**UNIVERSITI PUTRA MALAYSIA**

# *DIMENSIONAL EVOLUTION OF GRAPHENE-BASED NANOMATERIALS FOR SENSOR AND SUPERCAPACITOR APPLICATIONS*

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**FS 2018 27**

# DIMENSIONAL EVOLUTION OF GRAPHENE-BASED NANOMATERIALS FOR SENSOR AND SUPERCAPACITOR APPLICATIONS

By

**FOO CHUAN YI**

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

**February 2018**

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

# DIMENSIONAL EVOLUTION OF GRAPHENE-BASED NANOMATERIALS FOR SENSOR AND SUPERCAPACITOR APPLICATIONS

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The versatility of graphene and its derivatives from inventive synthesis method have evolved throughout the years, which provided avenues that precisely tune their structure and functionality for specific applications. Nevertheless, there are only several graphene-based products that have been successfully commercialized into the market. The main reason behind this is the lack of concrete performance metrics of graphene and its derivatives demonstrating their true value proposition in other segments. In this thesis, the investigation and justification of the evolution process of graphene derivatives were discussed in terms of graphene in different dimensions, which ultimately provide significant insights into diverse industrial applications.

In the first evolution stage, graphene with its natural 2D nanosheet structure was being employed as an "electronic blanket" in photoelectrochemical (PEC) sensing platform. The existence of 2D graphene (rGO) blanket in cadmium sulfide (CdS) modified carbon cloth (CC) electrode increased the photocurrent intensity by two orders of magnitude, compared to that of without graphene. 2D graphene blanket can also provide intimate integration between the nanoparticles and current collector substrate, thus contributing to a sensitive copper ion detector with a linear detection range of 0.1 to 40.0 μM and a detection limit of 0.05 μM.

In the second evolution stage, graphene was modified with nickel cobaltite (NCO) to produce a hierarchical 3D rGO/NCO nanostructure. Upon modification, the morphology of the graphene evolved from nanosheet into a petal-like nanostructure. This petal-like rGO/NCO nanostructure exhibits excellent supercapacitive performance (282.95 F $g^{-1}$), which is 1.5 times higher than that of pure NCO. Besides, intimate integration of NCO on the rGO nanosheet resulted in an efficient contact between the electrode/electrolyte interface, thus contributing to superior capacitance retention, which is 46% better than that of pure NCO.

In the third evolution stage, graphene was self-assembled and reinforced with polypyrrole (Ppy) into a free-standing 3D aerogel matrix. The self-agglomeration and oxidative polymerization of rGO and Ppy occurred synergistically in a controlled water bath environment, which resulted in an elastic and conductive compression sensor. The presence of flexible rGO nanosheets as an aerogel backbone provided a strong mechanical support which could be compressed more than 50% and recovered to its original structure in less than 5 seconds with minor mechanical deformations.

Lastly, in the final stage of evolution, graphene modified polylactic acid (PLA) was employed to develop a 3D printed electrode (3DE) using a commercial 3D printer. Graphene provided additional electrical conductivity properties to the insulating PLA matrix, which then proficiently fabricated into a supercapacitor and PEC sensor. They had a photocurrent response that exceeded expectations (~724.1 μA) and a lower detection limit (0.05 μM) than an ITO/FTO glass electrode.

In conclusion, the neoteric findings in this research provide a significant leap in functional graphene nanomaterial fabrications. Even though graphene has been extensively employed in laboratory research, the evolution and modification of graphene nanomaterials can eventually reveal its true potential in other industrial applications.

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

# EVOLUSI DIMENSI BAHAN NANO BERASASKAN GRAFIN DAN PENGGUNAANNYA DALAM BIDANG SENSOR DAN KAPASITOR

Oleh

**FOO CHUAN YI**

**Februari 2018**

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Kepelbagaian kegunaan grafin dan juga bahan-bahan berasaskan grafin yang dihasilkan daripada sintesis yang inovatif telah berkembang saban tahun di mana telah memberi laluan kepada pencorakan arkitektur dan fungsi bahan tersebut secara tepat bagi aplikasi yang tertentu. Namun begitu, sehingga kini hanya terdapat segelintir produk berasaskan grafin yang telah berjaya dikomersialkan di pasaran, dan punca utama di sebalik ini adalah kurangnya metrik prestasi grafin dan bahan-bahan berasaskan grafin ini dari segi keupayaan dalam menonjolkan kegunaan bahan-bahan tersebut di dalam cabang-cabang yang lain. Di dalam tesis ini, proses evolusi grafin dibincangkan dari aspek grafin di dalam dimensi yang berlainan di mana dapat memberikan pandangan untuk kegunaan industri yang pelbagai.

Pada peringkat perkembangan pertama, grafin dalam strukturnya yang asal iaitu lapisan nano 2D telah digunakan sebagai "selimut elektronik" dalam platform penderia fotoelektrokimia (PEC). Kewujudan selimut grafin 2D (rGO) di atas permukaan elektrod yang diperbuat dari fabrik karbon (carbon cloth) yang telah diubahsuai dengan kadmium sulfida telah meningkatkan keamatan fotoarus sebanyak dua kali ganda berbanding dengan keamatan tanpa grafin. Selimut grafin 2D juga boleh menyediakan integrasi mesra antara nano-partikel dan substrat pengumpulan arus, justeru menyumbang kepada pengesanan ion kuprum sensitif dengan julat pengesanan linear 0.1 ke 40.0 μM dan had pengesanan 0.05 μM.

Dalam peringkat perkembangan kedua, grafin telah diubahsuai dengan nikel kobaltit (NCO) untuk menghasilkan struktur hierarki 3D nano rGO/NCO. Setelah pengubahsuaian, morfologi grafin telah berkembang dari lapisan nano menjadi struktur nano mirip kelopak. rGO/NCO yang berstruktur nano mirip kelopak ini menunjukkan prestasi superkapasitif yang mengkagumkan (282.95 F $g^{-1}$), iaitu 1.5 kali lebih tinggi daripada NCO tulen. Selain itu, intergrasi mesra NCO pada nano-kepingan rGO menghasilkan interaksi yang cekap di antara permukaan elektrod/elektrolit.

Dalam peringkat perkembangan ketiga, grafin telah swa atur ke dalam matriks aerogel 3D berdiri sendiri yang diperkuatkan oleh polypyrrole (Ppy). Swa atur dan polimerisasi oksidatif rGO dan PPy berlaku secara sinergistik dalam persekitaran rendaman air yang terkawal, menghasilkan sensor mampatan yang elastik dan konduktif. Kehadiran nano-kepingan rGO yang fleksibel sebagai tulang belakang aerogel telah menyediakan sokongan mekanikal yang kuat di mana aerogel tersebut boleh dimampatkan lebih daripada 50% dan kembali ke struktur asalnya dalam tempoh kurang daripada 5 saat dengan deformasi mekanikal yang sangat kecil.

Akhir sekali, di peringkat perkembangan terakhir, grafin yang diubahsuai dengan asid polylactic (PLA) digunakan untuk menghasilkan elektrod cetakan 3D (3DE) mengunakan pencetak 3D komersial. Grafin memberikan tambahan sifat kekonduksian elektrik kepada matriks PLA yang bersifat penebat, yang kemudiannya dengan kemahiran yang tinggi telah difabrikasi ke dalam bentuk superkapasitor dan alat penderiaan PEC. Elektrod ini telah menunjukkan tindak balas fotoarus yang melebihi jangkaan (~724.1 μA) dan had pengesanan yang lebih rendah (0.05 μM) daripada electrod kaca ITO/FTO.

Kesimpulannya, penemuan neoterik dalam penyelidikan ini memberikan lonjakan penting dalam fabrikasi bahan nano grafin yang berfungsi. Walaupun grafin telah digunakan secara meluas dalam penyelidikan makmal, perkembangan dan pengubahsuaian bahan nano grafin akhirnya boleh mendedahkan potensi sebenarnya dalam aplikasi industri yang lain.

# ACKNOWLEDGEMENT

**"**For the ancestors who paved the path before me upon whose shoulders I stand". This is also dedicated to my beloved family and friends who have supported me on this fruitful journey. Thank you very much.

First of all, I would like to express my sincere gratitude to Assoc. Prof. Dr. Janet Lim Hong Ngee, my principle supervisor, who has the attitude of a genius who continually and convincingly conveyed a spirit of adventure in regards to research and an excitement in regards to teaching. Also, I would like to thank my Co-supervisory committee, Prof. Adzir bin Mahdi, Dr. Chong Kwok Feng and Dr. Hanif bin Wahid, for their constant encouragement and guidance throughout my entire PhD studies, without them this thesis would not have been made possible. For their unwavering support, I am truly thankful. I am also grateful to all the lecturers and officers in the Faculty of Science, Institute of Advanced Technology and also School of Graduate Study in particular, especially Mr. Zainal, Mr. Ali, Ms Roslinda and Ms. Sharifah for their support towards the successful completion of my research studies in UPM.

In addition, thank you to Dr. Huang Nay Ming from Xiamen University of Malaysia, who provided me the modified Hummer's method in graphene oxide (GO) synthesis, which is a precious material that plays an important role throughout my entire PhD research. I would like to also express my deep gratitude to Dr. Zhong-Tao Jiang and Dr. Mohammednoor from Murdoch University, thank you for providing me the opportunity to learn about the density functional theory (DFT), and also for your hospitality during my attachment in Western Australia. I am amazed with the working environment there and will definitely pay a visit again if I have another chance in the future. Besides, I would also like to thank the University of Nottingham for their kind assistance in FESEM analysis. Also, thank you MyPhD 2015 for the financial support throughout my PhD study. Hence, I would like to take this opportunity to express my heartfelt gratitude to the Ministry of Higher Education Malaysia for granting me the scholarship.

I would like to extend my appreciation to my friends, and research group members at UPM for their encouragement and moral support which made my studies here more enjoyable. Special thanks to Ms. Izwaharyani Ibrahim and Ms. Lau Siaw Cheng for bringing me a lot of entertainment and laughter throughout my research work in Lab 441. I want to thank my best friends Mr. Ng Leong Kee, Ms. Marilyn Yuen and Ms. Wong Jia Li for their love and understanding, who continuously supported me whenever I met troubles or difficulties during my research period. To them I say, "We meet to part, but more importantly, we part to meet."

Last but not least, I would like to thank my beloved family for their continuous support throughout my three years undergoing this PhD journey and for their love and encouragement that helped me to endure through my overwhelming and anxious moments during my studies. Thank you very much, I love you all.

I certify that a Thesis Examination Committee has met on 6 February 2018 to conduct the final examination of Foo Chuan Yi on his thesis entitled "Dimensional Evolution of Graphene- Based Nanomaterials for Sensor and Supercapacitor Applications" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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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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**Declaration by graduate student**

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# LIST OF ABBREVIATIONS

| | |
|---|---|
| 0D | Zero-dimensional |
| 1D | One-dimensional |
| 2D | Two-dimensional |
| 3D | Three-dimensional |
| 3DE | 3D-printed electrode |
| AACVD | Aerosol assists chemical vapour deposition |
| AFM | Atomic force microscope |
| BET | Brunauer-Emmett-Teller |
| CA | Chronoamperometry |
| CB | Conduction band |
| CC | Carbon cloth |
| CF | Carbon fibers |
| CTAB | Cationic cetyltrimethylammonium bromide |
| CV | Cyclic voltammetry |
| CVD | Chemical vapour deposition |
| DFT | Density functional modelling |
| DMAc | N,N-dimethylacetamide |
| DOP | Dioctyl phthalate |
| DSSC | Dye-sensitized solar cell |
| EDLC | Electrical double layer capacitor |
| EDX | Energy dispersive X-ray |
| EIS | Electrochemical impedance spectroscopy |
| FESEM | Field-Emission Scanning Electron Microscope |

| | |
|---|---|
| FT-IR | Fourier Transform Infrared |
| GCE | Glassy carbon electrode |
| GHA | Graphene hybrid hydrogel |
| GNR | Graphene nanoribbon |
| GO | Graphene oxide |
| GOQD | GO quantum dot |
| GQD | Graphene quantum dot |
| HGPAs | Holely graphene/polypyrrole hybrid aerogel |
| HM1H | 1-hexyl-3-methylimidazolium |
| HOPG | Highly ordered pyrolytic graphite |
| HRTEM | High-resolution transmission electron microscopy |
| ITO | Indium tin oxide |
| LED | Light-emitting diodes |
| LSV | Linear scan voltammetry |
| MWCNT | Multiwalled carbon nanotube |
| NapTS | Sodium p-toluenesulfonate |
| NCO | Nickel cobaltite |
| Ni-Fe-LDH | Nickel-iron layered double hydroxide |
| NMP | N-methyl-2-pyrrolidone |
| PCE | Power conversion efficiency |
| PDMS | Polydimethylsiloxane |
| PEC | Photoelectrochemical |
| PET | Polyethylene terephthalate |
| PGH | Polypyrrole/reduced graphene oxide hydrogel |
| PL | Photoluminescence |

| | |
|---|---|
| PLA | Polylactic acid |
| PMMA | Polymethyl methacrylate |
| Ppy | Polypyrrole |
| PVA | Polyvinyl alcohol |
| PVC | Polyvinyl chloride |
| PVDF | Polyvinylidene fluoride |
| PVP | Polyvinyl pyrrolidone |
| rGO | Reduced graphene oxide |
| rGOQD | RGO quantum dot |
| SDBS | Sodium dodecylbenzene sulfonate, |
| TEA | Triethanolamine |
| UV-Vis | Ultraviolet-visible |
| VB | Valance band |
| XPS | X-ray photoelectron spectroscopy |
| XRD | X-ray diffraction |

# CHAPTER 1

# INTRODUCTION

## 1.1 Graphene

The evolutions of both nanotechnology in the manufacturing industry and quality of fundamental materials are established drivers of disruptive innovations and developments, especially novel advanced materials. Research and developments in material sciences provided new growth prospects resulting from industrial, as well as commercial products and processes. Within the context of this trend, the emergence of new functional nanomaterials such as inorganic nanoparticles, quantum dots and carbon-based materials have resulted in nanotechnology advancements in various industries including electronics, catalysis, pharmaceuticals, biomedicine, energy conservation, cosmetics, medical and biological applications (K, 2015). Apart from these, graphene has attracted immense attention due to its potential as an extraordinary nanomaterial with abundant unique properties which will help to revolutionize fields such as functional composite materials, electronics, energy storage or solar conversion (Wang *et al*., 2012a).

Graphene is a one-atom-thick planar sheet of $sp^2$ hybridized carbon atoms which are tightly arranged in an extended two-dimensional (2D) honeycomb network. The first single layered graphene was mechanically exfoliated from its three-dimensional (3D) allotropes, graphite through a "Scotch tape" method in 2004 (Novoselov *et al*., 2004). Despite originating from graphite, single layer graphene possesses abundant delocalized electron clouds on the surface, which facilitate the electron mobility and provide low surface resistivity. These exceptional electrical advantages of graphene provide a significant improvement in electronic applications such as field effect transistors, conducting electrodes, integrated circuits and sensors (Avouris & Xia, 2012). In addition, graphene is also accredited as one of the strongest materials ever discovered, due to its highly ordered molecular structure and bonding arrangements between the $sp^2$ hybridized carbon atoms. Polymer matrix composites with graphene fillers have shown tremendous improvements in elastic properties, tensile strength, thermal stability and even electrical conductivity (Zhu *et al*., 2010).

## 1.2 Graphene derivatives

Graphene oxide (GO) is one of the graphene derivatives, whereby the graphene nanosheet is chemically modified by introducing oxygen functional groups within the hexagonal network. It first appeared as a by-product when a British chemist B. C. Brodie investigated the chemical reactivity of graphite flakes in 1895 (Brodie, 1859). After the discovery of graphene, GO was then being widely used as a precursor to synthesize graphene via a redox method. Unlike graphene, GO is an electrically insulating material due to the presence of oxygen functional groups which resulted in a disrupted $sp^2$ carbon network. However, the electrical conductivity can be easily recovered by restoring the $sp^2$ network through the reduction process and the product yield is called as reduced graphene oxide (rGO). This rGO comprises of graphene domains scattered with minimal oxygen functional groups and surface defects if compared to GO. Although rGO has lower electrical conductivity and mechanical strength in comparison to the pristine graphene nanosheets, the exceptional scalability of its synthesis has provide an alternative route in the preparation of new graphene-based products with the desired characteristics (Compton & Nguyen, 2010).

## 1.3 Synthesis and Applications of Graphene

Synthesis of graphite oxide through the oxidation of graphite flakes using different kinds of oxidants including concentrated sulfuric acid ($H_2SO_4$), phosphoric acid ($H_3PO_4$), nitric acid ($HNO_3$) and potassium permanganate ($KMnO_4$). These are the reagents for graphene-based materials (Chen *et al.*, 2013a; Marcano *et al.*, 2010; Zhang *et al.*, 2009). GO is also considered as a precursor for graphene synthesis through either thermal, chemical or microwave reduction process. It is mainly consisting of single layer of graphite oxide. Therefore, by removing the embedded oxygen functional groups, the $sp^2$ carbon network can be restored and the product formed (rGO) resembles graphene but contains residual oxygen and other structural defects.

Graphene produced through the reduction of GO has provided several important characteristics as it is prepared using low-cost graphite materials. By utilizing the benefits of its hydrophilic nature, GO can form a stable aqueous suspension. Even though the structural architectures of GO and rGO are more inferior compared to pristine graphene, these derivatives are still receiving close review in the research and development of graphene-based materials, especially the mass applications. This can provide a large scale and cost-effective method to synthesize graphene-based materials. The huge number of reported works and publications related to GO and rGO not only proved its superiority over other nanomaterials, but also progressively startling scientists' mind with new possibilities day by day.

Currently, rGO is being widely used in various research fields as a low-cost graphene alternative such as electrochemical capacitors, energy storage materials, catalysts, water purification and also electrode materials for photovoltaic devices. A flexible and free-standing cobalt oxide ($Co_3O_4$)/rGO hybrid paper was fabricated as an electrode material for electrochemical capacitor applications (Yuen *et al.*, 2012). It shows that rGO can provide a flexible and conductive platform for direct dispersion of nanoparticles. Also, rGO can provide an intimate integration between the active materials and the substrate which eventually enhances the capacitive performance and overall cycling stability of an electrochemical capacitor. Furthermore, the atomic-scaled thin rGO is highly transparent in the visible light spectrum. Alongside with the excellent optoelectronic properties, rGO has been widely investigated for transparent conducting materials as a potential alternative for indium tin oxide (ITO) in photovoltaic devices and light-emitting diodes (LED) applications (Loh *et al.*, 2010).

Nevertheless, graphene and its derivatives have shown significant importance in today's low-dimension research and eventually become a promising candidate for low-cost device fabrication in the electrical and electronic industry.

## 1.4 Nanomaterials

Functional nanomaterials such as colloidal semiconductor, metal and metal alloy nanoparticles has emerged as an important branch of material chemistry. Numerous techniques have been developed to control the chemical composition, morphology and surface chemistry of these functional nanomaterials. This include self-organization methods, template growth methods and colloidal syntheses methods (Yin & Talapin, 2013). Recently, the development of 3D nanostructure materials such as cadmium sulphide, cadmium telluride and zinc sulphide has been provided significant result in photovoltaic and energy storage devices. The aligned nanostructure arrays are regarded as a perfect architecture to facilitate the charge transport pathway as well as high photocurrent efficiency (Li *et al*., 2013d). Furthermore, their capability to provide high electrochemical activity and stability has been used for photoelectrochemical sensing platforms. On top of that, various metallic composite and spinel-type mixed oxides have been studied to provide beneficial effects in energy storage applications (Guene *et al*., 2007). For instance, ternary nickel cobalt oxide composites have attracted much attention due to the low-cost, naturally abundant and also possess high capacitive performances (Cheng *et al*., 2015).

## 1.5 Problem statements

Agglomeration and formation of nanoparticle clusters tend to occur during device fabrications. Nanoparticles such as titanium dioxide, $TiO_2$ (Chekli *et al.*, 2015), cadmium sulfide, CdS (Reyes-Esparza *et al.*, 2015) and zinc oxide, ZnO (Yuan *et al.*, 2016) have been widely used in electronic device fabrication. However, the high surface area to volume ratio of these nanoparticles may result in high surface energy. In order to minimize the surface energy, the nanoparticles create uncontrolled agglomerations due to the attractive van der Waals forces between particles. Consequently, the agglomeration of nanoparticles hinders their electrochemical characteristic which leads to difficulty of device fabrications and poor electronic performances due to the restricted electronic mobility, presence of charge recombination and limited transparency (Faure *et al.*, 2013). In view of this, a strategy using surfactant or coupling agent was used to bind nanoparticles onto the surface of growing species, thus controlling the growth rate and the degree of nanoparticles agglomeration (Sharma *et al.*, 2014). However, the presence of foreign species within the nanoparticle clusters requires additional modification and time-consuming post-treatment in order to remove the impurities or by-products.

In addition, the lack of flexibility platform for the electronic device fabrications has been given due attention recently. Several reports are available about the deposition of nanoparticles on a rigid conductive glass and metal foils substrate using different synthesis approaches. However, the use of rigid and expensive substrates is believed to restrict the flexibility and functionality, which ultimately hinders the development of low-cost electronic devices for their applications. In general, a flexible substrate has several advantages over a traditional rigid substrate, in terms of cost and processability. An enormous amount of effort has already been made by various research groups to design and fabricate various cost efficient electronic devices on flexible substrates such as carbon cloth and polyethylene terephthalate (PET) for practical applications (Fan *et al.*, 2009; J. Liu *et al.*, 2012; H. Su *et al.*, 2014).

Graphene represents a new inroad into low dimension research and offers new aspect for vast applications in electronic devices fabrication. It has the potential to address the agglomeration issue of nanoparticles due to the high surface area, optical transparency, reactive surface area and flexibility. Nevertheless, the applications of graphene are restricted to its 2D nanosheets form. Despite the great potential of graphene as a fascinating electronic material, the difficulties faced during material processing, insufficient reliable integrating techniques and their tendency to self-aggregate among graphene nanosheets remain as the major challenges. In contrast to the 2D nanosheets architecture, self-assembly of graphene materials can give rise to a unique 3D array, thus providing better aggregation resistance and high surface area while their excellent mechanical and electronic properties are maintained. Research into dimensional evolved graphene materials has been driven by their reinforced properties, which allow the materials to be conventionally applied in various electronic applications.

Although utilization of graphene with other nanomaterials shows promising results in promoting their overall physiochemical properties, the understanding in structural flexibility and their enhancement mechanism in electronic device are essential. Therefore, it is important to investigate the correlation between these characteristic with the dimensional evolution of graphene-nanomaterials changing from 2D to 3D architecture. It is also significant to develop a low-cost and versatile fabrication technique for graphene-based electrode materials.

## 1.6 Scope of research

In this research, we aim to prepare graphene-nanomaterials with different dimension to overcome the problems in electronic device fabrications. GO prepared from the modified Hummer's method will be tested to synthesize different graphene-nanomaterials such as CdS/rGO nanocomposites, rGO/NCO nanocomposites and polypyrrole (Ppy)/rGO nanocomposites, which will be utilized to produce visible-light-prompt photoelectrochemical sensor, high performance supercapacitor, flexible compression sensor and conductive electrode materials. The as-synthesized GO is less-toxic, economical and can be easily prepared using commercially available precursors. In addition, the presence of graphene also plays an important role in the morphology of the produced nanomaterials.

In order to test the workability of the as-synthesized graphene nanomaterials, a comparison study was carried out between the rGO modified nanocomposites and their counterpart. The performance of the as-fabricated electronic devices was also being evaluated by including parameter optimization, environmental study, performance comparison with commercial product and proof-of-concept study.

## 1.7 Research objectives

This research is to create an electrical device that relies on the combination of graphene properties. The main objective of this thesis is to investigate the dimensional evolution of graphene nanomaterials from 2D to 3D, and to use these graphene-nanomaterials in electronic devices fabrication. Special attention was devoted to overcome the current problems related to self-agglomeration of nanoparticles and to improve the overall performances. The specific objectives of the study are outlined as below:

i. To construct a 2D electronic blanket of graphene for enhancement in photoelectrochemical performance of CdS modified carbon cloth electrode.
ii. To develop a hierarchical 3D rGO/nickel cobaltite nanocomposites by modifying the 2D graphene with nickel cobaltite for high performance supercapacitor.
iii. To assemble a 3D graphene aerogel matrix reinforced with polypyrrole nanoparticles for a flexible compression sensor.
iv. To construct a 3D printed electrode by using graphene reinforced polylactic acid (PLA) *via* simplistic 3D printing technique.

## 1.8 Thesis outline

In Chapter 1, a brief introduction on graphene is given and its derivatives, problem statements and the main objective of the thesis. A comprehensive literature review on graphene-based materials, including the preparation methods and applications, is explained in Chapter 2.

Chapter 3 covers the experimental works used for fabricating a visible-light-prompt photoelectrochemical sensor, using 2D rGO nanosheets and CdS nanoparticles on flexible carbon cloth substrate. Aerosol assists chemical vapor deposition (AACVD) was employed to synthesized CdS nanoparticles on flexible carbon cloth substrate. A comparison study between the CdS/rGO and pure CdS modified carbon cloth substrate was carried out. The performance of rigid and flexible deposition substrate was also studied in this chapter.

In Chapter 4, the modification of 2D graphene with nickel cobaltite to obtain a hierarchical 3D rGO/NCO nanostructure *via* hydrothermal synthesis was conducted. Various tools such as FESEM, EDX, XPS and XRD were employed to evaluate the dimensional evolved rGO/NCO nanostructure. A comparison study between the as-fabricated rGO/NCO electrode and commercial KEMEX supercapacitor was carried out to prove the workability of rGO/NCO nanostructure for high performance supercapacitor applications.

In Chapter 5, the evolution of graphene from a hierarchical structure into a free-standing 3D architecture was developed. Graphene was synergistically assembled and reinforced with Ppy to produce Ppy/rGO aerogel matrix (PGA) via a simplistic water bath method. Density functional modeling (DFT) modelling was constructed to visualize the molecular interaction between Ppy and rGO. The physiochemical properties of as-synthesized PGA were also investigated using various characterization techniques.

In Chapter 6, a novel approach in electrochemical system was designed using a 3D-printed electrode (3DE) and electrochemically active nanomaterials. A 3DE was 3D printed using graphene reinforced PLA filaments. Surface modification of the 3DE for better workability was carried out in this chapter. In addition, the as-fabricated 3DE was developed into photoelectrochemical sensors and supercapacitor electrode materials.

Lastly, Chapter 7 contains the general conclusion and several future recommendations. The list of references cited in this thesis, appendices, biodata of students and a list of publications are given in post Chapter 7.

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