

Real-time optical fibre sensor for hydro-alcoholic solutions

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Abstract

The fast determination of ethanol–water concentration in alcohol distillation plants is a primordial requirement to preserve the quality and reduce production losses. The present research proposes an optical fibre sensor for the measurement of hydro-alcoholic concentration in liquids based on the Fresnel reflection principle. The reflection intensities of ethanol samples with 0–100% of water content were measured at different temperatures for 1310 nm and 1550 nm wavelengths. Calibration curves were prepared by fitting the experimental data and implemented in a computer algorithm. According to the functional tests, the sensor is capable of identifying samples with less than 1% error on concentration and providing practically real-time analysis.

Keywords: optical fibre sensor, ethanol production, Fresnel reflectometry

(Some figures in this article are in colour only in the electronic version)

1. Introduction

The sugar–alcohol agro industry in Brazil has been developing very fast since the beginning of the use of bioethanol as an alternative fuel for automotive engines. Today, Brazil has the largest worldwide production of sugarcane-based ethanol, for direct use in flex fuel automotive engines (hydrous ethanol fuel) or blending ethanol with gasoline as a fuel enhancer (anhydrous ethanol fuel) [1]. The alcohol production process is characterized by sequential procedures, starting from the extraction of sugar juice by sugarcane milling. After being filtered and sterilized, the clarified juice is added to yeast for fermentation. The gas produced on fermentation is sent to a scrubber in order to reduce the ethanol losses. The wine and the recovered ethanol obtained from the above steps are sent to the distillation column, resulting in hydrated ethanol. Afterwards, the hydrated ethanol is added to the molecular sieves, resulting in anhydrous ethanol. During the accomplishment of each stage, the hydro-alcoholic concentration of the product must be periodically monitored and compared under standardized conditions, in order to control the process and preserve the alcohol quality [2]. Currently, almost all ethanol industries use laboratorial techniques for the determination of concentration, such as chromatography [3] and infrared spectroscopy [4]

methods. However, these techniques do not allow the real-time monitoring of the product, demanding a long laboratorial analysis in order to achieve an accurate result. Thus, in most cases, the hydro-alcoholic concentration is not instantaneously available to feedback to the distillation plant, delaying the whole process and causing considerable losses to the alcohol production [5].

Nowadays, a large variety of optical fibre sensors to determine the concentration in liquid mixtures has been proposed since the concentration is correlated to the refractive index and temperature of the sample [6], as can be observed by long period grating fibre sensors [7], near-infrared miniaturized spectroscopic sensors [8] and reflectometric sensors [9]. In contrast to other methodologies, the use of fibre sensors presents many advantages, such as remote access, non-destructive measurements and immunity to electromagnetic interference [10]. Moreover, in the case of ethanol plants, optical fibres have resistance against the harmful environment and present no explosion risk. However, in spite of the high accuracy, most of these technologies demand the use of expensive equipment, complicated operational procedures and long time-consuming data analysis, and in general, do not allow real-time results.

Concerning these problems, the present research reports the development of an optical fibre sensor based on the Fresnel

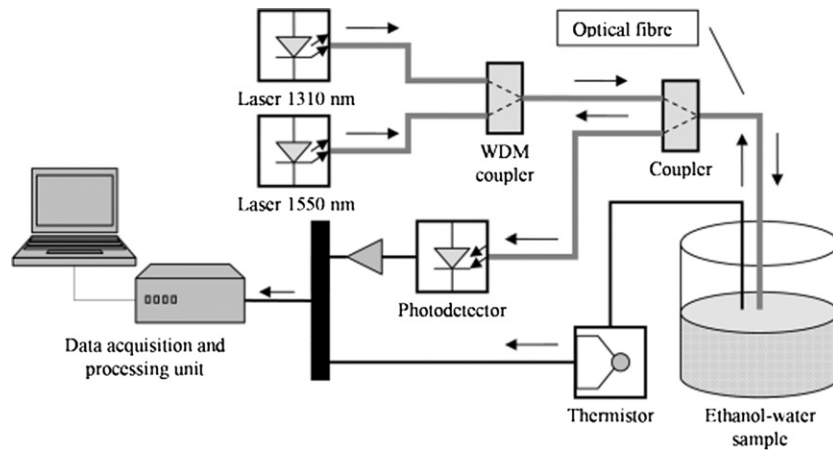


Figure 1. Schematic of the optical fibre sensor.

reflectometry principle [11], for the determination of hydro-alcoholic concentration in liquids. The proposed technology is constituted by non-expensive optoelectronic components and uses a fast algorithm to interpret the measured data, allowing a practically real-time analysis of water–ethanol mixtures and can be applied to the distillation (92.5–94% v/v of ethanol) and scrub recovering ($\sim 5.3\%$ v/v) stages in the ethanol plants.

2. Operating principle

2.1. System design

The optical fibre sensor is provided by two laser sources (1310 nm and 1550 nm), fibre couplers and a photodetector (figure 1). The extremity of a single-mode fibre ($n_D \approx 1.46$) actuates as the sensor head and it is immersed into the liquid sample. Part of the propagated light is reflected by the fibre–liquid interface, in such a manner that the intensity of returned light is a function of refractive indexes of the fibre and the liquid mixture [12]. Thus, the reflected intensity is converted to an electrical signal, and then evaluated by a data acquisition and processing unit. In order to correct the variations of the refractive index caused by temperature changes, the sample temperature is constantly monitored by a thermistor, and the data processing module correlates the reflected intensity to the temperature.

2.2. Measurement of ethanol–water concentration

According to the Fresnel principle [13], the intensity of the reflected light I is given by

$$I = K \left(\frac{n_f - n_l}{n_f + n_l} \right)^2, \quad (1)$$

where n_f is the effective refractive index of the fibre, n_l is the refractive index of the liquid and K is a hardware constant that depends on parameters such as the laser power, the electronic gain, the coupling ratio and the fibre connections. In the case of an ethanol–water mixture, n_l is defined as a function of its concentration C , temperature T and the wavelength λ [14]. Therefore, the study of process variables can be simplified to a

correlation of I , C , T and λ parameter values. In order to confer a model to the calibration curves of the sensor, the refractive index data for different ethanol–water concentrations available in the literature [15] were applied to the Fresnel equation (1). The study of such parameters revealed that the fifth-order polynomial function is well suited to fit the reflected intensity curves. The calibration curves that correlate the reflected intensity I to the concentration C are expressed by

$$I = \sum_{i=0}^5 a_i(T, \lambda) C^i \quad (2)$$

where a_i is the i th coefficient of the polynomial, calculated as a function of the temperature T and the wavelength λ .

The ethanol–water concentration of an unidentified sample can be determined by inputting I , λ and T values and solving (2) for C , considering only the solutions obtained from $C = 0$ –100%. However, due to the volume contraction of the ethanol–water mixture [16], a nonlinear correlation between n_l and C is observed, resulting in a minimum I value in the range from $C = 10\%$ to 30% of water depending on λ and T [15]. Figure 2 illustrates the nonlinear behaviour of the refractive index versus the concentration for $\lambda = 589$ nm and $T = 25$ °C, as well as the effect on the reflection intensity calculated from equation (1), using $n_f = 1.46$ and $K = 1$. At the volume contraction range, the volume decreases while the density of the liquid increases, causing the increment of the refractive index. In this case, two different concentration values from 0% to 30% are correlated to the same reflection intensity, causing an ambiguity on the sensor response. This problem was solved by measuring I for two different wavelengths since the C value that corresponds to the minimum reflection is shifted according to λ . Consequently, the algorithm should return a pair of possible solutions for each λ , and the correct concentration is determined by identifying the unique value that is the same in both pairs.

3. Experimental setup

Samples of ethanol–water were prepared mixing pure alcohol (anhydrous alcohol) and distilled water at 22 °C. The volume

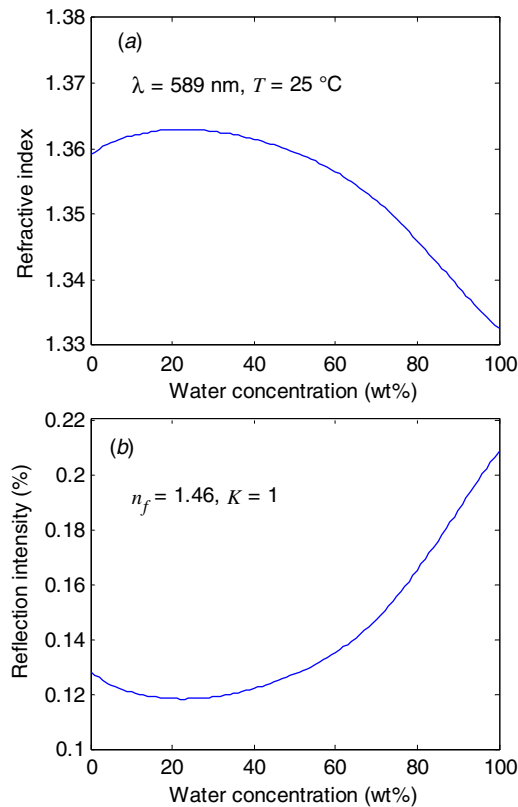


Figure 2. Effect of the volume contraction on the ethanol–water mixture on the (a) refractive index [15] and (b) reflection intensity, calculated from equation (1).

concentration of water was varied from 0% to 26% with 2% increment in order to improve the analyses for concentrations near to the volume contraction range, and from 30% to 100% with 10% increment. The reflection intensities were measured for both wavelengths ($\lambda = 1310$ nm and 1550 nm), while the solutions were maintained at different temperatures ($T = 10$ °C, 22 °C, 30 °C and 40 °C). The experiments were carried out by using a sampling rate of 1 kHz and acquiring 50 000 values for each combination of C , T and λ . Afterwards, the collected data were fitted in order to obtain the calibration curves.

The algorithm for the determination of ethanol–water concentration was implemented based on the fitting results. In order to verify the measurement error, the algorithm was tested for some concentrations using another data set, but under the same experimental conditions as the previous ones. The error was evaluated by computing the absolute difference between the nominal and measured concentrations.

4. Results

The measured reflection intensities and the calibration curves obtained by fitting the experimental data are shown in figure 3. The intensities were normalized and are expressed in percentages. Each point corresponds to a mean of 50 000 acquisitions, and the observed deviation was <1.95% for 1310 nm and <1.14% for 1550 nm. Fluctuations in the intensity values were noticed during the experiments,

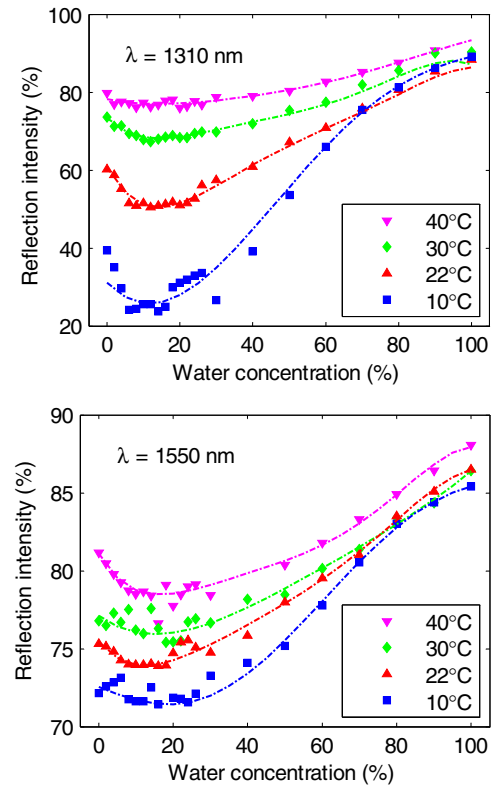


Figure 3. Calibration curves of intensity versus water concentration for 1310 nm and 1550 nm measured at different temperatures.

and later it was observed that most of these fluctuations occurred due to the influence of external environment, causing variations in the signal generated by the laser sources since the sensor electronics were not shielded. For the $\lambda = 1550$ nm and $T = 30$ °C condition, interference was caused by the operation of other equipment present in the laboratory, but this problem could be avoided in other experiments. As expected, the volume contraction behaviour of the mixture resulted in a nonlinear correlation between intensity and water concentration. In general, the intensities increase according to the increment of the liquid temperature. Moreover, the temperature influence is particularly less significant for solutions with higher water content because the thermo-optical coefficient of ethanol is higher than that of the water [17]. The temperature changes also affect the concentration related to the minimum reflection, but the values do not coincide for different wavelengths.

The results for the functional test of the algorithm at 22 °C are shown in figure 4. The water concentrations were calculated after a cumulative mean of 50 000 intensity values. The final concentration was determined as the mean values that were the same for both wavelengths. The methodology of using two wavelengths was effectively implemented since the sensor was able to retrieve the correct sample concentration even for the ambiguity range of volume contraction. An average error of 1.31% was observed, but for some concentration ranges the methodology can achieve an absolute error of less than 1%, for example, from 0% to 6% of water content. Specifically, this range corresponds

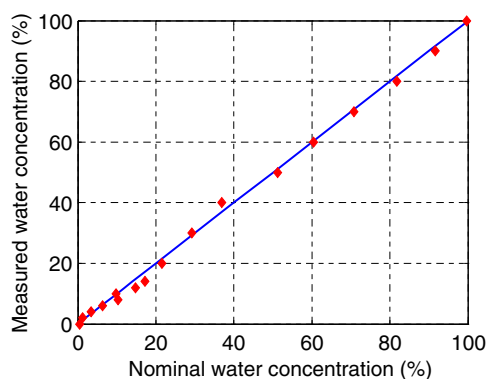


Figure 4. Plot of functional test results at 22 °C.

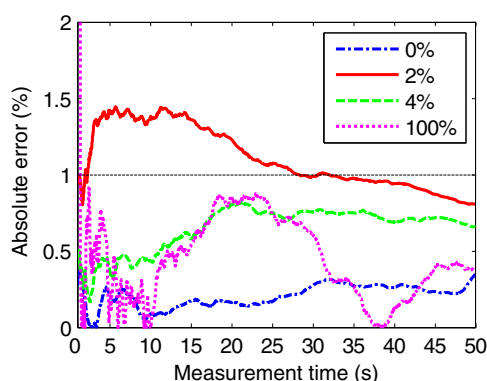


Figure 5. Convergence of the measurement error at 22 °C for ethanol samples with different water content.

Table 1. Temperature effect on the measurement error.

Nominal water concentration (%)	Absolute error at 10 °C (%)	Absolute error at 30 °C (%)	Absolute error at 40 °C (%)
0	3.00	0.98	0.11
10	1.79	2.09	0.49
20	6.87	1.24	3.06
30	1.91	1.34	7.06
40	1.01	0.99	0.66
50	1.69	0.46	2.43
60	0.50	0.83	1.13
70	0.33	1.49	1.30
80	0.19	3.24	0.18
90	0.10	1.35	2.29
100	0.10	0.23	0.15

to the operational hydro-alcoholic concentration in ethanol distillation plants in Brazil, during the production of anhydrous and hydrous ethanol fuels [2].

The comparison of the sensor response at different temperatures is summarized in table 1. The compensation of the thermo-optical effect was successfully accomplished. Particularly, a correlation between the measurement error and the increment of liquid temperature was not observed.

The correlation between the measurement time and the sensor response was also studied. Figure 5 shows the convergence of the sensor response for some mixtures measured at 22 °C. Considering a 1 kHz sampling rate, it

was possible to obtain an absolute error of less than 1% after 30 s. However, for samples with lower water content, as occurs in the distillation plants, the measurement error practically converges to less than 1.5% after 5 s, which leads to a practically real-time sensor operation, in spite of a small loss in precision.

5. Conclusion

The presented methodology proportionates the determination of ethanol–water concentrations with relatively low measurement errors, even in the range of volume contraction. The sensor operation is sensitive to the temperature variations, but the thermal effect can be compensated on the calibration. The methodology is also capable of retrieving the concentration with acceptable error after 5 s and presents a good resolution for lower water content, which is suitable for the automation of alcohol distillation plants, removing the monitoring delay of production and eliminating losses in the industrial process. This technology can be effective for online quality control and for improving the ethanol productivity.

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