PSEUDOCAPACITIVE AUGMENTATION OF PALM KERNEL SHELL CARBON FOR HIGH DENSITY SUPERCAPACITIVE CHARGE STORAGE

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SUPERVISOR'S DECLARATION

We hereby declare that We have checked the thesis and, in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Doctor of Philosophy.

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

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Thesis submitted in fulfillment of the requirements
for the award of the degree of
Doctor of Philosophy

Faculty of Industrial Sciences and Technology
UNIVERSITI MALAYSIA PAHANG

JANUARY 2021

DEDICATION

This thesis is dedicated to my beloved Mom and Dad,
Teachers and the Almighty God

ACKNOWLEDGEMENTS

First and foremost, praises and thanks to the God, the Almighty, for His showers of blessings throughout my research work to complete the research successfully.

I'm heartily grateful to my supervisor, Prof. Dr. Jose Rajan- my paragon for his immense support and encouragement in my postgraduate life, for his invaluable guidance that brings me into the wonderful world of advanced material science. His dynamism, vision, sincerity, and motivation have deeply inspired me. I express my deep gratitude towards my co-supervisor, Dr. Izan Izwan Misnon for his suggestions during the research period.

I am extending my thanks to Dr. Mohd Hasbi Ab Rahim, Dean of Faculty of Industrial Sciences and Technology, for his valuable inputs to my research. I also express my humble gratitude to Late Prof. Dato' Dr. Mashitah Mohd. Yusoff for all the help for the completion of this research work.

I would also like to express my sincere thanks to Prof. Dr. Chun-Chen Yang (BRCGE, Ming Chi University of Technology, Taiwan) for his invaluable advice during collaboration research (1st July to 31st December 2018). My special gratitude goes to Dr. Chelladurai Karuppaiah for his knowledge sharing during this period.

I also thank Dr. M.V. Reddy (CETEES, Hydro-Québec), Dr. Anil Kumar (R&D Center, Noritake Co. Ltd., Japan) and technical staffs (FIST lab, Central lab and CARIFF) for their help with instrumental facilities. Many thanks to the Institute of Postgraduate Studies (IPS) of UMP and staffs for their timely help during this research. I thank UMP Doctoral Scheme Scholarship (DSS), UMP Post Graduate Research Grants Scheme (GRS 170309), Noritake Co. Ltd – Universiti Malaysia Pahang co-funded project on ceramic reinforced porous carbon paste (RDU 192701), Strategic Leap 3 research grant (RDU 172201) of Universiti Malaysia Pahang and Fundamental Research Grant Scheme of the Ministry of Education, Govt. of Malaysia (FRGS/1/2019/STG07/UMP/01/1) for financial support.

I thank my lab-mates and friends, Dr. Baiju Vidyadharan, Dr. Midhun Harilal, Dr. Syam G Krishnan, Dr. Zinab Hassanien Bakr, Dr. Bhupender Pal, Dr. Amina Yasin and Miss. Nurul Khairiyyah for their knowledge and experience sharing.

I express my deepest gratitude to my parents and brother for their greatest love, support throughout my study. Last but not least; special thanks to my husband Mr. Varun Geetha Mohan for giving me the energy to overcome the challenges that I have come across in my postgraduate life and he has made an important contribution to the completion of my work.

ABSTRAK

Alotrop dan morfologi bahan karbon merupakan pilihan universal untuk fabrikasi elektrod peranti simpanan tenaga seperti bateri dan superkapasitor; bagaimanapun, kebanyakkan karbon daripada sumber biojisim mempunyai jumlah lompang pasif yang besar dan tidak menyumbang kepada fungsi akhir peranti atau struktur permukaan, dan masih boleh diubahsuai untuk peningkatan keupayaan. Oleh kerana ia boleh diperbaharui, pembangunan karbon teraktif (ACs) dari biojisim bukan makanan merupakan bidang penyelidikan yang aktif. Kajian kedoktoran ini memfokus kepada pengfungsian biojisim AC yang disintesis dari tempurung kelapa sawit (PKS) dengan sedikit (10 wt%) penambahan logam oksida atau logam untuk digunakan sebagai elektrod simpanan tenaga sama ada untuk peranti superkapasitor ataupun hibrid bateri – superkapasitor. Sasaran utama kajian adalah mencapai ketumpatan tenaga (ES) menyamai bateri dan ketumpatan kuasa (PS) seperti superkapasitor. Bagi mencapai objektif utama ini, lapisan tipis Mn2O3 dan metalik Co disalut kepada PKSAC dan seramik berstruktur nano MnCo2O4 dan TiO2 diisi ke dalam lompang AC. Bahan yang dihasilkan dicirikan menggunakan pembelauan sinar-X, mikroskop elektron pengimbasan pelepasan medan, spektrometri sebaran tenaga sinar-X, spektroskopi fotoelektron sinar-X, mikroskopi transmisi elektron dengan analisis pembelauan elektron ruang tertentu dan analisis penjerapan gas. Sifat elektrokimia AC, material tak organik dan komposit karbon – material tak organik dinilai menggunakan voltametri berkitar, kitaran galvanostatik cas-discas dan spektroskopi impedans elektrokimia dengan konfigurasi tiga elektrod dalam 1 M Na2SO4 dan 1 M LiPF6 dilarutkan dengan kadar campuran 1:1 (volum) etilena karbonat dan dietil karbonat. Elektrod AC memberikan kapasitan spesifik (CS) 150 F g-1 dengan kestabilan kitaran 97% selepas 5000 kitaran di dalam elektrolit neutral 1 M Na2SO4 (voltan ~1 V). Nilai CS meningkat beberapa ganda dengan penahanan kapasitan >97% selepas 5000 kitaran apabila lompang disalut atau diisi dengan material tak organik. Elektrod AC diisi dengan bunga-nano MnCo2O4 memberikan CS (510 F g-1) dan tetingkap voltan (~1.2V) paling tinggi dalam elektrolit neutral. Superkapasitor simetri dibina menggunakan elektrod optimum menunjukkan hampir empat kali ganda ES berbanding hanya AC disebabkan peningkatan dalam tetingkap voltan dan kapasitans. Selain itu, AC dan komposit karbon – material tak organik diuji sebagai katod untuk kapasitor ion litium (LIC). LIC dengan 10 wt.% MC@AC menunjukkan tetingkap voltan (~2.5 V), kapasitans superior (160 F g-1) dan kadar keupayaan lebih besar berbanding analog asli. Keputusan ini menunjukkan bahawa protokol semasa membolehkan fabrikasi elektrod simpanan cas superior dari material boleh diperbaharui difungsikan dengan kuantiti minimum material hasil bumi. Tambahan pula, peranti hibrid bateri – superkapasitor difabrikasi dengan bunga-nano MnCo2O4 sebagai anod dan komposit karbon dengan MnCo2O4 sebagai katod menunjukkan ES ~153 W h kg-1 pada PS ~214 W kg-1. Kajian ini menerangkan pendekatan skala penuh untuk memperbaiki kebolehan AC dengan modifikasi sifat permukaan untuk mempertingkatkan prestasi elektrokimia.

ABSTRACT

Allotropes and polymorphs of carbon are a universal choice to fabricate the electrodes of energy storage devices such as batteries and supercapacitors; however, most of these biomass-derived carbons have a large volume of passive voids which do not contribute to the final functionality of the devices or their surface structure could still be tailored for enhanced performance. Owing to their renewability, developments of activated carbons (ACs) from non-edible biomass is an active area of research. This doctoral research focuses on the functionalization of biomass activated carbon synthesized from palm kernel shell (PKS) with low quantities (10 wt.%) of metal oxides or metals for their application as an energy storage electrode either in a supercapacitor or in a battery – supercapacitor hybrid device. Achieving energy density (E_S) similar to that of batteries with similar power density (P_S) as that of supercapacitors has been the principal target in this research. Towards this primary objective, Mn_2O_3 and metallic Co are coated as a thin layer on PKSAC and $MnCo_2O_4$ and TiO_2 ceramic nanostructures loaded into the voids of AC. The materials were characterized using X- ray diffraction, field emission scanning electron microscopy, energy dispersive X-ray spectrometry, X- ray photoelectron spectroscopy, transmission electron microscopy with selected area electron diffraction analyses and gas adsorption measurements. Electrochemical properties of AC, inorganic materials and carbon – inorganic materials composites were evaluated using cyclic voltammetry, galvanostatic charge-discharge cycling and electrochemical impedance spectroscopy in three electrode configuration tested in 1 M Na_2SO_4 and in 1 M $LiPF_6$ dissolved in 1:1 (volume) mixture of ethylene carbonate and diethyl carbonate. The AC electrodes give a specific capacitance (C_S) of 150 F g^{-1} with a cyclic stability of 97% after 5000 cycles in 1 M Na_2SO_4 neutral electrolyte (potential ~1 V). The C_S increased to three-folds with a capacitance retention of >97% after 5000 cycles when the voids are coated or filled with the inorganic materials. The AC electrodes filled with $MnCo_2O_4$ nanoflowers offers one of the highest C_S (510 F g^{-1}) and potential window (~1.2 V) in neutral electrolytes. A symmetric supercapacitor developed using the optimum electrodes showed nearly four times higher energy density than the pure AC owing to the enhancements in voltage window and capacitance. Moreover, AC and carbon – inorganic materials composites are tested as the cathode materials of Lithium Ion Capacitor (LIC). LIC with 10 wt.% MC @AC shows larger voltage window (~2.5 V), superior capacitance (160 F g^{-1}) and rate capability than the pure analogue. These results demonstrate that the current protocol allows fabrication of superior charge storing electrodes using renewable materials functionalized by minimum quantity of earthborn materials. Furthermore, battery – supercapacitor hybrid device is fabricated with $MnCo_2O_4$ nanoflowers as anode and carbon composite with $MnCo_2O_4$ as cathode electrode shows E_S of ~153 W h kg^{-1} at a minimum P_S of ~214 W kg^{-1}. This research describes full scale approach for improving the performance of AC by modifying the surface properties for their enhanced electrochemical performance.

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τ	charge relaxation time
ν	scan rate
λ	wavelength
Θ	angle
C_d	double layer capacitance
C_S	specific capacitance
C_d	electric double-layer capacitance
CPE_{PC}	constant phase element
$f0$	charge relaxation frequency
p/p_o	relative pressure
R_{CT}	charge transfer resistance
R_S	series resistance
W	Warburg impedance
wt.%	weight percentage
Z_W	Warburg impedance
Z'	real impedance
Z''	imaginary impedance
$ZCPE$	constant phase element impedance

LIST OF ABBREVIATIONS

AC	activated carbon
ASC	asymmetric supercapacitor
BET	Brunaurer-Emmett-Teller
BJH	Barrett-Joyner-Halenda
BSH	Battery supercapacitor hybrid
CD	charge-discharge
CNT	carbon nano tube
CV	cyclic voltammetry
EDLC	electrochemical double layer capacitors
EDX	energy-dispersive X-ray spectrometry
EC	electrolytic capacitor
EIS	electrochemical impedance spectroscopy
FESEM	field emission scanning electron microscope
HRTEM	high-resolution transmission electron microscopy
HTAB	hexadecyl trimethyl ammonium bromide
HNWs	hybrid nanowires (MnCo2O4-CoO-MnO2)
LIB	lithium ion batteries
LIC	lithium ion capacitors
NR	Not reported
PANI	polyaniline
PC	pseudocapacitance
SAED	selected area electron diffraction
SC	supercapacitor
SSC	symmetric supercapacitor
TEM	transmission electron microscopy
XPS	X-ray photoelectron spectroscopy
XRD	X-ray diffraction

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