UNIVERSITI PUTRA MALAYSIA

INVESTIGATION OF DIFFERENT GRAPHENE OXIDES COATED ON TAPERED OPTICAL FIBER SENSOR FOR AQUEOUS ETHANOL DETECTION

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By

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Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master of Science

November 2016

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

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November 2016

Chair: Ahmad Shukri Muhammad Noor, PhD
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Sensing devices, especially optical sensors, are becoming increasingly popular in the last decade. Typically, evanescent-field optical fiber sensors utilize tapered or etched fiber structure to enhance the evanescent filed interaction with the sensing medium. This makes the fiber fragile and difficult to handle. This research focuses on designing and analysing a multimode fiber to be used as a sensor for the detection of aqueous ethanol. A nanostructured sensing layer is applied to further enhance the sensitivity of the sensor. This study investigates the use of tapered fiber tip sensor coated with different types of Graphene oxide (GO) which is GO (unsonicated), GO (sonicated) and GO 1-Ethyl-3-[3-dimethylaminopropyl] carbodiimide hydrochloride (EDC+NHS). The effect of nanostructured layers each types of (GO) are investigated towards the performance improvement of the optical sensor. GO nanostructures have huge surface area that enhances the sensor-analyte interaction and thus, improves the sensing performance. GO nanostructured materials also react chemically with ethanol molecules, resulting in an increase in the sensitivity of the sensor. Optimization of the each types of GO layer and the tapering parameters are done and the sensing capability of the device is tested using different concentrations of ethanol in water which are 20%, 40, 60%, 80% and 100% of ethanol concentration. The sensor demonstrates high sensitivity to aqueous ethanol when interrogated in the visible region using a spectrometer and light source. The GO coated tapered fiber sensor with 50 µm is compared with other works and its demonstrated high sensitivity in intensity and absorbance with a response and recovery time of approximately 25 and 35 seconds respectively.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

SIASATAN GRAPHENE OXIDE YANG BERBEZA DISALUT PADA SENSOR TIRUS FIBER OPTIK UNTUK MENGESAN EHTANOL DIDALAM AIR

Oleh

MOHD ANWARUL ARIF B MD ROSLI

November 2016

Pengerusi: Ahmad Shukri Muhammad Noor, PhD
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Peranti sensor, terutamanya sensor optik, semakin popular pada dekad yang lalu. Biasanya, *evanescent-field* sensor fiber optik menggunakan struktur fiber tirus atau terukir untuk meningkatkan interaksi *evanescent-filed* dengan sensor sederhana. Ini menjadikan gentian optik rapuh dan sukar untuk dikendali. Kajian ini memberi tumpuan kepada merekabentuk dan menganalisa gentian pelbagai mod untuk digunakan sebagai sensor bagi mengesan etanol di dalam perantara akueus. Satu lapisan *nanostructured* sensor digunakan untuk meningkatkan lagi kepekaan sensor. Kajian ini untuk mengkaji penggunaan sensor di hujung fiber tirus disalut dengan perlbagai jenis Graphene oxide (GO) seperti GO (tidak disonikasi), GO (disonikasi) dan GO 1-Ethyl-3- [3-dimethylaminopropyl] carbodiimide hidroklorida (EDC+NHS). Kesan lapisan berstruktur nano untuk setiap grafin oksida (GO) dikaji ke arah peningkatan prestasi sensor gentian optik. Struktur nano tersebut mempunyai luas permukaan yang besar yang meningkatkan interaksi antara permukaan sesnsor dan analit justeru, meningkatkan prestasi sensor. Bahan-bahan berstruktur nano tersebut juga berintreraksi secara kimia dengan molekul etanol dan meningkatkan kepekaan sensor. Optimasi lapisan untuk setiap jenis GO dan parameter tirus dilakukan dan keupayaan alat sensor diuji menggunakan kepekatan etanol yang berbeza didalam air yang mane 20%, 40, 60%, 80% and 100% kepekatan ethanol. Sensor ini menunjukkan kepekaan yang tinggi jepada etanol dalam air apabila berinteraksi di dalam rantau yg boleh dilihat dengan menggunakan a spektrometer dan sumber cahaya. Sensor gentian pelbagai mod bersalut GO dengan saiz ukur lilit 50 μm dibandingkan dengan projek yang lain telah menunjukkan kepekaan yang tinggi dalam keamatan dan serapan serta masa tindak balas dan pemulihan kira-kira 25 dan 35 saat masing-masing.

ACKNOWLEDGEMENTS

I would like to express my special appreciation and million thanks to my supervisor **Associate Prof. Dr. Ahmad Shukri b. Muhammad Noor** because of his dedication to help me with his advices and guidance throughout the entire project. His patience to explain many times without complaining is the best experience I have ever had and the knowledge I received is priceless. Without his supervision and constant help this dissertation would not have been possible.

A special gratitude as well to my co-supervisor, Prof. Dr. Janet Lim Hong Ngee for making my Master program a truly profitable experience. Her helpful suggestions and advices on various aspects of my research work have certainly been very constructive. Thank you also to **Dr. Punitavathi Thiruvakarasu**, a former Phd student for her helps in this project even though she already busy with her project. Without her help, this project will take more time to finish.

Also special thanks to my family. Words cannot express how grateful I am to my parents Md Rosli B Hj Malek and Foziah Bte shamsudin for all the sacrifices that they had made and also because of their understanding and toleration towards my study. Thanks also to all my colleagues and friends at the Photonic Lab and WIPNET for their help, providing a friendly and inspiring environment for conducting research and supported me all the way to strive towards my goal to finish up this project.

I would also like to thank Universiti Putra Malaysia for giving me the opportunity and financial assistance in completing this master. Last but not least thank you very much to all the people who are involved directly or indirectly in the completion of this project. Without them this project cannot be completed. Thank you.

I certify that a Thesis Examination Committee has met on 29 November 2016 to conduct the final examination of Mohd Anwarul Arif bin Md Rosli on his thesis entitled "Investigation of Different Graphene Oxides Coated on Tapered Optical Fiber Sensor for Aqueous Ethanol Detection" 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 Master of Science.

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Name of Chairman of Supervisory Committee:	Associate Professor Dr. Ahmad Shukri B Muhammad Noor

Signature:	
Name of Member of Supervisory Committee:	Associate Professor Dr.Janet Lim Hong Ngee

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LIST OF ABBREVIATONS

GO	Graphene Oxide
EDC/EDAC	1-Ethyl-3-(3-dimethylaminopropyl) carbodiimide
NHS	N-hydroxysuccinimide
TIR	Total Internal Reflection
RI	Refractive Index
MMF	Multi-mode Fiber
FBG	Fiber Bragg Grating
SMF	Single Mode Fiber
LPG	Long Period Gratings
CH_3CH_2OH	Ethanol
CNT	Carbon Nanotube
ZnO	Zinc oxide
WO_3	Tungsten Trioxide
SPR	Surface Plasmon Resonance
OSA	Optical Spectrum Analyser
UV	Ultraviolet
VIS	Visible
VOC	Volatile Organic Compound
GUI	Graphic User Interface
H_2O	Water
CO	Carbon Monoxide
PaH	Polycyclic aromatic hydrocarbons
H_2	Hydrogen
O_2	Oxygen

CHAPTER 1

INTRODUCTION

1.1 Overview

Optical fibers are strands of highly transparent glass or plastic that has the capability of transmitting light signals throughout using the principles of Total Internal Reflection (TIR) [1]. Optical fibers are commonly used in telecommunications [2][3], lighting [4], medicine [5], imaging [6], optical inspection [7] and optical sensing [8][9]. Compared to traditional electrical-based sensors, optical fiber sensors provide benefits such as high sensitivity [10], invulnerability to electromagnetic interference as well as being passive and simple to fabricate [11][12]. By modifying the surface or structure of an optical fiber cable, it can be made sensitive to external environments [13]. Among the popular optical fiber sensors are fiber Bragg gratings (FBG) and long period gratings (LPG) [14][15], which are popular in physical sensing applications. Optical fiber sensors are also gaining traction as an alternative in the field of chemical and biomedical sensing [16].

Optical fiber sensors are gaining popularity since it immune to electromagnetic interference, fast response time and miniature in size, therefore it is suitable to be used in hazardous and tight spaces [17]. The operating principle of optical fiber sensors lie in the geometry of the optical fiber itself. Optical fibers are made of a polymer or glass core surrounded by a layer of cladding material. The difference in density between these two structures enables the light propagation in a optical fiber which is in accordance with the principle of total internal reflection (TIR) [18] as mentioned earlier.

Refractive index [19] variation is also a way to generate perturbation other than reducing diameter size of the fiber [16]. Although optical fibers are mainly used as a sensors for physical changes, multimode fiber (MMF) can also be used to sense refractive index changes. However this commonly involves tapered MMFs and coating it with other materials to enhance the evanescent waves penetration to the surrounding area [20]. In this process the waist size of the MMF is reduced, to a point which the cladding becomes a new core, so that the sensing area can be immersed in the sample and the sample can act as the new cladding to the fiber. The sensitivity of the sensor increase as cladding thickness decrease [21].

Nowadays, a number of studies related to tapered optical fiber have been reported. Sensors based on tapered optical fibers have been intensively used for temperature [22], strain [23] and biomedical [24] applications due to its simplicity and high sensitivity [10]. Common parameter like refractive index (RI) can be effectively measured by tapered fiber sensors [25]. Moreover, the parameter of interest can be monitor in real time. Therefore, this has led to tapered fiber becoming an increasingly popular sensor of choice for practical studies.

Evanescent field absorbance optical fiber sensors have become increasingly popular for remote [26] and distributed sensing applications [15]. Evanescent based sensors [27][17] have been broadly used for chemical [28] and biological sensing [16]. The evanescent wave is formed when light propagating through the core of the fiber via TIR caused by a higher index medium bordered by a lower index medium. Although TIR conditions are met, some of the light still crosses the interface and propagates along the border of the two medium. This light, called the evanescent wave propagates parallel to the interface [29]. If the outer medium is an absorbing material, the evanescent wave intensity is attenuated, giving rise to a reduction in the power propagating in the denser medium [30]. Among many types of optical fiber sensors, the widely used ones are intensity modulated sensors because they are simple and easy to realize [19].

The unique optical and electronic properties of graphene attract tremendous interest. Graphene has high optical transparency and mobility [31]. In addition, graphene characteristics include flexibility, robustness and environmental stability [32]. The growth of graphene applications in photonics and optoelectronics is reflected by several recent results, ranging from solar cells and light-emitting devices to touch screens, photodetectors and ultrafast lasers.

Graphene oxide (GO) comprises of carbon merged with functional oxide groups [33]. GO is commonly used for the enhancement of electrochemical sensors and it is a remarkable material due to its capability of detecting different types of chemicals such as ethanol [34]. In addition, GO also high thermal conductivity and large surface-to-volume ratio as compared to other semi-conductors are some of the unique optical and physical properties of GO [35]. Since graphene and graphene oxide (GO) shares most of its beneficial optical and electronic properties, tapered MMF fiber coated with GO is experimentally exploited to study its sensing performance [32].

1-Ethyl-3-(3-dimethylaminopropyl) carbodiimide (EDC) alone, and in combination with N-hydroxysuccinimide (NHS) which is (EDC+NHS) is a coupling agent that helps GO to increase its sensitivity as well as fast response and recovery as a sensor [36]. One of the main advantages of EDC is its water solubility [37]. However, the coupling response has to be captured fast, as the responsive ester that is made can be quickly hydrolyzed in liquid solutions. To stabilize the response of this active ester, N-hydroxysuccinimide (NHS) can be used [38][37]. In this research, the experiments have been performed and the results have been proved that the sensor is enhanced by using this material.

Ethanol is a volatile organic compound that is normally created via fermentation process of sugars by yeast. It is also identified as Ethyl Alcohol or Ethanol and has a chemical formula C_2H_5OH. The mutual type of ethanol is found in alcoholic beverages, which can cause drunkenness after consumed [39]. It is a flammable, unstable, colourless liquid which is also used as an antibacterial, solvent and is currently gaining acceptance as a fuel alternative. Sensors to precisely monitor the concentrations of volatile organic compounds (VOCs) in liquid are very significant in order to avoid potential health and environmental problems [40]. Optical fiber sensors with real time and in situ sensing capabilities are perfect for this application. In dangerous or harsh surroundings, especially considering the risky nature of the VOCs, optical fiber sensors also have the capability of remote sensing that is perfect for this application.

1.2 Problem Statement

Most of the sensors to detect ethanol concentration are electrical based. These sensors are sensitive and reliable, however have several weaknesses, for example large in size and where the sample that has to be analyse in the lab. Electrical based sensors are also unsuitable to be installed in flammable surroundings such as for ethanol sensing. In situ monitoring of ethanol concentration requires an electrical source nearby to the sensing platform that would be a safety concern. Therefore the improvement of a safe, modest and reliable sensor for monitoring ethanol concentrations in aqueous is highly desirable.

Evanescent wave sensors harness the leak of the light energy from the core onto the cladding area but it is hard to handle it. The specific concept in evanescent wave device is to introduce interference in the fiber core to force the light to go out. Two methods can be implemented which are tapering [41] and etching [42] the fibers to increase the evanescent wave sensors. This will result in light will easily leaked out from the core because there is no cladding to confine the light in the fiber.

Although both evanescence and etched technique have been proven to be very sensitive, it lacks physical strength on its active area due to the size reduction. The diameter in the sensory region has been reduced because of the etching and the tapering process. Resultantly, its robustness becomes an issue for real world applications. Thus, a need for a optical fiber sensor utilizing GO layer [32] that will increase the potential of the sensor to be applied to the real world application without sacrificing its physical strength and sensitivity [21].

1.3 Research Scope of Study

To design and fabricate a sensitive ethanol sensor based on tapered optical fiber coated with of various nanostructured Graphene oxide application such as unsonicated GO, sonicated GO and GO with (EDC + NHS) coupling agent.

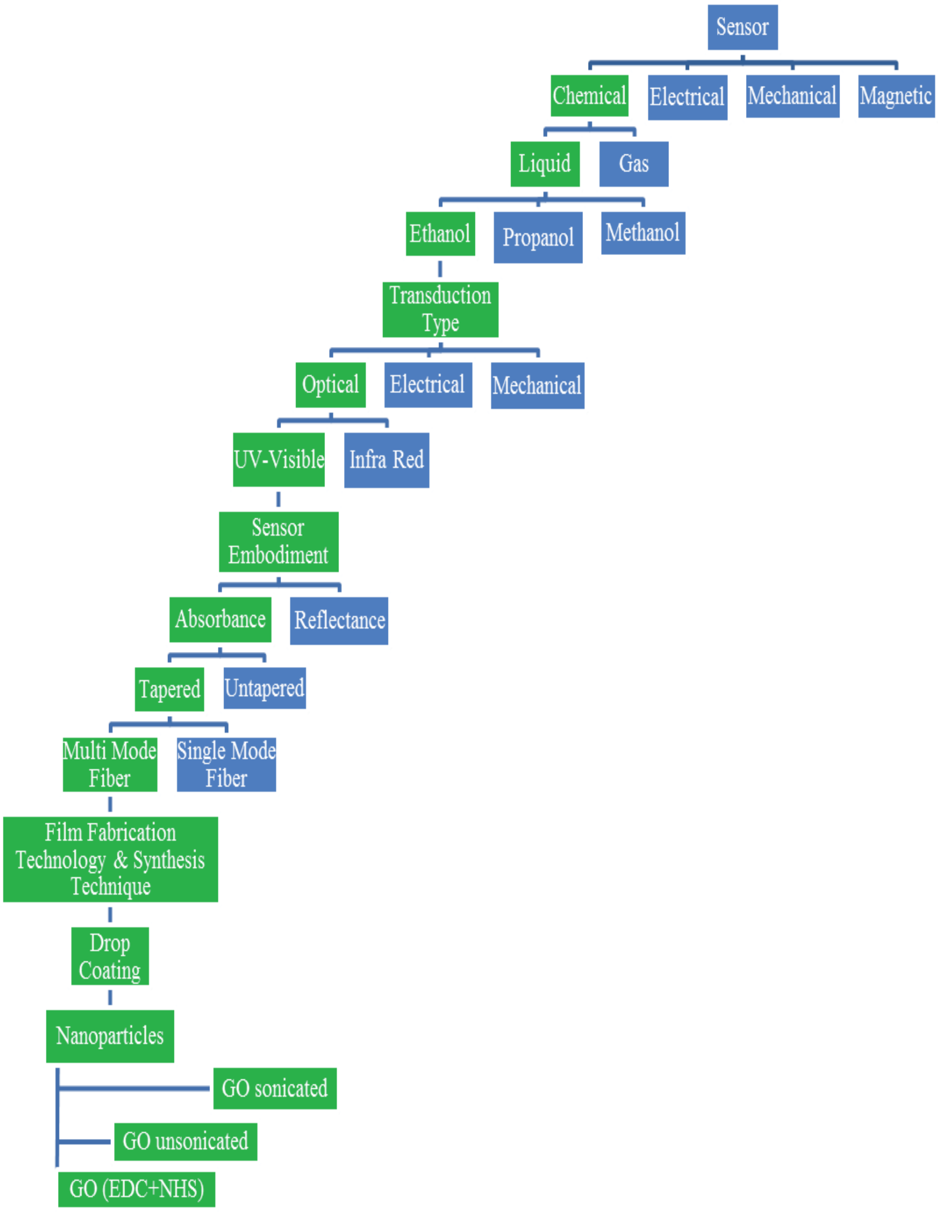

Figure 1. 1: Flow Chart Scope of Study

1.4 Objective

The purpose of this project is to study a new nanomaterial coating on tapered fiber to detect the concentration of ethanol. Observation and investigation on the differences between types of coating material are also conducted. To ensure this project run efficiently, therefore a few objectives have been formed, which are:

a. To design tapered based fiber sensor on MMF coated with GO for ethanol sensing.
b. To fabricate and evaluate the sensing performance of fiber coating with different types of graphene oxide (GO).
c. To investigate the effect of additional layer such as EDC+NHS.

1.5 Major Contributions

This research has managed to several significant contributions towards the existing work in the area of tapered optical fiber based chemical sensors. In this thesis, a comprehensive report is presented on the experiments and analysis of the developed optical sensor towards different concentrations of aqueous ethanol. The findings of this research can be outlined as follows:

- The development of a tapered optical fiber with high sensitivity towards various concentrations of ethanol.
- New investigation of various nanostructured GO application such as unsonicated GO, sonicated GO and GO with EDC + NHS coupling agent.

1.6 Summary

In this thesis, the chapter will be as follows:

i. Chapter 1: In this chapter introduces the project and outlines the objectives and problem statement.
ii. Chapter 2: In this chapter, background and theory supporting the project is discussed and other work related to this project will be reviewed and discussed.
iii. Chapter 3: In this chapter the project methodology will be outlined and the equipment and the method that will be used to complete the project will be discussed.
iv. Chapter 4: This chapter will present and discuss the result from the experiment that has been done.
v. Chapter 5: The project will be concluded and future work will be discussed in this chapter.

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