UNIVERSITI PUTRA MALAYSIA

OPTICAL MICROFIBER SENSOR COATED WITH NANOMATERIALS FOR AMMONIA SENSING APPLICATIONS

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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 Doctor of Philosophy

June 2021

DEDICATION

I dedicate this thesis to:
My Mother and my late Father.

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

OPTICAL MICROFIBER SENSOR COATED WITH NANOMATERIALS FOR AMMONIA SENSING APPLICATIONS

By

SAAD HAYATU GIREI

June 2021

Chairman : Mohd Hanif bin Yaacob, PhD
Faculty : Engineering

Ammonia (NH_3) is a colorless compound with a distinctive odor composed of nitrogen and hydrogen atoms that can be found in water as ammonia nitrogen (NH_3-N) and in the air as NH_3 gas. It is commonly used in several industrial processes, agricultural activity and several biological systems. However, at high concentrations, NH_3 can be toxic to plants, animals as well as human beings. Its detection is important for environmental and industrial safety. The previous development of NH_3 sensors were mostly concentrated on the thick films and electrical based sensors rather than optical.

In recent years, tapered optical microfibers sensors have been attracting greater attention due to their simplicity and immunity to various sources of interferences. This research work presents the development of a tapered optical microfiber sensor coated with nanomaterials for the detection of NH_3 in liquid and gaseous forms. The working principle of the sensor is based on the interaction between the evanescent fields of the tapered microfiber and different NH_3 concentrations. The interactions alter the properties of light propagating through the optical fiber and consequently producing a measurable response that allows quantifying the concentration of NH_3. To enhance the sensing performance of the developed sensor, zinc oxide (ZnO) and graphene oxide (GO) nanomaterials were deposited on the fiber surface providing a higher surface area and suitable chemical reaction between NH_3 and the sensing layer.

The GO nanostructures and ZnO nanorods were deposited using the optical and hydrothermal deposition techniques, respectively. The optical deposition technique was successfully implemented to produce uniform GO coating with corrugated and wrinkled structures on the cylindrical optical microfiber surface. The GO deposition is due to the occurrence of the thermophoresis effect resulting from the interaction of optical radiation with the GO solution at the tapered area of the microfiber interferometer (MFI). A unique hydrothermal method is designed for uniform coating around the cylindrical optical microfibers. This method is a simple and environmentally-friendly deposition technique

that produced ordered arrays of ZnO nanorods directing outwards from the surface of the tapered optical microfiber.

The NH_3-N sensing response investigated in a wide wavelength range of 1500 – 1800 nm by monitoring the wavelength shift shows sensitivities of 0.0894 nm/ppm and 0.1748 nm/ppm at 1785 nm for bare and GO-coated MFI sensor, respectively. The developed NH_3-N sensor showed excellent properties of high sensitivity, stability, and fast response at room temperature as compared to the conventional sensors. The ZnO nanorods and GO-coated optical microfiber sensor for NH_3 gas exhibit maximum absorbance response with the optimum sensing layer thickness of 750 nm and 692 nm, respectively. Sensing performance results reveal the sensitivities of 59.18 AU/% and 61.78 AU/% for sensors coated with ZnO nanorods and GO, respectively. The results of this investigation further reveal a promising method to improve NH_3 gas sensitivity by prolonging the hydrothermal growth duration of ZnO nanorod arrays on the optical microfiber. It was also discovered that the GO-coated sensor produced negative and positive absorbance responses at the visible and near-infrared wavelength regions, respectively. These interesting sensing characteristics provide a new understanding of the behavior of absorbance response of the GO-coated sensor to the operational bandwidth.

Above all, the combination of appropriate sensitive materials and optical fiber devices provides a convenient platform to detect the chemical concentrations of liquid and gas. As a result, these sensors may find applications in the manufacture of fertilizer, medical diagnostics, and in rivers and drinking water monitoring.

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

SENSOR MIKROFIBER OPTIK BERSALUT BAHAN NANO UNTUK APLIKASI PENDERIAAN AMMONIA

Oleh

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Ammonia (NH_3), suatu sebatian tidak berwarna, dengan bau tersendiri dan terdiri daripada atom nitrogen dan hidrogen, dapat dijumpai di dalam air dalam bentuk ammonia nitrogen (NH_3-N) dan di udara sebagai gas NH_3. Ammonia biasanya digunakan dalam beberapa proses industri, kegiatan pertanian, dan beberapa sistem biologi. Walau bagaimanapun, pada kepekatan tinggi, NH_3 boleh menjadi toksik kepada tumbuhan, haiwan dan juga manusia. Oleh itu, pengesanannya penting untuk keselamatan persekitaran dan industri. Perkembangan sensor NH_3 sebelum ini kebanyakannya tertumpu pada filem tebal dan berasaskan sensor elektrik berbanding yang berdasarkan optik.

Dalam beberapa tahun kebelakangan ini, sensor mikrofiber optik tirus telah menarik perhatian yang lebih besar kerana keringkasan dan ketahanannya terhadap pelbagai sumber gangguan. Kajian ini membentangkan kerja-kerja pembangunan sensor optik mikrofiber tirus disalut dengan bahan nano untuk mengesan NH_3 dalam bentuk cecair dan gas. Prinsip operasi sensor ini adalah hasil interaksi antara medan singkat daripada mikrofiber yang tirus dan NH_3 yang berkepekatan berbeza. Interaksi ini mengubah sifat-sifat penyebaran cahaya melalui gentian optik itu dan seterusnya menghasilkan tindakbalas terukur yang membolehkan penentuan kepekatan NH_3 . Untuk meningkatkan prestasi penderiaan sensor yang diuji ini, bahan nano zink oksida (ZnO) dan graphene oksida (GO) telah diselaput di permukaan gentian bagi menyediakan luas permukaan yang lebih tinggi dan sesuai untuk tindakbalas kimia antara NH_3 dan lapisan penderiaan.

Bahan struktur nano GO dan rod nano ZnO masing-masing telah terselaput menggunakan teknik pemendapan optik dan hidroterma. Teknik pemendapan optik juga telah berjaya dilaksanakan untuk menghasilkan lapisan GO yang seragam dengan struktur beralun dan berkedut di permukaan mikrofiber optik berbentuk silinder. Pemendapan GO berlaku hasil kesan termoforesis yang disebabkan oleh interaksi sinaran optik dengan larutan GO di kawasan interferometer mikrofiber terubah

(MFI). Kaedah hidroterma unik telah direka untuk lapisan seragam di sekitar mikrofiber optik silinder. Kaedah ini adalah teknik pemendapan yang mudah dan mesra alam yang menghasilkan susunan teratur rod nano ZnO yang mengarah keluar daripada permukaan mikrofiber optik tirus.

Tindakbalas penderiaan NH_3-N yang disiasat dalam julat panjang gelombang 1500 - 1800 nm dengan memantau perubahan panjang gelombang menunjukkan kepekaan 0.0894 nm/ppm dan 0.1748 nm/ppm pada 1785 nm masing-masing untuk sensor MFI tidak bersalut dan bersalut. Sensor NH_3-N yang dihasilkan menunjukkan sifat kepekaan tinggi, kestabilan, dan tindakbalas yang pantas pada suhu bilik berbanding dengan sensor konvensional. Sensor mikrofiber optik rod nano ZnO dan struktur nano GO untuk pengesanan gas NH_3 menunjukkan tindak balas serapan maksimum dengan ketebalan lapisan penderiaan optimum masing-masing 750 nm dan 692 nm. Hasil prestasi penderiaan menunjukkan kepekaan 59.18 AU/% dan 61.78 AU/% untuk sensor yang masing-masing dilapisi dengan rod nano ZnO dan GO. Hasil siasatan ini dengan lebih lanjut menunjukkan kaedah berpotensi untuk meningkatkan kepekaan terhadap gas NH_3 dengan memanjangkan tempoh pertumbuhan rod nano ZnO secara hidroterma pada mikrofiber optik. Ia juga mendapati bahawa sensor bersalut GO menghasilkan tindakbalas penyerapan negatif dan positif di kawasan panjang gelombang nampak dan infrared dekat. Ciri-ciri penderiaan yang menarik ini memberikan pemahaman baru mengenai sifat tindakbalas serapan sensor bersalut GO terhadap lebar jalur gelombang operasi.

Secara keseluruhannya, gabungan bahan-bahan sensitif sesuai dan peranti gentian optik menyediakan platform yang baru dan mudah untuk pengesanan kepekatan bahan kimia cecair dan gas. Oleh yang demikian, sensor ini mungkin dapat memenuhi aplikasi di dalam pembuatan baja, diagnosis perubatan, dan pemantauan sungai serta air minum.

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

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Declaration by graduate student

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Signature: ______________________________
Name of Member of Supervisory Committee: Prof. Dr. Hong Ngee Lim

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LIST OF ABBREVIATIONS

AFM	Atomic Force Microscopy
CNT	Carbon Nanotubes
DOE	Department of Environment
EDX	Energy Dispersion X-Ray
ER	Extinction Ration
FBG	Fiber Bragg Grating
FESEM	Field Emission Scanning Electron Microscopy
FFT	Fast Fourier Transform
FSR	Free Spectral Range
FWHM	Full Width Have Maximum
GO	Graphene Oxide
RH	Relative Humidity
ITU	International Telecommunication Union
LED	Light Emitting Diode
LMR	Lossy Mode Resonance
LOD	Limit of Detection
LPG	Long Period Grating
MFI	Microfiber Interferometer
MMF	Multimode fiber
NH_3	Ammonia gas
NH_3-N	Ammonia nitrogen
NIR	Near-infrared
OPD	Optical Path Difference

OSA	Optical Spectrum Analyzer
OSHA	Occupational Safety and Health Administration
PCF	Photonics Crystals Fiber
POF	Plastic Optical Fiber
rGO	reduced Graphene Oxide
RI	Refractive Index
SMF	Single mode fiber
SPR	Surface Plasmon Resonance
TCF	Thin Core Fiber
XRD	X-Ray Diffraction
ZnO	Zinc Oxide

CHAPTER 1

INTRODUCTION

This chapter discusses the background and motivation of the research, problem statement, PhD research objectives as well as the scope of the study.

1.1 Background and Motivations

In the past few years, there has been a growing interest in chemical sensing research. This is due to the wider use of chemicals in industries and the environment. A chemical sensor is a device or a subsystem that can be used to monitor or detect the changes or concentrations of some chemical specimen within a sample of interest [1]. Chemical sensor measurement plays a significant role in controlling environmental pollution and production processes. Chemicals are used in various industries as raw materials for production; they can also be harmful to the environment [2]. Ammonia (NH_3) is one of the widely used chemicals in industries and plays a significant role in environmental pollution.

NH_3 occurs naturally and can be found all through the environment in air, soil, and water. It has emerged as an important building block in the manufacturing of many products we use daily including plastics, textiles, dyes, and household cleaning solutions. It is also a nitrogen source for plant growth, which makes it an essential ingredient in fertilizers. About 80% of NH_3 produced is used for the manufacture of nitrogen-based fertilizer. Furthermore, it has been widely used as a preservative in the agriculture industry, nitrogen source in the beverage industry, curing agent in the leather industry, and anti-corrosive in the petroleum industry. It is also used in waste and wastewater treatment plants [3]–[5]. These various applications of NH_3 are supported by the statistics in Figure 1.1.

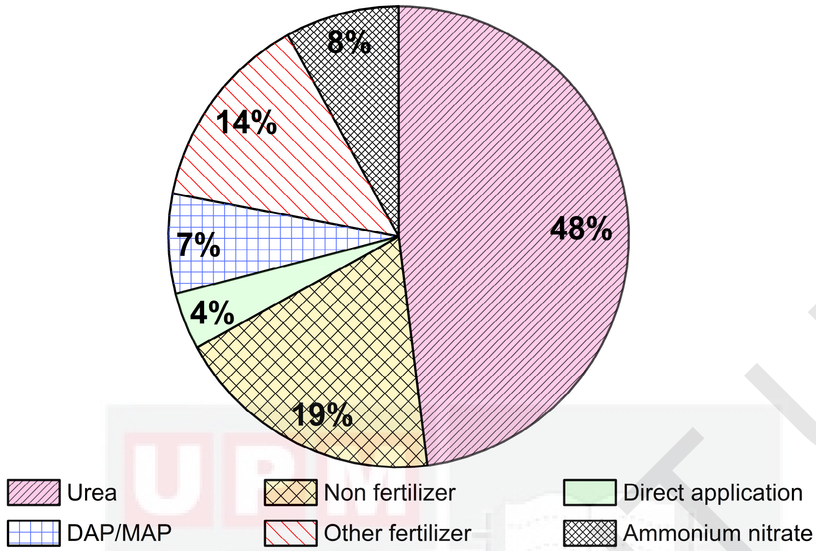

Figure 1.1: World NH_3 usage [4]

Nonetheless, NH_3 is one of the highly toxic chemicals given its implications on human health. According to the occupational safety and health administration (OSHA), the specified threshold limit value for NH_3 gas in the workplace is 25 ppm and 35 ppm for 8 h and 15 mins work shift, respectively [6], [7]. A high concentration of NH_3 constitutes a threat to the human body. NH_3 concentration of 500 ppm can cause immediate and severe irritation to the nose and throat while a higher concentration of 1000 ppm or more can cause pulmonary edema, accumulation of fluid in the lungs, and even death [8], [9]. NH_3 in the form of ammonium nitrate is also an ingredient in certain explosives. Recently, a devastating explosion at a port in Beirut, Lebanon, which killed more than 200 people and injured at least 5,000 was caused by tonnes of ammonium nitrate in a storage unit [10]. Additionally, NH_3 can directly upset the equilibrium of water bodies. Ammonia nitrogen (NH_3-N) is a measure of the amount of NH_3 found in water bodies. Levels of NH_3-N in water provide important information about water quality in rivers and water supply processing plants as well as in drinking water [11]. A high concentration of NH_3-N in water can create serious problems such as eutrophication, deterioration of water quality which can pose a potential hazard to aquatic life as well as human health [12], [13]. Therefore, it has become imperative to monitor NH_3 concentrations for environmental, health, and industrial safety.

A wide range of approaches has been developed for the detection of chemicals; these methods include colorimetry [14], mass spectrometry [15], and liquid chromatography [16]. Although these sensors can detect chemicals selectively, they exhibit some limitations such as high cost, bulky size, use of numerous samples, and is time-consuming [17], [18]. At the same time, low-cost electrochemical and conductometric sensors attain high sensitivity applications [19]. However, these sensors are also constrained by some downsides, such as selectivity and susceptibility to electromagnetic interferences which hinder their applications in rugged environments. Among all the sensing platforms, optical fiber sensors have achieved a high impact in the last decades because they offer several advantages over other sensors. Particularly, optical fiber sensors are highly sensitive, small, lightweight, resistant to high temperature and harsh environment, immune to electromagnetic fields and interferences, and have remote sensing capabilities [20], [21]. These unique features make them exceptionally suitable for some specific applications.

Over the past few years, optical fiber sensor technology has grown considerably and is increasingly playing a significant role in environmental and safety monitoring, as well as chemical and biological sensing [22]. For instance, optical fiber-based sensors are used for temperature [23] and humidity [24] monitoring and to detect and quantify the amount of ethanol in water [25] or even to monitor respiratory movement [26]. Figure 1.2 shows an illustration of the future development of optical fiber sensors. With the development of advanced sensing materials and microfabrication techniques, optical fiber NH_3 sensors can find applications in many areas of environmental monitoring, chemical and pharmaceutical industries, diagnosis, food safety, defense, healthcare, and so on, and significantly improve the quality of our living.

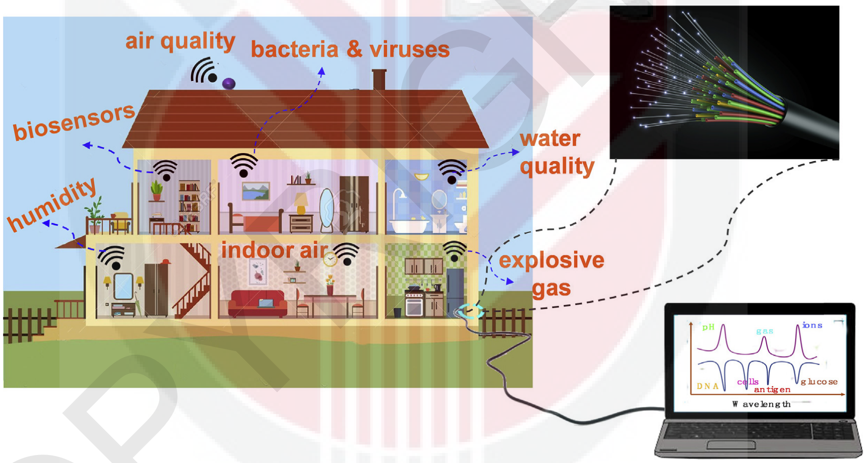

Figure 1.2: Application of optical microfiber sensors for chemical, biological, and environmental monitoring [20]

1.2 Problem Statement

Many chemical sensors are based on electrical sensing technology. Electrical-based sensors are widely used mainly because of their inexpensiveness and high sensitivity. Such sensors, however, suffer from poor selectivity and are not suitable to be deployed in a rugged environment. This is because they are prone to areas with a high risk of explosion or various sources of interference. In situ monitoring of NH_3 requires an electrical source close to the sensing platform, which will be a safety concern. Therefore, it is highly desirable to develop a safe, simple, and reliable NH_3 monitoring sensor, and an optical fiber sensor is a suitable candidate.

Most of the sensing layers used to detect chemicals in recent decades are based mainly on thick films (about 10 μm). However, the development of nanotechnology allows for the integration of sensing materials with transduction platforms in nanoscales. The nanoscale level is expected to change dramatically in the properties of materials including physical, chemical, electrical, and optical [27], [28]. Recent studies have found that using a nanostructured substance as an active sensing layer, in comparison to thick film sensing layers, will increase the chemical sensing efficiency in terms of operating temperatures, sensitivity, selectivity, and response time.

Most of the reported optical fiber sensors for NH_3-N sensing in water are based on special optical fibers such as fiber Bragg grating (FBG), photonic crystal fiber (PCF), and thin core fiber (TCF). These sensing devices involve complicated and expensive fabrication techniques. For example, in [29] and [30], thin-core fiber and small core fiber, respectively were sandwiched between standard single mode fiber forming and interferometric sensing devices for NH_3-N. Alternative optical sensing devices with simpler and cost-effective fabrication can be an attractive solution.

1.3 Objectives of the Study

The main objective of this research is to develop an optical microfiber sensor coated with nanomaterial for NH_3 sensing applications.
The specific objectives are:

1. To design, fabricate and characterize optical microfiber with optimized parameters for sensing of NH_3 concentration in water and air.

2. To synthesize and integrate nanomaterials onto tapered optical microfiber sensing area.

3. To investigate and evaluate the optical sensing characteristics of the nanomaterials towards liquid and gaseous NH_3.

To achieve these objectives, the following research questions were outlined.

1. What are the optimized optical microfiber dimensions for NH_3 sensing application?

2. What types of nanomaterials are suitable for NH_3 sensing applications?

3. How to integrate the nanomaterials onto tapered optical microfiber for sensing application?

4. How different are the optical sensing performances of tapered optical microfiber sensors with and without nanomaterials?

5. How different are the optical sensing performances of tapered optical microfiber coated with nanomaterials of different film thicknesses?

Based on these research questions, the research work focused on developing tapered optical microfiber as NH_3 sensor and investigating suitable nanomaterials that would enhance the performance of the optical microfiber sensor toward NH_3 in liquid and gaseous form. The author developed an optical microfiber interferometric sensor coated with graphene oxide (GO) for measurement of low concentration of NH_3-N in water. The author also developed an absorbance-based optical microfiber sensor coated with nanostructured sensing layers of Zinc Oxide (ZnO) nanorods and GO for measurement of different concentrations of NH_3 gas in the air. The nanomaterials were deposited on the proposed optical microfiber sensors by use of dip-casting and drop-casting techniques to analyze their sensing performance.

1.4 Scope of the study

This research project covers the development and fabrication of Opto-chemical sensors starting from materials leading to optical sensing of NH_3 in liquid and gaseous form. NH_3 was chosen due to its applications in the industry and its effect on animal health. The scope of the project can be explained with the tree diagram shown in Figure 1.3. The red, blue, purple, and green colors represent the direction followed in this thesis to achieve the goal and objectives of the work. The red lines represent the initial design consideration of the developed sensors. Optical fiber was chosen as the transducing platform due its simplicity and high sensitivity. The purples lines represent the sensing principles and performances investigated for the NH_3-N liquid sensing, while the blue lines represent the sensing principles and performances investigated for the NH_3 gas sensing. In this, GO and ZnO were used due to their excellent optical properties and high surface area. These properties make them one of the most appealing materials for optical based sensing devices. The sensing parameters under investigation are sensitivity, response time, and limit of detection. These parameters were chosen because they are crucial in providing reliable sensors. The green represents the micro characterization techniques used in investigating the morphology and structures of the developed sensors. The black lines refer to other research areas that are beyond the scope of this PhD research.

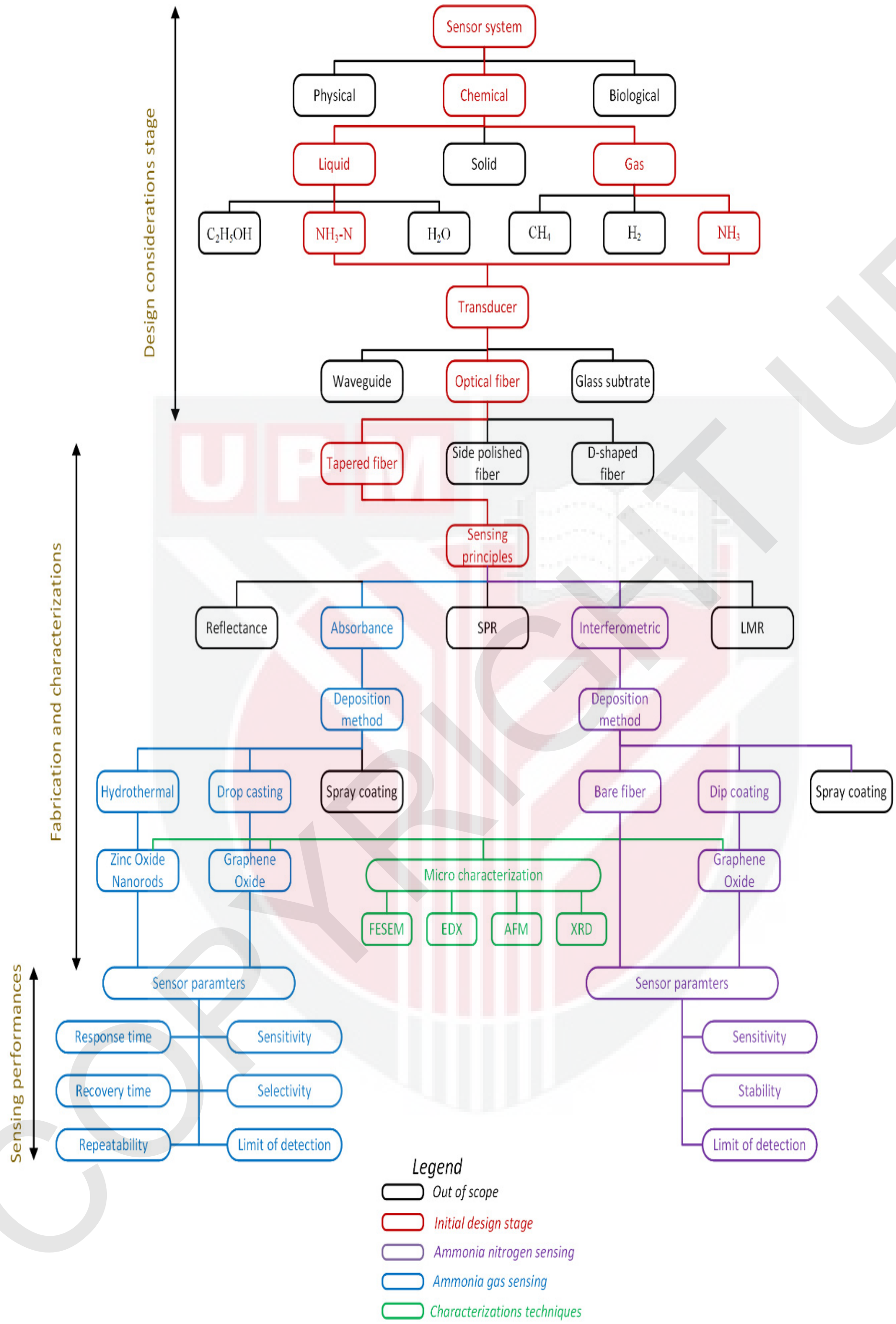

Figure 1.3: Scope of the study

This PhD project is limited only to experimental work.

1.5 Thesis Organization

This thesis is divided into five chapters and is outlined as follows:

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

Chapter Two presents the literature reviews that describe the rationale behind the project. It reviews optical fiber transducing platforms, optical fiber sensors, and their sensing principles. Different modification techniques of optical fiber are also presented and discussed. This includes a review of functional nanomaterials and their properties towards chemical sensing applications. The chapter also summarizes critical reviews of the latest works on NH_3 sensors in liquid and gaseous form.

Chapter Three covers the methodology and procedures used in implementing this project. This chapter gives a full description of the fabrication and design of the tapered optical microfiber sensors. The nanomaterials synthesis and deposition are also presented and discussed and followed by the measurement setups of the developed sensors.

Chapter Four presents the results and discussion on the outcomes of the research work and its findings. This chapter relates the characterization results of the optical microfiber structures and the deposited nanomaterials with the optical sensing performances. The sensing performance results and the sensing mechanism of the developed sensors towards liquid and gaseous NH_3 are also discussed in detail.

Chapter Five concludes the research findings with the outlined objectives for this PhD work. Future research to improve the developed sensors are also recommended in this chapter.

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LIST OF PUBLICATIONS

ISI Indexed Journals:

1. **S. H. Girei**, M. M. Alkhabet, Y. M. Kamil, H. N. Lim, M. A. Mahdi, and M. H. Yaacob, "Wavelength Dependent Graphene Oxide-Based Optical Microfiber Sensor for Ammonia Gas," Sensors, vol. 21, no. 556, pp. 1–11, 2021, doi: 10.3390/s21020556.

2. **S. H. Girei**, H. N. Lim, M. Z. Ahmad, M. A. Mahdi, A. R. M. Zain, and M. H. Yaacob, "High Sensitivity Microfiber Interferometer Sensor in Aqueous Solution," Sensors, vol. 20, no. 4713, pp. 1–9, 2020, doi: 10.3390/s20174713.

3. NH_3 Gas Sensing Properties of Tapered Optical Microfiber Coated with ZnO nanorods. (To be submitted)

4. Microfiber Interferometer Sensor Coated with Graphene Oxide for Ammonia nitrogen Sensing in Water. (To be submitted)

International Conference:

1. M. M. Alkhabet, **S. H. Girei**, S. Paiman, N. Arsad, M. A. Mahdi, and M. H. Yaacob, "Highly Sensitive Hydrogen Sensor Based on Palladium-Coated Tapered Optical Fiber at Room," 7th Int. Electron. Conf. Sensors Appl., pp. 3–8, 2020, doi: 10.3390/ecsa-7-08186.

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