MULTIPLE SAMPLE FLUORESCENCE SPECTROSCOPY USING A SIDE ILLUMINATED OPTICAL FIBER

Introduction

Fluorescence spectroscopy is an important tool in the analytical laboratory. It can be used in conjunction with fluorescent dyes, sensitive to a given chemical species, to determine the dye's spectral signature and the concentration of these species in almost any medium. When these two techniques are combined with a side illuminated optical fiber, the result is a powerful, and yet low cost, device capable of *multiple point sensing* along the fiber with a single detection system.

In this application note, we focus on the determination of chloride ions in water and concrete samples, along several sensing points of an optical fiber.

Background

The first fluorescent side illuminated optical fiber sensor was reported by a group of AT&T Bell Lab researchers in 1989[1] who described a fluorescent cladding sensor sensitive to molecular oxygen. Soon afterwards, in 1990[2-7], this author started working on this field and, in the process, reduced this technology to practice while discovering and patenting several other configurations of this sensor.

Unlike axial illumination, side illumination may use several light sources along the fiber length to generate multiple signals (see Fig. 1a and 1b). This allows the probing of the optical fiber at different positions along its length using simple switching techniques while, at the same time, increasing the fiber's signal, sensitivity and its measurement redundancy.

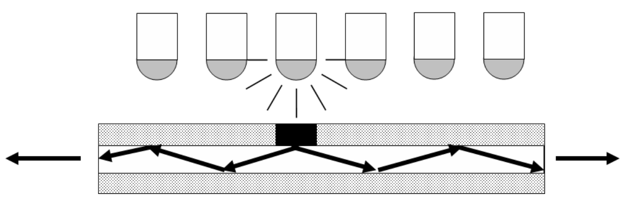

Figure 1a. Light source #3 probing a fluorescent cladding optical fiber and its associated fiber section.

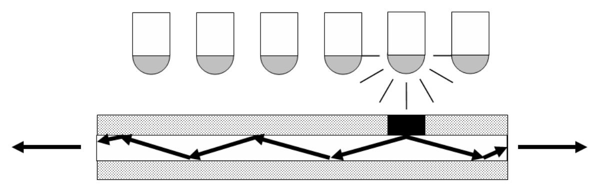

Figure 1b. Light source #3 probing a fluorescent cladding optical fiber and its associated fiber section.

In this note we describe how this technique can be used with an Ocean Optics USB-2000 mini-spectrometer, to determine the concentration of chloride ions.

Experimental Setup and Results

The setup used in this application is illustrated in Figs. 2a and 2b. It consists of

1. An optical fiber coated with a fluorescent indicator sensitive to chloride ions.
2. Three UV LEDs with peak wavelength at 370 nm (each LED illuminating its corresponding sensing point on the fiber).
3. A multichannel LED driver and
4. An Ocean Optics USB-2000 mini spectrometer.

Fig. 2b shows the fiber mounted over an LED support.

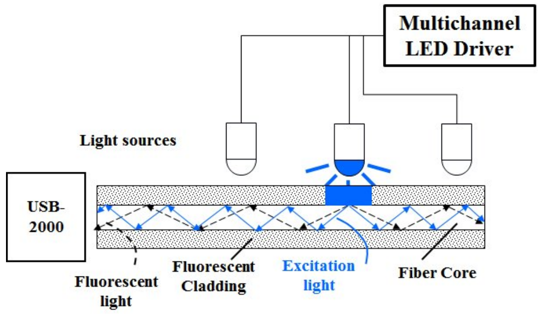

Figure 2a. Setup diagram illustrating the fluorescent coated optical fiber, the three LEDs, the multi-channel LED driver and the USB-2000 (not in scale).

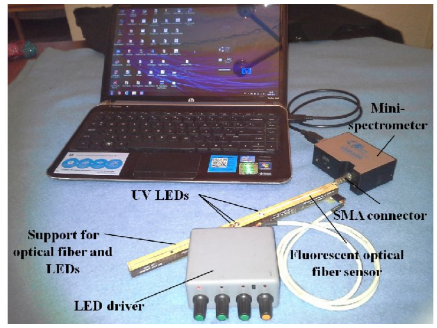

Figure 2b. Actual picture of the whole system illustrated in Fig. 2a.

The spectral distribution of the fluorescent dye of each sensing point is shown in Fig. 3a. This illustration shows the spectrum of the fiber when each point is illuminated separately by its corresponding LED. The further the illuminated spot is from the spectrometer, the lower the resulting signal. In this illustration, coating 1 is the one closest to the fiber tip that is connected to the spectrometer. The first peak from the left to the right corresponds to the spectral output of the UV LED, with peak wavelength at 370 nm. In this case, it can be seen that the UV light is not completely absorbed by the dye and still survives propagation towards the detector. The second band in Fig. 3b corresponds to the actual fluorescent signal from the fluorescent dye: this is the signal that is used to determine the concentration of chloride ions. *This result clearly illustrates one of the major advantages of this technique: that of allowing the measurement of multiple points, or samples, along the fiber with a single detection system.*

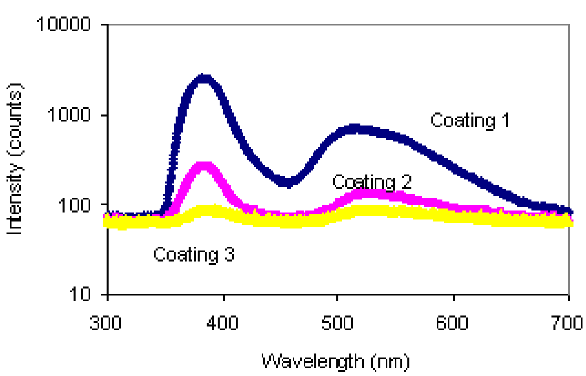

Fig. 3a. Spectral curves of the first, second and third sensing points of the fluorescent cladded optical fiber.

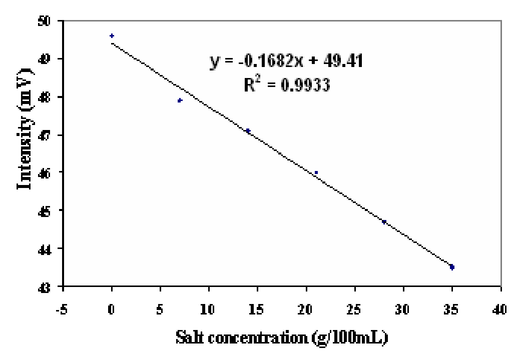

Fig. 3b. Spectral curves of the first, second and third sensing points of the fluorescent cladded optical fiber.

This setup was then used to obtain a calibration curve of the sensor in terms of chloride ion concentration (see Fig. 3b). At this stage, six sodium chloride salt solutions, of different concentrations, were prepared. A small container was then installed around the optical fiber sensing point, into which the calibrating solutions were poured while fluorescence intensity measurements were made. It can be clearly seen that the data is linear and decreases with the concentration of chloride ions, the so-called phenomenon of fluorescence quenching.

In the next step, the optical fiber sensor was embedded into a cylindrical sample of concrete (Fig. 4a), which was subjected to salt water intrusion at its top, and the fluorescence intensity was monitored during a period of 90 hours (see Fig. 4b). Notice that the fluorescent signal increases in the time interval 20*h*<*t*<50*h* corresponding to the period when clear water reaches one of the sensing points of the fiber. Conversely, the decrease in signal at $t > 50$ *h*, corresponds to the period when salt water reaches the same point.

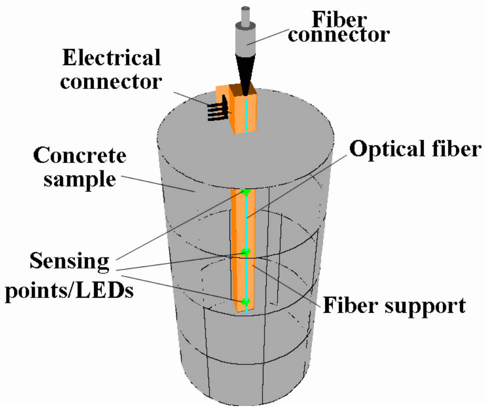

Fig. 4a. Optical fiber embedded in a cylindrical concrete sample.

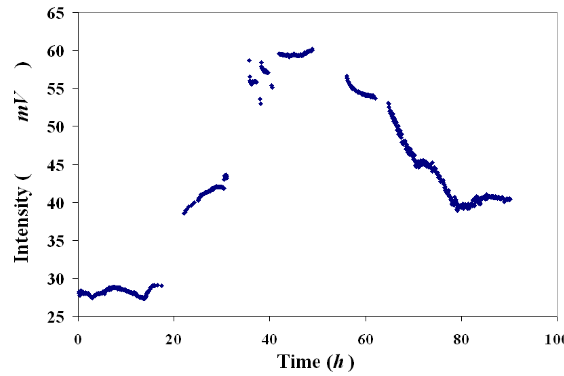

Fig. 3b. Fluorescence intensity against time for the chloride ions fiber sensor while embedded in concrete.

Conclusions

We demonstrated that multiple point fluorescence spectroscopy can be performed with a side illuminated optical fiber. This technique was then to the measurement of chloride ion concentration in water and concrete samples. This is a very inexpensive and flexible technique that can be used towards a multitude of measurements using Ocean Optics mini spectrometers as the detection system.

In future notes, we will describe other applications that use absorption, scattering, index of refraction measurements, how to perform simultaneous measurements with this device and a multiplexed spectrometric system that is capable of reading the signal from multiple cuvettes with a single spectrometer.

References

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[2] Egalon, C. O., "Injection Efficiency of Cladding Sources", Ph.D. Thesis and NASA Contractor Report 4333, November (1990).

[3] Egalon, C.O. and Rogowski, R.S., "Optical Fiber and Fluorosensors Having Improved Power Efficiency and Methods for Producing Same". US patent # 5,262,638, Filing date: 09/16/1991, Publication and Award date: 11/16/(1993).

[4] Albin, S., Bryant, . A.L., Egalon, C.O., and Rogowski, R.S. "Injection efficiency from a side-excited thin-film fluorescent cladding of a circular waveguide," *Optical Engineering*, Vol. 33, No. 4, pp. 1172-1175, April (1994).

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[6] Egalon, C.O., “Optical Fiber Sensors for the Detection of Chloride Ions in Concrete Structures”, PIPE Program Phase I grant, Fundação de Amparo à Pesquisa do Estado de São Paulo, FAPESP, 03/2003 to 12/2003.

[7] Egalon, C.O., "Improved, Reversible, Low Cost, Distributed Optical Fiber Sensor With High Spatial Resolution". International publication date: November 2nd, 2006. International Publication Number WO 2006/116590, Priority date: April 28th, 2005. This patent was issued in the following countries:

a. US Patent Number 7,473,906, January 6, 2009 and and its reissue RE43937, January 22, 2013.
b. Mexican Patent Number 275,404 issued on February 17, 2010.
c. European Patent Number EP1882178, granted December 7th, 2011 and validated in Germany, France, United Kingdom, Spain, Italy, The Netherlands, Switzerland, Liechtenstein, Greece, Portugal, Ireland, Denmark, Finland, Sweden, Cyprus and Luxembourg.
d. New Zealand Patent Number 563,641 issued on July 11th, 2011 and its divisional counterpart, number 591,014, issued on August 8th, 2011.
e. Japanese Patent Number 5,173,797, registration date January 11th, 2013.
f. Australian Patent Number 2006241097, issued on May 24, 2014.
g. This patent is pending in Canada and Brazil.