PMMA microball resonator for formaldehyde liquid sensing

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Abstract. This experiment reports the fabrication of the Polymethyl Methacrylate (PMMA) Microball Resonator (MBIR) as a liquid sensor tested on various level of concentration of the Formaldehyde liquid. The PMMA MBIR is fabricated via the “dipped and twirl” method to create the sphere-shaped ball with diameter Db = 320um. The MBIR is then optically excited using a 9μm PMMA microfiber and was found to have a Q Factor of >10⁴. The MBIR was then employed as a liquid sensor with the level percentage range between 0% to 4% of Formaldehyde liquid and the performance is compared with a non MBIR microfiber. The MBIR sensor was found to have a sensitivity of 6.94 dBm/%, linearity >90% and P-value of more than 10⁵. The PMMA MBIR liquid sensor was also found to have good repeatability and stability over a period of 60 seconds.

1. Introduction

In the complexity of the formaldehyde substance CH₂O, hydrogen, oxygen and carbon are three main compounds that exist in the Formaldehyde. Formaldehyde is a colourless, strong smell gas which is used in the making of many building materials and household products. It is used in pressed-wood products, such as particleboard, plywood, and fiberboard; glues and adhesives; permanent-press fabrics; paper product coatings; and certain insulation materials. The formaldehyde also exists in all substance and can be found in many by-products including plants, fish and human living. Formaldehyde can be broken down generally within hours in the air and easily dissolves in water. Formalin is the name of the substance when it is dissolved in water which is commonly used as an industrial disinfectant and as a preservative in funeral homes and medical labs. The formalin is also used as a preservative in some food products, such as antiseptics, medicine and cosmetics. Formaldehyde can be added as a preservative to food, but it can also be produced as the result of cooking and smoking.

Various sensing configurations of optical fiber sensors have been demonstrated such as fiber Bragg grating sensors, side polished optical fiber sensors, optical microfiber sensors, each of which has
specific advantages and disadvantages. One class of optical components, the optical micro-resonators, have also shown to have promising properties for sensing applications. Optical micro-resonators (OMR) are optical components which permit the circulation of light through a micron-length closed path fabricated in structures such as microspheres, microdisks and microrings loop and have employed for a verity of different biological or chemical sensors. One subclass of OMRs, namely the whispering gallery mode (WGM) resonators, which permit specific resonance (modes) of the waves along the ‘edges’ of the optical resonator, produces evanescent fields of the resonator. The resonating modes are also dependent on the refractive index of the surrounding media. As the light travels through the resonator multiple times, any change in both the absorption and refractive index of the surrounding medium is both amplified and is instantly measurable at the output. Multiple geometries of WGM resonators have been investigated but have primarily focussed on glass-based material.

In conjunction with serious needs, many studies have been reported on the detection of the formaldehyde liquid. Several available methods of detection of formaldehyde in air and water have been reported. For air samples is based on spectrophotometry, is one of the widely used methods, while for aqueous samples high-performance liquid chromatography is a preferred choice. The used of both of the techniques are highly sensitive but are costly. Therefore it is significant to develop a precise and sensitive sensing method for formaldehyde detection due to its implementation and applications is widely used in our daily lives.

2. Experimental Setup

First, a small amount of the Polymethyl Methacrylate (PMMA) powder was heated on a glass plate at around 150°C – 200°C. The PMMA powder was obtained from Across Organic with an Mw of 35 000. A silica microfiber was then fabricated from a single-mode fiber (SMF-28) using the flame brushing technique to produce a non-adiabatic optical microfiber with a diameter of 9 μm and a waist of 5 mm. The fiber was then broken into two and dipped into the molten PMMA. The two ends of the silica fiber were then lifted from the molten PMMA solution, which then forms a 'bridge' between the broken ends of the optical microfiber. To create a PMMA microfiber, the two ends were then pulled apart, producing a PMMA microfiber with an approximate diameter and length of 9 μm and 2 cm, respectively.

A PMMA microball resonator was then fabricated using a “dip-and-twirl” technique. This technique involves a tapered fiber tip being dipped into a small amount of molten PMMA and slowly twirled and pulled upwards to form a sphere-like structure. The fabricated structure was then heated by using a torch to form a microball-shaped resonator, shown in Figure 1. The PMMA Microball Resonator (MBIR) has a diameter (Dₐ) of 320 μm measured using an optical microscope as shown in Figure 2. This use of the PMMA for both the microfiber and MBIR reduces coupling losses due to refractive index differences, potentially increasing the performance of the MBIR sensor.

Figure 1. Fabrication of the PMMA Microfiber with a diameter of 9μm and 5mm waist
The PMMA MBIR was characterized by launching light from a tunable laser source (ANDO AQ4321D) with a working wavelength of between 1520 nm and 1620 nm to one end of the broken optical microfiber and collecting the resulting output power by using an optical power meter (THORLABS S145C) connected to the other end of the optical microfiber. The laser was then tuned from 1520.10 nm to 1520.30 nm with a wavelength interval of 1 pm, with the output power recorded. The resulting transmission spectrum is shown in Figure 3. The insertion loss was measured to be approximately 50 dB and includes losses from coupling into the PMMA microfiber as well as the PMMA MBIR. However, as the microfiber and resonator are both made of the same material, it is expected that coupling losses due to Fresnel reflection are relatively small and that the main loss originates from scattering losses due to air bubbles inside the PMMA MBIR as well as from the surface of the resonator. The Q-factor of the MBIR is then calculated from the definition of $Q = \Delta \lambda / \lambda$, where $\lambda$ is the resonant wavelength and $\Delta \lambda$ is the FWHM of the transmission dip and was found to be $1.689 \times 10^5$, similar to silica-based resonators such as silica microbottle and microball resonators.

Figure 2. Fabrication of the PMMA Microball Resonator (MBIR)

Figure 3. PMMA microfiber $9 \, \mu m$ diameter transmission modes.
The performance of the PMMA MBIR as formaldehyde sensor was then investigated by employing the setup as shown in Figure 4. The PMMA MBIR is placed in between the formaldehyde liquid and the microfiber. One end of the microfiber is connected to Tunable Laser Source (TLS) and the other end connected to the Optical Power Meter (OPM) to measure the transmitted power. The formaldehyde liquid concentration was then varied from 0% to 4%. The wavelength was set to $\lambda = 1520$ nm and the transmission of the PMMA MBIR at a different percentage of concentration were recorded. The experiment was carried out 3 times to reduce the random errors and investigate the repeatability, the linearity and the stability of the sensor. The stability of the sensor was also studied for 60 seconds by recording the transmission output at a different concentration level.

Figure 5. Experimental setup of the PMMA Microball resonator (MBIR) for formaldehyde concentration measurements.

3. Results and Discussions
Figure 6 shows the output signal of the Tunable Laser Source (TLS) transmitted through the PMMA with and without the MBIR at different formaldehyde concentration level. The transmission decreased with the increasing percentage of concentration of the formaldehyde for both with and without the MBIR. The linearity, sensitivity, standard deviation and P-Value of the PMMA MBIR is given in Table 1. The sensitivity of the PMMA MBIR is higher than the one without the resonator at 6.94dBm/\% as compared to 5.76dBm/\%. From the result, it can be seen that the standard deviation of the bare PMMA microfiber is less than the one with the PMMA MBIR resonator attached to it with the value of 9.2484dBm and 11.5184dBm respectively. This shows that the bare PMMA microfiber data-taking is
more stable than the one with attached MBIR resonator. The P-value both of the with and without PMMA MBIR was found to be $2.60 \times 10^{-6}$ and $3.83 \times 10^{-5}$ respectively indicates that the data collection is successfully conducted. This overall reduction may be attributed by the resonator loss, which increases with the increasing formaldehyde percentage of concentration due to the changes of the refractive index and the surface adsorption. However, as the light resonates around the MBIR, the losses accumulate for each round trip, thus increasing overall loss and increasing sensitivity. It can, therefore, be surmised that for liquid formaldehyde sensing, the PMMA MBIR demonstrates better performance compared to the bare PMMA microfiber.

Figure 6. The transmitted power value of the PMMA microfiber with and without the MBIR at varying formaldehyde concentration.

Table 1: Analysis of MBIR and without MBIR microfiber performance in formaldehyde concentration activity.

| Parameters                  | No-MBIR   | With PMMA MBIR |
|-----------------------------|-----------|----------------|
| Linearity (%)               | 98.49%    | 95.27%         |
| Sensitivity (dBm/%)         | 5.7607 dBm| 6.9400 dBm     |
| Standard deviation (dBm)    | 9.2484 dBm| 11.5184 dBm    |
| P-value                     | $3.83 \times 10^{-5}$ | $2.60 \times 10^{-6}$ |
| Linear Range (% concentration) | 0 - 4     | 0 - 4          |

4. Conclusions

This paper discussed the performance of PMMA MBIR and without MBIR microfiber as a liquid sensor tested on the formaldehyde liquid at different concentration level. First, a PMMA microfiber with a diameter of 9 μm and a length of 2 cm is fabricated by dipping two optical microfibers and pulling them apart. Then, a PMMA MBIR is fabricated using the “dip and twirl” method to create the sphere-shaped ball with diameter $D_b = 320\mu m$. The fabricated PMMA MBIR is then optically excited by a TLS via a 9 μm PMMA optical microfiber and characterized at the wavelength of $\lambda = 1520 \text{ nm} - 1530\text{nm}$. The PMMA microfiber sensor both with and without the MBIR were then employed for formaldehyde
sensing with a concentration of between 1-4%. The sensitivity, the linearity, standard deviation and P-Values were calculated, and the results indicate that the PMMA microfiber with the MBIR is superior in performance. The experiment was then repeated 3 times to investigate the accuracy and stability of the two sensor configurations for different formaldehyde concentrations. In general, the stability of both sensor configurations are comparable, but the sensor with the MBIR has superior sensitivity and repeatability, indicating that the PMMA MBIR is a promising sensor as a liquid formaldehyde sensor.

Acknowledgements
The authors would like to acknowledge the University of Malaya, Kuala Lumpur for their financial support. [Ni: RP039C-18AFR].

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