MICROFIBER LOOP RESONATOR FOR RELATIVE HUMIDITY AND FORMALDEHYDE SENSING

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FACULTY OF ENGINEERING
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MICROFIBER LOOP RESONATOR FOR RELATIVE HUMIDITY AND FORMALDEHYDE SENSING

ABSTRACT

Recently, optical micro-resonators (OMRs) gained noticeable importance due to the bright potential of using them in vital applications, and the simplest and most practical type of micro-fiber resonators are Micro-fiber Loop Resonators (MLRs) because of their simple fabrication process and high sensitivity. In this research, the sensitivity of the optical Micro-fiber Loop Resonators (MLRs) to relative humidity (RH) and Formaldehyde liquid is tested. The MLR is fabricated from the silica optical fiber (SMF-28) by a process of tapering to reduce its waist diameter using a flame brushing technique. The tapered fiber is manually twisted to create the MLR and placed on a movable glass stage. Two experiments are done to test the sensitivity for various fiber diameters based MLRs. The first one, is the test of the MLRs RH sensitivity using fibers with different diameters (3 µm, 4 µm, 5 µm and 7 µm). Also, the Straight micro-Fiber (SmF) RH sensitivity is tested for comparison purposes. Initially, the whispering gallery modes (WGMs) effect was observed in the MLR. Transmission mode spectra are examined to determine the number of resonated wavelengths and quality factor (Q-factor). The observed Q-factor for the MLRs is equal to $> 10^5$ which is good for sensing applications. By changing the RH and observing the MLR resonating behavior, it was concluded that the significant resonating response clearly occurs when the RH value ranges from 35% to 85%. All MLRs show higher stability than SmF when tested for a period of 600 seconds. As the RH increases, the sensitivity also increases, and it is 14 times for MLR based on 7 µm diameter fibers more than the value for the SmF. The output power decreases form -55.04 dBm to -65 dBm when the RH increase from 35%

to 85%. The MLR linearity is found to be 98.80% and its resolution is 0.0344%. The second experiment, is the testing of the MLR Formaldehyde sensitivity using fiber with (7 µm diameter) and Straight micro-Fiber. The Formaldehyde concentration used in this experiment range from 0% - 5%. The effect of WGMs was observed in the MLR. By examining the Transmission mode spectra, the MLR Q-factor was determined to be $> 10^5$. The MLR shows higher stability for all the formaldehyde concentration levels than the SmF for the 600 seconds testing time. the output power decreases from -36.26 dBm to -19.04 dBm with the increase of the formaldehyde concentration level, A high linearity value of 98.22% was calculated and the good sensitivity level of 0.0259% was observed.

Keywords: Micro-fiber Loop Resonator, Relative humidity, Formaldehyde liquid, straight micro-Fiber, Whispering gallery modes.

MIKROFIBER RESONATOR GELUNG UNTUK APLIKASI PENGESANAN RELATIVE DAN CECAIR FORMALDEHYDE

ABSTRAK

Akhir-akhir ini, mikro-resonator optik (OMRs) mendapat perhatian yang ketara kerana potensi yang cerah dalam aplikasi tertentu. Salah satu daripada OMR yang terkenal adalah Resonator Mikro Gelung (MLRs). Ia telah menarik perhatian yang sangat besar kerana proses fabrikasi yg mudah dan saiz kecil. Dalam kajian ini, prestasi pengesanan MLR kepada kelembapan relatif (RH) dan cecair Formaldehid telah dijalankan. MLR dibuat daripada gentian optik silika (SMF-28) dengan mengurangkan diameter pinggangnya menggunakan teknik menyikat api. Fiber mikro tersebut dipintal secara manual untuk membentuk gelung sentuhan diri. Eksperimen dijalankan dengan diameter berbeza (3 μm, 4 μm, 5 μm dan 7 μm). Fiber Mikro Lurus (SmF) diuji untuk tujuan perbandingan juga. Pada mulanya, kesan galeri berbisik (WGM) telah dilihat di MLR. Spektrum mod penghantaran diperiksa untuk menentukan bilangan panjang gelombang bergelombang dan faktor kualiti (Q-factor). Q-factor diperhatikan untuk MLRs bersamaan dengan> 10^5 yang baik untuk mengesan aplikasi. Dengan menukar RH dan memerhati kelakuan yang bergema MLR, disimpulkan bahawa tindak balas resonasi yang signifikan jelas berlaku apabila nilai RH berkisar antara 35% hingga 85%. Semua MLR menunjukkan kestabilan yang lebih tinggi daripada SmF apabila diuji selama tempoh 600 saat. Apabila RH meningkat, sensitiviti juga meningkat, dan ia adalah 14 kali untuk MLR berdasarkan gentian diameter 7 μm lebih daripada nilai untuk SmF. Kuasa output berkurangan bentuk -55.04 dBm hingga -65 dBm apabila peningkatan

RH dari 35% hingga 85%. Kadar lineariti MLR didapati 98.80% dan resolusinya adalah 0.0344%. Untuk aplikasi kedua, prestasi penginderaan Formaldehid MLR dilakukan dengan (diameter 7 μm) dan SmF. Kepekatan formaldehida yang digunakan dalam eksperimen ini berkisar dari 0% - 5%. Kesan WGM diperhatikan dalam MLR. Dengan memeriksa transmisi mode spektrum, faktor Q-MLR bertekad menjadi> 10^5. MLR menunjukkan kestabilan yang lebih tinggi untuk semua tahap kepekatan formaldehid daripada SmF untuk masa ujian 600 saat. kuasa output menurun dari -36.26 dBm kepada -19.04 dBm dengan peningkatan tahap kepekatan formaldehid. Nilai lineariti tinggi sebanyak 98.22% dikira dan tahap kepekaan yang baik 0.0259% telah diperhatikan.

Kata kunci: Resonator Loop serat mikro, Kelembapan relatif, cecair Formaldehid, lurus mikro-Fiber, mod galeri berbisik.

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LIST OF SYMBOLS AND ABBREVIATIONS

ASE	:	Amplified Spontaneous Emission
	:	Formaldehyde
DL	:	Detection Limit
DR	:	Dynamic Range
EDFA	:	erbium-doped fiber amplifier
LED	:	Light Emitting Diode
MEMS	:	microelectromechanical system
MF	:	Micro-fiber
MKR	:	Micro-fiber Knot Resonator
MLR	:	Micro-fiber Loop Resonator
MMR	:	Micro-fiber Multicoil Resonator
OMR	:	optical micro-resonator
Q-factor	:	Quality factor
R	:	Resolution
RH	:	Relative Humidity
RH	:	Relative humidity
S	:	Sensitivity
s	:	second
SMF	:	Single-mode Fiber
SmF	:	Straight micro-fiber
WGM	:	Whispering Gallery Mode
T	:	Response Time

CHAPTER 1: INTRODUCTION

1.1 Background

During the past 5 decades, the optical fiber sensing become preferable and powerful in the applications of fiber optics and sensing technologies (Zigang Duan & Wei Shi, 2010). Lately, with the fast progress in the micro and nanotechnologies and the heavy demand on the optical fiber sensors, there is a noticeable increase of fiber optics use because of their characteristics like high performance, versatility. The spatial miniaturization versatility made it one of the favorable choices of the fiber optic sensors. The size reducing is obviously an essential step in the design of sensor structure to give the sensor higher sensitivity, faster response, lower power consumption and higher spatial resolution. with all of these features the optical microfiber may be one of the superior candidates for these purposes (Guo, 2014; L. S. Tong, M., 2009; X. T. Wu, L, 2013).

Usually the micro-fiber resonators come in different shapes like loop, a knot, or a coil, their optical quality is very good and they are used in promising applications like modern filters and lasers (Zhe Chen, 2006). The use of fiber optic sensors is increasing in various technological fields. Due to the applications requirements and requests, high interest went towards the microfiber. Several types of fiber-optic sensors design presented and illustrated (Allwood, Wild, & Hinckley, 2017; L. Tong, 2018). The sensors based on the micro fibers are considered as simple devices compared to others, because they do not require complex and expensive fabrication procedures. The optical micro-ring (MLR) resonator is one of the types of optical resonators that have attracted high attention according to their simple structure and small size. The MLR is commonly used in many communication applications and optical devices (T. K. Yadav, M. A.

Mustapa, & Bakar, 2014; Zigang Duan & Wei Shi, 2010). The planar waveguide of micro-ring resonators is well developed, but they suffer from larger losses in connections with fibers and more expensive. Recently, new opportunities are opened up from the researches on the microfiber losses in the micro-photonic devices like resonators (Zhe Chen, 2011), couplers (Limin Tong, Lili Hu, & Zhang, 2006) , and sensors (Yuhang Li & Tong, 2008).

The optical micro sensors play a role in the modern society technology, their flexibility is mainly based on their speed and very low cost. Also, they are very wealthy in supporting optical technologies like optical fibers, photodiodes, and light sources. One of the properties of light is that confining it in a dielectric microstructure to interfere with itself, small range of frequencies can pass and resized inside the cavity without noticeable losses. The next generation of optical sensors that have very high performance is realized based on the use of resonant small power loss like interfering micro-cavities. If the geometry of the micro-cavity or the properties of its material change by deforming or heating for example, resonant parameters change will be detected. The detection may be achieved through monitoring light intensity levels. Thus, the micro-cavity may be considered as the transducer for optical signals. Optical resonators can be used or a variety of detection tasks depending on the geometry and material of the use micro-cavity for confining the light; for example, molecular receptors coated microcavities can respond to certain biomolecules.

One category of resonant optical sensors those based on microcavities that supports whispering gallery modes (WGMs), has created high level of interests since it shows very high level of sensitivity. Always needed goals for environmental monitors, biomedical detectors, and biosensors in the biomedical applications is the ability to detect the interaction of single molecules as well as their types. This aim is now being achieved with the aid of optical micro-cavities using WGMs. WGM sensors having extreme sensitivity have led to a bio detection breakthrough in addition to the capability of sensitive probing of some physical phenomena such as the quantum ground state read out of micromechanical oscillator through the use of optomechanical coupling (Matthew R. Foreman, 2015).

1.2 Objectives and Scope Research

The main objective of these experiments is to examine the effect of the micro-ring resonator with the surrounding medium (relative humidity, formaldehyde) sensing through the tapered optical micro-fiber and investigate the resonator influence. The research includes the following objectives:

a) The fabrication of microfiber loop resonators (MLR) for various waist diameter.
b) Setting up the experiment
c) Implementing in evaluation procedure that determines the best diameter of the MLR for the sensing applications using the various types of chemical materials.

1.3 Problem Statement

Microfibers have gain huge importance in optical sensing applications. In the microfiber operation, a laser light is introduced from one end, guided through the fiber and collected at the other end. However simple straight microfiber faces a problem which is the limited interaction between the microfiber and the materials that surrounding it leading to the sensitivity towards physical parameters. In order to increase the sensitivity, tapering procedures need to be conducted to improve access to the evanescent field of the wave propagating in a silica fiber. The diameter of the microfiber usually affects the sensing performance. The evanescent field in the fiber is a potential fiber for monitoring the change in refractive index. The propagation characteristics could be further enhanced by modifying the microfiber into a different geometry such as loop, ring, bottle and knot. This geometry enables the generation of the resonating signal which improves the sensing capability of the microfiber. For microfiber loop resonators (MLR), the characterization and optimization procedure need to be carefully conducted to maximize the sensing performance. It is done by measuring the quality factor (Q-factor) and sensing performance of various microfiber waist diameters.

1.4 Report outline

The thesis is organized into five chapters. Chapter one presents an introduction about the micro-fiber and sensing. Chapter two explains the theoretical fundamentals of micro-resonators, their types and characteristics. Chapter three describes the fabrication process of MLRs and examining these MLRs (with different waist diameters) and bare fiber to optimum MLR waist parameter according to their performance. Chapter four describes the sensing performance of MLR and bare fiber in the presence of the surrounding formaldehyde liquid. Finally, chapter five provides the main conclusions obtained from the presented work.

CHAPTER 2: LITERATURE REVIEW

2.1 Introduction

Earlier in 1960's the invention of the laser has been initiated and it becomes indispensable part for most of the present photonic applications. As the days go on, many inventors have developed the laser in order to be applicable for the high-end applications in a different majors, especially optical communications and sensor technologies. Several advantages offered by the using of optical fiber sensor over the conventional sensor, high sensitivity, small in size, lightweight, highly accurate, prevent electromagnetic interferences, geometric flexibility, safe to use it in hazardous environments, and the optical fiber compatibility to communications. The fiber optics sensing system technology is ideal for monitoring the structural health of aircraft (load, strain, temperature, and), Medical and Surgical Maneuvers (Biosensing, vascular procedures and detection, Placement and monitoring movement of tiny catheters) as shown in figure 2.1, buildings, and dams; improving the efficiency of turbines and industrial equipment; detecting instabilities within tunnels and power plants, and much more.

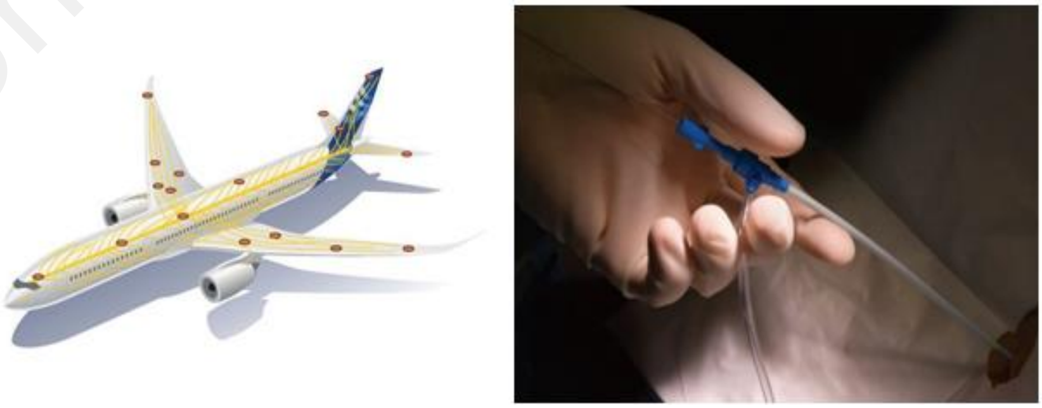


Figure 2.1: Some applications of optical fiber sensors

2.2 Optical Fiber

The simple normal optical fiber contains three concentric elements. The cylindrical core is made of silica glass coated by the cladding which is also made of silica glass but with a lower refractive index than in the core. Internal reflection occurs at the cladding-core boundary along the fiber length for most of the transmitted power. The light is confined through the optical fiber and does not run off the its boundary. The outer layer of the optical fiber is the buffer (jacket), which works as a mechanical support part that protects each of the core and cladding from environmental hazard and risk of damage. The buffer generally comprises one or more coats made of plastic material and sometimes covered by metallic sheaths to further strengthen the cable. The layers of the fiber are illustrated shown in figure 2.2

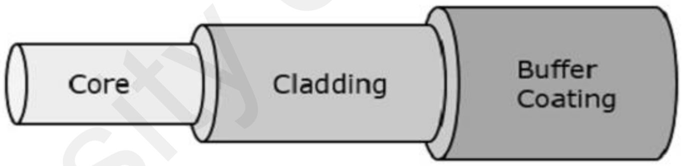


Figure 2.2: Optical Fiber Layers

Generally, there are two kinds of optical fibers according to the transmission characteristics: single mode fibers and multimode fibers. The difference between the transmission of light is explained in figure 2.3 (Hoss & Lacy, 1993).

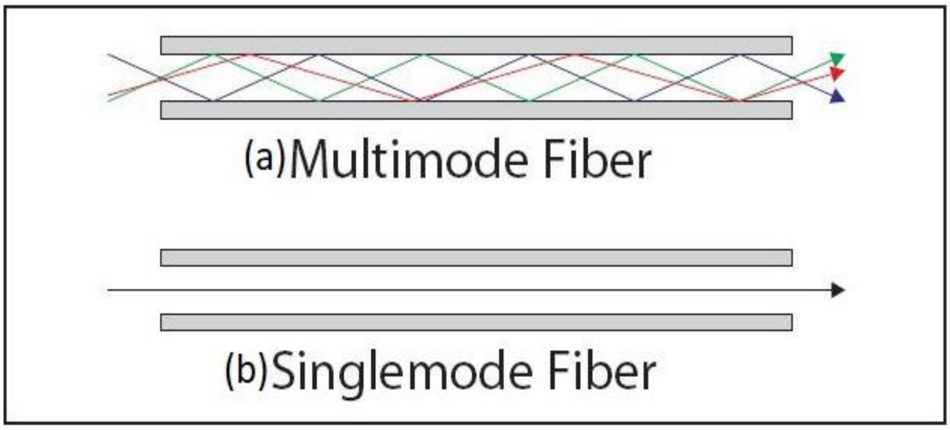


Figure 2.3: (a) Multimode and (b) single mode

The single mode fiber has a smaller core than the multimode one. the multimode allows hundreds of light modes to propagate through it. each mode represents a light beam that inters the fiber in a certain angle. While in a single mode fiber only one high directivity light beam passes through the fiber. The larger core of multimode fibers reduces the cost by using cheaper optical transmitters like light emitting diode (LED) and connectors. Single mode fibers may be considered as in ideal optical transmission medium for plenty of applications since it has huge information carrying capacity with low essential loss (Jeunhomme, 1983).

A single mode fiber is an interesting transmitting medium because it has broadband and low loss transmission characteristics (Kawana, Miyashita, Nakahara, Kawachi, & Hosaka, 1977). Single mode fibers type is having many advantages over multi-mode type in long distance telecommunications field, laser power delivery and sensor applications because of the carried signal light by the fiber cable fact that its travels in one mode only that leads to avoid intermodal dispersion problem that is encountered in multimode fibers. Also, the light intensity across a single mode fiber at a given wavelength is guaranteed to follow a single smooth, known and unchanging distribution. This is regardless of how light is launched into the fiber or of any disturbance of the fiber (Birks, Knight, & Russell, 2001).

2.3 Microfiber

The microfiber is a fiber with very small core diameter it ranges from hundred nanometers to a few micrometers. it is not accepted for a sensing purposes because of the cladding large diameter relative to mode field diameter. To get access to evanescent field due to removing the cladding part by the chemical itching or the mechanical polishing process. Even after the cladding removal, the optical fiber still suffering from residual face roughness and the limited control over the decladded fiber section diameter. The outgrow of the optical microfibers (MFs) create high effective and hopeful solutions to meet these challenges. a microfiber normally has prime diameter symmetry with sidewall smoothness and high-index variance between the microfiber material such as glass, polymer with the surroundings such as air, water. This type of the micro or nanoscale waveguide that guides light with low optical loss, prominent mechanical flexibilities, narrow optical confinement and huge fractional evanescent fields, make it a novel miniaturized platform for optical sensing with specific features such as fast response, high sensitivity and low power consumption.

Optical MFs small diameters can be manufactured using several techniques such as drawing fiber from bulk glass material (Neve et al., 2006), chemical etching (Zhang, Lou, & Tong, 2011), and the self-growth from silica nanomaterial (Naqshbandi, Khan, Rizwan, & Khan, 2012), or from tapered conventional optical fiber by scratching and heating. The latest method is the favored one because it has two main advantages: the low fabrication loss and the smooth tapered surface.

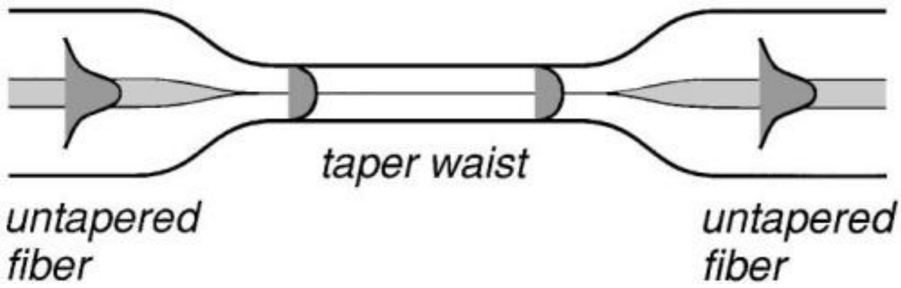


Figure 2.4: Tapered fiber

2.4 Flame Brushing Technique

Flame brushing technique is the commonly used method to fabricate fiber couplers or tapered fibers. It is usually chosen because it's easy to control the movement of the flame and the length of the fiber stretching, and it's in general consider as fast. The micro-fiber or tapered fiber in general dimensions can be fabricated with high accuracy and reproducibility. One of the most important things, is that the flame brushing technique enables the fabrication of a biconical tapered fiber having both connected to SMF. This biconical tapered fiber is usually used in the fabrication of low loss micro-fiber-based devices. The SMF coating is removed for several centimeters, then, it is placed horizontally and held by the two fiber holders. As illustrated in figure 2.5. The tapering is done by the moving the torch and heating the uncoated part of the fiber while it is being stretched. The torch movement provides a distributed heating along the uncoated fiber. This fabrication process results in a uniform and smooth tapered fiber along the heated region.

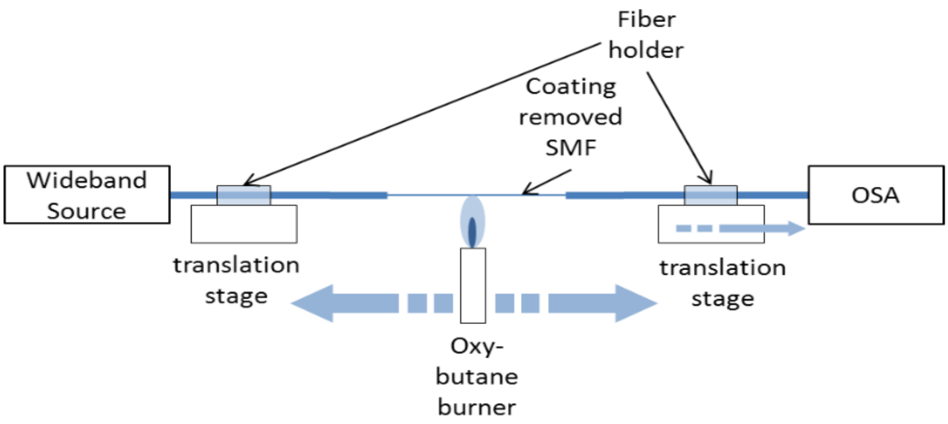


Figure 2.5: Flame brushing technique structure (K.-S. Lim et al., 2012)

2.5 Optical Micro-resonator

The optical micro-resonator (OMR) is the arrangement of mirrors that creates a cavity resonator in which incident light waves forms a standing wave. The light is confined in the cavity and is reflected many times by the mirrors generating the standing waves for certain resonance frequencies. There are different types of micro-resonators that can be differ from each other by the two mirrors focal length and the space between them. Normally the flat mirrors are not used because of the difficulty of aligning them to the exact position. Also, the resonator types are designed to meet some other criteria like the minimum beam waist or having the focal point being always outside the cavity.

Many research efforts have focused on the development of optical resonators based on microfibers, which are very useful for many applications in optical filters and sensors (G. Y. Chen, G. Brambilla, & T. P. Newson, 2013; Feng et al., 2011; Jiang, Chen, Vienne, & Tong, 2007; Y. Wu, Zeng, Rao, Hou, & Yang, 2009a). The optical resonators based on microfibers are very sensitive to any change in the surrounding environments due to the large evanescent field in the microfiber.

The optical micro-resonators are designed to have a high Q-factor. Since the light beam will reflect many times with limited attenuation. The beam frequency line width is very narrow actually as compared with the laser frequency.

2.5.1 OMR Circular Types

OMR circular types are made when the micro-fiber is shaped in a closed loop. the circular cavity based on the micro-fiber is formed because of the evanescent coupling occurring at the overlapping area. The Q-factor of the micro-fiber resonator depending on the coupling condition and the geometric parameters should varies between several hundreds to more than a million (J. Tong et al., 2009). According to their structure, circular micro resonators are divided in to the following kinds: Knot, multi-coil, and loop.

2.5.1.1 Micro-fiber Knot Resonator (MKR)

Jiang et al. suggested to tie the free-standing micro-fiber into a knot (Jiang et al., 2006). The maintaining of structure of the knot is done by the micro-fiber joint area friction under the high elastic bent knot tension, and high stability proved in water with Q-factor reaching 31,000 and a finesse value of 13. Many sensing applications are reported Based on the MKR structure (Ji, Liu, Tjin, Chow, & Lim, 2012; W. Tong et al., 2012). Wu et al. present a microelectromechanical system (MEMS) that is based on a combination of an optical accelerometer and a 386 μm diameter MKR that is fabricated from a silica fiber having a 1.1 μm diameter (Y. Wu, Zeng, Rao, Hou, & Yang, 2009b). The MKR Q-factor is equal to 8500 and was used for vibration measurement of the MEMS structure. In addition to the examples mentioned previously, micro-fiber optical sensors that are knot based are used for measurement of refractive index (Ji et al., 2012; Pal et al., 2011), humidity (Ji et al., 2012; Wang, Gu, Zhang, & Tong, 2011), magnetic field (Li & Ding, 2012), and temperature (K.-S. Lim et al., 2012;

XuZengYuWuChanglunHouJianBaiGuoguangYang, 2009; Yu Wu, 2012). Many opportunities are available via the use of MKRs for the optical sensing applications because of its high sensitivity, high robustness, fast response and its compact size in vibrating environment and/or liquid.

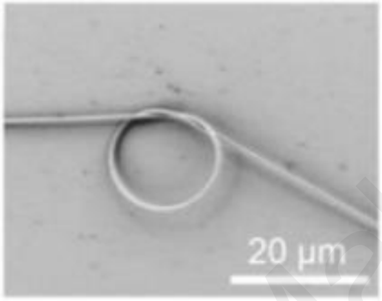


Figure 2.6: An optical MKR using a silica microfiber with 520 nm diameter (L. Tong et al., 2003b)

2.5.1.2 Micro-fiber Multicoil Resonator (MMR)

MMR was proposed for the first time by M. Sumetsky (M Sumetsky, Dulashko, & Hale, 2004). The fabrication of the three-dimensional MMR is usually done by microfiber wrapping around a rod with low-index for many turns as illustrated in figure 2.7.

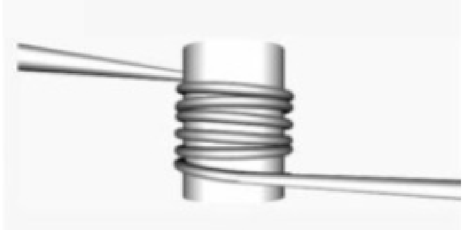


Figure 2.7: Optical MMR (Misha Sumetsky, Dulashko, & Fishteyn, 2007)

Xu et al. used a coated MMR, and he predicted a sensitivity to be around 700 nm/RIU (Xu, Horak, & Brambilla, 2007). Since then, the MMR optical-sensors are reported to be used in many optical sensing applications of temperature (G. Chen, G. Brambilla, & T. Newson, 2013), absorption (Lorenzi, Jung, & Brambilla, 2011), and acoustic waves (G. Y. Chen, Lee, Ismaeel, Brambilla, & Newson, 2012). The MMR can theoretically show a Q-factor around 1010, that means this may leads to a high optical sensitivity. However, because of the structure complexity and requirements accuracy, the experimental Q-factor is limited, leaving the space open for any further future improvements.

2.5.1.3 Micro-fiber Loop Resonator (MLR)

The micro-fiber loop resonator is the simplest resonator structure among all the micro-fiber structures. M. Sumetsky et al. reported an MLR with a loaded Q-factor around 120,000 and an intrinsic Q-factor around 630,000, and it was successfully used as a fast temperature sensor device that may be used in a direct contact with environments (M Sumetsky, Dulashko, Fini, Hale, & DiGiovanni, 2006). Due to its high Q-factor, its small and compact structure a very low temperature resolution can be obtained as low as (~0.1 mK) and small response time in the microseconds level.

Although the MLR fabrication is easy, the loop structure is preserved by electrostatic forces or van der Vaals at the joint zone, it is easy to be broken, especially in the liquid environment. To enhance the robustness of a microfiber loop for operating in liquid environment.

2.6 Sensors

Recently, the focusing on the optical resonators based on micro-fibers increased, because they are very useful for many applications especially in sensors and optical filters (Y. Chen, Xu, & Lu, 2011; Chung, 2013; Jiang et al., 2007; M Sumetsky, 2004; Yoon, Kim, Brambilla, & Han, 2012). The optical micro-resonators have a very high sensitivity when the surrounding environment change due to the large micro-fiber evanescent field (Xu & Brambilla, 2008; Xu et al., 2007). Several temperature sensors based on micro-fiber resonators have been successfully fabricated. a sensor based on an MKR has been reported for seawater temperature sensing (Yang, Wang, Wang, Wang, & Liao, 2014), with a maximum measured sensitivity of 22.81 pm/°C. Also, the MLR is used as a temperature sensor (M Sumetsky et al., 2006),and it is known for having a very fast response and high value of the Q-factor.

2.7 Sensors Parameters

Many parameters are usually used quantify the performance of the sensors, such as sensitivity, response time, repeatability, resolution, operating range, and detection limit.

2.7.1 Sensitivity

The sensitivity (S) represents the amount of change in the measuring variable due to the change in the monitored variable. For temperature sensing, the measuring variable maybe in nanometer if the measuring variable is a wavelength and the sensitivity will be in unit of nm/◦C since the monitored variable is temperature. Another possibility is the measuring variable being a power and this leads to a sensitivity being measured in dB/◦C. and a similar argument applies for the humidity sensing which may be measured either in nm/%RH or in dBm/%RH depending on wither the measuring variable is wavelength or power respectively.

2.7.2 Response time

The response time (τ) is the required time for a certain variable to rise to 90% of the its final value. The response time is usually measured from the starting time of the step change in the monitored variable to the time when the measuring variable follows 90% of the step change parameter up to monitor. It is usually measured in seconds(s).

2.7.3 Repeatability

Repeatability represents the detection parameter variation resulting from many measurements that are taken for the same sample and under the same experimental conditions. It is the agreement level indicator between two or more different carried out measurements for the same sample.

2.7.4 Resolution

Resolution (R) is the smallest detectable change in the parameter used for detection. The resolution is related to the precision with which the measurement is made and hence it is usually affected by the specifications of the detection system.

2.7.5 Dynamic range

Dynamic range (DR) is the difference between the maximum and minimum values of the examined parameter that the sensor is able to measure. Systems that have a large dynamic range may be used in a different type of environments.

2.7.6 Detection limit

Detection limit (DL) is the minimum change in the monitored parameter that the sensor is able to detect smaller change will not appear in the system output. It is related to sensitivity and resolution by:

$$DL = \frac{R}{S}$$

2.8 Formaldehyde

Formaldehyde is a basic compound that constitutes of hydrogen, oxygen and carbon. the common name of this substance is known as formalin. All life instances for example for instance microbes, plants, fish, and even people normally produce formaldehyde as a result of cell digestion. Formaldehyde has very additive and hostile to bacterial properties. However, the science based on formaldehyde properties is usually used to make a wide range of significant worthy items. Formaldehyde is very distinguished substance among most contemplated and newly discovered mixes in trade.

CHAPTER 3: MICRO-FIBER LOOP RESONATOR FOR HUMIDITY SENSING

3.1 Introduction

During the last 50 years, one of the prosperous technologies for the optics and sensing technologies is the optical fibers sensing (Schirmer, Hussein, Jekle, Hussein, & Becker, 2011). The fast progress and demands towards optical sensors, one of fiberoptic sensors trends currently is the spatial miniaturization. It is clear that the size reducing is an important step usually to bestow sensor together with faster response, more sensing, less power consuming and higher spatial resolution. the optical microfiber actually considered as one of best nominees for these applications (Nanowires, 2014; L. Tong & Sumetsky, 2011; X. T. Wu, L, 2013).

Fiber optics with nanotechnology combination, optical microfibers has been emerging as a novel platform for exploring fiber-optic technology on the micro or nanoscale (G Brambilla, 2010; Xu, Kou, Lu, & Hu, 2012). Fabrication of microfiber is done by taper drawing of polymer or glass materials. ordinarily the microfiber diameter ranges from hundred nanometers to a few micrometers. ideal microfiber must have highly uniform diameter and very smooth sidewall (L. Tong et al., 2003a), with a high index contrast between microfiber materials (e.g., polymer or glass) and its surroundings (e.g., water or air). this micro or nanoscale type waveguide leads light with little optical loss, and highly flexible for mechanical outstanding, also it has big fractional evanescent fields and tightly optical confinement (L. Tong, Lou, & Mazur, 2004; L. Tong & Sumetsky, 2011). The outstanding advantages like fast response, high sensitivity, and low power consumption, made it a miniaturized novel platform in optical sensing. when the microfiber assembled as a closed loop, the microfiber will form on the circular cavity forming at the overlapping area by the evanescent coupling. Depending on geometric parameters and coupling condition, the microfiber cavity Q-factor vary between several hundreds to more than one million (L. S. Tong, M., 2009). depending on their structures, so far there are many shapes of circular microfiber cavities: knot, loop and multi-coil.

Lately, the interests are directed towards the OMR due to its great possibilities in different applications like laser and sensing. Operated with continues spectral mode looping around the micro size of the resonator (Chiasera et al., 2010; Matsko, Savchenkov, & Maleki, 2005). A lot of OMR structures recently has been produced such as micro-ring, micro-disk, micropillar and microtoroids to achieve some predetermined research aims. the resonator activity according to the WGM resonating concept, makes the OMR having the least intersection losses which is necessary applicable for the sensing. Determining the resonating quality is based on finding the Q-factor which is calculated from the resonated wavelength. The OMR sensing capability mainly determined by the Q-factor and resonating depth.

This chapter describes the MLR optical characteristics during the transmitting mode related to the sensing applications such as Q-factor and the resonating wavelength. It also analysis the effect of using different MLR microfiber diameters in environments in which humidity ranges from 35% to 85%.

3.2 MLR Fabrication

The MLR cavity is the simplest cavity structure Among the microfiber cavities. The fabrication is done by tapering a single-mode fiber (SMF) to reduce fiber waist by heating and stretching technologies which is known as the flame brushing technique (Irawati, Rahman, Ahmad, & Harun, 2017).

The next step, the tapered fiber manually twisted to shape the MLR then it is placed on a movable stage (K.-S. Lim et al., 2012). After the fabrication process is done, the tapered fiber waist and the loop diameter can be verified by using a microscope with 20X magnification power. Figure 3.1 shows 5 µm diameter microfiber with its 300 µm diameter loop.

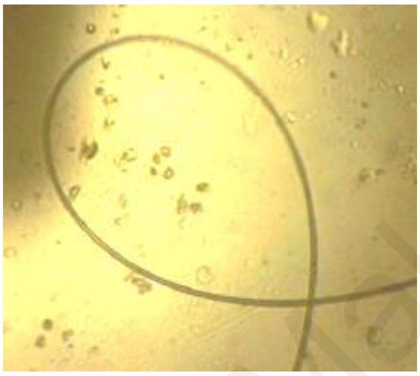


Figure 3.1: MLR Microscopic image of 5 µm diameter microfiber with its 300 µm diameter loop

3.3 Experimental Setup

The humidity sensing experimental setup is constructed by either bare microfiber or an MLR with different tapering diameters (3 µm, 4 µm, 5 µm and 7 µm), then connecting them as illustrated in the schematic depicted in figure 3.2. the MLR is placed inside the chamber. The Amplified Spontaneous Emission (ASE) with 1550nm wavelength from an erbium-doped fiber amplifier (EDFA) inject the light beam into one of the microfiber ends while the second one is connected to the Optical Spectrum analyzer (Anritsu: MS9710C) for measuring the output power and detecting the transmission mode. The probe of the Relative Humidity (RH) meter (Hygrometer RS 1365, Sensitivity: 1%) is placed in near the MLR for the purpose of monitoring the real value of the relative humidity of the MLR. The RH sensing is achieved by modifying the humidity from 35% to 85% and measuring output power in dBm unit. For every measurement, five readings are taken.

Figure 3.2: Experimental setup of MLR with Humidity Sensing

The stability is checked for five diameters MLRs (3 µm, 4 µm, 5 µm and 7 µm) at RH level equals to 45% with the resonating wavelength adjusted via the tunable laser source (ANDO AQ4321D) to the range (1550 nm to 1560 nm) at the input terminal. The wavelength incrementation is set to 0.001 nm. The transmitted power is collected from the output terminal through the optical power meter (THORLABS S145C). from the readings the insertion losses and transmission losses for every different MLR diameter were determined as shown in Figure 3.3.

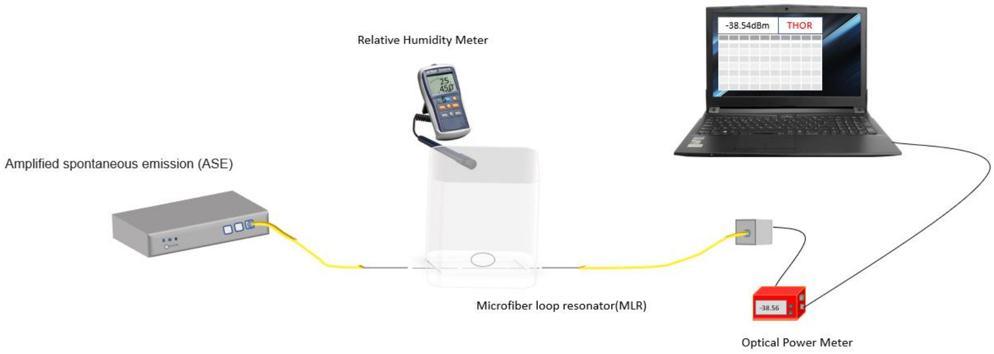


Figure 3.3: Experimental setup of MLR for taking WGM transmission mode

The number of resonated wavelengths is used to evaluate the sensor performance. The good resonator is usually recognized when a large number of resonated wavelengths is observed. this means that the sensor is highly sensitive.

3.4 Results and Discussion

The transmission mode of the MLRs is shown in Figure 3.4. The resonating wavelength with different depth observed for the MLR with different waist diameter. The insertion losses are -35dBm and transmission losses are -39dBm.

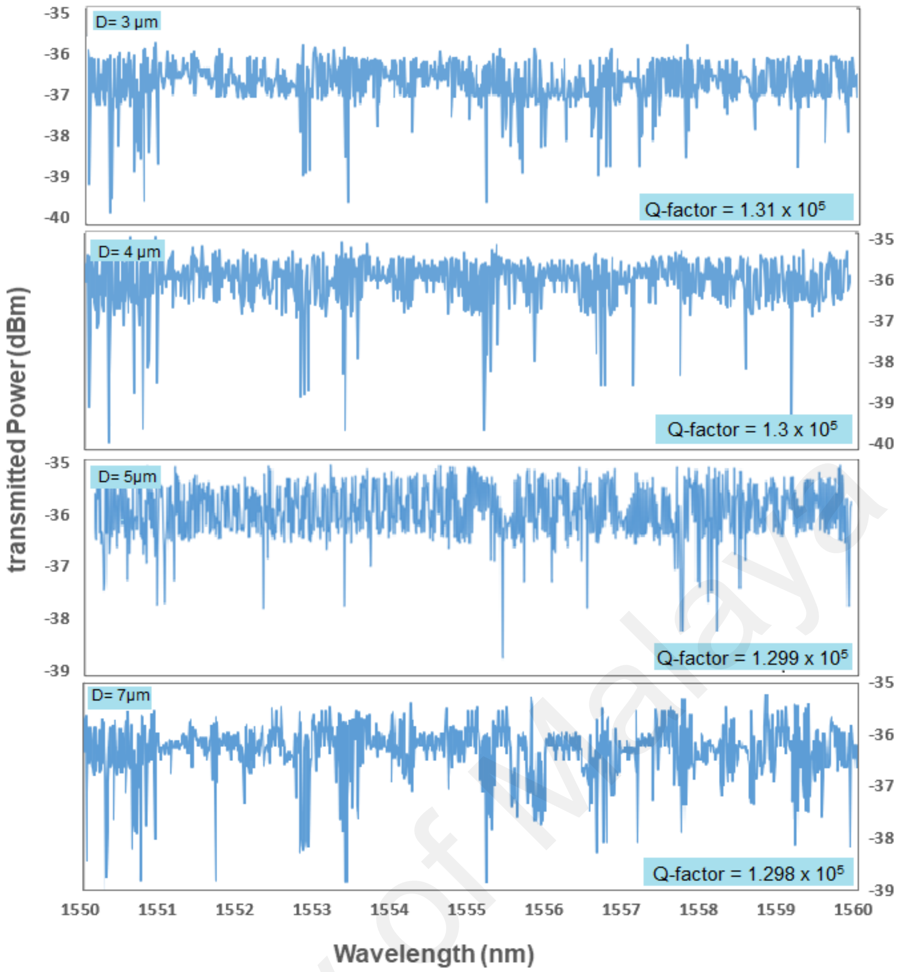


Figure 3.4: WGM transmission modes of different MLR waist diameter

The quality factor(Q-factor) of the MLR is equals to $\Delta\lambda/\lambda$ when λ is the resonant wavelength. The Q-factor for the smallest waist diameter (3 µm) is 1.31 x 10^5, which has the highest value and the Q-factor of the MLR with the largest waist diameter (3 µm) value equals to 1.298 x 10^5. The value of Q-factor depends on the micro-tapered fiber diameter, it increases when the used micro-tapered fiber diameter decreases (Matsko, Savchenkov, Strekalov, Ilchenko, & Maleki, 2005).

The real time response of the MLR and SmF is for the humidity range 35% to 85%. the recorded values are taken in a step of 5 % of the humidity as shown in Figure 3.5. depending on the obtained results it was realized that the output power decreases with the increase of the humidity the fiber is exposed to. The SmF values is show high linearity and sensitivity. However, the MLR linearity and sensitivity are much higher. The output power reduced value is caused by the reduction in transmission due to increased level of humidity of the MLR. The reduction of the transmission is caused by the added scattering losses made by water particles that absorb part of the power changing the refractive index of the micro fiber and resulting in increased sensitivity (Bariain, Matías, Arregui, & Lopez-Amo, 2000).

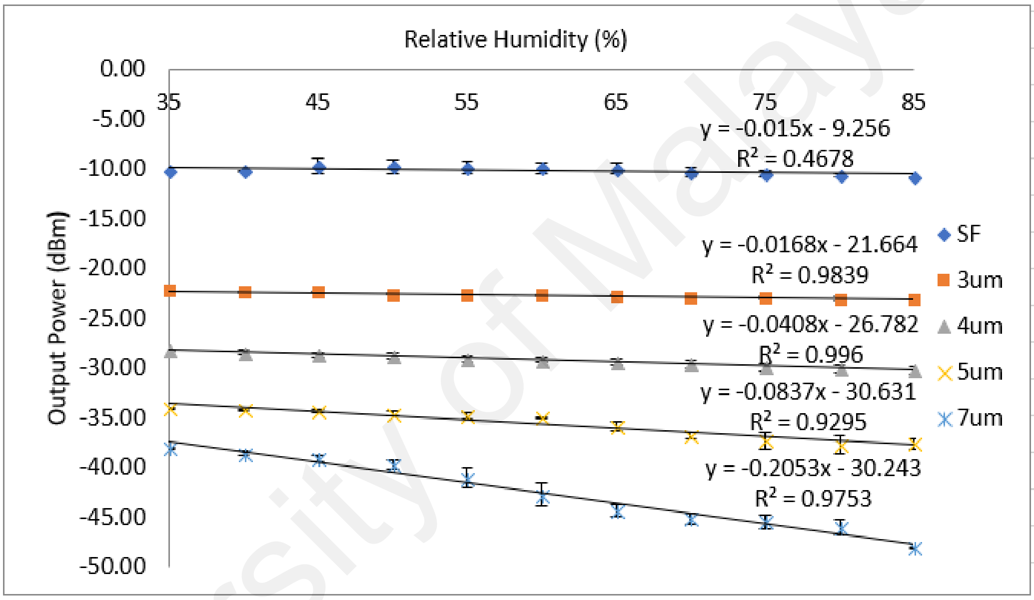
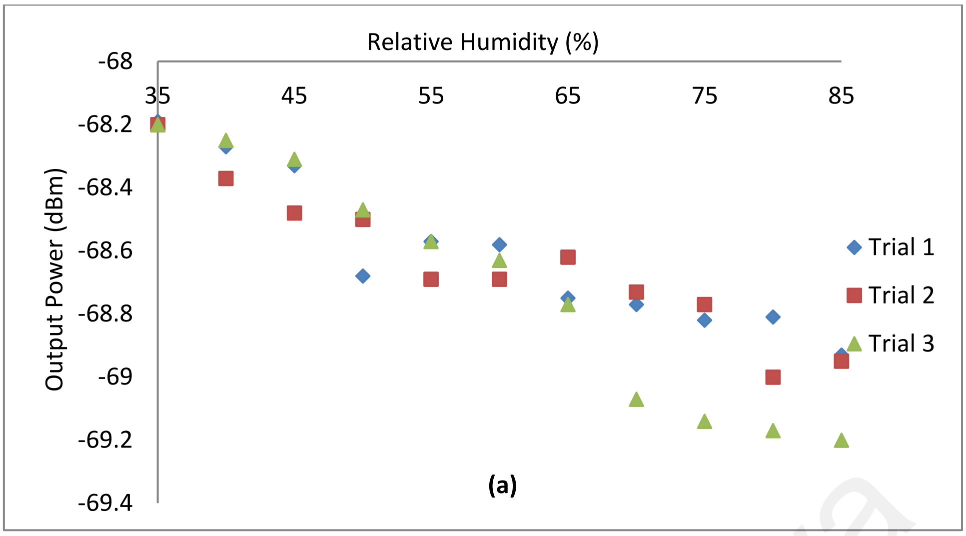
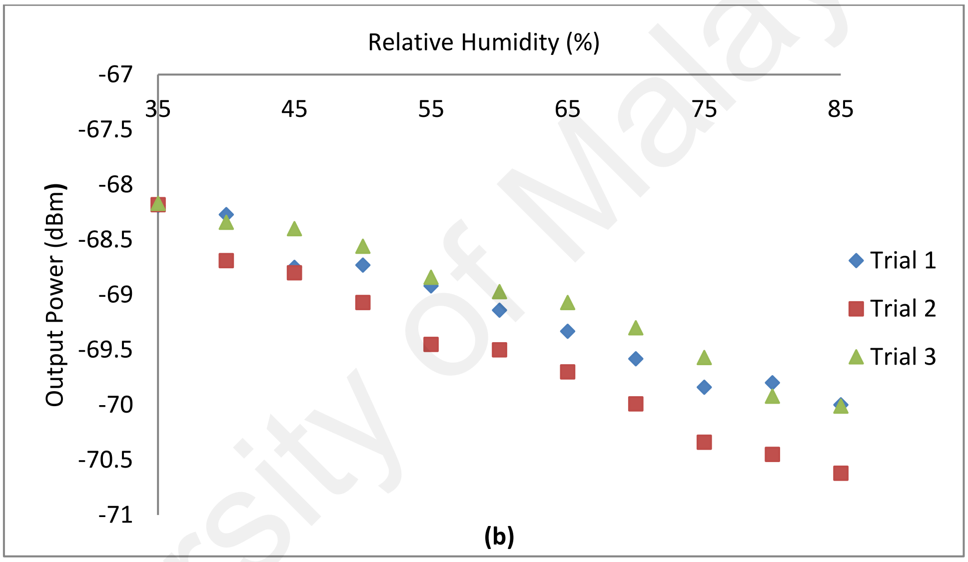
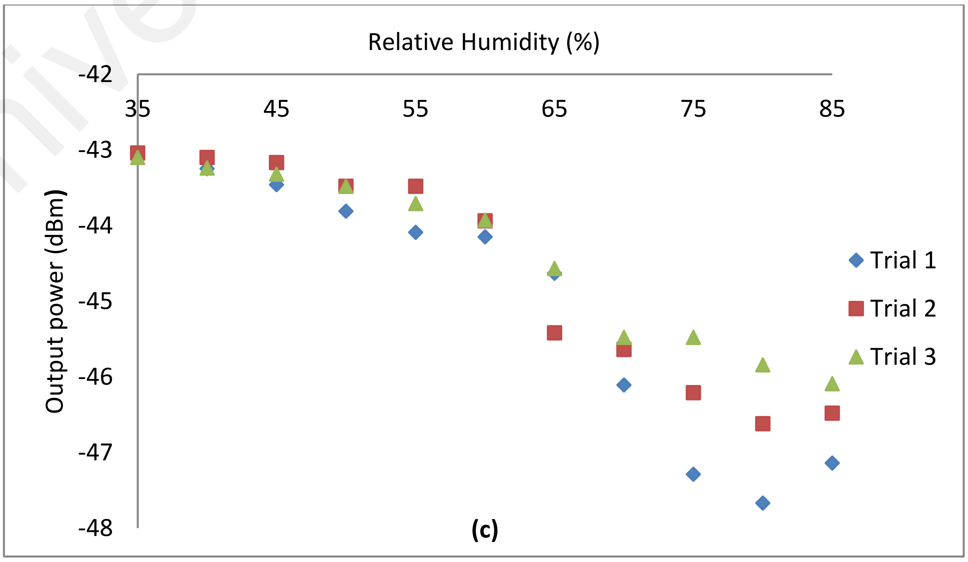


Figure 3.5: The MLR and SmF response towards the humidity sensing

Every RH experiment of the MLR is repeated three times for every fiber diameter based MLR. Based on these experiments the case of 3 µm showed more variance in the readings than for the MLR with a 7 µm fiber waist diameter of. All the recorded fiber outputs are illustrated in figure 3.6.


Relative Humidity (%)
Output Power (dBm)
-68
-68.2
-68.4
-68.6
-68.8
-69
-69.2
-69.4
35
45
55
65
75
85
Trial 1
Trial 2
Trial 3
(a)


Relative Humidity (%)
Output Power (dBm)
-67
-67.5
-68
-68.5
-69
-69.5
-70
-70.5
-71
35
45
55
65
75
85
Trial 1
Trial 2
Trial 3
(b)


Relative Humidity (%)
Output power (dBm)
-42
-43
-44
-45
-46
-47
-48
35
45
55
65
75
85
Trial 1
Trial 2
Trial 3
(c)

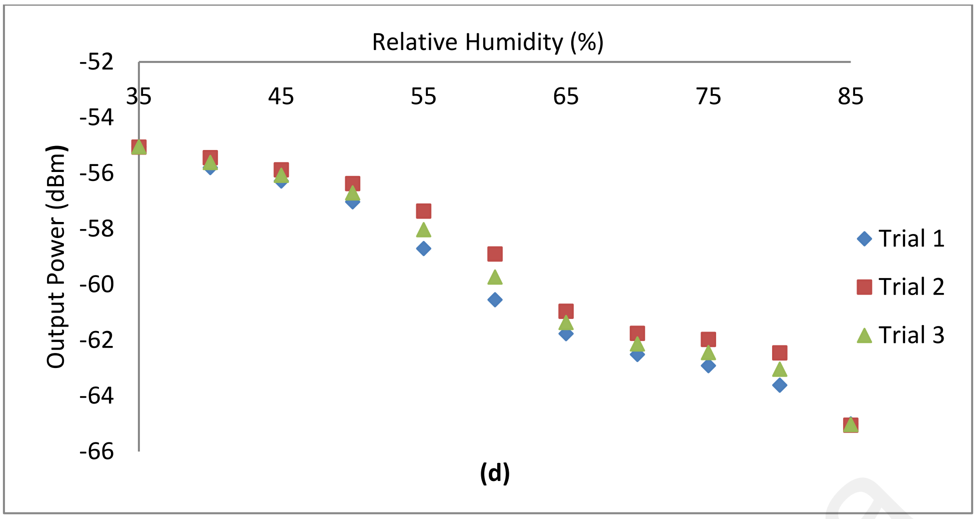


Figure 3.6: The MLR RH sensing repeatability for (a) 3 µm diameter (b) 4 µm diameter (c) 5 µm diameter (d) 7 µm diameter

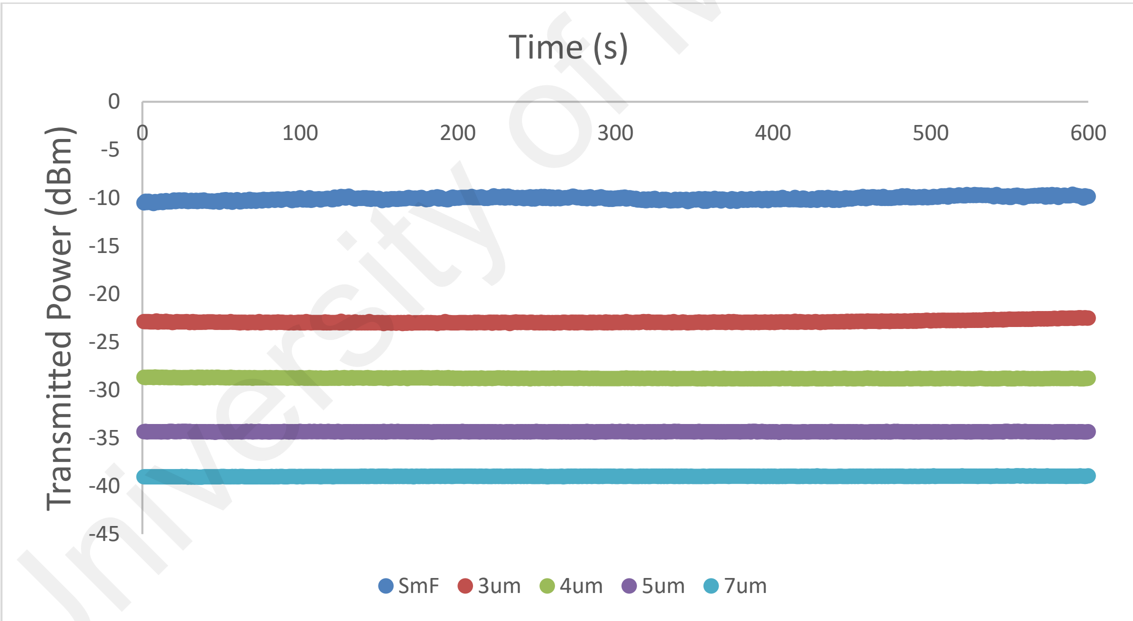


Figure 3.7: The MLR and SmF response towards the humidity sensing

As shown in Figure 3.5, the stability of the MLR and SmF as RH sensing recorded within 600 seconds (10 min). The MLR in different waist diameters shows better performance comparing with the SmF stability during the interval time. According to the MLR stabilities illustrated in the Figure the MLR with the waist diameter increases the stability decreases.

Table 3.1: Sensing performance of the humidity sensors

	SmF	3 µm	4 µm	5 µm	7 µm
Linearity (%)	68.40%	99.20%	99.80%	96.40%	98.80%
Sensitivity (dBm/%RH)	0.0150	0.0168	0.0408	0.0837	0.2053
Standard deviation(dBm)	0.0071	0.0058	0.0100	0.0306	0.0071
Resolution (%RH)	0.4714	0.3437	0.2451	0.3650	0.153
Linear Range (%RH)	45% - 85%	35% - 80%	35% - 80%	35% - 80%	35% - 80%

The performance of SmF and MLR is summarized in Table 1. It is worth mentioning that the humidity changes were significantly sensed by the MLR in different diameters with high level of linearity (96.40% for 5 µm diameter to 99.80% for 4 µm diameter). This linearity is higher than that in SmF which is only equals to 68.40%. And regarding to realize that the MLR has high sensitivity 0.2053 dBm/%RH for 7 µm diameter and decreases to 0.0168 dBm/%RH when the diameter waist is decreased to 3 µm diameter while the SmF is only 0.0150 dBm/%RH. The standard deviation of the output power of the MLR with diameters 5 µm and 4 µm is higher than that of SmF. However, for 7 µm diameter MLR the standard deviation of output power which is 0.0071 dBm will be the same as in the SmF case, and when smaller value of MLR like 3 µm diameter is used, the standard deviation which is 0.0058 dBm will be less than that in SmF case (It is always better to get small values for the output power standard deviation). the measurements show that the resolution of the humidity sensing is 3.1 times better in when using the MLRs as compared as using SmF.

3.5 Conclusions

In this chapter the humidity sensing using micro-fiber loop resonator with different fiber diameters and straight micro-fiber has been presented. The MLR proved to have higher performance than the SmF. The MLR showed better performance (around 14 times better) compared to SmF in humidity sensing. additional scattering losses due to absorption of water particles on the MLR changes the refractive index because of the interaction of MLR with water particles. This simple humidity sensor can offer wide range of applications such as in green house automatic control system (Park et al., 2011) and in extremely humid environments (Tan, Tay, Tjin, Chan, & Rahardjo, 2005).

CHAPTER 4: MICROFIBER LOOP RESONATOR FOR FORMALDEHYDE LIQUID SENSING

4.1 Introduction

Recently, researchers more attracted toward the tapered fiber technology specially focusing on the optical sensing branch due to the extensive outstanding performance and capabilities in many sensing applications (Udd & Spillman Jr, 2011). The fiber optics sensor is the favorite in the sensing applications due to its immunity to the electromagnetic interferences. Nowadays with the fast growth in micro and nanotechnologies, the fiber optics demands increases in the market. The tapered fibers with diameter range of micrometers (μm) are named microfibers while fibers with diameter less than 1 μm are usually named nanofiber (nm) (G Brambilla, 2010). Micro fiber characterized by their small diameters making them sensitive, responsive, have a high dynamic range, low attenuation loss, strong evanescent fields, and tight optical confinement (Gilberto Brambilla, Finazzi, & Richardson, 2004; Harun, Lim, Tio, Dimyati, & Ahmad, 2013; K. Lim et al., 2011; X. Wu & Tong, 2013). With these advantages made it become perfect for remote sensing applications as in humidity sensing (Rahman, Irawati, Abdullah, & Harun, 2015), refractive index sensing (Rahman et al., 2015), vapor sensing, chemical analysis, biomedicine, environmental engineering and in automotive industry.

In various technological field the fiber optics uses is increases. The micro fiber considered as the simplest sensors among other sensors, because they are not expensive and simply fabricated. In contrast, fiber Bragg grating sensors like require expensive phase-masks.

4.2 Fabrication of Formaldehyde

In this experiment formaldehyde is created using both the formalin volume and mineral water. Mineral water is known as distilled water which is made by boiling the water and condensing it back into liquid. Sometimes in industry, some chemical and biological laboratories use the deionized water and refer to it as distilled water. In this experiment a formalin with 37% purity is used (i.e it made of 37% formaldehyde with impurities) these impurities are usually aldehydes, ketones, methanol, and small amounts of formic acid, …etc. the following equation describes the constructed concentration levels:

$$V1 \times N1 = V2 \times N2$$

(Farrell Jr, 2009), Where:

V1 is the quantity of formalin

N1 is the pure formalin percentage which is 37%

V2 is the concentration total volume

N2 is the concentration level

In this work, 0% concentration of formalin is made of purely deionized water. A volume of formalin (2.7 ml) has been added to distilled water (97.3 ml) in order to make the required level of formaldehyde with 1% concentration (100 ml). To increase the concentration level (1% - 5%), more pure formalin quantity should be added with less volume of deionized water. The fabrication of different concentration levels shown in the following table 4.1.

Table 4.1: Formaldehyde concentration level materials quantities

Concentration level of formaldehyde (%), N2	Formalin Quantity (ml), V1	Volume of distilled water (ml)	Total Volume (ml), V2
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

4.3 Experimental Setup

The setup of part of the experiment that is used to evaluate the sensing of the MLR (the 7 µm diameter one) or the SmF when they are immersed in the Formaldehyde liquid as clarified in the following figure 4.1.

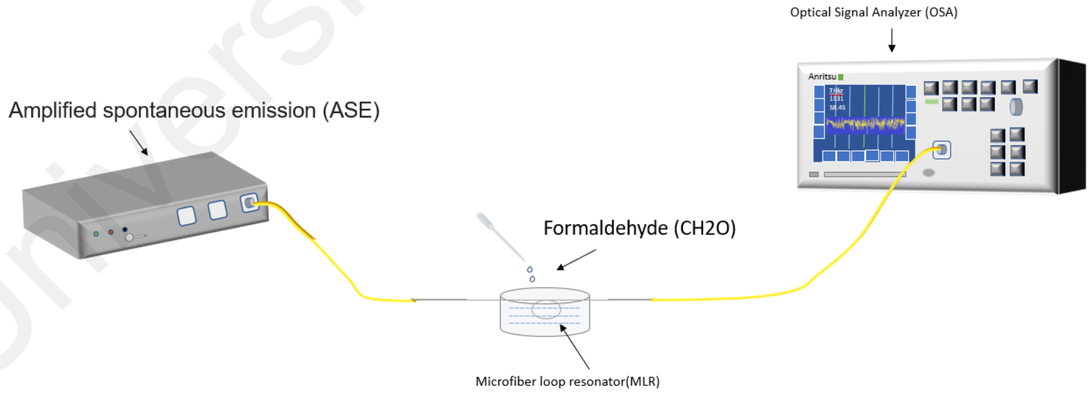


Figure 4.1: Experimental setup of MLR with formaldehyde Sensing

The MLR or the SmF is placed inside the experimental chamber with the formaldehyde liquid. The concentration levels are change from (0%-5%). The

Amplified Spontaneous Emission (ASE) with 1550nm wavelength from an erbium-doped fiber amplifier (EDFA) injects the light beam into one of the microfiber ends. The second end is connected to the Optical Spectrum analyzer (Anritsu: MS9710C) to measure the received power and detect the transmission mode.

4.4 Results and Discussion

The detected transmission modes of the MLR is shown in Figure 4.2. The resonating wavelength depths is plotted for different concentration level formaldehyde liquid.

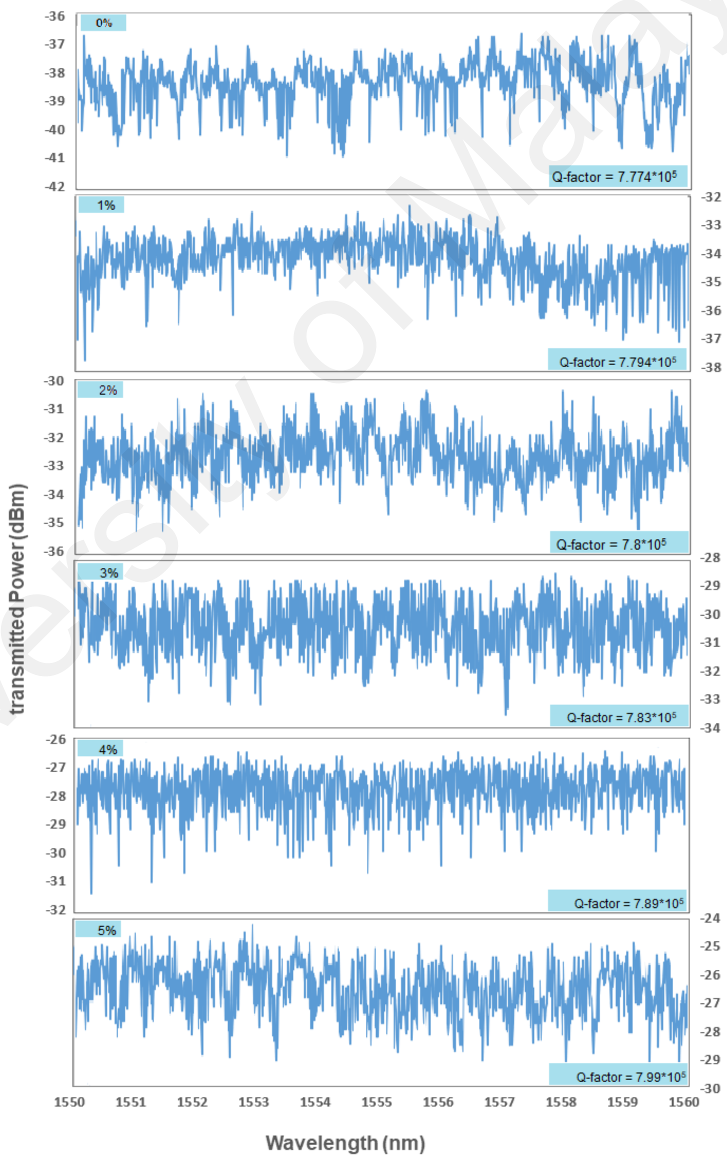


Figure 4.2: WGM transmission modes for different formaldehyde concentration levels

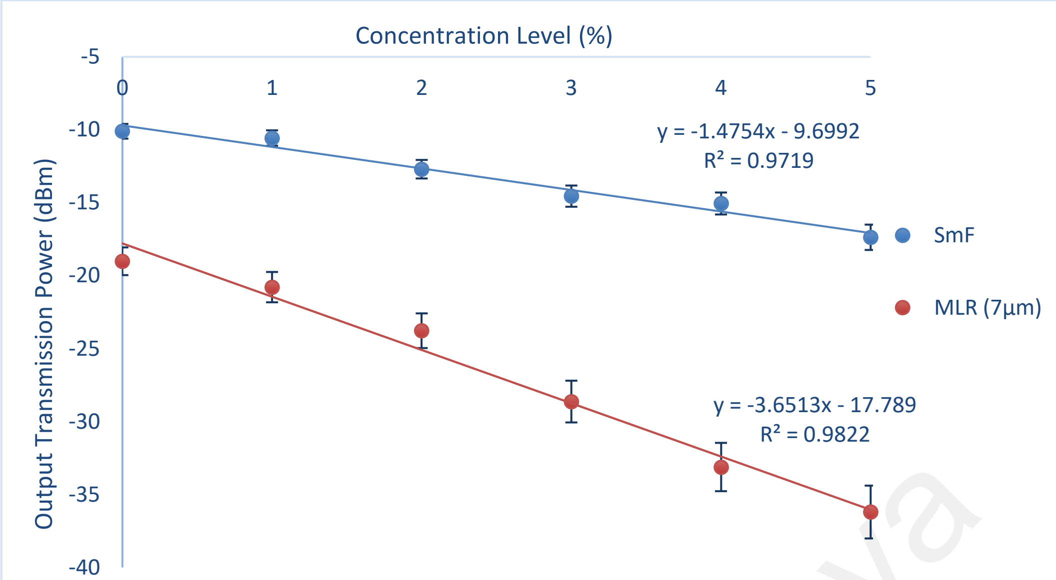
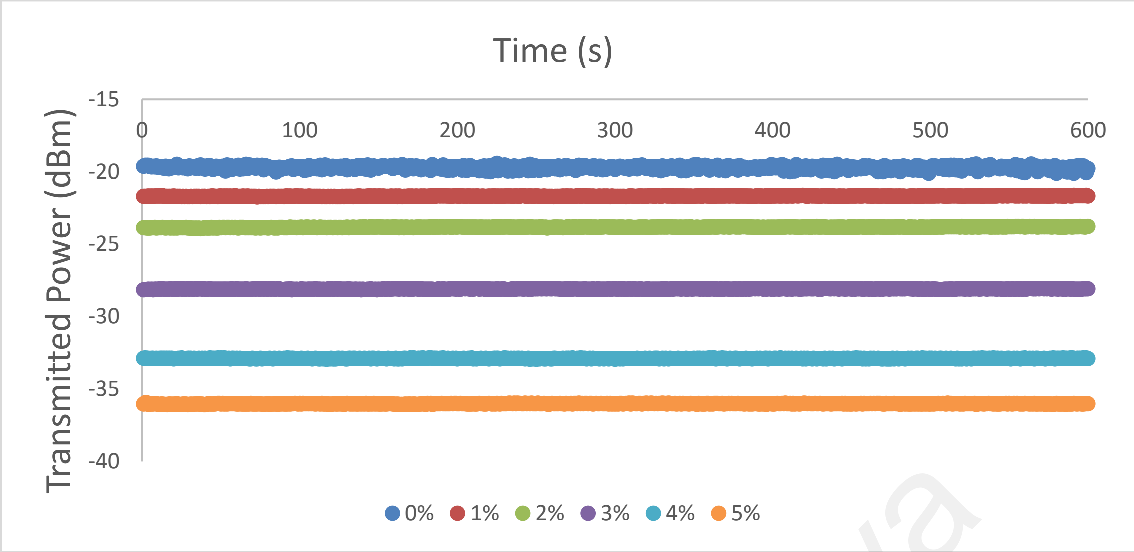


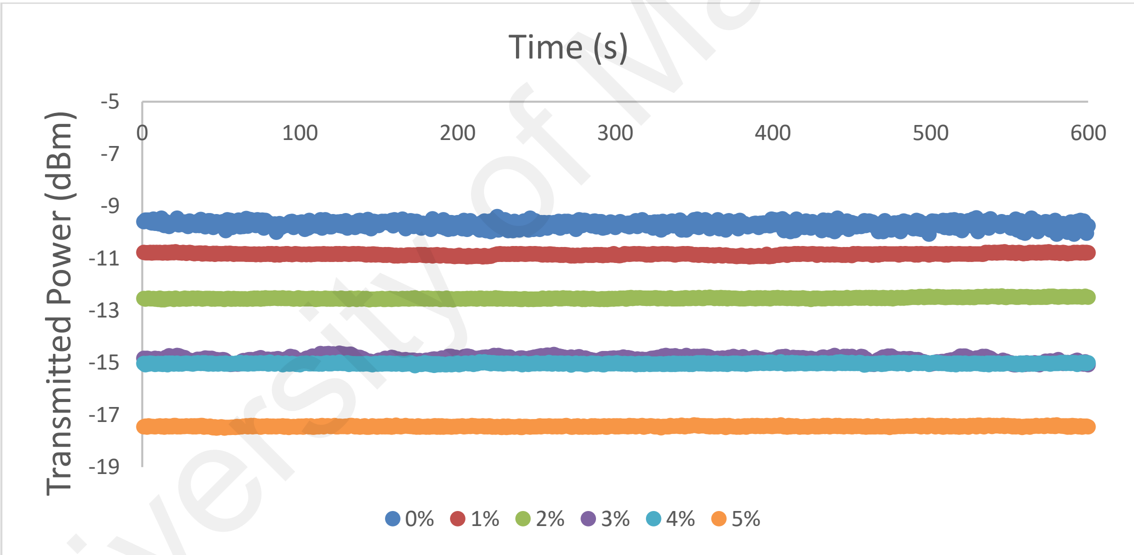
Figure 4.3: The MLR and SmF response towards the Formaldehyde sensing

As in figure 4.3 the graph shows trend decreasing with the increasing of formaldehyde concentration percentage level for both MLR and SmF. Depending on the obtained results it was realized that the SmF values is show high linearity and sensitivity. However, the MLR linearity and sensitivity are much higher.

The measured Q-factor for the concentration level 0% was 7.774 x 10^5, which is the lowest value. while a Q-factor of 7.99 x 10^5 was measured when the concentration level 5%, representing the greatest Q-factor value. The Q-factor increases with the increase of the when the increases of the formaldehyde concentration level.



(a)



(b)

Figure 4.4: The stability performance for (a) MLR (b) SmF

As illustrated in Figure 4.4, The stability is checked for the MLR (7 µm diameter) and the SmF with different formaldehyde concentration level using the recorded sensing values for 600 seconds (10 min) period of time. The MLR shows better stability than the SmF stability for all the formaldehyde concentration levels.

Table 4.2: Sensing parameters of the formaldehyde immersing experiment

	SmF	MLR (7 μm)
Linearity (%)	97.19%	98.22%
Sensitivity (dBm/RH%)	1.4754	3.6561
Standard deviation (dBm)	0.0408	0.0163
Resolution (%)	0.0276703	0.0045
Linear Range (%)	0% - 5%	0% - 5%

The performance of SmF and MLR is summarized in Table 4.2. The formaldehyde sensing by the MLR may have a high linearity 98.22%, which This is a bit higher than that sensed by SmF which is equals to 97.19%. And for the sensitivity analysis the MLR has high sensitivity equals to 3.6561 dBm/%RH for 7 μm diameter micro-fiber while the SmF sensitivity is only 1.4754 dBm/%RH. The standard deviation of the output power of the MLR with 7 μm diameter micro-fiber is 0.0163 dBm, which is better than the SmF standard deviation being 0.0408 dBm (It is always better to get small values for the output power standard deviation). the measurements show that using the MLR produce a better resolution as compared with the use of SmF.

4.5 Conclusions

The formaldehyde sensing using a micro-fiber loop resonator and a straight micro-fiber has been presented in this chapter. The MLR (7 μm diameter fiber) showed higher sensitivity value than the SmF by about 2.5 times. The quality factor for the MLR was calculated for every concentration level and it has a value $>10^5$. These results showed that the MLR sensor is suitable for wide range of applications.

CHAPTER 5: CONCLUSIONS

In this work two types of experiment were done. The first one was to test the sensitivity to the humidity (35% - 85%) using MLRs base on different micro-fiber diameters (3 µm, 4 µm, 5 µm and 7 µm) and straight micro-fiber. The main conclusions obtained on this experiment. All MLRs got high Q-factor > 10^5.the value of Q-factor increases with the decrease of the micro-fiber diameter, With the maximum Q-factor resulting in the case of 3 µm diameter micro-fiber. Increasing the micro-fiber diameter range of (3 µm - 7 µm) proved to enhance the sensitivity and stability. The 7 µm diameter micro-fiber that used in the experiment is the best choice from the range of diameters based on sensitivity and stability point of view.

The second experiment was to test the sensitivity for the formaldehyde liquid (0% - 5%) concentration level using MLR base on 7 µm diameter and straight micro-fiber. And the main conclusion drawn from the experiment are, the value Q-factor obtained in this experiment is comparable to these obtained in case of humidity sensing which is > 10^5. The sensitivity of MLR (3.6561 dBm/RH%) is higher than in the case of the SmF (4754 dBm/RH%).

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