

**SURFACE ENGINEERING OF COMMERCIAL
ACTIVATED CARBON FOR IMPROVING THE
CHARGE STORABILITY OF ELECTROCHEMICAL
CAPACITORS**

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MASTER OF SCIENCE

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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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ABSTRAK

Superkapasitor (SC) berasaskan karbon aktifan adalah contoh peranti mampan di kalangan peranti penyimpanan tenaga elektrokimia kerana bahan elektrod boleh diperbaharui, mesra alam, kitaran hayat yang lebih lama dan keupayaan kadar nyahcas caj yang unggul. Namun, untuk mengembangkan nilai komersil mereka, ketumpatan tenaga semasa mereka haruslah setanding dengan bateri Litium-ion yang terdapat di pasaran. Salah satu pendekatan bagi meningkatkan ketumpatan tenaga adalah dengan memaksimumkan bilangan liang untuk memudahkan penjerapan lebih banyak ion. Sebilangan besar penyelidikan mengenai SC telah menunjukkan keputusan yang sangat baik pada skala makmalmelalui peningkatan keliangan, di mana beban jisim elektrod yang dikaji mempunyai perbezaan yang mendadak daripada piawaian industri. Faktor-faktor ini merupakan sebahagian daripada cabaran inisiatif penkomersialan elektrod karbon biojisim, Untuk menangani isu ini, tesis ini memberi tumpuan kepada peningkatan performasi elektrokimia karbon aktifan cangkerang isi rong sawit melalui modifikasi permukaannya dengan cara yang mesra alam dan kos efektif. Dalam tesis ini, kaedah refluks asid nitrik telah digunakan untuk tujuan pengaktifan. Berbeza dengan kaedah konvensional, kaedah ini mengurangkan penggunaan bahan kimia yang keras, di mana bahan kimia ini boleh dikitar semula. Untuk mengoptimumkan prestasi elektrod, karbon teraktif telah direflukskan dengan pelbagai nisbah asid kepada pendahulu dan tempoh refluks. Prestasi elektrokimia bahan yang terhasil telah diuji dalam sistem konfigurasi tiga elektrod dalam 1 M elektrolit sodium sulfat. Kapasiti spesifik sampel optimum telah meningkat ~110% berikutan pengurangan ketara dalam impedans Warburg. Untuk memahami perubahan fizikokimia yang diperkenalkan semasa refluks, sampel karbon assintesis telah dicirikan menggunakan Difraksi sinar-X, Spektroskopi Inframerah Fourier Transformasi, Mikroskopi Elektron Pengimbasan, Spektroskopi Penyerakan Tenaga, dan ukuran penjerapan gas. Sampel refluks 72 jam telah mencatat kenaikan prestasi electrokimia sebanyak ~75% dan luas permukaan $\sim 722 \text{ m}^2 \cdot \text{g}^{-1}$. Setelah sampel tersebut dioptimumkan melalui sistem tiga-elektrod, sifat elektrokimia sampel tersebut dikaji dengan mempelbagaikan muatan jisim ($\sim 3, \sim 6, \sim 9, \sim 12$, dan $\sim 14 \text{ mg} \cdot \text{cm}^{-2}$). Semua peranti yang direka telah mencapai tetingkap berpotensi 1.8 V dalam 1M sodium sulfat. Peranti yang memiliki muatan jisim tertinggi ($\sim 14 \text{ mg} \cdot \text{cm}^{-2}$) telah menghasilkan kapasitans maksimum $\sim 494 \text{ mF} \cdot \text{cm}^{-2}$, ketumpatan tenaga $\sim 13 \text{ mWh} \cdot \text{cm}^{-3}$, dan ketumpatan kuasa maksimum $\sim 2189 \text{ mW} \cdot \text{cm}^{-3}$. Penyelidikan semasa telah menunjukkan pendekatan yang mesra alam dan ekonomi bagi kejuruteraan keliangan karbon aktifan komersil, untuk meningkatkan kebolehstoran cas bagi aplikasi praktikal.

ABSTRACT

Supercapacitors based on activated carbon are the representatives of sustainable devices among electrochemical energy storage devices because of their renewable electrode materials, eco-friendliness, longer life cycle and superior charge-discharge rate capabilities. However, to expand their commercial value, their current energy densities should be made comparable with the market leading Lithium-ion batteries. One of the approaches to increase the energy density is by maximizing the number of pores to incorporate more ions. A majority of the research on supercapacitors demonstrated excellent laboratory-scale results through improving porosity, where the mass loading of such electrodes has a staggering difference from the industrial standards. These factors predominantly suppressed the initiatives to lift the biomass-derived carbon-based electrodes into the commercial picture. To address this issue, the present thesis focuses on expanding the electrochemical properties of commercial activated carbon derived from palm kernel shells by engineering its porosity in an eco-friendly and cost-effective manner. Herein, we employ the nitric acid refluxing method for the activation purpose, which, unlike the conventional routes, not only limits the usage of harsh chemicals, but also enables recyclability. We have optimized the performance of the electrode materials by refluxing the activated carbon for various acid to precursor ratios and refluxing duration. The electrochemical performances of the resulting materials were examined in a three-electrode system configuration in 1 M sodium sulphate electrolyte. The specific capacitance of the optimum sample was increased ~110% following a significant reduction in Warburg impedance. To understand the physicochemical alterations introduced upon refluxing, the as-synthesized carbon samples were characterized using X-ray Diffraction, Fourier Transform Infrared Spectroscopy, Scanning Electron Microscopy, Energy Dispersive Spectroscopy, and gas adsorption measurements. With ~75% increment, a highest surface area of $\sim 722 \text{ m}^2 \cdot \text{g}^{-1}$ was recorded for the 72 hours refluxed sample, which aligns with the increased electrochemical performance in corresponding electrodes. Further, supercapacitor devices were fabricated using this optimized sample by varying the mass loading ($\sim 3, \sim 6, \sim 9, \sim 12$, and $\sim 14 \text{ mg} \cdot \text{cm}^{-2}$), and the electrochemical properties were studied. All the fabricated devices achieved a potential window of 1.8 V in 1 M sodium sulphate. The highest mass loaded ($\sim 14 \text{ mg} \cdot \text{cm}^{-2}$) device fabricated using the prepared material has delivered a maximum areal capacitance of $\sim 494 \text{ mF} \cdot \text{cm}^{-2}$, an energy density of $\sim 13 \text{ mWh} \cdot \text{cm}^{-3}$, and a maximum power density of $\sim 2189 \text{ mW} \cdot \text{cm}^{-3}$. The current research thereby demonstrates an environmentally friendly and economic approach for engineering the porosity of commercial activated carbon to enhance the charge storability for practical applications.

TABLE OF CONTENT

DECLARATION

TITLE PAGE

ACKNOWLEDGEMENTS	ii
-------------------------	----

ABSTRAK	iii
----------------	-----

ABSTRACT	iiiiiv
-----------------	--------

TABLE OF CONTENT	v
-------------------------	---

LIST OF TABLES	ix
-----------------------	----

LIST OF FIGURES	x
------------------------	---

LIST OF SYMBOLS	xiii
------------------------	------

LIST OF ABBREVIATIONS	xiv
------------------------------	-----

CHAPTER 1 INTRODUCTION	1
-------------------------------	---

1.1 Background	1
1.2 Problem statement	1
1.3 Research questions and hypotheses	3
1.4 Research objectives	3
1.5 Scope of research	4
1.6 Statement of contribution	5
1.7 Thesis outline	5

CHAPTER 2 LITERATURE REVIEW	7
2.1 Introduction to energy storage devices and their market prospects	7
2.2 Classification of electrochemical energy storage devices	8
2.2.1 Lithium-ion batteries	9
2.2.2 Electrolytic capacitors	10
2.2.3 Supercapacitors	12
2.3 Mechanism of supercapacitive charge storage	14
2.3.1 Electric double-layer capacitors	15
2.3.2 Pseudocapacitors	17
2.3.3 Hybrid capacitors	20
2.4 Electrode materials for Supercapacitors	21
2.4.1 Graphene	21
2.4.2 Carbon nanotubes	24
2.4.3 Activated carbon	23
2.4.3.1 Activation techniques	29
2.5 Summary	37
CHAPTER 3 MATERIALS AND METHODS	38
3.1 Introduction	38
3.2 Research methodology	38
3.3 Synthesis approach	40
3.4 Material characterizations	41
3.4.1 Electrochemical Characterization	41
3.4.1.1 Electrode preparation	41
3.4.1.2 Device fabrication	42

3.4.2 Electrochemical testing	43
3.4.2.1 Cyclic voltammetry	43
3.4.2.2 Galvanostatic Charge-Discharge analysis	44
3.4.2.3 Electrochemical Impedance Spectroscopy analysis	45
3.4.2.4 Electrochemical Impedance Spectroscopy data fitting	47
3.4.3 Physicochemical Characterization	48
3.4.3.1 X-ray Diffraction	48
3.4.3.2 Fourier Transform Infrared spectroscopy	49
3.4.3.3 Energy-dispersive X-ray spectroscopy	50
3.4.3.4 Field emission scanning electron microscope	51
3.4.3.5 Brunauer-Emmett-Teller analysis	52
CHAPTER 4 RESULTS AND DISCUSSION: ELECTROCHEMICAL AND PHYSICOCHEMICAL PROPERTIES OF MATERIALS	54
4.1 Introduction	54
4.2 Electrochemical characterization of materials in three-electrode configuration	54
4.2.1 Optimization of commercial AC: HNO ₃ ratio	54
4.2.1.1 Cyclic voltammetry	54
4.2.1.2 Galvanostatic Charge-Discharge analysis	57
4.2.1.3 Electrochemical Impedance Spectroscopy analysis	59
4.2.2 Optimization of refluxing durations	64
4.2.2.1 Cyclic voltammetry	64
4.2.2.2 Galvanostatic Charge-Discharge analysis	66
4.2.2.3 Electrochemical Impedance Spectroscopy analysis	69
4.3 Physicochemical properties of materials	72
4.3.1 X-Ray Diffraction analysis	73

4.3.2 Fourier Transform Infrared spectroscopy analysis	74
4.3.3 Field emission scanning electron microscopy analysis	76
4.3.4 Energy-dispersive X-ray spectrometry Analysis	77
4.3.5 Surface area and pore size distribution Analysis ((Brunauer-Emmett-Teller (BET))	78
4.4 Device fabrication and testing	81
4.4.1 Electrochemical characterization of devices	81
4.4.1.1 Cyclic voltammetry of SSC	81
4.4.1.2 Galvanostatic Charge-Discharge analysis of SSC	83
4.4.1.3 Electrochemical Impedance Spectroscopy analysis of SSC	86
4.4.2 Ragone plot	89
4.5 Summary	92
CHAPTER 5 CONCLUSIONS AND RECOMMENDATIONS	94
5.1 Conclusions	94
5.2 Recommendations for the future research	95
REFERENCES	94
APPENDIX A	132
Publications	132

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