

# Calibration of Fiber Grating Heavy Metal Ion Sensor Using Artificial Neural Network

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**Abstract:** An ultrasensitive hybrid fiber grating sensor has been designed and functionalized with a novel nanocomposite material for selective detection of lead ions in water. Sensor performance is enhanced using an artificial neural network-based calibration process.

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## 1. Introduction

Of the seventeen ‘Sustainable Development Goals’ announced by the United Nations, the 6<sup>th</sup> and 14<sup>th</sup> goals are the ‘Clean water and sanitation’ and ‘Life below water’ [1]. This emphasizes the global importance of water quality monitoring and addressing this a fiber optic heavy metal ion sensor has been designed, developed, and experimental results of its performance are reported in this paper. The intrinsic characteristics such as flexibility, compactness, immunity to electromagnetic interference, and insensitivity to a wide range of chemicals have made an optical fiber-based approach preferable for real-time remote monitoring of key toxic materials e.g. heavy metals, hormones, pesticides, and pharmaceutical residues in water [2]. In this work, a hybrid fiber optic grating-based sensor has been designed by combing a higher-order Long Period Grating (LPG) with LP<sub>0,9</sub> attenuation band and 1524 nm Fiber Bragg Grating (FBG) in series. The attenuation bands from the LPG and FBG were concurrently utilized for selective sensing of the heavy metal ion, in this case the Pb(II), and to allow for a correction to be made the ambient temperature. A FBG is immune to any surrounding refractive index changes, so the static response of its attenuation band was used as a reference point for making accurate measurements of the relative LPG shift ( $\Delta\lambda_{RF}$ ). The unique light-guiding mechanism of the LPG provides a reliable solution of surface sensing without tampering the structural integrity of the fiber [3]. The experimental data recorded, showing the sensor response was used to train an Artificial Neural Network (ANN) [4] to obtain the sensor calibration graph for metal ion concentration measurement.

## 2. Experiment results and discussion

Figure 1(a) shows  $H_y$  field distribution of the fibre LP<sub>0,9</sub> clad mode, simulated by using the Finite Element Method (FEM) [5]. The in-house excimer laser inscribed LPG was functionalized by using a layer-by-layer assembly of nanocomposite material and polyacrylic acid (PAA) of coating thickness of  $\sim 390$  nm, in order to optimize its sensitivity. The sensing surface of the coated LPG and the coating thickness were verified by using FE-SEM imaging, as shown in Figs. 1(b) and (c), respectively. The CCS-NGO nanocomposite, synthesized in the lab by using Nitrogen-doped Graphene Oxide (NGO), Cross-linked Chitosan (CCS) polymer, and PAA, containing major functional groups

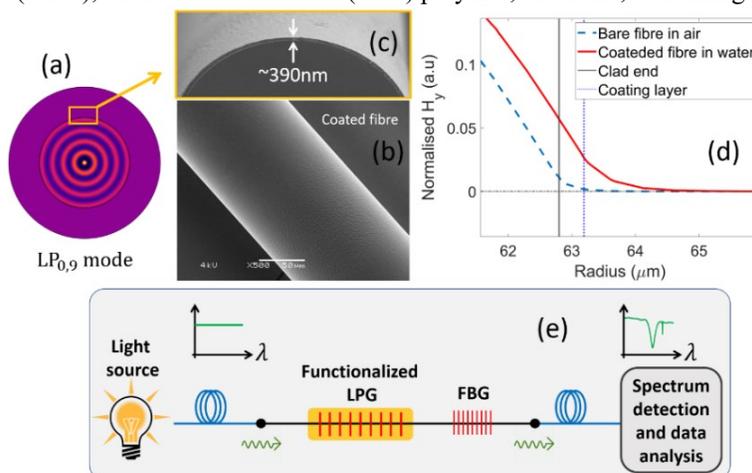


Fig. 1. Fibre grating-based heavy metal ion sensor. (a) Schematic of experimental setup. (b)  $H_y$  field distribution of LP<sub>0,9</sub> mode. (c) and (d) depict the SEM images of the coated LPG section. (e) Evanescent field enhancement due to sensing layer overlay.

such as  $-\text{NH}_2$ ,  $-\text{SH}$ , and  $-\text{COOH}$  that make the CCS-NGO/PAA coating sensitive to a small concentration of Pb(II) ion in a water environment. Moreover, the CCS-NGO/PAA multilayer coating enhances the light-matter interaction by  $\sim 30$  times by strengthening the evanescent field of the  $\text{LP}_{0,9}$  clad mode, as shown in Fig. 1(d). Figure 1(e) illustrates schematically the experimental setup where the response of the fibre grating sensor was interrogated and spectra analyzed, using an optical spectrum analyzer (OSA) and an ANN-based signal processing system.

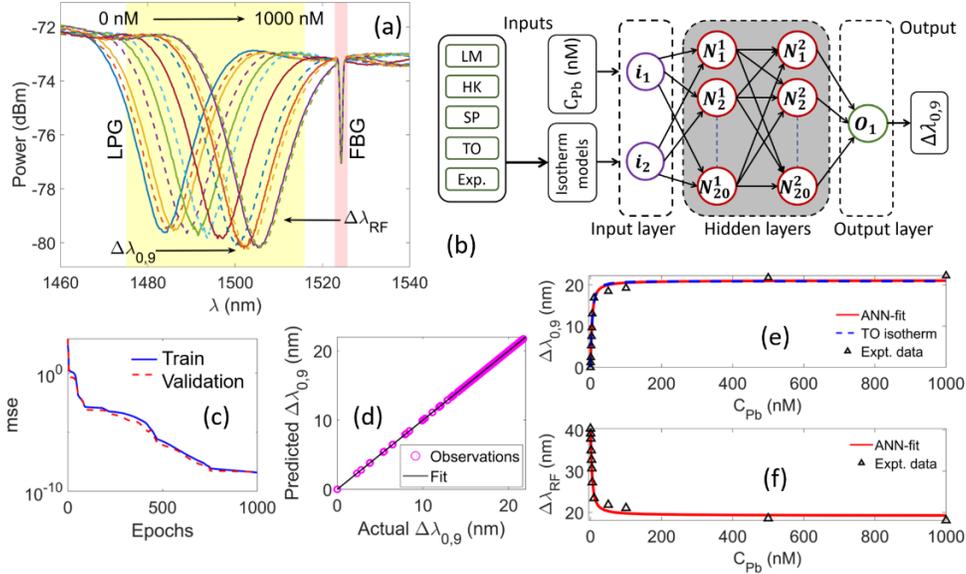


Fig. 2(a) Spectral response of the hybrid fibre grating sensor with  $C_{Pb}$ . (b) ANN framework block diagram. (c) mse vs epoch plot for training and validation sets. (d) Correlation plot of actual and predicted  $\Delta\lambda_{0,9}$ . (e) and (f) Calibration curves ( $\Delta\lambda_{0,9}$  and  $\Delta\lambda_{RF}$  vs  $C_{Pb}$ ) of the fibre grating-based Pb(II) ion sensor.

A real-time response (shown in Fig. 2(a)) of the fibre sensor was recorded with the variation of Pb(II) ion concentration ( $C_{Pb}$ ) ranging from 0 to 1000 nM. The experimental data on the LPG wavelength shift ( $\Delta\lambda_{0,9}$ ) as a function of  $C_{Pb}$  were fitted with Langmuir (LM), Holl-Krich (HK), Sips (SP), and Toth (TO) adsorption isotherm models that represent homogenous and heterogenous adsorption process on the multilayer adsorbent coating. These fitted and experimental data were further used to train an ANN model with one input, two hidden (20 nodes each), and one output layers, as shown in Fig. 2(b). The ANN performance plot in Fig. 2(c) shows a very low mean squared error ( $mse \sim 10^{-9}$ ) during training and validation, indicating a well-trained ANN model. The regression plot in Fig. 2(d), shows an excellent correlation between the actual and predicted value of  $\Delta\lambda_{0,9}$  with a correlation coefficient of  $R = 0.9999$ . Figures 2(e) and (f) represent the calibration curves for the  $\text{LP}_{0,9}$  ( $\Delta\lambda_{0,9}$ ) and relative ( $\Delta\lambda_{RF}$ ) spectral shifts, shown respectively against  $C_{Pb}$ . In both cases, the ANN fitted data showed an excellent agreement with the experimental data and Toth isotherm in sub-nanomolar and nanomolar concentration region of Pb(II) ion. Thus, from the Toth isotherm the limit of detection (LOD) of the sensor was determined to be 0.18 nM.

### 3. Conclusion

A new sensor system, using a synergy between fibre gratings (LPG and FBG) and a nanocomposite material have been exploited to design, develop, and experimentally demonstrate an ultrasensitive Pb(II) ion sensor. A trained ANN model was used as a metal-ion-concentration calibration tool that can accurately predict the nonlinear response of the sensor over a broad range of heavy metal concentrations in water.

### 4. Acknowledgement

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