UNIVERSITI PUTRA MALAYSIA

FLUORESCENCE QUENCHING OF OPTICAL FIBER CHEMICAL SENSOR DETECTING HAZARDOUS DINITROBENZENE MATERIAL

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By

ABDULLAHI MAGAJI YUSUFU

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

October 2017

DEDICATION

To my passed Parents,
my lovely Wife, Hajiya Asabe, and
my caring Children: Hauwa'u, Al-Amin, Mariya and Yusuf

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

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Chairman : Associate Professor Ahmad Shukri Bin Muhammad Noor,PhD
Faculty : Engineering

The consistent explosive and hazardous material monitoring is among the demanding aspect of security due to global terrorism activities in this era. Fluorescence sensing technology is among the best promising methods used for explosive detection nowadays because of its numerous advantages, which include simplicity of instrument, high sensitivity and short response time. This dissertation presents the development of optical fiber chemical sensors for detection of an explosive material 1, 4-dinitrobenzene (DNB) in acetone. Two polymers have been chosen to detect the explosive material. Chitosan polymer and conjugated polymer poly[2-methoxy-5-(2'-ethylhexyloxy)-p-phenylenevinylene (MEH-PPV) based optical fiber sensors are fabricated by modifying the fibers to enable a good formation of the polymer matrix on the sensing regions. First, a multi-mode fiber is tapered and the chitosan polymer is modified by cross-linking with glutaraldehyde to detect DNB in solution. The solution is deposited on the optical fiber tapered region to create a sensing portion using dip-coating method. Whereas, the conducting polymer MEH-PPV based sensors are developed by dip coating the tip of plastic optical fibers to detect the DNB as well. Unlike other fiber sensor developments, this study takes cognizance of fluorophore concentration in solvent for accessing the quenching ability of the analyte to improve sensitivity and performance of optical fiber sensors. Several chitosan and MEH-PPV polymer amount in solvents were characterized using UV-Vis and fluorescence intensity spectroscopy before coating processes. The morphology of cross-linked chitosan is done using scanning electron microscope (SEM). To characterize the changes in optical property due to modification of chitosan, ultraviolet (UV) laser and ultrafast femtosecond laser sources are used.

DNB concentration as low as 1ppm is detected when it was introduced to the chitosan based optical sensor. Time-dependent quenching results indicated that the sensor could operate in both linear and nonlinear regions with rapid quenching for nonlinear excitation source. For MEH-PPV based plastic optical fiber fabricated sensors, the developed devices are tested for DNB sensitivity and quenching ability with respect to time in terms of fluorophore amount in solvent. Preliminary results indicated that the initial fluorescence intensity generated increases with decrease of polymer/fluorophore concentration in solvent. This behavior is observed once more when sorted MEH-PPV samples are deposited as thin films on the plastic fiber tip to create a sensing region. When the sensors are introduced to 5ppm DNB in acetone for 10 seconds, there is a significant decrease of fluorescence intensity for sensor with thinner coating than that with thicker coating. This indicates that fluorescence of MEH-PPV can be quenched effectively in thinner films than in thicker films. All the developed sensors show high sensitivity and fast response with detection limit of 1.635ppm. The enhancement of sensing capability of DNB is explored using fluorophore amount in solvent and Stern-Volmer quenching analysis have been used to investigate their kinetic reaction.

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

PENDERIA KIMIA LIDAPAN PENDAFLOR GENTIAN OPTIC LAGI MENGESEN BAHAN MERBAHAYA DINITROBENZENA

Oleh

ABDULLAHI MAGAJI YUSUFU

Oktober 2017

Pengerusi : Profesor Madya Ahmad Shukri Bin Muhammad Noor, PhD
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Pemantauan bahan letupan dan bahan berbahaya secara konsisten adalah antara aspek yang amat penting dalam bidang keselamatan kerana aktiviti keganasan global dalam era ini. Teknologi penderia pendarfluor adalah antara kaedah terbaik digunakan untuk mengesan bahan letupan pada masa kini kerana banyak kelebihannya termasuk kesederhanaan instrumen, kepekaan yang tinggi dan masa tindak balas singkat. Disertasi ini membentangkan pembangunan penderia gentian optik kimia untuk mengesan bahan letupan 1, 4-dinitrobenzena (DNB) dalam aseton. Dua polimer telah dipilih untuk mengesan bahan letupan. Penderian gentian optik berdasarkan polimer Chitosan dan polimer konjugat poly[2-methoxy-5-(2'-ethylhexyloxy)-p-phenylenevinylene (MEH-PPV) direka setiap satunya secara berasingan dan dengan mengubah gentian tersebut untuk membolehkan pembentukan polimer matrik yang baik di kawasan penderiaan.

Pertama, bahagian tengah gentian mod-pelbagai ditiruskan pada klad dan kaedah salut celup digunakan bagi menyalut lapisan polimer chitosan dirangkai silangkan bersama glutaraldehyde. Manakala penderia gentian optik plastik berasaskan polimer konduktif MEH-PPV dibangunkan dengan cara yang sama pada hujung gentian optik plastik. Tidak seperti perkembangan penderia gentian yang lain, kajian ini menjurus kepada meningkatkan kepekaan dan prestasi penderia gentian optik dengan mengkaji tentang penlidapan pendaflor dengan mengubah kepekatan bahan pendaflor. Pengukuran ciri menggunakan UV-Vis dan spektroskopi keamatan pendaflor dijalankan kepada larutan chitosan dan larutan polimer konduktif MEH-PPV. Morfologi polimer saling rangkai chitosan dilakukan dengan menggunakan mikroskop pengimbas elektron (SEM). Cahaya ultraviolet (UV) dan laser femtosaat ultralaju digunakan bagi mengkaji sebarang perubahan ciri optik kepada chitosan tersebut.

DNB kepekatan serendah 1ppm dikesan apabila ia diperkenalkan kepada penderia optik berasaskan chitosan. Keputusan masa lidapan pedaflor penderia menunjukkan bahawa ia boleh beroperasi di kedua-dua rantau linear dan tak linear dengan lidapan pendaflor yang lebih pesat untuk sumber cahaya linear. Untuk penderia gentian optik MEH-PPV, peranti yang dibangunkan diuji untuk kepekaan DNB dan keupayaan masa lidapan pendaflor dari segi jumlah bahan pendaflor dalam pelarut. Keputusan awal menunjukkan bahawa keamatan pendarfluor dihasilkan meningkat dengan penurunan kepekatan polimer / bahan pendaflor dalam pelarut. Tingkah laku ini juga diperhatikan bagi penderia hujung gentian optik plastik dengan MEH-PPV. Apabila penderia diperkenalkan kepada 5ppm DNB dalam aseton selama 10 saat, terdapat penurunan ketara keamatan pendarfluor untuk penderia dengan salutan nipis berbanding dengan lapisan tebal. Ini menunjukkan bahawa lidapan pendarfluor MEH-PPV lebih berkesan untuk salutan nipis daripada dalam salutan tebal. Semua penderia yang dihasilkan menunjukkan kepekaan yang tinggi, tindak balas yang cepat dan masa pemulihan dan had pengesanan adalah 1.635ppm. Peningkatan keupayaan penderiaan DNB diterokai menggunakan jumlah bahan pendaflor yang digunakan dalam pelarut melalui analisis penlidapan pendaflor Stern-Volmer bagi menyiasat tindak balas kinetik mereka.

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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 Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

APF	Amplified Polymer Fluorescence
BE	Beam Expander
BS	Beam Splitter
BP	Beam profiler
CCD	Charge-coupled-Device
CW	Continuous wave
DNB	1, 4-Dinitrobenzene
DQE	DNB quenching efficiency
DNT	Dinitrotoluene
EDFL	Erbium doped fiber laser
EM	Electromagnetic
FL	Fluorescence
FRET	Fluorescence Resonance Energy Transfer
FWHM	Full Wave Half Maximum
GA–chitosan	Chitosan cross-linked with glutaraldehyde
GF	Green Filter
Glu	Glutaraldehyde
HRR	High Repetition Rate
IR	Infrared
LC	Liquid-crystalline
LOF	Lab-On-Fiber device
MEH-PPV	Conjugated polymer poly[2-methoxy-5-(2-ethylhexyloxy)-1,4-phenylenevinylene]

MMF	Multi-mode Fiber
NA	Numerical Aperture
NB	Nitrobenzene
NDF	Neutral Density Filter
NIR	Near Infrared Region
nm	Nanometer
NT	Nitrotoluene
PL	Photoluminescence
POF	Plastic optical fiber
PVC	Poly (vinyl chloride)
PMT	Photomultiplier Tube
PMMA	Poly(methyl methacrylate)
PET	Photo-Induced Electron Transfer
RFQ	Response after fluorescence quenching
RDX	hexahydro-1,3,5 trinitroazine
RL	Relay Lens
SEM	Scanning Electron Microscopy
SERS	Surface-enhanced Raman spectroscopy (SERS
TDQF	Time-dependent Quenching of Fluorescence
THF	Tetrahydrofuran
TIR	Total Internal Reflection
TCPOFs	Tip coated plastic optical fibers
TNT	Trinitrotoluene
UV	Ultra-Violet

CHAPTER 1

INTRODUCTION

1.1 Overview of Explosives

Nowadays, much attention is focused on consistent and precise method of detection of explosives and hazardous materials due to global issues concerning environmental pollution, homeland security and unexploded buried explosives in landmines as well as in military bases [1], [2]. For the sake of safety regard to national and environmental security, locating of hidden explosives within waters on earth and beneath, passenger luggage in airport or mails, commuter stations, public transports, recreational areas, hospitals, restaurants, beaches, laboratories as well as every relevant point is necessary and became significant dispute [3]. These emerging problems are due to the current situation of persistent worldwide terrorist activities [4]. Therefore, these reasons and so many others have inspired the necessity to continue scouting for a standstill sensitive, and low cost resources for real time monitoring and detection of explosives [5].

Meanwhile, detection of explosive is essential in forensic research preferably, when it comes to problems associated with health risks. For instance, when explosives residues are released from military sites or by any means, they can cause dangerous effects to all forms of lives [6]. A typical nitro-aromatic explosive is 1, 4-dinitrobenzene (DNB) originating from Trinitrotoluene (TNT) family [7]. It was observed that overexposure to even a very little quantity of these explosive materials can cause aneamia because of their ability to penetrate the skin and enter into the bloodstream directly resulting in liver problems and cataracts [8]. Obviously, a fast, sensitive and selective explosive detection enhances the facility to track and locate these analytes along with decreasing the continued loss of life of citizens and more importantly a quick alert regard to subsequent attacks by terrorists [9].

Any material either nuclear or chemical tenable for activation to take on a fast, and self-initial breakdown causing the creation of extra steady material, developing a rapid pressure effect and releases heat is an explosive by nature [10]. Explosives are categorized into different forms, such as low and high explosives. The smaller rate explosives are propellants, non-smoking ash, black dust, showpieces burn at moderately short degrees (cm s^{-1}), while great explosives explode on higher paces of (km s^{-1}). These are further categorized as shown in Figure 1.1 below.

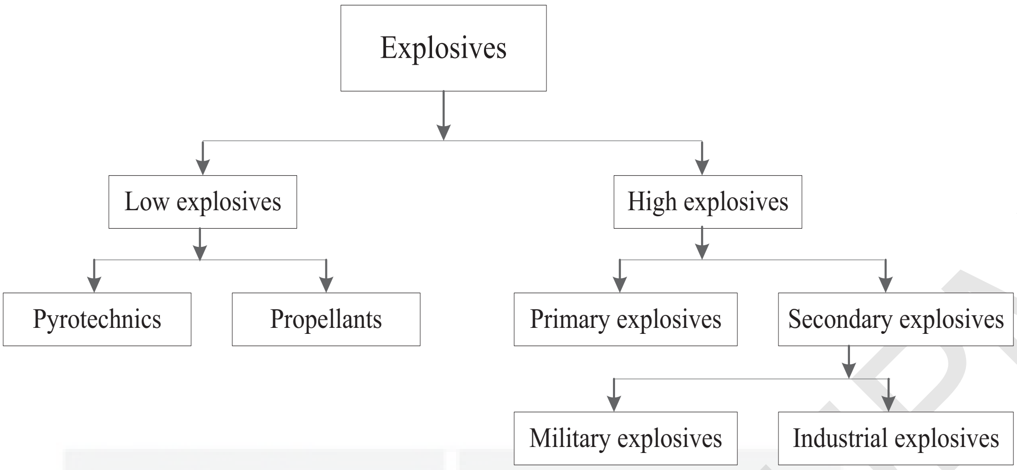

Figure 1.1 : Explosive grouping according to their structure and performance

Since detection of DNB is the focus of this dissertation, the discussion will be limited to secondary explosives among the high explosives category shown above which include nitroaromatics found predominantly by military locations. These remain dangerous and common explosives for current terrorist activities throughout the world. The secondary explosives are framed to detonate only under particular situations as leading control explosives. Their classification includes melt-pour explosives such as nitroaromatics, found on a folder and translucent explosive, like hexahydro-1,3,5 trinitroazine (RDX). For the secondary explosives, chemical reaction can spread with a hustle such that the range of response in the material can go beyond the rapidity of sound. There are several explosive detection schemes available such as the electrochemical method [11], the surface-enhanced Raman spectroscopy (SERS) [12], field-effect transistor [13], nanotechnology-based sensors [14], electrochemistry-based sensors [15], fluorescence quenching of conjugated polymers [16], semi-conductor-based gas sensors [17] among others. Because of its convenience, high sensitivity, easy visualization, low cost of sensor fabrication and the setup requires less materials, couple with its detection short response time, the fluorescence quenching method has recently become the most effective tool for sensing explosive materials [2], [3], [4], [18].

Chemical sensors based on optical fibers have gain ground in this type of research due to their unique characteristics such as low attenuation, inherently safe in chemically aggressive or explosive environments, resistance to radio frequency or electromagnetic intrusion, large bandwidth, wide dynamic range, high sensitivity and easy compliance for various sensing parameters on a particular platform [10], [11], [12], [14]. Further distinctive benefits of using optical fiber sensors is their low vulnerability to electromagnetic interference, electrically passive operation, light weight, miniature, geometrical versatility distributed sensing, and remote sensing capability where they can be used for explosive sensing application [15], [16], [17]. Hence, optical fiber sensors can easily be integrated into telecommunication systems and optical networks. Additionally, optical fiber sensors have the tendency to differentiate whether the analyte to be measured act outside (extrinsic sensing) or inside the fiber (intrinsic sensing).

1.2 Problem Statement

Although there has been a tremendous efforts recorded in terms of research concerning explosive and hazardous materials detection and monitoring using the aspect of sensing technology, but the demand for security against these analytes is still not met. That is why researchers are still investigating on ways and efficient methods for real time monitoring of these materials. For instance, many polymers are being modernized or synthesized and utilized either in liquid or solid thin films and mainly applied to detect the presence of explosives without accessing the effect of fluorophore content in a particular solvent before experimentation using fluorescence quenching sensing technique. This is important because fluorophore concentration plays a vital role in detecting analyte by fluorescence quenching method. Among the prevailing sensing technology usually used for this explosive detection is the use of optical fiber as sensing probe.

This thesis focuses on optimizing the chitosan copolymer for the first time in the authors' knowledge to trace the amount of DNB explosive using linear and nonlinear excitation laser sources to improve efficiency, sensitivity and performance. A multimode tapered fiber chitosan-based sensor is developed to do the work. Additionally, the work explores conjugated polymer MEH-PPV for the study of fluorophore weight/volume in solvent and how it influenced the quenching capability of DNB in solution. This analysis is extended to MEH-PPV thin films coated on plastic optical fibers and found to agree with the fact that higher polymer amount in solvent produces thicker coating, which results in slower and reduced quenching while less polymer amount in solvent facilitates quick and efficient quenching by DNB. This process increases device performance, sensitivity and fast detection of the analyte.

1.3 Research Motivation

Optical fiber sensor systems have been in use for sensing of chemical agents as reported [5] for over 50 years. A sensitive monitoring technique in fluorescence quenching process is successfully achieved when a fluorophore is evanescently excited. The resulting fluorescent radiation collected provides an extremely good result. For this type of technique, the fiber structure can simply be modified for a particular sensing function (for instance, intrinsic). Here, the optical fiber serves as a light guiding element within the core cladding and convey the generated signal to the detector. To achieve higher sensitivity and minimum interference, the development of an intrinsic sensor type to detect explosive material DNB is proposed for the work in this thesis.

Since there is a need to determine electron donating and accepting species, the optical fiber has to be modified in the cladding and by coating the fluorophore material along to serve as a sensing material. The light propagating in the core will penetrate into the cladding as evanescent wave and react with the usually coated polymer matrix within the sensing portion. The reflected light property from the cladding is then modulated into the optical fiber core by the polymer material. The sensing region can change the optical properties of light thereby bringing about change in the evanescent field in the cladding as well as the change in the light intensity in the fiber core.

A polymer material such as chitosan is not fluorescent but can be modified to produce fluorescence in solution and coated as sensing material on optical fiber. Similarly, a conductive conjugated polymer like MEH-PPV that is fluorescent can also be used directly in solution to coat the modified cladding of the fiber for function as a sensor. Initial fluorescence generated by both polymers after excitation can undergo increase or decrease of intensity due to introduction of an analyte such as DNB. The decrease of the fluorescence intensity is referred to as fluorescence quenching. The fluorescence quenching process can be used to trace the amount of analyte either in solution or in gas form. Thus, these polymers can act as sensitive materials towards DNB both in their solution form and when coated as thin films on optical fibers. The solutions of these polymers can also be utilized in cuvette to detect the analyte in solution by fluorescence quenching methods. In these ways, measurements of change of intensity is achieved either by steady state or by time-resolved quenching process.

The performance of these sensors depend on the parameters like type of a fiber, properties of the polymer matrix and the medium of sensing. The method used for coating, and coating thickness as well as the fiber sensor fabrication parameters are also considered. For sensing involving solutions, concentration of both polymer and the analyte play vital roles. Because these will form excellent material ambient stability and reduce random fluctuations of sensor signal because of small disturbance in the surrounding.

In this work, the parameters mentioned above were implemented to generate initial fluorescence intensity of chitosan and MEH-PPV polymers both in cuvette and by using coated Multi-mode tapered fibers as well as plastic optical fibers. Fluorescence quenching of these polymers take place when a chemical transducer converts the presence of analyte DNB to a change in optical information that lead to decrease in fluorescence intensity. These changes were achieved by varying both the analyte and the fluorophores concentrations in solvents and the coating thickness on the optical fibers. In this however, the intensity modulation in multi-mode optical fiber by chemical transducer that converts analyte presence to optical information has taken place. This process in turn leads to sensing by optical fibers. The intensity modulation is achieved by changes due to absorbance and conductivity of the transducer material owing to the presence of the analyte DNB.

1.4 Objectives of the Research

The main objective of this research is to develop an optical fiber chemical sensor intended for detecting 1, 4-dinitrobenzene explosive by fluorescence quenching method. The specified objectives are as follows:

1. To characterize the optical properties of chitosan polymer and MEH-PPV polymer.
2. To investigate and understand the dependence of fluorophore concentration in chitosan and MEH-PPV polymers towards the dinitrobenzene quenching capability.
3. To prepare and deposit polymers on multi-mode tapered fiber and plastic optical fiber.
4. To investigate the developed sensors performance using Stern-Volmer quenching analysis.

In order to achieve these objectives, the following research questions are outlined:

1. What types of fluorophore sensing layers can suitably be used to obtain response to explosive material DNB?
2. What characteristic of the polymer will influence the DNB quenching ability?
3. What features of multi-mode and plastic optical fibers can be manipulated to make it sensitive to the environment around it?
4. How can the performances of the developed sensors be understood and accessed?

1.5 Thesis Structure

This dissertation contains five chapters. Chapter One summarizes the background of the research and the set objectives on DNB explosive detection by fluorescence quenching using fiber optic sensors developed in this work.

Chapter Two underlined reviews on key areas of tapered optical fiber sensors, tip coated plastic optical fiber sensors and chemical sensors that utilized fluorescence quenching method. The study on fluorescence quenching, explosive material and significance for its detection via optical fiber sensors is elaborated.

Chapter Three discussed on the modernization and characterization of chitosan polymer with glutaraldehyde as a linking agent that is used as fluorescence materials for sensing DNB. This is followed by a thourough explanation on devices and materials characterization. Lastly, discussion on how chitosan-based multimode tapered optical fiber sensor is fabricated and used for detecting the nitroaromatic compound DNB in acetone solution within UV and NIR regions is enumerated.

Chapter Four focused on the results obtained from experiments of DNB detection by quenching fluorescence of MEH-PPV in numerous polymer and analyte concentrations in solvent. In addition, the fabrication and experimental results obtained from MEH-PPV polymer based plastic optical fiber sensors explained accordingly. The use of Stern-Volmer analysis for kinetic reaction for the results obtained also highlighted.

Finally, Chapter Five is the conclusion and discussion on the contributions of this work in the related research area and suggestions for possible future works.

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