OPTICAL MICROBOTTLE RESONATOR FOR FORMALDEHYDE (CH₂O) LIQUID SENSING

ABDULLAH AL NOMAN

FACULTY OF ENGINEERING UNIVERSITY OF MALAYA KUALA LUMPUR

2018

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RESEARCH REPORT SUBMITTED IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE DEGREE OF MASTERS OF TELECOMMUNICATION ENGINEERING

FACULTY OF ENGINEERING UNIVERSITY OF MALAYA KUALA LUMPUR

2018

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Name of Candidate: Abdullah Al Noman

Matric No: KQH160009

Name of Degree: Master of Telecommunication Engineering

Title of Research Report: Optical microbottle resonator for formaldehyde (CH₂O)

liquid sensing

Field of Study: Optical Fiber Sensor

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OPTICAL MICROBOTTLE RESONATOR FOR LIQUID FORMALDEHYDE (CH2O) SENSING ABSTRACT

In recent years, microbottle resonator (MBR) has attracted an excessive attention on account of its various applications such as sensors, lasing and filters. Compared to the other optical microresonators (OMRs) it has some advantages of fast tunability through the strain application, better control over the coupling by optical tapered microfiber and in the spectrum the probability of attaining a great number of equally-spaced modes. The MBR was fabricated with an SMF-28 silica optical fiber by "soften-and-compress" method which created a bulge area on the fiber to become formed as bottle shape. The formaldehyde (CH₂O) liquid which was used for this work has 37% purity of formalin with other ions. In this investigation, the influence of whispering gallery mode (WGM) on an optical microbottle resonator (OMBR) and its consequence on liquid (formaldehyde, CH₂O) sensing is demonstrated. The MBR then excited using a tapered optical microfiber of 5 µm waist diameter and for each concentration levels of liquid the Q-factor (quality factor) noted as $> 10^5$. Comparison between the MBR and the bare fiber has stated based on four specific parameters such as linearity, sensitivity, p-value and standard deviation where MBR has shown better potentiality than the bare fiber towards liquid sensing for the sensor. Moreover, the outcome of MBR was explained with two different diameters of tapered microfiber which are 8 µm and 10 µm. The MBR energized through both tapered fiber and also stated their effect for the liquid sensing in this work. For both experiments, the MBR was categorized by three significant constraints such as bottle distance D_b , stem width D_s and neck-to-neck length L_b where the bottle diameter was considered as 190 µm. The p-values were measured from both experiment as $> 10^{-5}$ which indicated that the research is in the right direction and the stability also calculated in terms of 60 second clock time. This finding shows that the MBR is a promising microresonator among other resonators for the formaldehyde liquid sensing which can be apply for the sensor in future.

Keyword: whispering gallery modes (WGM), optical microresonator (OMR), microbottle resonator (MBR), and formaldehyde (CH₂O).

PENGALUN GENTIAN BOTOL MIKRO SEBAGAI PENGESAN CECAIR FORMALDEHID (CH₂O)

ABSTRAK

Sejak kebelakangan ini, bahantara mikro-botol (MBR) telah menarik perhatian kerana kepelbagaian aplikasinya seperti pengesan, laser dan penapis. Jika dibandingkan dengan bahantara mikro optikal yang lain ia mempunyai kelebihan seperti kecepatan pengubahan dalam aplikasi penegangan, pengawalan yang baik dalam mencantumkan gentian mikro yang dinipiskan dan kehadiran spaktrum dengan jumlah yang besar dalam kesamaancahaya ruang. MBR dihasilkan dengan menggunakan gentian optik silika SMG-28 melalui teknik "melembut-dan-menekan" dimana akan membentuk kawasan gelembung pada gentian dengan bentuk seperti botol. Cecair formalin (CH₂O) yang digunakan dalam kajian ini mempunyai 37% tahap kesucian dengan ion yang lain. Di dalam kajian ini, pengaruh mod galeri berbisik (WGM) pada bahantara mikro-botol optikal (OMBR) dan kesannya terhadap mengesan cacair (formalin CH2O) telah di laksanakan. MBR digunakan bersama-sama gentian optik yang dinipiskan pada berketebalan 5 µm dan telah mendapat faktor kuality $> 10^5$ untuk setiap kepekatan cecair yang digunakan. Perbandingan diantara MBR dan gentian kosong telah dilaksanakan berdasarkan kepada empat parameter tertentu iaitu ketepatan, kepekaan, nilai-p dan sisihan piawai dimana MBR menunjukkan potensi yang lebih baik berbanding gentian kosong tehadap kebolehan mengesan bagi mengesan cecair. Dalam kajian lanjutan, keluaran hasil MBR diterjemahkan dari dua ukurlilit gentian mikro yang berbeza iaitu 8 µm dan 10 µm. MBR telah digunakan untuk kedua-dua gentian mirko tersebut dan juga kesannya terhadap kepekaan cecair ditunjukkan dalam kajian ini. Dikedua-dua kajian, MBR yang digunakan diketagorikan dengan tiga ukuran yang signifikan iaitu lebar ukuran botol D_h , lebar ukuran gentian D_s dan panjang ukuran botol L_b dimana ukuran botol adalah 190 µm.

Nilai-p yang telah diukur dari kedua-dua kajian adalah $> 10^{-5}$ dimana menyatakan bahawa kajian ini dilaksanakan pada paksi yang betul dan nilai kestabilan dikir adalam lingkungan 60 saat. Penemuan ini menunjukkan bahawa MBR adalah bahantara-mikro yang baik berbanding bahantara yang lain dalam mengesan cecair formaldehyde dimana ia boleh diaplikasikan sebagai pengesan dimasa akan datang.

Kata kunci: pengaruh mod galeri berbisik (WGM), bahantara mikro optikal (OMR), bahantara mikro-botol (MBR), formaldehid (CH₂O).

ACKNOWLEDGEMENTS

At the very first moment, I am expressing my gratitude to Almighty Allah for giving me the patience and competence to complete my research project successfully. In addition, I am really thankful to my parents whose allow me to fulfill my dream, inspire me and also support me in every stage of my life. During my research, I acknowledge the inspiration and assistance given by a number of people and my institute. I would like to thank my honorable supervisor Prof. Ir. Dr. Sulaiman Wadi Harun who motivated and driven me all the way to implement this research project.

Moreover, I am really grateful to Mr. Md Ashadi Md Johari who guided me as a mentor during my research. My appreciation goes to Mr. Mohd Hafiz Bin Jali and Mr. Haziezol Helmi Bin Mohd Yusof for their helping and advising me. I am also thankful to my friends Miss Sana Sulaiman Hamid and Miss Ummu Umairah for sharing their ideas with me.

At last, I would like to express gratitude towards the University of Malaya from giving me such platform where I can explore and expand my knowledge which will help me in my future.

Thank you very much.

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LIST OF SYMBOLS AND ABBREVIATIONS

- 2D : Two Dimensional
- BMR : Bottle Microresonator
- CaF_2 : Fluorite
- CH₂O : Formaldehyde
- CO₂ : Carbon Dioxide
- CQED : Cavity Quantum Electrodynamics
- D_b : Bottle Diameter
- D_s : Stem Diameter
- DUV : Deep Ultraviolet
- EBL : Electron Beam Lithography
- FSR : Free Spectral Range
- GaInp : Gallium Indium Phosphide
- L_b : Neck-to-neck or Bottle Length
- MBR : Microbottle Resonator
- NIL : Nano-imprinting Lithography
- nm : Nanometer
- OMR : Optical Microresonator
- OPM : Optical Power Meter
- Q : Quality
- RIU : Refractive Index Unit
- Si : Silica
- Si₂N₃ : Silicon Nitride
- SiO₂ : Silicon Dioxide
- SMF28 : Single Mode Fiber 28

- TLS : Tuneable Laser Source
- WGM : Whispering Gallery Mode
- WGR : Whispering Gallery Resonator
- XeF_2 : Xenon Difluoride
- μ : Micron or Micrometer

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CHAPTER 1: INTRODUCTION

1.1 Optical Whispering Gallery Mode

Whispering gallery modes (WGMs) or modes of a wave field (e.g. electromagnetic waves, sound waves etc.) or waves are particular resonances within a given cavity (a resonator) with smooth edges. They supported by the cavity surface during continuous total internal reelection and round the cavity correlate with wave circling that fulfill the requirement of the resonator (They meet at the same point after one round trip with the exact phase and therefore interfere constructively between each other, forming standing waves). These type of resonances of the resonator cavity depends greatly upon the geometry (Feron, 2004).

In the 19th century for the first time, the term WGM waves were used by Lord Rayleigh. It was located in London under the dome of St. Paul's Cathedral which describes the phenomenon of the WG. It was known that in one end of the dome if a whisper (a sound) uttered at the opposite side of the dome was able to hear the sound loudly despite of quite far away from the source. Inside the cathedral's dome (with R radius) the gallery with smooth curved which allows the sound waves (by resonance wavelength h_{rec}) above its surface for reflection through the circumference ($h_{rec} \cdot m = 2nR$) of an integer fraction m. Previously in electromagnetic waves the application of WGMs, the light which defines by optical microcavities repeated reflection over a long period of time to transform into either linear cavities (e.g. Fabry-Pérot, DFB, and VSCEL) (Kringlebotn, Archambault, Reekie, & Payne, 1994; Lott et al., 2000; Mohd Narizee Mohd Nasir, Yusoff, Al-Mansoori, Rashid, & Choudhury, 2009) or circular cavities (all fiber ring structure) (Duling, 1991). The light restricting properties of resonators depends on the quality factor Q and mode volume V. In order to gain high $\frac{Q}{V}$ ratio, the principles

of WGMs applying into several number of optical micro-cavities which have been expressed since the conception by Richtmyer was moved into the electromagnetic waves domain (Richtmyer, 1939).

Optical WGMs initiating work was demonstrated through employing a spherical (CF₂: Sm²⁺⁾ structure and coupling it into a free space master (Garrett, Kaiser, & Bond, 1961). The range of Qs (108-109) with high-quality resonators were later on explained the fused silica (SiO₂) through melting fiber tips in order to generate uniformity and high purity in optical WGM microspheres (Braginsky, Gorodetsky, & Ilchenko, 1989; Gorodetsky, Savchenkov, & Ilchenko, 1996). Cylinders are the simplest structure of optical WGM resonators. These type of structure can be made of through polymer coating of standard single mode fibers (SMFs) with striping-off (Birks, Knight, & Dimmick, 2000). Although, they affected by low Qs and high loss because of their longitudinal degree of freedom and into the resonators as the coupled light of WGMs spreads with the leak out and cylinder. With the beginning of microtoroid and microdisk WGM resonators the performance has been significantly increased. Because of high surface roughness was induce which limit the Q-factor of microresonator and increase the scattering loss. They executed the laser reflow to assure smooth surface for Q up to 108 during microtoroids fabrication (D. Armani, Kippenberg, Spillane, & Vahala, 2003). In addition, the microtoroid and microdisk resonators are more suitable in terms of geometry shape for integration on chip (T. Kippenberg, Spillane, & Vahala, 2004).

At present, within high temperature state the WGM microdiscus resonators via "squashing" microspheres has been described along Q where the range is 105 (Senthil Murugan, Wilkinson, & Zervas, 2012). Recently, a new kind of optical WGM microsresonators catches the attention which known as microbottle resonators (MBRs). The conception of MBRs is different than the other WGM resonators. It mainly lies in the WGMs survival state by plane deformation of spheroids. WGM microresonators that are described earlier can trap light with their circumference by 2D confinement whereas MBRs capable to support the combination of WG bouncing ball and WG ring principles through true 3D confinement (M Sumetsky, 2004).Improvement of the strong light field attached to the WGM modal confinement area which defines by two unique turning point of MBR (Louyer, Meschede, & Rauschenbeutel, 2005). As such, a free spectral range (FSR) with nondegenerate WGMs in a magnitude order lesser than the same diameter with microspheres which is able to sustain by MBRs (Ganapathy Senthil Murugan, Wilkinson, & Zervas, 2009). The dense is easily accessible in WGM generation ever since the various radius of MBR is able to capture the light even at the nanoscale which adjacent to its surface (Mikhail Sumetsky & Fini, 2011).

The fundamental method of MBR included "heat and pull" process. This process relies on the microtaper fiber two sections to form a bulge area with homogeneous diameter (Kakarantzas, Dimmick, Birks, Le Roux, & Russell, 2001; Ward et al., 2006; Warken, Rauschenbeutel, & Bartholomaus, 2008). Later on, the fabrication progression was enhanced and facilitated by the "soften and compress" thermos mechanical process. Whereas, to generate a strong bulge region along parabolic profile a portion of an ordinary optical fiber is compressed and heated (Ganapathy Senthil Murugan et al., 2009). Q-factor of MBRs intrinsically were calculated within the range of 108 (Pöllinger, O'Shea, Warken, & Rauschenbeutel, 2009) along 107 which was experimental values successfully demonstrated (Zervas, Murugan, Petrovich, & Wilkinson, 2011).

1.2 Problem Statement

Years after years, WGM resonators are not only contributing to fundamental research but also contributing widely in various applications such as micro-lasers, sensors, filters, an optical delay lines and cavity quantum electrodynamics (CQED) (Ilchenko & Matsko, 2006; Vahala, 2003). Simultaneously, several shapes of microresonators (e.g. spheres, toroids, disks and cylinders) have been discovered with the rationally symmetric configuration being broadly used.

Various work has been done regarding liquid sensing with different kind of microresonators. WGMs with microspheres resonator has been applied for the optical biomolecules sensing with the Q-factor of $\sim 10^5$ whereas sensitivity and reliability were not effective for biomolecules sensor (Nadeau, Ilchenko, Kossakovski, Bearman, & Maleki, 2002). A slot-waveguide microring resonator demonstrated an integration biochemical sensing for the use of the biomedical sensor. For that work, the microresonator was fabricated through silicon nitride (Si₂N₃) and silicon dioxide (SiO₂) which operated at the wavelength of 1.3 µm (Barrios et al., 2007). In addition, optical microring resonator has been utilized subwavelength wall thickness for optofluidic sensing such as earl-time bioanalytic sensing. In this way, they fabricated the microring resonator through silicon oxide or silicon dioxide which can be increased sensitivity and they able to produce a sensitivity of 400 nm/RIU (refractive index unit) by using ring microresonator (Huang et al., 2010). Coated microcoil has also been utilized for refractometric sensing where the sensitivity depended on the coil diameter waist and the thickness of the coating. They achieved a sensitivity of 700 nm/RIU (Xu, Horak, & Brambilla, 2007).

Nowadays a new kind of optical WGM microresonator named as bottle microresonators (BMRs) or microbottle resonators (MBRs) catch the attention compared to other optical resonators because of its distinguishable characteristics. Among other microresonators, it has some advantages such fast tunability through the strain application, better control over the coupling by optical tapered microfiber and in the spectrum the probability of attaining a great number of equally-spaced modes. To date,

as we notice that a wide range of investigation has been done for liquid sensing through various types of technique. In this thesis, the motivation behind this research to use the microbottle resonator for liquid sensing and develop a high sensitivity, cost-effective, high dynamic range and a non-tedious sensing system. The fabrication of the BMRs depends on three parameters which are: bottle diameter D_b , neck-to-to diameter L_b and stem diameter D_s (M Narizee Mohd Nasir, Ding, Murugan, & Zervas, 2013). For sensing application, the significant part is the light coupling into the cavity and out of the cavity. One of the technique is through microtaper fiber excite the modes of the cavity. For this study, several kinds of bottle diameter (5µm, 8 µm and 10µm) have been utilized towards sensing. Furthermore, this investigation can be effective in the field of the sensor.

1.3 Objectives

The principal aim of this experiment area is to investigate the influences of microbottle resonators for formaldehyde sensing through tapered optical microfibers and examine the effectivity of the resonators. The objectives which are given below have to met:

- 1. To understand the fundamental of the optical microresonator.
- To know about the phenomena of the microbottle resonator regarding formaldehyde sensing.
- 3. To conduct an experiment with consequences of the microbottle resonator for different concentration levels of formaldehyde.
- 4. To analyze the performance of the microbottle resonators as formaldehyde sensing.

1.4 Report Outline

The thesis is ordered into five chapters, each of which is then subdivided into sections and subsections. Chapter one presented an introduction of this work comprising the background study, problem statement, and aims of the research study. Chapter two explained the fundamental of microresonators, their properties and the characterizations of different types of microresonators. The fabrication process of MBR, use of MBR along with bare fiber and differences between MBR and bare fiber in terms of performance are briefly described in chapter three. Chapter four demonstrated the effects of different bare fibers with MBR for the formaldehyde liquid sensing. In the end, the overall summary was stated in chapter five along with references.

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CHAPTER 2: LITERATURE REVIEW

2.1 Optical Microresonator

Optical microresonator (OMR) has been made up based on the characterization of the WGMs. The optical microresonator or optical microcavity has a resonant frequency spectrum which is size dependent as similar its acoustic analog with the tuning fork. Volumes of microscale assure that frequencies of the resonant are more sparsely which is distributed during this spectrum compared to the corresponding resonator with microscale. A perfect cavity or resonator with microscale able to trap the light indefinitely which means without loss and also have frequencies of the resonant at specific values.

The confinement of the resonator in the spectra along narrow lines directed to the appearance of resonant electromagnetic modes. Label-free sensing is one of the promising applications of them, because of the optical modes sensitivity to external perturbations. Generally, the resonant devices sensitivity increases as the resonant features linewidth decreases. The Q-factor is inversely proportional and the optical losses are proportional to the linewidth of the resonant ($Q = \frac{\lambda_0}{\Delta \lambda}$, where the linewidth is $\Delta \lambda$ and the resonance wavelength at the center is λ_0). WGM microresonators in where the light confined with an axis-symmetric cross-section in a dielectric shape through total internal reflection, which has shown low intrinsic losses (A. B. Matsko & Ilchenko, 2006).Because of these losses, they are more potential for sensitive detection in extreme situation (Foreman, Swaim, & Vollmer, 2015). Different kind of WGM microresonator has already been used such as disks, tubes, rings, bottle, spheres, cylinders etc. For coupling, the light with high-efficiency tapered fiber replaced the side-polished fiber on the basis of the microresonator. Tapering process is done by heating and stretching technique which transforms the optical fiber to a narrow thread (Amitay & Presby, 1989).

The waist of the tapered fiber in the form of micron or micrometer as diameter. The microresonator placed at center of the tapered fiber. The signal or wave pass through the resonator and goes from one end to the other end of the tapered fiber.

2.1.1 Application of OMR

Basis on the WGMs microresonators has a vast range of useful applications. Such applications are (Ilchenko & Matsko, 2006) : spectroscopy, interferometry, fluorescence studies (due to their high finesses and quality factors), light storage devices (due to long storage of photon), metrology, in optical telecommunication sectors as filters, study about nonlinear effects of optical of optical frequency combs (at moderate powers pumping due to much high intensities circulating), research on non-classical cavity and light quantum electrodynamics or effects of CQED (where Q-factors with ultra-high are required), bio-sensing and several sensing applications(temperature, gas, molecule detection).

2.1.1.1 Photonic Filter

Usage of photonic filters built on optical WGRs is the best developed applications among the whispering gallery resonators. The intention is to utilize them in the optical communications filed for processing signals whereas Q-factors of ring resonators are more adequate. The general designs which are shown in figure 2.1 are a WGR coupled either into a taper bus of single fiber (drop filter) or into two buses (both add/drop filter) (Ilchenko & Matsko, 2006). Both add and drop filters are valuable for WDM (wavelength division multiplexing) (whereas numerous different wavelength signals convert into a single optical fiber), since then they single filter out those signal which equivalent the frequencies of resonant of the resonator and abandoned other signals in the fiber which is unchanged. In an add and drop filter, a resonant wavelength with a signal can able to be added to signals stream in the optical fiber (Gomilšek, 2011).



Figure 2.1 Comparison of an add/drop (right) and a drop filter (left) WGM design (Tobing & Dumon, 2010)

2.1.1.2 Sensors

Since the WGMs evanescent field protrudes externally the volume of the resonator as resonator modes are influenced by the environment in which the resonator is located. The atmosphere affects both the frequencies of resonator as well as WGMs quality factors. Due to WGRs may have exceptionally large quality factors a shift in their frequencies of resonant which is easily calculated. It means that they perform as sensors with high sensitivity which able to affected through things for instance the temperature, pressure and chemical structure of their surroundings (to enhance the selectivity we can treat the resonator surface therefore it binds only exact molecules) (Gomilšek, 2011).



Figure 2.2 By whispering gallery resonator detecting the presence of influenza A virus (A. M. Armani, Kulkarni, Fraser, Flagan, & Vahala, 2007)

An arrangement is presented below which detecting the presence of viruses which bind to the glass surface of a microsphere which is shown in the figure. A tapered microfiber has been utilized to achieve the coupling and tunable laser system sweeps across variant wavelengths to define the frequencies of resonance of the resonator (the laser beam transmission on resonance by the optical fiber drops) which straightly rely on the number of limited viruses (A. M. Armani et al., 2007).

2.1.1.3 Lasers

Through doping the WGR using a lasing medium (such as a quantum dots, a dye or nanocrystals), where laser operation can be achievable in such resonators (in solid state resonators or droplets). As laser resonators utilizing the WGRs has some advantages. Such as ultra-high quality factors and very small mode volumes which straightly transform into thresholds ultralow lasing (optical pimping power below one μ W and even smaller). Due to their compact size and easy on-chip integration, they are very promising and effective for lasing applications. Therefore we able to couple various such resonators into one optical fiber and even multi-wavelengths lasing also achievable (Ilchenko & Matsko, 2006). An example is given below regarding laser. An ultralow threshold laser with lasing threshold 65 nW (Ilchenko & Matsko, 2006) at $\lambda_0 = 1088.2$ nm with quality factors of 1.4x10⁸ that has been gained through a microsphere which is made of silica and doped with neodymium(3+): gadolinium oxide phosphors (Nd³⁺:Gd₂O₃). Sub-nanowatt threshold lasing is another example which is at 15k temperature with gallium indium phosphide (GaInp) microdisk and microresonators (Q = 5x10³ and diameters 1mm - 3mm) with embedded indium phosphide (Inp) quantum dots (Chu et al., 2011).

2.2 Different types of OMR

2.2.1 Toroid Micro-resonator

A toroid micro-cavity or microresonator is prepared though a dielectric material. The shape of the dielectric material is a solid toroid. Inside of the solid toroid light able to disseminate through constant bouncing by total internal reflection off the air interface of the toroid which is shown in figure 2.3 (T. Kippenberg et al., 2004). It uses the conception of the whispering galley modes which are almost same for the ring, disk and ball microresonators. Because of whispering gallery modes planarity shares a greater portion of the amenities of employing such resonators rather than spherical ones. The toroid micro-resonator fabrication part is slightly more included coupling than the disk or ring resonators, however is yet easy to chip integrable.



Figure 2.3 Fabricated a toroid microresonator from a thermal oxide (left) and an alignment of toroid microresonators (right) (T. J. A. Kippenberg, 2004)

Toroid microresonator has a lot of potential advantages. One of the biggest advantages compared to other resonators is they able to gain ultra-high Q factors in the sequence of $\sim 10^8$ and even $\sim 5 \times 10^8$ (compared to sphere microresonators because of the conduct of surface during their fabrication which present them a quiet soft surface and numerous

magnitude orders which is larger than the disk resonators Q-factors) (A. M. Armani & Vahala, 2007; T. J. A. Kippenberg, 2004), while sharing simplest fabrication of utmost advantages and integration of the resonators which is disk.

2.2.1.1 Fabrication

The constructing process for producing a microtoroid is demonstrated on figure 2.4, which is shown below. At the first step, a circular silica (SiO₂, silicon dioxide) disk is well defined through dry etching, after that a little amount of the Si (silicon) underneath of the disk which is shifted through isotropic etching utilizing XeF₂ (xenon difluoride) gas to confinement of the light vertically (the rest of the remaining Si functions as a post which use to assists the disk). Later on, at the final stage with the help of a CO₂ (carbon dioxide) laser the Si (silica) finally melted through irradiating process. Then the melted Si transforms into a soft toroidal shape by the use of surface tension at the disk edges section (the internal section do not restructure of the disk as much when they rapidly move their heat by the Si post, where the disk has maximum heat conductivity compared to silica) (Tobing & Dumon, 2010).



Figure 2.4 Demonstration of the fabrication process of the toroid microresonator and a view of completed microtoroid (Tobing & Dumon, 2010).

Toroid microresonators have been built up through this process with the fundamental phenomena D diameters among 80 μ m and 120 μ m, the torus shape thicknesses diameter (d) of 5 μ m and 10 μ m and also the Q-factors of ~10⁸ (compared to the sphere microresonators, because of the softness of the surface which is made of surface tension at the time of fabrication). The process of the fabrication which provides to easily control the size of that particular fabricated microtoroid than the fabrication processes for building sphere microresonators, whereas microtoroid planar geometry provides integration in much easier way into optical cavity then is probable for sphere microresonators (Tobing & Dumon, 2010).

2.2.2 Dielectric Sphere Resonator

Based on the WGMs one of the easiest resonators is a dielectric sphere resonator which has a higher refractive index compared to the surrounding material. From the perspective of a geometric optics, the incoming light which travels nearly to the sphere edge is constantly reflected. This happens inside the sphere through total internal reflection at the air interface cavity and cannot come out of the sphere. That means the light trapped inside the sphere. If the light beam which is circulating returns to the exact point along the exact phase then it intervenes constructively by itself and forms resonant waves (a resonance). Positioning the coordinate system is normal so that the light beam circulating around the sphere (azimuthally). The surface of the sphere also provides to concentrate the light into the vertical polar direction. Because the curvature of the polar direction of the polar sphere travels at the same optical path which is effective as it was zigzagging on all direction of the equator in place of going at a straight line which is shown in figure 2.5. This can be understood from the light polar confinement by an extra Gouy phase shift (an optical path with feasible lengthening obvious from the phase beam over the phase beam which is expected measured from the original distance that traveled) of usual Gaussian beams so they move by their focal points (Little, Laine, & Haus, 1999).

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Figure 2.5 Microsphere resonator fabrication through electric arc heating and tuned into a globe (left)(Laine, 2003), modes of the silica microsphere with 300µm (center) (A. Matsko, Iltchenko, Strekalov, Savchenkov, & Maleki, 2008) and approximation of the geometric optics to WGM propagation (right) (Little et al., 1999).

The approach of a wave optics essentials to be utilized for a spherical microresonator with full resonant behavior characterization instead of the description of geometry. For our understanding assume that light which provides us two primary corrections. First of all, inside the sphere truly the light is not bouncing off at the edge however it guided smoothly with the sphere edge. Secondly, at a curved edge of a wave with total internal reflection is certainly not complete that means the spherical interface associated with the bending losses and gradually the light leaks out from the sphere (set a boundary over higher attainable Q-factors instead of the material which made up from the sphere) (Gomilšek, 2011).

2.2.2.1 Fabrication

Sphere microresonator is normally fabricated through surface tension. It has been expressed by means of spheres which is made of materials in crystalline, liquid and amorphous structures. The optical microresonator at the early stage which is demonstrated simply is a micron (μ) sized droplet of liquid along a proximate perfect surface of spherical caused by surface tension (Tobing & Dumon, 2010). As WGMs the

most efficient use of droplets is hindered. The fact is they gradually evaporate and really hard to operate or manipulate compared to the solid state resonators (Tobing & Dumon, 2010). However resonators with liquid really effective in lasing, spectroscopy and fluorescence in dyes which already proved over the years. At present time, droplets of liquid crystal shown to be as possible as two magnitude orders more tunable over any resonator of solid state (externally at moderate voltages tuning through an electric field which has more spectral range) with a max Q-factor of 12,000 which probably opening the gate for the new fields such as lasers, sensors etc. (Humar, Ravnik, Pajk, & Muševič, 2009)

For the first time solid state MSR (microsphere resonator) was explained in fused SiO₂ (silica) (Tobing & Dumon, 2010). If the top point or tip of a SiO₂ optical fiber is melted through an electric arc or a flame then that particular melted area of the silica forms a soft sphere which minimizes the surface energy of the sphere which shown in figure 2.5. Later on when the flame or arc removed then the melted area of silica solidifies into a shape of microsphere where the radius of the sphere controlled through adjusting the fiber tip size. The shape and size of microsphere which is reproducible have been expressed with diameters of sphere between 50µm and 100µm along the Q-factors of ~10⁹. Microspheres with fused silica are so sensitive to outer contaminates for instance -OH absorption, water absorption, and has to be careful to certify an inert atmosphere for the microresonators (Tobing & Dumon, 2010).

Recently, a record has been made by spherical resonators. It has the most measured Q-factors for a WGR (whispering gallery resonator). For fused silica Q-factors of 8×10^9 (with the finesse of 2.3×10^6) at $\lambda_0 = 633$ nm has been measured and for CaF₂ (fluorite) crystalline Q-factors of 3×10^{11} (with the finesse of 2.1×10^7) at $\lambda_0 = 1.55$ µm has also been explained (Savchenkov, Matsko, Ilchenko, & Maleki, 2007).

2.2.3 Disk, Cylinder and Ring Microresonator

The whispering gallery modes also supported by the disks microresonator (low height with cylinders) and dielectric cylinders. Cylinders almost act like as sphere according to the WGMs which they support (by an analogous way light spreads all over the place of the cylinder to light rounding around the sphere equator) however the fundamental phenomenon is not similar. While the modes of the sphere below perturbations away from the propagation which is equatorial, in the same way, is not accurate for cylinders of the dielectric. For a sphere, while the curvature with polar form in sphere surface focuses and confines light in the way of polar direction in a light beam of the cylinder of dielectric which is appropriately perturbed surrounding the equator by propagating. They do this so that they can escape over the bottom or top of the dielectric cylinder and therefore, it leaves the microresonator (Gomilšek, 2011).

Because of the instability of the mode in the polar direction on account of the lack of focusing, and it is difficult to fabricate disks with parallelly a spherical resonator which has less surface roughness, the cylinder resonator Q-factors, and disk microresonators are generally much less than the spheres (normally for disks). However, the fabrication process is much easier, more able to control and much faster due to their planar geometry and easier to integrate into an optical integrated network or over a chip, whereas at the same moment considering much lower space than the radii of the microspheres with smaller volumes of the mode. In this ways, it makes them so effective according to the practical applications. (Tobing & Dumon, 2010)

Variations of resonators with the disk are ring resonators (at the middle of disk resonators with a circular hole) which are shown in the figure. Since WGMs are extremely restricted at cavity air interface ring which has exactly the same structure of whispering gallery modal as resonators with disk while radial with high order modes are better suppressed. Ring resonators have an additional advantage. It permits numerous times smaller mode volumes at only a volume fraction of the material (dielectric) (Gomilšek, 2011).

2.2.3.1 Fabrication

The fabrication of cylinder, ring and disk resonators can be done through one of the three processes: DUV (deep ultraviolet) lithography, EBL (electron beam lithography) or NIL (nano-imprinting lithography). DUV lithography which has maximum throughput, which is easily matched with CMOS however because of the only ~100nm feature resolution which creates some surface roughness. EBL which has resolution feature of ~10nm and has fewer effects for nearly packed structures than deep ultraviolet lithography. NIL which has both high feature resolution and high throughput. (Tobing & Dumon, 2010).

DUV lithography utilizes UV light at the wavelength of 193nm or 248nm to define the structure of resonator by etching the substrate whereas for etching EBL uses speeded or accelerated electrons. NIL first need the structure of resonator which to be fabricated utilizing either EBL or DUV lithography. After that around the structure, a polymer is molded and solidified to produce a solid mold. Later on, this mold can be utilized as a resonator.



Figure 2.6 Ring resonator (right) (Tobing & Dumon, 2010) and disk resonator (left) (Srinivasan, Borselli, Painter, Stintz, & Krishna, 2006)

These three processes can able to create the structure of resonator with the same intrinsic quality factor of $\sim 3x10^5$ and Q-factor of $\sim 5x10^4$ which is total. DUV lithography also able to build resonator with the finesse of ~ 3.600 and loaded finesse of ~ 600 . (Tobing & Dumon, 2010)

2.2.4 Microbottle Resonator

An OMR (optical bottle resonator) or MBR (microbottle resonator) is kind of microresonator which is made by an optical fiber (a lengthy dielectric fiber which made of plastic or silica). At the middle of the resonator, it has a bulge area whereas in the fiber the bottle thickness is a little bit increased compared to the surrounding fiber thickness. Incoming light circulates alongside the fiber circumference and perpendicularly to the optical fiber symmetry axis. It is radially confined the light continuously through total internal reflection (like in a cylinder or disk resonator) however additional axial confinement is gained through the slowly changing of the optical fiber thickness (similarly to the spherical resonators polar confinement and in opposition with same optical fiber where light is not confined in the direction of axial to allow light guiding down the fiber) (O'Shea, Junge, Nickel, Pöllinger, & Rauschenbeutel, 2011).



Figure 2.7 Geometry of microbottle resonator (O'Shea et al., 2011)

2.2.4.1 Spectrum

Generally, the thickness profile of the optical fibers around the bottle resonator is almost parabolic in the axial direction $z : R(z) = R_0 (1 - \frac{1}{2} (\Delta k \cdot z)^2)$, in where the bottle

maximum radius is $R_0 = R(0)$ and Δk is the resonator axial curvature. This fibers thickness profile produces an effective LHO (linear harmonic oscillator) as potential in the axial direction. Therefore, complete light confinement inside the resonator is gained which holds in the adiabatic (also can call Born-Oppenheimer) estimation ($\left|\frac{dR}{dz}\right| << 1$). (O'Shea et al., 2011)

The electromagnetic field Eigen-modes can be written as coordinates of cylindrical (r, φ , z) inside the optical fiber as (utilizing the first kind Bessel functions J_m and linear harmonic oscillator Eigen-functions Z_q):

$$\Psi_{m,q}(r,\varphi,z) = A e^{im\varphi} J_m\left(\frac{mr}{R(z)}\right) Z_q(z)$$
(2.1)

Where q is the axial mode number and m is the azimuthal and they alongside with the polarization p (transverse magnetic or transverse electric) which define the uniqueness of the mode (the multi-index mode is $\zeta = (m, p, q)$). Inside the LHO we can imagine visualize that light bouncing to and fro in the axial direction which is shown in figure 2.8, creating a standing wave in where the resonant condition is met. Light introduces a caustic (an area which significantly increased intensity) on the resonance of the resonant or at the tuning point $\pm z_c$ inside an LHO for classical motion. In this way, we can think light "bouncing back", as it hitting the mirror (such as Fabry-Pérot interferometer). (O'Shea et al., 2011)



Figure 2.8 Comparison between the OMR and Fabry-Pérot resonator (O'Shea et al., 2011) and a monograph of q = 1,2,3,4 modes (Pöllinger et al., 2009)

The OMR spectrum is given through the number of the wave inside the bottle k_1 as (O'Shea et al., 2011):

$$k_{1,m,q} = \frac{2\pi n}{\lambda_0} = \sqrt{\frac{m^2}{R_0^2} + \left(q + \frac{1}{2}\right)\Delta E_m} = \frac{m}{R_c}$$
(2.2)

Here LHO energy spacing is $\Delta E_m = \frac{2m\Delta k}{R_0}$, λ_0 is the light wavelength in vacuum and $n=\sqrt{\epsilon\mu}$ is the optical fiber refractive index. At the caustic $R_c = R$ ($\pm z_c$) indicates the bottle radius. Due to the higher m and higher q the radius of the fiber getting shrinks and the caustic axial position proportional to the mode numbers (Gomilšek, 2011).

2.2.4.2 Fabrication

The fabrication of MBR has been done through "soften and compress" method. It has some advantages which include simply manufacture process (fibers are easily fabricable and the fiber thickness can straightforwardly be improved through stretching and heating the fiber) and higher tunability (mechanically during stretching process the fiber thickness changes itself and also the microbottle resonant frequencies, alternative way is electrical thermo-optic tuning), while also sustaining the typical toroid and spherical resonators ultra-high Q-factor (O'Shea et al., 2011). The actual fabrication of MBR has been briefly demonstrated in Chapter 3.

2.3 Microresonators Parameters

Microcavities or microresonators rely on several significant parameters. But quality (Q) factor and free spectral range (FSR) are really crucial among them.

2.3.1 Q-factor

Microresonator quality factor is a unit-less parameter. It is used to determine the damping strength of its oscillations and corresponds to the light confinement inside the resonator. It is usually described as the stored energy ratio to the power loss (Jung,

Brambilla, & Richardson, 2010) and how long inside a cavity or resonator a photon can be stored is also measured by

$$Q = 2\pi \frac{\text{stored energy}}{\text{power loss per roundtrip}}$$
(2.3)

To calculate the overall quality factor of a WGM microresonator many mechanisms involved with it. They are connected through

$$\frac{1}{Q_{total}} = \frac{1}{Q_{WGM}} + \frac{1}{Q_{mat}} + \frac{1}{Q_{cont}} + \frac{1}{Q_{ss}} + \frac{1}{Q_{coupling}}$$
(2.4)
$$= \frac{1}{Q_{coupling}} + \frac{1}{Q_{intrinsic}}$$

In where Q_{total} is the total cavity Q-factor. Intrinsic Q-factor is the combination of three parameters of the resonator which are Q_{mat} (material loss), Q_{WGM} (radiation loss because of the dielectric cavity curvature), Q_{ss} (surface scattering) and Q_{cont} (any contamination over the resonator). $Q_{coupling}$ defines the energy loss because of the input or output coupling. Based on the OMR mechanisms it can affect values of Q-factor through material intrinsic absorption and radiation losses due to roughness scattering of the surface or waveguide bending. Externally the tapered fiber and microresonator Q-factor can be measured (Mohd Narizee Mohd Nasir, G Senthil Murugan, & Michalis N Zervas, 2016b; Vahala, 2003) by following way

$$Q_e = \frac{m\pi}{k^2} \tag{2.5}$$

The Q-factor of the resonator also associated with the resonance $\Delta\lambda$ linewidth at operating wavelength λ and the lifetime of photon inside cavity τ by

$$Q = \frac{\Delta\lambda}{\lambda} = \omega_0 \tau \tag{2.6}$$

Here ω_0 ($\omega = 2\pi c / \lambda$) represents the optical frequency.

2.3.2 Free Spectral Range

The FSR of a cavity is generally measured as the spacing of frequency of its axial cavity modes. Physically when the size of the resonator decreases (depends on path length) then it's FSR increases which means they are inversely proportional to each other. The mode defines the free spectral range are the successive modes which have the exact structure of transverse mode. The azimuthal ($\Delta v_m = v_{m+1,q} - v_{m,q}$) and axial ($\Delta v_q = v_{m,q+1} - v_{m,q}$) FSRs can be extracted from the wave function eigenvalues $k_{m,q}$. They can be estimated through

$$\Delta v_m = \frac{c}{2\pi n} \left(k_{m+1,q} - k_{m,q} \right) \approx \frac{c}{2\pi n R_0}$$
(2.7)

$$\Delta v_q \approx \frac{c\Delta k}{2\pi n} \tag{2.8}$$

CHAPTER 3: MICROBOTTLE RESONATOR FOR FORMALDEHYDE (CH₂O) LIQUID SENSING

3.1 Introduction

Optical microresonator (OMR) in the several structures, for instance, microdisc, microsphere and microrings have undergoing demanding inquiry research in chemical and biological sensor application recently (Hanumegowda, White, Oveys, & Fan, 2005; Krioukov, Greve, & Otto, 2003; Nadeau et al., 2002; Frank Vollmer, Arnold, Braun, Teraoka, & Libchaber, 2003; Fea Vollmer et al., 2002). By utilizing the total internal reflection of the modes the whispering gallery modes (WGMs) construct inside the resonator on the surface of arc borderline. The WGM has a fleeting field outside the OMR with a trademark length of tens to several nanometers and consequently is delicate to the refractive index change prompted by the authoritative of natural or potentially synthetic atoms to the resonator surface. Because of the light reusing nature and high Q-factor related with the WGMs, the light-matter cooperation is improved altogether. The Q-factor of a resonator does not have any unit which means it is dimensionless. It defines the damping quality of its motions and in the resonator compares to the worldly light repression. The quality factor is, for the most part, characterized as the proportion of put away vitality towards the power misfortune. It is also a dimension for to what extent a photon be able to put aside in a pit (Michelitsch et al., 2011). Subsequently, an OMR sensing with sensor has high affectability, a little impression, low test utilization, and multiplexing capacity.

Investigation of optical OMR supporting WGMs has additionally been stretched out to incorporate barrel-shaped structures, for example, optical strands, or OMR framed on filaments for their unmistakable way in restricting light and in addition for simple dealing with and consolidation in viable applications (Birks et al., 2000; Ilchenko, Gorodetsky, Yao, & Maleki, 2001). As of late, there has been expanded action on another tube-shaped microresonator compose, to be specific the bottle microresonator, which, conversely with the cases above. Micro-bottle resonators are strong, solid prolate spheroid structures, which bolster exceptionally non-decline WGMs. Contingent upon the excitation course of action, a rich assortment of modes can be productively energized, not at all like microspheres where mode-decline covers such excitation (Ganapathy Senthil Murugan et al., 2009). Specifically, noteworthy are modes that display two all-around isolated spatial areas along the MBR hub with upgraded field quality, comparing to modular defining moments. By mixing up the whispering-gallery ball and ring standards, genuine 3-D WGM light confinement can be bolstered by MBRs (M Sumetsky, 2004).

This chapter examines the execution of MBR based formaldehyde (CH₂O) liquid sensing. The MBR was manufactured by the supposed "soften-and-compress" procedure from a standard SMF28. The MBR is first portrayed by utilizing a $5\mu m$ microfibre before the being utilized for a range of concentrations of 0% - 5%, and afterward contrasted and exposed fiber for detecting execution.

3.2 Fabrication of MBR

The fabrication process of the microbottle resonator for this work has been done through a method which is called "soften-and-compress" (Zervas et al., 2011). An SMF-28 with continuous length in clamped in a manual splicer on two sides (Furukawa Electric Fitel S178A) whereas a small section of the fiber is heated under a plasma arc. At the same time, the two ends of the optical fiber are compressed inward in the plasma arc direction and as a result, it transforms into a structure of bottle which is depicted in figure 3.1.After the fabrication process, the characterization of MBR determined by three parameters which are, the stem diameter D_s , the bottle diameter D_b and the neck to neck length L_b . By increasing the plasma arcs number that employed on the fiber, it is possible to produce various type of bottle size D_b .



Figure 3.1 Microbottle resonator with D_b , D_s and L_b

3.3 Formaldehyde

Formaldehyde is a basic substance compound made of hydrogen, oxygen and carbon. It is commonly known as formalin. All life frames for instance microbes, plants, fish, creatures and people normally deliver formaldehyde as a feature of cell digestion. Formaldehyde is maybe best known for its additive and hostile to bacterial properties. However, formaldehyde-based science is utilized to make an extensive variety of significant worth included items. Formaldehyde is a standout amongst the most allaround contemplated and surely knew mixes in trade.

3.3.1 Fabrication of CH₂O

The creation of formaldehyde for this experiment has been done by using both the formalin volume and distilled water. Distilled water is a kind of water which boiled into the steam and evaporated back into the liquid in a distinct container. In industries, chemical & biological laboratories as well as for many other purposes, deionized water has been used which referred to as distilled water. The formalin has been used for this

experiment has 37% purity which means it is made of 37% formaldehyde and impurities such as methanol, small amounts of formic acid, aldehydes and ketones. So, each concentration levels constructed based on the following equation:

$$V_1 \times N_1 = V_2 \times N_2 \tag{3.1}$$

Whereas V_1 is the formalin quantity, N_1 is the percentage of pure formalin which is 37%, V_2 is the total volume of the concentration and N_2 is the concentration level (Farrell, 1993). In this work, 0% concentration of formaldehyde is made of pure deionized water. For making 100 ml of 1% concentration level of formaldehyde, 2.7 ml volume of formalin has been added with 97.3 ml distilled water. Therefore, as the concentration level (1% - 5%) increasing then the addition of formalin quantity also increasing while the volume of deionized water decreasing. The fabrication of different CH₂O concentration levels shown in the following figure 3.1.

Concentration level of formaldehyde (%),	Formalin Quantity (ml),	Volume of distilled water (ml)	Total Volume (ml),
N2	\mathbf{V}_1		\mathbf{V}_2
0	0	100	100
1	2.7	97.3	100
2	5.4	94.6	100
3	8.1	91.9	100
4	10.8	89.2	100
5	13.5	86.5	100

Table 3.1 Fabrication different concentration level of formaldehyde

3.4 Experimental Setup

The experiment continues by forming MBR using standard silica fiber SMF-28 using technique called "soften-and-compressed" (G Senthil Murugan, Petrovich, Jung, Wilkinson, & Zervas, 2011). This technique used manual splicing machine (Furukawa Electric Fitel S178A) which able to form bottle structure in the middle of fiber by

applying for several arc numbers. This makes a lump in the focal point of the fiber, with the size being dictated by the quantity of bends utilized (Ganapathy Senthil Murugan et al., 2009). The WGM utilized in this experiment on bottle shape resonator after been coupled with $5\mu m$ bare fiber, which created by tapering fine process. The subsequent MBR structure is then physically described by three parameters, to be specific, the bottle distance across, the stem width D_s , the bottle diameter D_b and the neck-to-neck length L_b , as shown in the figure 3.2. In this work, D_b was set at 190 µm. A biconical optical microfibre with a midriff distance across of 5 µm, manufactured by the fire brushing strategy, is utilized to optically energize the MBR (Lim, Harun, Arof, & Ahmad, 2012).



Figure 3.2 Fabricated optical MBR with $L_b = 182 \ \mu m, D_s = 190 \ \mu m, D_b = 125 \ \mu m$

The tuneable laser source (ANDO AQ4321D) utilized the wavelength range from 1520 nm to 1620 nm to characterized the MBR through a non-adiabatic microfiber with a 5 µm waist diameter tapered fiber. By 0.001 nm wavelength interval, the laser was adjusted between 1551.0 nm to 1551.7 nm and at the end, the transmitted power collected through the optical power meter (THORLABS S145C). Figure 3.3 depicted the MBR transmission spectral in which the peaks of sharp resonant can be simply noticeable (Mohd Narizee Mohd Nasir, Ganapathy Senthil Murugan, & Michalis N Zervas, 2016a). For each stage of the concentration level, the insertion loss is approximately from -

43dBm to -47 dBm, which can be control by adjusting the space between MBR and tapered microfiber (Cai, Painter, & Vahala, 2000). The MBR quality factor can be define as $\Delta\lambda/\lambda$ (λ is the resonant wavelength), and also be found that the quality factor for each of concentrations is not similar, which compared to past work (Mohd Narizee Mohd Nasir et al., 2016b). Because of the microfiber non-adiabaticity, the thought was giving significant insertion loss of microbottle.



Figure 3.3 Micro-bottle resonator transmission spectral coupled on 5 μm waist diameter of tapered fiber for different concentration levels.



Figure 3.4 Experimental setup of MBR with formaldehyde and a tapered microfiber of 5 μm waist diameter.

The experimental setup of the MBR has shown in figure 3.4 for formaldehyde liquid sensing performance investigation. The microfiber and the MBR are placed inside control chamber, which every level of formaldehyde concentration been tested. The tuneable laser source is connected to one end of the microfiber and optical power meter connected to another end for transmitted power measurement. The formaldehyde liquid was then varied from 0% to 5%. Initially, the transmitted power of the MBR on each level of liquid concentrations are recorded with the wavelength at 1551 nm. To investigate the repeatability of liquid sensing and to reduce random error, the experiment was repeated three times. The liquid was then replaced by another concentration which the performance between MBR and microfiber as the liquid sensing for the sensor on different concentration level was investigated as a comparison. Finally, the transmission on different concentration level is recorded for a 60 seconds period of times for sensor stability investigation.

3.5 Results and Discussion

The average transmission of the bare microfiber and the MBR at different concentration levels presented in figure 3.5. Generally, the graph showed decreases trend with increasing levels of formaldehyde concentration percentage for both bare microfiber and MBR. However, the linearity, standard deviation, p-value and even the sensitivity value of the MBR is notably better than the bare microfiber as compared, which recorded in table 3.2. The sensitivity of the MBR which manage to have 4.397 dB/%, is four times higher compared to 0.517 d/% recorded by the bare microfiber. Indeed, the linearity being almost 90% grater received by MBR than the bare microfiber. As formaldehyde liquid sensing, the MBR indicated fine result than bare microfiber. The losses increased during the transmission at high concentration levels happened due to reduction on surface absorption. The light experienced multiple circulated in the MBR which magnified high losses and losing more power for every circulation, thus increasing the sensitivity of the

sensor sensing (Arregui, Liu, Matias, & Claus, 1999; Batumalay, Harun, Irawati, Ahmad, & Arof, 2015).



Figure 3.5 Transmitted power value with different concentration levels of formaldehyde for MBR and bare microfiber.

Table 3.2 Performance analysis of MBR and bare microfiber in formaldehyde
sensing activity.

Parameters	Bare Fiber	With MBR
Linearity (%)	50.45%	98.23%
Sensitivity (dB/%)	0.517	4.397
Standard deviation (dBm)	2.644	8.059
P-value	1.48 x 10 ⁻⁵	5.30 x 10 ⁻⁵
Linear Range (%)	0 - 5	0 - 5

The experiment repeated three times for the MBR and bare microfiber to investigate repeatability of the setup and it had applied for each concentration levels (Isa, Irawati, Rahman, Yusoff, & Harun, 2018). Figure 3.6 showed repeatability results for MBR and bare microfiber, which is more than 3.0 dB/% for MBR and less than 0.8 dB/% for the bare microfiber, severally. Even though repetition has been made up to three times, the

MBR show well-balanced as liquid concentration sensing compared to the bare microfiber.



Figure 3.6 Transmitted power value of (a) MBR and (b) bare microfiber for repeatability performance of varies with liquid concentration level.

As shown in figure 3.7, the stability of MBR and bare microfiber as liquid concentration sensing recorded within 60-second duration. Fortunately, both the MBR and bare microfiber marked stable performance during this time interval. The transmission variation is lower than 5% which happened in MBR setup and for the bare microfiber, is really noticeable on every level of concentration.



Figure 3.7 The performance of (a) MBR and (b) bare microfiber varies with time for stability results.

3.6 Summary

This chapter discussed the performance of bare microfiber and MBR as formaldehyde liquid sensing. A technique called "soften-and-compress" applied on silica fiber to create

bounce structure with the diameter of D_b = 190 µm, stem diameter of D_s = 125 µm and bottle length of L_b = 182 µm. The MBR is then excited via tapered microfiber with wrist diameter of 5µm non-adiabetic by using TLS and been characterized by managed the TLS wavelength with step interval of 0.001 nm on wavelength range from 1551.0 nm until 1551.7 nm. The quality factor then received at five different values of concentrations, were initially recorded to have >10⁵ for every concentration. The performance of the MBR and the bare microfiber then investigated by comparing both ability to become a liquid sensor. Four listed parameters used for evaluation purpose which are linearity, sensitivity, standard deviation and P-value were calculated. Moreover, the MBR was found to be superior to the bare microfiber for each parameter. In addition, the p-value which is >10⁻⁵ for the MBR and the bare microfiber fairly undergoing 60 second procedure for stability testing. In conclusion, we can use these effectiveness of MBR towards the formaldehyde liquid sensor.

CHAPTER 4: EFFECT OF TAPERING DIAMETERS WITH MICROBOTTLE RESONATOR FOR FORMALDEHYDE (CH₂O) LIQUID SENSING

4.1 Introduction

Optical microresonator (OMR) has captured recent interest for past years. By supporting Whispering gallery mode (WGMs), have created much potential toward application in micro-system of optical and miniaturization (A. B. Matsko & Ilchenko, 2006; Vahala, 2003). Microtoroid, microsphere and microdisc representing several geometries of microresonator which able to coupling the mode in lowest volume with high-quality factor (Q-factor) value. The process completed by having total internal reflection between the formation of WGMs and microcavity surrounding medium. These microresonators are been considered as 2-D resonator while confining the mode in equatorial planes and allowed spectral properties defined by its diameters.

Investigation of optical MRs supporting WGMs has likewise been reached out to incorporate cylindrical shaped structures, for example, optical filaments, or OMRs framed on strands for their particular way in confining light and in addition, for easy handling and useful applications (Ilchenko et al., 2001; Misha Sumetsky, Dulashko, & Windeler, 2010). Micro-bottle resonator (MBR) recently increased attention among another type of fabricated optical MRs, which is due to ability on WGMs supporting manner. MBR also capable supporting 3-D light confinement of WGM through the combination of WG-bouncing ball and WG-ring principle (M Sumetsky, 2004).

In this chapter, the experiment conducted on formaldehyde (CH₂O) liquid sensing using MBR coupled with different tapering diameter. The two diameter of bare tapered microfibers used for the setup which are 8 μ m and 10 μ m. The MRB formed by using a procedure which called "soften-compress" which create bottle structure from standard SMF 28 fiber. The level of the formaldehyde liquid used for this work between 0% - 5% which prepared by mixing the formaldehyde liquid with distilled water. The MBR was exposed to these liquids for sensing purpose.

4.2 Experimental Setup

As same as the previous experiment, the fabrication method of MBR has done through "soften-and-compress" process by a splicing machine (Furukawa Electric Fitel S178A) on an SMF-28 optical silica fiber which creates a bulge area at the center of the fiber by arcing with high temperature (Zervas et al., 2011). Then the MBR sized defined physically by three specific parameters such as the bottle distance across D_b , the stem width D_s and the neck-to-neck length L_b , as shown in figure 4.1. Bottle diameter was set at 190 µm for this work. The fine tapering process created MBR structure will be apply on the bare microfiber with two different diameters, 8 µm and 10 µm, which allowed the bundle of modes bouncing on MBR surface and utilizing WGM (Lim et al., 2012).



Figure 4.1 SMF-28 structure changed to MBR after arc procedure with $L_b = 182 \ \mu m, D_s = 190 \ \mu m, D_b = 125 \ \mu m$

The wavelength range utilized from 1520 nm until 1620 nm for tuneable laser source (ANDO AQ4321D) which is used for MBR characterization on non-adiabatic bare microfiber with the different sizes which are 8 μ m and 10 μ m respectively. The interval

scale used is 0.001 nm for wavelength range between 1551.3 nm to 1551.6 nm for all concentration level, while the output collected in power value through optical power meter (THORLABS S145C).

Figure 4.2 (a) showed the sharp resonant depth of transmission spectral with 8 μ m bare microfiber used for every level of liquid concentrations (Mohd Narizee Mohd Nasir et al., 2016b). In each stage of concentration level, the insertion loss was approximately from -22 dBm until – 38 dBm, where the value was decreased while increasing concentrations level (Cai et al., 2000). The insertion loss was significantly not same for every concentration level, which was influenced by non-adiabatic microfiber and the concentration of the liquid.

In Figure 4.2 (b), the waist diameter of bare microfiber used for this experiment is 10 μ m, which manage to get sharp depth resonation of transmission modes for every concentration used, which is similar with Figure 4.2 (a) (Mohd Narizee Mohd Nasir et al., 2016b). However, the insertion loss was approximated from -6.2 dBm to -9.4 dBm, much higher than the previous size of bare microfiber (Cai et al., 2000). Same goes for the previous reference, the insertion loss decreased with the increasing liquid concentrations value. The size of bare microfiber which formed with non-adiabatic structure gave much influenced to the insertion loss.



Figure 4.2 The MBR transmission spectral coupled on 8 µm waist diameter of tapered fiber (a) and 10 µm waist diameter of tapered fiber (b) for different concentration levels.

Figure 4.3 showed the experiment setup for formaldehyde liquid concentration level sensing used different bare microfiber. The MBR is placed between bare microfiber and liquid surface, where the MBR at the bottom side was dipped into the liquid while the top of the MBR attached with bare microfiber. The idea is to allow transmission spectra resonated on the MBR surface and experienced WGM with formaldehyde molecule

adsorb along MBR surface. The optical power meter connected to end of setup for output data collections, while tuneable laser source on another end fiber, supplied the light source respectively. The liquid of formaldehyde was varied from 0% to 5%. The wavelength of 1551.3 nm is used for every liquid level as transmitted power. The experiment repeated by three cycles to minimize random error and record as repeatability test on all condition. For stability testing, the transmission of spectral is recorded for 60 second period on different concentrations. All the testing was conducted on two different bare microfibers for comparison purpose.



Figure 4.3 MBR with formaldehyde and a bare microfiber of 8 µm and 10 µm waist diameter for concentration liquid sensing.

4.3 **Results and Discussion**

The average of transmission 8 μ m (presented as A) and 10 μ m (presented as B) bare microfiber with the D_s = 190 μ m MBR for different concentration level showed in figure 4.4. The graph showed a decreased trend for both bare microfiber with increasing concentration level of liquid, with the 8 μ m size showed more critical slope than the 10 μ m size. As mentioned in table 4.1, the size of 8 μ m tapered microfiber showed better performance for all parameter tested such as linearity, sensitivity, standard deviation and also p-value. The MBR with 8 μ m bare microfiber manages to have 3.6251 dB/%, which is higher than the MBR with 10 μ m bare microfiber, which only gets 0.278 dB/%, three times lower. The linearity of the MBR with 8 μ m also manage to have over 95% while the other setup only received less than 60%. The MBR with 8 μ m bare microfiber able to indicate better result than the MBR with 10 μ m bare microfiber. However, the losses of the 10 μ m showed higher than 8 μ m bare microfiber. This is because of the tapering waist diameter used are different and this would lead to experienced more losses for every concentration tasted (Arregui et al., 1999; Khaliq, James, & Tatam, 2001; Zhao, Deng, & Wang, 2014).



Figure 4.4 The transmitted power value with 8 µm bare microfiber (A) and 10 µm bare microfiber (B) with different concentration levels of formaldehyde for MBR.

Table 4.1 Performance analysis between 8 μ m and 10 μ m bare microfiber γ	with
MBR for formaldehyde sensing.	

Parameters	8 µm Bare	10 µm Bare
	Microfiber	Microfiber
Linearity (%)	99.10%	33.18%
Sensitivity (dB/%)	3.6251	0.278
Standard deviation (dBm)	6.365	1.497
P-value	8.3 x 10 ⁻⁷	7.59 x 10 ⁻⁵
Linear Range (%)	0 - 5	0 - 5

The performance of the sensing is depending on the accuracy of data collected. For this, the experiment was repeated by three cycles for all condition respectively (Isa et al., 2018; Ohno, Naruse, Kihara, & Shimada, 2001). It also to reduce random error while probably happened during the experiment. The results showed in figure 4.5, where three cycles represented by the three-line graph for both 8 μ m and 10 μ m bare microfiber used with MBR. Here, figure 4.5 (a) represents 8 μ m bare microfiber showed fine decreased line than 10 μ m bare microfiber by the figure 4.5 (b). The fine line somehow influenced the analysis of bare microfiber in sensing performance and capability. The bare microfiber of 8 μ m with MBR showed balance as concentration sense even though it has repeating three times.



Figure 4.5 Transmitted power value of (a) 8 µm and (b) 10 µm bare microfiber with the MBR for repeatability performance of varies with liquid concentration level.

Figure 4.6 (a) and (b) showed the stability test for 8 μ m and 10 μ m bare microfiber with the MBR on liquid concentration sensing for 60-second duration. The MBR with 8 μ m bare microfiber showed less stable than the bare microfiber 10 μ m diameter. The diameter of bare microfiber influenced the stability of sensing performance. Here, with larger diameter used for liquid sensing, the MBR with 10 μ m bare microfiber promised stable reaction with the different concentration of liquid.



Figure 4.6 Transmitted power value of (a) 8 μ m and (b) 10 μ m bare microfiber with the MBR for stability performance of varies with 60 second time data collection.

4.4 Summary

The performance of two different diameters of microfiber with MBR utilizing as formaldehyde liquid sensor discussed in this part. A method is known as "soften-and-compress" has applied to a silica fiber which created a bulge area on the fiber called MBR where the stem diameter D_s is 125 µm, bottle diameter D_b is 190 µm and bottle length L_b is 182 µm. The MBR then excited through two tapered microfibers which diameter are 8 µm and 10 µm via tuneable laser source and also characterized by shifting the wavelength of TLS from 1551.30 *nm* to 1551.60 *nm* with the wavelength interval of 0.001 *nm*. The comparison between two different diameters of tapered fiber has reported based on the four parameters which are linearity, sensitivity, standard deviation and p-value. In addition, basis on the results it was found that 8 µm tapered microfiber with MBR has more efficiency than the waist diameter of 10 µm tapered microfiber through MBR. The p-value for each dimeter has depicted as > 10⁻⁵ which ensures that the research going on the right way and also the stability of both tapered microfiber diameters measured by 60 second in this experiment.

CHAPTER 5: CONCLUSION

At present, optical microresonators are contributing to the fundamental research through its distinguishable structures which followed the phenomena of whispering gallery modes. OMRs which tolerate whispering gallery modes are capable of generating exceptionally high Q-factors on account of minimum scattering losses and leakage, and low material absorption. Through tailoring the size or diameter, material composition, and shape of the resonator, OMRs has shown great perspective based on the low power, compact size, and high speed. OMRs are widely demonstrated that it not only contributing the fundamental research but also broadly uses for device applications such as microlasers, sensors with small-scale, filters etc.

Among all the OMRs, a new kind of OMR called "microbottle resonator (MBR) or bottle microresonator (BMR)" has been given much attention because of its distinct features rather than the other optical microresonators. Advantages of BMR over other resonators are fast tunability through the strain application, better control over the coupling by optical tapered microfiber and in the spectrum the probability of attaining a great number of equally-spaced modes. The MBR was fabricated by a procedure which referred as "soften-and -compress" method. Then it was characterized based on the three specific diameters such as bottle diameter, stem diameter, and neck-to-neck length.

Firstly, the experiment has been done to evaluate the performance of the bare fiber and the MBR through a 5 μ m tapered optical microfiber towards formaldehyde (CH₂O) liquid sensing whereas the bottle diameter D_b was set as 190 μ m. The BMR was excited by the tuneable laser source with the wavelength range between 1551.0 nm – 1551.7 nm where the interval was 0.001 nm. The performance measured in terms of four parameters for

instance p-value, sensitivity, linearity and standard deviation. Based on the performance, it was noticed that MBR along with tapered microfiber has much more competency than the bare microfiber. The quality factor was found as $>10^5$ for all the concentration levels of formalin.

In an additional experiment, we investigated the effect of the microbottle resonator (MBR) based on whispering gallery modes (WGM) with two different diameters (8 μ m and 10 μ m) of tapered microfiber and its experimentation for the formaldehyde liquid sensing. In this work, the MBR energized with both diameters tapered fiber by the TLS range of 1551.30 nm to 1551.60 nm with the same interval as the previous experiment. In terms of performance, we have seen that the 8 μ m tapered microfiber with MBR has more efficiency than the waist diameter of 10 μ m tapered microfiber through MBR.

As a future work, this work could be tested for formaldehyde liquid sensor although there are some other challenges to produce a sensor.

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