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Chapter

Modified Single Mode Optical Fiber Ammonia Sensors Deploying PANI Thin Films

Husam Abduldaem Mohammed and Mohd Hanif Yaacob

Abstract

Modified optical fiber sensors received increasing attention because of their superior properties over electrical sensors. These properties include their immunity towards electromagnetic interference and the ability to be deployed in corrosive and volatile environment. Several optical fiber platforms have been developed for chemical sensing applications based on modifying optical fiber cladding layer such as etched, tapered, D-shaped and etched-tapered. The modifications purpose is to extend the evanescent wave propagating out of the core physical dimensions. Thus, evanescent wave interaction with analyte is enhanced. Modified optical transducing platforms are integrated in gas sensing applications, such as ammonia. Modified optical fiber sensors coated with nanostructured thin films have been developed and gained popularity as practical devices towards gases with low concentrations. The development and characterization of the modified SMF sensing platforms including etched, tapered and etched-tapered platforms against ammonia will be presented in this chapter. These platforms were coated with PANI nanostructured thin film. The 50 μm etched-tapered SMF coated with PANI produced response, recovery times, and sensitivity of 58 s, 475 s, and 231.5%, respectively, in the C-band range. The limit of detection of the modified fiber sensor was 25 ppm. The developed sensors exhibit good repeatability, reversibility, and selectivity.

Keywords: optical fiber sensors, ammonia sensors, gas sensors, polyaniline, modified SMF sensors, etched-tapered optical fiber, C-band sensors

1. Introduction

The conventional ammonia (NH_3) sensors are electrical type. The electrical sensors have simple structure and low cost but they have poor selectivity as they respond to other gases. Moreover, electrical sensors are susceptible towards electromagnetic interference. Because of signal ignition opportunity, the electrical sensors are not appropriate to be used in oil and gas volatile environment [1]. There is a critical demand to develop an alternate type of sensors to avoid disasters resulted from ammonia leakages or drawbacks related to electrical signal based sensors {Mohammed, 2019 #3943}. The optical fiber sensor is an outstanding alternate [2]. Mostly, modified multimode optical fiber (MMF) is deployed to fabricate current NH_3 optical fiber sensors. Generally, the MMF sensors have lower sensitivity than the single optical fiber SMF sensors that not extensively explored for NH_3 sensing [2].

Researchers showed intensive focus on modified optical fiber platforms as sensing tools since they are more sensitive compared to the conventional fibers. Cladding modified SMF sensors with high sensitivity integrated with nanostructured thin films against ammonia can be deployed to avoid crises resulted from gas leakage such as ammonia [2]. These sensors have been gained popularity as practical tool to detect chemicals with low concentrations such as gases. By utilizing these configurations, it is expected to fabricate sensors with high sensitivity and fast response.

The aim of this chapter is to design and demonstrate an etched-tapered SMF optical fiber gas sensor for remote monitoring application. The gas under testing is ammonia due to its high severity and deployment in the industry. The objectives to achieve this research project are as follows:

- To present different modified optical fiber transducing platforms and nanomaterials used as sensing layers particularly, polyaniline.

- To design, fabricate and characterize modified SMF transducing platforms, that are etched, tapered and etched-tapered SMF platforms.

- To synthesize, deposit, characterize and evaluate gas sensing characteristics of the PANI nanofiber as a sensing layer onto developed modified SMF transducing platforms towards different concentrations of NH3 gas within C-band wavelength ranges.

- To compare performance criteria of developed SMF sensors with the reported sensors in some previous studies.

The next section presents a description of the modified optical fiber platforms as sensing tools since they are more sensitive compared to the conventional fibers. The etched, tapered, etched-tapered platforms as modified optical fiber platforms will be elaborated. After that, the Polyaniline nanofiber employed as a sensing layer is introduced in details. The properties of PANI thin films will be discussed by highlighting its attraction and factors that influence the sensing performance. Later, a detailed review on previous works that use PANI as a sensing layer for ammonia sensors will be presented. Moreover, PANI nanostructured thin film preparation and deposition onto the SMF transducing platforms will be highlighted. Several micro-characterizations of the fabricated nanostructured thin films were carried out to investigate sensing layer morphology and thickness of the nanostructured thin film. These parameters affect gas sensing performance. The sensing performance of the modified SMF sensors including etched, tapered and etched-tapered sensors coated with sprayed PANI nanofibers will be investigated and analyzed when exposed to ammonia with different concentrations. The investigation performed in the range of C-band wavelengths at room temperature. Based on author's knowledge, the investigation of the SMF sensors coated with PANI in C-band wavelengths ranges is not explored yet. Finally, chapter conclusions will be summarizing the performance properties behind the deployment of modified SMF sensing platforms Integrated with PANI nanofibers at room temperature.

2. Modified single mode optical fiber (SMF) sensors

Researchers showed intensive focus on modified optical fiber platforms as sensing tools since they are more sensitive compared to the conventional fibers. The high sensitivity in the modified fiber is a consequence of the evanescent wave or some portion of the optical power propagates outside of the core layer [3].

Evanescence wave-based intensity sensors require fiber modification to expand the penetration depth of the evanescence wave to increase its interaction with the surroundings. As a result, different optical fiber modification techniques are deployed including side polishing [4], D-fibers [5], chemical etching [6, 7] or tapering [8, 9] as shown the **Figure 1** [10] to improve the evanescence field for sensing applications [11].

2.1 Etched optical fiber sensor

Etched optical fiber is widely used for evanescent based sensors. The common process is to etch the cladding part of the fiber. One way to remove the cladding is to immerse the optical fiber using a strong acid in a chemical bath. Chemical etching produces shorter tapers with larger cone angles, resulting in higher transmission efficiency [12]. Depending on the composition of the glass, different acidic solutions are used. The etching rate depends on the dopant concentration in the structure of the optical fiber and concentration of the chemical solution. Etching parameters such as solvent type, acid concentration, etching time, and temperature are critical factors for the resultant optical and geometrical characteristics of the optical fiber [11].

Even though the modified optical fiber sensors enhance the evanescent wave to interact with the surrounding, each technique has its drawbacks. Side-polished fiber is made through polishing a fiber that is implanted on a block, such as a quartz block. The weakness of this technique is that the long time consumed in the fabrication procedures and it is difficult to produce a long sensing region. Hence, it difficult to develop a high sensitivity sensor [13]. The D-shape fiber is an optical fiber with removing half of the cladding layer. It has an advantage of long evanescent field interaction length. The removed-clad, namely etched fiber, sensor offers a simple and inexpensive fabrication method, especially compared with mechanical pulling and D fibers. On the other hand, chemical etching process is not easily controllable. The fabrication of tapered optical fibers is more reproducible and the controllable with the advance in tapering machine technology. However, the tapering machine itself is very expensive.

2.2 Tapered optical fiber sensors

In tapered optical fiber, the fiber diameter is reduced at a specific area called taper waist through heating and pulling the fiber ends in opposite directions as

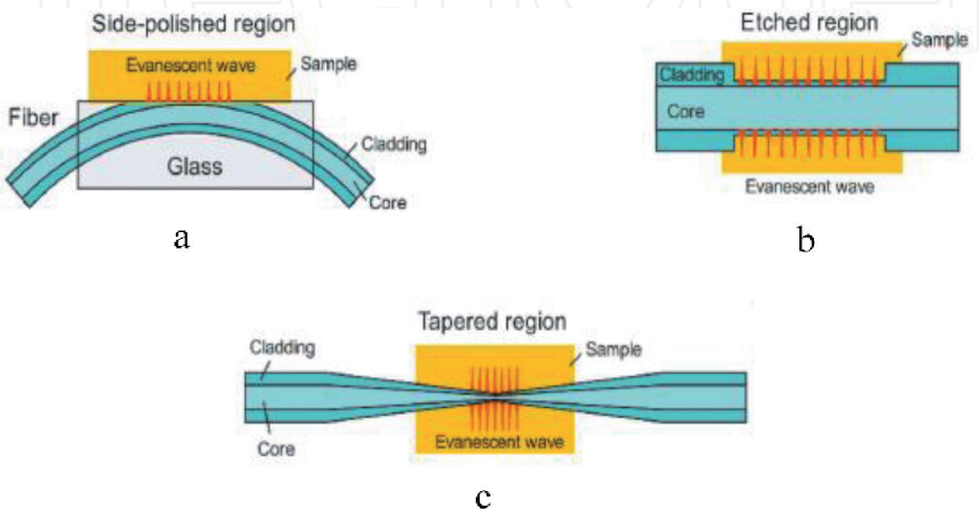

Figure 1.
Modified optical fiber sensors (a) side-polished, (b) etched and (c) tapered fibers [10, 11].

shown in **Figure 2** [11, 14]. Based on the figure, D_t, W_l and U_t represents the down taper, waist length and up taper regions which are the tapered fiber profile [11].

Several techniques have been developed to fabricate the tapered optical fibers such as etching [15] and flame heating. The flame heating technique has been verified to be the most flexible technique that results in robust physical properties. Later, the flame was replaced with microheater that is more controllable. Recently, computer-controlled machines that are able to fabricate tapers with desired dimensions are available in the market. Conventional SMF or multimode optical fiber (MMF) with standard diameter of 125 μm can be tapered down to 5 μm using the machine [16, 17].

Tapering process improves the sensitivity of the optical fiber sensors by easing the access to the evanescent field, which enables strong interaction between the light and the analyte. To prepare a qualified tapered fiber based devices, the tapered fibers used should be fabricated with high adiabaticity, uniform microfiber diameter and suitable microfiber diameter with large evanescent wave. Fundamentally, strong evanescent wave is obtainable with thinner tapering fiber diameters. Accordingly, the tapered fibers are made with small diameter in the range of 0.8–3 μm for most devices uses tapered fibers.

The strong evanescent wave on the taper waist surface make it more sensitive to its surrounding. The optical fiber sensors fabricated either by etching or tapering processes can be coated with suitable nanostructured sensing layer in order to improve the sensitivity. When sensing layer reacts towards the target analyte, its optical properties may change. Hence, the amount of evanescent wave absorbed by the sensing layer is changed according to the analyte concentrations [11].

2.3 Etched-tapered optical fiber sensors

The Etched-tapered optical fiber platform comprises of the etching and tapering processes abovementioned. Firstly, hydrofluoric acid is used to remove some of the cladding of the SMF as illustrated previously. The etched area is then tapered using different tapering methods based on the recommended configurations. Dealing with etched fibers to perform the tapering process is a critical and more challenging as compared to tapering the standard fiber [11]. The removal of the cladding layer of the SMF increases its fragility and increase difficulty of fiber handling. This is may be overcome by utilizing a customized holder in which the etched fiber is positioned while it is tapered.

Optical fiber sensors are deployed for detection of different hazardous gases including ammonia (NH_3). Ammonia is widely used gas in different applications such as chemical industries, agricultures and medicines [18, 19] . Natural NH_3 level present in the atmosphere is in low ppb (1–5 ppb) levels. NH_3 can be characterized by its colorless, pungent smell, and explosive, toxic at a high-concentration NH_3 atmosphere [20, 21]. Generally, upon exposure to around 50 ppm NH_3 gas in air

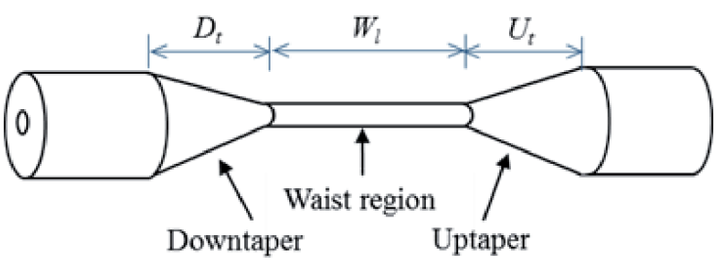

Figure 2.
Demonstration of tapered optical fiber [11].

may cause acute poisoning or life-threatening situations such as permanent blindness, lung disease, respiratory disease, skin disease. The Occupational Safety and Health Administration (OSHA) has set a limit of 25 ppm in the workplace throughout an 8 hours shift and a short-term limit (15 min) of 35 ppm [11, 22]. Additionally, NH_3 sensors remain the potential candidates employed in agriculture, chemical industries, pharmaceutical, hydrogen fuels, defense and food processing industries to monitor the NH_3 leak in controlled atmospheres. Hence, the development of highly-sensitive and reliable NH_3 sensors to continuously detect leakages NH_3 is a key issue for the safety of environments [19].

More worrying, the gas is flammable at 50°C at very high concentration (150,000 ppm) [11, 23]. In chemical leakage alarm for NH_3 detection, the detection limit ≥1000 ppm with operating temperature range up to 500°C and required response time may be in the range of minutes. The concentrations can be very high at NH_3 plants and can even be explosive [24]. Recently, on Aug 2016, Petronas Chemicals Group stated that two workers were killed and three injured by an ammonia leak at a Malaysian chemical plant [25]. Hence, the development of highly-sensitive and reliable sensors to continuously detect leakages of NH_3 is a key issue for the safety of environments [19, 26–29].

In general, the performance of the nanomaterial based sensors, particularly sensitivity, is controlled by three factors, material characteristics, transducer function and variability for sensor development [30]. The material characteristics indicate its surface ability to detect a specified chemical. The transducer function refers to the ability of converting the response of interaction between the analyte and nanomaterial surface into readable signal [31]. For the purpose of effectively upgrading the performance of the sensor, the surface properties might be enhanced by depositing nanostructured thin film as a sensing layer. In nanometer dimensions, the majority of the particles (atoms) are surface or near surface of the sensing platform. Therefore, the effective number of existing sites to interact with analyte molecules is high [11]. The deployment of nanomaterial sensing layers reduces the size of the detecting parts and transducer as well as reduced cost and response time. This results in scaling down of the detecting devices and simplicity. Moreover, nanomaterial sensing layers provides high surface to volume ratio leads to better detection limits [32]. The sensors incorporating the nanostructured sensing layer including conducting polymers such as polyaniline (PANI) has showed ability to integrate with different transducing platforms [11, 32].

3. Polyaniline nanostructures

Conducting polymers have become popular since early 1980s [33] due to their low cost, ease of synthesis and processing with ability to sense in room temperature [34]. Polyaniline (PANI) is of the important conducting polymers exploited extensively and studied as sensing materials. The light weight, high conductivity, mechanical flexibility and low-cost leads to the use of PANI in many applications.

PANI exist in several oxidation states with different colors. Generally, the fundamental form of polyaniline known as emeraldine. Emeraldine forms can either be in emeraldine base (EB) or protonated emeraldine salt (ES) forms. Reducing emeraldine base generates the leucoemeraldine base (LEB) or pernigraniline base (PEB) in oxidized forms. [35]. The acid/base doping response makes PANI attractive for acid/base chemical vapor sensors, super capacitors, as well as biosensors. Potential aspects of PANI make it promising for sensing applications since it is presents different oxidation states each with different color, changes and conformations [11].

PANI-ES is the only conducting form of PANI with approximately 15 S cm^{-1} conductivity. Meanwhile, other forms are normally insulating with conductivity below 10–5 S cm-1 [36, 37]. The PANI EB and ES form can be identified through their colors, where EB is blue and ES is green [36]. PANI-ES can be obtained through doping process, either by oxidation of leucoemeraldine base or by protonation of the PANI-EB [36]. The protonation is carried out by processing the PANI-EB with a strong acid such as HCl that induces the protonation of the imine sites.

PANI is attractive to be used as a sensing layer because it can rapidly switch between the EB and ES forms as it is exposed to certain analytes. This reversible process is also known as doping (ES) or dedoping (EB). This reversible pH-switching property not only changes its electrical conductivity, but also its optical property. The change in optical properties can be observed through the change in the absorbance spectrum.

PANI has been proposed for sensing NH_3 since there were variations in the electrical conductivity and optical absorption on exposure to NH_3. The properties change with the condition of oxidation and protonation of the polymer. At the point when exposing PANI-ES (the acid form) to NH_3, it will be deprotonated and transferred into a non-conducting PANI-EB [11, 21]. While there are many reported studies on the PANI based electrical sensors [38–41], the optical fiber based NH_3 sensors employing PANI is not as popular as the electrical one [42, 43].

Sensors that use PANI in nanostructure forms such as nanofibers or nanorods have shown a significantly better performance in terms of response time and sensitivity compared to the ones that use conventional PANI films [44]. This is as a result of increased surface area, high porosity, and small structure diameter which enhances the diffusion of the analyte molecules into the nanostructures [44]. PANI nanofibers can be obtained through various methods such as template synthesis, phase separation and electrospinning [45]. Several approaches have been adopted to enhance the PANI sensing performance (sensitivity and selectivity) [46]. This includes polymer molecular structures modification, using different dopants, and integrating the conducting PANI with different types of inorganic materials such as graphene-like materials [47]. The conductivity of PANI can be also enhanced using a highly conductive filler as graphene and graphite [48].

4. Review of ammonia sensors based on polyaniline

Limited SMF based NH_3 sensors employing PANI nanocomposite have been proposed so far. The developed optical sensors utilized a few types of substrates including glass substrate, waveguide, and modified optical fibers. Different optical measurement techniques such as absorption, transmittance, reflectance, resonance wavelength shift and fluorescence are used in the development of NH_3 optical sensors coated with PANI. The development of NH_3 optical sensors coated with PANI can be carried out using different deposition methods. This includes in-situ deposition, drop casting, dip coating, spray, electrochemical deposition, or spin coating.

The influence of synthesis methods, deposition methods, dopant types on PANI morphology and NH_3 sensing properties was studied in [49]. Glass substrate was used and absorbance measurement was done at wavelength of 632 nm. They experimented with three synthesis methods (interfacial, rapid mixing and dropwise mixing), two deposition methods (in-situ and drop-coating) with three types of dopants (HCl, CSA, and I_2). The results demonstrated that in-situ deposited PANI formed a cauliflower-like nanoparticles structure with a thickness of approximately 400 nm and diameter of 300 nm. While in the case of drop-casted PANI, a PANI nanofibers was formed with measured diameters of approximately

60–90 nm and 350 nm length. The in-situ deposited PANI nanostructure showed a shorter response time with higher sensitivity compared to drop-casted PANI, mainly because of more uniform coating. Drop-casting method suffered from the problem of non-uniform coating due to agglomeration of the PANI at certain areas on the glass surface. In-situ deposition of PANI-HCl was used for other experiments to investigate the best synthesis method. Rapid mixing method with oxidant-to-monomer mole ratio of equal to 1 was found to give the best result. This was contributed from the highest porosity and highest surface area of PANI nanogranules with size of 200 nm – 300 nm. PANI-CSA was found to give the highest sensitivity and the most stable NH_3 sensor as compared to PANI-HCl and I_2-doped PANI. The UV–Vis and FTIR measurements also confirmed that the sensing mechanism is based on the deprotonation process. From this work, it is learnt that to achieve high sensitivity, the highest surface area nanostructure is desirable together with high doping level.

In [50], a super Fiber Bragg grating (FBG) NH_3 sensor was developed based on optical reflectance measurement method. The FBG sensor was fabricated by removing some part of its cladding using chemical etching with 14 μm reduced diameter. The PANI sensing layer was deposited on the etched area with 300 nm thickness. The FBG showed a blue shift in Bragg's wavelength towards the shorter wavelengths as exposed to higher NH_3 concentration with 0.073 pm/ppm sensitivity. The value is low so that it is hardly to be measured.

Surface plasmon resonance-based NH_3 plastic optical fiber sensor coated with PANI as sensing layer was reported in [51]. The sensor was fabricated by uncladding 1 cm length of a 600 μm fiber diameter. The unclad area was coated with different thickness of indium tin oxide (ITO) and PANI on top with the use of thermal evaporation technique. Then, the ITO coated fiber was dipped into ammonium hydroxide (as adhesive), followed by PANI solution. They found that the resonance wavelength increases as the NH_3 concentration increases and the sensor with ITO layer of 60 nm gave the best response.

Fiber sensors based on evanescent wave absorption were proposed using bent optical fiber [52] and removed-clad fiber [53]. In [52], NH_3 sensors were proposed using bent optical fibers with PANI and Fe (III) porphyrin-doped PMMA have detection limit in the range of ppm. The cladding of silica MMF was removed using chemical etching and replaced with thin PANI layer (less than 1 μm) in [53] for NH_3 sensing. It was observed that the absorbance spectra increase over certain wavelength (between 500 to 800 nm) as the sensors were exposed to NH_3. However, there is no detailed explanation on the synthesis methods and the type of PANI used in this work. Even though this work is quite dated (2003), but it gave a useful indication that thin PANI layer is a good candidate as an absorbance-based NH_3 sensor.

5. Optical fiber modification, nanomaterials deposition and characterizations

In this section, the development and characterizations of the modified SMF sensing platforms including etched, tapered and etched-tapered platforms will be elaborated. The etching process based on the use of chemical to remove some of the cladding layer. These platforms were characterized in term of output optical power. In the second section, PANI nanostructured thin film preparation and deposition onto the SMF transducing platforms will be highlighted. Finally, the PANI nanofibers fabrication and deposition onto the SMF transducing platforms will be explained. Several micro-characterizations of the fabricated nanostructured thin films were carried out to investigate sensing layer morphology and thickness of the

nanostructured thin film. These parameters affect gas sensing performance which will be discussed extensively here.

5.1 Modified SMF fabrication and characterizations

Three types of modified SMF platforms including etched, tapered and etched-tapered platforms were developed and investigated towards ammonia at room temperature. A standard SMF-28 single mode silica fiber (Lucent All-Wave Fiber, 9/125 μm core/cladding diameter ratio) is modified as the optical transducing platforms for NH_3 sensing applications. Each transducing platform was made of 1 m length of SMF. The Tafzel ® polymer jacket enfolding the SMF was removed mechanically over 8 cm by a fiber a stripper. The fabrication processes for the three types of modified SMF platform will be elaborated in the following subsections [2, 11].

5.1.1 Etched SMF sensor

A 48% hydrofluoric acid (HF) (Sigma Aldrich) was used as an agent for chemically etching the SMF. For better holding of the optical fiber platform, both ends of the fiber are fixed using a metal racks so the fiber dangle into the vessel containing the acid used for the etching as shown in **Figure 3**. This is fixed inside a fume hood to prevent direct exposure to HF vapor and creation of aerosols. The etching process was started by filling 100 μl of HF acid in a container using a Pasteur pipette. To fabricate the etched optical fiber transducer, the stripped fiber is fixed as depicted in the figure to control its emersion in HF acid. The fiber is etched in two stages process to control its modification. The first stage includes immersing the stripped area in 48% HF acid at a specific time to produce different etched diameters. After that, the fiber was taken out and cleaned with deionized water for 30 minutes to remove the HF acid residual. The etched fiber is left to dry at room temperature. In the second stage, the fiber was immersed in HF with less concentrations of 12% to reduce the etching rate and hence, more control on the etching dimensions. A variety of parameters affect the etching rate of the cladding such as acid concentration, humidity and temperature. The humidity and temperature are fixed at 67% and 25 C, respectively. As the second stage of etching is finished, the fiber is immersed in deionized water for 30 minutes to remove the remaining of the HF acid as well as to prevent further etching due to residual HF [2, 11].

5.1.2 Tapered SMF sensor

Tapering the SMF is a critical part in the work. Vytran Glass Processing System workstation (GPX-3000, USA) was used to taper transducing platforms. This

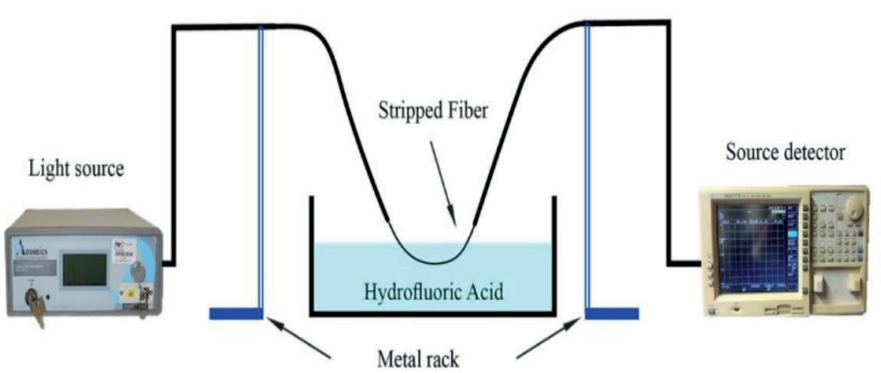

Figure 3.
Setup for etching optical fiber using HF acid [2, 11].

system implies a heating element which is a filament of graphite and two movable fiber holders blocks as a part of tapering control process to generate the recorded profile parameters. The tapered SMF was fabricated using heat-pulling technique which suggests that the fiber diameter changes smoothly and is adiabatically slow as a function of fiber length. Before tapering, the plastic jacket and polymer coating of the fiber are removed approximately 8 cm length. The machine was controlled using a proprietary software on a computer, where the desired tapered fiber profile (waist diameter, waist length, down taper length, and up taper length) can be specified. The waist length, up and down transitions setting for tapering SMF platforms are 10, 2 and 2 mm, respectively. The dimensions of the modified SMF transducing platforms are verified using the CCD camera of Vytran workstation [2, 11].

5.1.3 Fabrication of etched-tapered SMF transducing platform

Fabrication of the etched-tapered SMF (ETSMF) sensing platform was done by combining the two processes; etching and tapering. Firstly, the SMF was etched using HF acid in the same manners described in SubSection 5.1.1 Afterward, the etched SMF was tapered using Vytran workstation according to the proposed settings used in Section 5.1.2 The ETSMF platform is shown in **Figure 4**. Tapering the etched SMF is critical and more challenging than tapering the standard fiber. The reduction in the fiber diameter and the weaken fiber structure due to the etching process increases the difficulty of fragility the modified fiber. Thus, customized holder that fixed the etched fiber during the tapering process was used. The diameters of the optical fibers used in the research fabricated by both etching and tapering were confirmed using the Vytran Glass Processing System workstation GPX- 3000 [11].

In order to verify the compatibility of modified SMF platforms for gas sensing, many experiments were carried out using these sensors coated with PANI nanostructures thin films as sensing layer for NH_3. **Table 1** summarizes the design parameters for the fabricated sensors used in this PhD project. Sensors S1-S4 are ETSMF sensors while Sensors S5 and S6 are the tapered only and etched only sensors. Referring to **Table 1**, it is observed that the core to cladding ratio is found to be varied according to the different modification techniques. It is noted from **Table 1** that in the etched only sensing platform (S6), the original core diameter is unchanged at 9 μm [2, 11]. During the etching process, the cladding layer was dissolved to produce 6 μm thickness diameter. The core to cladding ratio is 0.6. On the other hand, the core diameter is modified as well as the cladding diameter in the case of tapered only sensing platform (S2). The core diameter after tapering became 1.08 μm to give core-cladding ratio as the lowest (0.07) for the tapered only platform that is the lowest relative to other modified fibers [11].

The ETSMF sensing platforms (S1-S4) possess unique core-cladding ratio as compared to the etched only and tapered only platforms. During the etching process, some of the cladding was removed and hence the core becomes more

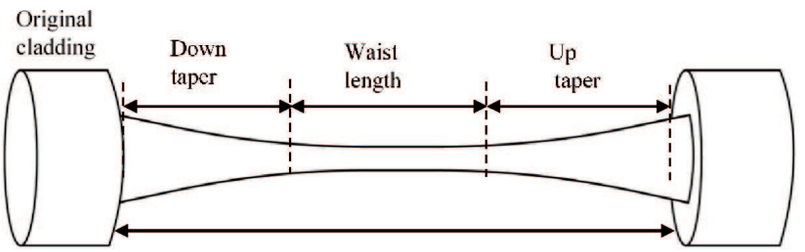

Figure 4.
Etched-tapered sensing platform schematic diagram [2, 11].

Label	Etched fiber diameter (μm)	Tapered fiber diameter (μm)	Fiber core diameter after tapering (μm)	Core to cladding ratio
S1	30	15	4.5	0.3
S2	40	15	2.95	0.2
S3	50	15	2.44	0.16
S4	60	15	2.01	0.15
S5	No	15	1.08	0.07
S6	15	No	9	0.6

Table 1.
Design parameters of the modified SMF sensors [2, 11].

sensitive to the surroundings and the ratio of core-cladding is modified. Afterward, the core diameter is reduced when tapered to the specified settings. The tapering process does not change the core-cladding ratio of the sensing platforms which is equal to the previous etched fiber. For example, S1 sensing platform with 30 μm etched diameter has a core diameter of 9 μm. By tapering process, the cladding and core diameters are changed so that the core diameter is about 4.5 μm core diameter. Consequently, combination of etching and tapering processes yields to a reduction in cladding layer thickness surrounding the core allowing the latter to be more sensitive to the variations in environmental parameters. It is found that the core to cladding ratio for sensor S1 is 0.3 which the highest ratio for the ETSMF platforms [2, 11]. The core cladding ratio for the ETSMF sensing platforms S2-S4 are found to be 0.2 (S2), 0.16 (S3) and 0.15 (S4) as listed in **Table 1**. Stronger response is expected from the ETSMF sensing platforms as a consequence of both surface area and evanescent field enhancement via combination of etching-tapering processes. These three different groups of optical fiber platform were characterized in terms of their optical transmission in terms of the output optical power.

5.2 Characterization of modified SMF transduction platforms

The surface investigation of the unprocessed and modified single mode optical fibers is shown in **Figure 5** based on scanning electron microscopy (SEM - Hitachi SU1510, Japan). The unprocessed single mode optical fiber with a diameter of 125 μm is shown in **Figure 5(a)**. **Figure 5b** depicts the etched SMF using hydrofluoric acid with total fiber diameter of 9.7 μm. **Figure 5(c)** and **(d)** shows the etched-tapered with a diameter of 77.7 μm after etching and waist diameter of 15 μm after tapering. The tapered part of the etched-tapered fiber is shown in **Figure 5(c)**. The fiber exhibited a uniform transition with surface roughness. The downward transition of the etched-tapered SMF with 15 μm waist diameter is shown in **Figure 5(d)**. it can be noted that the unprocessed SMF has a smooth surface. On the other hand, the etched fiber exhibited a rough surface due to its processing with hydrofluoric acid. The surface roughness of the modified SMF is superior to enhance the surface area of sensing layer deposited onto it which allows stronger interaction between sensing layer molecules and the gas molecules [2, 11].

5.3 Synthesis, deposition and characterization of polyaniline (PANI) nanofiber thin film sensing layer

PANI nanofiber was deposited on modified SMF transducing platforms as sensing layers towards NH_3. In the next subsections, the synthesis and

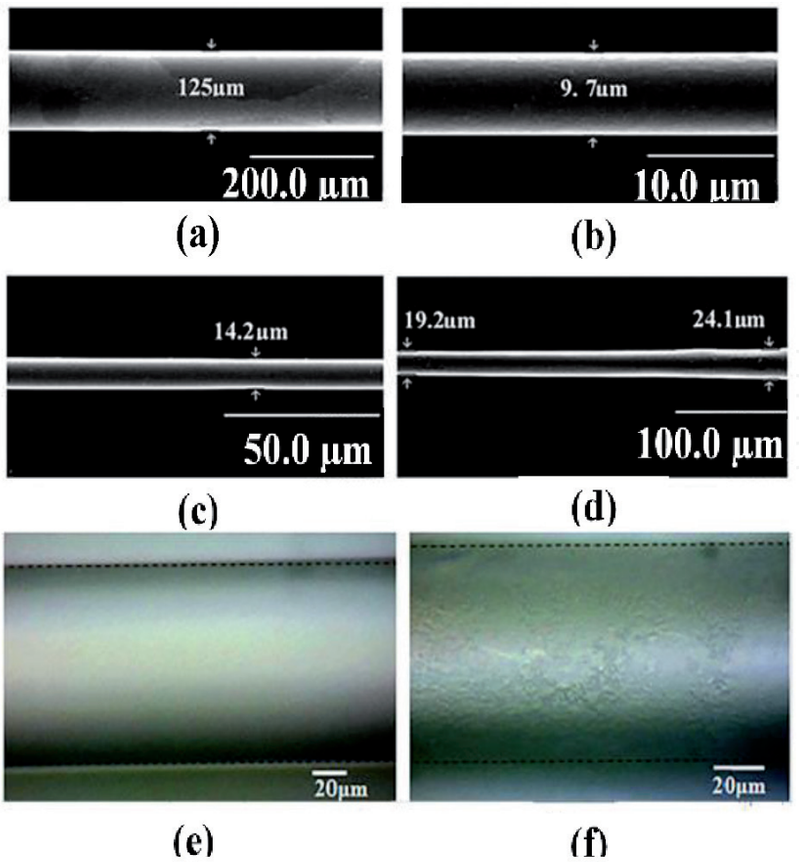

Figure 5.
SEM images of modified SMF platforms, (a) original SMF, (b) the etched SMF, (c) the etched-tapered SMF and (d) the down ward transition for etched-tapered SMF in (c) and microscopic images of modified SMF platforms (e) original SMF and (f) a 77.7 µm etched SMF [2, 11].

micro-characterization of these nanostructured thin films will be presented. The modified SMF transducers were coated with PANI nanofiber thin films as a sensing layer towards NH_3. PANI solution was prepared by dissolving and dispersing a 15 mg PANI powder with 15 mg camphor sulfonic acid (Sigma Aldrich) in 8 ml of chloroform ($CHCl_3$). This resulted in CSA-doped PANI nanofiber of green color solution with concentrations of 3.75 mg/ml [11]. The camphor sulfonic acid was implied to boost the ability of solving PANI in chloroform. The resulted solution was stirred for 1 hour using magnetic bar.

To generate a homogeneous solution to be used to deposited on the SMF transducers and glass substrates, the solvent was sonicated for 1 hour at room temperature using Hielcher, Ultrasound Technique, UPS2005 ultra sound processor. The fabricated PANI-CSA solution had dark green color. This indicates the doping with CSA was carried out successfully. Glass is chosen as one of the substrates for PANI micro-characterization. The optical fibers and glass substrates were heated up to 50°C for 30 minutes prior to deposition of PANI using hotplate. The heating is important to increase the binding of the nanomaterial and generating uniform films. The prepared samples were left to dry for 1 hour at room temperature. The coating process was done using a fume hood [2, 11].

The polyaniline coated on the glass substrate was investigated using scanning electron microscopy as described in **Figure 6(a)** to prove its morphology. As can be noted from the figure, PANI deposited on the glass exhibits a random distribution over the substrate surface in cluster forms with different sizes. As can be noted from the **Figure 6(b)**, the non-uniform nanofibers agglomerated to produce the cluster morphology. These PANI nanofibers exhibits a typical length of 2.5–3.5 µm with diameter in the range of 180–200 nm. **Figure 6(c)** presents the image of the PANI

thin film taken with the aid of an atomic force microscope (NT-MDT Solver NEXT AFM). The average thickness of PANI thin film was found to be 400 nm while the its surface roughness was approximately 228.2 nm [11]. Surface roughness is significant in the applications of gas sensing as it enhances the surface area which rises the active interaction sites between the gas molecules and the sensing layer. Consequently, increases the sensor sensitivity [11].

5.4 Ammonia sensing performance of modified SMF coated with PANI nanofiber

The setup used to investigate the optical response of the modified SMF sensors towards NH_3 is outlined in **Figure 7**. This setup is used to prove the behavior of the sensors in the visible wavelength range (600–750 nm) and C-band wavelength range (1535–1565 nm). Based on the setup, the modified SMF sensor is placed inside a gas chamber which contains a gas inlet and outlet as well as FC/PC connecting adapters to fit the sensors. The sensor is connected to a light source (tungsten-halogen lamp (Ocean Optics HL2000) for visible and C-band (Ammonics) for C-band) and the other end is connected to a detection system spectrophotometer (Ocean Optics USB4000) for visible and OSA for C-band). A proprietary software is used to record and measure the responses from the detection system. A gas calibration system (AALBORG) is deployed to vary the gas concentrations and purging time, automatically. NH_3 of 1% concentrations in 99% synthetic air is purged into the chamber via the MFCs. Another pure synthetic air is used as the reference gas. The gas flow is fixed at a rate of 200 sccm. This is completely

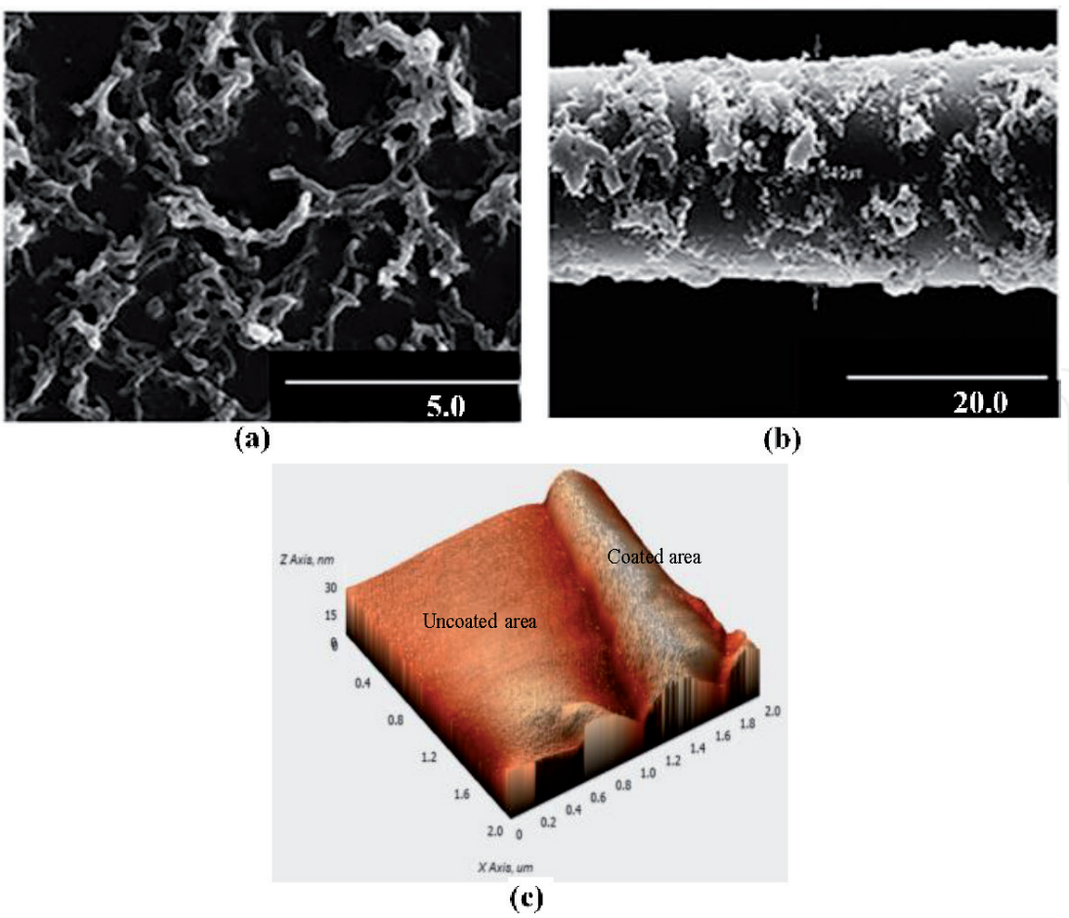

Figure 6.
SEM images of (a) PANI nanofibers on glass, (b) etched-tapered SMF transducer coated with PANI nanofibers and (c) 3D AFM image of uncoated and PANI coated areas on a glass substrate [2, 11].

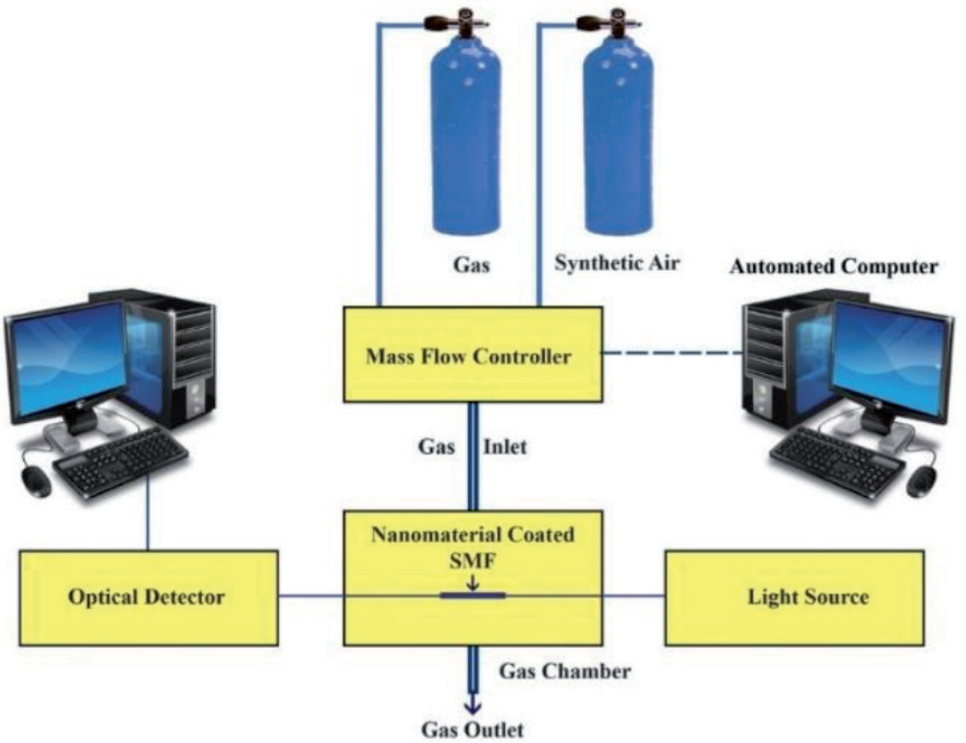

Figure 7.
Experimental setup for modified SMF ammonia sensing coated with PANI thin film [2, 11].

computerized using Labview control program. Certified NH_3 and synthetic air gas cylinders (Linde, Malaysia-Singapore Sdn Bhd) were used in the mixing and purging of the gases into the chamber. The mixing was done for the purpose of changing the concentration of ammonia in the range of 0.125–1%. The dynamic response of the SMF sensors was investigated upon exposure to NH_3 with different concentrations at room temperature. This was carried out through measuring cumulative absorbance of the sensor as it exposed to a NH_3 at abovementioned concentrations. Each gas concentration cycle was persisted for 8 minutes while the sensor air regeneration lasted for 15 minutes [2, 11].

In order to verify the compatibility of modified SMF sensors for gas sensing, many experiments were carried out using these sensors towards NH_3. The sensors were coated with sprayed PANI nanostructure thin films as a sensing layer. The sensing performances were investigated and analyzed in both visible and C-band wavelength ranges. The details of morphology and thickness the of the PANI sensing layer was introduced in Section 5.3. Different modified SMF, namely etched, tapered and ETSMFs were investigated towards NH_3 gas with different concentrations. The design parameters for the fabricated sensors used are summarized in **Table 1**. These platforms' dynamic response was investigated towards NH_3 in the C band wavelength range [11].

Figure 8 demonstrates the dynamic responses of the etched-tapered SMF (S1-S4) while **Figure 9** represents that of tapered and etched SMF (S5 and S6) sensors against different concentrations of ammonia, respectively. SMF sensors with different modifications proved proportional increase in the output optical power against NH_3 concentrations. The etched-tapered sensors exhibited superior response magnitude over that of the sensors with other modifications. The etched-tapered sensors (S1-S4) shows response of 1.6, 1.5, 1.39, and 1.29 dBm, respectively, when exposed to 1% of ammonia while the tapered and etched sensors response is 0.84 and 0.68 dBm, respectively. Lower increases are noted at lower NH_3 concentrations. As exposed to 0.125% NH_3 concentration, the modified sensors (S1-S6) exhibited a change

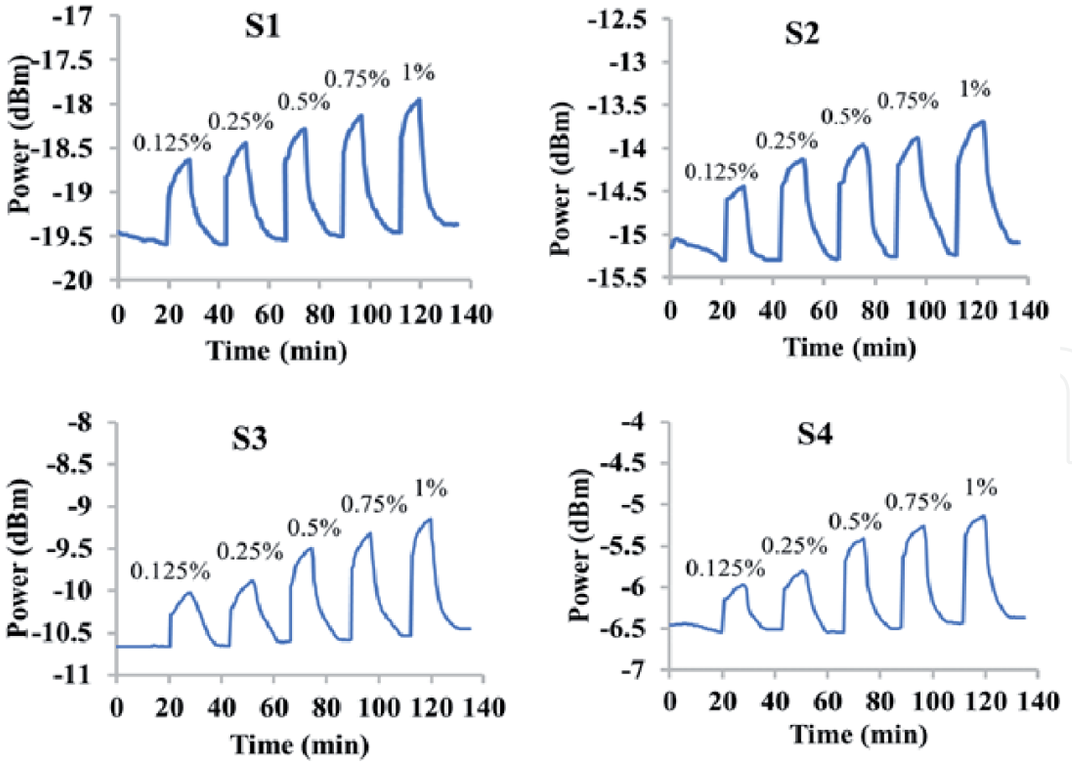

Figure 8.
Dynamic response of the etched-tapered SMF sensors (S1-S4) exposed to different NH_3 concentrations in the C-band wavelength range [2].

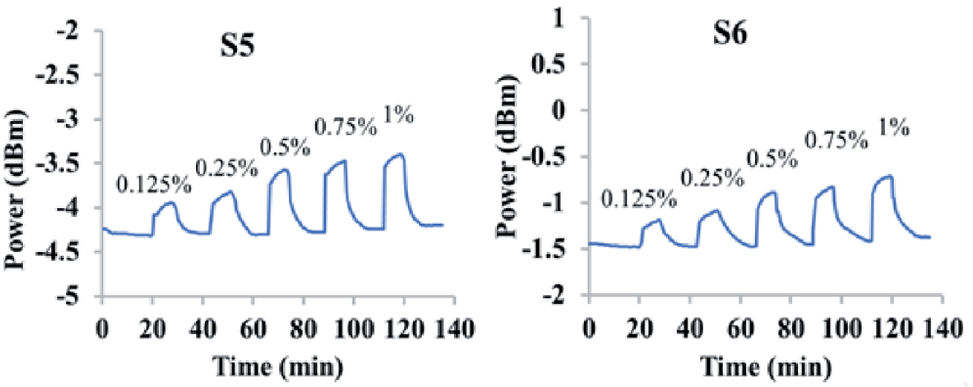

Figure 9.
Dynamic responses for tapered and etched SMF sensors (S5 and S6) when exposed to NH_3 with different concentrations [2].

of 0.96, 0.86, 0.68, 0.55, 0.36, and 0.29 dBm, respectively [2, 11]. Generally, SMF sensors with different modifications proved different responses and recovery times are. Moreover, the response time is inversely proportional to the ammonia concentrations while the recovery time is linearly proportional to it for all SMF sensors with different modifications investigated in the C-band range. The core-to-cladding ratio due to etching and tapering processes of the modified SMF sensors affects the response and recovery times as shown in **Figures 8** and **9** [2, 11].

The average response time for the modified sensors (S1-S6) against ammonia are 58–71 s, 79 s, and 92 s, respectively. The average recovery time designates the opposite performance for the sensors with different modification techniques. The values for modified sensors (S1-S6) against ammonia are 466–453 s, 380 s, and 360 s, respectively. Different sensing performances for different types of modified SMF sensors is attributed to different rates of ammonia molecules adsorption/

desorption on each kind of the sensors surfaces. For example, fastest response but slowest recovery is observed for the etched-tapered sensors. Contrary, the etched-only sensor shows the reverse time responses. The response of the modified SMF sensors presented here and investigated in the C-band wavelength range were significantly improved compared with the tapered MMF sensors described in a previous study investigated in the visible wavelength range [54]. The response time of etched-tapered SMF sensors (62 s) was more than twice that of the tapered MMF sensors (2.27 minutes or 136.2 s) [11, 54]. The recovery time of the SMF and the MMF sensors were 453 and 583.8 s, respectively [54]. The sensor introduced by Airoudj et al. [55] investigated in the visible to near-infrared wavelengths (632.8–980 nm) based on the single mode planar polymer waveguide coated with PANI exhibited response and recovery times of 180 s and 480 s, respectively [2, 11].

The normalized cumulative Δ optical power of the modified SMF sensors coated with PANI thin film against ammonia is depicted in **Figure 10**. The etched-tapered SMF sensors (S1–S4) proved significant enhancement in response compared with other modifications. On the contrary, the etched-only sensor exhibited the lowest response. The etched-tapered SMF response as exposed to ammonia is improved when the SMF cladding thickness is reduced due to etching process. For example, sensor (S1) with smallest etching diameter shows the strongest response among the etched-tapered sensors. This result principally is a consequence of the strong evanescent field energy propagating out of the core physical dimensions for the modified optical fiber sensors into the sensing layer of PANI. Additionally, the modified SMF sensors integrated with PANI show a variation in response that is proportional to the ammonia concentrations. The normalized cumulative optical powers for the sensors (S1–S6) are 17.7%, 14.6%, 10.8%, 9.5%, 6.3%, and 1.9% at the limit of detection of the sensors [2, 11]. The practical of the gas system used in the work, the limit of detection for the SMF sensors is found to be 0.04% or 400 ppm. Based on established technique introduced by Mola et al. [11, 56], the limit of detection for the etched-tapered SMF Sensor S1 is 0.0025%, which is equal to 25 ppm. Accordingly, the established PANI coated SMF sensor can detect ammonia gas concentration below the ammonia lowest tolerable exposure limit reported by OSHA [11].

The sensitivities for the SMF sensors (S1-S6) are found to be 231.5 (S1), 209.7 (S2), 172.1 (S3), 146.6 (S4), 100.4 (S5) and 81.2 (S6). The ETSMF sensors (S1-S4) proved considerably higher sensitivity towards NH_3 compared to the tapered only (S5) and etched only sensors (S6). The sensitivity of the ETSMF sensor (S1-S4) are 2.8, 2.3,1.7 and 1.5 times the sensitivity of the tapered only SMF sensor (S5)

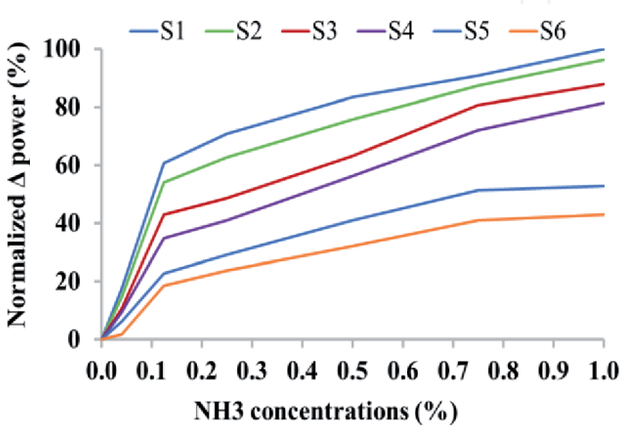

Figure 10.
Normalized Δ power versus NH_3 concentrations for the modified SMF sensors (S1-S6) in the C-band wavelengths range [2, 11].

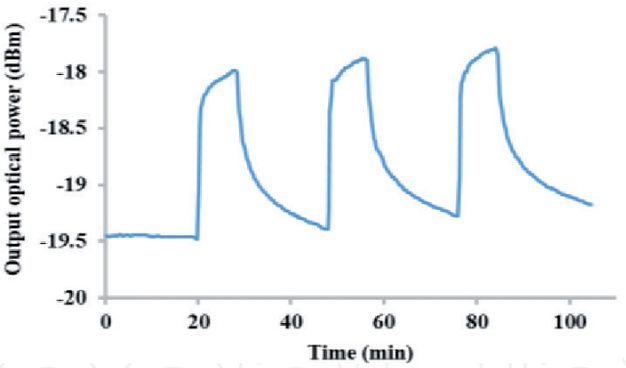

Figure 11.
Repeatability for the ETSMF sensor S1 coated with PANI nanostructured thin film as exposed to 1% NH_3 for three cycles [2, 11].

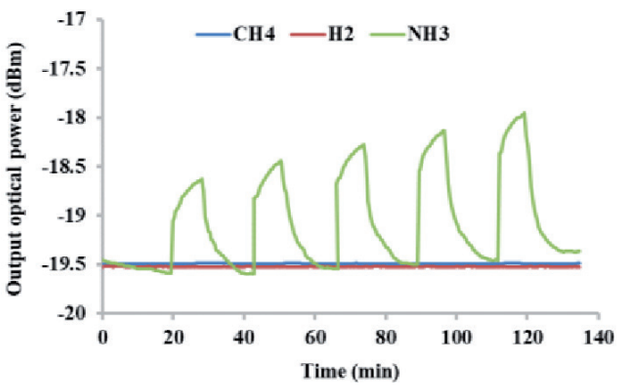

Figure 12.
Optical response for ETSMF sensor S1 coated with PANI nanostructured thin film towards CH_4, H_2 and NH_3 [2].

and 9.3, 7.7, 5.7 and 5 times that of the etched only SMF sensor (S6) [2, 11]. Based on **Figure 10**, the sensitivities for the SMF sensors (S1–S6) are found to be 231.5, 209.7, 172.1, 146.6, 100.4, and 81.2, respectively [2, 11]. As compared with the tapered and etched sensors, the etched-tapered SMF sensors have higher sensitivity towards NH_3 [2].

Figure 11 shows The repeatability and reversibility of etched-tapered Sensor S1 against 1% ammonia concentration for three cycles each lasts for 8 minutes of ammonia, followed by purging purified air for 15 minutes. These three cycles in the figure exhibits slight difference when exposed towards 1% ammonia. Furthermore, the base time experienced slight shift as a result of incomplete elimination of ammonia from the PANI sensing layer when the air was purged for 15 min. The etched-tapered SMF sensor shows good repeatability and reversibility as demonstrated in the figure.

The etched-tapered Sensor S1 was examined towards methane (CH_4), hydrogen (H_2), and ammonia (NH_3) to prove its selectivity towards ammonia. The range of concentrations of these gases utilized in this test was from 0.125–1% at room temperature. The sensor exhibits superior response towards ammonia compared to that due to methane and hydrogen as depicted in **Figure 12**. Accordingly, the sensor can be described to be highly selective towards NH_3.

6. Conclusion

Simple and low-cost modified SMF platforms were successfully designed, developed and investigated for optical sensing towards NH_3 with low concentrations.

The modification processes performed on the SMFs were etching, tapering and combination of etching-tapering. The sensing performance of the modified SMF sensors coated with PANI nanofibers thin films was investigated towards NH_3 at room temperature for C-band wavelengths range (1535–1565 nm). The investigation showed that the principle of the gas sensors used is the change in the light characteristics because of the interaction between the NH_3 molecules and PANI sensing layer. The interaction between the NH_3 molecules and the PANI sensing layer reduces the absorbance in the C-band range and inversely proportional to the NH_3 concentrations. Consequently, the transmitted optical power increased. These PANI nanofiber thin films show high sensitivity towards NH_3 with low concentration as well as good repeatability indication. The performance of the modified SMF sensors was found to be dependent on the modification technique used in the fabrication of the SMF platform as well as the thickness of the cladding layer after modification (core/cladding ratio). The investigation on the NH_3 optical sensing performance using absorbance measurement proved that the ETSMF showed superior response than the other modified fibers and thus, highly potential for novel optical transducer. The ability of the ETSMF coated with PANI thin films operates at room temperature indicates its promising candidate for NH_3 sensing applications such as chemical plant leakage remote sensor. Particularly, its response in the C-band wavelength range allows easy integration with the existing all optical fiber communication networks infrastructures. The modification technique used in the fabrication of the SMF platform namely etching, tapering or combination of both strongly affects the performance of the modified SMF sensors. Furthermore, the performance of the modified SMF sensors is also dependent on the thickness of the cladding layer after modification and core/cladding ratio. The investigation on the ammonia optical sensing performance demonstrated that the response of etched-tapered SMF is the stronger among that of other modified sensors. Thus, the developed etched-tapered SMF sensors show high potential to be a novel optical transducer. The ability of the etched-tapered SMF coated with PANI to perform at room temperature makes it a good candidate for ammonia sensing and remote monitoring applications. Particularly, its strong response in the C-band wavelength range makes it is easy to be integrated with all well-established optical fiber communication network infrastructures such as fiber to the home. The developed sensor exhibits good repeatability and reversibility. The limit of detection of the modified SMF sensor was 0.0025% (25 ppm).

Author details

Husam Abduldaem Mohammed[1*] and Mohd Hanif Yaacob[2]

1 Electronic and Communication Engineering Department, College of Engineering, University of Baghdad, Baghdad, Iraq

2 Wireless and Photonics Network Research Centre, Faculty of Engineering, Universiti Putra Malaysia, Serdang, Selangor, Malaysia

*Address all correspondence to: husam.a@coeng.uobaghdad.edu.iq

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