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Refractive index measurements of ethanol-water binary liquid solutions using a graded-index fiber tip sensor

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Abstract. The refractometric analysis of ethanol-water mixtures is hampered because this type of binary solution does not present a linear behaviour. In this work it is proposed a multimode Graded-Index Fiber (GIF) tip sensor for the measurement of ethanol in binary liquid solutions of ethanol-water. The probe is fabricated by fusion-splicing a 500 µm GIF to a Single Mode Fiber (SMF) and it operates as a refractometric sensor in reflection. Samples of ethanol-water mixtures were measured at different temperatures (20°C to 60°C) to evaluate the probe's capability to detect variations in ethanol refractive index. The samples have different % (v/v) of ethanol, in a range between 0% and 100%.

1 Introduction

Ethanol is an element used as a solvent in several industries because it is miscible in non-polar and polar substances [1].

Nowadays, the laboratory analysis performed to control the quality of ethanol-based products are time consuming and difficult to apply; these are the main reasons for the need of new monitoring systems [2]. The mixture of a hydrophobic solute, such as ethanol, in an aqueous solution creates a non-common increase of entropy, associated with the formation of hydrogen bonding clusters. Because of this, the refractive index of ethanol-water mixtures has a non-linear variation with the increase of ethanol concentration. There is a critical value of ethanol in solution, from which the determination of the refractive index is very ambiguous [2].

In this work, a multimode GIF tip sensor is proposed to measure the refractive index of binary solutions of ethanol-water, for the range between 0 and 100% (v/v) of ethanol in water, at different temperatures (20ºC to 60ºC).

2 Results

2.1. Sensing probe and experimental setup

The sensing probe proposed consists of a 500 µm-length multimode GIF section spliced to a standard SMF and interrogated in reflection – Figure 1.

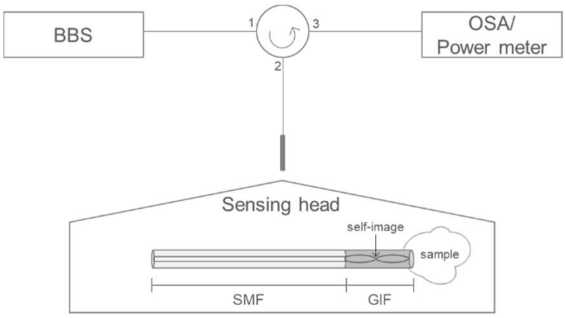

Fig. 1. Scheme of the experimental setup.

It was characterized in two ways: (1) first using an Optical Spectrum Analyzer (OSA) and then (2) an optical power meter. The OSA was used to acquire the optical spectrum response of the sensing probe during its fabrication; afterward, the power meter was used to monitor the integrated optical power of the sensor when submitted to different refractive index solutions. The experimental setup is based on an optical Broadband Source (BBS), with a central wavelength at 1550 nm and a bandwidth of 100 nm, connected to the OSA/power meter by means of an optical circulator. The sensing head operating mechanism relies on the self-imaging phenomenon. The light propagates through the incoming SMF and then it enters in a section of GIF, which self-focuses (self-image) the light beam. The end of the GIF is cleaved to ensure the Fresnel reflection. The intensity variation of the Fresnel reflection at the fiber-to-liquid sample interface allows to determine the samples

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refractive index variations. Figure 2 shows the output spectrum of the configuration proposed. It is possible to see the parabolic refractive index profile of the multimode GIF, related to the output focusing or collimating, according to the GIF length (500 µm).

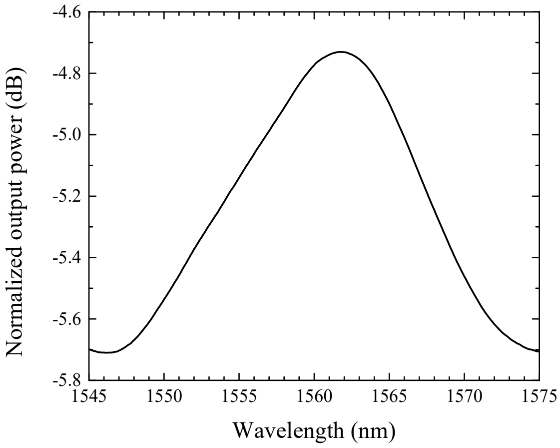

Fig. 2. Experimental spectrum response of the fiber tip sensor proposed (fiber tip is only in contact with air, at room temperature, ~20°C).

2.2 Refractive index measurements

The refractive index measurements were performed using 11 binary liquid solutions of ethanol 96%-deionized water (30 mL), for the range between 0 and 100% (v/v) of ethanol in water. The sensing head was vertically immersed in each sample and the output power was obtained through the optical power meter. The samples were measured at different temperatures (20ºC to 60ºC). For that, each sample was subjected to a water-bath.

Initially, a refractive index characterization of the ethanol-water mixtures, at room temperature (~20°C), was performed. The refractive index was measured using an Abbe refractometer. A third-order polynomial approximation of the obtained calibration was carried out. Figure 3 (a) shows the optical response obtained during the samples measurements and in Figure 3 (b) is represented the refractive index variation, according to the ethanol concentration. The refractive index was determined using the previous calibration performed. The results were fitted with a third-order polynomial function, with coefficients of determination equal to or higher than 0.990. To evaluate the influence of temperature on the sensor output power response, the sensing head was placed in an oven. During the heating (~25°C to 50°C), the sensing head was only in contact with air and the output power response was obtained using an optical power meter. The output power response remains approximately constant (~ 0.8 µW), which proves that the sensor response is only related to the refractive index variations.

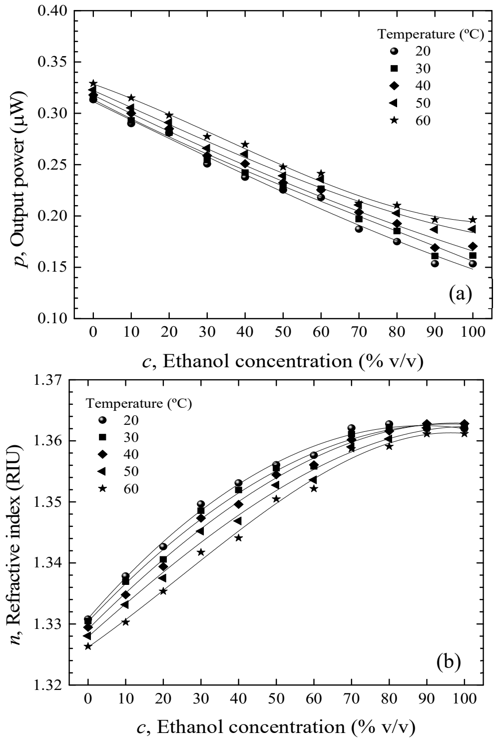

Fig. 3. Refractive index dependence with ethanol concentration and temperature variations. (a) fiber tip output power response, (b) ethanol-water samples refractive index.

3 Conclusions

The proposed sensor allows for the performance of refractive index measurements of ethanol-water mixtures, even above the critical value of 80 % v/v ethanol and is not influenced by external factors such as temperature. A non-linear dependence of refractive index with the increase of ethanol concentration, associated with the ethanol-water clusters formation, was obtained. Also, the temperature increase caused a reduction in the refractive index, associated with the reduction of intermolecular interactions' intensity and an increase in the speed of light in the propagation medium. This proves the potential of GIF tip sensors to perform in-line measurements of refractive index.

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