AIP Conference Proceedings

Detection of trace elements in DI water and comparison of several water solutions by using EF-FLRD chemical sensors

Cite as: AIP Conference Proceedings **1809**, 020027 (2017); https://doi.org/10.1063/1.4975442
Published Online: 24 February 2017

M. Kaya, and C. Wang

View Online Export Citation

ARTICLES YOU MAY BE INTERESTED IN

Synthesis and testing of a conducting polymeric composite material for lightning strike protection applications
AIP Conference Proceedings **1809**, 020026 (2017); https://doi.org/10.1063/1.4975441



AIP Publishing

© 2017 Author(s).

Detection of Trace Elements in DI Water and Comparison of Several Water Solutions by Using EF-FLRD Chemical Sensors

M. Kaya[1,2,*] and C. Wang[1]

[1]*Department of Physics and Astronomy, Mississippi State University, Starkville, MS 39759 USA*
[2]*Department of Physics, Eskisehir Osmangazi University, Eskisehir, 26480, TURKEY*
[*]*Corresponding author: malikkaya@ogu.edu.tr*

Abstract. Detection of trace elements in DI water and comparison of several types of water respons such as DI water, tap water, and ocean water were studied by using EF-FLRD chemical sensors. Solutions of Mg, Fe, P, and Cd elements with a concentration of 1000 μg/ml (1 mg/ml) which corresponds to 1000 ppm by weight were tested. DI water, tap water, and real ocean water were utilized as medium to observe refractive index difference of solutions. The EF sensing technique which is based on the EF scattering effect was employed for the detection of trace chemicals and recording of solution responses. Due to the refractive index differences between solutions, optical loss of the sensor was different when the sensor head was immersed into solutions of approximately 10 ml each. The differences of the refractive indices are presented by recording ringdown times when the sensor head was immersed into the solutions. Fast response (~1s), high sensitive, and reproducible EF-FLRD chemical sensors were fabricated and tested in laboratory conditions. High sensitivity of the sensors due to being capability of detecting smaller differences between measurands without any additional components or treatment makes the EF-FLRD sensors more attractive. Enhanced sensitivity of the EF-FLRD chemical sensors as a result of the multi-pass nature of the FLRD technique has potential application in detecting of trace elements in monitoring of water quality.

Keywords: Trace elements; Water testing; FLRD technique; EF scattering.
PACS: 92.20.Wx, 92.40.Bc,42.79.-e, 42.81.Dp.

INTRODUCTION

Controlling the quality of drinking water or any important liquid have an increasing attention of specialists as much as consumers all around the world. Controlling the quality is extremely important due to the intrusion of toxic chemicals from industrial discharge, chemical spills, leaking underground storage tanks, etc. [1]. Therefore, measurement of several chemical elements in water has required great significance. For determination of chemical species or elements in water, there are various techniques such as atomic absorption spectroscopy (AAP), inductively coupled plasma optical emission spectroscopy (ICP-OES), laser induced breakdown spectroscopy (LIBS), fiber optic evanescent wave spectroscopy (FEWS), laser induced fluorescence spectroscopy (LIFS), etc. [2-6]. Detection of trace elements was studied by using several aforementioned techniques but FLRD technique was utilized the first time with

Proceedings of the 6th International Advances in Applied Physics and Materials Science Congress & Exhibition
AIP Conf. Proc. 1809, 020027-1–020027-8; doi: 10.1063/1.4975442
Published by AIP Publishing. 978-0-7354-1477-8/$30.00

bare single mode fiber without any additional treatment such as coating the sensor head, or any additional optical devices such as optical spectrum analyzer.

Trace detection of chromium, nitrite, chlorine, iron, etc. elements have been studied [1, 7-10]. Kasik et al. studied on monitoring chlorine to control water quality [8]. They used a commercially available absorption transducer electrochemically immobilized onto fiber optic substrates. Their control points for detection of chlorine are around 1000 ppb which corresponds to 1 mg/ml. Kumar et al. designed and developed LED based fiber optic evanescent wave sensor for trace detection of chromium and nitrite in water [1]. They utilized multimode plastic clad silica fibers and presented LED sourced optical fibre sensor simultaneous measurement of chromium and nitrite concentrations in water ranging from 0.1 to 20 mg/dl and from 0.01 to 10 mg/dl, respectively. Xiong et al. [10] developed small-volume fiber optic evanescent-wave (EW) absorption sensor based on Griess–Ilosvay reaction and evaluated for determination of nitrite. The EW field produced on the fiber core surface sent to the surrounding medium and interacted with the azo dye, which was generated by nitrite and nitrite-sensitive reagents reaction. Nitrite concentration they used was in the range of 50 to 10000 ppb, corresponds to 0.05 μg/ml to 10 mg/ml and their detection limit was 0.2 mg/dl (3σ).

In another study, DI water, drinking water, and ocean water was tested in laboratory conditions by utilizing FLRD chemical sensor. The sensor was immersed into each water solution of 10 ml and the sensor responses to each solution in terms of ringdown time were compared. In early 90s, Goyet et al. [11] was the first time developed a fiber optic sensor for pCO_2 measurement in seawater. They first illuminated water and generated fluorescence, then detected returned fluoresced light by a single optical fiber. Thus they demonstrated an optical sensor for detection oceanic signals. Very recently, George et al. [12] studied variation of refractive index of chlorinated water by employing a microbend fiber optic sensor. In the study, the refractive index of the solution changed by releasing chlorine from water and therefore the sensor detected the change.

In the present work, detection of trace elements such as Mg, Fe, P, and Cd and refractive index difference of different types of water solutions such as DI water, tap water, and real ocean water are investigated. First, element solutions with a concentration of 1000 μg/ml (1 mg/ml) were tested by using FLRD chemical sensors. Detection of trace elements are resulted by presenting and explaining in detail in the results and discussion section. Later, responses of different types of water solutions were recorded by continuous monitoring of ringdown time. DI water, tap water, and real ocean water solutions were tested based on bulk index sensing technique [13]. Response of each solution was recorded in terms of ringdown time. Different ringdown time represented by different solution by means of different refractive index.

EXPERIMENTAL SETUP

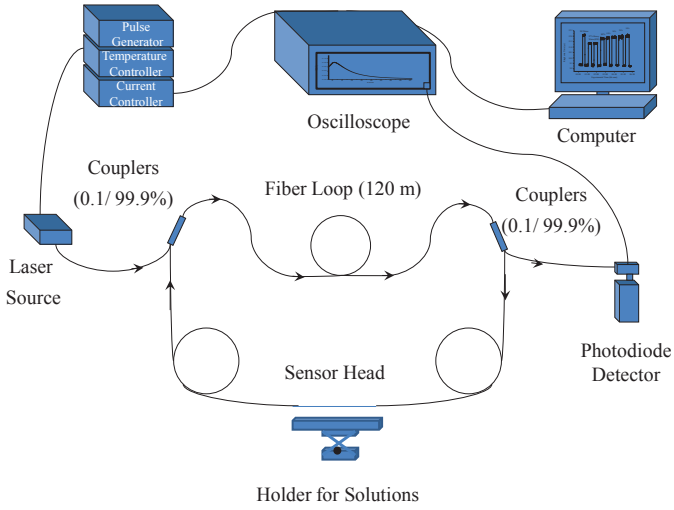


Figure 1. Schematic illustration of the EF-FLRD system.

Figure 1 shows a schematic illustration of the EF-FLRD system similar to the one described in our previous study [14]. The system consists of a continuous wave (CW) diode laser light source, a section of fused silica single mode fiber, two 2×1 fiber couplers, a photodetector, and an electronic control. The cladding and the fiber core of the single mode fiber, SMF-28e, Corning, Inc., have diameters of 125 μm and ~8.2 μm, respectively. The two identical 2×1 fiber couplers, Opneti Communications Co., were fabricated with a split ratio of 0.1:99.9 in the 2-leg end of the fiber couplers. The two 1-leg ends and the two 99.9% legs of each of the two couplers were spliced together, FITEL S321, Optical Fiber Cleaver; Ericsson 925, Fusion Splicer, to form a fiber loop. The total optical loss, including absorption loss, fiber connectors' insertion losses, and fiber couplers' losses, was estimated to be <0.45 dB. The splicing loss was estimated by the splicer as 0.02 to 0.04 dB. A narrow band, ~30 MHz, single mode laser beam from the single mode fiber of the pig-tailed distributed feedback laser diode, NEL, Model NLK1S5AAA, was coupled into the fiber loop through the 0.1% leg with an FC/APC fiber connector, and the 0.1% leg of the second coupler was coupled to the InGaAs photodetector, Thorlabs, PDA50B. The total length of the loop was 120 m. Identical fiber loops were used in this work, but after several rounds of cutting and splicing, the length of each loop and the total optical loss of each loop were slightly different. Consequently, each fiber loop gave a different ringdown time, τ_0. The absorption loss rate of the fiber was quoted to be 0.3 dB/km at 1515 nm, and the wavelength of the laser diode had a tunable range of ±1.5 nm around the central wavelength of 1515 nm. In the present work, the diode laser wavelength was tuned precisely to 1515.25 nm. The output wavelength was monitored by a wavemeter, Burleigh, WA-1500, that had an accuracy of ±0.001 nm. Water and high abundance atmospheric molecules, such as oxygen, nitrogen, and carbon dioxide, have no absorption at 1515.25 nm.

Commercially available element solutions (Sigma) were used for trace element detection. The ruler was carefully cleaned after each element solution was tested to eliminate contamination effect. Response of each solution was recorded in terms of ringdown time. Results are plotted and explained in detail in following section. Moreover, real ocean water (Petco Co.), DI water and tap water were compared by recording the sensor's responses in the solutions. Results are shown and explained in following section as well. Principle of FLRD technique is explained in detail in our previous work [14].

RESULTS and DISCUSSION

Figure 2 (a) shows the detection of two different elements in DI water solution. First, the sensor head was immersed into DI water to test sensor quality and reproducibility. When the response of the sensor in air was 6.1 μs, it was 9.8 μs in DI water. Second, the sensor head was immersed into Magnesium (Mg) elemental solution. The response of the sensors was changed to 9.45 μs in Mg solution. Then the sensor head was cleaned with DI water and immersed into Iron (Fe) elemental solution. The response of the sensor was 9.98 μs in Fe solution. Finally, the reverse process was applied and the reproducibility and repeatibility of the sensor were tested successfully. As seen from Figure 2 (a), each chemical solution including DI water solution has different refractive indexes. Therefore, the response of the sensor was different in each solution.

Figure 2 (b) presents detection of two elements in DI water solution. Initially, fabricated FLRD chemical sensor was immersed into DI water to detect sensor's response in DI water solution in terms of ringdown time. The sensor response was 11.21 μs in DI water solution. After DI water detection, the sensor head was immersed into phosphorous (P) elemental solution of 1000 μg/ml in DI water. Response of the sensor to P elemental solution in terms of ringdown time is higher than response of DI water solution. Sensor's response was 11.64 μs in P elemental solution. Response of the sensor is higher in P solution than in DI water solution because refractive indices of the solutions are different. Optical loss of the sensor is higher when the sensor was immersed into DI water, resulting lower ringdown time and lower when the sensor was immersed into P solution, resulting higher ringdown time. Then, cleaning of the sensor head in DI water solution is presented in Figure 2 (b). The sensor was cleaned to get rid of contaminated particles on the sensor head due to trace element detection of P solution. The sensor was immersed into fresh DI water solution after cleaned to show that sensor response is the same for DI water solutions. Later, Mg elemental solution was tested and recorded response of the sensor in terms of ringdown time was 10.88 μs, which is lower than responses of both DI water and P elemental solutions. The cleaned sensor head was immersed into Mg elemental solution and response of the sensor was recorded in terms of ringdown time. Differences of 0.43 μs between DI water and P solution, 0.33 μs between DI water and Mg solution, and 0.76 μs between P and Mg solutions were reported by recording sensor responses in terms of ringdown time when the sensor was immersed into each solution. The sensor head was immersed into solutions twice to show repeatability of the sensor and trace element detection was

repeated for P and Mg to show reproducibility. The sensor was carefully cleaned after each element test to make sure the sensor was protected from contamination.

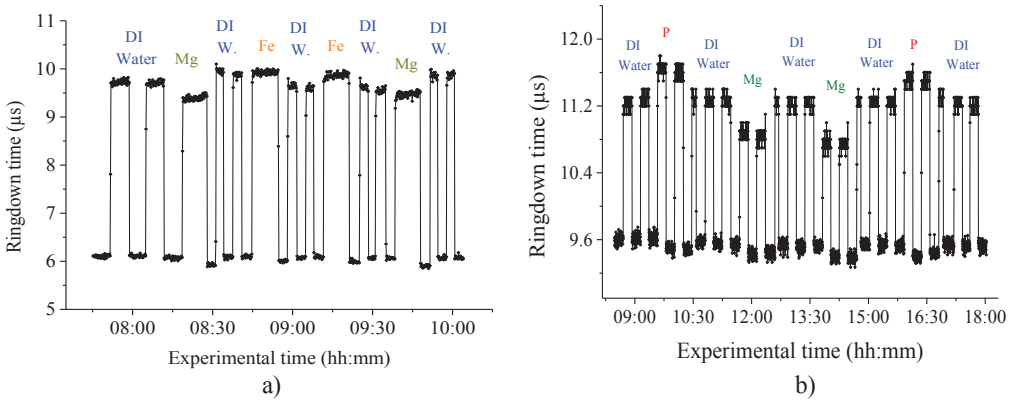


Figure 2. a) Detection of trace elements (for the solutions of Mg and Fe elements); b) Detection of trace elements (for the solutions of P and Mg elements).

Figure 3 (a) shows detection of two elements, P and Cd, in DI water solution. Initially, FLRD chemical sensor was immersed into DI water to detect DI water response of the sensor in terms of ringdown time. The sensor response was 11.25 µs in DI water solution. After DI water detection, the sensor head was immersed into phosphorous (P) elemental solution of 1000 µg/ml in DI water. Response of the sensor to P elemental solution in terms of ringdown time is higher than response of DI water solution. Sensor's response was 11.67 µs in P elemental solution. Response of the sensor is higher in P solution than in DI water solution because refractive indices of the solutions are different. Optical loss of the sensor is higher when the sensor was immersed into DI water, resulting lower ringdown time and lower when the sensor was immersed into P solution, resulting higher ringdown time. Then, cleaning of the sensor head in DI water solution is presented in Figure 3 (a). The sensor was cleaned to get rid of contaminated particles on the sensor head due to trace element detection of P solution. The sensor was immersed into fresh DI water solution after cleaned to show that sensor response is the same for DI water solutions. Later, Cd elemental solution was tested and recorded response of the sensor in terms of ringdown time was 11.04 µs, which is lower than responses of both DI water and P elemental solutions. The cleaned sensor head was immersed into Cd elemental solution and response of the sensor was recorded in terms of ringdown time. Differences of 0.42 µs between DI water and P solution, 0.22 µs between DI water and Cd solution, and 0.63 µs between P and Cd solutions were reported by recording sensor responses in terms of ringdown time when the sensor was immersed into each solution. The sensor head was immersed into solutions twice to show repeatability of the sensor and trace element detection was repeated for P and Cd to show reproducibility. The sensor was carefully cleaned after each element test to make sure the sensor was protected from contamination.

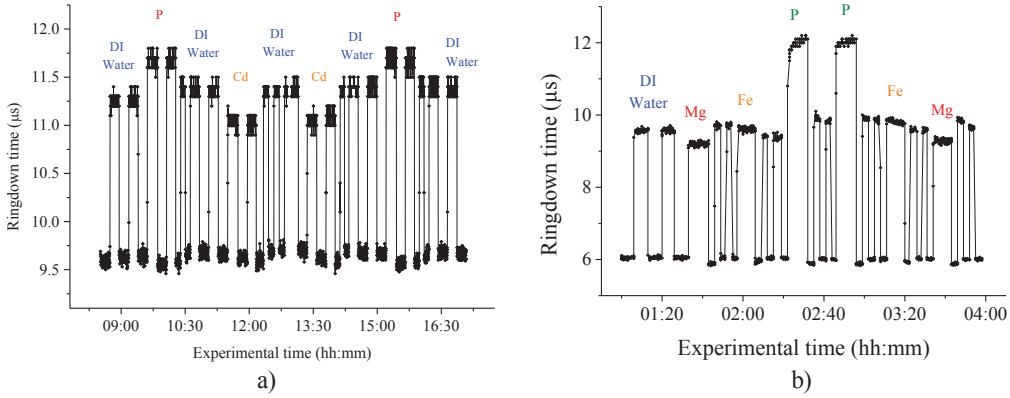


Figure 3. a) Detection of trace elements (for the solutions of P and Cd elements); b) Detection of trace elements (for the elements of Mg, Fe, and P elements).

Figure 3 (b) shows trace detection of three elements, Mg, Fe, and P, in DI water solution. Initially, FLRD chemical sensor was immersed into DI water to detect DI water response of the sensor in terms of ringdown time. The sensor response was 9.48 µs in DI water solution. After DI water solution, the sensor head was immersed into Mg elemental solution of concentration of 1000 µg/ml in DI water. Response of the sensor to Mg elemental solution in terms of ringdown time is lower than response of DI water solution. Sensor's response was 9.19 µs in Mg elemental solution. Response difference of the sensor is due to difference of refractive indices of the solutions. Optical loss of the sensor is lower when the sensor was immersed into DI water, resulting higher ringdown time and higher when the sensor was immersed into Mg solution, resulting lower ringdown time. Then, cleaning of the sensor head in DI water solution is presented in Figure 3 (b). The sensor was cleaned to get rid of contaminated particles on the sensor head due to trace element detection of Mg solution. The sensor was immersed into DI water solution twice for cleaning process. First step of cleaning was to get rid of contaminated particles, and second one was to make sure that it was perfectly clean. Second cleaning step was carried out with fresh DI water. Later, the cleaned sensor head was immersed into Fe elemental solution and response of the sensor was recorded in terms of ringdown time. The sensor was tested in Fe elemental solution and recorded response of the sensor in terms of ringdown time was 9.62 µs. Furthermore, the sensor was immersed into P elemental solution and response of the sensor was recorded as 12.00 µs in terms of ringdown time. Differences of 0.29 µs between DI water and Mg solution, 0.14 µs between DI water and Fe solution, 2.52 µs between DI water and P solution, 0.43 µs between Mg and Fe solutions, 2.81 µs between Mg and P solutions, and 2.38 µs between Fe and P were reported by recording sensor responses in terms of ringdown time when the sensor was immersed into each solution and results were compared with each ringdown time. The sensor was carefully cleaned after each element test to make sure the sensor was protected from contamination.

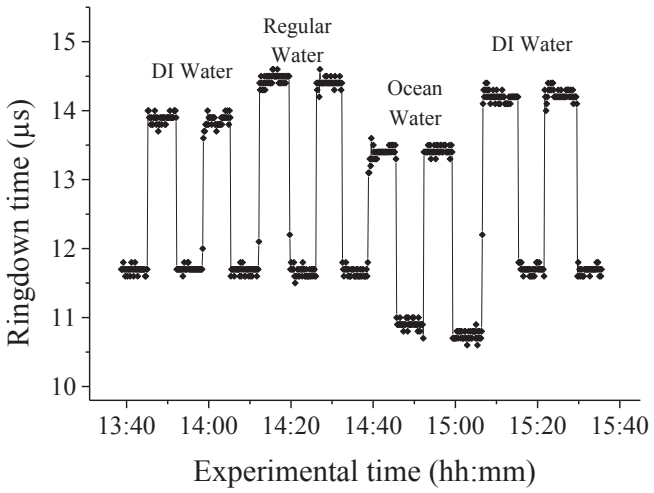


Figure 4. Detection of responses for different type water solutions.

Figure 4 present response of a FLRD chemical sensor to three different type of water which are DI water, tap water (regular water), and ocean water. Real ocean water purchased from Petco Company was used for comparison with other water samples. Difference of sensor responses can be obviously seen not only in Figure 4 but also the previous figures due to different refractive index of each solution. Trace element detection and water responses were recorded very accurately and diligently due to fast response and high sensitivity feature of the EF-FLRD chemical sensors.

In comparison with other techniques, FLRD technique has high sensitivity, near-real time fast response, and cost efficiency. For example, while detection limit in pCO_2 detection experiment [11] is in the range of 400-500 ppm, FLRD sensors' detection limit is in the range of 50-1000 ppm [14]. In microbend sensing technique [12], while more quantitative measurement is not possible, with FLRD technique, detection of optical index change up to $3.2x10^{-5}$ is possible [15]. Moreover, potential detection limit can be increased to the order of 10^{-6}. On the other hand, when FLRD system is compared to the other systems in response time, FLDR sensors have ~0.1 sec near-real time response time while microbend sensor for pCO_2 detection has 10 sec. It is obviously seen that FLRD technique has more distinguish features over its counterparts. Another comparison can be hold between cost efficiencies. When those mentioned on the introduction part and above need some extra devices such as a torch lamp and lenses in addition to fiber optic, plasma, pump, spectrometer, etc. A FLRD system needs only a single mode fiber optic and usual electronics used in each system such as oscilloscope, photodetector, computer, etc. As a result, FLRD sensors have unique merits in terms of sensitivity, response time, and cost efficiency to be used in the new age for micro-level detections such as trace element detection.

CONCLUSION

In this study, several EF-FLRD chemical sensors were developed and tested for the purpose of detection of trace elements and different kind of water samples. Traces of

Fe, Mg, Cd, and P elements of concentration of 1000 µg/ml (1 mg/ml) were detected. Comparisons of trace element detection results show that each element solution has different refractive index. Therefore, EF-FLRD chemical sensor can distinguish each solution and differences can be clearly observed by using recorded ringdown times. Furthermore, several water samples such as DI water, tap water, and ocean water were used to test EF-FLRD chemical sensor. The sensor responded to each solution differently and the response of the sensor was recorded in terms of ringdown time. Potential application of the chemical sensors showed that EF-FLRD chemical sensors have promising future for detection of trace elements in DI water and in drinking water with high sensitivity, fast response, and low cost.

REFERENCES

1. P. S. Kumar, S. T. Lee, C. P. G. Vallabhan, V. P. N. Nampoori, P. Radhakrishnan, *Opt. Commun.*, **214**, 25-30 (2002).

2. V. F. Hodge, S. L. Seidel, E. D. Goldberg, *Anal. Chem.*, **151**, 1256-1259 (1979).

3. J. C. A. de Wuilloud, R. G. Wuilloud, M. F. Silva, R. A. Olsina, L. D. Martinez, *Spectrochimica Acta Part B: Atomic Spect.*, **57**, 365-374 (2002).

4. G. Arca, A. Ciucci, V. Palleschi, S. Rastelli, E. Tognoni, *Applied Spectroscopy*, **51**, 1102-1105 (1997).

5. R. Krska, R. Keliner, U. Schiessl, M. Tacke, A. Katzir, *Appl. Phys. Lett.*, **63**, 1868-1870 (1993).

6. J. Bublitz, A. Knaack, M. Dickenhausen, M. Gratz, W. Schade, T. Baumgartl, R. Horn, *Adv. Remote Sens.*, **3**, 179-187 (1995).

7. P. S. Kumar, C. P. G. Vallabhan, V. P. N. Nampoori, V. N. S. Pillai, P. Radhakrishnan, *J. Opt. A: Pure Appl. Opt.*, **4**, 247-250 (2002).

8. I. Kasik, J. Mrazek, O. Podrazky, M. Seidl, J. Aubrecht, P. Tobiska, M. Pospisilova, V. Matejec, B. Kovacs, A. Markovics, M. Szili, *Sens. Actuat. B: Chem.*, **139**, 139-142 (2009).

9. S. T. Lee, P. S. Kumar, K. P. Unnikrishnan, V. P. N. Nampoori, V. P. N. Nampoori, C. P. G. Vallabhan, S. Sugunan, P. Radhakrishnan, *Meas. Sci. Technol.*, **14**, 858-861 (2003).

10. Y. Xiong, D-Q. Zhu, C-F. Duan, J-W. Wang, Y-F. Guan, *Anal. Bioanal. Chem.*, **396**, 943-948 (2010).

11. G. Goyet, D. R. Walt, P. G. Brewer, *Deep Sea Research Part A. Oceanographic Research Papers*, **39**, 1015-1026 (1992).

12. N. A. George, A. M. Paul, M. S. Saranya, *Light Elect. Opt.*, **125**, 301-303 (2014).

13. C. Wang, M. Kaya, C. Wang, *J. Biomed. Opt.*, **17**, 037004/1-10 (2012).

14. M. Kaya, P. Sahay, C. Wang, *Sensors Actuat. B: Chem.*, **176**, 803-810 (2013).

15. C. Wang, C. Herath, *Opt. Lett.*, **35**, 1629-1631 (2010).