

# **UNIVERSITI PUTRA MALAYSIA**

SIDE-POLISHED PLASTIC OPTICAL FIBER COATED WITH NANOMATERIALS FOR CHEMICAL SENSING APPLICATIONS

AHMED LATEEF KHALAF

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# SIDE-POLISHED PLASTIC OPTICAL FIBER COATED WITH NANOMATERIALS FOR CHEMICAL SENSING APPLICATIONS

By

AHMED LATEEF KHALAF

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

November 2017



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# DEDICATIONS

То

The Memory of My Late Father for His Love and Endless Support, My Mother, Number One for Me My lovely wife, My Soul Mate, for her patience and encouragement, My beloved sons (Alkaram & Lateef), The Reasons to Get Through Another Day, My beloved brother (Husham), The Shoulder to Lean On, Sisters, My Day and Night,

My Supervisor,

All my Supervisory Committee,

All of My Friends,

My beloved first and second country Iraq and Malaysia

Without Your Support and Encouragement, My Success Wouldn't Have Been Possible.

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

## SIDE-POLISHED PLASTIC OPTICAL FIBER COATED WITH NANOMATERIALS FOR CHEMICAL SENSING APPLICATIONS

## By

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November 2017

Chairman : Mohd Hanif Yaacob, PhD Faculty : Engineering

Gaseous and liquid pollutants such as ammonia (NH<sub>3</sub>) gas and ethanol liquid, are ubiquitous in daily human activities and have been extensively studied because of their high toxicity and wide use in many fields. Common NH<sub>3</sub> gas and ethanol liquid detectors are electrical based. Although these electrical or conductometric sensors attain high sensitivity, they suffer from drawbacks that include poor selectivity, high operating temperature, and being prone to electromagnetic interference, which can be addressed by optical sensor. Optical fiber sensors present advantages in certain aspects as compared with electrical sensor, such as their compact size, capability to work in harsh environment, and capacity for remote and distributed sensing. However, chemical sensing using optical fiber has not been fully explored.

Presently, nanotechnology enabled chemical sensors have been increasingly used to enhance the sensing performance as compared with the conventional sensors toward target analytes owing to their high surface area. The sensing layer based on nanostructures has been identified to work at low temperature with high sensitivity. Therefore, this research project aims to design and comprehensively analyze optical fiber based NH<sub>3</sub> gas and ethanol liquid sensors with the incorporation of different nanostructure coatings as sensing layers. Plastic optical fiber (POF) was selected as the transducing platform for the sensor because of its low cost, ease in fabrication, and suitability for remote sensing applications. The sensitivity of POF based sensors can be improved by simply polishing part of the fiber to form side-polished optical fiber (SPPOF) using simple mechanical polishing technique. Thus, the light interaction upon coating with the sensing layer will significantly improve and great absorbance response will be achieved upon exposure to different target chemical concentrations. The influence of nanostructures morphology and roughness on the sensing performance was also studied in this PhD research.

The nanostructures under investigation were tungsten oxide (WO<sub>3</sub>), graphene oxide (GO), and carbon nanotubes (CNTs). Chemically synthesized aluminum oxide  $(Al_2O_3)$ /polyaniline (PANI) and graphene/PANI nanocomposites were also considered as sensing layers. The different nanostructured sensing layers were integrated with the polished area of the POF via radio frequency sputtering and drop-casting deposition techniques. Micro-nano characterization techniques such as SEM, EDX, AFM, Raman spectroscopy and XRD were utilized to obtain detailed structural properties of these nanostructures to fundamentally understand their functionalities with respect to the optical sensor performance.

The response of the sensors towards target chemicals at different concentrations was measured using absorbance change within the wavelength range of 400 - 800 nm at room temperature. The sensing performance was evaluated in terms of response time, recovery time, sensitivity, and repeatability. The chemical sensing performance of the developed SPPOF sensors was compared with the performance of another modified fiber, which is uncladded POF (UCPOF), using absorbance measurement. The optical sensing mechanisms of the analyte molecules and nanostructured sensing layer coated onto the polished fiber region towards NH<sub>3</sub> and ethanol with concentrations of 0.125% – 1% and 20% – 100%, respectively, at room temperature were explained.

For the first time, according to the author's knowledge, an SPPOF  $NH_3$  sensor coated with sputtered gold (Au)/WO<sub>3</sub> nanostructure thin films was successfully developed. The obtained sensitivity, response time and recovery time were 29.26/vol%, 1.2 min, and 7.3 min, respectively. Novel  $NH_3$  sensor based on SPPOF coated with graphene/PANI nanocomposite demonstrated significant sensitivity of 55.47/vol%. The remote sensing performance of the developed SPPOF sensors was also investigated by connecting them to 1.1 km multimode silica optical fiber. The SPPOF remote sensors coated with graphene/PANI and CNT exhibited excellent sensitivities of 16.63/vol% and 0.23/vol% toward different concentrations of  $NH_3$  and ethanol, respectively, at room temperature with high selectivity and long shelf life. The developed chemical sensors using SPPOF coated with nanomaterials showed superior performance as compared with the electrical based sensors. The excellent sensing performance of the optical fiber sensors via low cost and simple techniques indicates its high efficiency for remote chemical sensing in various industrial and environmental applications.

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

## TEKNIK PENGILAPAN SEBAHAGIAN PERMUKAAN GENTIAN OPTIK PLASTIK DENGAN UNSUR NANO DALAM APLIKASI SENSOR KIMIA

Oleh

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Gas dan cecair berbahaya seperti gas ammonia (NH<sub>3</sub>) dan cecair etanol, terdapat dalam aktiviti manusia seharian dan kajian meluas telah dijalankan kerana tahap keracunan yang tinggi dan penggunaannya yang meluas dalam pelbagai bidang. Biasanya, medium pengesanan gas NH<sub>3</sub> dan pengesan cecair etanol adalah berasaskan elektrik. Walaupun sensor elektrik atau konduktor metrik ini mencapai sensitiviti yang tinggi, ia mempunyai kelemahan termasuk pemilihan yang terhad, suhu operasi yang tinggi, dan terdedah kepada gangguan elektromagnetik, dimana ini boleh diatasi menggunakan sensor optik. Kelebihan sensor optik dalam aspek tertentu berbanding dengan sensor elektrik, seperti saiz yang kompak, keupayaan untuk bekerja dalam persekitaran yang mencabar, dan boleh beroperasi dalam kawalan jarak jauh dan serta pengesanan secara berkelompok. Walau bagaimanapun, sensor kimia yang menggunakan gentian kaca optik masih belum diterokai sepenuhnya.

Pada masa ini, nanoteknologi membolehkan sensor kimia semakin digunakan untuk meningkatkan prestasi pengesanan berbanding dengan sensor konvensional ke arah kejituan pada kawasan permukaannya yang luas. Lapisan penginderaan berdasarkan struktur nano telah dikenal pasti untuk beroperasi pada suhu yang rendah dengan kepekaan yang tinggi. Oleh itu, projek penyelidikan ini bertujuan untuk membuat analisis dan menganalisis sensor cecair gas NH<sub>3</sub> dan etanol cecair etanol secara terperinci dengan memasukkan lapisan struktur nano yang berbeza sebagai lapisan penginderaan. Gentian optik plastik (POF) dipilih sebagai platform pengantara untuk sensor kerana kosnya yang lebih rendah, kemudahan dalam fabrikasi, dan kesesuaian untuk aplikasi pengesanan jarak jauh. Sensitiviti sensor berasaskan POF boleh ditingkatkan dengan menggilap sebahagian daripada gentian fiber untuk membentuk medan optik berpilingan sisi (SPPOF) menggunakan teknik penggilap mekanikal mudah. Oleh itu, interaksi cahaya apabila salutan dengan lapisan penginderaan akan

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memberi kesan yang lebih tepat serta tindak balas penyerapan yang baik boleh dicapai apabila terdedah kepada kepekatan kimia yang berlainan. Pengaruh morfologi struktur nano dan status prestasi penderiaan juga dikaji dalam penyelidikan PhD ini.

Bahan-bahan nano yang digunakan adalah tungsten oxide (WO<sub>3</sub>), graphene oxide (GO), dan nanotube karbon (CNTs). Aluminium oksida sintetik (Al<sub>2</sub>O<sub>3</sub>)/polyaniline (PANI) dan graphene/PANI komposit nano juga digunakan sebagai lapisan penginderaan. Lapisan penginderaan struktur nano yang berbeza telah diintegrasikan dengan kawasan yang digilap oleh POF menerusi teknik pendepositan frekuensi dan spekteran frekuensi radio. Teknik pengekstrakan mikro-nano seperti SEM, EDX, AFM, spektroskopi Raman dan XRD digunakan untuk mendapatkan maklumat terperinci tentang struktur nano bagi memahami fungsinya dengan prestasi sensor optik.

Tindak balas sensor terhadap bahan kimia pilihan pada kepekatan yang berbeza diukur dengan memanupulasi penyerapan dalam jarak panjang 400 - 800 nm pada suhu bilik. Prestasi penginderaan dinilai dari segi masa tindak balas, masa pemulihan, kepekaan, dan kebolehulangan. Prestasi penginderaan kimia sensor SPPOF dibandingkan dengan prestasi gentian optik yang lain, seperti POF (UCPOF), dengan menggunakan teknik pengukuran penyerapan. Mekanisme penderiaan optik molekul analitik dan lapisan penderiaan struktur nano yang yang telah digilap ke dalam NH<sub>3</sub> dan etanol dengan kepekatan 0.125% – 1% dan 20% – 100%, pada suhu bilik dijelaskan.

Untuk pertama kali, sensor SPPOF NH<sub>3</sub> yang dilapisi dengan emas (Au)/ WO<sub>3</sub> struktur nano nipis berjaya dibangunkan. Sensitiviti, masa tindak balas dan masa pemulihan yang diperolehi masing-masing adalah 29.26/vol%, 1.2 min, dan 7.3 min. Sensor Novel NH<sub>3</sub> berdasarkan SPPOF yang disalut dengan graphene / PANI nano composite menunjukkan kepekaan yang signifikan 55.47/vol%. Prestasi penderiaan jauh dari sensor SPPOF yang maju juga dikaji dengan menyambungkannya ke gentian optik multimode silika sepanjang 1.1 km. Sensor penderiaan jauh SPPOF yang disalut dengan graphene/PANI dan CNT mempamerkan sensitiviti yang sangat baik dari 16.63/vol% dan 0.23/vol% dalam kepekatan NH<sub>3</sub> dan etanol yang berbeza, masing-masing pada suhu bilik dengan selektiviti tinggi dan jangka hayat yang panjang. Sensor kimia yang dibangunkan menggunakan SPPOF yang disalut dengan kemasan nano menunjukkan prestasi yang unggul berbanding dengan sensor berasaskan elektrik. Prestasi penderiaan optik yang sangat baik dengan kos yang rendah dan teknik mudah menunjukkan kecekapan tinggi untuk penginderaan kimia jarak jauh dalam pelbagai aplikasi perindustrian dan persekitaran.

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#### AHMED LATEEF KHALAF

I certify that a Thesis Examination Committee has met on 16 November 2017 to conduct the final examination of Ahmed Lateef Khalaf on his thesis entitled "Side-Polished Plastic Optical Fiber Coated with Nanomaterials for Chemical Sensing 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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## LIST OF ABBREVIATIONS

AFM Al<sub>2</sub>O<sub>3</sub> APS Ar Au BCB С C<sub>2</sub>H<sub>5</sub>OH  $CH_4$ CNT  $CO_2$ COOH CSA Cu CuO DC EDX EMI GO  $H_2$ HCl HF ITO MWCNT NA NAP NH<sub>3</sub> NIR 0 **OSHA** PANI Pd PLD **PMMA** POF Pt RF RI SEM SiO<sub>2</sub> SnO<sub>2</sub> SOF SPPOF

SWCNT

Atomic Force Microscopy Aluminum oxide Ammonium peroxydisulfate Argon Gold Bromocresol purple Carbon Ethanol Methane Carbon nanotube Carbon dioxide Carbolic acid groups Camphorsulfonic acid Copper Copper oxide Direct current Energy Dispersive X-Ray Spectroscopy Electromagnetic interference Graphene oxide Hydrogen Hydrochloric acid Hydrofluoric acid Indium tin oxide Multi walled carbon nanotube Numerical aperture Nano-Analytical Platform Ammonia Near Infrared Oxygen Occupational safety and health administration Polyaniline Palladium Pulsed laser deposition Polymethyl methacrylate Plastic optical fiber Platinum Radio frequency Refractive index Scanning Electron Microscopy Silicon dioxide Tin oxide Silica optical fiber Side-polished plastic optical fiber Single walled carbon nanotube

TiO<sub>2</sub> TIR UCPOF UMP V<sub>2</sub>O<sub>5</sub> V<sub>7</sub>O<sub>16</sub> VOC W WO<sub>3</sub> XRD YSZ ZnO

PN

Titanium dioxide Total internal reflection Uncladded plastic optical fiber Universiti Malaysia Pahang Vanadium(V) oxide Vanadium(V) oxide Volatile organic compound Tungsten Tungsten trioxide X-Ray Diffraction Yttria-stabilized zirconia Zinc oxide

## **CHAPTER 1**

#### **INTRODUCTION**

This chapter presents an overview on the research conducted by the author. Section 1.1 presents a background on optical fibers, with a focus on the importance of their use in chemical sensing applications. The motivation behind the research is discussed in the same section, which explains how the problem statements are formed as a result of the limitations found in conventional chemical sensors. Section 1.2 reviews the advances in optical sensors integrated with nanostructured coatings. Section 1.3 presents the problem statements. Section 1.4 discusses the research objectives and research questions. Finally, Sections 1.5 and 1.6 present the research scope and thesis organization, respectively.

#### 1.1 Research Background and Motivations

Air and liquid pollutants are ubiquitous in daily human activities, such as agriculture, food processing, industrial coolants, and household detergents. Two important chemicals for industrial applications are ammonia  $(NH_3)$  and ethanol. Advanced  $NH_3$  gas and ethanol liquid real-time sensing methods have been continuously developed over the past few decades because of their high toxicity and wide use in many fields. Thus, it is critical to develop highly sensitive and reliable sensors to measure these chemicals in their applications and to prevent safety hazards.

 $NH_3$  is a natural gas that exists in the earth atmosphere. This natural gas is colorless flammable gas with strong odor and is commonly produced from various sources, such as chemical plants, livestock farming, and motor vehicles [1]. The world market production of  $NH_3$  is estimated to grow at an average rate of 3.5% in 2017, with a global capacity of approximately 198 million tons, which leads to revenues of approximately one hundred billion US dollar [2, 3].

NH<sub>3</sub> is an irritant gas, and its permissible exposure limit is 35 ppm according to occupational safety and health administration (OSHA) [4]. The inhalation of large quantities of NH<sub>3</sub> can cause harmful effects on human health, such as eye irritation, pulmonary edema, and respiratory arrest [5]. Recently, in India, five people were killed and over 140 others complained of breathing problems and eye irritation after they inhaled large amounts of NH<sub>3</sub> gas that leaked from an NH<sub>3</sub> gas tanker. The leakage of gas also can lead to the death of plants and animals within 4 km radius [6]. Therefore, NH<sub>3</sub> concentrations should be monitored using reliable and low cost sensors that can be operated at room temperature to prevent environmental pollution risks and comply with the safety regulations.

Ethanol is a volatile organic compound (VOC) that is commonly used in the food, beverage, fuel and pharmaceutical industries [7, 8]. The ethanol production has risen substantially over the past decade as a result of the rise in demand for ethanol blend fuels. A report indicated that 14.7 billion gallons of ethanol was transported in the United States of America (USA) either by rail or barges from production facilities in 2015 [9]. Ethanol is currently considered one of the largest volume hazardous materials shipped by rail in the USA aside from crude oil and chlorine [10, 11]. Figure 1.1 shows the increase in production volume of ethanol in the USA from 2000 to 2012. One of the major concerns is ethanol leakage or spills during transportation or storage. Liquid ethanol is flammable, with a lower explosive limit to an upper explosive limit range of 3.3% - 19% [12]. In addition, ethanol is colorless and is completely miscible in water. Eleven major spills were reported in the USA from 2006 to 2010, which mostly resulted in water contamination [12]. Exposure to high concentrations of ethanol may cause irritation to the skin and inflammation of the nasal mucous membrane. Therefore, ethanol detection in a liquid medium is crucial for the quality control of food, beverages and medicines [13, 14].



Figure 1.1: Ethanol production from 2000 to 2015 [12].

A wide range of real-time approaches have been developed for sensing applications, such as liquid chromatography and mass spectrometry, to meet such an ever-increasing demand [15]. However, despite their good selectivity, these approaches suffer from limitations that include high cost and bulky size. Meanwhile, low cost conductometric and electrochemical sensors attain high sensitivity in chemical sensing applications [16, 17]. However, these sensors are limited by several drawbacks, such as poor selectivity, high power consumption, and susceptibility to electromagnetic interference (EMI), which restrict their use in practical application. In addition, all the aforementioned sensors require an elevated working temperature (> 300 °C) and several mechanical moving elements, thereby leading to implementation difficulties for

 $\bigcirc$ 

remote sensing applications. Thus, to overcome these drawbacks, optical fibers have been explored as an alternative to the conventional chemical sensors owing to their numerous advantages. Optical fiber sensors are easily integrated into optical networks and telecommunications systems with the added capability of distributed and remote sensing. The introduction of chemical based optical fiber sensors in the last few decades has gained great attention because of their micro-sized, immunity to EMI, and their capability to work in harsh environments [18]. Since then, optical fiber based chemical sensors have been adopted in various applications and enormous efforts have been made to enhance their performance.

#### 1.2 Optical Fiber Sensor with Nanostructured Sensing Enhancements

Optical fiber sensors have gained much popularity and market acceptance in recent years due to the number of advantages compared with their electrical counterparts. These advantages include miniaturization, flexibility, and immunity to EMI in addition to their capability to operate in hostile environments, such as high temperature or chemically reactive surroundings [19]. Optical fibers are also inert, passive, and electrically nonconductive. As a result, optical fiber is safe to be used near flammable or explosive materials, such as in the oil and gas industries [20].

The recent advances in optical sensing with nanostructured material enhancements are making a significant impact on the direction of future sensor technology. The rapid progress in nanofabrication methods and nanoparticle synthesis is leading to the development of various new nanostructured materials with unique physical and chemical properties. Nanostructures are materials that have at least one dimension in nanoscale range, which is less than 100 nm [21]. These materials are particularly suitable for chemical and biological sensing applications owing to the fact that many of the reactions occur in nanoscale [22].

When materials are reduced to nanoscale dimensions, they reveal new and unique properties such high surface-to-volume ratio, high heat capacity, mechanical strength, and changes in magnetic behavior. Distinctive changes in optical properties include reflectivity, absorbance, and luminescence [23].

Optical sensors with nanostructured enhancements are new and an interesting path for developing chemical sensors. Integration of nanostructured material thin films on optical transducing platforms has demonstrated improvements in sensing performance. The optical techniques generally employed to measure response in chemical sensing applications are based on reflectance, absorbance, fluorescence, surface plasmon resonance, and luminescence changes, which are caused by the interaction between the nanostructured coatings and diverse chemical molecules [24].

## 1.3 Problem Statement

Most of chemical sensors are electrical based. The main reason electrical sensors are widely used is due to their high sensitivity and low cost. Nonetheless, these sensors suffer from poor selectivity and limited deployment in a volatile environment, especially where high risk of explosion or proneness to EMI is expected. Dangerous chemicals, such as NH<sub>3</sub> gas and ethanol liquid, are highly toxic and flammable. The limitations of electrical sensors to detect these chemicals can be addressed with the use of optical transducers, such as optical fiber.

Common optical fiber sensors are based on glass or silicon dioxide  $(SiO_2)$  material. Glass sensor is highly deployed in optical communications because of its high temperature and low cost. Nevertheless, its high fragility owing to small waist diameter (<100 µm) and the complexity in its modification can restrict the platform deployment in sensing applications.

Plastic optical fiber (POF) offers numerous advantages, including low cost, ease of manipulation, and high degree of flexibility [25]. In optical fiber chemical sensors, strong interaction between the sensing layer and light propagates in the fiber core is highly required to improve their sensitivity. This can be achieved by modifying the POF through removing part of the fiber structure via a simple mechanical polishing technique and coating it with an active sensing layer. Changes in the optical, physical, and chemical properties may occur as a result of the interaction between the analyte molecules and the sensing layer coated on the exposed area of the optical fiber.

Most of the sensing layers deployed in chemical detection during the last decades are dominantly based on thick films (approximately more than 10  $\mu$ m). Nevertheless, the advancement of nanotechnology provides opportunity to integrate sensing materials at nanoscale with transducing platforms. Nanostructured materials have numerous advantages that include large surface-to-volume ratio, high specific surface area, and several surface active sites [26, 27]. Recent studies have identified that the use of nanostructured material as active sensing layer can improve the chemical sensing performance with regard to sensitivity, selectivity, and response time in comparison with thick film sensing layers [28].

Remote sensing using optical fibers has attracted great attention in many applications in recent years because of the distinctive features of these optical fibers that make them efficient candidate for applications in harsh and combustible environments [29]. Moreover, the telemetry channel based on optical fiber is immune to EMI in addition to its capability to avoid reflections from natural and artificial obstacles and bad weather interruptions [30]. Generally, the main concept of optical fiber remote sensing is to continuously observe a structure from a central station located away from the sensor critical site without the need for electrical power feeds in the remote locations. Thus,

optical fiber remote sensor allows instant hazard detection as compared with the currently used electrical remote sensor that suffers from complexity and slow response times [31]. Apart from that, the use of optical fiber remote sensing for chemical sensing applications remains in its infancy stage.

## 1.4 Research Objectives and Questions

The aim of this research is to design and fabricate SPPOF for chemicals detection by integrating nanostructured materials as sensing layer. The objectives to achieve this include:

- 1. To fabricate and characterize side-polished POF (SPPOF) for NH<sub>3</sub> and ethanol sensing.
- 2. To synthesis and deposit the nanomaterial onto the polished sensing area.
- 3. To investigate and relate the micro-nano characteristics of the nanomaterials and their influence on the optical sensor's performance.
- 4. To analyze and evaluate the optical sensing performance of the developed in-situ and remote SPPOF sensors towards NH<sub>3</sub> and ethanol at room temperature.
- 5. To propose and explain the chemical sensing mechanism of all the developed novel optical fiber sensors.

To achieve these objectives, the following research questions are outlined:

- 1. What sensing layers are suitable to be used in order to obtain a response to NH<sub>3</sub> and ethanol?
- 2. What are the suitable synthesis and deposition methods accessible to coat nanomaterials onto optical fibers?
- 3. What are the optimized dimensions of the SPPOF as chemical sensor?
- 4. How different are the sensing performances of side-polished optical fiber sensors coated with various nanostructured sensing layers?
- 5. How to understand the nature of interaction between the target chemical and the sensing layer?

Based on the research questions, the research project focused on developing SPPOF as a chemical sensor and investigating suitable nanostructured materials that would enhance the performance of the optical sensor toward NH<sub>3</sub> gas and ethanol liquid detection. The author developed an SPPOF-assisted chemical sensor coated with nanostructured sensing

layers of gold (Au)/tungsten oxide (WO<sub>3</sub>), graphene oxide (GO) and carbon nanotubes (CNT) in addition to aluminum oxide (Al<sub>2</sub>O<sub>3</sub>)/polyaniline (PANI) and graphene/PANI nanocomposites. The nanostructured materials were deposited on the proposed optical fibers by use of drop-casting and radio frequency (RF)-sputtering techniques to analyze their sensing performance.

### 1.5 Research Scope

This research project covers the development of opto-chemical sensor starting from materials leading to optical devices. The scope of the project can be best explained with the tree diagram in Figure 1.2. The solid lines represent the direction followed in this thesis to achieve the goal and objectives of the work, while the dotted lines refer to other research areas that are out of the scope of this work. The highlighted boxes represent the elements deployed to achieve the research objectives proposed for the development of optical fiber chemical sensor.

### 1.6 Thesis Organization

This thesis is divided into six chapters. Chapter 1 presents an overview of the research area, focusing on the problems that motivated this work. In addition, this chapter demonstrates the problem statements, aim and objectives of the research. Chapter 2 indicates a comprehensive review on the optical fiber types and properties and the principles of optical measurement. A critical review on optical fiber sensors used for chemical applications is also discussed in this chapter. The nanostructured materials utilized in this PhD work are included, and their properties and applications are discussed. The reported research, most relevant to this project, is critically reviewed, showing the trade off in the performance of these works. Chapter 3 gives the full description for the fabrication and design of SPPOF and uncladded POF (UCPOF) sensors. The nanostructured material synthesis and deposition are also presented and discussed, followed by the testing setup for the developed optical fiber sensors. Chapter 4 provides the characterization results of the nanostructures deposited on SPPOF and UCPOF. Chapter 5 discusses the sensing results obtained from the SPPOF and UCPOF sensors performance integrated with nanostructured materials toward NH<sub>3</sub> and ethanol. The effect of the morphology of the nanostructured materials on the sensor responses toward different concentrations of  $NH_3$  and ethanol is elucidated. The performance of the remote sensing of SPPOF is discussed and configured for different nanomaterial coatings. This chapter also discusses the sensor performance in terms of sensitivity, selectivity, repeatability, and response and recovery times. The optical sensing mechanisms for NH<sub>3</sub> gas and ethanol liquid interaction with different nanomaterials are explained. Chapter 6 concludes the thesis and presents a list of contributions of this work. Potential ideas to be pursued as future works are also suggested.



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