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ETHANOL SENSOR USING TAPERED OPTICAL FIBER COATED WITH GRAPHENE-BASED NANOMATERIALS

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By

SAAD HAYATU GIREI

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

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DEDICATION

This work is dedicated to;
My Parents; Mal. Saadu Muhammadu and Hajiya Aishatu (Hajja Dudu)
My brothers; Dr Gidado, Alhaji Abdullahi, Aliyu and Shagari.
My Sisters; Maryam and Fadimatu. May Allah bless them all… ameen

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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SAAD HAYATU GIREI

November 2014

Chairman: **Mohd Hanif Yaacob, PhD**

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Recently, studies based on tapered optical fiber have received much attention as compared to conventional fiber. Tapered optical fiber was found to be more sensitive as compared to the conventional fiber due to the manner of light propagation in the tapered optical fiber core. Combining the tapered fiber with a nanomaterial based sensing layer can produce novel and highly sensitive optical sensor. Graphene and graphene oxide (GO) has emerged as a leading materials in wide variety of applications including chemical sensing due to its exceptional thermal, optical and mechanical properties. Their nanostructures have a huge surface area that enhances the sensor-analyte interaction and thus, improves the sensing performance. However, their potentials as a sensing layer in the optical fiber sensor, system especially using tapered fiber towards volatile organic compounds such as liquid ethanol are yet to be fully explored. Ethanol is used in various industries such as pharmaceutical, food industries and medical. It is also increasingly used as a biofuel to replace the conventional fossil based fuels. In this thesis, ethanol sensors using tapered multimode optical fiber coated with graphene and GO were successfully developed.
Tapered optical fibers were fabricated using Vytran glass processing workstation to achieve tapers with different waist diameters. The multimode tapered optical fibers were coated with graphene and graphene oxide as the sensing layer using the drop casting technique. Graphene was produced using sodium dodecyl benzene sulfonate (SDBS) while GO was produced using the simplified Hummers method. Graphene and GO thin films were characterized using scanning electron microscopy (SEM), Raman and ultraviolet-visible (UV-Vis) spectroscopies. The working principle of the optical sensor is based on the absorbance changes upon exposure to various ethanol concentrations in water.

Sensing results indicate that the absorbance response changes linearly when the sensor was exposed to ethanol concentrations in the range of 5% to 40% in water. While both tapered optical fiber with and without the sensing layer reacted towards

ethanol, the ones coated with graphene and GO showed higher sensitivity and displays high repeatability and reversibility. As compared to the fiber coated with graphene, the ones coated with GO exhibited fast sensing performance by having both response and recovery times of less than 40 s. The GO coated sensor also exhibited higher sensitivity, as much as 0.814/vol% ethanol concentrations, than graphene coated sensor, 0.669/vol% ethanol concentrations. The developed sensors could be a suitable candidate for the practical applications of environmental monitoring and safety requirements in industries.

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

PENDERIA ETANOL MENGGUNAKAN GENTIAN OPTIK TIRUS BERSALUT BAHAN NANO BERASASKAN GRAPHENE

Oleh

SAAD HAYATU GIREI

November 2014

Pengerusi: Mohd Hanif Yaacob, PhD

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Sekarang ini kajian berdasarkan gentian optik tirus lebih mendapat perhatian berbanding gentian optik konvensional. Gentian optik tirus telah didapati lebih sensitif berbanding dengan gentian konvensional kerana cara pergerakan cahaya di dalam terasnya. Penggabungan gentian tirus dengan lapisan deria berasaskan bahan nano boleh menghasilkan pederia optik baru yang sangat sensitif. *Graphene* dan *graphene* oksida (GO) telah muncul sebagai bahan utama dalam pelbagai aplikasi termasuk penderiaan kimia kerana sifat-sifat haba, optik dan mekanikalnya yang luar biasa. Struktur nano *graphene* mempunyai kawasan permukaan besar yang meningkatkan interaksi penderia-sasaran sekaligus meningkatkan prestasi penderiaan. Walau bagaimanapun, potensi mereka sebagai lapisan deria dalam sistem penderia gentian optik khususnya gentian tirus terhadap sebatian organik meruap seperti cecair etanol masih belum diterokai sepenuhnya. Etanol digunakan dalam pelbagai industri seperti farmaseutikal, industri makanan dan perubatan. Ia juga semakin digunakan sebagai bahan api bio untuk menggantikan bahan api fosil konvensional. Dalam tesis ini, penderia etanol menggunakan gentian optik tirus bersalut *graphene* dan GO telah berjaya dibangunkan.

Gentian optik tirus ini diperbuat dengan stesen kerja pemproses kaca *Vytran* untuk mencapai gentian dengan diameter pinggang yang berbeza. Gentian optik tirus pelbagai mod telah disalut dengan *graphene* dan GO sebagai lapisan penderiaan menggunakan teknik titisan. *Graphene* dihasilkan menggunakan *natrium dodecyl benzena sulfonate* (SDBS) manakala GO telah dihasilkan menggunakan kaedah Hummers teringkas. Filem nipis *Graphene* dan GO dianalisa dengan menggunakan mikroskop imbasan elektron (SEM), Raman dan spektroskopi ultralembayung-bolehnampak (UV-Vis). Prinsip kerja penderia optik adalah berdasarkan perubahan kuantiti apabila terdedah kepada pelbagai kepekatan etanol dalam air.

Keputusan penderiaan menunjukkan bahawa kuantiti tindak balas berubah secara linear apabila penderia didedahkan kepada kepekatan etanol dalam julat 5% kepada 40% dalam air. Walaupun kedua-dua gentian optik tirus dengan lapisan deria dan tanpa lapisan deria bertindak balas terhadap etanol, gentian yang bersalut dengan *graphene* dan GO menunjukkan kepekaan yang lebih tinggi serta memaparkan kebolehulangan dan kebolehbalikan yang tinggi. Berbanding dengan gentian yang disalut dengan *graphene*, peranti yang disalut dengan GO pula menunjukkan prestasi penderiaan yang lebih baik dengan kestabilan yang tinggi apabila menunjukkan kedua-dua masa tindak balas dan masa pemulihan kurang daripada 40 s. Penderia optik bersalut GO juga mempamerkan kepekaan yang lebih tinggi dengan bacaan kepekaan ke atas kepekatan etanol sebanyak 0.814/vol% berbanding penderia bersalut *graphene* dengan bacaan kepekaan ke atas kepekatan etanol 0.669/vol%. Penderia optik termaju ini berpotensi digunakan untuk aplikasi praktikal bagi tujuan pemantauan dan keselamatan alam sekitar dan industri.

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This thesis submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

CCD	Charge-couple device
GO	Graphene oxide
rGO	Reduced graphene oxide
SDBS	Sodium dodecyl benzene solfonate
SEM	Scanning electron microscopy
SPR	Surface Plasmon resonance
TIR	Total internal reflections
UV-Vis	Ultraviolet-visible
VOC	Volatile organic compound

CHAPTER ONE

INTRODUCTION

1.1 Introduction

This chapter outlines the motivation and rationale of the research. It also includes objectives of the project. The organization of the thesis is also presented in this chapter.

1.2 Motivation and Problem Statement

In recent years, there has been an increasing interest in research on chemical sensing as wider use of chemicals are deployed in the industries. A chemical sensor is a device that can be used to monitor the concentrations or activity of some chemical specimen within a sample of interest [1]. Chemical sensor measurement plays a significant role in controlling the inputs of many production processes. Chemicals are used in various industries as raw materials for production; they can also be harmful to the environment [2]. One of the important chemicals for industrial applications is ethanol. Therefore, accurate measurement of ethanol concentrations in a given sample is of great demand.

Ethanol is mainly produced by fermentation of carbohydrate or by hydration of ethylene. It is a colorless, volatile and flammable liquid. Mixture of ethanol and water with up to 40% ethanol by volume will ignite if heated up to about 26^0 C [3]. Ethanol is widely used in many fields such as in medicine, pharmaceuticals, food industries, wine quality monitoring and in organic chemistry. Ethanol is one of the well known biofuels; it is a good alternative to petroleum as energy source. Petroleum products and its derivatives are one of the indispensible energy sources in the world. However, due to the increasing used and demand of petroleum and its derivatives, supply of crude oil has decreased dramatically in recent years [4]. This leads to increase in prices of petroleum products. Meanwhile, pollution from petroleum and its derivatives is increasingly affecting global ecosystem. Because of these drawbacks, more countries are interested in providing an alternative renewable energy source based on ethanol bio-fuels that is eco-friendly [5]. The use of ethanol can reduce the over dependence on petroleum and this will diminish the harmful gases resulting from petroleum which are believed to contribute to green house effect [6]. The use of ethanol as bio-fuel requires that it must be at least 93% pure [7]. Ethanol vapor can be toxic at certain level of concentrations. It can caused irritation of skin, inflammation of nasal mucous membrane and conjunctiva [8]. For this reason, it is of extreme important to develop ethanol sensor for environmental and industrial safety applications.

Several types of traditional chemical sensing techniques have been studied and employed in the industries. These techniques include gas/liquid chromatography, ion mobility and mass spectrometry [9]. Even though, these techniques provide accurate chemical detection, their setups are complex, expensive and not suitable for in-situ monitoring [9]. Furthermore, most chemical sensors were developed using electrical based transducers. Electrical sensors are well established and highly sensitive, but it has limitations on its deployment in the environment especially the one with high risk of explosion. Also it cannot be used in the environment that is prone to electromagnetic interference. Therefore, the development of simple, fast and safe sensors for monitoring ethanol concentrations in rugged environment is required.

From the previous view points, optical fiber sensor becomes a promising candidate due to its unique advantages such as immunity to electromagnetic interference, high sensitivity, light weight and compact size [10]. Optical fiber sensors are also robust and more resistant to harsh environments and have the capability of fast, in-situ and remote sensing application [11]. In recent years, optical fiber sensors based on tapered optical fiber has received more attention in the field of optical sensing than the conventional optical fiber sensors. This is because optical fiber sensors based on tapered optical fiber is more sensitive to the surrounding environment [12]. It is believed that highly sensitive and fast response sensors will be realized by employing tapered optical fiber sensor in a volatile environment for aqueous ethanol sensing. As a result, reducing the risk associated with leakage of ethanol.

1.3 Nanotechnology Enabled Sensors

Nanotechnology is a term that is used to describe the science, engineering and technology performed at dimension range of nanometer level. At nanoscale level, properties of materials such as physical, chemical, electrical and biological are expected to change significantly [13]. A current trend in recent years for enhancing the performance of chemical sensors is by deploying nanomaterials on sensor transducers. Nanomaterials are defined as materials with dimensions of about 100 nm and below. It is reported that sensors based on nanostructured thin film has higher surface to volume ratio. As a result higher sensitivity and lower operating temperature are expected from sensors based on nanostructured thin film. In addition, nanomaterials minimized interaction time between the analyte and the sensing layer [14]. Consequently, sensors with nanostructured thin film show fast response and recovery. This is a prerequisite in preventing potential chemical disaster.

In spite of that, most chemical sensors were based on bulk materials. For this project, the author believed that with the rapid development of nanotechnology, it is envisaged that integrating nanomaterials onto tapered optical fiber will yield optical fiber sensors with small size, fast response and high sensitivity towards ethanol.

1.4 Objectives

The aim of this research project is to develop optical fiber sensor via integration of nanomaterials onto tapered optical fiber for ethanol sensing. The specific objectives are as follows;

1. To fabricate tapered optical fiber structure for absorbance based sensor.
2. To deposit nanomaterials on the tapered optical fiber for ethanol sensing application.

3. To test and verify the sensing performance of the developed optical fiber sensor towards aqueous ethanol.

In order to achieve this objective, the following research questions were outlined.

1. How to taper optical fiber for sensing application?
2. What are the types of nanomaterials suitable for ethanol sensing?
3. How to integrate nanomaterials onto tapered optical fiber for optical sensing application?
4. How different are optical sensing performances of the tapered optical fiber sensor with and without nanomaterials?

1.5 Scope of the study

This research project will cover the fabrication of optical fiber with various taper dimensions for ethanol sensing applications. Active layer consists of graphene or GO nanostructures will be integrated onto the optical transducer and exposed towards liquid ethanol in the concentration range of 5 – 40% in water. Drop-casting technique will be deployed to deposit the nanomaterials onto tapered fiber and glass substrates. The micro-characterizations techniques such as Raman spectroscopy, UV-Vis spectroscopy and scanning electron microscopy (SEM) will be deployed to analyze the material properties. The sensing performances under consideration for the developed optical sensor are sensitivity, response and recovery times as well as sensor's stability. Figure 1.1 shows the detailed scope of the study.

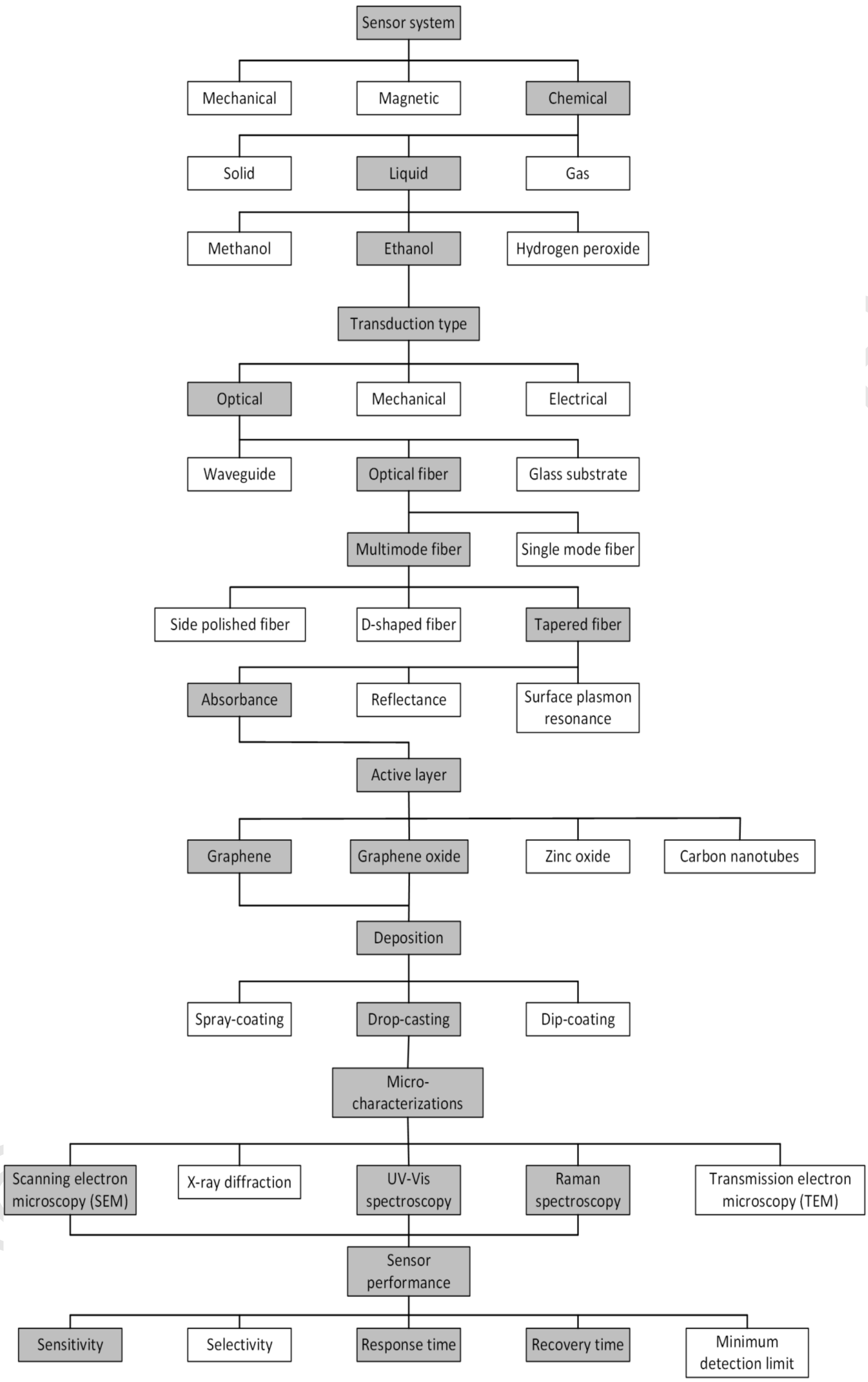

Figure 1.1. Chart showing the scope of the study

1.6 Thesis Organization

This thesis is divided into five chapters.

Chapter one is the introduction chapter that describes the motivation, problem statement, objectives, as well as the scope of the work.

Chapter two is the literature reviews that describe the rationale behind the project. This chapter presents reviews on optical fiber, optical fiber sensors and their sensing techniques. The chapter also includes review on ethanol sensors as well as nanomaterials used in this project.

Chapter three is the methodology that covers the procedures and methods used in implementing this project.

Chapter four is the results and discussion that mentions about the outcomes of the research work and its findings.

Chapter five presents the concluding part of the research work and also recommendations for future works.

REFERENCES

[1] M. A. Arnold, "Fiber-Optic Chemical Sensors," *Anal. Chem.*, vol. 64, no. 21, pp. 1015–1025, 1992.

[2] J. Janata, *Principles of chemical sensors*. New York, NY, USA: Springer, 2009.

[3] "Flash points of ethanol-based water solutions," *The engineering tool box. Retrieved 20th February 2014 from http://www.engineeringtoolbox.com*. .

[4] N. L. C. Silva, G. J. V. Betancur, M. P. Vasquez, E. D. B. Gomes, and N. Pereira, "Ethanol production from residual wood chips of cellulose industry: acid pretreatment investigation, hemicellulosic hydrolysate fermentation, and remaining solid fraction fermentation by SSF process.," *Appl. Biochem. Biotechnol.*, vol. 163, no. 7, pp. 928–36, Apr. 2011.

[5] W. Chao, "Ethanol sensor development using three-dimensional single-walled carbon nanotube networks," University of Texas, MSc thesis, 2011.

[6] M. García-Aparicio, K. Trollope, L. Tyhoda, D. Diedericks, and J. Görgens, "Evaluation of triticale bran as raw material for bioethanol production," *Fuel*, vol. 90, no. 4, pp. 1638–1644, Apr. 2011.

[7] S. K. Srivastava, R. Verma, and B. D. Gupta, "Surface plasmon resonance based fiber optic sensor for the detection of low water content in ethanol," *Sensors Actuators B Chem.*, vol. 153, no. 1, pp. 194–198, Mar. 2011.

[8] K. Mitsubayashi, T. Kon, and Y. Hashimoto, "Optical bio-sniffer for ethanol vapor using an oxygen-sensitive optical fiber," *Biosens. Bioelectron.*, vol. 19, no. 3, pp. 193–198, Nov. 2003.

[9] C. Ho, M. Itamura, M. Kelley, and R. Hughes, "Review of chemical sensors for in-situ monitoring of volatile contaminants," 2001.

[10] S. Yin, P. B. Ruffin, and F. T. S. Yu, *Fiber optic sensors*. CRC Press, 1993.

[11] K. Fidanboylu and H. S. Efendioglu, "Fiber optic sensors and their applications," in *Proceedings of 5th International Advanced Technologies Symposium*, 2009, pp. 1–6.

[12] G. Brambilla, "Optical fibre nanotaper sensors," *Opt. Fiber Technol.*, vol. 16, no. 6, pp. 331–342, Dec. 2010.

[13] P. H. Haick, *Nanotechnology and Nanosensors: Introduction to Nanotechnology*. 2013.

[14] K. Kalantan-zadeh and B. Binjamin, *Nanotechnology enabled sensors*. New York: Springer Science, 2008.

[15] R. Abdurahman, A. Yimit, H. Ablat, M. Mahmut, J. De Wang, and K. Itoh, "Optical waveguide sensor of volatile organic compounds based on PTA thin film," *Anal. Chim. Acta*, vol. 658, pp. 63–67, 2010.

[16] A. Yimit, K. Itoh, and M. Murabayashi, "Detection of ammonia in the ppt range based on a composite optical waveguide pH sensor," *Sensors Actuators B Chem.*, vol. 88, pp. 239–245, 2003.

[17] K. Iizuka, *Element of Photonics for Fiber and Intedrated Optics*. New York: John Wiley, 2002.

[18] M. H. Yaacob, "Investigation of Metal Oxide Nanostructured Thin Films Based Optical Hydrogen Sensors," RMIT University Australia, PhD thesis, 2012.

[19] A. Yimit, A. G. Rossberg, T. Amemiya, and K. Itoh, "Thin film composite optical waveguides for sensor applications : a review," *Talanta*, vol. 65, pp. 1102–1109, 2005.

[20] A. Airoudj, D. Debarnot, B. Be, and F. Poncin-epaillard, "Design and Sensing Properties of an Integrated Optical Gas Sensor Based on a Multilayer Structure," *Anal. Chem.*, vol. 80, no. 23, pp. 9188–9194, 2012.

[21] M. Yasin, S. W. Harun, and H. Arof, *FIBER OPTIC SENSORS*. Rijeka, Croatia: InTech, 2012.

[22] C. DeCusatis and C. J. S. DeCusatis, *Fiber Optics Essentials*. Elsevier, 2006.

[23] M. Azadeh, *Fiber Optics Engineering*. New York: Springer, 2009.

[24] J. I. M. Jachetta, "Fiber-Optic Transmission Systems Related Links :," in *NAB Engineering handbook*, 2007.

[25] K. Grattan and T. Sun, "Fiber optic sensor technology: an overview," *Sensors Actuators A Phys.*, vol. 82, pp. 40–61, 2000.

[26] S. Guo and S. Albin, "Transmission property and evanescent wave absorption of cladded multimode fiber tapers," *Opt. Express*, vol. 11, no. 3, pp. 215–223, 2003.

[27] S. Xue, M. A. van Eijkelenborg, G. W. Barton, and P. Hambley, "Theoretical, Numerical, and Experimental Analysis of Optical Fiber Tapering," *J. Light. Technol.*, vol. 25, no. 5, pp. 1169–1176, May 2007.

[28] K. Kieu and M. Mansuripur, "Biconical fiber taper sensors," *Photonics Technol. Lett. IEEE*, vol. 18, no. 21, pp. 2239–2241, 2006.

[29] M. Sumetsky, "Optical fiber microcoil resonator," *Opt. Express*, vol. 12, no. 10, pp. 2303–2316, 2004.

[30] G. Brambilla, F. Koizumi, X. Feng, and D. J. Richardson, “Compound-glass optical nanowires,” *Electrochim. Acta*, vol. 41, no. 7, 2005.

[31] H. S. Haddock, P. . Shankar, and R. Mutharasan, “Fabrication of biconical tapered optical fibers using hydrofluoric acid,” *Mater. Sci. Eng. B*, vol. 97, no. 1, pp. 87–93, Jan. 2003.

[32] L. Tong, R. Gattass, J. Ashcom, S. He, and J. Lou, “Subwavelength-diameter silica wires for low-loss optical wave guiding,” *Nature*, vol. 426, no. December 2003, pp. 2004–2007, 2003.

[33] H. Y. Lin, C. H. Huang, G. L. Cheng, N. K. Chen, and H. C. Chui, “Tapered optical fiber sensor based on localized surface plasmon resonance.,” *Opt. Express*, vol. 20, no. 19, pp. 21693–701, Sep. 2012.

[34] T. Birks and Y. Li, “The shape of fiber tapers,” *Light. Technol. J.*, vol. 10, no. 4, pp. 432–438, 1992.

[35] M. I. Zibaii, H. Latifi, M. Karami, M. Gholami, S. M. Hosseini, and M. H. Ghezelayagh, “Non-adiabatic tapered optical fiber sensor for measuring the interaction between α-amino acids in aqueous carbohydrate solution,” *Meas. Sci. Technol.*, vol. 21, no. 10, p. 105801, Oct. 2010.

[36] S. W. Harun, K. S. Lim, C. K. Tio, K. Dimyati, and H. Ahmad, “Theoretical analysis and fabrication of tapered fiber,” *Opt. - Int. J. Light Electron Opt.*, vol. 124, no. 6, pp. 538–543, Mar. 2013.

[37] G. Brambilla, F. Xu, P. Horak, Y. Jung, F. Koizumi, N. P. Sessions, E. Koukharenko, X. Feng, G. S. Murugan, J. S. Wilkinson, and D. J. Richardson, “Optical fiber nanowires and microwires: fabrication and applications,” *Adv. Opt. Photonics*, vol. 1, no. 1, p. 107, Jan. 2009.

[38] J. Villatoro, D. Monzón-Hernández, and D. Luna-Moreno, “In-line optical fiber sensors based on cladded multimode tapered fibers.,” *Appl. Opt.*, vol. 43, no. 32, pp. 5933–5938, Nov. 2004.

[39] M. Ahmad and L. L. Hench, “Effect of taper geometries and launch angle on evanescent wave penetration depth in optical fibers.,” *Biosens. Bioelectron.*, vol. 20, no. 7, pp. 1312–9, Jan. 2005.

[40] C. . Singh, Y. Shibata, and M. Ogita, “A theoretical study of tapered, porous clad optical fibers for detection of gases,” *Sensors Actuators B Chem.*, vol. 92, no. 1–2, pp. 44–48, Jul. 2003.

[41] J. Golden and G. Anderson, “An evanescent wave biosensor. II. Fluorescent signal acquisition from tapered fiber optic probes,” *IEEE Trans. Biomed. Eng.*, vol. 41, no. 6, pp. 585–591, 1994.

[42] Y. Tai and P. Wei, “Sensitive liquid refractive index sensors using tapered optical fiber tips,” *Opt. Lett.*, vol. 35, no. 7, pp. 944–946, 2010.

[43] S. S. Chong, A. R. A. Aziz, and S. W. Harun, “Fibre Optic Sensors for Selected Wastewater Characteristics,” *Sensors*, vol. 13, pp. 8640–8668, 2013.

[44] A. W. Snyder and J. D. Love, *Optical Waveguide Theory*. New York, NY, USA: Chapman & Hall, 1983.

[45] L. Bansal, “Development of fiber optic chemical sensor for detection of toxic vapors,” Drexel University, PhD thesis, 2004.

[46] B. Culshaw, “Fiber optics in sensing and measurement,” *IEEE J. Sel. Top. Quantum Electron.*, vol. 6, no. 6, pp. 1014–1021, Nov. 2000.

[47] X. Wang and O. Wolfbeis, “Fiber-optic chemical sensors and biosensors (2008–2012),” *Anal. Chem.*, 2012.

[48] K. T. V. Grattan and B. T. Meggitt, *Optical fiber sensor technology*. Springer Science, 1999.

[49] H. A. A. Rahaman, S.W. Harun, S.W. Phang, S.S.A. Damanhuri, H. Arof, “Tapered plastic multimode fiber sensor for salinity detection,” *Sensors Actuators A Phys.*, vol. 171, pp. 219–222, 2012.

[50] W. Cao and Y. Duan, “Optical fiber-based evanescent ammonia sensor,” *Sensors Actuators B Chem.*, vol. 110, no. 2, pp. 252–259, Oct. 2005.

[51] A. Og, P. A. Atanasov, A. T. Andreev, and B. S. Zafirova, “ZnO thin film on side polished optical fiber for gas sensing applications,” *Appl. Surf. Sci.*, vol. 254, pp. 1087–1090, 2007.

[52] W. Jin, G. Stewart, M. Wilkinson, B. Culshaw, F. Muhammad, S. Murray, and J. W. Norris, “compensation for Surface Contamination in a D-Fiber Evanescent Wave Methane Sensor,” *J. Light. Technol.*, vol. 13, no. 6, pp. 1177–1183, 1995.

[53] J. M. Corres, I. R. Matias, J. Bravo, and F. J. Arregui, “Tapered optical fiber biosensor for the detection of anti-gliadin antibodies,” *Sensors Actuators B Chem.*, vol. 135, pp. 166–171, 2008.

[54] W. Jin, H. Ho, Y. Cao, J. Ju, and L. Qi, “Gas detection with micro-and nano-engineered optical fibers,” *Opt. Fiber Technol.*, 2013.

[55] C. D. Hussey and J. D. Minelly, “Optical fibre polishing with a motor driven polishing wheel,” *Electron. Lett.*, vol. 24, no. 13, pp. 805–807, 1988.

[56] J. Villatoro, D. Luna-Moreno, and D. Monzón-Hernández, “Optical fiber hydrogen sensor for concentrations below the lower explosive limit,” *Sensors Actuators B Chem.*, vol. 110, no. 1, pp. 23–27, Sep. 2005.

[57] J. Villatoro and D. Monzón-hernández, “Fast detection of hydrogen with nano fiber tapers coated with ultra thin palladium layers,” *Opt. Express*, vol. 13, no. 13, pp. 5087–5092, 2005.

[58] F. B. Xiong and D. Sisler, “Determination of low-level water content in ethanol by fiber-optic evanescent absorption sensor,” *Opt. Commun.*, vol. 283, no. 7, pp. 1326–1330, Apr. 2010.

[59] F. B. Xiong, W. Z. Zhu, X. G. Meng, H. F. Lin, X. H. Huang, and Y. Q. Huang, “Monitor light water concentration in deuterium oxide by evanescent absorption sensor,” *Opt. - Int. J. Light Electron Opt.*, vol. 124, no. 15, pp. 2008–2012, Aug. 2013.

[60] S. Harun, M. Batumalay, and F. Ahmad, “Tapered plastic optical fiber coated with single wall carbon nanotubes polyethylene oxide composite for measurement of uric acid concentration,” *Sensor*, vol. 34, pp. 75–79, 2014.

[61] R. M. Chyad, M. Z. Mat Jafri, and K. Ibrahim, “Fabricated nano-fiber diameter as liquid concentration sensors,” *Results Phys.*, vol. 3, pp. 91–96, Jan. 2013.

[62] M. Consales, A. Crescitelli, M. Penza, P. Aversa, P. D. Veneri, M. Giordano, and A. Cusano, “SWCNT nano-composite optical sensors for VOC and gas trace detection,” *Sensors Actuators B Chem.*, vol. 138, no. 1, pp. 351–361, Apr. 2009.

[63] H. Zhang, A. Kulkarni, H. Kim, D. Woo, Y.-J. Kim, B. H. Hong, J.-B. Choi, and T. Kim, “Detection of Acetone Vapor Using Graphene on Polymer Optical Fiber,” *J. Nanosci. Nanotechnol.*, vol. 11, no. 7, pp. 5939–5943, Jul. 2011.

[64] J. I. Perterson, S. R. Golstein, and R. V Fitzgerald, “Fiber Optic pH Probe for Physiological Use,” *Anal. Chem.*, vol. 52, no. 33, pp. 864–869, 1980.

[65] J. Z. Ou, M. H. Yaacob, J. L. Campbell, M. Breedon, K. Kalantar-zadeh, and W. Wlodarski, “H sensing performance of optical fiber coated with nano-platelet WO3 film,” *Sensors Actuators B Chem.*, vol. 166–167, pp. 1–6, May 2012.

[66] C. Bariain, I. R. Matías, C. Fdez-Valdivielso, C. Elosúa, A. Luquin, J. Garrido, and M. Laguna, “Optical fibre sensors based on vapochromic gold complexes for environmental applications,” *Sensors Actuators B Chem.*, vol. 108, no. 1–2, pp. 535–541, Jul. 2005.

[67] A. Mike, F. Paul, and S. Daniel, *Nanomaterials, Nanotechnologies and Design*. 2009.

[68] C. Zamarreño, I. Matias, and F. Arregui, “Nanofabrication Techniques Applied to the Development of Novel Optical Fiber Sensors Based on

Nanostructured Coatings," *Sensors Journal, IEEE*, vol. 12, no. 8, pp. 2699–2710, 2012.

[69] M. Gautam and A. H. Jayatissa, "Gas sensing properties of graphene synthesized by chemical vapor deposition," *Mater. Sci. Eng. C*, vol. 31, no. 7, pp. 1405–1411, Oct. 2011.

[70] F. J. Arregui, I. R. Matias, J. M. Corres, I. Del Villar, J. Goicoechea, C. R. Zamarreño, M. Hernáez, and R. O. Claus, "Optical fiber sensors based on Layer-by-Layer nanostructured films," *Procedia Eng.*, vol. 5, pp. 1087–1090, Jan. 2010.

[71] B. Renganathan, D. Sastikumar, G. Gobi, N. Rajeswari Yogamalar, and A. Chandra Bose, "Nanocrystalline ZnO coated fiber optic sensor for ammonia gas detection," *Opt. Laser Technol.*, vol. 43, no. 8, pp. 1398–1404, Nov. 2011.

[72] S. Prezioso, F. Perrozzi, L. Giancaterini, C. Cantalini, E. Treossi, V. Palermo, M. Nardone, S. Santucci, and L. Ottaviano, "Graphene oxide as a practical solution to high sensitivity gas sensing," *J. Phys. Chem. C*, vol. 117, no. 20, pp. 10683–10690, May 2013.

[73] J. A. Francisco, *Sensors based on nanostructured materials*. New York, NY, USA: Springer Science, 2009.

[74] X. Liu, S. Cheng, H. Liu, S. Hu, D. Zhang, and H. Ning, "A Survey on Gas Sensing Technology," *Sensors*, vol. 12, no. 7, pp. 9635–65, Jan. 2012.

[75] R. Bogue, "Nanomaterials for gas sensing : a review of recent research," *Sens. Rev.*, vol. 1, pp. 1–8, 2014.

[76] V. Singh, D. Joung, L. Zhai, and S. Das, "Graphene based materials: past, present and future," *Prog. Mater. Sci.*, vol. 56, pp. 1178–1271, 2011.

[77] K. S. Novoselov, A. K. Geim, S. V Morozov, D. Jiang, Y. Zhang, S. V Dubonos, I. V Grigorieva, and A. A. Firsov, "Electric field effect in atomically thin carbon films.," *Science (80-.).*, vol. 306, no. 5696, pp. 666–9, Oct. 2004.

[78] Y. Zhu, S. Murali, W. Cai, X. Li, J. W. Suk, J. R. Potts, and R. S. Ruoff, "Graphene and graphene oxide: synthesis, properties, and applications.," *Adv. Mater.*, vol. 22, no. 35, pp. 3906–24, Sep. 2010.

[79] S. Basu and P. Bhattacharyya, "Recent developments on graphene and graphene oxide based solid state gas sensors," *Sensors Actuators B Chem.*, vol. 173, pp. 1–21, Oct. 2012.

[80] F. Schedin, A. K. Geim, S. V Morozov, E. W. Hill, P. Blake, M. I. Katsnelson, and K. S. Novoselov, "Detection of individual gas molecules adsorbed on graphene.," *Nat. Mater.*, vol. 6, no. 9, pp. 652–5, Sep. 2007.

[81] Y. Dan, Y. Lu, N. J. Kybert, Z. Luo, and a T. C. Johnson, "Intrinsic response of graphene vapor sensors.," *Nano Lett.*, vol. 9, no. 4, pp. 1472–5, Apr. 2009.

[82] G. Lu, L. E. Ocola, and J. Chen, "Gas detection using low-temperature reduced graphene oxide sheets," *Appl. Phys. Lett.*, vol. 94, no. 8, p. 083111, 2009.

[83] F. A. Chowdhury, T. Morisaki, J. Otsuki, and M. Sahabul Alam, "Annealing effect on the optoelectronic properties of graphene oxide thin films," *Appl. Nanosci.*, Jul. 2012.

[84] D. Marcano, D. Kosynkin, and J. Berlin, "Improved synthesis of graphene oxide," *ACS Nano*, vol. 4, no. 8, 2010.

[85] J. Zhang, H. Yang, G. Shen, P. Cheng, J. Zhang, and S. Guo, "Reduction of graphene oxide via L-ascorbic acid.," *Chem. Commun.*, vol. 46, no. 7, pp. 1112–4, Feb. 2010.

[86] S. Some, Y. Xu, Y. Kim, Y. Yoon, H. Qin, A. Kulkarni, T. Kim, and H. Lee, "Highly sensitive and selective gas sensor using hydrophilic and hydrophobic graphenes.," *Sci. Rep.*, vol. 3, p. 1868, Jan. 2013.

[87] N. F. Lokman, A. A. A. Bakar, F. Suja, H. Abdullah, W. B. W. A. Rahman, N.-M. Huang, and M. H. Yaacob, "Highly sensitive SPR response of Au/chitosan/graphene oxide nanostructured thin films toward Pb (II) ions," *Sensors Actuators B Chem.*, vol. 195, pp. 459–466, May 2014.

[88] Y. Weng, J. Rick, and T. Chou, "A sputtered thin film of nanostructured Ni/Pt/Ti on Al 2 O 3 substrate for ethanol sensing," *Biosens. Bioelectron.*, vol. 20, pp. 41–51, 2004.

[89] B. Tao, J. Zhang, S. Hui, and L. Wan, "An amperometric ethanol sensor based on a Pd–Ni/SiNWs electrode," *Sensors Actuators B Chem.*, vol. 142, no. 1, pp. 298–303, Oct. 2009.

[90] S. Khan, M. Rahman, and K. Akhtar, "Novel and sensitive ethanol chemi-sensor based on nanohybrid materials," *Int J Electrochem …*, vol. 7, pp. 4030–4038, 2012.

[91] C. Shan, H. Yang, D. Han, Q. Zhang, A. Ivaska, and L. Niu, "Electrochemical determination of NADH and ethanol based on ionic liquid-functionalized graphene.," *Biosens. Bioelectron.*, vol. 25, no. 6, pp. 1504–8, Feb. 2010.

[92] B. Chen, H. Liu, X. Li, C. Lu, Y. Ding, and B. Lu, "Applied Surface Science Fabrication of a graphene field effect transistor array on microchannels for ethanol sensing," *Appl. Surf. Sci.*, vol. 258, no. 6, pp. 1971–1975, 2012.

[93] Y.-M. Lee, C.-M. Huang, H.-W. Chen, and H.-W. Yang, "Low temperature solution-processed ZnO nanorod arrays with application to liquid ethanol sensors," *Sensors Actuators A Phys.*, vol. 189, pp. 307–312, Jan. 2013.

[94] E. Fujiwara, R. T. Takeishi, a Hase, E. Ono, J. S. Santos, and C. K. Suzuki, "Real-time optical fibre sensor for hydro-alcoholic solutions," *Meas. Sci. Technol.*, vol. 21, no. 9, p. 094035, Sep. 2010.

[95] M. Morisawa and S. Muto, "Plastic Optical Fiber Sensing of Alcohol Concentration in Liquors," *J. Sensors*, vol. 2012, no. 1, pp. 1–5, 2012.

[96] "GPX-3000 glass processing system - Operator manual."

[97] C. Qian, "Impact study of multimode fibre taper in modal noise under restricted offset launch conditions," Queen's University, Ontario Canada, MSc thesis, 2007.

[98] W. H. Jr and R. Offeman, "Preparation of graphitic oxide," *J. Am. Chem. …*, vol. 208, p. 1339, 1958.

[99] F. C. Krebs, "Fabrication and processing of polymer solar cells: A review of printing and coating techniques," *Sol. Energy Mater. Sol. Cells*, vol. 93, no. 4, pp. 394–412, Apr. 2009.

[100] H. N. Lim, N. M. Huang, S. S. Lim, I. Harrison, and C. H. Chia, "Fabrication and characterization of graphene hydrogel via hydrothermal approach as a scaffold for preliminary study of cell growth.," *Int. J. Nanomedicine*, vol. 6, pp. 1817–23, Jan. 2011.

[101] T. Owen, "Fundamentals of modern UV-visible spectroscopy," *Agil. Technol.*, 2008.

[102] K. Kudin, B. Ozbas, and H. Schniepp, "Raman spectra of graphite oxide and functionalized graphene sheets," *Nano Lett.*, vol. 8, no. 1, pp. 36–41, Jan. 2008.

[103] D. Yang, A. Velamakanni, G. Bozoklu, S. Park, M. Stoller, R. D. Piner, S. Stankovich, I. Jung, D. A. Field, C. A. Ventrice, and R. S. Ruoff, "Chemical analysis of graphene oxide films after heat and chemical treatments by X-ray photoelectron and Micro-Raman spectroscopy," *Carbon N. Y.*, vol. 47, no. 1, pp. 145–152, Jan. 2009.

[104] S. J. Wang, Y. Geng, Q. Zheng, and J.-K. Kim, "Fabrication of highly conducting and transparent graphene films," *Carbon N. Y.*, vol. 48, no. 6, pp. 1815–1823, May 2010.

[105] S. Gurunathan, J. W. Han, V. Eppakayala, and J.-H. Kim, "Biocompatibility of microbially reduced graphene oxide in primary mouse embryonic fibroblast cells.," *Colloids Surf. B. Biointerfaces*, vol. 105, pp. 58–66, May 2013.

[106] A. R. Marlinda, N. M. Huang, M. R. Muhamad, M. N. An'amt, B. Y. S. Chang, N. Yusoff, I. Harrison, H. N. Lim, C. H. Chia, and S. V. Kumar, "Highly efficient preparation of ZnO nanorods decorated reduced graphene oxide nanocomposites," *Mater. Lett.*, vol. 80, pp. 9–12, Aug. 2012.

[107] C. Wang, L. Zhang, Z. Guo, J. Xu, H. Wang, K. Zhai, and X. Zhuo, “A novel hydrazine electrochemical sensor based on the high specific surface area graphene,” *Microchim. Acta*, vol. 169, no. 1–2, pp. 1–6, Feb. 2010.

[108] Y. Shen and A. C. Lua, “A facile method for the large-scale continuous synthesis of graphene sheets using a novel catalyst.,” *Sci. Rep.*, vol. 3, p. 3037, Jan. 2013.

[109] L. Zhang, F. Gu, J. Lou, X. Yin, and L. Tong, “Fast detection of humidity with a subwavelength-diameter fiber taper coated with gelatin film.,” *Opt. Express*, vol. 16, no. 17, pp. 13349–53, Aug. 2008.

[110] M. Gautam and A. H. Jayatissa, “Detection of organic vapors by graphene films functionalized with metallic nanoparticles,” *J. Appl. Phys.*, vol. 112, no. 11, p. 114326, 2012.

[111] Y. Chang, Y. Yao, B. Wang, H. Luo, T. Li, and L. Zhi, “Reduced graphene oxide mediated SnO2 nanocrystals for enhanced gas-sensing properties,” *J. Mater. Sci. Technol.*, vol. 29, no. 2, pp. 157–160, Feb. 2013.

[112] V. S. Langford, A. J. Mckinley, and T. I. Quickenden, “Temperature dependence of the visible-near-infrared absorption spectrum of liquid water,” *j. phys. chem. A*, vol. 105, pp. 8916–8921, 2001.

[113] J. I. Paredes, S. Villar-Rodil, a Martínez-Alonso, and J. M. D. Tascón, “Graphene oxide dispersions in organic solvents.,” *Langmuir*, vol. 24, no. 19, pp. 10560–4, Oct. 2008.

[114] H. Bi, K. Yin, X. Xie, J. Ji, S. Wan, L. Sun, M. Terrones, and M. S. Dresselhaus, “Ultrahigh humidity sensitivity of graphene oxide.,” *Sci. Rep.*, vol. 3, no. 5 V, p. 2714, Jan. 2013.

[115] C. Elosúa, C. Bariáin, I. R. Matías, F. J. Arregui, A. Luquin, and M. Laguna, “Volatile alcoholic compounds fibre optic nanosensor,” *Sensors Actuators B Chem.*, vol. 115, no. 1, pp. 444–449, May 2006.

[116] C. Balamurugan, a. Subashini, G. N. Chaudhari, and a. Subramania, “Development of wide band gap sensor based on AlNbO4 nanopowder for ethanol,” *J. Alloys Compd.*, vol. 526, pp. 110–115, Jun. 2012.

[117] R. Wu, Y. Huang, M. Yu, T. Lin, and S. Hung, “Application of m-CNTs/NaClO4/Ppy to a fast response, room working temperature ethanol sensor,” *Sensors Actuators B Chem.*, vol. 134, no. 1, pp. 213–218, Aug. 2008.

[118] J. Yuan and M. El-Sherif, “Fiber-optic chemical sensor using polyaniline as modified cladding material,” *Sensors Journal, IEEE*, vol. 3, no. 1, pp. 5–12, 2003.

[119] M. El-Sherif, L. Bansal, and J. Yuan, “Fiber Optic Sensors For Detection of Toxic and Biological Threats,” *Sensors*, vol. 7, no. 12, pp. 3100–3118, Dec. 2007.

[120] F. a. Chowdhury, T. Morisaki, J. Otsuki, and M. S. Alam, “Optoelectronic properties of graphene oxide thin film processed by cost-effective route,” *Appl. Surf. Sci.*, vol. 259, pp. 460–464, Oct. 2012.

[121] K. Khun Khun, A. Mahajan, and R. K. Bedi, “SnO thick films for room temperature gas sensing applications,” *J. Appl. Phys.*, vol. 106, no. 12, p. 124509, 2009.