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Elemental analysis of wastewater effluent using highly sensitive fiber Bragg grating sensor

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ABSTRACT

Industrial effluent is largely being used for irrigation of crops. Hence, monitoring its pollution level is essential to check its suitability for the purpose. We propose a simple and highly sensitive Fiber Bragg grating (FBG) sensor to determine the concentration of Chloride (Cl) and Lead (Pb) ions present in the treated waste water effluent of sugar factories. It is based on the principle of shift in the Bragg wavelength of an etched FBG with change in the ambient refractive index. The etched FBG was calibrated by known concentration of solutions containing the element under study and then was used to quantify the element in the waste effluent. Proper reagents were used for selective determination of the element. The concentrations of Cl and Pb ions in the effluent sample determined by our designed FBG sensor are 95.73 ppm and 0.008 ppm respectively which are in good agreement with the results obtained by APHA (American Public health association) standard techniques employed for wastewater analysis. Sensitivities of Cl and Pb sensors are calculated as 0.76 pm/ppm and 38.6 pm/ppm respectively. Sensitivity of the sensor for Pb ions being about 50 times greater than that for Cl ions, suggests a higher sensitivity of sensor in lower range of concentration in ppm. Limit of detection of our sensor for Cl and Pb are 1.2 ppm and 0.003 ppm respectively which are lower than that of APHA techniques, suggesting the superiority of our sensors. Simple working, miniaturization, cost-effectiveness and ability of online measurement are the most important features of FBG chemical sensors that give them an edge over existing techniques for continuous effluent monitoring.

1. Introduction

Industrial effluent has a significant contribution in environmental pollution which is identified as one of the major global problems today. Rapid urbanization and population growth as well as scarcity of fresh water has resulted in rising dependence of agriculture on Industrial waste water [1]. Sugar industry being an economically important agro based industry in our country produces a huge amount of waste water (1000 dm^3 of waste water per ton of sugarcane crushed) [2,3], which is mostly dumped into water bodies, either in the treated or untreated form which is then used for irrigation of crops [4]. This effluent consists of oil and grease, dissolved solids, micro-organisms, organic and inorganic chemicals. Although some of these are useful micronutrients and are beneficial for crop yield, an excess of any of the constituents may lead to soil deterioration and may have toxic effect on crop yield and hence on human health [5,6]. The suitability of effluent for agriculture and reuse is determined by measurement of physico-chemical parameters like pH, color, temperature, odor, COD (carbon oxygen demand), BOD (biological oxygen demand), TSS (total suspended solids), TS (total solids), TDS (Total dissolved solids), heavy metals and trace elements. TS, TDS and TSS are composed mainly of carbonates, bicarbonates, chlorides, sulphates, nitrates, Ca, Mg, Na, K, Mn and organic matter silts and other particles. Heavy metals in the effluent may include Cu^{2+}, Fe^{2+}, Zn^{2+}, Cr^{3+}, Ni^{2+} and Pb^{2+}. Many research papers have reported that the values of parameters, specially dissolved salts and heavy metals of treated sugar factory effluent exceeded the limits specified by the water regulatory bodies [1,3,7,8]. Hence, it is essential to monitor the quality of the effluent by checking the concentration of individual parameters as each one has its own ill effects on crop and soil quality when present in excess of upper limits specified for them.

We have designed Fiber Bragg grating (FBG) sensor to detect and quantify two of the elements, namely Chloride ions (Cl) and Lead ions (Pb) in sugar factory effluent. These are present in trace

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amounts in ppm levels and hence are usually ignored parameters. Determination of concentration of Chlorides (a part of total dissolved solids) in effluent is important as its gradual accumulation prompts inability of roots to absorb water, resulting in unfavorable growth of plants. High concentration of salts also makes the agricultural land non productive [9,10]. According to Food and Agricultural organization, United Nations (FAO) Guidelines for agricultural use (1985) the permissible limit of Cl in irrigation water is 142 ppm for surface irrigation and 106 ppm for sprinkle irrigation [11].

Lead is the most toxic heavy metal of all and high concentration of heavy metals in irrigated water causes adverse effects in plants. Although not essential for plant system, it is easily absorbed and accumulated by different parts of plants, causing retardation of growth, chlorosis and blackening of roots [5,12]. Lead can easily get into the human food, especially through contaminated vegetables, causing anemia, kidney malfunction, tissue damage of brain and even death in extreme poisoning situation. The permissible limit of Pb in irrigation water given by Row and Abdel (1995) as well as FAO (1985) is 5 ppm [9,11]. The permissible limit for Pb in wastewater, given by Environmental Protection Agency (EPA), is 0.05 ppm [13].

FBG sensors are well established in the field of strain, temperature and humidity sensing [14–16]. Several papers have reported work on Tilted FBG chemical sensors and FBG refractive index sensors [17]. Our previous work has presented highly sensitive FBG sensors for determination of toxic metal ions in drinking water [18,19]. Existing techniques for effluent analysis like APHA (American Public health association) standard methods [20,21], Spectrophotometric methods, Open and closed reflux methods, Titrimetric and Colorimetric method, microbial method, Atomic absorption method, etc. are good but some may be expensive, involve cumbersome procedures, need technicians for careful laboratory handling, may produce secondary hazardous waste, and are inadequate for remote and continuous on line monitoring of analysis [22,23]. On the other hand FBG sensors are simple, portable, are not affected with radiations, highly sensitive and above all facilitate uninterrupted online monitoring of effluent even in remote places [24]. Since the Bragg wavelength of FBG is its characteristic wavelength, the sensor is insensitive to fluctuations in the intensity of light coupled to it and hence capable of absolute measurement of the measurand. This advantage distinguishes FBG sensors from other fiber optic sensors. Also, their mass production can make them cost effective making them competitive to other conventional sensors [25].

1.1. Principle of FBG sensor

FBG is a photosensitive single mode optical fiber with a periodic modulation of refractive index (RI) inscribed along the length of its core by exposing it to UV light and acts as a selective mirror for a particular wavelength [26,27]. There are several methods of fabrication of FBG like holographic method, Amplitude mask method, point by point method and Phase mask method. Phase mask method which is the most commonly used fabrication method for FBG has been employed for our experiment. Phase mask technique makes use of an optical phase mask made of silica glass with a suitable grating period, to write the grating on a small bare portion of a single mode photosensitive optical fiber. The Phase mask, specially designed to suppress zero order diffraction is placed above the optical fiber in close proximity. The UV laser of wavelength range 244–256 nm is made to pass through the phase mask at normal incidence. This results in diffracted beams in the order of ±1 beams that superimpose to produce interference pattern on the core of fiber. This pattern photo-imprints periodic modulation of refractive index onto the core of the photosensitive fiber and thus

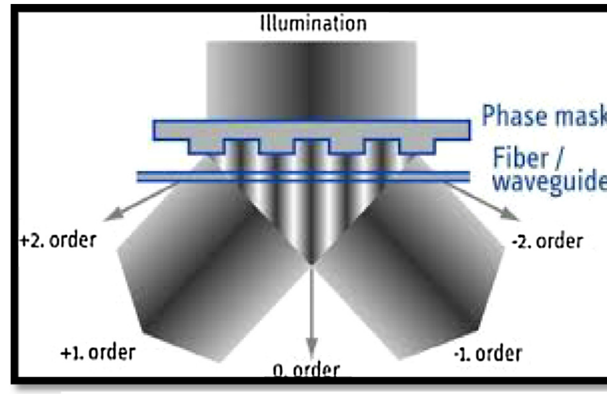

Fig. 1. FBG fabrication by Phase mask method.

forms Fiber Bragg grating [28]. Fig. 1 shows the schematic representation of the Phase mask method.

FBG has a short period of RI modulation which is less than 100 μm and it supports coupling between modes traveling in opposite directions. When light from a broadband source, consisting of multiple wavelengths is passed through the FBG, it reflects only a narrow band of wavelengths with a characteristic central wavelength, when Bragg condition is satisfied, which is given by [26]:

$$\lambda_B = 2n_{eff}\Lambda \qquad (1)$$

where, n_{eff} is the effective refractive index of the fiber core and Λ is the grating pitch. Principle of FBG is shown in Fig. 2. Normally, the core of the FBG is insensitive to the RI of the surroundings and hence the cladding is removed (etched) partially or completely in order to induce interaction between the core mode and external environment. Etching of the FBG makes n_{eff} depend on RI of external medium whereas keeps Λ unaltered [24,29]. Thus, when the concentration of medium surrounding the cladding varies, only the effective refractive index varies and brings about a shift in Bragg wavelength according to Eq. (1). However shift is accompanied by modification in the intensity of peak [29]. The shift in Bragg wavelength can be related to the concentration of the medium surrounding the etched FBG as RI is a linear function of concentration, for small concentrations [30]. Thus, basically RI sensitivity of the FBG is applied for designing our sensor to determine the concentration of analytes in waste water effluent.

Our experiment is based on the principle of immersing the FBG in solutions of varying known concentration of the element under study, measuring the corresponding Bragg wavelengths and using the Bragg wavelength – concentration graph to find unknown concentration of the sample. For selective measurement of the concen-

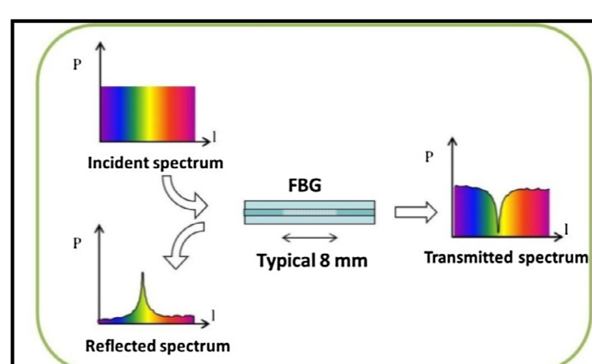

Fig. 2. Principle of FBG as a wavelength selector.