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Etched optical fiber for measuring concentration and refractive index of sucrose solutions by evanescent waves

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Abstract
The study of the reduction of an optical fiber by chemical etching has been suggested to determine the concentrations of sucrose in water and their refractive indices by evanescent waves using a coherent infrared source. The cladding of a single-mode optical fiber was removed at a rate of ~3.27 μm min^{-1} using hydrofluoric acid until it reached a diameter of 7.3 μm, similar to the core of the fiber. This fiber was used to characterize sucrose solutions at different amounts employing a continuous wave infrared laser source at 1550 nm. The sucrose was dissolved in water to evaluate the quantitative sensor response based on the transmission relationship. The experimental results showed that the refractive indices obtained by the evanescent absorbance were in the range of 1.31–1.44 for concentrations of sucrose between 0% (water) to 65%. Additionally, it was determined that for concentrations higher than 65% of sucrose, the refractive index of the solution is similar to the core of the fiber, and therefore the total internal reflection was not possible. The results obtained in this work suggest that the etched optical fiber can be used as a refractive index sensor, which may play an important role in chemical applications.

Keywords: evanescent sensor, optical fiber, evanescent wave, chemical etching, sucrose

(Some figures may appear in colour only in the online journal)

1. Introduction

Optical fibers have been widely used as sensing devices because they offer immunity to electromagnetic interference as well as offering electrical insulation, resistance to corrosion, low cost and other advantages [1–3]. Optical fiber sensors based on the evanescent wave phenomenon have received considerable attention due to the ability of the evanescent wave to penetrate the surrounding environment of the fiber and interact directly or indirectly with the analyte [4–6]. When these sensors are immersed in an aqueous media, the penetration depth of the wave is strongly linked to the refractive index of the solution in which it is immersed [6–9]. These types of sensors have already found applications in the evaluation of physical parameters, analysis of biological samples and chemical reagents [10–15].

Chachlani and Chhattopadhay recently described the study and design of an evanescent wave absorption sensor and its applications in the detection of pollutants in water. The results showed that the absorption coefficient is proportional to the solution concentration and does not depend on the length of the uncovered optical fiber [16]. Patrialova and coworkers reported an optical fiber sensor, which was folded into a U-shape to measure the concentration of alcohol using the principle of multimode interference and an evanescent field. It was determined that the sensitivity can be increased, bending the sensor, and it was shown that the concentration of alcohol is dependent on the mechanical properties of the folding of the fiber into the U-shape [17].

In this paper, we present a simple, repeatable and controllable approach to fabricate a sensor reduced by chemical etching to measure the concentrations of sucrose by evanescent waves. The experimental data indicate that when the optical fiber is immersed in hydrofluoric acid (HF), the diameter of the fiber decreases linearly with time. Using an optical fiber without cladding, it was possible to characterize sucrose solutions at different concentrations in a range from 0% (water) to 65%, as well as their respective refractive indices. The proposed arrangement has the advantage of being a low-cost device, and it is easy to manufacture.

2. Evanescent wave sensor principle

Optical fiber sensors based on evanescent wave absorption are typically used to monitor the concentration of absorbing fluids. According to Punjabi *et al*, evanescent waves are energy losses at the core/solution interface caused by differences in their refractive indices [18]. A fundamental parameter is the energy that resides in the cladding (P_{clad}), whose power ratio, transported by the fiber, is given by:

$$P_{clad} = P - P_{core}, \tag{1}$$

where P_{core} is the power in the core of the optical fiber, and P is the input power. Due to the cladding being completely removed from the optical fiber in our experiment; P_{clad} is considered as the optical power that is transported through the solution (P_{sol}). This fraction of energy is very important in evanescent wave absorption sensors, which can be approximated by calculating the evanescent fractional power present in the cladding with the following equation [18].

$$\frac{P_{sol}}{P} = \frac{4\sqrt{2}}{3}\frac{\lambda}{2\pi r\sqrt{n_{core}^2 - n_{sol}^2}}, \tag{2}$$

where λ is the wavelength of the radiation source, r is the radius of the optical fiber and n_{sol} and n_{core} are the refractive indices of the solution and the core, respectively. From equation (2), it is possible to obtain the refractive indices between the light and some interaction medium by using the next expression:

$$n_{sol} = \sqrt{n_{core}^2 - \left(\frac{4\sqrt{2}\lambda P}{6\pi r P_{sol}}\right)^2}. \tag{3}$$

Ahmad and Hench suggest that to increase the intensity of the evanescent wave it is necessary to increase the penetration depth *Dp* in the sensing region [19]. It is convenient to remove the cladding from the region surrounding the core and place the fiber in an aqueous medium, which will be acting as a new cladding. The penetration depth depends on three important parameters: (i) the incident angle θ_i formed at the core/cladding interface, (ii) the wavelength and (iii) the refractive index of the medium, as shown in the following equation [20, 21]:

$$D_p = \frac{\lambda}{2\pi n_{core}\sqrt{\sin^2\theta_i - \left(\frac{n_{sol}}{n_{core}}\right)^2}}. \tag{4}$$

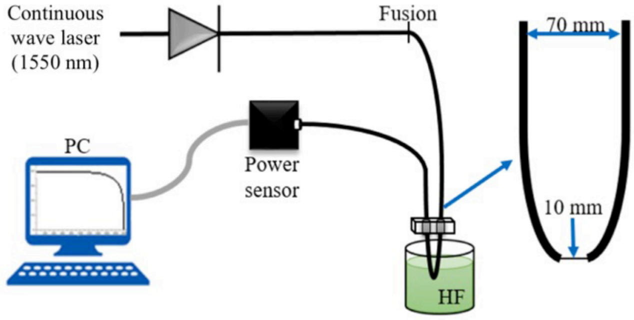

Figure 1. Experimental setup reduction of an optical fiber using HF.

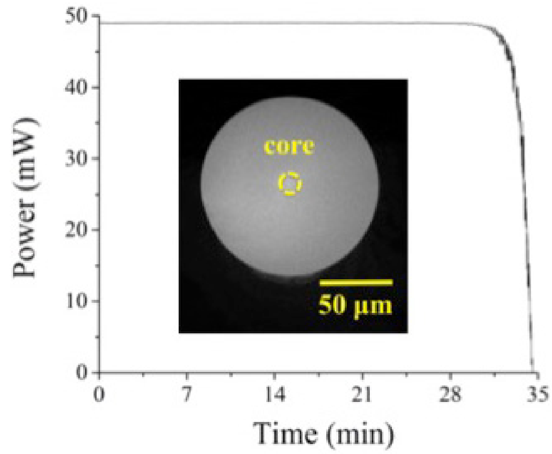

Figure 2. Transmission of the optical fiber during the etching process and cross-section of a single-mode optical fiber obtained by SEM (inset).

3. Experiment

A single-mode optical fiber (SMF-28e+) with 7.5 and 125 μm in the core and cladding diameters was used in this work. A laser diode with an optical fiber output (Thorlabs model FPL1009S) and continuous wave emission at a wavelength of 1550 nm was employed as the radiation source. A power meter (Thorlabs model PM100D) with an InGaAs sensor (Thorlabs model S145C) and an integrating sphere was used as the measurement system. The reduction of the optical fiber was carried out by chemical etching using HF 48.0%–51.0% (JT BAKER 9560-06) and for the cleaning of the fiber, a piranha solution of hydrogen peroxide, sulfuric acid (1:2) and tridistilled water (HYCEL) were used.

To manufacture the optical fiber sensor, approximately 1 cm of fiber coating was removed. Then, the fiber was placed

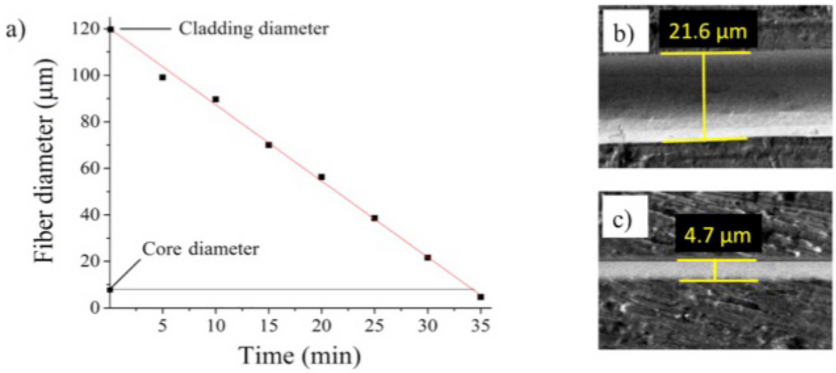

Figure 3. (a) Dependence of the reduction for seven optical fibers immersed into HF, the red solid line indicate a linear fit to the data, and (b) and (c) images obtained by SEM of fiber reduced with HF.

on a plastic holder where one terminal of the optical fiber was spliced into the laser system. The other terminal was connected to the power sensor in order to monitor the transmission of the signal, as shown in figure 1.

To reduce the optical fiber, it was immersed in the HF solution at room temperature with the laser on. The transmitted power was measured and the data were collected and recorded on a computer. The result of the reduction process is shown in figure 2. It can be seen that the power transmitted is constant until the 28th min, after this time an abrupt decay in the transmitted power was observed. According to our measurement, it was observed that the reduction of the fiber depends on the HF concentration, temperature and characteristics of the optical fiber. This behavior of the transmission through the optical fiber, during the etching process is similar to the behavior reported in 2012 by Kbashi, with the difference that the HF concentration is approximately 20% higher in our experiment, for this reason the core of the fiber is reached in a shorter time [22].

The study about the rate of etching was performed using seven optical fibers with the same characteristics, which were immersed in the HF solution at the same time. Later, each optical fiber was withdrawn from the solution at intervals of 5 min. Immediately after, the reduced fibers were cleaned using a piranha solution and water. The diameters of the fibers were measured using a scanning electron microscope (SEM), as shown in figure 3. It is possible to observe that when the optical fiber is immersed in HF at a concentration of 48%–51%, the diameter of the fiber decreases linearly with time, obtaining a rate of etching of ~3.27 μm min^{-1}. Using this information, it was possible to determine that the time in which the cladding was completely removed from the optical fiber, discovering the core, was approximately 34 min. Figure 3(b) and (c) shows images obtained by an SEM of two fibers, which were immersed in HF for more than 30 min.

The reduced fiber was used as a sensing device to different sucrose concentrations. The solutions were prepared making a mixture of tridistilled water with sucrose. For our purpose, 13 solutions of 20 ml were prepared at different sucrose concentrations; these concentrations were increased at each 5%. A reduced optical fiber with a diameter of 7.3 μm was used to perform the characterization of the different testing solutions. The sensing process consisted of immersing the reduced fiber

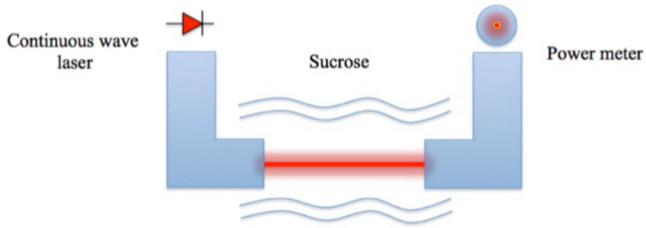

Figure 4. Experimental setup of the sucrose sensor.

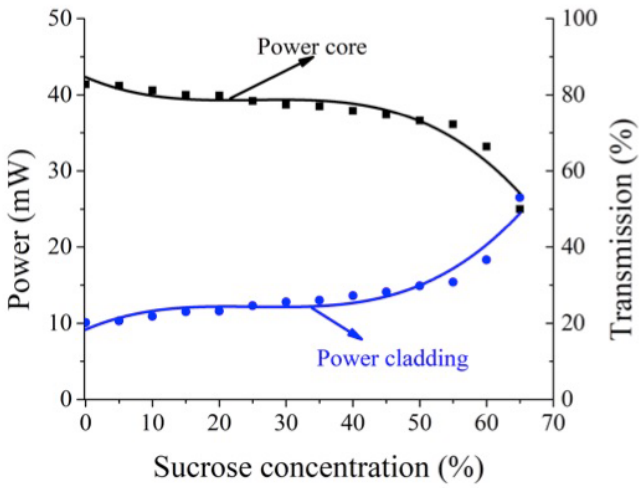

Figure 5. Immersed sensor response in different sucrose concentrations, experimental data (black) and calculated data (blue). Solid lines represent fits to the experimental and calculated data.

into each container and measuring the response through the power meter. This is depicted in figure 4.

4. Results and discussions

The results obtained by the sensor to different sucrose concentrations are shown in figure 5. The sucrose concentrations used were 0%–65%. The black squares represent the optical power measured at the end of the sensor when the fiber was immersed in different sucrose concentrations, and the blue circles are the power in the solution obtained using equation (1). The reported results show a good repeatability. It can be observed that when the sensor is immersed in water, the power

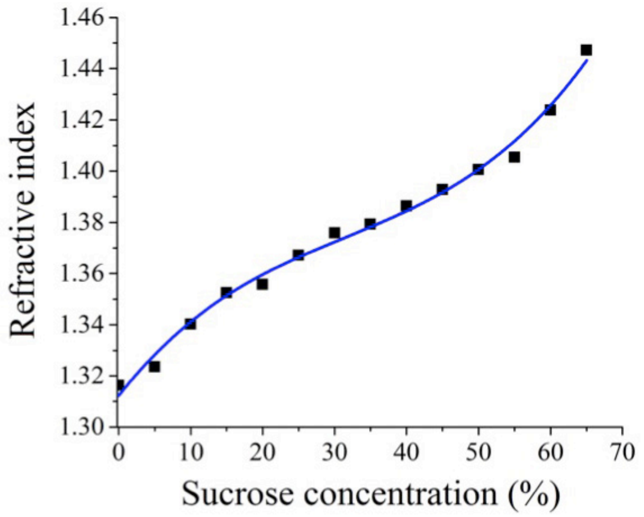

Figure 6. Refractive indices measured for different concentrations of sucrose, the solid line depicts the theoretical fit.

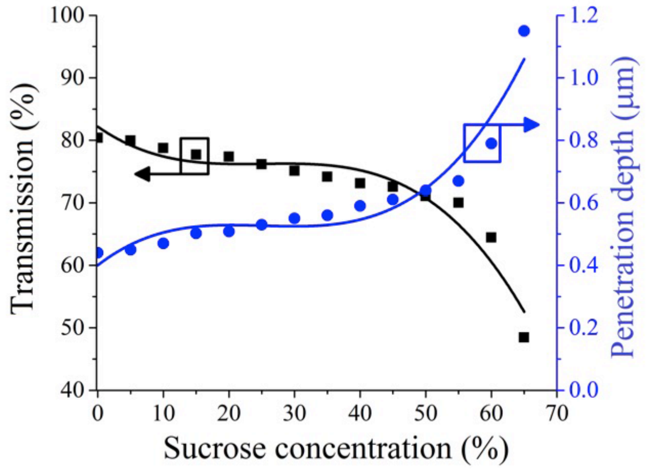

Figure 7. Transmission and penetration depth at different concentrations of sucrose. Experimental data are indicated by points and the continuous lines stands for the theoretical fitting.

in the core is bigger than the power in the cladding. However, when the sucrose concentration is increased the transmission of the sensor decreases This means that the sensor transmission is inversely proportional to the sucrose concentration.

The refractive indices for different sucrose concentrations were calculated using equation (3). The results showed that the refractive indices obtained by the evanescent absorbance were in the range from 1.31–1.44 for concentrations of sucrose between 0% (water) to 65%, as shown in figure 6. The calculations showed that for high levels of sucrose concentration, the refractive index increases; however, for concentrations higher than 65% it is not possible to carry out a characterization. This is due to the fact that refractive index of the solution is beginning to be similar or higher than the refractive index of the core of the fiber, avoiding the fact that there is a total internal reflection inside fiber. The results of the refractive indices are similar to those reported by Saunders *et al* in 2016 using a large-angle refractometer [23].

We also performed a study into the penetration depth of the evanescent wave versus the concentration of sucrose as it is shown in figure 7. It is possible to observe that the penetration depth increases and the transmission decreases as a function of the concentration of sucrose. This means that the relationship between the transmission and the penetration depth is inversely proportional to the concentration of sucrose. Taking into account the relationship between refractive index and the penetration depth, it can be seen that an increase in the cladding refractive index due to the increase of the sucrose concentration will result in an increase in the penetration depth or portion of light travelling in sucrose.

Although the penetration depth is typically less than the wavelength, it is clear that, according to equation (4), its value rises sharply as the angle of incidence approaches the critical angle. This exponential decay of the field is more important when choosing the core and cladding material for a sensor. Therefore, the differences between the cladding (analyte) and the core should be as small as possible to have more energy for an interaction with the external medium. In fact, there are more options to improve the response of the detection technique, such as making a U-bent, increasing the sensing area, using plasmonic nanoparticles, among others.

5. Conclusion

In the present work, an optical fiber sensor reduced by chemical etching was developed to measure different concentrations of sucrose by evanescent waves. The cladding of an optical fiber was removed using HF in approximately 34 min at a rate of 3.27 μm min^{-1}. Using a reduced fiber with a diameter of 7.3 μm it was possible to determine the transmission in sucrose concentrations of up to 65%, as well as the dependence of the refractive indices for each concentration. The refractive indices obtained were 1.31–1.44 for sucrose concentrations of 0%–65%. The results obtained showed that the fibers reduced by chemical etching could be used as devices for the sensing of glucose in the blood; because glucose is a simple type of sugar that can be found in various aspects of life.

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