$ /PANI as a positive electrode and watermelon rind biochar (WRB) as a negative electrode was fabricated to form a hybrid device (supercapattery) that operated in a stable potential window of 1.5 V. The energy density and power density of the device measured at a current density of 4 A g^{-1} were estimated to be 22.45 Wh kg^{-1} and 833.19 W kg^{-1} , respectively. The fabricated supercapattery showed excellent cyclability with 97.46% specific capacity retention after 5,000 cycles.

ABSTRAK

Oksida logam dan polimer pengalir terkenal sebagai beberapa bahan berpotensi tinggi bagi peranti storan tenaga elektrokimia (EES), termasuk bateri, superkapasitor dan peranti EES hibrid, disebabkan oleh sifat redoks yang unik, kapasiti teori yang ketara, serta tidak berbahaya kepada alam sekitar. Walau bagaimanapun, oksida logam yang tidak disokong dan struktur nano polimer konduktif mengalami pengagregatan zarah, yang mengurangkan luas permukaan elektrokimia bahan. Dalam beberapa tahun kebelakangan ini, penyediaan EES daripada biojisim boleh diperbaharui telah dibangunkan dengan mengambil kira kebolehlaksanaan ekonomi dan alam sekitar. Biochar merupakan salah satu produk utama penukaran termokimia biojisim. Penggunaan biochar dalam bidang pertanian dan alam sekitar telah dikaji dan disemak secara meluas. Walau bagaimanapun, biochar bagi bahan storan tenaga masih belum diterokai dan disemak secara meluas. Dalam kajian ini, kulit tembikai (WR) telah ditukar kepada biochar magnetik melalui kaedah pirolisis pembersihan-sendiri laluan-tunggal. Oksida logam binari (BMO), seperti ferit nikel (NiFe_2O_4) dan ferit kobalt (CoFe_2O_4), telah diimpregnat dalam kulit tembikai kering untuk memasukkan ion logam ke dalam biochar magnetik. Metodologi permukaan tindak balas (RSM) telah digunakan untuk menentukan keadaan sintesis biochar kulit tembikai magnetik (MWRB) (suhu pirolisis, masa pirolisis dan nisbah WR:BMO). Biochar bermagnet yang dioptimumkan telah digabungkan dengan polyaniline (PANI) bagi menghasilkan komposit biochar bermagnet terner dengan PANI (TC-MWRB/PANI) melalui pempolimeran *in-situ* untuk meningkatkan lagi prestasi elektrokimianya. RSM juga digunakan untuk menentukan keadaan sintesis TC-MWRB/PANI (kepekatan PANI, masa sonikasi, dan amplitud sonikasi). Pencirian dilakukan melalui mikroskop elektron pengimbasan pelepasan medan (FESEM), spektroskopi sinar-X penyebaran tenaga (EDX), pembelauan sinar-X (XRD), spektroskopi inframerah transformasi Fourier (FTIR), luas permukaan Brunauer-Emmett-Teller (BET), dan magnetometer sampel bergetar (VSM). Selain itu, penilaian elektrokimia dilakukan melalui voltametri kitaran (CV), nyahcas cas-galvanostatik (GCD), dan spektroskopi impedans elektrokimia (EIS). Keputusan XRD dan FTIR mengesahkan pembentukan MWRB dan TC-MWRB/PANI. Imej FESEM mendedahkan struktur berliang MWRB dan rupa PANI bergentian yang tertanam pada permukaan TC-MWRB/PANI, manakala keputusan EDX menunjukkan komposisi unsur yang berkaitan. Penyiasatan elektrokimia mendapati prestasi elektrokimia yang sangat baik dalam MWRB dan TC-MWRB/PANI bagi aplikasi storan tenaga. Berdasarkan kapasiti khusus elektrod, model regresi dan keputusan eksperimen untuk fabrikasi MWRB NiFe_2O_4 dan MWRB CoFe_2O_4 , masing-masing telah ditentukan pada 5 mV s^{-1} sebagai 191 C g^{-1} dan 187 C g^{-1} , dan 200.05 C g^{-1} dan 200.96 C g^{-1} . Di samping itu, kapasiti khusus elektrod berdasarkan model regresi dan keputusan eksperimen untuk TC-MWRB CoFe_2O_4 /PANI yang direka dan didapati sebagai 488.22 C g^{-1} and 491.29 C g^{-1} . Konfigurasi dua elektrod dengan TC-MWRB CoFe_2O_4 /PANI sebagai elektrod positif dan biochar kulit tembikai (WRB) sebagai elektrod negatif telah direka untuk membentuk peranti hibrid (*supercapattery*) yang beroperasi dalam tettingkap berpotensi stabil 1.5 V. Ketumpatan tenaga dan ketumpatan kuasa peranti yang diukur pada ketumpatan arus 4 A g^{-1} masing-masing dianggarkan 22.45 Wh kg^{-1} dan 833.19 W kg^{-1} . *Supercapattery* yang difabrikasi menunjukkan kebolehkitaran yang sangat baik dengan pengekaln kapasiti spesifik 97.46% selepas 5,000 kitaran.

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LIST OF ABBREVIATIONS

AC	-	Activated carbon
BAWDC	-	Boron-doped activated wood-derived carbon
BET	-	Brunauer Emmett Teller
BMO	-	Binary metal oxide
CAD No.	-	Chemical Abstracts Service Number
CCD	-	Central Composite Design
CNT	-	Carbon nanotube
CoFe ₂ O ₄	-	Cobalt ferrite
CV	-	Cyclic voltammogram
CVD	-	Chemical vapor deposition
DIB	-	Dual-ion battery
EDLC	-	Electrochemical double-layer capacitor
EDS	-	Energy dispersive spectroscopy
EES	-	Electrochemical Energy Storage
EIS	-	Electrochemical impedance spectroscopy
ESR	-	Equivalent series resistance
ESS	-	Energy storage system
EV	-	All-electric vehicle
FESEM	-	Field emission scanning electron microscopy
FTIR	-	Fourier transform infrared spectroscopy
GCD	-	Galvanostatic charge-discharge
GHBC	-	Grape husk biochar
GO	-	Graphene oxide
GSBC	-	Grape stalk biochar
GWR	-	Ground watermelon rind
HEV	-	Hybrid electric vehicle
HMB	-	Hierarchically porous magnetic biochars
JCPDS	-	Joint committee on powder diffraction
MB	-	Magnetic biochar
MOP	-	Magnetic orange peel

MWCNT	-	Multi-wall carbon nanotube
MWRB	-	Magnetic watermelon rind biochar
MWRB _{CoFe2O4}	-	Magnetic watermelon rind biochar with Cobalt ferrite
MWRB _{NiFe2O4}	-	Magnetic watermelon rind biochar with Nickel ferrite
NKWC	-	Wood-derived nitrogen-doped porous carbon
NiFe ₂ O ₄	-	Nickel ferrite
NF	-	Nickel foam
NSBC	-	Nut shield biochar
NMP	-	N-methyl-2-pyrrolidone
PANI	-	Polyaniline
PEDOT	-	Poly(3,4-ethylenedioxythiophene)
Ppy	-	Polypyrrole
PSBC	-	Plum stone biochar
PVDF	-	Polyvinylidene fluoride
rGO	-	Reduced graphene oxide
SWCNT	-	Single-wall carbon nanotube
TMO	-	Transition metal oxide
UC	-	Ultra capacitor
VSM	-	Vibrating sample magnetometer
WR	-	Watermelon rind
WRB	-	Watermelon rind Biochar
WSBC	-	Wheat straw biochar
TC- MWRB _{CoFe2O4} /PANI	-	Ternary composite of magnetic watermelon rind biochar with cobalt ferrite/polyaniline
XRD	-	X-ray dispersive

LIST OF SYMBOLS

A	-	Surface area
B	-	Applied magnetic field
C_s	-	Specific capacitance of electrode
C	-	Coulomb
C	-	Material-specific curie constant
d	-	Thickness of double layer
dV	-	Potential window
f	-	Response function
F	-	Farad
Hz	-	Hertz
I	-	Current
k	-	Number of variables
m	-	Mass loading
M	-	Resulting magnetisation
M_s	-	Saturation magnetisation
M_r	-	Retentivity
pH	-	Potential of hydrogen
Oe	-	Coercive field
Q_s	-	Specific capacity of the electrode
Q_d	-	Specific capacity of the device
R_{ct}	-	Charge transfer resistance
R^2	-	Correlation coefficient
v	-	Scan rate
T	-	Absolute temperature
T_c	-	Curie's temperature
V_i	-	Initial potential
V_f	-	Final potential
Z_{re}	-	Real part of impedance
Z_{im}	-	Imaginary part of impedance
Δt	-	Discharge time

ΔV	-	Voltage window
α	-	Absorption coefficient
λ	-	X-ray wavelength of radiation
θ	-	Diffraction angle
ε	-	Experimental error or residuals
ε_0	-	Electrical permittivity of free space
ε_r	-	Dielectric constant
Ω	-	Ohm for impedance
χ	-	Magnetic susceptibility

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CHAPTER 1

INTRODUCTION

1.1 Background

The emergence of electronics has rapidly increased the demand for energy storage devices. On the other hand, the aggravation of environmental pollution and the depletion of fossil fuels have stimulated the development of green, renewable, and favorable energy storage devices (Agudosi et al., 2020; Dong et al., 2020; Xie et al., 2018). Supercapacitors, batteries, fuel cells, and conventional capacitors are standard smart energy technologies. Extensive research and exploration are being executed worldwide on each of these devices. Supercapacitors have drawn intensive attention due to their rapid charge-discharge rate, high power density, long cycle life, and eco-friendliness (Kalyani & Anitha, 2013). One of the impediments to employing supercapacitors on a large scale is the expense encountered. Thus, replacing commercial activated carbon with biochars can be considered an option due to the environmental considerations (Thomas et al., 2020).

Biochar is the solid carbon-rich material produced through the thermochemical conversion of biomass (Xiu et al., 2017; Zhang et al., 2014a; Zhang et al., 2014b). Biomass can be referred to as organic materials originating from living matters such as animals and plants or organic and inorganic tissues, such as excrement of animals, sludge, and waste wood (Cha et al., 2016; Tripathi et al., 2016). Over the years, biochar has gained substantial interest in developing materials for the applications ranging from carbon capture and storage, hydrogen storage, environmental remediation, and energy storage devices due to its unique properties such as high absorption capacity, variability in microporosity, high specific surface area, and high ion exchange capability (Balahmar et al., 2015; Blankenship et al.; Li et al., 2018a; Wang & Wang, 2019; Xiu et al., 2017).

The various methodological approaches developed and established throughout the years for biochar production include pyrolysis, carbonisation, and gasification. Essentially the production routes significantly affected the properties of the produced biochar. For instance, biochar yield greatly depends on pyrolysis type. For example, slow pyrolysis performed at a longer residence time and a moderate temperature (350–550 °C) in the absence of O₂ results in a higher yield of biochar (30%) than the fast pyrolysis (12%) or gasification (10%) (Inyang & Dickenson, 2015). Besides, the production temperature and the types of biomass precursors also play a vital role in determining the properties of biochar (Sun et al., 2014). The biochar needs some modifications to increase its specific surface area (SSA) and pore fraction or form functional groups before use to expand its applications. Pre/Post-synthetic changes are conducted based on as-synthesised biochar, like physical or chemical activation, surface functionalisation, or nanomaterials/biochar composites. In recent years, researchers have shown an increased interest in ternary composites by incorporating conducting polymers such as polyaniline, polypyrrole, polyacetylene, etc., as measures to enhance the electrochemical capacitance (Thomas et al., 2020; Yang & Ionescu, 2017).

This study converted the watermelon rinds into magnetic biochar through the self-purging pyrolysis method. Binary metal oxide, nickel ferrite (NiFe₂O₄) and cobalt ferrite (CoFe₂O₄) were impregnated on dried watermelon rind to incorporate metal ions into the magnetic biochar. The operating parameters (pyrolysis temperature, pyrolysis time and watermelon rind to binary metal oxide ratio) were optimised to produce magnetic biochar with high specific capacity for supercapacitor's electrode. The optimised magnetic watermelon rind biochar (MWRB) was then combined with polyaniline via in-situ polymerisation to produce a ternary composite of magnetic watermelon rind biochar and polyaniline (TC-MWRB/PANI) to enhance its electrochemical performance. This enables faradaic redox reaction, and charging occurs throughout the electrode materials. The electrode fabrication method is employed as adopted by Kouchachvili and Entchev (2017) and Thines et al. (2016), which is further elaborated in Methodology. In addition to these, Response Surface Methodology (RSM) will be carried out using a Design Expert to optimise the parameters in producing MWRB and TC-MWRB/PANI composites for supercapacitor's electrode application.

1.2 Problem Statement

Detrimental environmental consequences of fossil fuels usage and their depletion issue have motivated serious concerns leading to enormous research focusing on determining adequate alternatives for efficient energy storage systems from various abundantly available renewable and sustainable materials for energy sources (Kim et al., 2021). Among impending measures to meet increasing global energy demand, energy conversion applications, including hydrogen fuel cells, photovoltaic cells, wind power and energy storage applications, including metal-ion batteries and supercapacitors, are considered the most viable options (Iqbal et al., 2020b). The rapid pace in the emergent trend of portable electronic devices and hybrid vehicles is another cause to explore efficient energy storage systems.

As a class of promising energy storage systems, supercapacitors have received much attention owing to their expansive applications for storing intermittent electrical energy (Chen et al., 2015a). Thus, the electrode material as the key contributor to the energy storage of supercapacitors is attracting considerable research interest from the materials science and energy storage field (Chen et al., 2013; Liu et al., 2015b; Zhu et al., 2014).

Generally, activated carbon is commonly used as electrode material for supercapacitors. However, the drawbacks of using activated carbons as electrode materials are slow mass transport of electrolyte ions because of space confinement imposed by small pore sizes, low conductivity because of the presence of enormous surface functional groups and defects due to activated process, and collapse of porous structures during high temperature treatments is low capacitance stability at high current densities which limits their commercial applications.

In the use of biochar in energy storage applications, surface area, pore structure, and conductivity behavior of the porous carbon material are the most important properties that affect electrochemical performance. As the surface area increases, the capacitance increases due the higher amount of charge accumulated on the electrode surface. However, high surface area alone is not sufficient for high

capacitance. It is also crucial that the pores are to be 'open' as well as their sizes. Not all pores on the surface are electrochemically accessible. Apart from carbon materials, conducting polymers such as polyaniline (PANI) has proved to be a promising supercapacitor electrode due to their high charge density and ease of fabrication. However, PANI experiences significant volumetric swelling and shrinking during the charge-discharge process due to ion doping and de-doping, leading to structural breakdown and flaking off PANI. Thus, compositing biomass carbon with metal oxides and conducting polymer lead to enhanced supercapacitor properties, as evident from previous research since the effective carbon support is expected to provide good electrical conduction with metal oxides, large surface area to disperse them, and suitable pore channels for their deposition and for electrolyte ion transportation (Thomas et al., 2020; Yang & Ionescu, 2017).

It was hypothesised that the preparation of MWRB via pyrolysis in vacuumed conditioned electric muffle furnace gives a great advantage as facile single route synthesis of magnetic biochar rather than conventional pyrolysis in the presence of inert gas. To date, biochar derived from biomass waste such as coconut shell, rice husk, coffee shell, durian rind, and the banana peel has been studied to evaluate its potential as a supercapacitor's electrode. However, such information is limited for watermelon rind biomass. The introduction of binary metal oxides of NiFe_2O_4 and CoFe_2O_4 into the biochar framework was expected to improve the biochar's porosity and promote active sites, which are expected to enhance the electrochemical properties of biochars. Lastly, the introduction of PANI was also anticipated to enhance electrochemical properties due to the synergetic combination of the excellent mechanical strength of carbon materials and the high pseudocapacitance of PANI.

1.3 Research Objectives

The research aims to develop the magnetic watermelon rind biochar with binary metal oxide (nickel ferrite, NiFe_2O_4 and cobalt ferrite, CoFe_2O_4) and a ternary composite of magnetic watermelon rind-polyaniline (TC-MWRB/PANI) for the fabrication of electrode materials for supercapacitors. It is known that the properties of magnetic biochars and magnetic biochar composites are found to be dependent on processing parameters, materials combination and proportion. Thus, a comprehensive selection of materials, synthesising parameters and materials ratio should be made. To achieve this key objective, several explicit aims need to be addressed:

- i. To optimise MWRB synthesis parameters such as pyrolysis temperature, pyrolysis time and binary metal oxide to biomass ratio for specific capacity measurement using Response Surface Methodology (RSM) by Design Expert® Version 13.
- ii. To synthesise TC-MWRB/PANI composites through in-situ polymerisation and optimise the parameters such as PANI ratio, sonication time and sonication amplitude for supercapacitor's electrode using RSM by Design Expert® Version 13.
- iii. To study morphology, chemical, elemental, magnetic and electrochemical properties of the MWRB and TC-MWRB/PANI composites.
- iv. To fabricate and investigate the performance of the synthesised TC-MWRB/PANI composites for energy storage application through CV, GCD, lifecycle and EIS measurements

1.4 Scopes of Research

Magnetic biochars were prepared from waste watermelon rind (WR) with the impregnation of binary metal oxide via self-purging pyrolysis by attaining limited oxygen conditions in an electric muffle furnace without using any carrier gas. There

are two types of binary metal oxide used in the preparation of MWRB, which are NiFe_2O_4 and CoFe_2O_4 . The RSM was implemented using Design-Expert[®] software Version 13 (Stat-Ease, Inc.) to optimise MWRB production. The pyrolysis independent variables and their ranges were pyrolysis temperature (600 °C-900 °C) and pyrolysis time (10-60minutes), and watermelon rind to binary metal oxide ratio ,WR:BMO (25-75 weight %). In contrast, the dependent variable was the specific capacity (Qs) response. The MWRB produced at optimum condition were then undergo extensive material characterisations involving field emission scanning electron microscopy-energy dispersive x-ray spectroscopy (FESEM-EDX), X-ray diffractometry (XRD), Fourier transforms infrared (FTIR), Brunauer–Emmett–Teller (BET) surface area and Vibrating sample magnetometer (VSM).

The optimised MWRB samples were then were added with PANI to produce TC-MWRB/PANI composites via in-situ polymerisation. Likewise, the RSM by Design Expert[®] software Version 13 software (Stat-Ease, Inc.) was employed to optimise TC-MWRB/PANI synthesis. The factors such as the PANI concentration, sonication amplitude, and sonication time were optimised within 3-7 mmol, 30-80 %, and 15-30 minutes, respectively. The electrochemical performance of the composites in terms of their specific capacity was designated as the response. The characterisations of optimised TC-MWRB/PANI composites were done through FESEM-EDX, XRD, FTIR, BET surface area, and VSM studies.

The electrochemical evaluations of both optimised MWRB and TC-MWRB/PANI composite were investigated through cyclic voltammetry (CV), galvanostatic charge-discharge (GCD), and electrochemical impedance spectroscopy (EIS). A solution of potassium hydroxide (KOH), 1M, was used as an electrolyte. At the same time, the as-synthesised MWRB and TC-MWRB/PANI composites, saturated calomel electrode (SCE), and platinum wire were employed as working reference, and counter electrodes, respectively.

1.5 Significance of Research

This recent research developed MWRB from waste watermelon rinds and TC-MWRB/PANI composites for supercapacitor's electrode material. Transition metal oxides of NiFe_2O_4 and CoFe_2O_4 were used to produce MWRB since the pseudocapacitive oxides of transition metal are widely explored due to their high theoretical capacitance, low cost, and reversible faradaic redox reactions compared to carbonaceous materials. Thus, incorporating these transition metal oxides was expected to enhance the specific capacitance of synthesised MWRB.

It is well known that, among various electrode materials, PANI has been proved to be a favorable supercapacitor's electrode material due to its low cost, high charge density, good electrical conductivity in doped states, and facile fabrication for large scale devices (Yu et al., 2015). However, PANI experiences significant volumetric swelling and shrinking during the charge-discharge process due to electrochemical instability. Consequently, the combination of MWRB with PANI becomes favorable in increasing the electrochemical performance of the electrode materials. Thus, the incorporation of earth-abundant carbon materials such as biomass with PANI is one of the effective ways to improve the electrochemical stability of PANI and enhance energy output.

Besides, the demand for watermelon in Malaysia is not decreasing due to its delicious taste and broad availability, especially during hot weather. As more fruits are being consumed daily, the availability of the rinds is increasing as well. Managing these rinds is quite tedious, and humans generally opt for the dumping and burning choice to overcome this problem. This option has already led to several environmental issues (Khedari et al., 2003). Dumping food waste in the landfill will gradually rot and release methane, a strong greenhouse gas. Hence this approach is expected to contribute to minimising the issues mentioned above.

1.6 Thesis Outline

This thesis comprises five chapters.

Chapter 1 provides a brief background about the energy storage devices and the problems encountered due to the shortage of fossil fuels. Then comes the introduction of biochar as an alternative. The types of biochar, its processing methods, and applications in the various field are delineated. The biochar's modification to enhance its properties is also deliberated in this chapter.

Chapter 2 presents the details of the literature, and the feasibility study of this research is given and elaborated. It also explains the general concepts related to Response Surface Methodology (RSM), electrochemical analysis and the characterisations involved in biochar specifically for energy storage applications.

Chapter 3 highlights the overall research methodology and procedures. The experimental procedures for the synthesis and production of MWRB and TC-MWRB/PANI composites are reported. The experimental methods, materials, and design factors for optimisation and their ranges are stated with justifications. The material characterisations (FTIR, XRD, BET, VSM, and FESEM-EDX) and the electrochemical evaluation (CV, GCDs, and EIS) techniques for analysing optimised samples for both the as-synthesised MWRB and TC-MWRB/PANI composites are articulated. A flowchart illustrating the summary of this chapter is presented in this chapter.

Chapter 4 provides in-depth discussions and thorough analysis and the results of statistical RSM analysis of MWRB and TC-MWRB/PANI and elemental, morphological, structural, and electrochemical analysis of the optimised MWRB and TC-MWRB/PANI composites.

Lastly, Chapter 5 summarily presents the findings based on the set of objectives and recommendations for future research based on present findings.

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LIST OF PUBLICATIONS

Journal with Impact Factor

- 1 **Omar, N.**, Abdullah, E.C., Petrus, A.A. *et al.* Single-route synthesis of binary metal oxide loaded coconut shell and watermelon rind biochar: Characterizations and cyclic voltammetry analysis. *Biomass Conv. Bioref.* (2021). <https://doi.org/10.1007/s13399-021-01367-3> **Q2, IF: 4.987**
- 2 **Omar, N.**, Abdullah. E.C., Arshid, N., Mubarak, N.M., Khalid, M., Aid, S.R., and Agudosi, E.S. (2022). Facile synthesis of a binary composite from watermelon rind using response surface methodology for supercapacitor electrode material. *Journal of Energy Storage*, 45 <https://doi.org/10.1016/j.est.2022.104147> **Q1, IF: 6.583**
- 3 **Omar, N.**, Abdullah. E.C., Arshid, N., Mubarak, N.M., Khalid, M., Aid, S.R., and Agudosi, E.S. (2022). Recent Development of Biochar-based nanocomposites for Electrochemical Energy Storage Application. *Journal of Energy Storage* (Under review) -
- 4 Agudosi, E. S., Abdullah, E. C., Numan, A., Mubarak, N. M., Khalid, M., & **Omar, N.** (2020a). A Review of the Graphene Synthesis Routes and its Applications in Electrochemical Energy Storage. *Critical Reviews in Solid State and Materials Sciences*, 45(5), 339-377. <https://doi.org/10.1080/10408436.2019.1632793> **Q1, IF: 8.344**
- 5 Agudosi, E. S., Abdullah, E. C., Numan, A., Mubarak, N. M., Aid, S. R., Benages-Vilau, R., Gómez-Romero, P., Khalid, M., & **Omar, N.** (2020b). Fabrication of 3D binder-free graphene NiO electrode for highly stable supercapattery. *Scientific Reports*, 10(1), 11214. <https://doi.org/10.1038/s41598-020-68067-2> **Q1, IF: 4.011**
- 6 Agudosi, E. S., Abdullah, E. C., Numan, A., Khalid, M., Mubarak, N. M., Aid, S. R., & **Omar, N.** (2021). Optimising the fabrication of 3D binder-free graphene electrode for electrochemical energy storage application. *Surface and Coatings Technology*, (413), 127080 <https://doi.org/10.1016/j.surfcoat.2021.127080> **Q1, IF: 4.158**

- 7 Agudosi, E. S., Abdullah, E. C., Numan, A., Khalid, M., Mubarak, N. M., Benages-Vilau, R., Gómez-Romero, P., Aid, S. R & **Omar, N.** (2021) Optimisation of NiO electrodeposition on 3D graphene electrode for electrochemical energy storage using response surface methodology. *Journal of Electroanalytical Chemistry*, (482) <https://doi.org/10.1016/j.jelechem.2021.114992> **Q1, IF: 4.46**