

Determination of thermo-optic coefficient of ethanol-water mixtures with optical fiber tip sensor

Susana Novais*, Marta S. Ferreira, João L. Pinto

I3N & Physics Department, University of Aveiro, Campus de Santiago, 3810-193 Aveiro, Portugal

ARTICLE INFO

Keywords:
Fiber optic sensors
Interferometry
Temperature
Thermo-optic coefficient

ABSTRACT

In this work, the thermo-optic coefficient (TOC) of ethanol-water mixtures, through refractive index and temperature measurements are determined using an etched optical fiber tip based on a multimode interferometer. The proposed probe is fabricated by fusion-splicing a 5.2 mm long coreless fiber section to a single mode fiber. To reduce the sensor dimensions and improve its sensitivity towards external medium variations, the fiber tip is subjected to wet chemical etching using a solution of 40% hydrofluoric acid, presenting a final diameter of 24.4 μm . The TOC of each solution is estimated and, in the case of deionized water and pure ethanol, its value is of $-1.128 \times 10^{-4} \text{ } ^\circ\text{C}^{-1}$ and $-3.117 \times 10^{-4} \text{ } ^\circ\text{C}^{-1}$, respectively.

1. Introduction

The fact that ethanol is an element miscible in non-polar and polar substances makes the ethanol a versatile solvent used in several industrial sectors such as chemical, pharmaceutical or fuel. Due to the inevitable depletion of the world's petroleum supply (in the fuel sector), there is an increasing worldwide interest in alternative, non-petroleum-based sources of energy. In order to preserve the product quality, the water-ethanol proportion must be periodically monitored and compared to standardized conditions taking into account that the ethanol production process is characterized by sequential procedures, and in several cases the water is a constituent of the final product [1].

According Nish et al., the refractometric analysis of ethanol-water mixtures is hampered because this kind of mixtures presents deviations from a linear behavior, and the existence of anomalous physical-chemical properties. During the process of the mixture of water and ethanol the entropy of the system increases differently from that expected for an ideal solution of randomly mixed molecules. Thereby, the refractive index of the ethanol-water mixture shows a non-linear dependence with the ethanol concentration, and it is expected that as the ethanol concentration rises beyond a critical value, the refractive index relation becomes a two-valued function, presenting an ambiguous region for the determination of the ethanol proportion in the mixture [2]. Nowadays, a huge variety of optical fiber sensors to determine the concentration in liquid mixtures has been suggested, since de concentration is correlated to the refractive index and temperature of the samples [3], and several methods have been used in the literature to

study and measure water and ethanol mixtures.

The measurement of the refractive index dependence with temperature, at a given wavelength in liquid solutions (thermo-optic coefficient (TOC)) has attracted a lot of attention, due to its importance in chemical and bio-chemical analysis [4]. As for water-ethanol mixtures, due to the high TOC of ethanol, the temperature fluctuations contribute to the uncertainty in the refractive index measurements [5].

Several optical fiber-based configurations have been already proposed for the TOC determination, using etched fiber Bragg gratings [6,7], long period gratings (which have also demonstrated high sensitivity to the refractive index of surrounding medium) [8,9], surface plasmon resonance effects [10], a two-mode interferometric probe based on a special fiber [4] and using a hollow core Fabry-Perot interferometer [11].

The use of coreless fiber has also been addressed for sensing applications, namely for high sensitivity refractive index [12] and temperature [13] measurements. The configurations reported are usually based on a transmission scheme, where the coreless fiber is spliced between two sections of single mode fiber [14]. The reflection scheme, where the coreless fiber tip is spliced to one section of single mode fiber, has also been proposed. Typically, a metal coating is applied to the sensor to increase its reflectivity and performance [15].

In this work, an etched coreless fiber tip is proposed for measurement of refractive index of ethanol aqueous mixtures as well as for the determination of their thermo-optic coefficient. The sensing device is previously calibrated using water-glycerin mixtures with known refractive index at the 1550 nm wavelength range. The TOC for each

* Corresponding author.

E-mail addresses: novais@ua.pt (S. Novais), marta.ferreira@ua.pt (M.S. Ferreira), jlp@ua.pt (J.L. Pinto).

solution is estimated through refractive index and temperature measurements.

2. Experimental results

The sensor head was obtained by fusion splicing a section of coreless fiber (with an initial diameter of 125 μm , supplied by Thorlabs) to standard single mode fiber (SMF), using the manual program of the splicing machine (Fujikura 62S). Afterwards, the fiber was cleaved, presenting a length of 5.2 mm. The tip was then subjected to wet chemical etching for 61 min, using a solution of 40% hydrofluoric acid. After the chemical etching procedure, the sensor presented a final length of 5.1 mm and a diameter of $\sim 24.4 \mu\text{m}$. To determine the thermo-optic coefficient (TOC) of ethanol-water mixtures, a set of 5 solutions with ethanol mass fractions ranging from 0 wt% to 100 wt% were prepared under a controlled laboratory environment. After stirring the solutions for 60 min with a magnetic stirrer (NAHITA, model n° 690/1), they were stored for 24 h, to allow their stabilization. The sensing device was inserted vertically in the 5 different solutions with aid of capillary glass tube, to ensure the sensor robustness as well as the stability of the fiber. The spectral response of this sensing structure was observed in a typical reflection scheme, as shown in Fig. 1. The experimental setup consisted on a broadband optical source, an optical spectrum analyzer (OSA Anritsu MS9740A) and the sensing head, connected by means of an optical circulator. The optical source was centered at 1570 nm, with a bandwidth of 80 nm. All readings were done with a resolution of 0.1 nm.

When the light guided through the SMF enters the coreless fiber section, several modes are excited. These modes are reflected at the coreless fiber/surrounding medium interface, and recoupled to the SMF fundamental mode. The reflected wavelength, λ_0 , can be tailored according to Eq. (1):

$$\lambda_0 = n_1 D^2 p / (2L), \quad (1)$$

where n_1 , D , L and p are the coreless fiber refractive index, the fiber diameter, the length, and the interference order number, respectively [13]. For the present work, given the sensor dimensions and the reflected wavelength in air, p was estimated to be 19. According to the literature, the self-image of the multimode interferometer occurs at multiples of $p = 4$, and lower losses are obtained. However, at other interference order numbers (no self-image), two consecutive modes with high coupling efficiency can constructively interfere, originating a spectrum that is suitable to perform measurements. This sensor revealed to be adequate to perform high sensitivity measurements and it presented a fast response and high stability in the liquid media. The thinner the coreless fiber section, the larger the evanescent field in the sensing area, which interacts with the external medium. Therefore, a large sensitivity is expected, when compared to an unetched fiber section [13].

In Fig. 2a) and b) it is possible to observe the spectra obtained at room temperature for each ethanol aqueous solution, and the

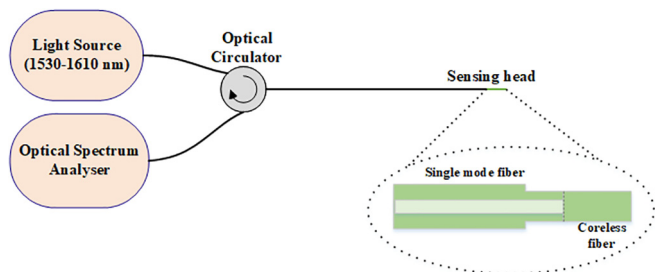


Fig. 1. Scheme of the experimental setup.

wavelength shift dependence with the mass fraction for all solutions, respectively. The sensor response to temperature and refractive index variations was determined by following the shift of the dip wavelength (located at $\sim 1551 \text{ nm}$ for pure deionized water). The decrease in the wavelength shift exhibited for 100 wt% ethanol, evidenced in Fig. 2b) is related to the decrease of the refractive index at that concentration.

The variation of mass fraction in a mixture of water and ethanol translates in a change of the medium refractive index. This parameter was determined for each solution and measured with an Abbe refractometer (KRÜSS optronic refractometer) operating at 589 nm. However, since the optical source was centered in the 1550 nm region, it was necessary to calibrate the sensor, thus estimating the refractive index of each solution in this wavelength range. This was achieved by placing the sensor in glycerin aqueous solutions that ranged from 0 wt% to 51 wt% glycerin, which were previously calibrated for the proper operation wavelength [16]. Fig. 3 presents the wavelength shift dependence with the refractive index variations. As expected, a non-linear response is observed. Nevertheless, the behavior for the refractive index range between 1.315 RIU and 1.365 RIU, is approximately linear. The ethanol-water mixtures refractive index range is expected to be located within this region [5]. The sensor presented a linear sensitivity of 627.9 nm/RIU, with a fitting correlation factor of 0.994.

Once the relationship between wavelength shift and refractive index was established, the sensor spectral response was evaluated for each water-ethanol solution. The correspondent refractive index was determined through the sensitivity obtained in the calibration procedure. Fig. 4 presents the refractive index dependence with mass fraction for the ethanol-water mixtures, for the experimental (589 nm) and calibrated (1550 nm). As can be seen from the results when the ethanol mass fraction increases, the refractive index also increases, until a mass fraction of $\sim 80\%$ [17], after this value, there is a decrease of the refractive index. It is also possible to observe that the behavior is consistent for both wavelengths.

To perform the experiments, the sensor device was inserted vertically in the ethanol-water solutions which were placed in a temperature controlled hot plate (resolution of 0.1 $^{\circ}\text{C}$) and subjected to temperature variations from 30 $^{\circ}\text{C}$ until 80 $^{\circ}\text{C}$, with steps of 5 $^{\circ}\text{C}$. Fig. 5 presents the wavelength shift with temperature, for each solution. A shift towards shorter wavelengths (blue shift) with temperature increase is observed in all cases. On the other hand, it is observed that with the increase of ethanol mass fraction, the wavelength shift becomes more pronounced, which indicates that the sensor sensitivity also increases. Notice that the response is non-linear for the solutions with a mass fraction of 0 wt%, 21 wt%, and 44 wt% ethanol. For higher concentrations, the behavior becomes linear. This is consistent with the behavior already reported in the literature [1,5,10].

To improve the TOC determination, the silica TOC and thermal expansion effects were compensated by performing temperature measurements in air, in the same wavelength range as for the liquids measurements. The sensor was placed in a thermal chamber (Model 340, Challenge Angelantoni Industry), and the readings were done with a resolution of 0.1 $^{\circ}\text{C}$. Fig. 5(a) also shows the sensor response in air (red solid circles), from which a linear sensitivity, $\Delta\lambda_1/\Delta T = k_{T1} = 12.75 \text{ pm}/^{\circ}\text{C}$ was attained. On the other hand, when the sensor is placed in the liquid, the wavelength shift can be attributed to two components: one due to the coreless thermal expansion and TOC, k_{T1} , previously determined, and the other one due to the liquids TOC. The wavelength shift can be estimated through the expression $\Delta\lambda_2 = (k_{T1} + k_{T2})\Delta T$. Using the two equations, the sensitivity due to the liquid contribution can be determined through $\Delta\lambda_2 - \Delta\lambda_1 = k_{T2}\Delta T$. Fig. 5(b) presents the calculated wavelength shift due the liquids contribution.

Table 1 summarizes the sensitivities to refractive index (k_n), and to

Download English Version:

<https://daneshyari.com/en/article/6888233>

Download Persian Version:

<https://daneshyari.com/article/6888233>

[Daneshyari.com](https://daneshyari.com)