

Lossy Mode Resonance Based Fiber Optic Sensor for the Detection of Acetone Concentration

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Abstract. Diabetic ketoacidosis (DKA) is a serious complication arising due to the shortage of insulin that allows blood sugar into cells to be used as energy. As a result, the liver begins to break down fat for energy, thus producing acids called ketones. The severity of DKA influences the number of ketones produced in the human body. Therefore, the acetone level in the human body samples has the potential to be used as a biomarker towards the analysis of diabetic levels. The present work reports the development of a Lossy Mode Resonance (LMR) based fiber optic sensor to detect acetone concentration in liquids. A sensing region was developed by coating Aluminium doped Zinc Oxide (AZO) over the unclad core region using sputtering technique. The structural, morphological, and optical properties of the AZO coating were analyzed using X-ray Diffraction (XRD), Scanning Electron Microscope (SEM), and an ellipsometer device. The sensor probe used to measure the acetone concentration ranging from 10 $\mu\text{l/ml}$ to 800 $\mu\text{l/ml}$ recorded resonance wavelength shifts in LMR1, LMR2, and LMR3 of 38 nm, 19 nm, and 9 nm, respectively. The wavelength shift was larger for LMR1 than for the other peaks with a sensor response of 4.73% and a sensitivity of 0.3 nm/($\mu\text{l/ml}$). © 2023 Journal of Biomedical Photonics & Engineering.

Keywords: optical sensor; lossy mode resonance; acetone, diabetes.

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1 Introduction

Diabetic ketoacidosis (DKA) is a chronic condition that frequently affects people with type 1 diabetes. DKA occurs when the body cannot effectively utilize blood sugar (glucose) for energy because of insufficient insulin production. Because the cells cannot use blood sugar as the energy source, they consume fat instead. The decomposition of fat results in the production of blood byproducts known as ketones [1]. Acetone is one of the ketone bodies, and its elevated level during diabetic ketoacidosis (above 80 mg/dL or 4.4 mmol/L (1mg/dL = 0.055 mmol/L) i.e., 1.0191 $\mu\text{l/ml}$ (1 ml of acetone weighs 0.7855 g)) indicates a metabolic disorder. Therefore, it is essential to monitor the acetone concentration in diabetics to determine the severity of DKA. The laboratory diagnostics for DKA includes blood glucose assays, serum electrolyte determinations,

blood urea nitrogen (BUN) evaluation, urine ketone concentration, and arterial blood gas (ABG) measurements [2]. Among these, the detection of urinary ketone levels is one of the non-invasive techniques that is painless and comfortable [3]. Due to their potential for accurate disease diagnosis and treatment, analytical methods employing non-invasive nanotechnology-based techniques have gained popularity. In the present investigation, we have developed an LMR-based optical fiber sensor that can potentially replace the intrusive and unpleasant standard blood tests.

Standard methods such as gas chromatography and mass spectrometry that are available for the detection of acetone are often cumbersome and take up a great deal of space. Further, the sampling procedure is complicated and time-consuming. Another type of detector, namely the flame and light ionization reacts negatively to oxygen-containing organic substances [4].

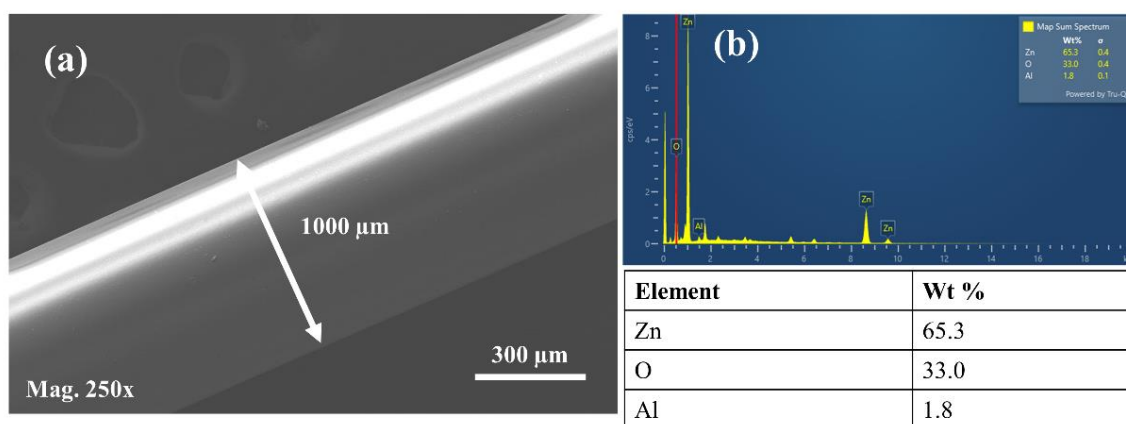


Fig. 1 (a) SEM image of AZO coated probe and (b) EDS spectrum of AZO.

In addition to these methods, chemiresistive-based sensors are used to measure the acetone concentration [5]. However, the requirement for a high working temperature for such sensors poses a major disadvantage. Hence, the detection of acetone requires a sensor that is basic, sensitive, and capable of operating at room temperature.

In this scenario, fiber optic sensors with rapid response time, small size, multiplexing sensing capability, versatility, and immunity to electromagnetic interference, can serve as alternative to the aforementioned sensing methods. Several interrogation techniques, including evanescent waves [6], Surface Plasmon Resonance (SPR) [7], and Localized Surface Plasmon Resonance (LSPR) [8], have been used to build optical fiber sensors. According to the reports, the evanescent wave technique is one of the most cost-effective methods for determining the quantity of water in ethyl alcohol [9]. However, the evanescent mode optical fiber is dependent on intensity modulation, and hence, variations in the intensity of the light source can affect the output of the sensor. In such cases, the SPR approach could be employed to detect liquid sample Volatile Organic Compounds (VOCs) with higher sensitivity than the evanescent mode technique [10]. Nevertheless, the SPR-based fiber optic sensor with higher sensitivity and quicker response time is often difficult to fabricate [11] as it necessitates the use of noble metals and transverse magnetic polarized light. Therefore, for the purpose of acetone detection, we propose the development of an LMR-based sensor that offers numerous advantages, such as polarization independence, ease of fabrication, and cost-effective sensing layer [12]. LMR can only be produced when the real component of the permittivity of the supporting material is positive, greater in magnitude than its own imaginary component, and greater than that of the real components of the optical fiber core and the surrounding medium. Metal oxides such as zinc oxide (ZnO) [13] and indium tin oxide (ITO) [14] are widely used as LMR supporting materials for refractive index measurements. Despite the fact that the refractive index of the supporting material has a substantial effect on the sensitivity of LMR

sensors, the performance of the sensor [15] is optimized by selecting materials of suitable thickness. For the present study, Aluminum-doped zinc oxide (AZO) with exceptional electrical conductivity, high transparency, and outstanding chemical and thermal stability was chosen as the LMR supporting material.

The present study aims to develop LMR-based sensor for the detection of acetone concentrations below a specific limit. The proposed sensor device was evaluated for acetone in the concentration range of 10 $\mu\text{l/ml}$ to 800 $\mu\text{l/ml}$, and the corresponding resonance wavelength shift was measured. To the best of the authors knowledge, this is the first experimental report on an LMR-based acetone sensor. The efficacy of the sensor probe was determined by evaluating its sensing characteristics, namely its sensitivity and sensor response.

2 Preparation of Sensor Probe

The sensor probe was fabricated using clad modification technique, which involves removing 2 cm of the clad and replacing it with an AZO layer using Radio Frequency (RF) magnetron sputtering technique.

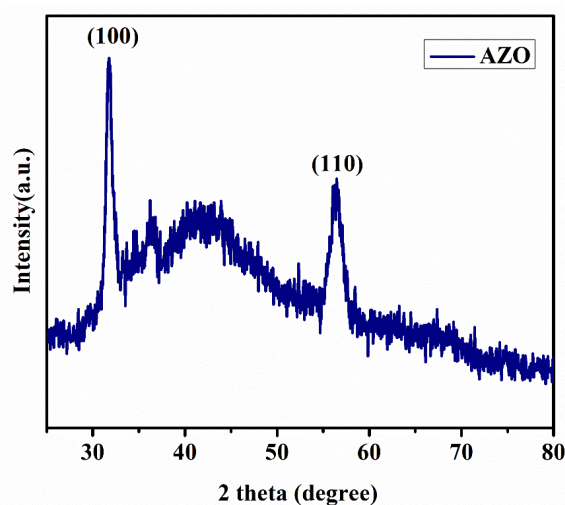


Fig. 2 XRD pattern of AZO coated thin film.

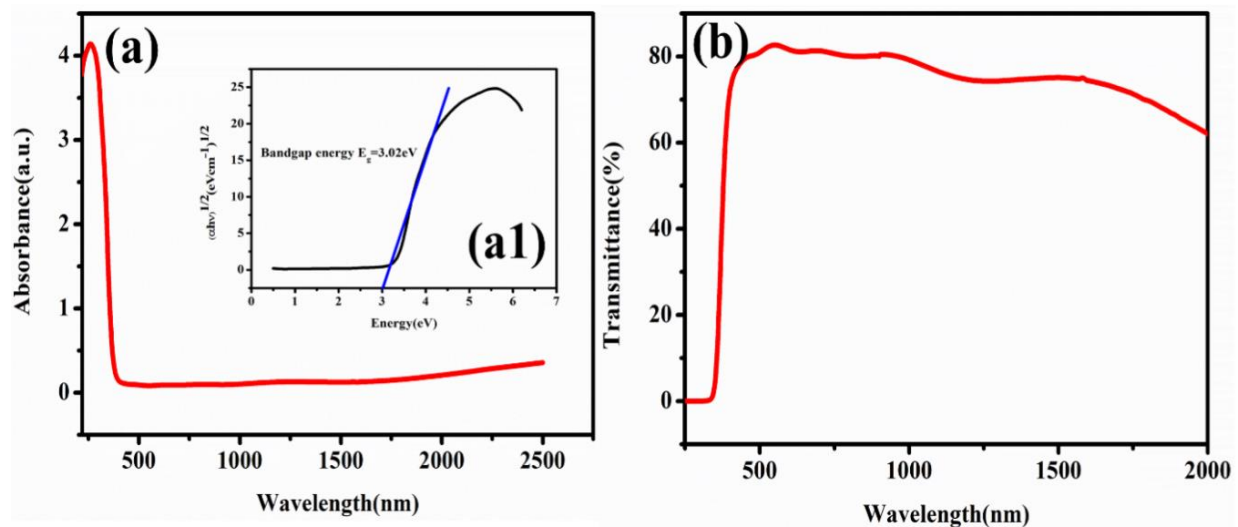


Fig. 3 (a) Absorption and (b) transmission spectra of AZO thin film.

For the preparation of the sensor probe, plastic-clad silica core (PCS) optical fiber (0.39 NA) with a 1000 μm core diameter (FMT1000, Thorlabs) was utilized. The index of refraction of the plastic-clad is 1.406, while that of the optical fiber core is 1.453. Using chemical etching method, the plastic-clad portion of the optical fiber was removed by immersing it for 5 min in an HF bath. The unclad portion of the optical fiber was fixed inside the RF Magnetron deposition chamber and the 99.99% pure AZO target (98% ZnO, 2% Al) was inserted 7 cm away from the fiber. AZO was coated for 25 min with a steady flow of 10 SCCM of Argon (Ar) and 5 SCCM of Oxygen (O_2) at pressures of 3×10^{-5} mbar (Base) and 25×10^{-3} mbar (Deposition), respectively over the interior of the fiber. During deposition, the optical fiber within the chamber was continuously rotated using a DC-motor-driven rotating system to ensure uniform coating thickness.

3 Characterization of the Sensing Layer

3.1 SEM & EDS Analysis

SEM measurements were carried out to determine the uniformity of the coating. The SEM images shown in Fig. 1 revealed uniform coating of the top surface of the optical fiber with AZO layer. The EDAX analysis confirmed the presence of aluminium (Al), zinc (Zn), and oxygen (O) molecules.

3.2 X-ray Diffraction

The crystalline properties of the prepared AZO layer were investigated using X-ray diffraction studies. The XRD pattern in Fig. 2 showed two major peaks at angles of 31.7° and 56.5° that can be attributed to the (100) and (110) diffraction planes.

3.3 UV-Vis Spectroscopy

UV-Vis spectroscopy was used to analyze the optical properties of the AZO layer. A sharp absorption edge at

~ 265 nm as shown in Fig. 3 was observed. The optical bandgap energy (E_g) of the AZO thin film was calculated using Tauc's equation given by Eq. (1).

$$\alpha h\nu^{1/2} = A(h\nu - E_g), \quad (1)$$

where A is a proportionality constant, $h\nu$ is the incident photon energy, α is the absorption coefficient, and E_g is the optical bandgap. The inset of Fig. 3(a) shows the plot of $\alpha h\nu$ vs $h\nu$ from which the optical bandgap of the AZO thin film was determined to be 3.02 eV. Further, the measured optical transmission of the AZO coated layer showed a maximum transmission of $\sim 80\%$ as seen from Fig. 3(b). The optical constants (n and k) of the thin films were determined using an ellipsometer (J. A. Woollam, Alpha-SE). Fig. 4 shows the dispersion characteristics of the AZO thin film obtained using the Lorentz model. The real parts of the refractive indices of the modified clads (AZO thin films) were higher than their imaginary parts. This confirmed that AZO is a supportive material for LMR sensing [16].

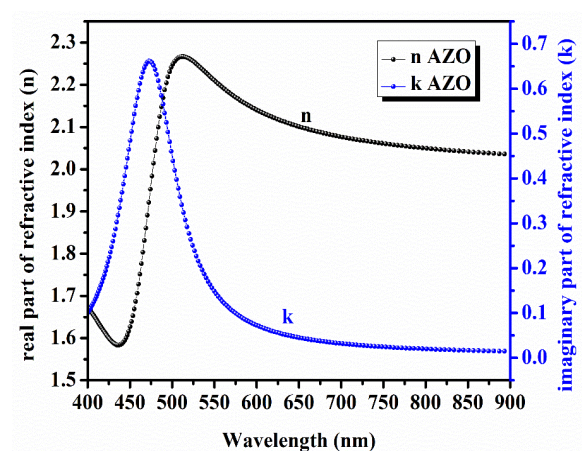


Fig. 4 Dispersion characteristics of AZO thin film.

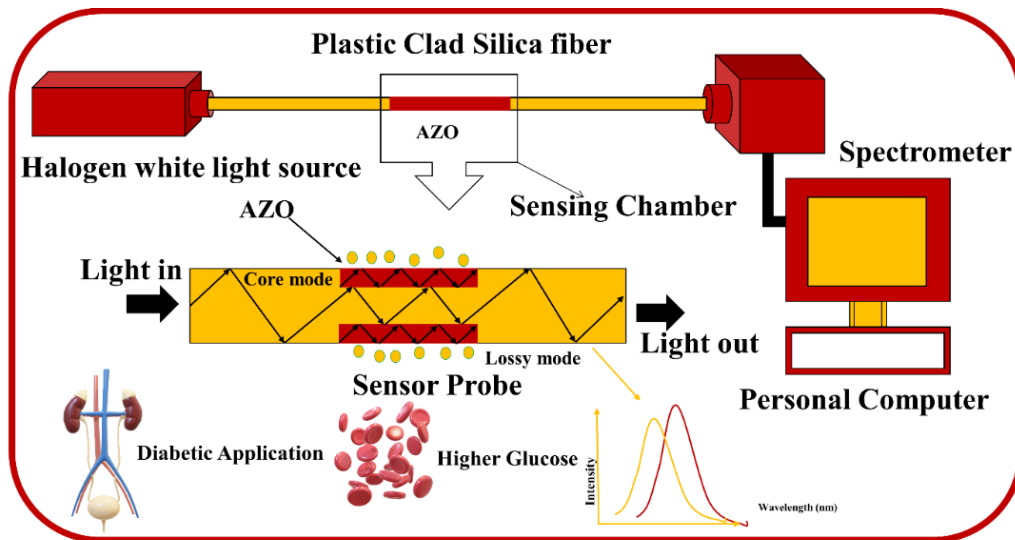


Fig. 5 Schematic representation of the experimental setup of AZO coated LMR based acetone sensor.

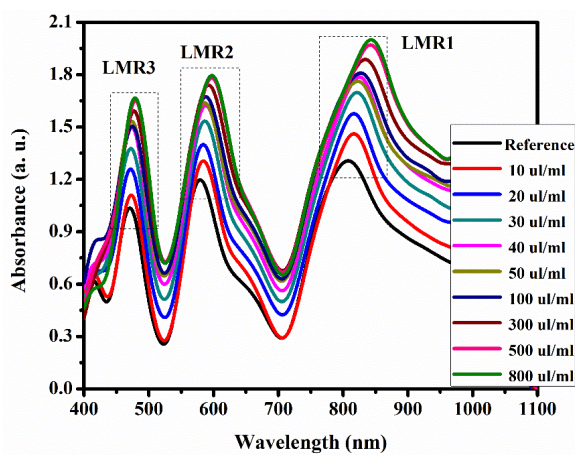


Fig. 6 Wavelength shift spectra of AZO coated LMR based acetone sensor.

4 Experimental Analysis

The schematic representation of the experimental setup of the fiber optic-based sensor for the detection of acetone is shown in Fig. 5. One end of the optical probe is connected to a broadband halogen white light source (200–2200 nm), while the other end is connected to a spectrometer (200–1100 nm). Acetone samples of different concentrations were prepared by diluting it with distilled water. The corresponding resonance wavelength shift for each concentration was measured.

4.1 Spectral Response of the Sensor Probe (SP)

Fig. 6 represents the LMR absorbance spectra of the SP for different concentrations of acetone (0–800 $\mu\text{l/ml}$) in water. Three maximum LMR peaks were observed at

806 nm (LMR1), 579 nm (LMR2), and 470 nm (LMR3) for 0% of acetone (water).

Each of these maxima correspond to a new mode referred to as a 'lossy mode' that passes the 'cut-off' condition and is governed by a 250 nm layer thickness. At a wavelength of 806 nm, the first mode is near to the cutoff condition, and the spectrum generates LMR1. As the wavelength decreases, a second mode overcomes the condition and passes through the coating. This is displayed in the spectrum as LMR2 at 579 nm. Similarly, the condition for guiding a third mode (LMR3) in the coating arises at a wavelength of 470 nm. Interestingly, it has been observed that with increase in the thickness of the AZO coating, a wider range of lossy modes can be guided through it. As the concentration of acetone increases, the change in the Surrounding Medium Refractive Index (SMRI) impacts the LMR resonance peaks. Since LMR sensors are extremely sensitive to SMRI, a minor change in the refractive index has a significant impact on the shift of the resonance wavelength as shown in Fig. 7.

The LMR resonance wavelength increases as the concentration of acetone rises, as shown in Fig. 7(a), (b), and (c). With a change in acetone concentration from the reference to 800 $\mu\text{l/ml}$, the resonance wavelength of LMR1 shifts from 806.3 nm to 844.5 nm, while the resonance wavelength of LMR2 changes from 579.7 nm to 597.7 nm. For the same range of acetone concentrations, the LMR3 resonance wavelength goes from 470.3 nm to 479 nm. From the observation, it has been inferred that an increase in the refractive index of the surrounding medium causes an increase in the resonance wavelength (i.e., redshift) up to an acetone concentration of 800 $\mu\text{l/ml}$. LMR1 shows a maximum wavelength shift of 38 nm as compared to LMR2 and LMR3 with wavelength shifts of 19 nm and 9 nm, respectively for acetone concentration ranging from reference to 800 $\mu\text{l/ml}$.

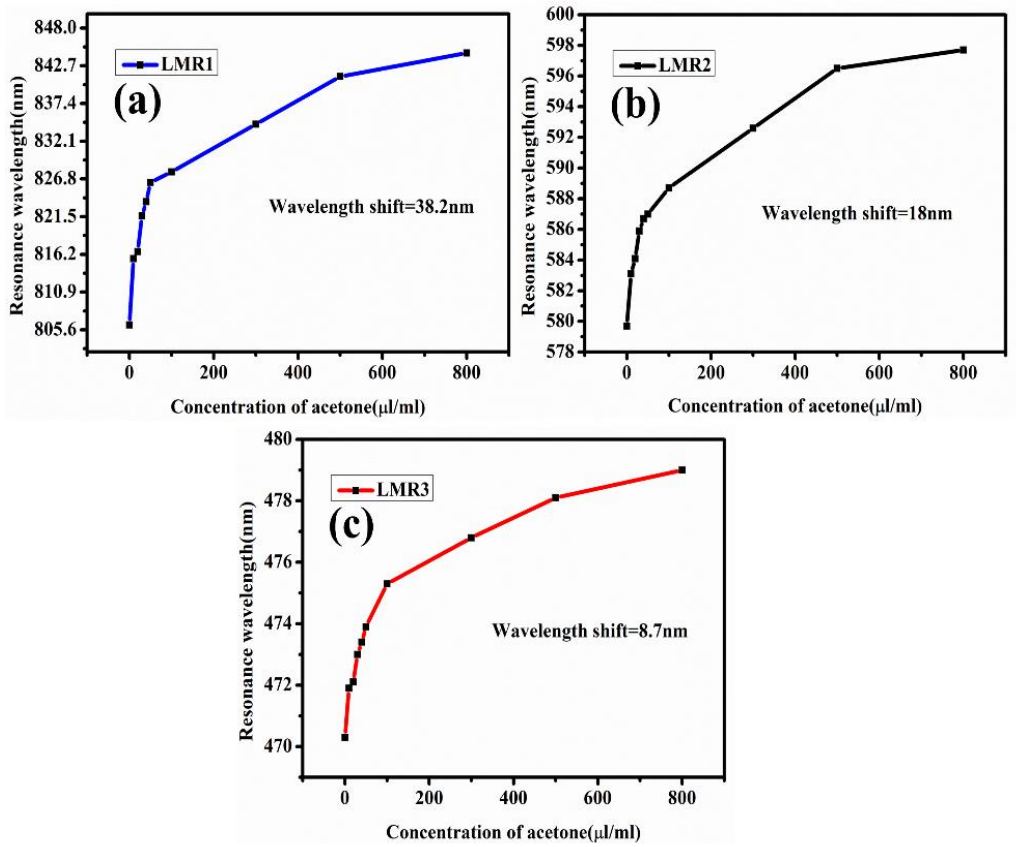


Fig. 7 Variation of resonance wavelength of AZO coated sensor probe (a) LMR1, (b) LMR2, and (c) LMR3.

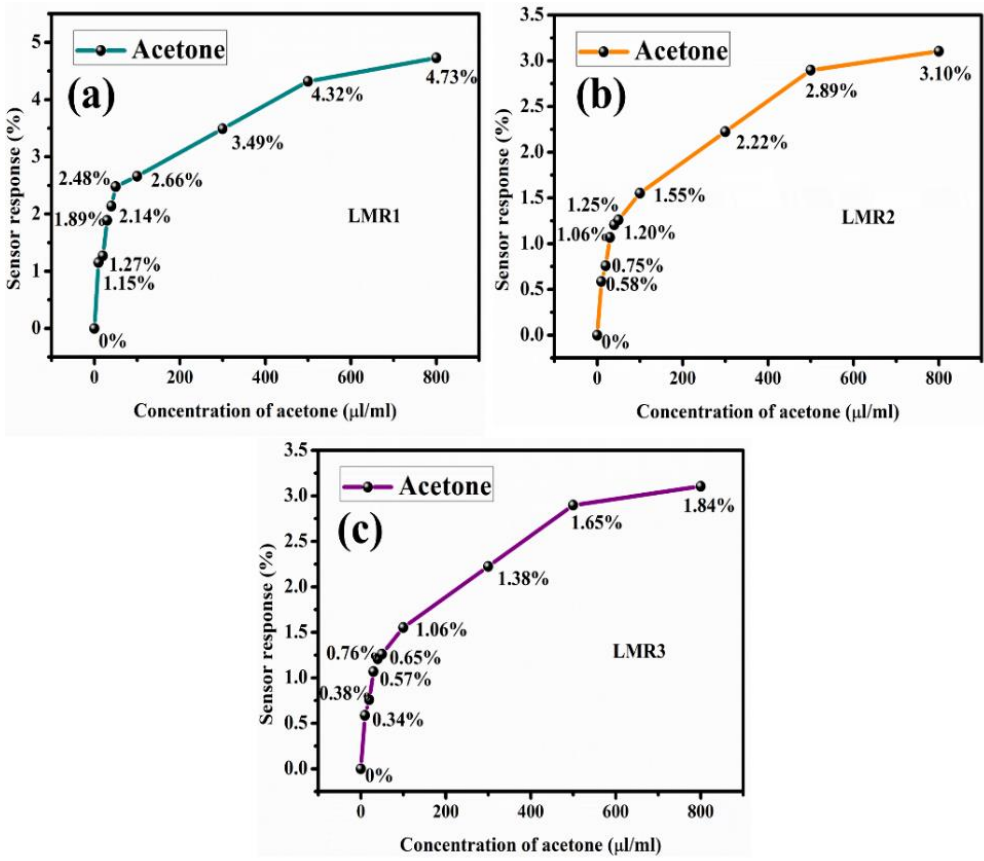
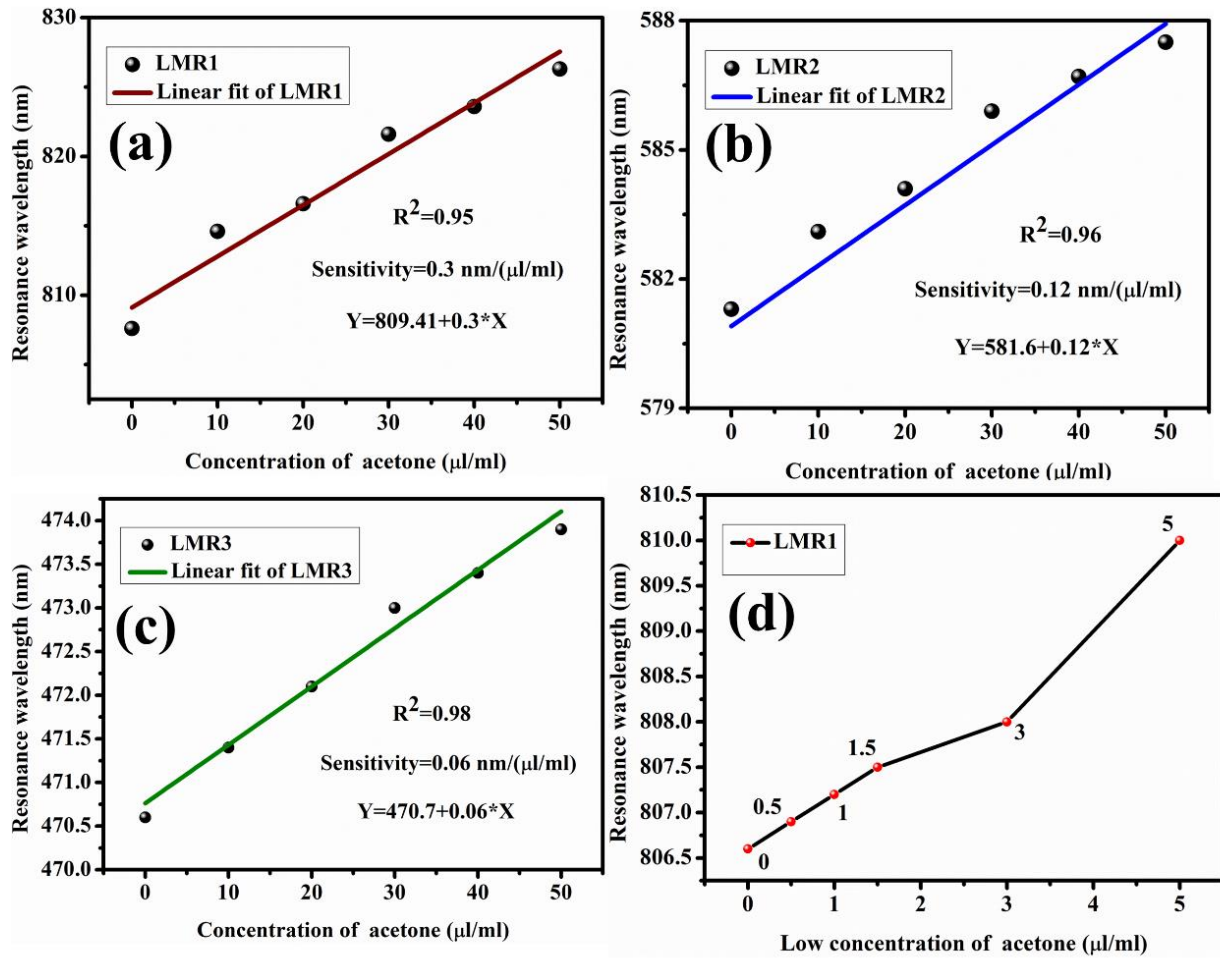


Fig. 8 Change in sensor response of prepared AZO coated sensor probe (a) LMR1, (b) LMR2, and (c) LMR3.



*Sensitivity= Wavelength (nm)/ Acetone concentration (μl/ml)

Fig. 9 Sensitivity plots of acetone sensor for (a) LMR1, (b) LMR2, (c) LMR3, and (d) change in resonance wavelength for lower concentrations (LMR1).

5 Sensor Response

The sensor response is defined as the relative change in the resonance wavelength in the presence of acetone over the resonance wavelength of the reference. It is determined using Eq. (2) given below [17].

$$\text{Sensor response (\%)} = \frac{S_w - S_a}{S_w} \times 100 \%, \quad (2)$$

where S_w is the resonance wavelength of the initial concentration of acetone and S_a is the resonance wavelength at different acetone concentration.

Fig. 8 shows the sensor response for varying acetone concentrations, from reference to 800 μl/ml. The sensor response for LMR1 was enhanced from 0% to 4.73% as the amount of acetone goes up from reference to 800 μl/ml. In the same way, the sensor response for LMR2 was enhanced by 3.10% while that for LMR3 was enhanced by 1.84%. Further, an improved sensor response was observed at elevated concentration of acetone and comparatively, the sensor response at LMR1 was higher than that at LMR2 and LMR3.

6 Sensitivity

The sensitivity of the proposed acetone sensor is defined as the change in resonance wavelength per change in acetone concentration as expressed by Eq. (3) [18].

$$S_n = \Delta\lambda_{res} / \Delta C_a, \quad (3)$$

where $\Delta\lambda_{res}$ is the resonance wavelength shift and ΔC_a is the difference in concentration of acetone.

The sensitivity of AZO coated LMR probe towards acetone detection was determined from the plot of resonance wavelength vs. acetone concentration shown in Fig. 9 (a–c). The sensitivities were found to be 0.3 μl/ml, 0.12 μl/ml, and 0.06 μl/ml, respectively, for LMR1, LMR2, and LMR3. The shift in resonance wavelength per volume percentage of acetone was significantly greater for LMR1 because higher wavelength resonances are more sensitive to the surrounding medium's refractive index than lower wavelength resonances. Further, the sensor could also detect concentration of acetone as low as 0.5 μl/ml as shown in Fig. 9(d).

7 Summary and Conclusion

A lossy mode resonance (LMR)-based optical fiber sensor for acetone detection in liquid samples under diabetic ketoacidosis was developed. RF Magnetron sputtering was used to deposit the AZO sensing layer over the unclad core of the optical fiber to fabricate the LMR-based optical fiber sensor probe. Using the characterization techniques such as SEM, XRD, UV-Vis spectroscopy, and ellipsometry, the uniformity, crystalline structure, and thickness of the AZO sensing layer were determined. Different concentrations of acetone ranging from 10 $\mu\text{l/ml}$ to 800 $\mu\text{l/ml}$ were used to examine the LMR wavelength shift using a sensor instrument. The sensor probe's absorption spectrum revealed three LMR peaks namely, LMR1, LMR2, and LMR3 when it was immersed in 0% acetone (DI water). With increase in the concentration of acetone from 10 $\mu\text{l/ml}$ to 800 $\mu\text{l/ml}$, the resonance wavelengths for each of the three LMR peaks exhibited shifts. The

maximum shifts in resonance wavelengths for LMR1, LMR2, and LMR3 were 38 nm, 19 nm, and 9 nm, respectively. The observations further indicate that LMR shifts with acetone concentration was higher at longer wavelengths (LMR1 > LMR2 and LMR3). The maximal sensor response of the LMR1 probe was 4.73% with a sensitivity of 0.3 nm/($\mu\text{l/ml}$).

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Disclosures

The authors declare no conflict of interest.

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