Development of a MEMS-based Piezoresistive Cantilever Sensor for Lead (Pb(II)) Detection in Drinking Water

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Received: 1 July 2024 | Revised: 31 July 2024 and 16 August 2024 | Accepted: 22 August 2024

DOI: https://doi.org/10.48084/etasr.8275

ABSTRACT

One of the most hazardous pollutants of natural water resources is lead -Pb (II)- which poses a significant threat to human health and environmental safety. The accumulation of this heavy metal in an organism affects a number of systems and is particularly dangerous for children. At low levels of intake over short periods, it induces diarrhea, abdominal pain, and renal damage, with the potential for fatal outcomes in extreme cases. The principal sources of lead pollution are industries, coal-fired power plants and motor vehicles. In response to the critical demand for effective lead detection, researchers have developed advanced Micro-Electromechanical Systems (MEMS) piezoresistive cantilever sensors that make use of the chelating properties of Ethylenediaminetetraacetic Acid (EDTA) and the superior electrical properties of reduced Graphene Oxide (rGO). It has been proven that this composite can be effectively immobilized on a MEMS cantilever surface, enabling the selective removal of Pb (II) ions from wastewater. This adsorption process exerts stress on the surface of the cantilever, resulting in variations in resistance that can subsequently be measured. A sensitive and selective MEMS piezoresistive cantilever sensor for Pb (II) has been developed, offering significant potential as a lead monitoring tool in water samples. The sensor demonstrated high sensitivity and selectivity, with a detection limit of 1 ppb and a linear response range of 10-100 ppb. This novel approach has the potential to significantly enhance pollution monitoring and provide substantial benefits for public health by enabling real-time, on-site mapping of lead contamination across aqueous environments. This technological advancement in the environmental surveillance domain offers a new perspective on the safety of water and the reduction of potential health hazards associated with lead consumption.

Keywords-lead (Pb(II)) contamination; MEMS sensors; piezoresistive cantilever; environmental monitoring; water safety

I. INTRODUCTION

The detection and quantification of heavy metal ions (e.g., lead Pb (II), cadmium Cd (II), and mercury Hg (II)) in water sources is of great importance, given their high toxicity and substantial impact on human health and the environment. These contaminants are hazardous when present as trace compounds in drinking water. Even at minimal levels, they were found to exert powerful effects on human health, causing neurological disorders and kidney damage. This has driven the need for the development of sensitive and accurate detection methods [1]. Electrochemical sensors have been the subject of extensive study due to their high sensitivity, rapid diffusion on the surface and low-cost detection efficiency in different protocols among the various detection methods available [2]. Meanwhile, the usage of nanomaterials, particularly rGO and its composites, has enabled a further enhancement in the performance of these sensors [3]. It is of considerable interest to examine the surface properties of rGO, which is an excellent choice for modifying sensors, thus improving their sensitivity and selectivity. Authors in [4], used an electrochemical sensor for the sensitive and selective detection of lead ions (Pb2+), which was developed using a nanocomposite film, exhibiting a notable synergistic effect on the sensor's sensitivity and selectivity for Pb (II) detection. Similarly, the functionalization of an electrochemically reduced graphene oxide with amino groups indicated a notable enhancement in the sensitivity of the sensors for the detection of trace Pb (II) species, when compared to the Pb (II) ions detection limit. The surface functionalization facilitated the electrochemical sensing of trace metal ions to a considerable extent [5]. Moreover, the incorporation of rGO with gold and palladium nanoparticles resulted in the enhancement of sensor activity [6]. Authors in [7] reported the one-step synthesis of AuPdNPs/ERGO nanocomposites by hydrothermal synthesis for the electrochemical sensing of Cr (VI) in water, which is well suited to lead detection in drinking water [8]. Additionally, authors planned to detect a specific individual metal without interference from the surrounding elements, which demonstrates the simultaneous detection of different HM, highlighting the potential of a graphene base for electrochemical sensors. Furthermore, the potential of graphene in conjunction with ZnO for the simultaneous detection of Pb (II) and Cd (II) has been explored [9]. The ZnO/ErGO-modified electrode demonstrated this capability, suggesting the possibility of these materials for multi-analyte detection [10]. Authors in [11] developed a method for functionalizing DNA aptamers over electrochemically reduced graphene oxide for the ultra-trace sensitive detection of lead cations. This approach provides insights into the biofunctionalization of sensors for improved specificity [12]. Furthermore, the ultra-trace determination of Cd (II) was achieved through the development of electropolymerized ion-imprinted PoPD/ERGO composite-based sensors [13]. Simultaneous quantitation of lead and cadmium using EDTA-reduced graphene oxide-modified glassy carbon electrodes. The selective electrochemical sensing of arsenite using rGO/Fe_3O_4 nanocomposites was used, demonstrating the versatility of graphene-based materials for sensing toxic heavy metals. Piezoresistive cantilevers were developed into versatile structures for a variety of applications, including force measurement, pressure sensing and environmental monitoring. These generate a voltage through mechanical strain, which is employed for the dependent monitoring of minor displacements and forces [14]. Authors in [15] optimized the design of piezoresistive cantilevers for force sensing in both air and water environments. Their findings demonstrated that cantilever geometry and material properties are critical for the proper performance of such sensors. Further research was focused into the noise and sensitivity bands of piezoresistive cantilevers, which facilitated the development of a sophisticated design and implementation of the sensors [16].

This constitutes a significant step forward in the field of research, as the development of ultrathin piezoresistive cantilevers [17] has enabled the achievement of high sensitivity through the reduction of thickness [18]. The use of piezoresistive cantilevers in advanced applications has revealed the capacity for two-dimensional force sensing. Recently, a cantilever capable of evoking forces in all possible directions was developed by researchers, and differential pressure sensing were carried out. Authors in [19] validated the accuracy and reproducibility of their cantilever-based sensor through theoretical proof and experimentation. Authors in [20] reviewed the performance of piezoresistive cantilevers in nanomechanical sensing and demonstrated their capability in a variety of high-precision applications [21]. More recently, studies have examined the efficient piezoresistive design of microcantilevers for improved stress sensing and reduction/compensation of temperature influence. It is of critical importance to optimize the sensor in order to enhance readings in different environmental conditions, thereby ensuring the reliability and accuracy of the sensor [22]. Furthermore, the performance of MEMS cantilevers was investigated for the specific task of VOC detection, thereby demonstrating the potential and capability of environmental monitoring. Piezoresistive cantilevers were previously demonstrated in Atomic Force Microscopy (AFM), which initially created a silicon cantilever beam with a piezo resistor integrated into the IPS tech system. This enabled the measurement of distances from surfaces with greater accuracy, thereby enhancing the specificity of AFM for surface spectra [23]. Researchers put forth the concept of portable microforce calibration standards and adopted the use of piezoresistive cantilevers for applications requiring high precision measurements [24-26].

The present paper examines an innovative nanomechanical sensor based on the CMOS MEMS piezoresistive microcantilevers, coated with PEDOT for the detection of Pb (II) ions. The sensor displays excellent sensitivity, with a remarkable ability to monitor lead concentrations in the range of 0.01-1000 ppm, with a detection limit as low as 5 ppb. Consequently, this sensor can be conveniently employed for on-site water quality monitoring, facilitating the development of a portable solution for real-time detection of lead in drinking water [27]. In this study, the piezoresistive cantilever sensor is developed for the detection of small molecule concentrations. The incorporation of a membrane-bridge structure enhances the sensitivity of the microcantilever sensor, as compared to traditional sensor designs. The technique is applied to the detection of trace amounts of contaminants in water, thereby providing a more sensitive and reliable method for environmental monitoring [28]. This paper describes the development of an inkjet-printed flexible electrochemical sensor. The sensor was equipped with an electrochemical detection system based on gold for the purpose of detecting lead (II) ions in aqueous environments. The sensor has demonstrated satisfactory performance for lead levels below the limits set by the United States Environmental Protection Agency, making it suitable for the practical detection of lead in drinking water in both laboratory and field settings [29].

II. PROPOSED FRAMEWORK

A. Synthesis of ErGO Nanocomposites

The ErGO nanocomposite was synthesized in three stages [30]. This process commenced with the synthesis of Graphene Oxide (GO) using the well-established simplified Hummers method. A quantity of graphite powder (0.4 g) was added to a mixture of concentrated sulfuric acid and ortho-phosphoric acid (40 ml: 10 ml) and stirred for a period of five minutes. Then, 2.4 grams of $KMnO_4$ was slowly added, resulting in a purplish mixture that was stirred continuously for a period of three days at room temperature in order to facilitate the oxidation process [31]. The color of the mixture underwent a change, from purple to dark brown. The reaction was terminated under cold conditions to halt the oxidation process. The mixture was then poured slowly into 100 mL of cold deionized water containing 5 mL of 30% H_2O_2, which resulted in a change in solution color to yellow, confirming the oxidation of graphite [25]. To remove any residual impurities and unreacted reactants, the sample was subjected to a series of centrifugations, first with 1 M HCl (three times) and then with DI (seven times). This process also resulted in the thickening of the GO, yielding a dark brown gel. The gel was subsequently subjected to a drying process at 60°C in a vacuum oven for a period of six hours [31]. Subsequently, the EGO nanocomposite was prepared by dispersing the purified graphene oxide in ethanol at a concentration of 5 mg/mL and subjecting it to ultrasonication for a period of 60 minutes. Furthermore, a solution of Ethylenediaminetetraacetic Acid (EDTA) was prepared in ethanol at a concentration of 20 mg/mL. Subsequently, 10 mL of the GO solution and 10 mL of the EDTA solution were combined and stirred for 15 hours to functionalize the GO, which was rinsed three times with deionized water [32]. The resulting product was then dried in a vacuum oven at 60°C for a period of 12 hours. Lastly, electrochemical reduction was employed to transform EGO into ErGO. EGO was dispersed in deionized water (5 mg/mL) and ultra-sonicated for 2 hours. Subsequently, 5 µL of the dispersed EGO was drop cast onto polished glassy carbon electrodes and left to dry. Thereafter, under a nitrogen environment using 0.5 mM potassium nitrate as electrolyte, cyclic voltammetry was performed between 0 and -1.5 V at a scan rate of 50 mV/s for 30 scan cycles [33]. Following 30 scan cycles, EGO was fully converted into ErGO, as shown in Figure 1. The modified glassy carbon electrode was dried in a nitrogen environment and employed for the detection of Pb (II) [34].

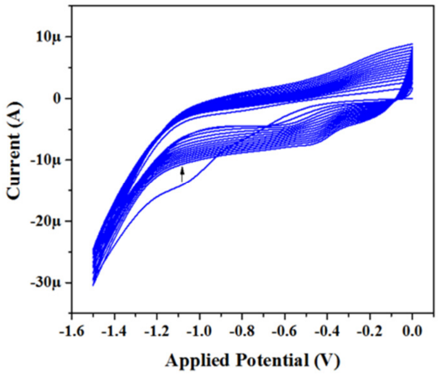

Fig. 1. Electrochemical synthesis of ErGO nanocomposite.

B. *Electroanalytical Determination of Pb (II)*

In order to examine the behavior of ErGO-modified glassy carbon electrodes in the presence of Pb (II), a square wave voltammetry technique was deployed using 0.3 M nitric acid as electrolyte within the voltametric cell. The optimal working parameters for the square wave were identified as an amplitude of 0.025 volts, a frequency of 40 Hz and a step potential of 0.01 V. All experiments were performed in a nitrogen environment. The electrochemical determination of Pb (II) involves two distinct steps. First, the Pb (II) ions are reduced to the surface of the electrodes using a deposition potential of -0.7 V for a period of 150 seconds. During the stripping step, a square wave is applied within the range of -0.7 V to -0.2 V [24]. Furthermore, the modified electrode was investigated with regard to its selectivity for Pb (II) by performing sensing studies in the presence of potential interferants, namely Cu (II), Cd (II), Fe (II) and Hg (II), at concentrations 10-fold and 20-fold.

III. RESULTS

The electrochemical performance of the synthesized EGO and ErGO nanocomposites was evaluated through the utilization of Cyclic Voltammetry (CV) in a 5 mM ferri-ferro solution. The objective of this study was to investigate the electron transfer kinetics of the ferri-ferro-redox probe, with a particular focus on the influence of the electrode/electrolyte interface. The results are presented in the following:

A. *Electrochemical Characterizations*

The electron transfer kinetics of the synthesized EGO and ErGO were examined in greater detail through the use of CV, with measurements taken at 5 mM Ferri-Ferro, as shown in Figure 2. ErGO exhibited superior areal capacitance and enhanced conductivity when compared to EGO, as evidenced by frequency response analysis and Nyquist plots, respectively. Additionally, an increase in peak current density was observed upon increasing the scan rates (1.773E-6 cm^2/s). These enhancements can be attributed to electron-limited processes, as well as diffusion-controlled enhancement properties. Moreover, ErGO exhibits a markedly reduced charge transfer resistance of 20.2 kΩ, in comparison to EGO, which is 54 kΩ, as shown in Figure 2. The ErGO-modified glassy carbon electrode exhibited enhanced electrochemical characteristics compared with EGO, due to the increased electrical conductivity and high surface area of the ErGO species. Furthermore, the impact of the scan rate on the CV characteristics of the ErGO-modified glassy carbon electrode was investigated, as shown in Figure 3.

B. *Effect of Experimental Variables*

The influence of the deposition potential on the current response at a Pb (II) concentration of 50 ppb was examined on an ErGO-modified glassy carbon electrode between -0.4 V and -0.8 V. The deposition was performed for 150 s, as shown in Figure 3. All measurements were conducted under the optimal conditions. At potentials below -0.6 V, an elevated current plateau was discerned for the ErGO-modified glassy carbon electrode, which is linked to the comprehensive coverage of the surface. This phenomenon can be attributed to the formation of multiple layers of EDTA on the electrode. For this working electrode, multiple peaks are observed at potentials other than -0.7 V, which also show comparable current, thus demonstrating the successful determination of trace Pb (II). At potentials lower than -0.8 V, the reduction of the proton to hydrogen (H_2) commences at the working electrode. Consequently, the impact of the deposition potential on the working electrode in response to Pb (II) was effectively discerned, and –0.7 V was identified as the optimal deposition potential.

C. *Sensing Studies*

The detection limit for Pb (II) was found to be 1 ppb using square wave stripping voltammetry and 0.3 M nitric acid as the electrolyte. The electrolyte (20 mL) was placed in a voltametric cell and purged with nitrogen for one minute to remove any dissolved oxygen.

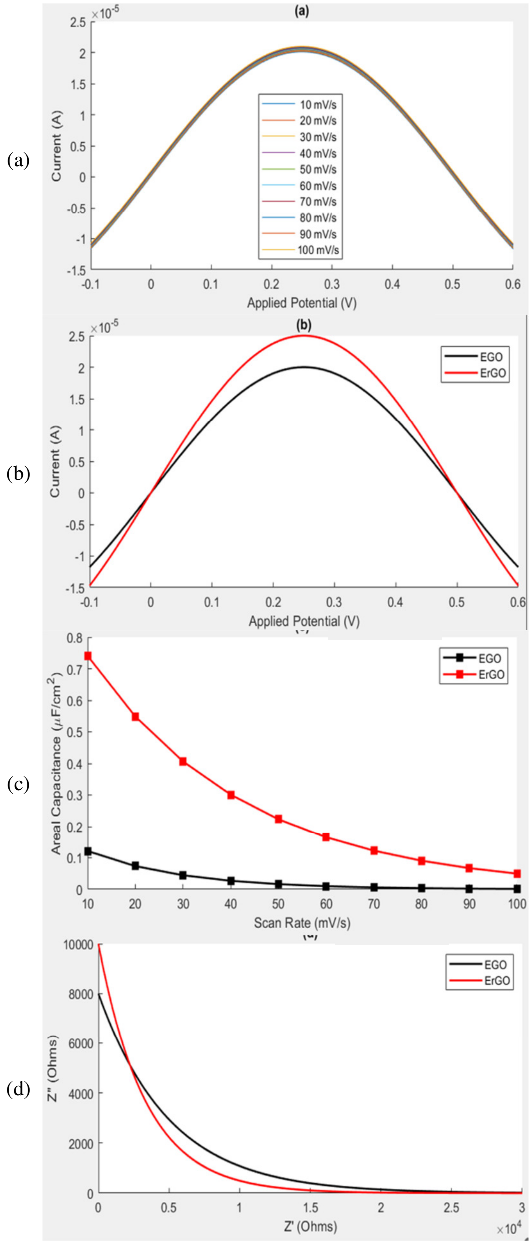

Fig. 2. Electrochemical characteristics (a) effect of scan rate on CV characteristics of ErGO nanocomposite, (b) comparison of CV characteristics of EGO and ErGO nanocomposite, (c) comparative effect of scan rate on areal capacitance of EGO and ErGO nanocomposite and (d) comparison of frequency response analysis of EGO and ErGO nanocomposite.

Stock solutions of 10 ppm Pb (II) were prepared, and 10 ppb of Pb (II) were added for the standard addition. Additionally, 20 µL of 10 ppm Pb (II) were added to 20 mL of electrolyte in each experiment. The analysis of Pb (II) involves two distinct stages: deposition and stripping. The deposition step was performed at a potential of -0.7 V for 150 seconds, after which the stripping step was initiated. This was achieved by applying a square wave with a frequency of 40 Hz and an amplitude of 25 mV in the potential range of -0.7 V to -0.2 V, with a step potential of 10 mV.

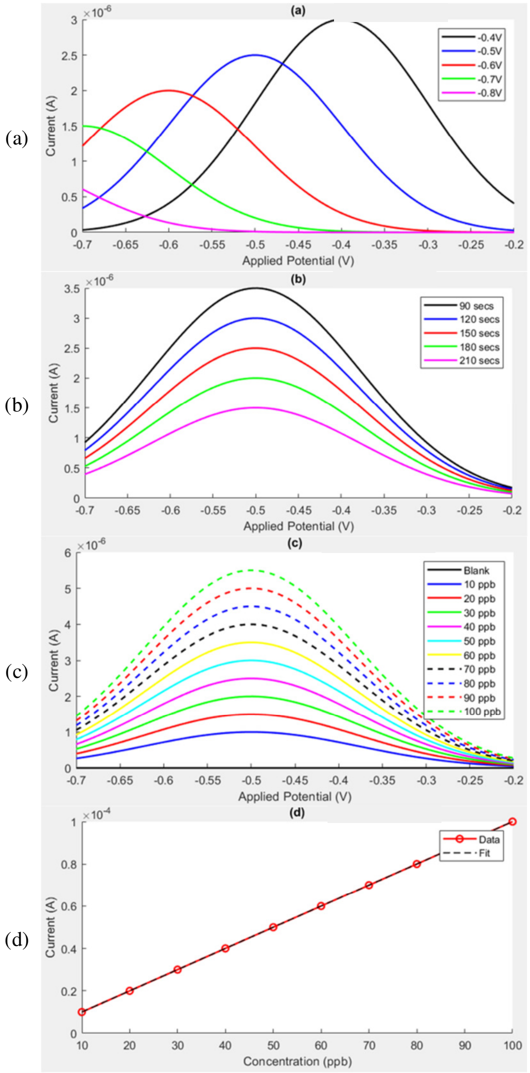

Fig. 3. Sensing studies towards Pb (II),(a) effect of deposition potential on 50 ppb Pb (II) concentration,(b) effect of deposition time on 50 ppb Pb (II) concentration,(c) effect of Pb (II) concentration at optimized sensing parameters, and (d) analytical characteristics of ErGO nanocomposite towards Pb (II) in the 10-100ppb range.

Subsequently, the current response of the ErGO-modified glassy carbon electrode to varying Pb (II) concentrations (10–100 ppb) was documented under optimal conditions, as shown in Figure 3. The peak current exhibited a linear increase in response to the increment of Pb (II), which is likely attributable to the enhanced availability of electroactive Pb (II) species in the solution. The notable increase in current is likely attributable to the adsorption of Pb (II) onto the ErGO-modified GC electrode, whereby the adsorbed layer remained unoxidized. This behavior is likely to result in a linear increase in current with increasing Pb (II) concentration. The experimental determination of the calibration range yielded a value of 10–100 ppb, while the sensitivity was found to be 38.2 nA/ppb. The theoretical limit of detection, calculated as 3 × slope/standard deviation, was determined to be 1.61 ppb. In comparison to the majority of electrochemical probes, the current chemosensory system is more straightforward, cost-effective, sensitive and selective.

D. Piezoresistive MEMS Cantilever Platform for Pb (II) Estimation in Potable Water

The single-layer piezoresistive cantilever, based on a MEMS cantilever, was designed to mimic the structure of a diving board. When compressive and tensile stresses are applied, the cantilever exhibits a deflection. The stress experienced by the cantilever results in piezoelectric resistance, which is quantified as a change in resistance between two points in contact with the nano-material. This process ultimately converts nano-mechanical motion into electrical signals, thereby enabling precise measurements to be made. A high-affinity selective receptor for Pb (II) ions is chemically attached to the surface of the cantilever. To reduce the occurrence of false signals, the Pb (II) ions present in the water solution are adsorbed on the cantilever surface by depositing a receptor layer on the cantilever. The water sample presumed to contain Pb (II) ions is placed into the analyte chamber. By subjecting the sample to heat generated by the platform's temperature control system, the Pb (II) ions are vaporized and transferred from the liquid to the vapor phase into the detection chamber. The vapors are pulsed into the detection chamber via mass flow controllers. These controllers are designed to regulate specific types of flow, thereby ensuring accuracy and consistency in flow rate. The vapours of contaminants are transported by nitrogen in order to prevent interaction during the detection process. In the detection chamber, the functionalized cantilever is exposed to vapors containing Pb (II) ions. The Pb (II) ions physically adsorb or chemically interact with the selective receptor layer on the cantilever surface, thereby inducing electrostatic surface stress and causing the cantilever to bend. The Pb (II) cation binds to piezoresistive nanofilaments, thereby generating mechanical strain in the piezoresistive element and resulting in a change in resistance. The graphical representation of the variation in resistance over time allows for the real-time detection of the presence or concentration of Pb (II) ions in the water sample. The platform provides optimized experimental conditions, thereby enhancing the sensitivity and selectivity of the lead sensor. It is thus essential to control the temperature of both the cantilever reaction chamber and the analyte vial in order to guarantee optimal measurement precision, which is dependent on the adsorption of Pb (II) ions on the cantilever surface. The system records all monitoring data, which can be examined subsequent to the conclusion of the experiment. The device's internal memory is capable of storing up to 40 hours of data in Comma-separated Values (CSV) format, which can then be downloaded, analyzed and interpreted at a later stage. The PC software oversees the experimental process, generates real-time graphs, and collates the data in an accessible and straightforward manner. The determination of Pb (II) is performed with an auto-calibration function, thereby ensuring the consistency and reproducibility of the measurements. Moreover, the system can be adapted for use with alternative carrier gases, thereby extending its versatility to encompass a more expansive range of potential applications. The process is capable of detecting Pb (II) in drinking water, and it may be developed further to create a range of new applications and products based on cantilever technology. This functionality is enabled by the nature of the functionalization layer, which is the sole component of the device that requires customization for specific analytes. Consequently, this platform can be employed for a multitude of environmental and industrial processes by simply switching the cantilever. The technology will permit the sensitive and straightforward detection of heavy metals in water, thereby enhancing water quality and public health status. The fully integrated arrangement, which is supported by VEGA, features reliable real-time monitoring and analysis for Volatile Organic Compounds (VOCs) and gases, as well as for dissolved Pb (II) in potable water. The latter is of particular importance globally, as it is a key indicator of water quality. The arrangement also employs reliable methodologies for Pb (II) sensing, which is based on piezoresistive MEMS technology. Furthermore, it is capable of monitoring and analyzing ceiling-air for VOCs and gases, which is a crucial aspect of ensuring the safety of drinking water.

E. Coating and Testing of ErGO on Microcantilever

The cantilevers are affixed to a Printed Circuit Board (PCB) through a wire bonding process. The cantilevers are enclosed in a liquid cell, which is equipped with an inlet and outlet for the regulation of liquid flow. The system is equipped with a dual-channel syringe pump, capable of delivering a flow rate that ranges from 55 µL/hr to 37 ml/min. The syringe pump is equipped with the capability to connect two syringes, which can be operated independently. The range of resistance that can be detected by the cantilever is from 27 kΩ to 90 kΩ. The system is battery-operated and incorporates a data storage facility for the data generated during the course of the experiment. Figure 4 shows a block diagram of the Omnicant cantilever, indicating the connections and pathways. The experiments are downloaded to the personal computer (PC) using the PC software in CSV format, and Figure 5 show the configuration for determining the resistance change in the piezoresistive cantilever. The objective is to calculate the resistance change in the piezoresistive cantilever. The software offers the option of calibrating the cantilever and initiating the experiment. The data can be observed in real time in the form of a graph, as shown in Figure 6, which presents the resistance plot of the piezoresistive cantilever for Pb detection.

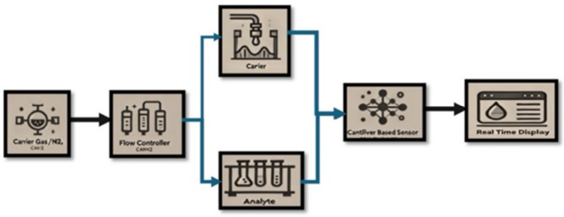

Fig. 4. Block diagram of the mnicant cantilever.

Fig. 5. Setup for determining the resistance change in piezoresistive cantilever.

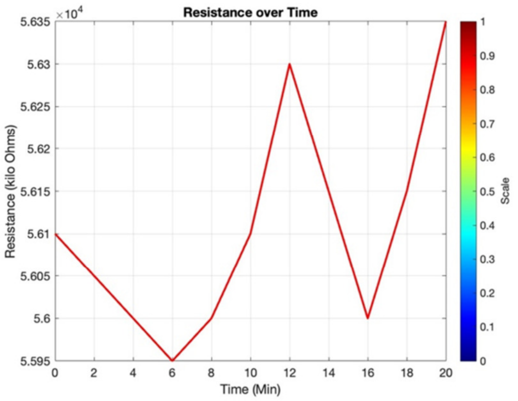

Fig. 6. Resistance plot of the piezoresistive cantilever for Pb(II) detection.

F. Determination of Trace Concentrations of Lead with ErGO

The fabricated cantilever is coated with ErGO, and the coating structure is observed under the Omnicant. The observed variations in resistance were found to be comparable to the values determined from the chemical analysis. Table I presents a comparative analysis of the readings obtained from the proposed single-layer piezoresistive cantilever and the electrochemical chemosensory platform using an ErGO coating on the electrode for the determination of Pb (II) traces in drinking water. A comparison of the outputs of the proposed piezoresistive cantilever sensor and the conventional electrochemical chemosensory system for the detection of Pb (II) in potable water is presented in Table I. The sensor output, expressed in microamperes (µA), was measured at varying concentrations of Pb (II). A similar comparison reveals that the new sensor exhibits markedly enhanced sensitivity and accuracy across a range of concentrations, thereby outperforming the conventional sensor. This comparison was crucial for validating the performance and efficacy of the recently developed sensor, which exhibited a validation value of approximately 10%. The piezoresistive sensor demonstrated superior sensitivity compared to the electrochemical sensory system.

TABLE I. COMPARISON OF OMNICANT AND ELECTROCHEMICAL CHEMOSENSORY SYSTEM OUTPUT

S.No.	**Pb(II) Concentration (ppb)**	**Output due to proposed piezoresistive cantilever (µA)**	**Output due to electrochemical chemosensory system (µA)**
1	10	1.57	1.4
2	20	1.88	1.6
3	30	2.23	2.1
4	40	2.54	2.4
5	50	2.78	2.6
6	60	3.23	3.1
7	70	3.59	3.5
8	80	3.88	3.8
9	90	4.43	4.2
10	100	4.79	4.6

IV. CONCLUSIONS

The electrochemical chemosensory system using ErGO (ethylene diamine tetra acetic acid reduced graphene oxide) displays high sensitivity for the detection of Pb (II) ions in water. The extensive detection range is extended by a calculated theoretical limit of 1.61 ppb, which allows the anti-MCH-I nanobody, in principle, to exceed the World Health Organization (WHO) recommended level for mercury ions by at least tenfold. These low-cost, straightforward-to-deploy, highly sensitive systems are ideal for monitoring lead contamination in drinking water. At present, techniques such as Atomic Absorption Spectroscopy (AAS), Inductively Coupled Plasma Mass Spectrometry (ICP-MS), and Anodic Stripping Voltammetry (ASV) are the established methods for the sensitive detection of metal ions. However, these methods are not without significant drawbacks. However, these techniques often necessitate the use of large sample volumes and complex, time-consuming protocols, which are costly to maintain and require expensive equipment. Furthermore, the experiment requires precise conditions to ensure accurate results. The proposed piezoresistive microcantilever sensor offers a promising avenue for ultra-low detection of lead ions (Pb (II)) in drinking water. It is intended that this sensor will be used as a component of a Total Analytical System (TAS), and it is capable of performing quantitatively at a level comparable to that of traditional chemical analysis techniques, with a variation of approximately 10%. This microcantilever sensor represents a significant advancement in the field of environmental monitoring technologies. This provides an accessible method for establishing benchmarks when examining lead contamination in municipal water supplies, facilitating the implementation of a high-throughput test for one of the most pressing public health concerns related to water. The integration of these sensor devices into our monitoring systems will significantly enhance our ability to ensure the safety and quality of drinking water, thereby protecting public health.

It can be argued that microcantilever sensor technology represents a significant advancement in water quality monitoring, offering the potential for integration with IoT devices for the acquisition of real-time data and mobile applications for remote tracking. Further development may result in the creation of a sensor capable of detecting multiple analytes, thereby increasing its versatility and suitability for monitoring a range of contaminants. Research is focused on the development of sensors with enhanced sensitivity, selectivity, and long-term robustness. The expansion of deployment in both urban and rural settings will facilitate the widest possible access to clean water monitoring. It will facilitate the attainment of regulatory compliance and the advancement of community education initiatives, thereby promoting safer drinking water standards on a global scale.

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