Measurement of sucrose and ethanol concentrations in process streams and effluents of sugarcane bioethanol industry by optical fiber sensor

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ABSTRACT

The measurement of process streams and effluents from sugar-ethanol industry by using optical fiber sensor based on Fresnel reflection principle is reported. Firstly, binary sucrose-water and ethanol-water solutions were measured in order to determine the calibration curves. Secondly, the co-products from various processing stages were analyzed in order to identify the sucrose or ethanol concentration. The absolute error was calculated by comparison between the nominal concentration values obtained by plant laboratory analysis and the sensor response, yielding errors ≤ 5 wt% and ≤ 5 vol% for sucrose and ethanol content, respectively. The fiber sensor provided reliable results even for samples with more complex compositions than pure sucrose or ethanol solutions, with perspectives of application on the several stages of the plant facility.

Keywords: optical fiber sensor, Fresnel reflectometry, sugarcane bioethanol, process monitoring

1. INTRODUCTION

The great majority of current sugarcane-based sugar and ethanol production plants (mainly in Brazil) rely on the "1st generation technology", which accomplished of a variety of processes, such as sugarcane milling, juice clarification, evaporation, sucrose extraction, fermentation, wine centrifugation, distillation and ethanol dehydration¹. However, several losses on the sucrose and ethanol contents still occur during the different processing stages², making indispensable the development of more efficient measurement techniques for the improvement of the process control and to preserve the quality of final products. In this sense, optical fiber sensors present an interesting technology to the monitoring of the industrial process, offering remote sensing, with low disturbance and no explosion risks. Particularly, reflectometry-based fiber sensors have been demonstrating high sensitivity and real-time response on the analysis of sugar and alcohol solutions, with a non-expensive and simple implementation³⁻⁶. In this research, the measurement of process streams and effluents (co-products) from sugar-ethanol industry by using a Fresnel-based optical fiber sensor is reported. The sensor was calibrated according to the sucrose-water or ethanol-water binary mixtures and then the concentration of the co-products from the different stages of the plant was determined by measuring the intensity of reflected light in the fiber-liquid interface.

2. METHODOLOGY

2.1. Sensor design

The optical fiber sensor consists of a reflectometer for measurement of liquid mixtures, illustrated in Figure 1, in which the analyzed sample is identified by its refractive index. The light emitted by the laser source is propagated through a single-mode fiber. Since the fiber end face is placed directly in the liquid, part of the light is reflected for the sake of the refractive indexes difference of the fiber-sample interface. Then, the reflected wave returns to the fiber, whose intensity is measured by a photodetector. Furthermore, the laser intensity and the liquid temperature are also monitored by an additional photodiode and a thermistor, respectively, and all acquired signals are digitalized and post-processed. The correlation between the reflected intensity I_R and the sample refractive index n_s is given based on the Fresnel equation³

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$$I_{R} = K I_{0} \left(\frac{n_{f} - n_{s}}{n_{f} + n_{s}} \right)^{2},$$

$$\tag{1}$$

where I_0 is the laser source intensity, n_f is the fiber refractive index and K is a constant that depends on the fiber losses, coupling ratio and electronic gain. Considering that the sample is a binary mixture, n_s can be defined as a function of its concentration C, temperature T, and the laser wavelength λ^7 .



Figure 1. Configuration of the optical fiber sensor.

2.2. Samples preparation

Two sets of calibration curves were prepared, regarding the sucrose-water and ethanol-water binary mixtures. Samples were mixed at 22°C by using sucrose and anhydrous ethanol diluted on distillated water. The concentration of solute was varied from 0 to 50 wt% for sucrose solutions, and from 0 to 100 vol% for ethanol solutions. Subsequently, co-products from different stages of the sugar-ethanol plant¹ were analyzed. In this research, the measured samples were: clarified juice (CJ), sterilized juice (SJ), syrup (SY) for sugar production, fermentation vat content (FC), CO₂ wash water (WW) from the scrubber, clarified wine (CW), vinasse (VN), flegmass (FL) and hydrous ethanol (HE). It is observed that each of these products have a particular sucrose or ethanol concentration. Afterwards, subsets of samples were prepared by diluting the co-products with different amounts of distillated water.

2.3. Experimental

All measurements were carried out at 22°C, by using the 1550 nm laser source. Firstly, the binary sucrose-water and ethanol-water solutions were measured in order to determine the calibration curves. In particular, in the case of the ethanol-water samples, the experiments were also conduced for the 1310 nm source in order to compensate the effect of volume contraction on the refractive indices⁸. Secondly, the subsets of co-products were tested under the same measurement conditions. The I_R, I₀ and T values were acquired at a 1 MHz sampling rate. Each experimental point was obtained by calculating the mean value of 50000 acquisitions and by applying noise reduction and normalization procedures. The calibration curves (I_R versus C) were achieved by applying 3rd and 5th degree polynomial fittings onto the experimental data of sucrose and ethanol solutions, respectively. Given the calibration curves, the sucrose or ethanol concentration of the samples from the plant could be calculated by input the measured I_R values and solving the polynomial functions for C. In order to evaluate the efficacy of the optical fiber sensor, the results were compared to the nominal concentration values, obtained from analysis at plant laboratory. In this case, the sucrose content was determined by polarimetry, while the ethanol volume concentration was determined by densitometry. Both tests were performed at ~20°C. The absolute error was calculated as the difference in modulus between the nominal concentration values (from the laboratorial analysis) and the measured values.

3. RESULTS

The calibration curves for both binary solutions are displayed on Figure 2. Regarding the sucrose solutions, Figure 2 (a), these samples presented a tendency to adhere to the fiber end surface, making it necessary to clean the sensor probe after each experiment. The same occurred in case of the co-products from the sugar-ethanol plant with higher sucrose

concentration, suggesting that some attention must be paid, considering the hypothesis of installing the fiber sensor directly on the sugar-ethanol production facility. In spite of this problem, it can be observed that the sensor was capable to identify the different concentrations of sucrose on the plant samples. Concerning the calibration curves for ethanol-water binary system, Figure 2 (b), the 1550 nm laser exhibited a higher sensibility on the detection of variations for volume contents lower than \sim 30 vol%. On the other hand, the 1310 nm laser was more sensitive for concentrations higher than \sim 90 vol%. Specifically for the application on sugar-ethanol plants, since most of the products have concentrations from 0 to 20 vol%, only one laser source is necessary on the sensor system, because the volume contraction effect is not significant on this range. On the other hand, the utilization of two different wavelengths is still required in the monitoring of the HE, in order to eliminate the ambiguity on the determination of ethanol content.



Figure 2. Measurement at 22°C of (a) sucrose-water solutions for 1550 nm, and (b) ethanol-water solutions for (i) 1550 nm and (ii) 1310 nm. The vertical lines indicate the measured data of products from sugar-ethanol plant: (FC) fermentor vat content, (CJ) clarified juice, (SJ) sterilized juice, (SY) syrup, (WW) CO₂ wash water, (VN) vinasse, (FL) flegmass, (CW) clarified wine and (HE) hydrous ethanol.

As the calibration curves were defined, the sensor was tested for the subsets of samples from the plant diluted in water. Figure 3 illustrates the absolute error for each subset. The fiber sensor proportionates a good response on the determination of sucrose concentration values, with error values lower than 4 wt%. For example, the nominal values for CJ, SJ and SY were 17.5, 27.2 and 61.0 wt%, respectively, while the sensor results indicated 16.7, 24.0 and 60.3 wt%. A possible reason for the slightly lower measurement values can be attributed to the fact that the samples were subjected to environment variations for some time before measurements, which caused an inevitable natural fermentation and, consequently, a small decrease on the sucrose concentration. This effect can be probably avoided in case of in-situ measurements. Another aspect is the chemical composition, since there are some compounds other than sucrose and water on the samples, such as small concentrations of ethanol, fructose, glucose, minerals and contaminants⁹, which could affect the refractive index.

Regarding the FC, the solution was tested to sucrose and ethanol, since there was no guarantee that the sucrose content was entirely converted to ethanol in the analyzed sample. The calculated measurement errors were considerable for the FC (results from plant laboratory analysis were 2.80 wt% for sucrose and 9.88 vol% for ethanol) mostly because of the calibration curves were prepared regarding only binary solutions. In case of the FC, besides the sucrose and ethanol, other components are not negligible, such as the case of the yeast¹, that could modify the composition and the refractive index of the sample. However, in spite of the complexity of the FC composition, the presented reflectometric fiber sensor was capable to measure with tolerable precision.

The measurement of ethanol percentage for the WW and CW also resulted in tolerable absolute errors ($\leq 5 \text{ vol}\%$). The expected results for WW and CW were 2.5 and 10.2 vol%, respectively. In particular, the higher measurement error for the CW is probably attributed to the minerals and impurities contained on this sample¹.



Figure 3. Measurement error on the determination of (a) sucrose and (b) ethanol concentrations in co-products of sugar-ethanol plant diluted in water.

4. CONCLUSION

The optical fiber sensor was successfully employed on the measurement of samples collected from various processing stages of a sugarcane based sugar-ethanol plant, allowing the identification of different sucrose and ethanol concentrations. The sensor was able to detect variations on the concentration even for the analysis of samples with complex compositions (other than sucrose-water or ethanol-water mixtures), resulting in relatively low measurement errors. Even though some developments still must be accomplished before implementing the system on the plant facility, the optical fiber sensor technologies definitively presents an interesting alternative for monitoring the sugar and ethanol productions, making possible the improvement of the productivity by reducing the processing loss.

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