Synthesis and Characterization of a Two-Step Reduced Graphene Oxide for Antenna Applications in the Microwave Regime

by

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LIST OF ABBREVIATIONS

CST	Computer Software Technology
C/O	Carbon to Oxygen Ratio
GO	Graphene Oxide
HIrGO	Hydroiodic acid reduced Graphene Oxide
HIMrGO	Hydroiodic acid Microwave reduced Graphene Oxide
MGO	Microwaved Graphene Oxide
rGO	Reduced Graphene Oxide

LIST OF SYMBOLS

Ω	Ohm
ε_R	Efficiency (Antenna)
ε_T	Efficiency (Total)
H	Planck's Constant
^{o}C	Celsius
Σ	Conductivity
V	Magnitude of Electric Field
I	Magnitude of Current

Sintesis dan Pencirian dua-langkah *Graphene* Oksida Terturun Bagi Aplikasi Sebagai Antena Dalam Julat Gelombang Mikro

ABSTRAK

Tesis ini adalah berkaitan tentang pembangunan dua-langkah *graphene* oksida terturun yang akan digunakan sebagai unsur menyinar bagi sebuah antena tampal. Dua-langkah *graphene* oksida terturun ini di sediakan dari serbuk grafit yang dioksidakan menggunakan kaedah Tour. *Graphene* oksida yang terhasil daripada prosedur ini dibersihkan dan dineutralkan sebelum menjalani penyinaran gelombang mikro bagi memulakan proses penurunan, dimana kumpulan berfungsi yang terdapat oksigen dibuang dari bahan ini. Langkah kedua proses penurunan merangkumi penggunaan asid hidrohalik yang lebih dikenali sebagai asid hidroiodik bagi menurunkan graphene oksida dalam sebuah pasang refluk. Juzuk dan hasil akhir graphene oksida terturun dicirikan menggunakan pancaran medan mikroskopi elektron penskanan (FESEM), analisis termogravimetrik (TGA), ultrungu spektroskopi (UV-vis), spektroskopi inframerah transformasi fourier (FTIR), 4-titik prob, belauan sinar – X analisis (XRD) dan tenaga serakan sinar-X analisis (EDX). Hasil dari proses dua-langkah penurunan memberi produk *graphene* oksida terturun dengan jumlah konduktiviti, 4.8 x 10^2 Sm^{-1}. Kerintangan dan kekonduksian elecktrik yang diukur memberi keputusan yang lebih tinggi bagi kaedah dua-langkah dibandingkan dengan pengukuran yang diperoleh daripada bahan pelopornya. Tesis ini menerangkan kesan penyinaran gelombang mikro dan penurunan kimia terhadap darjah penurunan dan struktur keseluruhan *graphene* oksida terturun. Hasil akhir akan digunakan sebagai unsur menyinar dalam antena tampal mikrojalur bagi menentukan prestasi antenna dalam kawasan gelombang mikro. Teknologi simulasi computer (CST), perisian *Microwave Studio Suite* digunakan untuk menjana dua model antenna mikrojalur; satu memodelkan antenna yang menggunakan *graphene* sebagai unsur menyinar dan satu memodelkan kuprum sebagai unsur menyinar. Keputusan dari simulasi dibandingkan dengan pengujian hidup nyata yang dijalankan dengan menghasilkan prototaip antena dalam kebuk tak bergema. Prototaip ini dicirikan berdasarkan prestasi antena melalui pengukuran pantulan koefisien, pertambahan antenna dan corak radiasi dengan menggunakan ENA *Vector* analisis (*Vector Analyzer*). Berdasarkan keputusan ujian, *graphene* oksida antenna yang dihasilkan dari kaedah dua-langkah penurunan beroperasi pada lebar jalur antara 1.3 GHz to 11 GHz, nilai q perolehan adalah antara 4.2 dB to 15.2 dB, dan mempunyai corak radiasi yang dikehendaki. .Keputusan bagi prestasi antenna mengesahkan bahawa *graphene* oksida terturun mampu berfungsi sebagai unsur menyinar dalam julat gelombang mikro.

Synthesis and Characterization of a Two-Step Reduced Graphene Oxide for Antenna Applications in the Microwave Regime

ABSTRACT

This thesis presents the development of a two-step reduced graphene oxide that is to be implemented as the radiating element for a patch antenna. The two-step reduced graphene oxide is prepared with graphite powder and is oxidized using the Tour's method. The graphene oxide produced from this procedure is cleaned and neutralized before microwave irradiation procedure is carried out to begin the reduction process, by which oxygen containing functional groups are removed from the material. The second step of the reduction process consists of using the strongest hydrohalic acid, hydroiodic acid, to reduce graphene oxide in a reflux setup. The two-step reduced graphene oxide and its precursors are characterized using Field Emission Scanning Electron Microscopy (FESEM), Thermogravimetric analyzer (TGA), Ultraviolet-Visible Spectroscopy (UV-Vis), Fourier Transform Infrared Spectroscopy (FTIR), 4-Point Probe Conductivity Test, X-Ray Diffraction analyzer (XRD), and Energy Dispersive X-Ray analyzer (EDX). The results of the two-step reduction process yielded a reduced graphene oxide with a net conductivity measured at 4.8×10^2 Sm^{-1}. The measured resistivity and electrical conductivity results were higher in the two-stepped method than any measurement yielded from its precursor materials. This thesis explains the effects that microwave irradiation and chemical reduction has on the degree of reduction and the overall structure of reduced graphene oxide. The two-step reduced graphene oxide was used as the radiating element in a microstrip patch antenna to determine the antenna performance in the microwave region. Computer Simulation Technology (CST) Microwave Studio Suite software was used to generate two microstrip antenna models; one that models an antenna that uses graphene as the radiating element and another that models copper as the radiating material. The results from the simulation was compared to real life testing carried out by a fabricated antenna prototype inside an anechoic chamber. The prototype was characterized for its antenna performance through the measurement of the reflection coefficient, antenna gain, and the radiation pattern using an ENA Vector Analyzer. It was found that the two-step reduced graphene oxide antenna operated on a bandwidth between 1.3 GHz to 11 GHz, measured q gain between 4.2 dB to 15.2 dB, and contained a favorable radiation pattern. The results of the antenna performance confirms that the reduced graphene oxide was capable of functioning as a radiating element in the microwave range and could be used for further antenna applications.

CHAPTER 1

INTRODUCTION

1.1 Background

Graphene, the carbon network composed of hybridized sp^2 orbitals, is gaining momentum as the next generation material for tomorrow's applications. In the same vein silicon served during the rise of the computer chip, graphene's multifaceted uses and favorable material properties make it an ideal candidate for various technological advances and applications. Already graphene is being sought after for its applicable uses in biosensors (Huang et al., 2011b), sensors (Reed et al., 2012), antennas (Thampy et al., 2015), solar cells (Kahng et al., 2014), and batteries (Zhang et al., 2016). However, due to the high cost and technical difficulty required in the fabrication process, the use of graphene is limited to mostly research purposes.

Recently, graphene has been of special interest in advanced communication technology. In industries requiring continuous miniaturization of technological parts to match current technological demands, such as the health care industry, advanced sensors and antennas research are turning to graphene to meet these demands. The use of graphene for antennas offers the benefits of providing increasing miniaturization on a nanoscale, efficient dynamic tuning, monolithic integration with graphene RF nanoelectronics, and improved flexibility (Perruisseau-Carrier, 2012). However, despite the increased interest in studying the various applications it possesses, graphene is limited by its fabrication methodology.

As a result, special interests needs to be directed toward the many different fabrication methods available. Through better understanding of the material, its' derivatives, and methods of manufacturing, can research in graphene synthesis continue to drive toward facile and greener methodologies. Therefore, in order to further the study of graphene and its applications, it is necessary to first understand the manufacturing process required to fabricate graphene.

The large scale synthesis of graphene is still a limitation that needs to be addressed. Currently, the four main methods of fabricating graphene consists of chemical vapor deposition (CVD), epitaxial growth, micromechanical exfoliation, and the usage of colloidal suspensions (Park and Ruoff, 2009). Despite the number of methods available for graphene synthesis, each of these methods all suffer from one main drawback, high cost of production. The costs of manufacturing include, but are not limited to, the use of harsh chemicals and expensive equipment, make manufacturing pristine graphene a costly, tedious, and time consuming procedure. Of course, with ongoing research, scientists are not very far away from conceiving an easier route to produce graphene.

In order to develop a safer and efficient route for graphene synthesis, it is necessary to study all the chemical derivatives of graphene. Following a bottom-up approach, by studying the precursor materials and various procedures that lead from graphite to graphene oxide to graphene-like reduced graphene oxide, then perhaps it is possible to discover an alternate pathway to manufacture applicable graphene on an industrial scale.

Reduced Graphene oxide (rGO), a stable derivative of graphene that many researchers are turning to in substitute of pristine graphene, contains various mechanical and material properties, including favorable electrical conductivity, which makes it an ideal candidate for applications where pristine graphene would be ideally utilized. While graphene is defined as having a sp^2 honey comb lattice structure, reduced graphene oxide's structure is markedly different. Due to the atomic arrangement and various functional groups, the properties are comparable to graphene while being easier to produce on a large scale.

However, the chemical process used to produce graphene oxide from graphite renders it electrically inert. For applications that require high electrical conductivity, graphene oxide is not a viable material, and requires further treatment before it can be applicable.

Although the properties of graphene are vastly superior to graphene oxide, there are numerous methods available to use to return the electrical conductive properties to graphene oxide. This research focuses on the potential of utilizing two previously carried out procedures to reduce graphene oxide. The first procedure consists of chemically reducing graphene oxide with the reducing agent hydroiodic acid. The second procedure consists of exposing graphene oxide to microwave irradiation until partial or full reduction occurs. Lastly, the two procedures will be used together to form a two-stepped reduced graphene oxide to determine the characteristic effects and benefits of such a procedure.

In order to determine the applicability of such a procedure, the final compound will be used in conjuncture with a microstrip patch antenna. Graphene has recently attracted attention toward antenna applications partially due to is unique chemical, thermal, mechanical, electronic, and optical properties. The rGO, manufactured through this procedure, will be used as the radiating element. The antenna performance will be examined and compared to simulation data.

1.2 Problem Statement

In order to meet the demand of various industries (healthcare, technological, etc.) that require the use of graphene, it is necessary to develop alternative methods for large scale production that are safe, cost-efficient, and economically viable. In communications, the use of graphene for antennas and other electromagnetic passives could bring significant benefit such as extreme miniaturization, monolithic integration with graphene RF nanoelectronics, efficient dynamic tuning, and even transparency and mechanical flexibility (Perruisseau-Carrier, 2012). Current single step reduction methods (e.g. solvothermal methods) are extremely costly. They require the use of expensive machinery, time, and a skilled laborer to ensure the end product is of high quality. Even then, there is the problem that single step methods produce impure quality graphene. Currently reduced graphene oxide, a derivative of the highly regarded graphene, is such a material that can serve multiple applications within sensor technologies and advanced communications.

However, the fabrication process is tedious and sometimes requires the use of hazardous materials that are both dangerous to the individual and the environment. It is the goal to synthesize graphene oxide using processes that conserve reactants, time, and energy (Viana et al., 2015). The most widely used reducing agent, hydrazine hydrate, is highly carcinogenic and volatile. Incorrect usage of hydrazine hydrate can lead to laboratory accidents and permanent health damages. In addition, the use of hydrazine hydrate is not suitable for applications where flexibility is necessary due to disintegration and stiffening of graphene oxide films (Pei et al., 2010).

Another method to reduce graphene oxide involves the use of microwave irradiation. The main advantage of microwave irradiation over other conventional heating methods (e.g. solvothermal) is that the material is heated uniformly and rapidly. Microwave irradiation provides significant enhancement in the transfer of energy directly to the reactants, causing the internal temperature to rise instantaneously (Pokharel et al., 2014).

In order to meet the demands for a reduced graphene oxide that is suitable for antenna applications in the microwave regime, a two-step reduced graphene oxide is synthesized and characterized. In order to improve the production of graphene based functional materials via chemical reduction, the material properties were determined through characterization testing. The electrical conductivity measurement of the two-step reduced graphene oxide was determined due to its heavy reliance in antenna applications (e.g. radiating element). In this study, a microstrip patch antenna is fabricated from a two-step reduced graphene oxide made using microwave irradiation followed by chemical reduction using hydroiodic acid. The antenna's performance is tested against a simulation of copper and of itself using Computer Simulation Software (CST) Microwave Suite before undergoing analysis to determine the antenna performance with respect to gain, radiation pattern, and directivity.

1.3 Objectives of Research

The general objective of this study was to synthesize and characterize a reduced graphene oxide material that had been treated with a chemical and microwave process. As well as test this material's capabilities as a radiating element. The specific objectives are as follows:

1. To synthesize and characterize a two-step process of rGO prepared using microwave irradiation technique followed by hydroiodic acid reduction.

2. To determine the electrical conductivity characteristics of each stage of the GO prepared from the two-step process.
3. To model and simulate a microstrip patch antenna made of rGO (prepared from the two-step process) using Computer Simulation Technology (CST) Software Version 2.1.
4. To fabricate and test a microstrip patch antenna made of rGO prepared from the two-step process.

1.4 Scope of Research

In this study, graphene oxide will be used in lieu of pristine graphene for the sole use of creating a radiating element in a microstrip antenna. Graphene oxide will be reduced using a two-step process that includes microwave expansion followed by chemical reduction. The resulting material will be used to fabricate the radiating element in a microstrip antenna to be analyzed within the microwave spectrum. The results will be compared to simulation results generated with CST and contrasted against another common materials used as radiating elements in antennas, copper.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Antennas are a crucial component in many communication systems. An antenna is a device that can transform an RF signal, traveling on a conductor, in an electromagnetic wave in free space (Carr, 2001). Due to the advancement in materials and communications technologies, there is no limit to the number of designs that can be made to improve an antenna's performance. Recently, the rediscovery of graphene has brought about a renewed interest in antenna design.

2.2 Graphene

Graphene is an allotrope of the element carbon. Pristine graphene is composed of atomically thick conjugated sp^2 carbon atoms arranged in a two dimensional honeycomb lattice (Stankovich et al., 2006). Recent excitement over graphene's applications began when it was determined that graphene contained an unusual linear dispersion at the π-band at the Fermi level, which parallels to its exotic electrical properties (Batzill, 2012). Its high interest stems from is wide range of potential applications given its excellent electrical, mechanical, and thermal properties. It is reported that graphene is 100 times stronger than steel. In addition, graphene holds properties that are reported to include its Young's modulus (1,000 GPa), fracture strength (125 GPa), thermal conductivity ~ ((4.84

± 0.44) x 10^3 to (5.30 ±0.48) x 10^3 W m^{-1} K^{-1}) (Balandin et al., 2008) and specific surface area (2,630 m^2 g^{-1}) (Zhu et al., 2010a). Due to its high electrical conductivity, graphene is being sought after for uses in battery, composites, semiconductor, and other electronics based industries.

Through the study of theoretical predictions, it is understood that graphene contains an extremely high carrier mobility and an ambipolar field effect. In addition, it has been determined that graphene sheets prepared from graphene oxide have good field effect transmitter (FET) properties (Rao et al., 2010).

The knowledge of Graphene has been known since 1940, but it was not until 2004 when Konstantin Novoselov and Andre Geim first isolated graphene onto SiO_2 substrate using micromechanical cleavage, which would late be coined the “Scotch Tape” method (Novoselov et al., 2004). Previously, the obtainment of graphene was a very expensive and complex procedure requiring chemical vapor deposition. This reason alone limited the amount of research being done at the time. However, in 2012, a new technique was discovered that greatly reduced the cost and time necessary to fabricate the material. As a result, the development of graphene based technologies and research began to take shape. However, despite the many applications that are being researched for graphene, a fundamental problem that prohibits the mass manufacturing of graphene is its high cost of fabrication.

Currently, production of graphene is limited depending on the process being used. Graphene requires a tedious amount of work and high costs to produce small scale amounts. As a result, producing graphene on a commercial scale is not yet a reality. Nevertheless, with the work being done today, and through ongoing research, the fabrication methods continue to make strides towards a safe and reliable protocol for mass production of graphene (Kuila et al., 2012).

2.2.1 Fabrication Methods

Graphene is fabricated from mainly one of four procedures. Although there are several methods to produce graphene, the primary four include chemical vapor deposition (CVD), micromechanical exfoliation of graphite, epitaxial growth, and colloidal suspensions (Alam et al., 2016). Additional methods include nanotube slicing, carbon dioxide reduction, spin coating, microwave-assisted oxidation, intercalation, and laser treatments.

2.2.1.1 Chemical Vapor Deposition

CVD, or chemical vapor deposition, is a procedure that can be used to produce graphene. In a typical setup, a predetermined substrate is placed inside a reaction chamber at a specific pressure and temperature. Afterwards, gases are introduced into the reaction chamber and a small film develops on the material substrate's surface. Recently, Chen et al. (2014) began experimenting using CVD to deposit graphene onto nickel foam. However, the typical substrates used are copper and silica. After completion, the remaining gases are pumped from the chamber. The quality and size of the graphene film depends on the control of the volume, temperature, and pressure during the specific time duration. Despite this method requiring careful monitoring, it is a promising method for producing large scale amounts of high quality graphene. However, since the use of CVD requires that graphene be synthesize on metallic surfaces (e.g. Nickel or Copper), the then finalized material requires transfer to a Si/SiO_2 or polymer substrate (Kaplas et al., 2012). As a result, the surface of graphene contains defects and irregularities. In order to combat this, either the transferring step needs to be removed or a final surface treatment is added.

McEvoy et al (2013) used plasma treatments to functional the surface of graphene produced by chemical vapor deposition. The incorporation of nitrogen into the graphene lattice proved effective in modifying the surface for further functionalization and applications.

CVD coatings display imperviousness, fine grained, and increased hardness in comparison to other fabrication processes. The main drawbacks to using this method is the usage of harsh precursor chemicals. In order to initiate the reaction, volatile chemicals need to be used to react with the substrate. Nevertheless, afterwards the chemical gas is pumped out of the system and becomes relatively safe to handle. In addition, the CVD method is relatively expensive and the final product may suffer from surface irregularities that could reduce the overall electrical conductivity of the final product (Nekahi et al., 2014).

Lastly, problems arise when graphene is separated from the substrate material. Fine tuning the number of layers that grow on a CVD surface is another challenge researcher's face (Zhu et al., 2010b). Multilayer graphene is of particular interest in microwave applications since the electrical conductivity increases with the number of layers (Wu et al., 2014). Currently, this process is not perfect and requires further research in order to understand the binding that occurs at the interface between the substrate's surface and the graphene's surface. For research purposes, CVD is more than capable of studying simulated data and new models. Factor in the cost of expensive equipment, CVD is a less than ideal methodology for the mass production of graphene based antennas.

2.2.1.2 Micromechanical Exfoliation

Micromechanical exfoliation, also known as micromechanical cleavage, is a flawed yet easy to use method to produce films of several layers of graphene. Micromechanical cleavage allows the extraction of several layers of graphene from graphite. In essence, it is a simple peeling procedure where highly ordered pyrolytic graphite sheet (HOPG) is dry etched in oxygen plasma and, subsequently, stuck onto a photoresist sheet before being peeled off layer by layer using scotch tape (Singh et al., 2011). The aforementioned method roughly entails the famous scotch tape method for micromechanically exfoliated graphene flakes. However, commercial scale production using this process is infeasible and is used mainly for research or proof of concept purposes.

Chemical oxidation of graphite powder, and the resulting exfoliated graphene oxide, commonly suffers from structural defects that are typically foreign in pristine graphene monolayers. As such, these samples have adherently poor electronic properties that would make a poor for application in semiconductors and electronic properties. Even with the use of thermal annealing or chemical reduction, it is not possible to fully restore the carbon crystal lattice to that of pure graphene. In the end, mechanical exfoliation is only useful when studying graphene's structure (Singh et al., 2011).

2.2.1.3 Other Methods

One method used to synthesize graphene is laser ablation. Focused lasers are used to remove particles from the surface of a material. In principle, the application of laser ablation to graphite flakes can produce graphene if the laser is tuned to the correct laser