

Silver Nanoparticles as Coating for Fiber Bragg Gratings in Lead Detection

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Abstract

Water pollution from heavy metals, including lead (Pb^{2+}), remains a serious problem for humanity due to their use in various industries. Although the development of water detection sensors has also been widely pursued, including etched fiber Bragg gratings (e-FBG) and tilted FBG (TFBG). FBG is unique because the optical fiber is modified, enabling it to act as a sensitive sensor for detecting Pb^{2+} . However, the existing devices require modifications that entail high costs, the need for skilled operators, and even complex preparation. Therefore, this study reports an FBG-based sensor utilizing AgNPs as a functional material for Pb^{2+} detection. Notably, the AgNPs were synthesized using laser ablation. In this study, AgNPs coating on FBGs was done to utilize localized surface plasmon resonance (LSPR), which was subsequently used for lead detection, showing a minimum detection limit of 0.65 ppm, with a sensitivity of 9.2×10^{-3} ppm/dBm, and quite selective to Pb^{2+} . Although the detection limit is not below the WHO threshold, these results demonstrate that Ag-coated conventional FBGs have potential for Pb^{2+} detection through simple, low-cost, and eco-friendly modifications. Therefore, further modifications are required to enhance the Pb detection performance of the nanoparticle coating on the FBG.



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Introduction

The modern world is closely tied to the use of minerals extracted from the earth for various applications [1]. Heavy metals such as lead (Pb^{2+}), arsenic (As^{3+}), cadmium (Cd^{2+}), chromium (Cr^{2+}), zinc (Zn^{2+}), and mercury (Hg^{6+}) from industrial use have been released into the water, air, and soil, causing undeniable pollution [2], [3]. To date, water pollution remains a serious problem that requires rapid and periodic detection [4], [5]. In Indonesia, many rivers have been reported to be polluted by heavy metals, especially Pb^{2+} . For example, in the Jeneberang river, the Pb^{2+} content is quite high, ranging from 30 to 50 ppm [6]. Meanwhile, the maximum threshold for Pb^{2+} in water, according to the World Health Organization (WHO), is 0.01 ppm

[7]. It has been widely used to detect water quality from heavy metals using an Atomic Absorption Spectrophotometer (AAS) [8], [9], Inductively Coupled Plasma Mass Spectrometry (ICP/MS) [10], [11], and Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP/AES) [12]. Although effective, these methods require high costs, complex preparation, and expert operators. This condition has led to the development of techniques for detecting heavy metals in water that are easier and more effective in various ways.

Optical sensors have advantages, including resistance to electromagnetic interference and high stability even when exposed to extreme temperatures, pressure, and humidity [13]. One of the efforts to develop heavy metal detection is to utilize fiber bragg gratings (FBG), which specifically use evanescent waves in surface plasmon resonance [14], [15]. Although FBG-based sensors are often used to measure load, strain, voltage, and temperature, coating certain nanomaterials as external modifications can also improve their efficiency in heavy metal detection [16], [17]. In addition, some attempts to make it into a heavy metal sensor, in addition to coating nanomaterials directly, are by etching FBG (e-FBG) and tilted FBG (TFBG) [18], [19]. These efforts have demonstrated that fiber optics can detect heavy metals. Previous research showed that the use of e-FBG is able to detect Pb^{2+} with a low detection limit of up to 10 fM [20].

Both eFBGs and TFBGs exhibit enhanced sensitivity to interactions between the optical field and the surrounding environment, driven by changes in the surface refractive index. These changes are achieved by modifying the fiber structure, such as by reducing the cladding in eFBGs or by using tilted gratings in TFBGs. In eFBGs, the etching process using strong hydrofluoric acid renders the fibers brittle and prone to damage, whereas TFBGs involve more complex data interpretation due to their sensitivity to bending direction. Furthermore, both types require specialized structural modifications, resulting in higher costs [21], [22]. In contrast, this study employs conventional FBGs and evaluates the extent to which nanocomposites can serve as active materials without structural modifications. Furthermore, conventional FBGs are less expensive than structurally modified FBGs, which are quite costly despite still requiring coating modifications. Even so, the study still needs further development to detect heavy metals.

To enhance the performance of the FBG-based heavy metal detector, this study modified the FBG with nanoparticles. Nanoparticles have various advantages that can improve performance. Silver nanoparticles (AgNPs) are one of the materials that have been in demand due to their many unique properties, including their extraordinary plasmonic properties [23]. The optical properties of these nanoparticles, which exhibit specific absorption of localized surface plasmon resonance (LSPR), make them very likely to be able to detect chemical substances [24], [25]. Previous research has also proven that AgNPs sensors can detect heavy metals Pb^{2+} in water up to 13.6 μM [26],[27]. This reinforces the use of AgNPs as FBG coatings for further development of heavy metal detection. However, in that study, AgNPs were synthesized using chemicals such as silver nitrate, sodium borohydride, hydrazine hydrate, sodium citrate, and lithium aluminum hydride.

The use of these chemicals does not rule out the possibility of secondary pollutants that are harmful to the environment. Therefore, in this study AgNPs were synthesized by laser ablation, a method considered safer and more environmentally friendly than chemical methods because it requires no additional chemicals and uses only water and a laser [28]. In addition, the AgNPs-coated FBG sensor configuration features a simpler structure and is easier

to fabricate than several previous studies that require optical fiber modifications, a distinct advantage of this study. Therefore, this study was conducted to demonstrate the performance of AgNPs-coated FBGs for Pb^{2+} detection in water, which offers structural simplicity, sensor stability, environmental friendliness, and potential for further development.

Experimental Method

In this experiment, an FBG with a center wavelength of 1559.887 nm is used, coated with AgNPs. The synthesis of AgNPs is carried out by laser ablation, and before firing, the bulk 99.9% silver is first rubbed with sandpaper and sonicated for 10 minutes. Laser ablation was performed with a Ytterbium laser fiber RFL 20 at a wavelength of 1064 nm with 90% power (20 watts of the maximum energy) and a speed of 1000 mm/s for 2 minutes. A cleaned piece of pure silver bulk was placed in a cuvette with 4 mL of water for laser irradiation. The parameters used, including the time, were kept constant during nanoparticle synthesis.

Subsequently, AgNPs were prepared for XRD, UV-Vis, and SEM-EDX analysis. The AgNPs solution was deposited onto a 1×1 cm glass slide. This is because the AgNPs, initially in liquid colloid form, need to be solidified for characterization by XRD and SEM, as these techniques cannot be performed on liquid samples. Then, the sample was characterized using XRD, UV-Vis, and SEM-EDX to determine its structure, absorbance, morphology, and elemental composition.

Before coating, FBG is cleaned with Isopropyl Alcohol and methanol. After drying, the FBG is soaked in 1 M NaOH and then cleaned with deionized water. Next, after cleaning and drying again, the FBG is soaked in Mercaptopropyl-trimethoxysilane (MPTMS) for 24 hours, then cleaned again with ethanol and soaked with AgNPs until dry. Once dry, FBG can be used to detect heavy metals using an Optical Spectrum Analyzer (OSA). Pb^{2+} samples were prepared by making 10 – 50 ppm solutions from $\text{Pb}(\text{NO}_3)_2$. The Pb^{2+} solutions were placed in the well of the container with an FBG connected to an OSA and a light source. After connection, the Pb^{2+} concentration was varied, and the sample was positioned at the Bragg location, and OSA data were then collected to determine sensitivity. Selectivity was assessed by varying the heavy metals while keeping the concentration constant.

Result and Discussion

The analysis of the diffraction pattern and structure of AgNPs is shown by the X-ray diffraction results in Figure 1. Based on Figure 1 (a), AgNPs have a crystalline diffraction pattern visible at the apex $2\theta = 37.99^\circ$, 44.15° , 64.21° , and 77.10° with hkl planes (111), (200), (220), and (311), with lattice parameters of 4.0995 Å. This pattern is in accordance with the AMCSD database No. 0011135, which also indicates that the crystal structure of AgNPs is face-centered cubic (FCC) with the space group Fm-3m ($\alpha = \beta = \gamma = 90^\circ$), as in Figure 1(b). This is also in line with the diffraction pattern of AgNPs observed in previous research, which reported AgNPs diffraction patterns corresponding to hkl (111), (200), (220), and (311) and a cubic crystal structure [26], [29]. These analysis results show that AgNPs were successfully synthesized without any impurities.

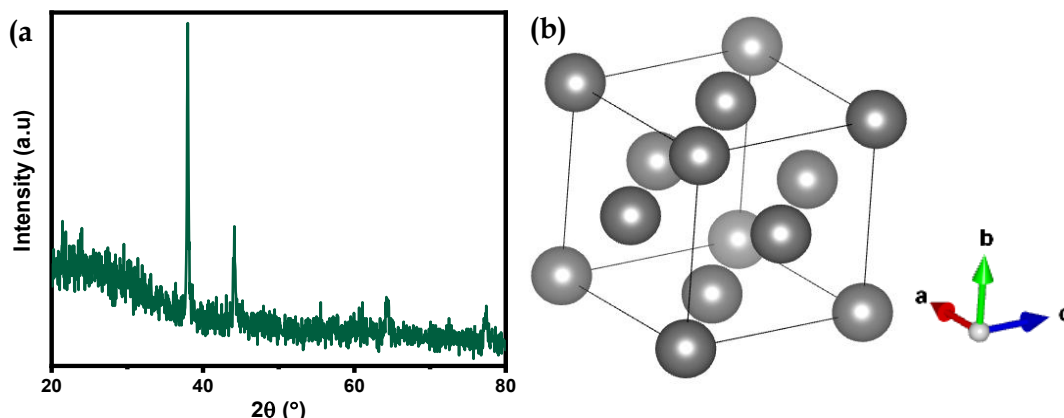


Figure 1. (a) XRD Analysis (b) Crystal Structure of AgNPs

Furthermore, the UV-Vis spectrum of AgNPs is shown in Figure 2. AgNPs appear to have a peak around 400 nm. The peak indicates the presence of LSPR, which is characteristic of AgNPs. Furthermore, this peak is close to the absorbance results previously studied, namely at a wavelength of 421 nm [30]. AgNPs are metallic materials; the absorption band originates from LSPR. These characteristics support the formation of AgNPs with the synthesis method in this study, although results from other characterizations are also needed to confirm that. Then, the high absorbance intensity indicates the significant formation of AgNPs in the colloid [31].

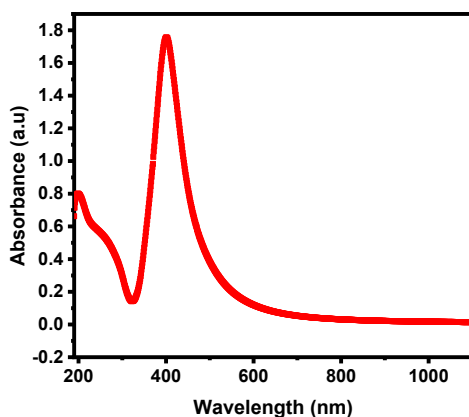


Figure 2. Analysis of UV-Vis Spectrometer

The morphology of AgNPs was analyzed using a Scanning Electron Microscope-Energy Dispersive X-ray (SEM-EDX), and the results are shown in Figure 3. These nanoparticles were synthesized in colloidal form and required drying on a glass substrate for SEM-EDX testing, which requires a solid sample. EDX analysis revealed the presence of AgM and AgL elements at around 0.5 keV and 2.8 keV, respectively, representing the AgNPs in the colloid [32]. Si, O, Mg, Ca, and Na were also found in this colloid in significant amounts due to the glass sample preparation used in the test [33].

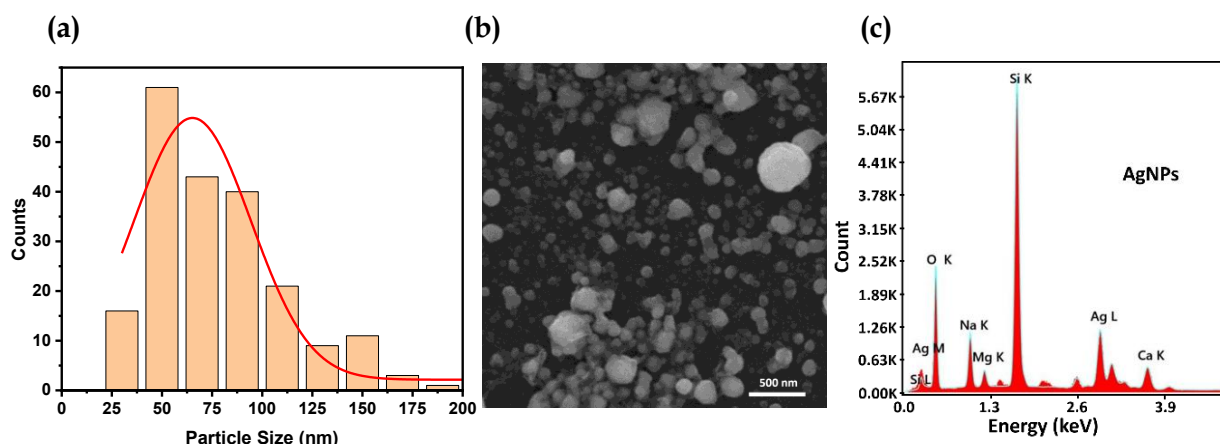


Figure 3. SEM-EDX Analysis of AgNPs.

Based on SEM analysis, the average particle size of AgNPs is 65.2 nm with a spherical shape. The average AgNPs size commonly found is 1-100 nm, and previous research has reported an average AgNPs size of 48 nm [34]. AgNPs can possess a large surface area due to their nanoscale dimensions, resulting in enhanced light absorption and effective interaction with target molecules [35]. This size can be adjusted, or made smaller or larger, by changing the ablation parameters [36]. However, SEM images indicate that each particle tends to agglomerate. In theory, colloidal nanoparticles often easily experience aggregation between particles because the ratio between the surface and volume is too large [37]. Therefore, AgNPs coating on the FBG must be performed on a new sample to avoid nanoparticle agglomeration.

In this study, the sensor's performance in detecting Pb^{2+} was evaluated using sensitivity and selectivity tests. The performance of the AgNPs-coated FBG as a Pb^{2+} heavy-metal detection sensor has been tested, with the results shown in Figure 4. Based on Figure 4 (a), it is known that the AgNPs-coated FBG is able to detect the presence of Pb^{2+} with a sensitivity of 9.2×10^{-3} dBm/ppm (Figure 4 (b)). Furthermore, the lowest detection limit of this sensor has been calculated using Equation (1), yielding 0.65 ppm within the 10–50 ppm detection range. As with previous studies, AgNPs have been proven to be able to detect Pb^{2+} with a detection limit of 37×10^{-9} M [38], [39]. Figure 4(c) is a graph of sensor selectivity, showing that the sensor exhibits a significant response to Pb^{2+} relative to Cd^{2+} , Zn^{2+} , and Cu^{2+} . These results prove that the use of AgNPs also provides quite good selectivity to Pb^{2+} compared to other heavy metals [40].

$$LoD = \frac{3.3 \times \sigma}{s} \quad (1)$$

Where σ is the standard deviation or standard error, while s is the slope of the sensor linearity [41].

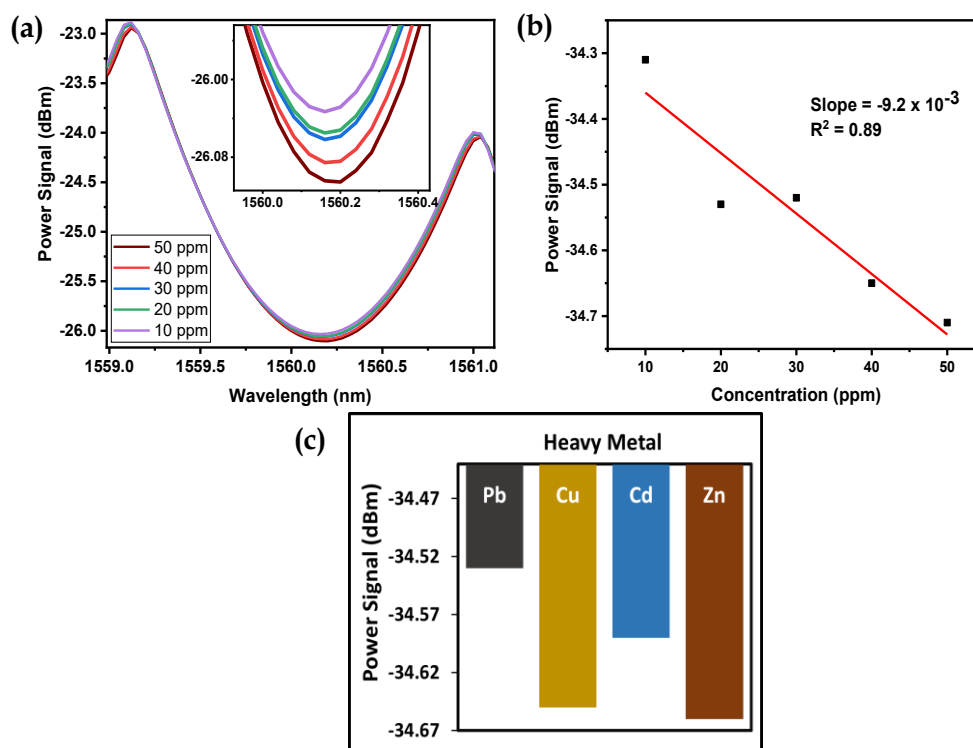


Figure 4. (a) Peak Position, (b) Sensitivity, and (c) Selectivity of Pb^{2+} Sensor.

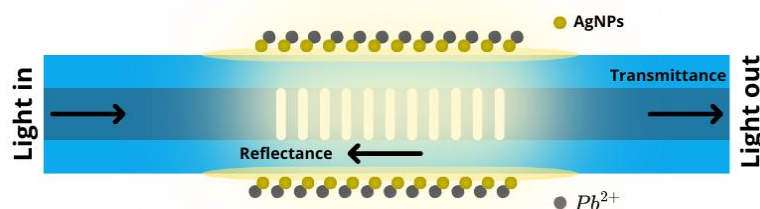


Figure 5. Pb^{2+} Sensor Detection Mechanism.

Next, the heavy metal detection mechanism using FBG-based AgNPs is shown in Figure 5. AgNPs will cooperate with Pb^{2+} , so that it changes the external conditions of the FBG and makes a change in wavelength compared to before the presence of AgNPs, as well as a change in signal strength if there is a difference in heavy metals [16], [42]. MPTMS, in this case, functions as an activator of the -OH group to bond AgNPs to the FBG [43]. By coating MPTMS first, AgNPs can adhere well to the FBG, thereby improving its performance in detecting Pb^{2+} . In the FBG concept, incoming light waves exit in two paths; some are transmitted, and others are reflected. This occurs due to the principle of Bragg reflection with periodic refractive index modulation that is capable of reflecting very well, and the other is transmitted through the grating [44]. But, this experiment shows that detecting Pb^{2+} based on signal intensity change, because the change in Pb^{2+} concentration only causes loss of the cladding mode and not enough to change the global refractive index, so the wavelength does not shift [19].

Several previous studies have also successfully applied FBGs to detect Pb^{2+} in water using various techniques such as FBG etching and TFBG. FBG etching-based sensors have demonstrated the ability to detect Pb^{2+} with a sensitivity of 5×10^{-3} nm/ppm over the concentration range of 0–50 ppm [45]. In addition, it has also been reported that Black Phosphorus coating on TFBG showed a sensitivity of 0.5×10^{-3} dB/ppb and a detection limit of 0.25 ppb, and when TFBG was coated with gold (Au) and thrombin aptamer, the detection limit was low, reaching 0.001 ppb [46], [47]. Not only that, TFBG with graphene oxide (GO) and Au coating showed a sensitivity of 0.018 dB/ μM and a detection limit of 0.711 μM on Pb^{2+} [19]. This demonstrates that coating FBGs with various materials can improve sensor performance and that high sensitivity arises from structural modifications in TFBGs and eFBGs. Without structural modifications, this research will focus more on the effects of the AgNPs coating. However, this research focuses primarily on the functionality of AgNPs as an FBG coating for Pb^{2+} detection. Although the detection limit of this study does not yet meet the WHO-recommended maximum Pb^{2+} threshold of 0.01 ppm, this sensor has the potential to serve as an initial monitoring tool for detecting Pb^{2+} contamination using a simpler system.

Conclusion

FBG, which is usually a strain and temperature sensor, can also detect Pb^{2+} ions well. The use of AgNP coatings as functional materials exhibiting the LSPR phenomenon is very helpful for FBGs in detecting heavy metals. The AgNPs produced via laser ablation are spherical and 65.2 nm in size. It is also driven by the small particle size and the remarkable optical properties of AgNPs produced by laser ablation. In this case, the detection of Pb^{2+} showed sensitivity and selectivity, with a detection limit of 0.65 ppm. Although the resulting detection limit remains higher than the WHO threshold for the heavy metal Pb^{2+} in water. This study has contributed to the development of AgNPs as a functional material for FBG-based Pb^{2+} detection. Even so, improving the performance of FBG remains necessary, and it is hoped that future research will develop FBG coatings using AgNPs compositions combined with other nanomaterials.

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