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Performance analysis of gold/graphene oxide surface plasmon resonance interface for trace level detection of cadmium ions

Muhammad Fahmi Anuar[1], **Yap Wing Fen**[1,2,*], **Josephine Liew Ying Chyi**[1,2], **Tan Sin Tee**[2], **Wan Mohd Ebtisyam Mustaqim Mohd Daniyal**[3] and **Mohd Adzir Mahdi**[4]

[1] Functional Nanotechnology Devices Laboratory (FNDL), Institute of Nanoscience and Nanotechnology (ION2) UPM 43400 Serdang Selangor, Malaysia
[2] Department of Physics, Faculty of Science, Universiti Putra Malaysia 43400 UPM Serdang Selangor, Malaysia
[3] Physics section, School of Distance Education, Universiti Sains Malaysia (USM), 11800, Minden, Pulau Pinang, Malaysia
[4] Wireless and Photonics Networks, Faculty of Engineering, Universiti Putra Malaysia, 43400 UPM Serdang, Selangor, Malaysia
[*] Author to whom any correspondence should be addressed.

E-mail: yapwingfen@upm.edu.my

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Abstract

This study explores the integration of graphene oxide (GO) into a gold surface to form a gold/graphene oxide (Au/GO) bilayer as the sensing interface in the surface plasmon resonance (SPR) sensor for cadmium ions (Cd^{2+}) detection in aqueous solutions. The sensor displays a solid reaction within a wide concentration range of 0–1000 ppm, making it suited for environmental and industrial monitoring. The sensor demonstrated a sensitivity of 0.04°/ppm, with a limit of detection (LOD) of 0.37 ppm and a limit of quantification (LOQ) of 0.01 ppm. In addition, the high binding affinity constant (0.6037/ppm) indicates strong interactions between Cd^{2+} ions and the Au/GO sensing interface. Additional performance metrics, including detection accuracy (DA) and a signal-to-noise ratio (SNR) were also investigated. These results highlight the potential of the Au/GO-based SPR platform as a promising tool for real-time, label-free monitoring of cadmium contamination in environmental samples.

1. Introduction

Water is one of the most vital natural resources, essential for sustaining humans, plants, and animals' lives. However, rapid industrialisation, agricultural expansion, and urban development have significantly increased the risk of water contamination [1]. Pollutants such as pesticides, dyes, heavy metals, chemicals, and pathogens often enter water sources through surface runoff [2], improper chemical disposal [3], and untreated industrial effluents [4]. Such contamination not only makes water unsafe for human use [5] but also causes serious harm to the environment and aquatic ecosystem [6].

Among various detrimental pollutants, heavy metal ions are some of the most hazardous due to their toxicity, persistence in the environment, and bio-accumulative nature [7]. Notably, lead (Pb^{2+}) [8], chromium (Cr^{6+}) [9], mercury (Hg^{2+}) [10], and cadmium (Cd^{2+}) [11] are recognised as particularly harmful and have consistently been the focus of numerous studies aimed at their detection and monitoring [12]. For instance, cadmium ions pose a significant health risk as they readily bind to functional groups and accumulate in biological tissues, particularly in the kidney [13] and cause subsequent tissue damage [14]. They may also disrupt the protein and enzyme activity through heavy metal poisoning and bioaccumulation [15]. Cadmium ions have also been shown to inhibit several key enzymatic processes, indirectly contributing to the formation of free radical oxygen radical species [16], which are associated with tumour development [17]. Moreover, it exerts detrimental effects on the reproductive system, reducing fertility and contributing to sexual dysfunction [18], ultimately leading to physiological disturbances and functional impairments in both males and females [19]. Given its severe impact on human health and the environment, sensitive and reliable detection of Cd^{2+} in water sources is essential to ensure the safety and sustainability of water resources.

Traditional water monitoring methods, including atomic absorption spectroscopy (AAS) and inductively coupled plasma mass spectrometry (ICP MS), provide high sensitivity measurement but often involve complex procedures and time-consuming laboratory analysis. In recent years, optical sensor-based Surface Plasmon Resonance (SPR) has emerged as a promising alternative that offers a reliable, highly efficient, high-sensitivity performance and label-free detection method [20]. SPR technology has been widely applied in various fields, mainly for precise sensing applications in clinical [21] and environmental fields [22]. Integrating SPR sensor into the water monitoring system enables real-time detection of contaminants such as cadmium ions, facilitating early intervention and supporting the provision of safe drinking water.

Despite its extensive capabilities, reliable detection of target analytes at trace-level concentrations remains a significant limitation of conventional SPR without any surface modification. Previous studies have attempted to overcome this challenge by incorporating advanced materials such as graphene oxide [23], carbon quantum dots [24], and perovskite [25], which indeed improved the sensor performance but often required complex and time-consuming fabrication processes. While these nanomaterials offer clear advantages in terms of enhanced sensitivity and selectivity, they also present drawbacks such as increased fabrication costs, reduced reproducibility, and limited scalability for practical applications.

Among these, graphene oxide (GO) has emerged as a promising candidate for enhancing the performance of SPR sensors, particularly due to its unique properties such as a large specific surface area, good adsorption capacities, outstanding photoluminescence, facile surface functionalisation, good biocompatibility, and low toxicity [26]. This nanomaterial has been widely applied across various fields, including drug delivery [27], as bio-imaging tools [28], dental applications [29], and chemical sensing [30]. When integrated onto the surface of the gold thin film, these nanomaterials can significantly improve the sensor performance. In the present work, the gold thin film was functionalised with the GO as a single-layer surface modifier to enhance the detection of Cd^{2+}.

The novelty of the present work lies in the use of a single-layer GO as a surface modifier on a gold thin film for SPR-based Cd^{2+} detection. Unlike previous studies that employed complex composite or multilayer nanomaterials, this approach demonstrates that a simple GO layer can achieve effective detection at environmentally relevant concentrations, simplifying fabrication while maintaining competitive sensitivity and limit of detection. This simple design overcomes the challenge of detecting very low levels of Cd^{2+} with conventional SPR sensors and provides a practical approach for monitoring water quality.

2. Materials and methodology

2.1. Materials

Graphene oxide and a stock solution of 1000 ppm cadmium (Cd^{2+}) ions were purchased from (Sigma Aldrich, USA), and Fisher Scientific, (United Kingdom) respectively. Glass substrates (24 mm × 24 mm) were bought from Menzel-Glaser (Germany) and deionised water. Cd^{2+} stock solution were diluted by using the dilution formula $M_1V_1 = M_2V_2$ using deionised water to produce different concentrations of Cd^{2+} solutions.

2.2. Fabrication of sensing layer

A 24 mm × 24 mm glass substrate from Menzel-Glaser with 0.13–0.16 mm thickness was used as the sensor platform. To ensure a consistent metal interface sensor layer, the glass surface was cleaned with acetone before it was coated with a layer of gold using a sputtering machine. Then, to create a sensor film and investigate its ability to detect Cd^{2+}, a thin layer of GO was added to the pristine gold thin film using the spin coating technique. The prepared sensor films were labelled and identified as Au/GO.

2.3. Surface plasmon resonance sensor

A specially constructed setup utilising the Kretschmann configuration [31], as shown in figure 1, was used to conduct the SPR experiment. The setup consists of several equipment such as a laser, optical chopper, polariser, stepper motor, photodiode and a lock-in amplifier. The experiment used a He-Ne laser (632.8 nm), which was directed through a prism and reflected after striking the thin film. To obtain a different incident angle of measurement, MATLAB software was used to operate the rotational stage on which the prism and thin films were installed. The reflected light intensity will be collected by a photodiode as a function of angle, and the signal will be converted into measurable data through a lock-in amplifier. The thin film in this experiment was positioned between the prism and the dielectric layer, which in this case was a solution of cadmium ions. The analyte chamber was filled with a solution of cadmium ions at varying concentrations sequentially, and the intensity of the reflected beam was measured. Every measurement was repeated three times to ensure accuracy and reproducibility.

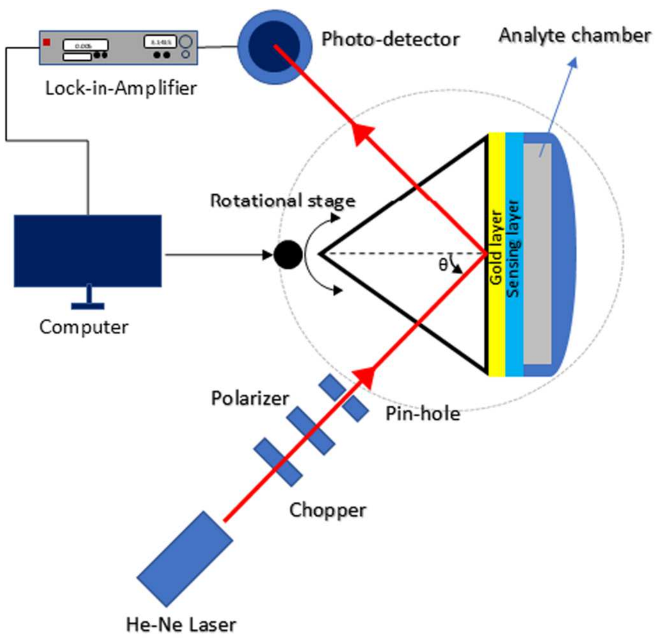

Figure 1. Schematic diagram of Kretschmann-based surface plasmon resonance spectroscopy used for detection of Cd^{2+} for this work.

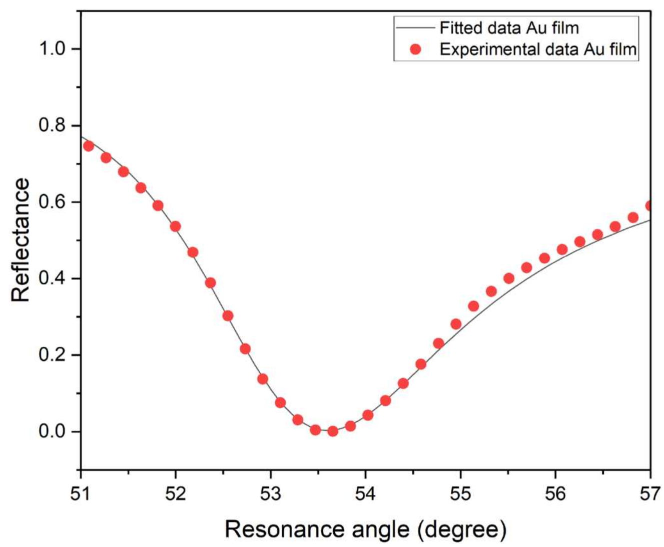

Figure 2. SPR reflectance curves of the gold thin film in deionised water (DW), showing experimental data (points) and theoretical fitting (solid line). The fitting was performed using Fresnel's equations, yielding a gold layer thickness of 40.10 nm.

3. Results and discussion

This section presents the experimental results obtained from the development of the SPR sensor, along with a comprehensive discussion of their implications. The sensor's performance of the developed SPR sensor was evaluated based on key parameters such as SPR angle shift, sensitivity, signal-to-noise ratio (SNR), and detection accuracy across varying concentrations of Cd^{2+} ions.

A critical factor governing sensor performance is the thickness of the metallic interface and the sensing layer. The SPR experimental data was fitted theoretically based on Fresnel's equation, yielding a gold layer thickness of 40.10 nm for the sensor in contact with deionised water (DW), as illustrated in figure 2. This value falls within the range of the optimal range reported for efficient SPR excitation [32], thereby validating the fabrication process.

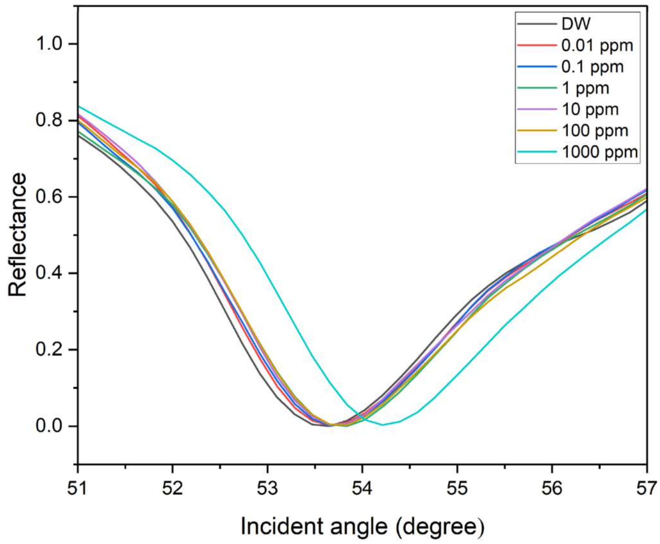

Figure 3. The SPR reflectivity curve for the Au thin film shows no noticeable SPR angle shift across the Cd^{2+} concentration range of 0–100 ppm.

The SPR response was first characterised using a gold thin film in contact with deionised water (DW) to establish a reference resonance angle. Figure 3 shows the SPR curve, plotted with the incident angle on the x-axis and the reflectance intensity on the y-axis. The resonance angle is defined by the minimum points of the dip in the curve, which correspond to the phenomenon of surface plasmon resonance. At this specific angle, the momentum of the incident photon is equal to the surface plasmon wave. Consequently, the reflectance intensity reaches its minimum due to the efficient transfer of energy from the incident light to the surface plasmon oscillations at the gold interface [33].

The reflectance curve for the reference sample (DW) exhibits a clear resonance angle at 53.650°. This angle served as the baseline for monitoring subsequent shifts induced upon exposure to different concentrations of Cd^{2+}. Notably, the SPR reflectivity curve showed no significant shift in the resonance angle for concentrations below 1000 ppm concentration. This finding suggests that the interaction between the metal ions and the bare gold surface is minimal and resulting in no measurable change in the local refractive index, indicating limited binding at lower concentrations. This lack of response aligns with expectations, as gold alone lacks sufficient affinity for Cd^{2+} binding at trace levels and therefore does not significantly affect the optical properties of the thin film [34]. The evidence presented gives the idea that detection of Cd^{2+} at low concentrations using gold is not practicable due to limited surface reactivity and the refractive index remaining unaltered or being nearly identical to that of deionised water [35].

The SPR characterisation was extended to the Au/GO thin film, beginning with a baseline measurement of deionised water (DW). The resonance angle of the Au/GO thin film in contact with DW was established at 54.707°, a value notably higher than that of the bare gold film. This angular increase is attributable to the higher refractive index introduced by the graphene oxide (GO) layer. This baseline measurement served as the critical reference for subsequent evaluations of the sensor's response to varying Cd^{2+} ion concentrations.

The thickness of the GO layer was determined to be 5.41 nm through fitting of the experimental SPR data with the theoretical model. Figure 4 illustrated the close agreement between the simulated and experimental reflectance curves, validating the obtained thickness value.

As illustrated in figure 5, the simulation shows the influence of GO layer thickness on the SPR curve. An increased thickness of the GO layer results in a broader SPR reflectance curve, degrading the sensor sensitivity and detection accuracy. Conversely, a thinner sensing layer leads to limited binding interaction between the sensing surface and the target analyte [36]. Therefore, both factors need to be considered to obtain the optimum thickness of the sensing layer to get the best SPR signal for better sensitivity and accuracy.

Following this, a systematic series of measurements was conducted by introducing Cd^{2+} solutions of increasing concentrations. The resultant shift in the resonance angle was monitored relative to the DW baseline. The reflectance spectra are illustrated in figure 6, and the difference between the SPR angle for each Cd^{2+} concentration and that of deionised water (DW) was calculated and documented, as presented in table 1.

Figure 6 demonstrates a positive correlation between the Cd^{2+} concentration and the resonance angle, which shifts to higher values from 54.728° at 0.01 ppm to 56.352° at 1000 ppm, with the highest increment of

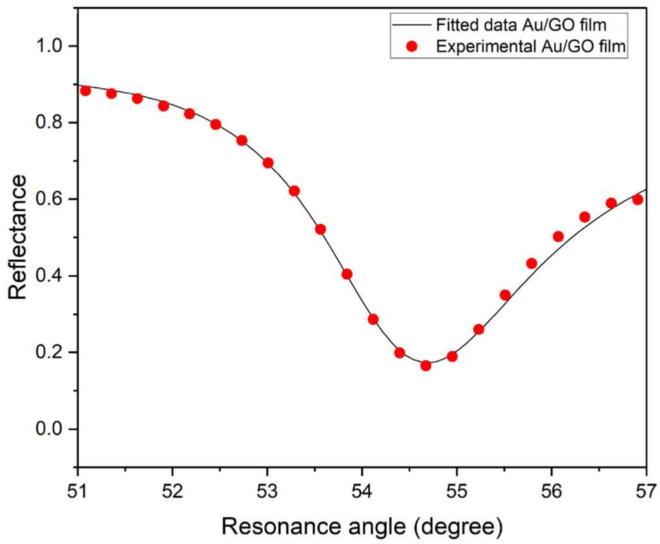

Figure 4. SPR reflectance curves of the Au/GO thin film in deionised water (DW), showing experimental data (points) and theoretical fitting (solid line). The fitting was performed using Fresnel's equations, yielding a GO layer thickness of 5.41 nm.

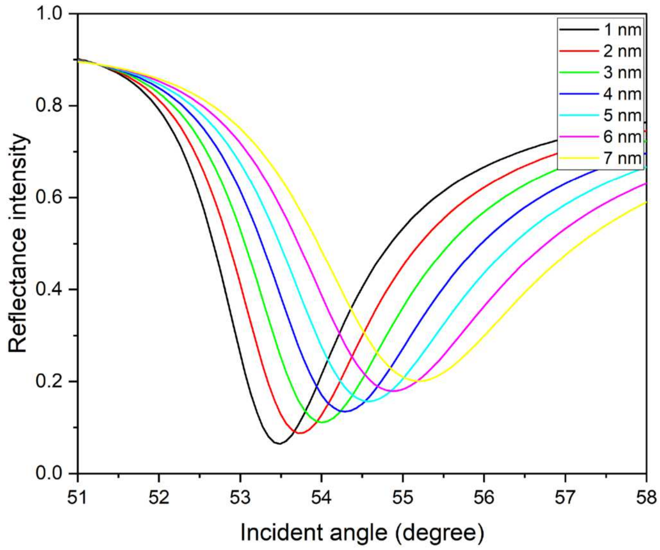

Figure 5. Simulation-based optimisation of SPR response for different thicknesses of GO layers. Increasing GO thickness results in broader SPR curves, indicating loss of sensitivity and signal accuracy.

1.645°. This progressive angular shift is attributed to the interaction between the sensing layer and the Cd^{2+} solution that changes the refractive index near the metal surface of the thin film.

A progressive decrease in reflectance in the resonance dip was observed with increasing Cd^{2+} concentration. This phenomenon is attributed to the increase in the local refractive index near the metal surface upon binding interaction between the sensing layer and target ions and thus resulted in lower reflectance [37]. For a given wavelength and incident light, energy conservation requires the total of these three qualities (transmittance, reflectance, and absorptance) to equal one. The minimum reflectance at this dip corresponds to the strongest coupling between incident photons and surface plasmons at the metal-dielectric interface [38]. This dip signifies the energy transfer to surface plasmon [39]. A deeper resonance dip implies enhanced light energy absorption, which can result from improved surface plasmon excitation or from strong exciton–plasmon coupling [40], particularly when functional materials such as graphene oxide are introduced. Consequently, at higher Cd^{2+} concentrations, the increased interaction between the sensing layer and the analyte leads to greater absorption, thereby reducing the measured reflectance.

Figure 7 depicts the linear regression graph, plotting the SPR angle shift ($\Delta\theta$) against the concentration of Cd^{2+}. A good linear relationship between the Au/GO and Cd^{2+} is observed across the concentration range of 0

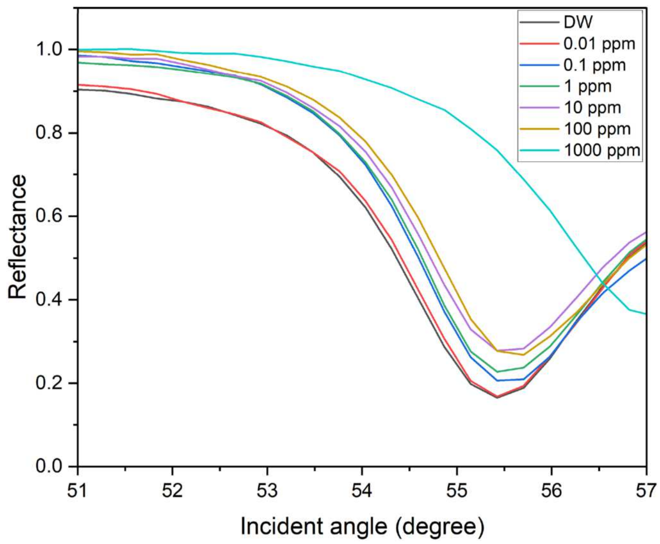

Figure 6. The SPR reflectivity curve of Au/GO thin film observed a slight shift to the right in the resonance angle for detection of Cd^{2+} within the range of 0–100 ppm.

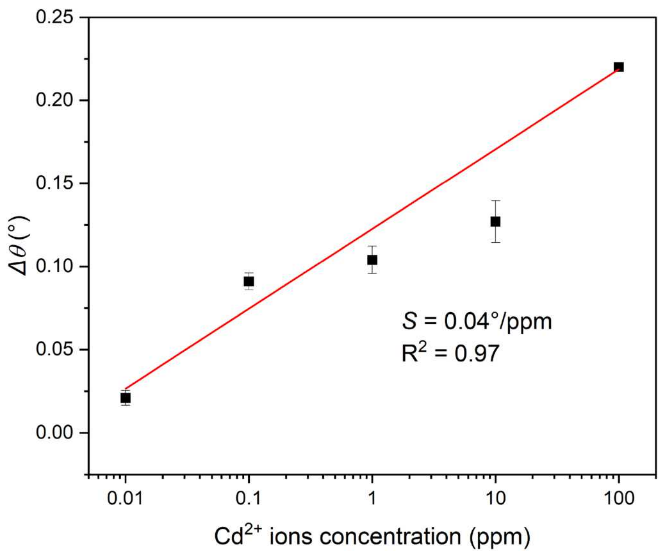

Figure 7. Linear regression graph of the Au/GO sensor for Cd^{2+} detection in the concentration range of 0–100 ppm, where the slope (S) indicates the sensor's sensitivity. Error bars denote the standard deviation from three repeated measurements.

Table 1. The SPR angle and angle shift response of Au/GO thin film in contact with varying concentrations of cadmium ions from 0–1000 ppm.

Concentrations of Cd^{2+} (ppm)	SPR angle (°)	SPR angle shift, $\Delta\theta$ (°)
0	54.707	0.000
0.01	54.728	0.021
0.1	54.798	0.091
1	54.811	0.104
10	54.834	0.127
100	54.927	0.220
1000	56.352	1.645

Table 2. Comparison of detection range, sensitivity, and the detection limit of previously reported SPR-based cadmium (Cd^{2+}) sensors with the present Au/GO sensor.

Sensing layer	Range (ppm)	Sensitivity (°/ppm)	LOQ/LOD (ppm)	References
Urease-functionalised N-succinimidyl 3-(2-pyridyldithiol) propionate (SPDP)	0–10	Linear	2.0/–	[43]
Metallothionein	0.22–134.89	Linear	0.22/–	[44]
pHEMA thin film based SPR	0.001–0.05	—	0.001/0.0005	[45]
Au/TiO_2-CNC composite	0–1000	0.48	0.01/–	[37]
Au/GO	0–1000	0.04	0.01/0.37	This work

to 100 ppm. The sensitivity of the sensor, defined by the slope of this linear fit, was quantified at 0.04°/ppm. The linear regression yielded a strong correlation coefficient of 0.97, confirming the excellent reliability of the sensor's response within this range. These results confirm that the functionalisation of gold film with GO enhances the detection capability of the SPR sensor for cadmium ions detection.

The limit of detection (LOD) value represents the lowest concentration of an analyte that can be reliably distinguished from a blank measurement, with a defined level of confidence [41]. Furthermore, the limit of LOD of this proposed sensor was calculated by using equation (1) [42] :

$$LOD = \frac{3\sigma}{S} \tag{1}$$

where σ is the standard deviation of the reflectance intensity measured from the DW solution and S is the slope of the calibration or the sensitivity of the proposed sensor. For the concentration range of 0–100 ppm, the LOD of the Au/GO was determined to be 0.37 ppm, while the limit of quantification (LOQ), defined as the lowest concentration detected in this work, was 0.01 ppm

Table 2 presents a comparative summary of previously reported SPR-based Cd^{2+} sensors, outlining their detection range, sensitivity, and detection limits in relation to the present work. Sensors such as metallothionein-based films have achieved a very low detection limit of 0.22 ppm, while Au/TiO_2–CNC composites have demonstrated both high sensitivity (0.48°/ppm) and an ultra-low LOQ of 0.01 ppm. In contrast, the Au/GO thin film proposed in this work exhibited a sensitivity of 0.04°/ppm and an LOD of 0.37 ppm with LOQ of 0.01 ppm, which, although not as low as some advanced nanocomposite systems, remains within a practical and competitive range for Cd^{2+} detection. Importantly, this performance was achieved using a streamlined single layer GO structure, without the need for complex functionalization or multilayer composites. These results indicate that the Au/GO platform offers a favourable compromise between sensitivity, detection capability, and fabrication simplicity, making it a viable option for cost-effective and scalable sensor application.

The analysis of binding interactions between the analyte and the sensor surface is a critical aspect of SPR sensor characterisation. Fitting the experimental data to adsorption isotherm models enables the extraction of the quantitative binding parameters, offering valuable insights into the nature and strength of the interaction. In this context, the binding affinity of the Au/GO sensor was evaluated by using conventional isotherm models such as Langmuir and Sips. The Langmuir isotherm model was given by equation (2).

$$\Delta\theta = \frac{\Delta\theta \text{ max } C}{\frac{1}{K} + C} \tag{2}$$

where $\Delta\theta$ is the SPR angle shift, $\Delta\theta_{max}$ is the maximum SPR angle shift at saturation, C denotes the concentration of the analyte (Cd^{2+}), and K is the binding affinity constant that reflects the strength of interaction between the analyte and the sensor surface. On the other hand, the Sips isotherm model can be expressed as equation (3).

$$\Delta\theta = \frac{\Delta\theta \text{ max}(KsC)^n}{1 + (KsC)^n} \tag{3}$$

where K_s is the Sips affinity constant and n is the heterogeneity index of the system. The experimental data were fitted to the Sips isotherm model, and the goodness of fit was evaluated by comparing the coefficients of determination (R^2) from both the Langmuir and Sips models. The higher value indicates a better fit of the data with the corresponding model, thus providing insight into the nature and uniformity of the analyte and sensor layer interaction.

The plotted figure 8 presents the experimental data fitted to the isotherm model of Langmuir and Sips. Based on the R^2, the Sips model provided the best fit to the experimental data with R^2 of 0.98. The Sips affinity

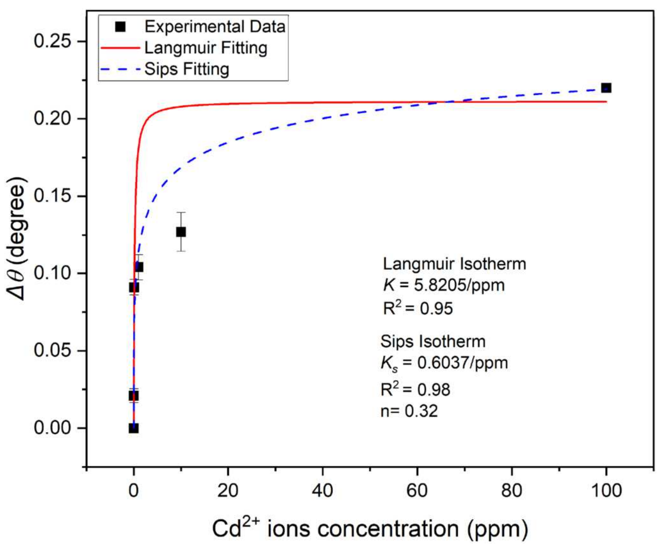

Figure 8. Comparison of Sips and Langmuir isotherm fits to the SPR response of the Au/GO thin film in contact with Cd^{2+} (0–100 ppm). The Sips model provides a superior fit to the experimental data, and the affinity constant (K_s) can be determined from the graph. Error bars denote the standard deviation from three repeated measurements.

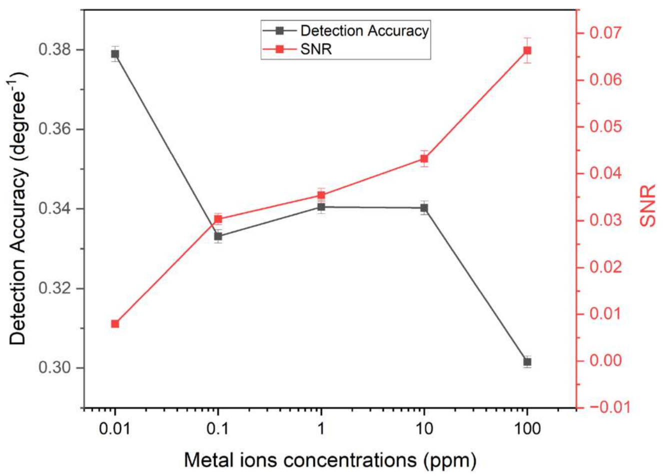

Figure 9. Detection accuracy (DA) of the Au/GO sensor decreases while the signal-to-noise ratio (SNR) increases with higher Cd^{2+} concentrations from 0.01 to 100 ppm. Error bars denote the standard deviation from three repeated measurements.

constant, K_s was determined to be 0.6037/ppm. In addition, the n value obtained was 0.32 suggesting high heterogeneity relationships at the interface of the sensor surface [46].

The detection accuracy (DA) of the SPR sensor was characterised by measuring the full width at half maximum (FWHM) of the reflectance curve. FWHM refers to the width of the reflectance curve at half of the maximum intensity. Since the detection accuracy is inversely proportional to the FWHM of the SPR reflectivity, a narrower FWHM indicates higher precision and improved detection accuracy [47]. It is to note that the FWHM values vary depending on the design and implementation of the SPR sensor [48]. Figure 9 presents a combination graph of the detection accuracy (DA) and signal-to-noise ratio (SNR) of the SPR between the Au/GO sensing film and the Cd^{2+} of different concentrations. Notably, the maximum DA value was observed to be at the lowest concentration of 0.01 ppm at 0.38/°, and the value of DA decreased as the concentration went up. This trend is attributed to the increase in the thickness of the sensing layer surface due to enhanced binding activity between the sensor and target at higher concentrations. As a result, the surface plasmon oscillation loses electron energy, leading to damping and broadening of the SPR curve [49].

In a sensor system, SNR is defined as the ratio of the desired signal strength in comparison to the unwanted or noise signal. In the case of SPR, the signal is the reflected light intensity or wavelength captured by a photodiode while the noise is the undesirable fluctuation of the background. As expected, the proposed sensor shows a relatively good SNR trend when exposed to Cd^{2+}, showing improved values at higher target Cd^{2+} concentrations. This enhancement is attributed to stronger binding interaction at the surface of the sensor [50]. In essence, increasing Cd^{2+} concentration led to an improvement in signal with better noise reduction [51]. Therefore, higher SNR values observed in this sensor indicate enhanced sensitivity, improved detection accuracy, and overall reliability in sensing performance [52].

In conclusion, the demonstrated Au/GO sensor performance towards detections of Cd^{2+} highlights its potential for real-world environmental monitoring applications. Its capability to detect Cd^{2+} and possibly other heavy metals in aqueous medium is essential towards a safer and cleaner environment. This SPR sensor offers several advantages including high sensitivity, label-free detection, and real-time monitoring. However, further modifications are necessary to enhance its detection performance, particularly at trace levels. Future works will focus on modifying the sensing interface with advanced sensing materials such as quantum dots and metal organic frameworks (MOF). These materials offer excellent adsorption properties, which are expected to enhance sensor performance, especially lower the detection limit. Thereby expanding the sensor's application in environmental monitoring and public health monitoring.

4. Conclusion

Overall, this work demonstrated the proposed SPR sensor (Au/GO) for potential detection of Cd^{2+} at different concentrations. The investigation was conducted to evaluate the performance of proposed sensors such as sensitivity, binding affinity, detection accuracy, and signal-to-noise ratio. The sensitivity of the proposed sensors was valued at 0.04°/ppm with the corresponding limit of detection (LOD) and limit of quantification (LOQ) of 0.37 ppm and 0.01 ppm, respectively. Furthermore, the proposed sensor with the target Cd^{2+} has a binding affinity constant, which was calculated based on the Sips isotherm model, K_s of 0.6037/ppm. Despite the lower detection accuracy at higher concentrations, the SNR value improved, indicating a good and dependable sensor film for detection. Hence, the proposed sensor demonstrates a promising capability for recognising Cd^{2+}, confirming its suitability for sensitive detection of Cd^{2+}.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Declaration of generative AI in scientific writing

The author does not use generative AI in the writing of this scientific writing.

Author contributions

Muhammad Fahmi Anuar
Formal analysis (lead), Investigation (lead), Methodology (equal), Writing – original draft (equal)

Yap Wing Fen 0000-0003-3475-7469
Conceptualization (equal), Supervision (lead), Writing – review & editing (lead)

Josephine Liew Ying Chyi 0000-0003-1577-4773
Methodology (supporting), Resources (supporting)

Tan Sin Tee
Resources (supporting), Supervision (supporting)

Wan Mohd Ebtisyam Mustaqim Mohd Daniyal 0000-0002-8047-2037
Formal analysis (supporting), Methodology (supporting)

Mohd Adzir Mahdi
Resources (supporting), Supervision (supporting)

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