

PLASTIC OPTICAL FIBERS AS GAS SENSORS

by

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ABSTRACT

PLASTIC OPTICAL FIBERS AS GAS SENSORS

This thesis is mainly about polyethylene glycol (PEG) coated simple optical fibers and their use as gas sensors. In this study, a simple plastic optical fiber is coated with gamma-isocyanatopropyltriethoxysilane end-capped polyethylene glycol (PEG) and experiments with different chemical compounds are performed to check how the refractive index of the coated cladding layer changes in accordance with the conditions imposed. An intrinsic, intensity-based evanescent wave gas sensor is constructed and responses of plastic optical fiber (POF) gas sensor to toluene, xylene, methanol and acetone vapors are observed. POF gas sensor gives a different response pattern for each vapor. POF gives the highest response to methanol due to a decrease in the refractive index of the PEG film. This effect reveals itself as an increase in the output voltage intensity for the case of methanol. For acetone, the POF gas sensor response pattern reveals the fact that there occurs a slight decrease in the refractive index of the coated region of the cladding and this causes a minor growth in output voltage and then, a sharp increase in the refractive index takes place and generates a strong downward movement in output voltage intensity. For toluene and xylene there follows a similar pattern to each other. A decrease in output voltage occurs due to an increase in the refractive index of PEG thin film.

ÖZET

PLASTİK OPTİK FİBERLERİN GAZ SENSÖRÜ OLARAK KULLANIMI

Bu tez genel anlamda polyethylene glycol (PEG) kaplı optik fiberler ve bunların gaz sensörü olarak kullanımı üzerine yapılan çalışmaları içermektedir. Basit plastik optik fiber PEG ile kaplanıp, değişik kimyasal gazlara maruz bırakıldığında, PEG kaplı filmin kırılma indisinin değiştiği gözlenmektedir. Oluşturulan gaz sensörünün toluen, ksilen, metanol, aseton gazlarına tepkisi ölçülmüştür. Fiber optik gaz sensörü her gaz için farklı bir tepki vermiştir. Fiber optik gaz sensörü en büyük tepkiyi metanol gazına vermiş ve metanole maruz kalan PEG filminin kırılma indisinde bir düşüş gözlenmiştir. Kırılma indisindeki bu düşüş, toplam ışık çıktısında bir artış olarak görülmüştür. Aseton için fiber optik gaz sensörünün tepki grafiğine göre aseton gazı PEG filminin kırılma indisinde küçük bir düşüğe yol açmış ve bu kendisini toplam ışık çıktısında bir artış olarak göstermiştir. Daha sonra aseton kırılma indisinde sert bir artışa sebep olarak toplam ışık çıktısında keskin bir düşüğe yol açmıştır. Toluen ve ksilen içinse birbirlerine benzer tepkiler oluşmuştur. Bu gazlar filmin kırılma indisinde bir artışa neden olmak suretiyle toplam ışık çıktısında düşüğe yol açmışlardır.

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LIST OF ABBREVIATIONS

PCS	Plastic clad silica
PEG	Polyethylene glycol
FOS	Fiber optic sensor
FOSs	Fiber optic sensors
VOCs	Volatile organic compounds
LED	Light emitting diode
POF	Plastic optical fiber
POFs	Plastic optical fibers
DOAS	Differential absorption spectroscopy

1. INTRODUCTION

Apart from their use in communication technologies, the popularity of optical fibers is increasing day by day. There is a growing interest towards optical fibers in sensor construction .

Detection of volatile organic compounds(VOCs) has a significance today. Many research groups are designing various types of optical fiber sensors towards environmental applications, electronic noses, food or chemical industry [1]. Those sensors can help to determine if the ripen process of a fruit is completed [2], or if the concentration of a chemical product in air exceeds the safety limits. For instance, researchers have used resistive sensors [3] to measure the swelling of the polymer; capacitive sensors [4,5] to measure changes in polymer permeability, just to mention a few.

Fiber optic sensors have more advantages than electronic sensors. Some of these electronic sensors need to be heated at 673K to detect VOCs [6] and at least all of them need to be biased to operate. Moreover, electronic sensors are not preferred in some explosive environments as chemical plants due the fact that they need an electrical signal to get modulated. Besides these, electronic sensors do not operate properly in the presence of electromagnetic interferences.

A fiber optic sensor is based on a sensing material. Material changes its optical properties, such as color or refractive index, when exposed to some chemical compounds. Choosing an appropriate sensing material is crucial in POF sensor construction [6,7].

In this study, polyethylene glycol is used as the sensing material. PEG is known to be highly hydrophilic in its response to water vapor. This feature of PEG makes it suitable in relative humidity detection. However, PEG has a stability problem. It dissolves easily at high concentrations of certain VOCs or water vapor. In order to make PEG more durable on plastic fiber surface, very low amount of gamma-

isocyanatopropyltriethoxysilane is mixed with PEG for this work [7]. In this study, an intrinsic, intensity-based evanescent wave gas sensor is designed and PEG is used as the reagent. Moreover, PEG responses to VOCs such as acetone, methanol, toluene, and xylene are determined.

This thesis contains three chapters. The first chapter is the introduction and it gives a brief summary of the study and states the aim of the study, and the necessity of it. The second chapter explains the theory of the study. The third chapter includes the experimental work and results. The final chapter consists of a brief conclusion on the study.

2. REVIEW

2.1. POF Description

An optical fiber is a cylindrical waveguide which consists of a core and a cladding with a lower refractive index than that of the core [8,13].

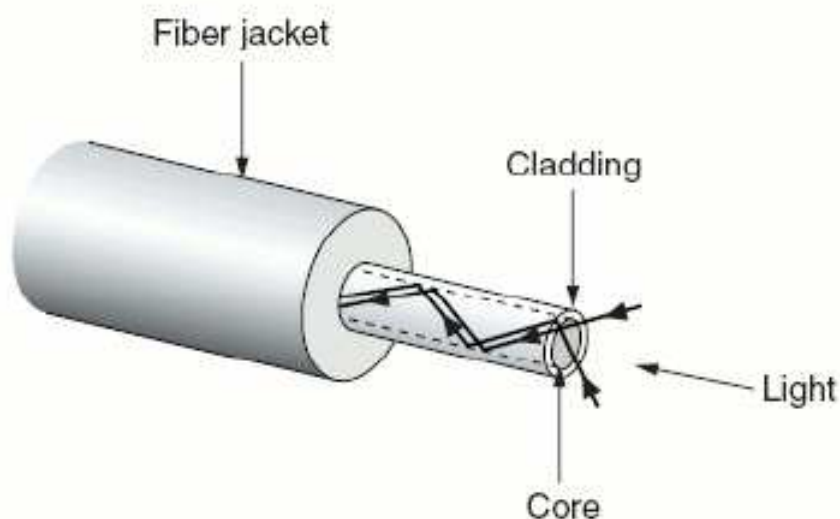


Figure 2.1. A general view of an optical fiber

In order for the light to stay in the core the index of refraction of the core must be greater than that of the cladding. Optical fibers can be examined in three categories [14]. These are fibers made of glass, plastic clad silica (PCS) fibers and plastic fibers. Glass (silica) fibers consist of single-mode step index fibers, multi-mode graded index fibers and multi-mode step index fibers. Plastic fibers are large in diameter and the signal is much slower compared to that of glass fibers. Plastic optical fibers (POFs) have many advantages such as low cost, light weight, high geometrical versatility and immunity to electromagnetic interference [14]. Plastic clad silica (PCS) fibers have a glass core and a plastic cladding. PCS fibers can be placed in between glass and plastic fibers in size and performance. PCS and plastic fibers are low cost fibers with respect to glass fibers. Glass fibers also far outweigh PCS and plastic fibers in signal transmission efficiency. Due to these reasons plastic and PCS fibers are generally used

in cars, sensors and short distance communication devices [15]. Optical fibers can be put into two categories according to the relationship between the indices of the core and the cladding. These are step index and graded index fibers. A step index fiber has a core of uniform refractive index and the index of refraction sharply decreases at the core-cladding boundary. Step index fiber has a high bandwidth and low losses. A graded index fiber, on the other hand, is one in which the index of refraction decreases with increasing radial distance from the central axis of the fiber. A light ray slows down when it passes close to the center and accelerates when it passes through the outer regions of the core in a graded index optical fiber. Light rays following a longer path through the fiber are called higher modes. In a graded index fiber higher order modes arrive at approximately the same time as the lower modes which travel closer to the central axis of the fiber. This is simply because lower mode light rays are slower due to the increasing refractive index towards the central axis. Optical fibers can be divided into three groups according to modal distribution profiles. These are multi-mode step index, multi-mode graded index and single-mode step index fibers. Multi-mode fiber means light can follow different paths through the core of the fiber. Light rays enter and leave the fiber at various angles [15,16].

2.1.1. Total Internal Reflection

Total internal reflection is a mechanism which enables optical-fiber communications possible. The attenuation in optical fibers can only be reduced to practical levels via the total internal reflection phenomenon. When light faces a discontinuity in the index of refraction, as it travels through an optical fiber, it refracts according to Snell's Law.

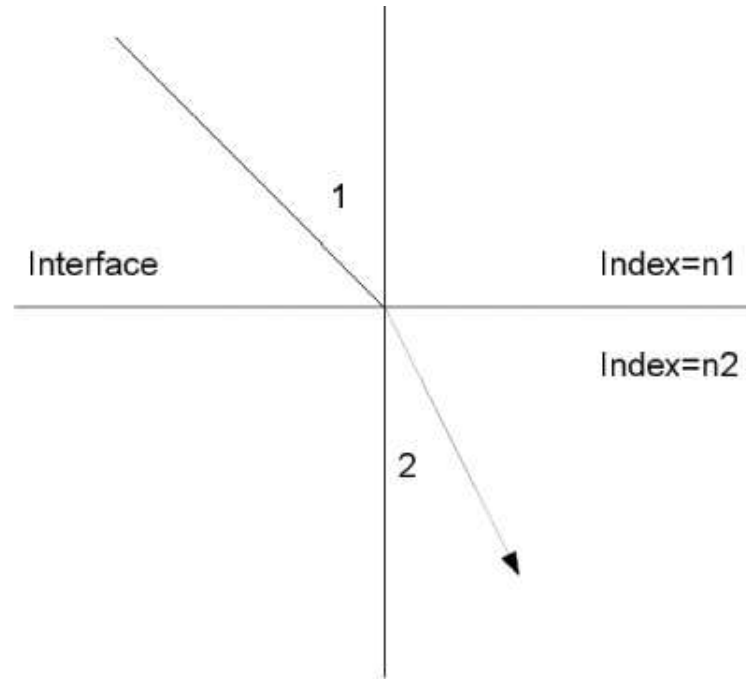


Figure 2.2. Snell's law of refraction

As it is seen from the Figure 2.2 light bends toward the normal when transmitting into a higher index and it bends away from the normal when transmitting into a lower index. Snell's Law states that

$$n_1 \sin \theta_1 = n_2 \sin \theta_2 .$$

Where θ_1 and θ_2 are the incident and the transmission angles respectively. Then the transmission angle θ_2 can be defined as

$$\theta_2 = \arcsin\left(\frac{n_1}{n_2} \sin \theta_1\right).$$

And using the formula above, it is possible to show that the total internal reflection occurs when the incident angle at the end face of the fiber is less than the critical angle defined as

$$\theta_c = \arcsin\left(\sqrt{n_2^2 - n_1^2}\right).$$

Equation above gives the critical angle of the fiber when n_1 is index of refraction of the core and n_2 is that of the cladding. The critical angle is the angle of incidence above which total internal reflection takes place. Critical angle is an important criterion

determining whether there can total internal reflection take place or not as light travels through an optical fiber. Since the index of refraction of the core in an optical fiber is greater than that of the cladding a ray hitting the interface with an angle greater than the critical angle conforms to the rules of the total internal reflection. Light rays entering the optical fiber within a cone defined by this critical angle are trapped in the fiber through internal reflection and they become guided rays. Light rays entering the fiber outside this cone on the other hand are attenuated by partial transmissions and lost from the optical fiber. The sine of this critical angle defines the numerical aperture of the fiber [16]. Numerical aperture is then defined as

$$NA = \sqrt{n_1^2 - n_2^2}.$$

2.2. Fiber Optic Sensors

Optical fibers can be used to sense properties such as moisture, temperature, flow, strain, etc. When they are designed to work as such sensing mechanisms they are called fiber optic sensors (FOSs). Working principle of fiber optic sensors is plain. Light from an optical source whose relevant optical properties remain constant is sent into a fiber by means of a stable coupling mechanism and guided to the point at which measurement is taken [17]. In other words, in a fiber optic sensor (FOS), one or more of the characteristics such as intensity, phase, frequency (color) is altered and correlated to an external physical or chemical parameter. There are different approaches in categorization of optical fiber sensors. The most common approach of all is subdividing them into two groups as intrinsic and extrinsic sensors. The criterion that they are named as extrinsic and intrinsic sensors is whether the transduction between the light and the measurand takes place in or outside the fiber [18]. According to the principle of operation both sensor groups can further be divided into two large categories of intensity-modulated and phase-modulated FOSs.

2.2.1. Extrinsic Sensors

In extrinsic type of sensors the optical fiber functions as a data transmission line. The fiber resembles a delivery and collection system. The propagating light leaves the fiber, interacts with the environment and is collected again by the same (or another) fiber. The alignment of the input and output fibers is crucial in these sensors.

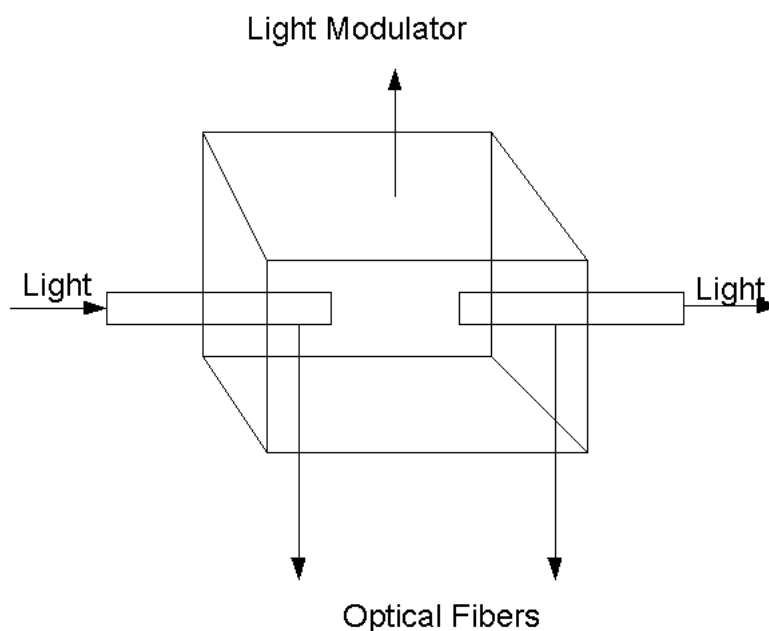


Figure 2.3. An extrinsic FOS configuration

The figure above illustrates an extrinsic FOS configuration. The basic components are simple as a light modulator and two pieces of optical fibers. As seen in the figure, light is taken to a modulation region using by means of an optical fiber and modulated there in by a physical, chemical or biomedical phenomena, and the modulated light is transmitted back to a receiver, detected and demodulated [18,19].

2.2.2. Intrinsic Sensors

Unlike extrinsic sensors, in intrinsic sensors, the fiber itself acts as a sensing medium. A chemical dye usually acts as an interface between the light and environment. In this case optical signal either passes through the dye and is collected by a

receiving fiber or it is reflected from the dye interface and coupled back into the same fiber.

2.2.3. Evanescent Wave Intrinsic Sensors

In this type of sensors there is a segment of the fiber that acts as the sensing area so there is no open path between the source and the detector. The cladding layer of the optical fiber is removed sensitively and coated with a chemical dye which acts as an interface between the light and the environment. So, any change in the optical and structural characteristic of the chemical dye due to the vapors sent into the environment of the fiber provokes a change in the effective index of the optical fiber, changing its transmission properties. The sensitivity of these sensors is dependent on the optical power transferred into the evanescent field and also the penetration depth of these waves into the sensing cladding. If the modified (coated or changed) cladding has a lower index of refraction than the core, then the total internal reflection takes place [20].

2.3. Intensity-Modulated Sensors

The working principle of intensity-modulated FOSs depends on detecting the variations of the intensity of the light associated with the perturbing environment. In other words, some kind of perturbation (physical, chemical or biomedical) interacts with the fiber or a mechanical transducer attached to the fiber. And the perturbation changes the intensity of the received light. This change due to the environmental perturbation is a function of the phenomenon being measured. To clarify this, intensity-modulated sensors detect the amount of light that is a function of the perturbing environment. The light loss may be due to transmission, reflection, microbending concepts as well as other phenomena such as absorption, scattering or fluorescence. Transmission, reflection, and microbending sensor concepts are the most commonly used ones with intensity-modulated sensors [19,20].

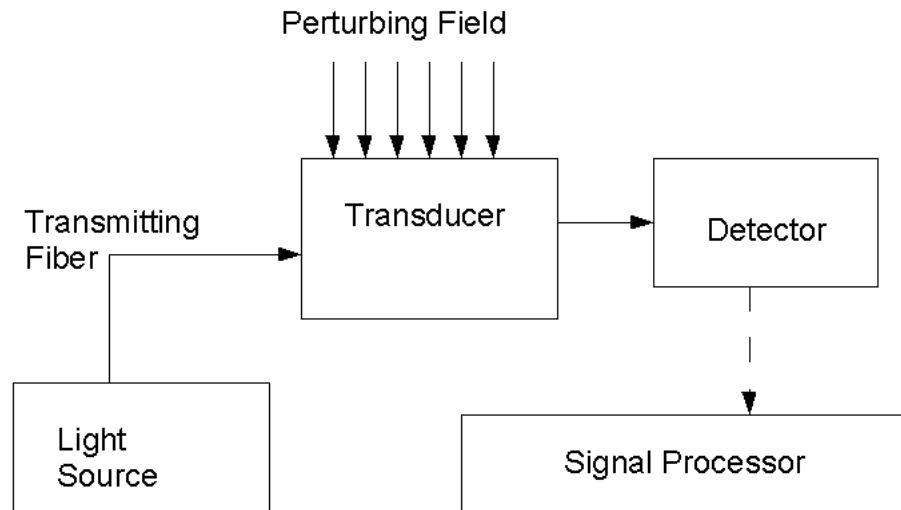


Figure 2.4. The configuration of an intensity-modulated FOS

2.4. Phase-Modulated Sensors

The working principle of Phase-Modulated FOSs is based on comparing the phase of light in a sensing fiber to a reference fiber, which is in an interferometer. In general, a phase-modulated sensor has a coherent laser light source and two single mode fibers. The light is split and directed into two separate fibers. These fibers are capable of measuring the phase difference with an extreme sensitivity. One fiber is exposed to measurand field and is called the sensing fiber. Another fiber is isolated from the surrounding and is called the reference fiber. When the environment perturbs one of the fibers a phase shift takes place. This phase shift is detected very precisely by an interferometer. There are four different configurations for interferometers. These are the Mach Zehnder, the Michelson, the Fabry-Perot, and the Sagnac interferometers. Compared to the intensity-modulated sensors phase-modulated sensors are much more accurate. However, they are generally more expensive [19,20].

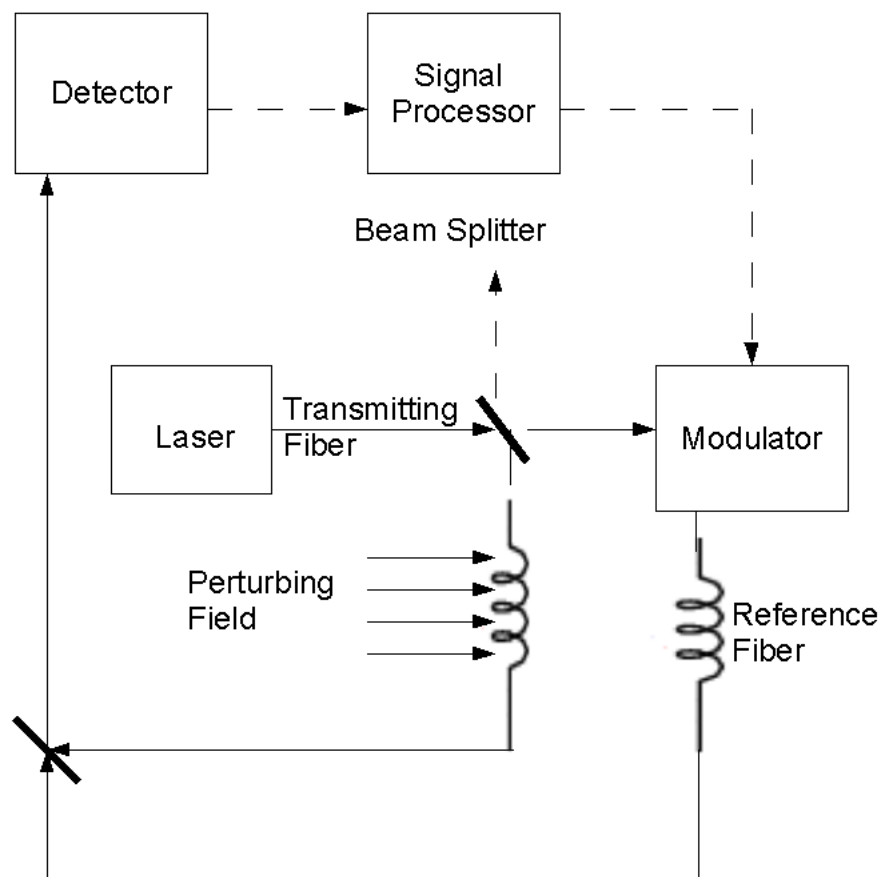


Figure 2.5. The configuration of a phase-modulated FOS

2.5. Fiber Optic Gas Sensors

Optical fibers can be used as gas sensing devices. There are several reasons for the fact that optical fiber gas sensors are more popular than electronic gas sensors. Some of electronic sensors need to be heated at 673K so as to detect gases or organic vapors. Furthermore, an electric signal is needed to get the sensor head modulated, so they are not preferred in some explosive environments like chemical plants. In addition to these, electronic sensors are vulnerable to electromagnetic interferences. FOS, on the other hand, are not only immune to electromagnetic interferences but also much cheaper than electronic sensors [8]. POF gas sensors can be configured in different ways. They can be designed as extrinsic or intrinsic as well as intensity modulated or phase modulated. They can also be designed to employ reflective or transmissive fiber optic configurations. No matter how they are classified or which type of sensors they

are POF gas sensors, in general, make use of four techniques which are fluorescence, scattering, absorption and refractive index change.

2.5.1. Fluorescence-based gas sensors

In this type of sensors sensing parts are made of fluorescent materials. As the light goes through the fiber it hits the sensing fluorescent material and the fluorescent target gives off a characteristic emission. When the fluorescent sensing target interacts with the sample gas, a change in the emission of fluorescent target is used as the sensing response. The fluorescent technique can be applied by using different configurations such as transmissive or reflective. Fluorescence sensors are widely used in detecting oxygen [18]. So as to detect oxygen a fluorescent dye which is quenched in the presence of oxygen is used [19]. Fluorescent technique can also be used in evanescent wave sensors. In this type of sensors evanescent wave interacts with the fluorescent coating material and a resulting low-level fluorescence is scattered in all directions. Using evanescent wave sensors with fluorescent technique is inefficient because only those rays that fall within critical angle for total internal reflection can be used for sensing [19].

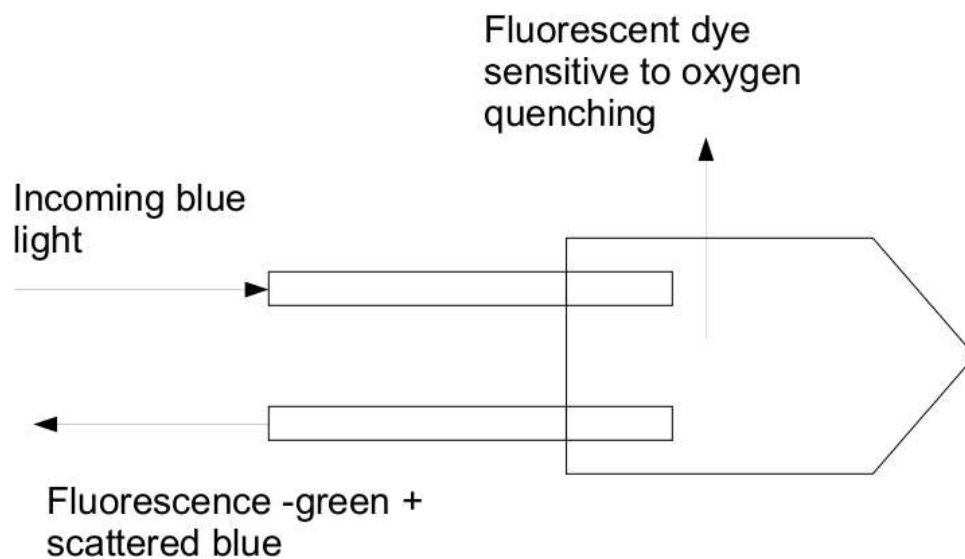


Figure 2.6. Oxygen detection using fluorescent dye

2.5.2. Absorption-based gas sensors

Absorption-based gas sensors rely on the phenomenon that gases have characteristic absorption bands [7]. Gas species absorb light at characteristic wavelengths and for each molecule there exists a unique absorption band [9]. For weakly absorbing gases, the light path passing through the gas is set long in order to increase the sensitivity. A faceted target allowing multiple reflections through the material being analysed can also be used to enhance the sensitivity. This type of sensors are used in a wide range of areas. Absorption gas sensors based on ultraviolet (UV) differential absorption spectroscopy (DOAS), for instance, are used in the automotive sector [9]. Besides their superior features, absorption-based gas sensors have a major limitation that optical fibers can only transmit between the UV and mid IR. That is, the primary peaks of many gases are beyond the range of present fiber optic systems [19].

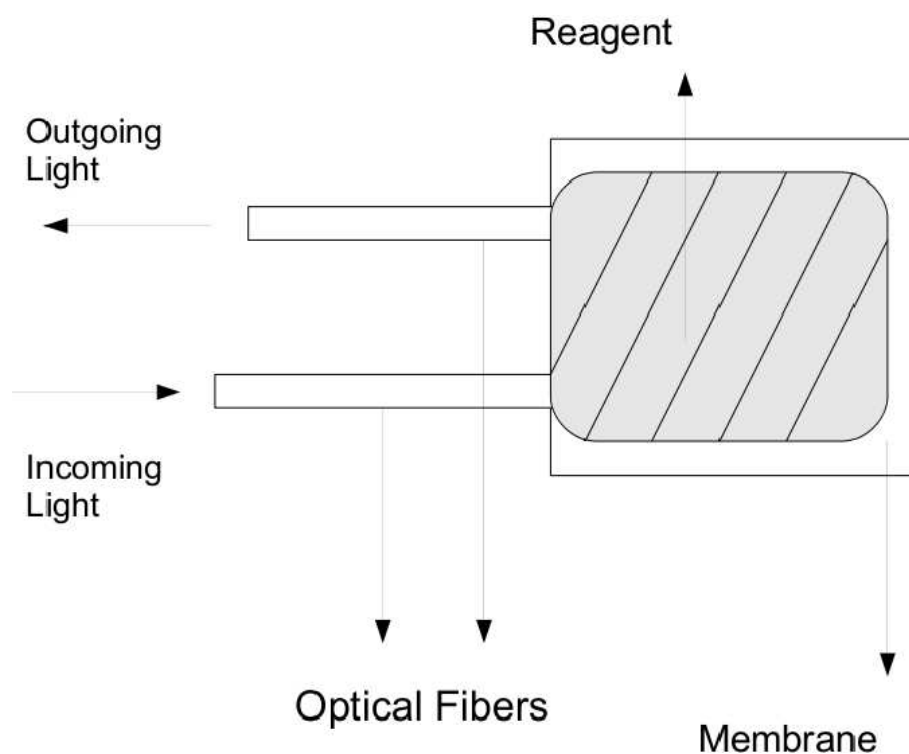


Figure 2.7. Absorption-based sensor configuration sample

The figure illustrates an example of absorption-based gas sensor sample. The sensing material is called reagent. The reagent is an immobilized dye which changes colour in the presence of the gas [19].

2.5.3. Scattering-based gas sensors

Scattering-based sensors are grounded on the process of sending light through a gas sample and collecting the scattered light back. After the spectrum analysis of the collected light the concentration change of the gas sample is determined. Raman laser scattering, for instance, is used to detect small concentrations of various gases. The system employs a high intensity monochromatic light source coupled to the transmitting fiber that illuminates the gas sample. The receiving fiber collects the Raman-scattered radiation that is characteristic of the particular gas being measured [19].

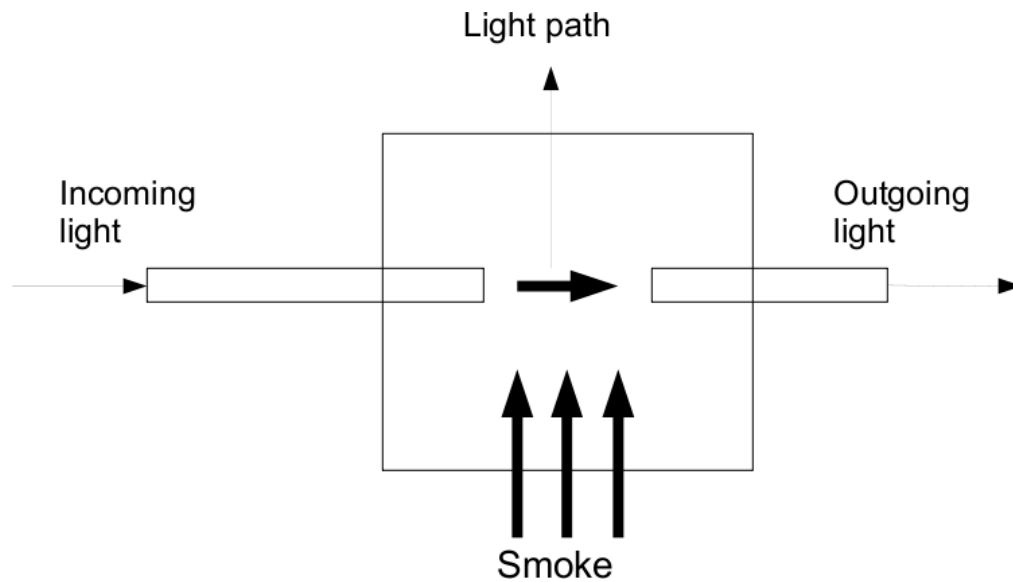


Figure 2.8. A sample configuration for scattering-based sensors

Figure 2.9 shows a sample configuration for a scattering-based POF gas sensor. Light hits vapour molecules and excites them. Due to the excitation of vapour molecules, a specific radiation is emitted and this radiation is collected.

2.5.4. Refractive index change-based gas sensors

Refractive index change is another property used in fiber optic(FO) gas sensors. The working mechanism of these sensors depends on the index of refraction change of the reagent.

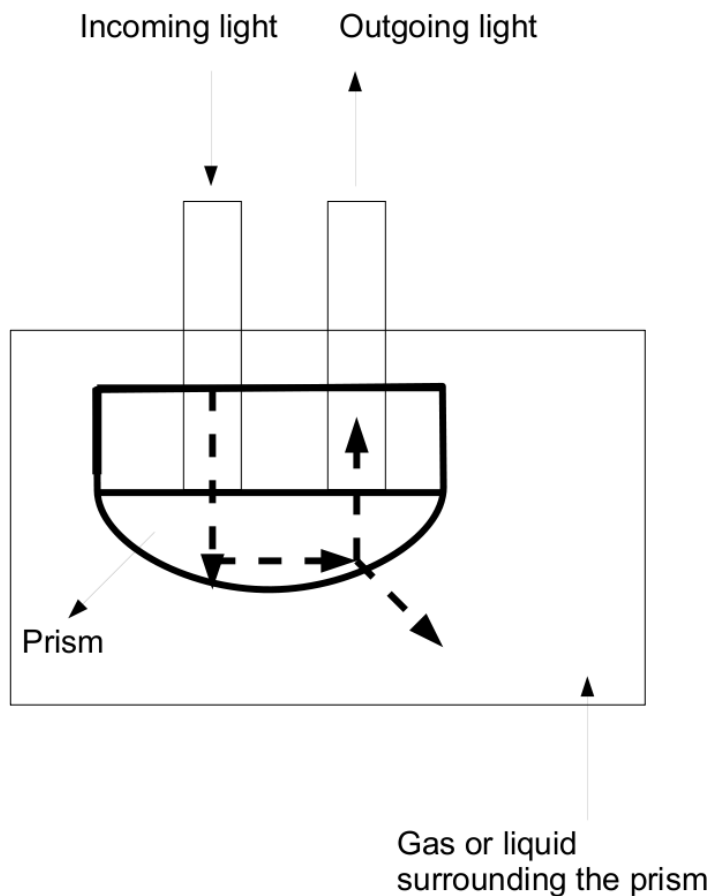


Figure 2.9. A refractive index change-based gas sensor configuration

Figure 2.9 depicts A fiber optic gas sensor which makes use of the index of refraction change. Light is transmitted through a fiber. Two parallel optical fibers are coupled to a prism and the prism acts as the sensor head. The prism is isolated and through gas or liquid injection the concentration of the atmosphere around the prism is changed. As the concentration changes around the prism the index of refraction changes as well. Then the amount of outgoing reflected light varies in accordance with the medium of the prism [19].

3. EXPERIMENTAL

3.1. POF Preparation

As it is mentioned in the theory section, there are various POF sensor configurations. In this work, we design an intrinsic, intensity-based evanescent wave gas sensor. We use PEG as the sensing material, and we use a POF with the following features:

Core Material	Polymetyl-Methacrylate
Cladding Material	Fluorinated Polymer
Core refractive index	1.49
Numerical Aperture	0.5
Refractive index profile	Step Index
Core Diameter	1.470 μm
Cladding Material	1.500 μm
Approximate weight	2,2 g/m

Table 3.1. Plastic optical fiber features

A certain cladding region of the POF surface about 2cm is gently removed with a knife. Then using the method of dip coating, the POF is coated with PEG reagent. Dip-coating is vital to ensure that POF is coated uniformly. In this method, the plastic optical fiber is stabilised vertically within the coating solution and the solution is allowed to flow out of the gas tube gradually from the bottom end. After dip-coating, one further step is left to accomplish the coating process. This is, leaving the PEG coated POF in an oven at 323K for two hours. During this process, the solvent of the PEG , which is water, evaporates and the POF coating process is accomplished.

3.2. Optical Setup

The experimental setup is depicted in figure 3.1. The setup consists of a bell jar vacuum chamber and a vacuum pump to evacuate the bell jar before data taking. A POF gas sensor is set into vacuum chamber which contains a POF with sensor head inside the bell jar. Besides these, there is a glass reservoir containing chemical compounds in liquid phase which can vaporise quickly. An infrared light-emitting diode (LED) is used as the guiding light source in the POF. A power supply provides the LED with a voltage of 5V. A photodetector (Thorlabs PDA55) is used to measure the intensity of the light leaving the bell jar. As the last component of the setup there is a manometer(Lutron PM-9100) to measure the pressure inside the bell jar due to vaporising compounds coming from the glass reservoir into to the vacuum chamber.

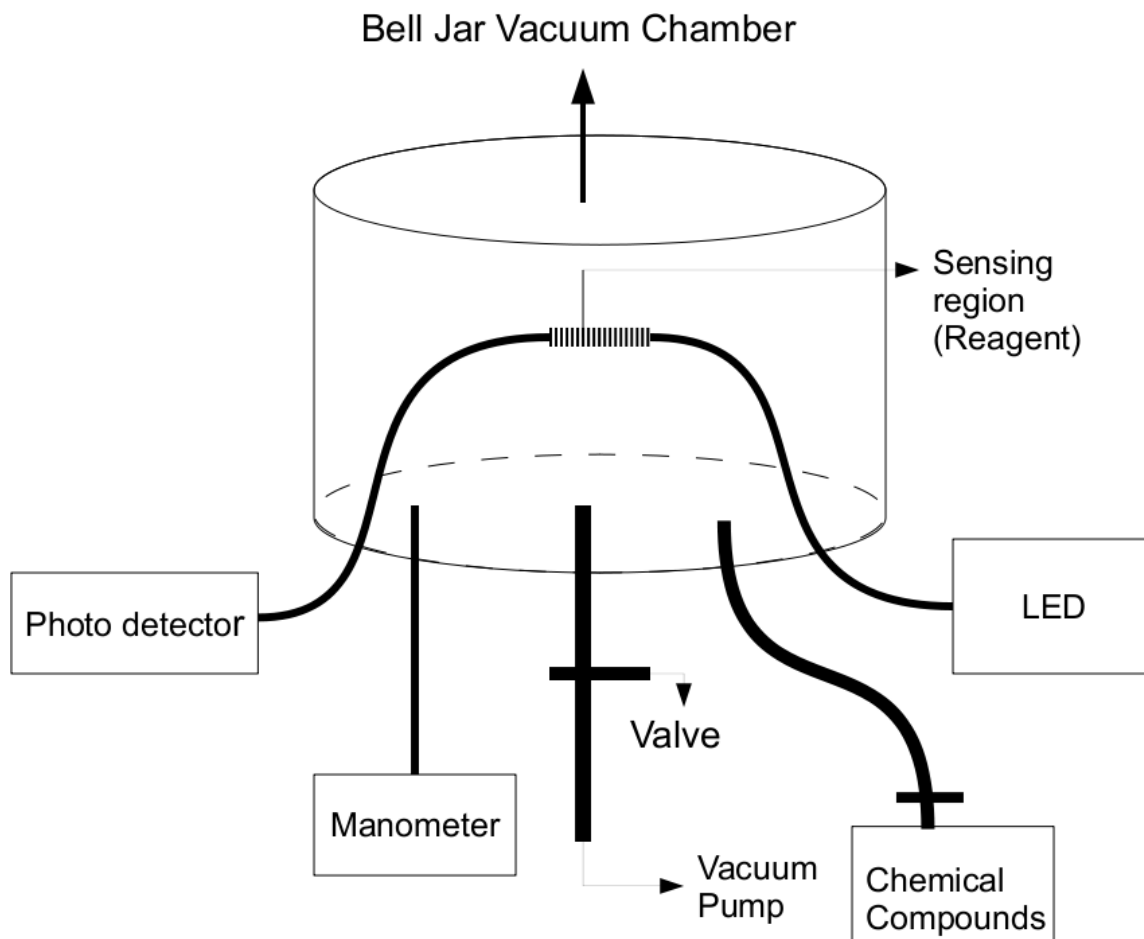


Figure 3.1. Setup

Before the data taking process, the bell jar is evacuated with a vacuum pump, and light is sent through a plastic optical fiber from a LED. At this point, there exists no gas inside the bell jar. Light is guided through the vacuum chamber, and the output intensity of light is measured to be constant initially via the photodetector, as there is no gas to effect the sensing mechanism initially. At this stage, the valve of the vacuum pump is closed and the valve of the glass reservoir containing easily vaporised liquid phase compounds is opened gradually. Then liquid phase chemical compounds vaporise quickly and fill into the bell jar vacuum chamber. Vapor phase chemical compounds surrounding the sensing matter PEG change its refractive index properties, and output light intensity measured by the photodetector shows variations. This intensity may increase or decrease depending on the effect of the gas on PEG. Finally output intensity versus gas pressure inside the vacuum chamber data are taken manually. This finishes the data taking process. This setup is superior to those measuring effect of the gas on PEG-Si without a vacuum chamber. The vacuum chamber has a crucial role in this setup because in order to observe the exact effect of the gas on PEG, the reagent must be isolated first. Otherwise one can not be sure of whether other factors, such as humidity, change the correct data pattern or not.

3.3. Results

3.3.1. PEG response to acetone

Various gases are used to test the POF gas sensor mechanism with vapor phase solvents. Liquid phase acetone(CH_3COCH_3) is one of them. Acetone, in liquid phase, is introduced into the glass reservoir. When the bell jar is evacuated, the reservoir valve is opened gradually and liquid phase acetone is vaporised quickly as it moves into the chamber and surrounds the PEG thin film, which is coated on the POF.

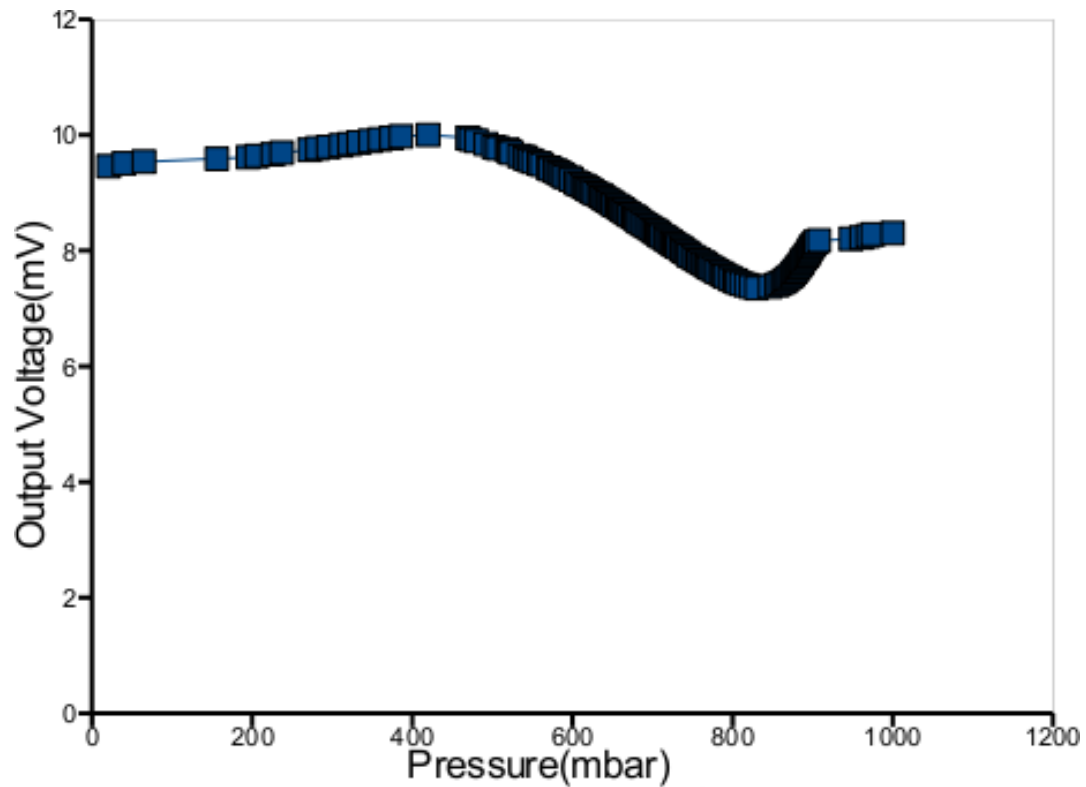


Figure 3.2. Output voltage vs pressure graph

Figure 3.2 shows the PEG response to acetone vapor. As acetone fills into the chamber, the pressure inside the chamber increases gradually. A manometer records the pressure data. There are three parameters playing an overwhelming role in the response pattern of the polymer to the applied gas. These are concentration of the gas, type of the gas and type of the polymer [10].

3.3.2. PEG response to methanol

An identical procedure is followed for the case of methanol. Figure 3.3 shows the response of PEG to the methanol vapor.

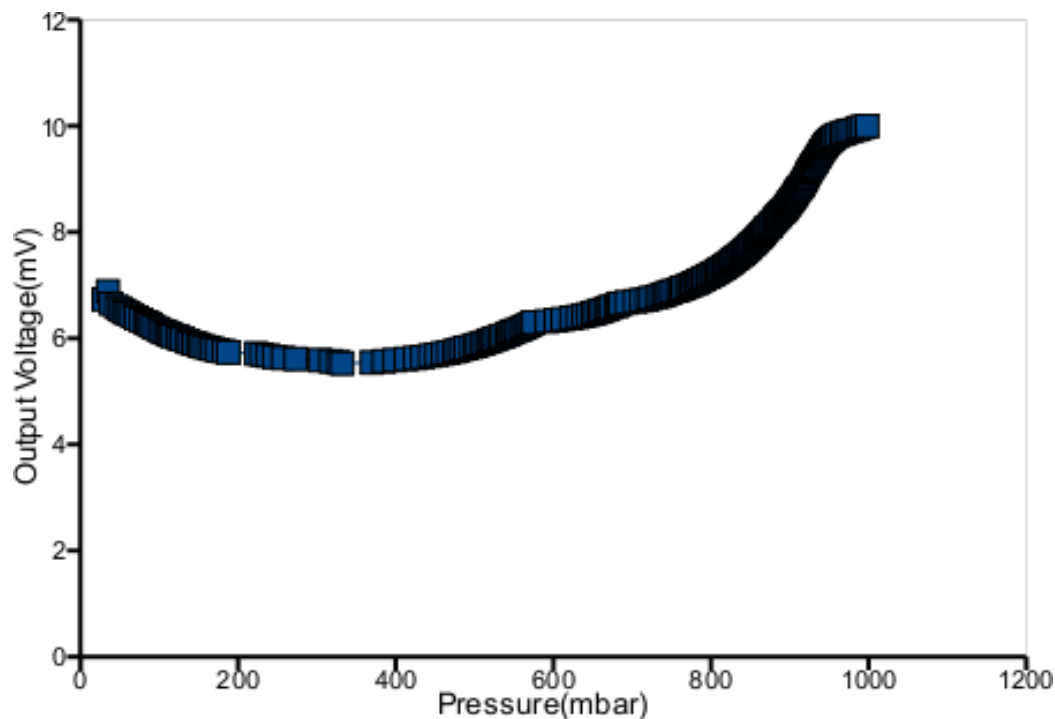


Figure 3.3. Output voltage vs pressure graph

The fact that PEG is soluble in methanol plays an important role in the response pattern of PEG to methanol. The refractive index of methanol is 1.3284; and the refractive index of the POF core is 1.49. The refractive index of PEG is slightly less than that of the core, which is 1.445. Therefore, as the methanol vapor is sent into the vacuum chamber, the refractive index of PEG decreases, and POF functions as a wave guide, and hence, the output voltage increases. POF sensor response to methanol far outweighs the responses shown to other vapors. This result is related to the fact that PEG is more sensitive to alcohols than aromatic hydrocarbons or ketones. An important factor contributing to methanol penetration into PEG is that PEG and methanol have polar characteristics. Since methanol is the compound with the highest polarity, it is easier for it to penetrate into PEG. As methanol diffuses into PEG, its lower refractive index causes a decrease in the effective refractive index of the PEG. This causes an increase in the output voltage. PEG has a hydrophilic characteristic. Moreover, PEG has an inclination to form hydrogen bonds with numerous species including methanol. As methanol vapor gathers around the PEG sensing film, it quickly forms reversible hydrogen bonds with the OH groups of the PEG. This explains the

decrease in the refractive index of the cladding layer, which, in turn, causes an increase in the output voltage [11].

3.3.3. PEG response to toluene

The experimental response pattern of PEG sensing film to toluene is given in figure 3.4. As seen from the figure, fiber output voltage decreases with increasing amount of gas gathering inside the vacuum chamber. In order to get this pattern, the refractive index of the cladding layer which consists of PEG should decrease. Toluene(C_7H_8) is an aromatic hydrocarbon. The refractive index of toluene is 1.4960. This is very close to the refractive index of the core, which is 1.500.

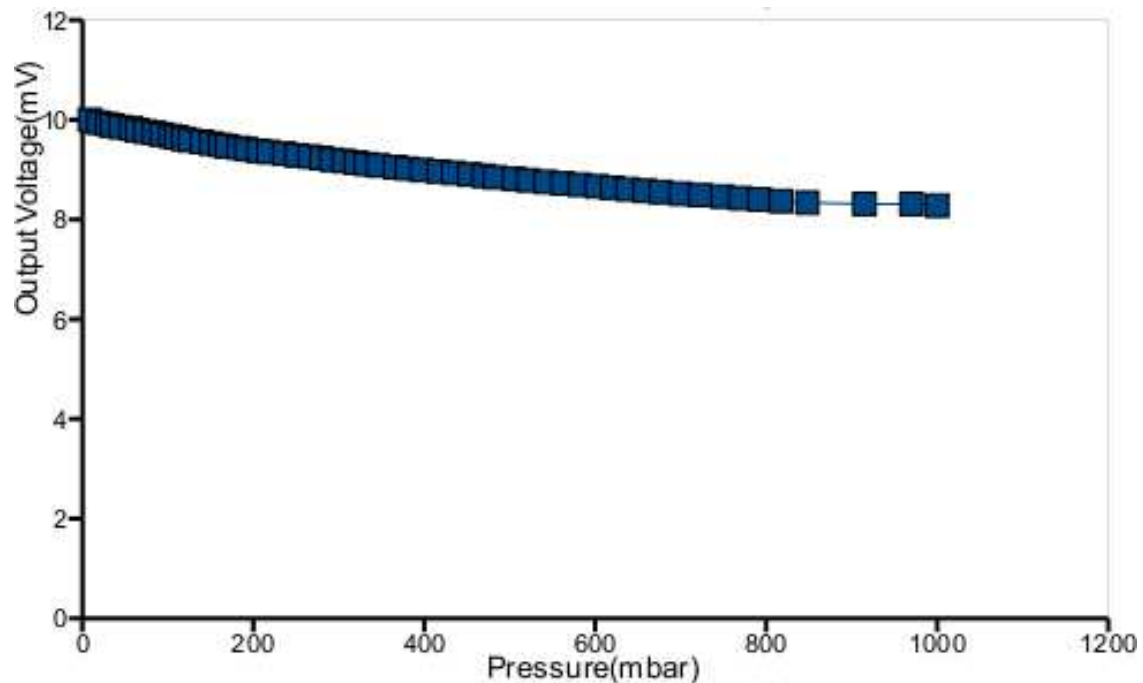


Figure 3.4. Output voltage vs pressure graph

Although we see a response to the toluene vapor, this response is attained after waiting long enough for the adequate concentration of toluene to gather around PEG. Toluene is a non-polar cyclic hydrocarbon, and therefore is unlikely to diffuse into PEG, which is polar. Furthermore, toluene has a ring structure and this is another factor making it harder to penetrate into PEG compared to methanol and acetone. As

toluene diffuses into PEG, it increases the density of the PEG sensing film and due to this increase of density in the cladding layer, POF turns into a leaky type and output voltage intensity decreases.

3.3.4. PEG response to xylene

As shown in figure 3.5, PEG response to xylene vapor follows a similar pattern to that of toluene with a difference.

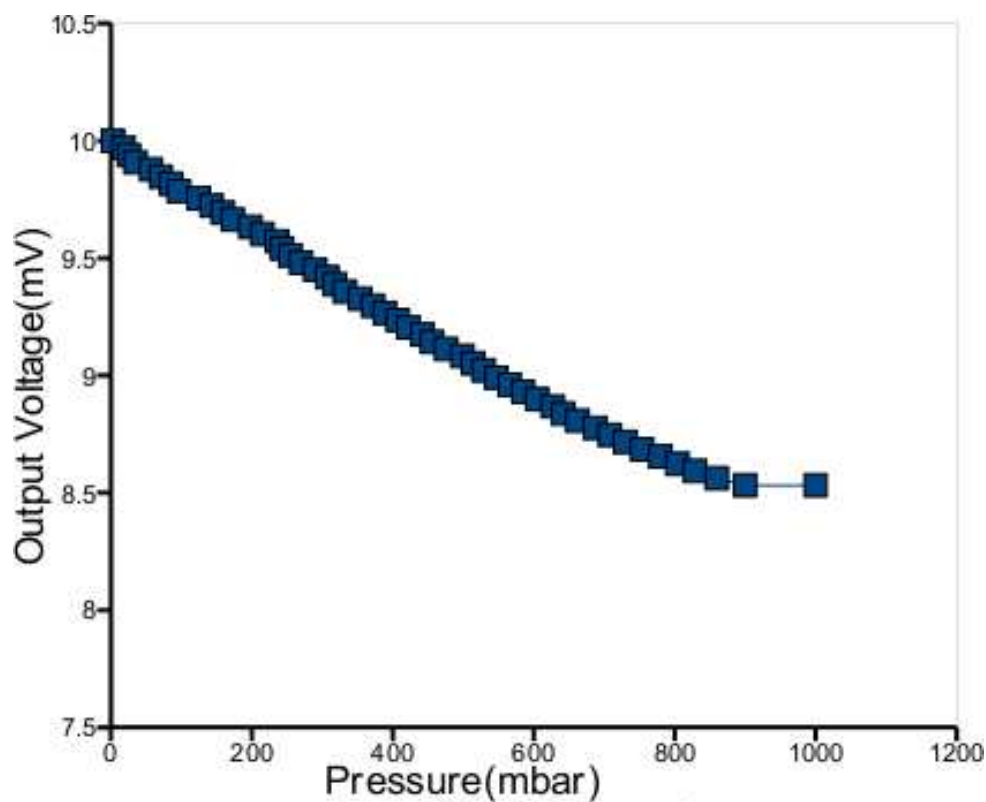


Figure 3.5. Output voltage vs pressure graph

The difference is that with increasing pressure, the xylene effect on output voltage is achieved at lower concentrations compared to toluene. Xylene (C_8H_{10}) with refractive index of 1.500 is an aromatic hydrocarbon like toluene. The change in PEG response pattern of xylene with respect to toluene may be due to its higher refractive index. The refractive index of xylene which is higher than toluene is equal to that of the core. Because toluene has a bigger molecular structure than xylene, we expect xylene

molecules to penetrate into PEG harder and slower than toluene molecules do. But this is not the only factor having a role in the determination of this response pattern. The refractive index of xylene is higher than that of toluene and if the effect of this factor surpasses, the former one POF gas sensor may have a response pattern shown in figure 3.5 for xylene.

4. CONCLUSIONS

In this study, an intrinsic, intensity-based evanescent wave gas sensor is designed. Using the setup described in the experimental section, responses of the POF gas sensor to acetone, methanol, toluene, and xylene are investigated. The factors contributing to the different response patterns are analysed and the possible reasons are suggested. It is observed that the evanescent wave POF gas sensor gives the highest response to the methanol vapor. This may be linked to the fact that both methanol and PEG are polar, and this allows methanol to penetrate into PEG easier compared to other chemical compounds. Another factor contributing to the penetration ability of methanol into PEG is that methanol is a short chain compound. Methanol diffuses into PEG by means of reversible hydrogen bonds. These bonds are not of strong type, and as the methanol concentration around PEG decreases or the bell jar is evacuated, methanol can easily leave PEG. The response patterns of xylene and toluene show similarities and a decrease in output voltage is observed. Xylene and toluene are long chain chemical compounds and they are unlikely to penetrate into PEG as effectively and fast as methanol does. Xylene and toluene cause an increase in the refractive index of the cladding which, in turn, generates a decrease in output voltage. In the case of acetone both an increase and a decrease are observed in the output voltage intensity. Initially, there occurs an increase in the output voltage intensity and then a decrease takes place. These changes in output voltage intensity correspond to an initial decrease and a following increase in the refractive index of the PEG thin film. PEG, giving different responses to each volatile organic compound proves to be a reliable sensing material in this study. As a future study, POF gas sensor responses to volatile organic compounds can be verified by using the thin film setup integrated to the vacuum chamber.

REFERENCES

1. Bariain, C., *et al.*, "Behavioral experimental studies of a novel vapochromic material towards development of optical fibre organic compounds sensor", *Sensors and Actuators*, pp.25-31, 2001.
2. Schweizer-Beberich, P.M., *et al.*, "Characterisation of food freshness with sensor arrays", *Sensors and Actuators*, pp.282-290, 1994.
3. Lonergan, M.C., *et al.*, "Array based vapor sensing using chemically sensitive carbon black-polymer resistors", *Chem. Mater.*, pp.2298-2312, 1996.
4. Endres, H.E., *et al.*, "A capacitative CO_2 sensor system with suppression of the humidity interference", *Sensors and Actuators*, pp.83-87, 1999.
5. Delapierre, G., *et al.*, "Polymer based capacitative humidity sensor: characteristics and experimental results", *Sensors and Actuators*, pp.97-104, 1983.
6. Grate, J.W., B.M. Wise, M.H. Abraham, "Method for unknown vapor characterization and classification using a multivariate sorption detector", *Anal. Chem.*, pp.4544-4553, 1999.
7. Yogun, H.U., Y. Ercil, Y. Menciloglu, N. Inci, "Coating material and a fiber optic sensor in which this coating material is used", *European Patent*, Publication No.WO2005090253, 2005.
8. Elousa, C., C. Bariain, *et al.*, " Volatile alcoholic compounds fibre optic nanosensor ", *Sensors and Actuators*, pp.444-449, 2005.
9. Dooly, G., C. Fitzpatrick, E. Lewis, "Optical sensing of hazardous exhaust emissions using a UV based extrinsic sensor", *Energy*, pp.657-666, 2007.
10. Bariain, C., *et al.*, "Optical fiber sensors based on vapochromic gold complexes for

- environmental applications”, *Sensors and Actuators B*, pp.535-541, 2005.
11. Wright, Y.J., A.K. Kor, Y.W. Kim, C. Scholz, M.A. George, ”Study of microcapillary pipette-assisted method to prepare polyethylene glycol coated microcantilever sensors”, *Sensors and Actuators B*, pp.242-251, 2005.
 12. Morisawa, M., *et al.*, ”Plastic optical fibre sensor for detecting vapour phase alcohol”, *Meas. Sci. Technol.*, pp.877-881, 2001.
 13. Agrawal, G.P., *Fiber Optic Communication Systems*, Third Edition, New York, Wiley Interscience, 2002.
 14. Kasap, S.O., *Optoelectronics and Photonics*, New Jersey, Prentice Hall, 2001.
 15. Hayes, J., *The Fiber Optic Technician’s Manual*, Third Edition, Delmar, 2006.
 16. Anderson, D.R., L. Johnson, F.G. Bell, *Troubleshooting Optical-Fiber Networks*, Second Edition, San Diego, Elsevier Academic Press, 2004.
 17. Dakin, J., B. Culshaw, *Optical Fiber Sensors: Principles and Components*, Volume One, Massachusetts, Artech House, 1988.
 18. Elosua, C., I.R. Matias, *et al.*, ” Volatile Organic Compound Optical Fiber Sensors”, *Sensors*, pp.1440-1465, 2006.
 19. Krohn, D.A., *Fiber Optic Sensors*, Second Edition, North Carolina, Instrument Society of America, 1992.
 20. Yuan, J., A. El-Sheriff, ”Fiber Optic Chemical Sensor Using Polyaniline as Modified Cladding Material”, *IEEE Sensor*, pp.5-12, 2003.