



UNIVERSITI PUTRA MALAYSIA

**VALORIZATION OF OIL PALM BY-PRODUCT MATERIALS AS A NEW
SUSTAINABLE CARBON SOURCE FOR CARBON-BASED
NANOMATERIAL SYNTHESIS AND ENERGY STORAGE APPLICATION**

SALISU NASIR

ITMA 2019 5



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By

SALISU NASIR

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

April 2019

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DEDICATION

This thesis is dedicated to the beloved memory of my late parents: Mrs. Fatima Binti Adam Alhassan and His Eminence Muhammad Nasir Ibn Usman Isma'il, for introducing me to the world, and setting up the right footings in my life. May Allah (S.W.T.) reward them, be pleased with them, accept all their good deeds, forgive all their shortcomings and make their graves spacious, full of light and a garden of paradise, amen.



Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Doctor of Philosophy

VALORIZATION OF OIL PALM BY-PRODUCT MATERIALS AS A NEW SUSTAINABLE CARBON SOURCE FOR CARBON-BASED NANOMATERIAL SYNTHESIS AND ENERGY STORAGE APPLICATION

By

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April 2019

Chairman : Professor Mohd Zobir Hussein, PhD
Faculty : Institute of Advanced Technology

Energy and environmental issues are the two major problems that our world is facing today. These, combined with developing consumer demands have stirred researchers' interest in inexpensive, environmentally friendly functional materials. With Malaysia as an indicator, and based on a projected annual production of palm oil in Malaysia of over 15.4 million metric tons by 2020, it is estimated that about 46.6 tons of lignocellulosic wastes will be generated. Transforming these wastes into wealth could be integrated into a global paradigm shift towards sustainable development.

Thus, in this research, a new approach was proposed to produce reduced graphene oxide (rGO) from graphene oxide (GO), and activated carbons (AC) using various oil palm by-product materials, namely oil palm leaves (OPL), palm kernel shells (PKS) and empty fruit bunches (EFB). The effect of heating temperature on the formation of graphitic carbon and the yield was examined prior to the GO and rGO synthesis. Carbonization of the starting materials was conducted in a furnace under nitrogen gas for 3 h at temperatures ranging from 400 to 900 °C and a constant heating rate of 10 °C/min. The GO was further synthesized from the as-carbonized materials using the 'improved synthesis of graphene oxide' method. Subsequently, the GO was reduced by low-temperature annealing reduction at 300 °C in a furnace under nitrogen gas for 1 h to produce rGO.

It was found that the I_G/I_D ratio calculated from the Raman spectral analysis increases with the increasing of the degree of the graphitization in the order of rGO from oil palm leaves (rGOOPL) < rGO palm kernel shells (rGOPKS) < rGO commercial graphite (rGOCG) < rGO empty fruit bunches (rGOEFB) with the I_G/I_D values of 1.06,

1.14, 1.16 and 1.20, respectively. The surface area and pore volume analyses of the as-prepared materials were performed using the Brunauer Emmett Teller (BET) nitrogen adsorption-desorption method. The lower BET surface area of 8 and 15 m²g⁻¹ observed for rGO CG and rGO OPL, respectively could be due to partial restacking of GO layers and locally-blocked pores. Relatively, this lower BET surface area is inconsequential when compared to rGO PKS and rGO EFB, with a surface area of 114 and 117 m² g⁻¹, respectively.

Furthermore, electrochemical energy storage performances of the rGOs, and also the as-prepared activated carbons were also all carried out using cyclic voltammetry (CV) method, and were found to be good electrode materials for supercapacitors applications. One of the OPL-based AC electrodes was found to have very high capacitance values of 434 F g⁻¹ at 5 mVs⁻¹, which is much higher than the specific capacitance value (343 F g⁻¹) of the only oil palm leaf-derived porous carbon nanoparticles ever reported in the literature.

On the other hand, a novel phase change material (PCM) made of n-nonadecane infused by capillary forces in a compressed reduced graphene oxide-activated carbon composite matrix was also investigated. Reduced graphene oxide (rGO EFB) - activated carbon (AC) composite was successfully prepared and exhibited an improved thermal conduction property, and was used as a matrix or framework for the fabrication of shape-stabilized composite phase change material (SCPCM). During this SCPCM set-up, which was achieved by simple impregnation method, the pores of the rGO EFB-AC composite serves as the matrix/framework while n-nonadecane as the central envelope. The molten n-nonadecane was successfully stabilized by this porous carbon matrix via the capillary force and surface tension forces which mitigated the seepage of the molten n-nonadecane throughout the phase change cycle. The phase change temperatures and latent heats of composite SCPCM-5 were 37.25 and 25.58 °C and 82.72 and -62.22 J/g, respectively. On the whole, the novel carbon-based nanomaterials produced in this project demonstrate excellent features that enabled them to be used as both thermal and electrochemical energy storage materials.

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

PENAMBAHAN NILAI PRODUK SAMPINGAN KELAPA SAWIT SEBAGAI SUMBER KARBON BARU YANG MAMPAN UNTUK SINTESIS BAHAN NANO BERASASKAN KARBON DAN APLIKASI PENYIMPANAN TENAGA

Oleh

SALISU NASIR

April 2019

Pengerusi : Profesor Mohd Zobir Hussein, PhD
Fakulti : Institut Teknologi Maju

Antara permasalahan besar yang dihadapi masyarakat dunia hari ini adalah isu-isu berkaitan tenaga dan alam sekitar. Di samping itu, permintaan pengguna yang semakin meningkat turut menggalakkan kajian berkaitan bahan berfungsi yang murah dan mesra alam. Dengan menggunakan Malaysia sebagai indikator dan berdasarkan jangkaan produksi minyak kelapa sawit Malaysia yang akan melebihi 15.4 juta tan matrik pada tahun 2020, dijangka bahawa sebanyak 46.6 tan sisa lignoselulosa akan dijana. Kemampuan untuk menukar sisa tersebut kepada bahan bernilai mungkin boleh diintegrasikan ke dalam usaha perubahan paradigma dunia ke arah pembangunan mampan.

Sehubungan dengan itu, kajian ini memperkenalkan kaedah baru untuk penghasilan grafen oksida terturun (rGO) daripada grafen oksida (GO) dan karbon teraktif (AC) menggunakan produk sampingan industri kelapa sawit iaitu daun kelapa sawit (OPL), tempurung sawit (PKS) dan tandang kosong kelapa sawit (EFB). Kesan suhu pemanasan terhadap penghasilan karbon grafitik dikaji sebelum proses sintesis GO dan rGO. Karbonasi bahan mentah telah dijalankan di dalam relau berisi gas nitrogen selama 3 jam pada suhu antara 400 ke 900 °C dengan kadar pemanasan 10 °C/min. Kemudian GO telah disintesis menggunakan bahan yang telah dikarbonasi dengan menggunakan kaedah Hummers' yang telah ditambah baik. Seterusnya, GO diturunkan menggunakan kaedah penurunan penyepuhllindungan suhu rendah pada suhu 300 °C di dalam relau berisi nitrogen selama 1 jam untuk menghasilkan rGO.

Didapati bahawa nisbah I_G/I_D yang dikira daripada analisis spektroskopi Raman bertambah dengan pertambahan kadar penggrafitan mengikut urutan berikut; rGO

daripada daun kelapa sawit (rGOOPL) < rGO daripada tempurung sawit (rGOPKS) < rGO daripada graphit komersil (rGOCCG) < rGO daripada tandan kosong kelapa sawit (rGOEFB) dengan nilai I_G/I_D masing-masing 1.06, 1.14, 1.16 and 1.20. Analisis luas permukaan dan isipadu liang bahan-bahan yang disintesis telah dikaji menggunakan kaedah penjerapan-penyahjerapan nitrogen Brunauer Emmett Teller (BET). Luas permukaan yang rendah telah didapati untuk rGOCCG dan rGOOPL (8 dan $15 \text{ m}^2\text{g}^{-1}$) adalah berkemungkinan disebabkan oleh penyusunan semula lapisan GO serta liang yang tersumbat. Secara relatif, luas permukaan BET yang rendah ini tidak penting jika dibanding dengan nilai luas permukaan BET rGOPKS dan rGOEFB, iaitu masing-masing, 114 and $117 \text{ m}^2 \text{ g}^{-1}$.

Di samping itu, prestasi penyimpanan kuasa elektrokimia oleh rGO dan karbon teraktif yang dihasilkan telah dikaji menggunakan kaedah voltametri berkitar (CV). Didapati bahawa semua rGO dan karbon teraktif menunjukkan prestasi baik sebagai elektrod untuk aplikasi superkapasitor. Salah satu elektrod AC berasaskan OPL menunjukkan nilai kapasiti yang tinggi iaitu 434 F g^{-1} pada 5 mVs^{-1} . Nilai ini lebih tinggi jika dibandingkan dengan nilai kapasiti bahan partikel nanokarbon berasaskan daun kelapa sawit tunggal yang pernah dilaporkan dalam literatur (343 F g^{-1}).

Selain itu, bahan berubah fasa (PCM) baru yang dihasilkan menggunakan komposit grafen oksida terturun (rGOEFB) /karbon teraktif (AC) dan n-nonadekana turut dikaji. Komposit rGOEFB-AC bertindak sebagai matrik sementara n- nonadekana bertindak sebagai bahan berubah fasa yang utama. Pencairan n- nonadekana telah berjaya distabilkan menggunakan karbon matrik berongga melalui daya kapilari dan daya tegang permukaan yang mengurangkan rembesan pencairan n- nonadekana sepanjang kitaran perubahan fasa. Perubahan fasa suhu dan pengumpulan haba komposit masing-masing, SCPCM-5 adalah 37.25 dan $25.58 \text{ }^\circ\text{C}$ dan 82.72 dan -62.22 J/g . Secara keseluruhannya, bahan-bahan nanokarbon baru yang dihasilkan di dalam kajian ini menunjukkan ciri-ciri unggul untuk digunakan sebagai bahan penyimpanan tenaga haba dan juga tenaga elektrokimia.

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Eternally yours,
Salisu Nasir



Declaration by graduate student

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LIST OF ABBREVIATIONS

0-D	Zero Dimensional
1-D	One Dimensional
2-D	Two Dimensional
3-D	Three Dimensional
AC	Activated Carbon
BET	Brunauer-Emmet-Teller
BJH	Barrett– Joyner–Halenda
CG	Commercial Graphite
CV	Cyclic Voltammetry
CVD	Chemical Vapour Deposition
DSC	Differential Scanning Calorimeter
DTG	Differential Thermogravimetric
EDLCs	Electric Double Layer Capacitors
EFB	Empty Fruit Bunch
EFBAC	Empty Fruit Bunch-Derived Activated Carbon
EFB(rGOAC)-M	Reduced Graphene Oxide-Activated Carbon Matrix
EPA	Environmental Protection Agency
ESR	Equivalent Series Resistance
ETES	Electrochemical and Thermal Energy Storage
FAO	Food and Agriculture Organization of the United Nations
FESEM	Field Emission Scanning Electron Microscope
FTIR	Fourier Transforms Infrared Spectroscopy
GCD	Galvanostatic Charge and Discharge
GCE	Glassy Carbon Electrode

GO	Graphene Oxide
GOCG	Graphene Oxide Derived from Commercially Acquired Graphite
GOEFB	Graphene Oxide Derived from Empty Fruit Bunch
GOOPL	Graphene Oxide Derived from Oil Palm Leaves
GOPKS	Graphene Oxide Derived from Palm Kernel Shells
ΔH_f	Enthalpy Change of Freezing
ΔH_m	Enthalpy Change of Melting
HRTEM	High Resolution Transmission Electron Microscopy
I_D/I_G	Ratio of the Intensity of the Raman D-Peak to that of the G-Peak
I_G/I_D	Ratio of the Intensity of the Raman G-Peak to that of the D-Peak
IUPAC	International Union of Pure and Applied Chemistry
J/g	Joule Per Gram
MPOC	Malaysian Palm Oil Council
mV/s	Millivolts Per Second
MWCNT	Multiwall Carbon Nanotubes
nm	Nanometer
OPL	Oil Palm Leaf
OPLAC	Oil Palm Leaf-Derived Activated Carbon
PA	Phosphoric Acid
PCNs	Porous Carbon Nanoparticles
PKS	Palm Kernel Shell
PKSAC	Palm Kernel Shell-Derived Activated Carbon
rGO	Reduced Graphene Oxide
rGOCG	Reduced Graphene Oxide Derived from Commercial Graphite
rGOEFB	Reduced Graphene Oxide Derived from Empty Fruit Bunch

rGOOPL	Reduced Graphene Oxide Derived from Oil Palm Leaves
rGOPKS	Reduced Graphene Oxide Derived from Palm Kernel Shells
rpm	Revolution Per Minute
SEM	Scanning Electron Microscopy
SCPCM	Shape-Stabilized Composite Phase Change Material
SCPCM-1	SCPCM derived from 10 wt % of n-Nonadecane
SCPCM-2	SCPCM derived from 30 wt % of n-Nonadecane
SCPCM-3	SCPCM derived from 50 wt % of n-Nonadecane
SCPCM-4	SCPCM derived from 70 wt % of n-Nonadecane
SCPCM-5	SCPCM derived from 90 wt % of n-Nonadecane
SWCNT	Single Wall Carbon Nanotube
TCE	Thermal Conductivity Enhancer
TES	Thermal Energy Storage
TEM	Transmission Electron Microscopy
TGA	Thermogravimetric Analysis
UN	United Nations
WEC	World Energy Council
wt	Weight
w/v	Weight by Volume
XRD	X-ray Diffraction
μm	Micrometer

CHAPTER 1

INTRODUCTION

1.1 General Background of the Research

Oil palm-derived wastes are often becoming environmental problems in the Southeast Asian developing countries, especially, Malaysia, Indonesia and Thailand. The use of natural bioresources or biomass for carbon-based nanostructures fabrication as well as their energy storage-related applications are currently receiving a great deal of attention [1]. As a result, problems associated with the generation of bio-agricultural wastes are prompting researchers to find ways of utilizing them in order to protect the environment. Most of the natural components (especially those of lignocellulosic inclination) from those wastes can be utilized as carbon sources for creating innovative materials as a function of cost. Thus, high technology industries are compelled to find alternative materials to substitute the conventional materials due to economic and environmental challenges. These alternative materials are believed to hold some advantages over their counterparts (i.e. the conventional materials) in terms of availability/sustainability, renewability, cost effectiveness and compatibility with the natural environment. In summary, the establishments of sustainable and innovative solutions are required to tackle those sprouting problems. The main intention of this thesis is to contribute with new knowledge to a better understanding of the huge potentials of oil palm waste-based biomass materials for the fabrication of carbon-based nanomaterials (graphene oxide, reduced graphene oxide and activated carbons) and to explore their thermal and electrochemical energy storage potentials.

To begin with, nanomaterials (in general) are materials that have distinctive or novel properties, due to the nanoscale structuring. The materials are usually produced by integrating or structuring of nanoparticles. They are subdivided into nanopowders, nanocrystals and nanotubes and can have zero, one, two or three dimensions on the nanoscale. The current interest on nanomaterials is ascribed to the small scale dimension of the materials. Materials with nanoscale size have essentially different properties as compared to the bigger scale and this is attributed to increased surface area to volume ratios. In essence, increased interaction and reactivity is expected with these materials and thus potentially using less of the materials [2].

1.2 Graphitic Nanostructures

Typical graphitic nanostructures comprise graphene, graphene oxides (GO), reduced graphene oxide (rGO), graphene quantum dots (GQDs), carbon nanotubes (CNTs), carbon nanofibers (CNFs), carbon nanohorns and onion-like carbon (OLC). They are usually synthesized using a variety of carbon sources (as the precursors) with different methods. In the context of this thesis, we focus largely on the preparation of GO, rGO and activated carbon with renewable and economical materials (biomass) as carbon source.

1.3 Carbon Nanomaterials at a Glance

An evidence of the synthesis and application of carbon-based materials went back to several centuries ago. The material under discussion encompasses complete and diverse crystallographic structures with various shapes, dimensions, geometries and chemical bonds. A typical example of these materials are but not limited to, various sorts of graphite, synthetic and naturally occurring diamond, activated carbon, carbon-based aerogels, carbon fibres, and their composites, carbon nanotubes, fullerenes, graphene and other graphene-related and derived materials, etc. In recent times, the controlled manipulation, reduction and modification of sample dimension into a small number of nanometers is getting so much attention. Researchers are captivated in the nanoscale due to the fact that physical and chemical properties of materials at nanoscale change considerably from those at a larger scale. Therefore, nanoscaled materials could be designed and developed through the modified, controlled and size-selective production of nanoscale building block with tunable and enhanced physical and chemical properties.

1.4 Carbon Nanomaterials on the Global Energy Perspective

According to world energy council report (WEC) [3], it is projected that the world's population will expand from nearly 7 billion in 2013 to about 8.7 billion in the Jazz scenario and around 9.4 billion in the Symphony scenario in 2050, respectively, which is equivalent to a 26% boost up (36% respectively). As a result, the world energy demand will keep on increasing. It is forecasted that the demand may double by 2050. This calls for a new and efficient means to also double the energy supply in order to meet the challenges that forge ahead. Carbon nanomaterials are believed to be appropriate and promising (when used as energy materials) to cushion the threat. Consequently, the need and the challenge to expand and revolutionize the energy future to clean and sustainable one is enormous and necessary. The future outlook for major energy intermingles in 2050 demonstrates that increase rates will be supreme for renewable energy sources. The contribution of renewable energy supply will rise from approximately 15% in 2010 to about 20% in Jazz in 2050 and nearly 30% in Symphony in 2050. For clarity, "Jazz as an energy scenario, has a focus on energy equity with priority given to achieving individual access and affordability of energy through economic growth. Symphony as an energy scenario on the other hand has a focus on achieving environmental sustainability through internationally coordinated policies and practices".

In an attempt to offer a substantial solution to those challenges, to achieve the global aspiration of affordable, sustainable, and secure energy supply for all, this thesis has explored the potential valorization of by-product materials from oil palm to be an alternative and sustainable carbon sources of carbon-based nanomaterials synthesis and energy storage applications.

1.5 Carbon-based Supercapacitors

Energy storage systems are a central pillar for the success of the energy revolution. They contribute significantly to the integration of renewable energies and ensure a stable grid. The field of materials science and nanotechnology has witnessed a significant expansion in recent years across virtually all industrial sectors. This is due to number of key advantages that traditional manufacturing cannot offer. These include mass customization, geometrical complexity, etc. Historically, the first supercapacitor based on double layer mechanism was developed in 1957 by Becker H. I. (an engineer at General Electric) using a porous carbon electrode. Although the mechanism was not known at that time until 1966, however, it was believed that the energy was stored in the pores of the carbon material and it exhibited high capacitance value. Thus, for quite long time, carbon has garnered substantial attention due to its unique properties. It has been identified to comprise of different low-dimension allotropes including graphite, activated carbon, carbon nanotubes, and the C₆₀ family of buckyballs, polyaromatic molecules and graphene. This distinctive ability for existence under diverse forms (i.e. from powders to fibres, forms, fabrics and composite) denotes an excellent material for electrochemical applications, particularly for the storage of energy in supercapacitors. More so, the amphoteric nature of carbon also allows use of the rich electrochemical properties of this element from donor to acceptor state. In addition, carbon-based materials are eco-friendly. In the last couple of years, a huge attention has been directed on the application of renewable and sustainable carbons sources for electrode materials owing to their accessibility, transformation operational simplicity and inexpensiveness. Mostly, carbon-based materials are chemically stable in different solutions (from strongly acidic to basic) and able for performance in a wide range of temperatures. A well-established chemical and physical method of preparation and activation (as the case may be) allow fabricating materials with a developed surface area and a controlled distribution of pores that influence the electrode/electrolyte interface for electrochemical applications.

In recent times, carbon-based electrode materials for electrochemical capacitors (EC) have been successfully developed owing to the growing demand for an innovative type of accumulators of electrical energy with a high specific power and a long durability. The fundamental benefit of this storage appliance is the capacity of a high dynamic of charge transmission which is essential in the hybrid power sources for electrical vehicles, digital telecommunication systems, UPS for computers, etc. Another important benefits of the EC device is a likelihood of full discharge, and a short-circuit between the two electrodes is also not harmful [4].

In general, carbon nanomaterials (when used as electrode materials for supercapacitors) are very efficient energy conversion and storage devices owing to their good thermal, electrical, mechanical and chemical properties [5–7]. Therefore, they are generally believed to be the potential materials that could solve the global energy demand by 2050. Energy conversion and storage ability could be enhanced using materials with excellent morphological, electrical, optical and mechanical properties. In this study, carbon nanomaterials have been experimentally tested and

proven to possess all the requisite qualities needed to be used in this course. It should be emphasized that carbon-based materials were and still are the primary supplier of energy consumed by mankind. In sum, the amazing properties of these materials and greatest potentials towards greener and environment friendly synthesis methods and industrial scale production of carbon nanostructured materials is undoubtedly crucial and can therefore be glimpsed as the focal point of many researchers in science and technology in the 21st century.

1.6 Synthesis, Properties and Applications of carbon nanomaterials

The two foremost approaches employed for the fabrication of carbon nanomaterials like graphene and other graphene-related materials are generally known as the bottom-up and top-down approaches. The quality and the yield of the graphene produced via these methods have a contrasting stance which entirely relies on the how the processes are used. In bottom-up approach, for example the graphene or graphene related materials are mainly generated using uncomplicated carbon molecule such as alkanes (e.g. methane) and alkanols (e.g. ethanol). However, in top-down approach (as employed in this work), graphene or graphene-like material layers are extracted from graphite. The materials produced in this thesis, and their corresponding application, could be extended to address the rising demand of new types of clean and sustainable energy storage systems, especially for electric vehicles with minor or no exhaust emissions.

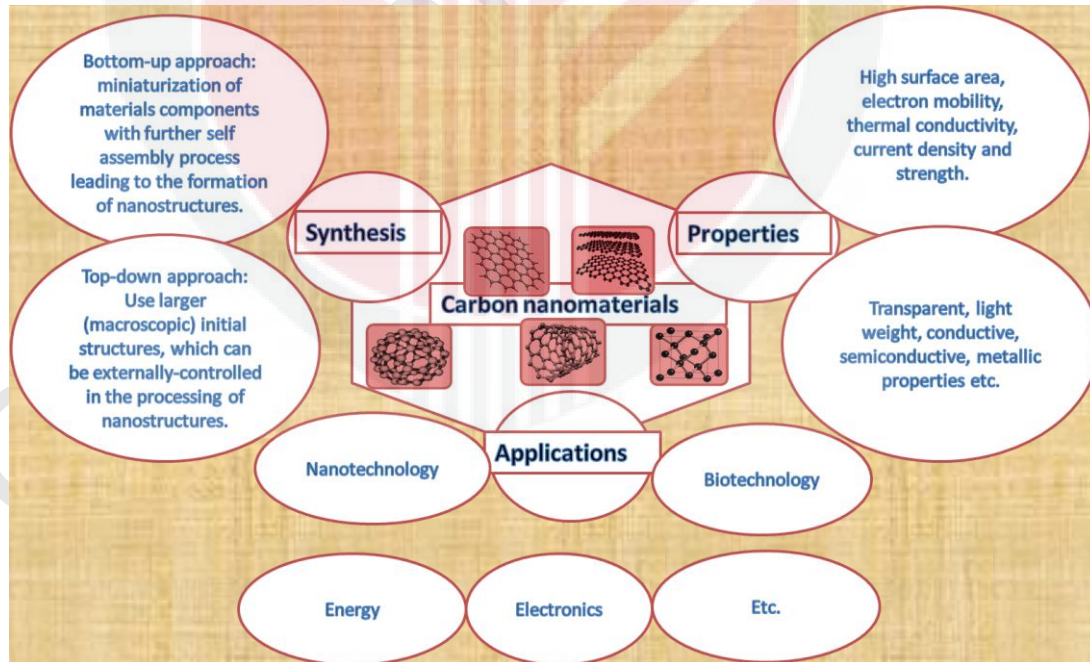


Figure 1.1 : Schematic illustration of synthesis, properties and applications of carbon nanomaterials

1.7 Problem Statement

There is an increasing demand for carbon-based materials, particularly activated carbon, graphene and other graphene-related and derived nanomaterials globally, such as in the automotive, aerospace, energy storage and consumer product industries, etc.

Secondly, Malaysia is one of the world's largest palm oil producers with over 950,000 ha of oil palm land cultivated. It is estimated that annual production of palm oil in Malaysia may reach about 15.4 million tonnes between 2016 to 2020. In essence, a lot of lignocellulosic biomass are generated every day from the oil palm industries. These include oil palm trunks (OPT), oil palm fronds (OPF), empty fruit bunches (EFB) and palm pressed fibres (PPF), palm shells and palm oil mill effluent palm (POME) etc.

It is alarming that the waste generated from oil palm activities is posing major disposal problems concerns.

Consequently, there is a need for technological, cost-effective, energy balance and environmental considerations in a balanced proportion in order to resolve utilization of oil palm wastes.

For this reason and coupled by the researcher's inquisitiveness to explore new knowledge, I felt it was essential to devise an experimental means of enhancing the utilization and production of profitable materials from those 'green wastes' generated by oil palm industries that can match the trendy from 'Wastes to wealth' mantra which is focused towards zero waste industry. This is believed to stimulate economic growth more especially in the developing countries and mitigate environmental degradation, and there by heading towards green technology, which is integrated into global paradigm shift towards sustainable development.

1.8 Objectives

Herein, four objectives were set and will follow the general objective as presented below:

1.8.1 General Objective

The main goal of this project is to produce carbon-based nanomaterials particularly graphene oxide (GO), reduced graphene oxide (rGO) and activated carbon from a low-cost natural carbon feedstock (exclusively oil palm waste precursors) using unique, improved and highly novel process (i.e. energy saving reactions) and explore their new potentials in thermal and electrochemical energy storage applications. This is targeted towards stimulating economic growth (i.e. creating wealth from waste) and

mitigating environmental degradation to corroborate with the global paradigm shift towards sustainable development.

1.8.2 Specific Objectives

1. To use a scalable and cost-effective feedstocks and synthetic pathway to produce carbon nanomaterials (graphene oxide, reduced graphene oxide and activated carbon) all from various oil palm waste precursors.
2. To characterize the as-synthesized materials using modern characterization techniques.
3. To assess the appropriate reaction conditions for the synthetic pathways and compare the physico-chemical properties of the as-prepared carbon-based nanomaterials.
4. To evaluate the electrochemical and thermal energy storage (ETES) potentials of the as-prepared materials.

1.9 Hypothesis

Natural precursors as a source of hydrocarbon (which are renewable and inexpensive), have the potentials to be the green alternative for laboratory and industrial scale production of carbon nanomaterials such as graphene, GO, rGO and activated carbon for ETES applications. The perception behind using natural precursors as a carbon feedstock for synthesizing the aforementioned materials is they are cheap, commonly available and have no likelihood of scarcity in the near future. Hence, this work will test the possibility of using oil palm wastes to synthesize carbon-nanostructured materials, particularly GO and rGO (as these materials have never been synthesized from those class of waste before), and AC and test their potential application as electrochemical and thermal energy storage materials

1.10 Scope of the Thesis

This project covered only a selective synthesis and characterization of some carbon nanomaterials (i.e. GO, rGO, and AC) from oil palm-derived byproduct materials. Thermal and electrochemical energy storage system of rGO and the activated carbon were also studied.

1.11 Thesis Layout

This thesis format is organized into six chapters. The first chapter (introduction) gives the general background on carbon nanomaterials; this was followed by a problem statement of this project, general and specific objectives of the work, hypothesis, scope and the thesis layout. The second chapter (Literature review) begins with extensive review of relevant available literature in the area. The first part of this chapter begins with the description of oil palm activities in Malaysia in relation to lignocellulosic waste

generation. The second part of the chapter talks about the potential valorization of the by-product materials from oil palm as an alternative carbon sources for carbon-based nanomaterials synthesis, current global energy crisis and the role of carbon-based materials as a useful materials to mitigate this problem. The last part of this chapter describes the commonest synthetic pathways to prepare carbon nanomaterials.

The third chapter focused mainly on the synthesis of graphene oxides and reduced graphene oxides from different oil palm precursors. The results of the synthetic pathways and the conditions were interpreted and discussed under this chapter. This chapter also reported the utility of the as-prepared reduced graphene oxides as carbon-electrode materials for supercapacitors application.

The fourth chapter describes mainly the synthesis and characterization of activated carbon from oil palm leaf and palm kernel shell precursors, and their use as electrode materials for electrochemical energy storage application.

The fifth chapter described the synthesis and application of new, inexpensive framework/ matrix based on n-nonadecane/biomass wastes-derived reduced graphene oxide-activated carbon composites for the preparation of shape-stabilized phase change material. The thermal energy storage potential of the composite material was studied under this chapter.

The sixth or the last chapter is dedicated to the general conclusion and recommendation. Summary of all the results was given here and future recommendations were suggested under this chapter.

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BIODATA OF STUDENT

The researcher was born and grew up in a small rural town called Babura, on the north edge of Jigawa State, Nigeria. At the age of five in 1989 he was enrolled into Babura Arewa Primary School (BAPS) and finished in 1995. From BAPS, he proceeded to a Government Secondary School for his junior school education in the same town. Upon completion of junior school education at the age of fourteen, he moved to Science Secondary School Kafin-Hausa also in Jigawa State, where he completed his secondary school education at the age of seventeen. After finishing secondary school, Nasir worked as a clerical officer at the Jigawa State Investment and Property Development Company Ltd., before he moved to Kano, where he gained a first degree in Chemistry from the famous Bayero University, Kano (a.k.a. BUK) in 2009. Upon successful completion of one year mandatory National Youth Service (NYSC) in 2010 at the Nigerian National Petroleum Corporation (NNPC), he was awarded a Scholarship by the Petroleum Technology Development Fund (PTDF) to study in the United Kingdom. Under this award (Scholarship), he earned a master's degree in Petroleum Geochemistry from the University of Newcastle upon Tyne in 2011. He started a lecturing career as assistant lecturer in January 2013 at the Department of Chemistry, Faculty of Science, Federal University Dutse, Nigeria. He continued his postgraduate at the PhD level in April 2016 at the Institute of Advanced Technology (ITMA), University Putra Malaysia, under the able supervision of Prof. Dr. Mohd Zobir Hussein. His research topic was ‘Valorization of oil palm by-product materials as a new sustainable carbon source for carbon-based nanomaterials synthesis and energy storage applications’. He's happily married to Rahmah binti Sabo Yusuf and blessed with a child named Muhammad Nasir II (a.k.a. Affan).

LIST OF PUBLICATIONS

Publications

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