

# REAL-TIME OPTICAL FIBER SENSOR FOR HYDRO-ALCOHOLIC SOLUTIONS

Eric Fujiwara<sup>1</sup>, Rafael T. Takeishi<sup>1</sup>, Eduardo Ono<sup>1</sup>, J. S. dos Santos<sup>1</sup>, Aya Hase<sup>1</sup>, Carlos K. Suzuki<sup>1</sup>

<sup>1</sup> UNICAMP-The State University of Campinas, Faculty of Mechanical & Mechatronics Engineering, Laboratory of Photonic Materials & Devices, 13083-970 Campinas, Sao Paulo, Brazil

## ABSTRACT

The real-time determination of hydro-alcoholic concentration in alcohol distillation plants is a primordial condition in order to preserve the quality and reduce production losses. Presented research proposes a Fresnel reflectometric optical fiber sensor for the determination of hydro-alcoholic concentration in liquids. The intensity of reflected light and the sample temperature are continuously measured and processed by a fast algorithm. Calibration curves were prepared for a range from 0 to 100% of water in alcohol (ethanol) and adjusted to second order polynomials. According to functional tests, sensor provides maximal error of 1.3 % for concentration values and proportionates practically real-time analysis.

**Keywords:** Optical fiber sensor, ethanol production, Fresnel reflectometry

## 1. INTRODUCTION

Since the introduction of sugar cane-based ethanol as an alternative fuel for automotive engines in Brazil, the production scale in sugar agro industry has increased substantially. Today, Brazil presents the largest worldwide production of sugar cane-based ethanol, for direct use in converted automotive engines (aqueous ethanol) or to be added to gasoline as a fuel enhancer (anhydrous ethanol) [1]. The alcohol production process in distillation plants is characterized by sequential procedures, starting from the fermentation of sugar cane followed by continuous distillation stages. During the accomplishment of each stage, the hydro-alcoholic concentration of product must be periodically monitored and compared to the standardized conditions, in order to keep the process control and preserve the quality of the alcohol [2]. However, laboratorial techniques for the determination of concentration, such as the chromatography [3] and infrared spectroscopy [4] methods, does not allow the real-time monitoring of the product, demanding a long time of laboratorial analysis in order to achieve an accurate result. Thus, in most of the cases, the hydro-alcoholic concentration is not instantaneously available to feedback the distillation plant, delaying the whole process and causing in gigantic losses to the alcohol production [5].

Nowadays, a large variety of optical fiber sensors for the determination of concentrations in liquid mixtures was proposed, since the concentration is correlated to the refractive index and temperature of the sample [6], as can be observed on Long Period Grating fiber sensors [7], near infrared miniaturized spectroscopic sensors [8], and reflectometric sensors [9]. However, in spite of the high accuracy, most of these technologies demands the use of expensive equipments, complicated operational procedures and long time-consuming data analysis, not allowing real time results.

Concerning these problems, presented research reports the developments of an optical fiber sensor based on the Fresnel reflectometry principle [10], for the determination of hydro-alcoholic concentration in liquids. The proposed technology is constituted by no expensive optoelectronic components and uses a fast algorithm to the measured data interpretation, allowing a practically real-time analysis of water-ethanol mixtures.

## 2. METHODOLOGY

The optoelectronic system is provided by closed loop controlled laser sources (1310 nm and 1550 nm), fiber couplers and InGaAs photodetectors (Figure 1). The extremity of a single-mode fiber for telecommunication ( $n_D \approx 1.46$ ) actuates

as the sensing probe and is immersed in the sample, allowing remote measurements. The lightwave emitted by the laser is propagated from the source to the sensing probe. Since the fiber tip is immersed in the liquid fuel, according to the Fresnel principle, part of the propagated light is reflected on the fiber-fuel interface, in such manner that the intensity of returned light is function of refractive indexes of the fiber and the liquid mixture [11]. Thus, the reflected amount is converted to electrical signal by the photodetector, and then evaluated by a data acquisition and processing unit. In order to correct the variations on the refractive index of the fuels caused by temperature changes, an additional temperature measuring system was implemented, by placing a thermistor directly on the liquid. The detected temperature signal is also delivered to the data processing unit, allowing the instantaneous correlation between the variables.

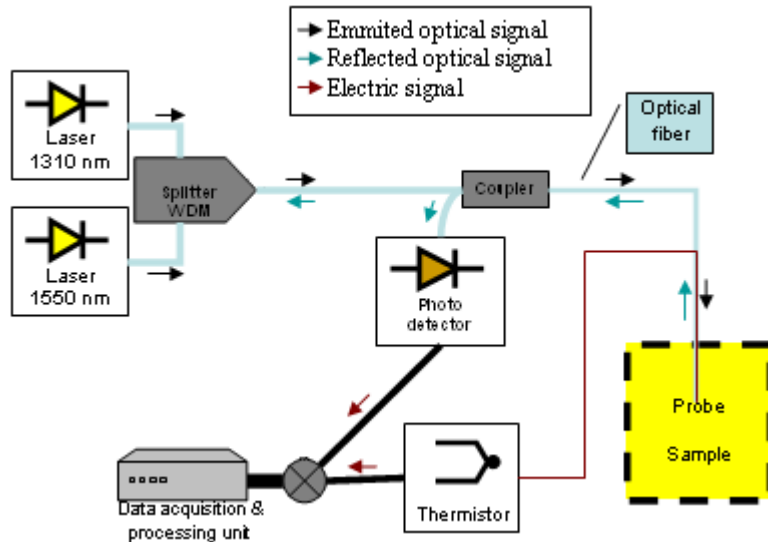


Figure 1. Schematic of the optical fiber sensor.

For the determination of calibration curves, several samples of water-alcohol solutions were prepared by using anhydrous ethanol and distilled water, covering a range from 0 % to 100 % of water in hydro-alcoholic concentration, with an increment step of 1 %. The measurements were conducted by using a sampling rate of 1000 hertz and performed during 100 seconds for both wavelengths of laser sources. The solutions were also submitted to temperatures from 10 °C to 50 °C during the measurements, in order to generate calibration functions for different operational conditions. All the collected data were post processed and adjusted by curve fitting. The study of fitting parameters revealed that the second order polynomial provides a good balance between precision and computational processing time. The calibration functions are expressed as

$$I_{1310} = f_{1310}(T) * C_{1310}^2 + g_{1310}(T) * C_{1310} + h_{1310}(T) \tag{1}$$

$$I_{1550} = f_{1550}(T) * C_{1550}^2 + g_{1550}(T) * C_{1550} + h_{1550}(T) \tag{2}$$

where  $I$  is the reflected intensity for each wavelength,  $C$  is the water concentration in hydro-alcoholic solutions,  $T$  is the temperature and  $f$ ,  $g$  and  $h$  are coefficients defined by third order polynomial functions of the temperature.

Considering an input array of measured reflected intensities and temperatures, the corresponding hydro-alcoholic concentration can be determined by finding roots for the calibration functions. It is observed that each function outputs two different concentration values, so the correct value can be determined by verifying the coherence of each result. For example, values out of the range from 0% to 100% are automatically discarded, as well as complex numbers. In these cases, the analyzed liquid might not be a hydro-alcoholic solution, or it probably occurs an operational error during the measurements, because the input intensity value does not fit to the calibration functions. On the other hand, in case of valid values, the correct concentration is determined by the root value that best matches for both wavelengths. Moreover, in order to avoid fluctuations on the reflected intensity values, which could lead to erroneous concentrations, the optical

fiber sensor performs the continuous measurement and processing of input data and the correction of the output value by statistics.

The accuracy of presented methodology was evaluated by calculating the difference between the nominal hydro-alcoholic concentration of the sample and the value output by the system, based on the analysis of different hydro-alcoholic solutions, by using the same sampling rate but carrying out the measurements during 10 seconds, in an environment with controlled temperature.

### 3. RESULTS

The calibration curves for 1310 nm and 1550 nm are illustrated in Figure 2. As expected, it was observed a non-linear behavior of the functions, as observed on theoretical curves of refractive index for water-ethanol solutions [12]. In case of the sample temperature, it can be concluded that the effect of this variable is very significant to the sensor response, since the reflected intensity increases for higher temperature values. Comparing the results for each wavelength, it was observed that the concentration value that corresponds to the minimum intensity is different for 1310 nm and 1550 nm. This effect can be attributed to the influence of the wavelength on the refractive index. Moreover, due to the higher responsivity of photodetector for 1550 nm, the reflected intensities for this wavelength were higher than the measured one for 1330 nm. In spite of the small fluctuations on the data acquired from the photodiodes, the collected data fitted successfully to the second order polynomials, granting the validity of calibration curves.

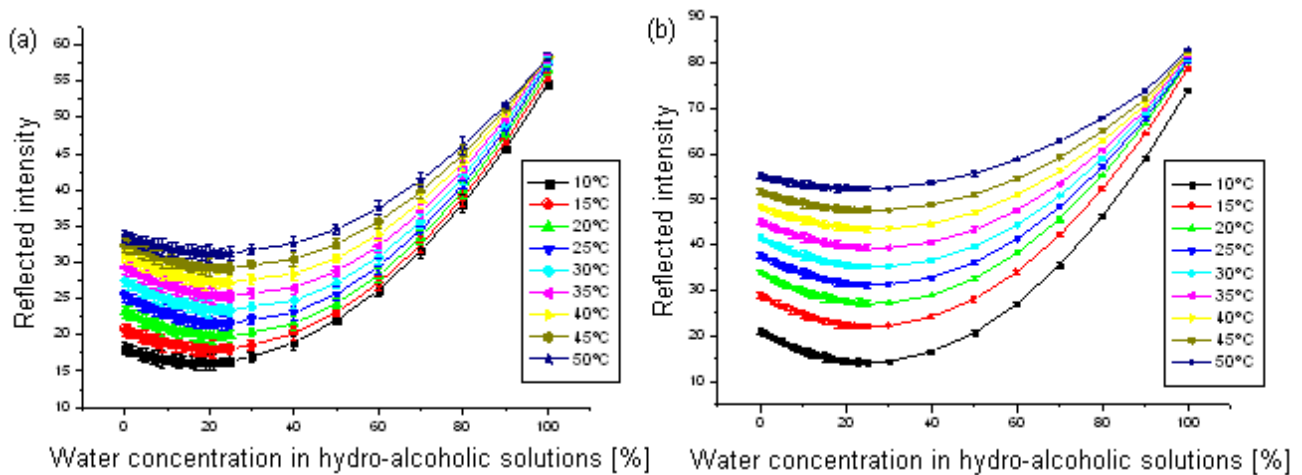


Figure 2. Calibration curves of reflected intensity response for (a) 1310 nm and (b) 1550 nm.

The calibration functions were loaded on the processing unit and applied to the optical fiber sensor. The functional test revealed that the system accuracy varies according to the concentration range of measured solution and the sample temperature. The highest errors occurred for hydro-alcoholic concentrations correspondent to intensity values near to the lowest reflected intensity, because of the perceptible decreasing on the curves inclinations. The same behavior can be observed for higher temperature values, since the increase of this parameter also reduces the inclination for whole reflected intensity curve. Moreover, the velocity of sensor response is also affected by the operational conditions. For some cases, the algorithm rapidly converged to the correct concentration value, as for other cases, the output oscillated around values near to the nominal concentration. However, in all cases, the sensor response matched to the coherent value before 4 seconds, providing practically real-time measurements. Variations of  $\pm 1.3\%$  in hydro-alcoholic concentration were observed in the concentration range from 10% to 30% and  $\pm 0.7\%$  out of this region. Otherwise, the accuracy of the sensor can be enhanced by increasing the number of acquired samples.

### 4. CONCLUSIONS

The presented methodology for the determination of concentration of hydro-alcoholic mixtures proportionate

accurate results, allowing the real-time measurement of samples. The calibration functions provided a maximal error of 1.3 % for the concentration values, but this result can be enhanced by improving the calibration functions by using smaller increments of concentration, for example. Another way to increase the sensor sensitivity would consist on replacing the ordinary optical fiber ( $n_D \approx 1.46$ ) by a fiber with higher refractive index ( $n_D \geq 1.5$ ), since according to the Fresnel principle, the reflected intensity is function of the fiber index, besides the liquid mixture. Therefore, increasing the difference of refractive indexes results on a higher intensity value, improving the optical system output. The optical fiber sensor presents an attractive alternative to be implemented on alcohol distillation plants, providing real-time response to the control of production, and eliminating most of the losses on the industrial process.

## 5. ACKNOWLEDGEMENTS

The authors would like to acknowledge the financial support of FAPESP, CAPES, CNPq, and FINEP.

## REFERENCES

- <sup>1</sup> B. Kamm, P.R. Gruber, M. Kamm, *Biorefineries - Industrial Processes and Products: Status Quo and Future Directions Vol. 1*, 964 p., Wiley-VCH, Weinheim - Germany, 2006.
- <sup>2</sup> E. Gusken, R.M. Salgado, C.E.V. Rossel, T. Ohishi, C.K. Suzuki, "Hybrid optical fiber sensor and artificial neural networks system for bioethanol quality control and productivity enhancement". *In: 19th International Conference on Optical Fibre Sensors*. Perth, 2008.
- <sup>3</sup> P.R. Prasad, K.S.R. Rao, K. Bhuvaneswari, N. Praveena, Y.V.V. Srikanth, "Determination of ethanol in blend petrol by gas chromatography and Fourier transform infrared spectroscopy", *Energy Sources, Part A: Recovery, Utilization, and Environmental Effects*, 2008.
- <sup>4</sup> T. Yano, T. Aimi, Y. Nakano, M. Tamai. "Prediction of the concentrations of ethanol and acetic acid in the culture broth of a rice vinegar fermentation using near-infrared spectroscopy", *J. Ferm. Bioeng.*, **84**, n. 5, pp. 461-465, 1997.
- <sup>5</sup> C.A. Cardona, O.J. Sanches, "Fuel ethanol production: Process design trends and integration opportunities", *Bioresource Technology*, **98**, pp. 2415-2457, 2007.
- <sup>6</sup> C.B. Kim, C.B. Su, "Measurement of the refractive index of liquids at 1.3 and 1.5 microns using a fibre optic Fresnel ratiometer", *Meas. Sci. Technol.*, **15**, pp. 1683-1686, 2004.
- <sup>7</sup> B. Lee, "Review of present status of optical fiber sensors", *Opt. Fiber Technol.*, **71**, n. 10, pp. 3864-3868, (2000).
- <sup>8</sup> S. Cho, H. Chung, Y.A. Woo, H.J. Kim, "Determination of water content in ethanol by miniaturized near-infrared (NIR) systems", *Bull. Korean. Chem. Soc.*, **26**, n. 1, pp. 115-118, (2005).
- <sup>9</sup> D. King, W.B. Lyons, C. Flanagan, E. Lewis, "An optical-fiber sensor for use in water systems utilizing digital signal processing techniques and artificial neural network pattern recognition", *IEEE Sens. J.*, **4**, n. 1, pp. 21-27, 2004.
- <sup>10</sup> C.K. Suzuki, E. Gusken, A.C. Mercado, E. Fujiwara, E. Ono, "Fiber Optics Sensing System For Liquid Fuels", OMPI Patent, Prot. PCT/BR2008/000231, 2007.
- <sup>11</sup> R.D. Meyer, G.L. Eesley, "Optical fiber refractometer", *Rev. Sci. Instrum.*, **58**, n. 11, pp. 2047-2048, 1987.
- <sup>12</sup> T.A. Scott Jr., "Refractive index of ethanol-water mixtures and density and refractive index of ethanol-water-ethyl ether mixtures", *J. Phys. Chem.*, **50**, n. 5, pp. 406-412, 1946.