



Original scientific paper

## Enhanced anodic stripping determination of Cd(II) using NH<sub>2</sub>-functionalized MOF and GO-modified glassy carbon electrode

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### Abstract

*In this work, an amino-functionalized metal-organic framework (NH<sub>2</sub>-functionalized MIL-101(Fe) MOF) and graphene oxide (GO) were employed to modify a glassy carbon electrode (GCE) to prepare a simple and sensitive electrochemical sensing platform for detecting Cd(II) ions in drinking water samples. It is important to note that the high surface area, porous structure, and amino-functional groups in the structure of MIL-101(Fe) MOF can significantly facilitate the enrichment and accumulation of Cd(II) ions at the GCE surface, while the utilization of GO enhances the electrochemical performance and conductivity of the modified electrode. Due to these facts, the NH<sub>2</sub>-functionalized MIL-101(Fe)/GO-modified GCE exhibited good ability for detecting Cd(II) in comparison with the unmodified GCE. The sensing capability of the NH<sub>2</sub>-functionalized MIL-101(Fe)/GO-modified GCE was evaluated using differential pulse anodic stripping voltammetry under optimized conditions. Based on quantitative measurements, the as-prepared sensing platform was capable of detecting Cd(II) with a detection limit of 0.004 μM and a linear response range of 0.01 to 12.0 μM. Importantly, the ability of NH<sub>2</sub>-functionalized MIL-101(Fe)/GO-modified GCE to determine Cd(II) in the real drinking water sample further confirms the practicability of the prepared sensing platform. These results demonstrate the feasibility of a simple, cost-effective electrochemical sensing platform for the sensitive determination of Cd(II) ions, contributing to environmental protection and human health.*

### Keywords

Environmental monitoring; water analysis; heavy metal ions; electrochemical sensor

### Introduction

Environmental surveillance is mainly directed to the orderly identification, measurement and assessment of contaminants possibly found in parts of the environment, such as ground, water and air. Pollution may comprise chemical pollutants, biological contaminants, microorganisms, and

radioactive materials, all of which can have adverse effects on ecosystems and human health. The global requirement for safe, fresh water is increasing constantly and so the importance of precise and permanent control over the quality of water has become even more important. The introduction of new pollution sources can significantly degrade water quality, posing a severe challenge to environmental protection and public health. Heavy metal ions (HMIs) are among the most serious environmental pollutants and have attracted increasing attention [1]. HMIs are very stable, bio-resistant and have high toxicity potential that could affect various living organisms. The principal origins of HMIs are linked to anthropogenic activities, such as industrial effluents, mining and smelting operations, discharge of urban wastewater, and agricultural practices using agrochemicals. Of all HMIs, arsenic, lead, cadmium, chromium, and mercury are most notable for their extraordinary toxicity and persistence in the environment [2].

In particular, Cd(II) is identified as one of the most toxic heavy metal ions (HMIs) that can cause equally hazardous effects at ppb level, and even below, including nephrotoxicity, bone demineralization syndrome, renal tubular dysfunction and life-threatening diseases such as cancer, heart attack and diabetes mostly through contaminated water and food [3, 4]. Although Cd is highly toxic and its health effects can be observed at very low concentrations, it is commonly used in diverse applications such as batteries, pigments, plastics, metal plating *etc.* [5]. Thus, accurate monitoring and quantification of Cd(II) in environmental samples are essential for risk evaluation and for developing viable strategies to reduce human exposure. Currently, there are many due analytical methods applied for Cd(II) measurement in the various samples which they can be attributed to atomic absorption spectrometry (AAS) [6], inductively coupled plasma-mass spectrometry (ICP-MS) [7], high performance liquid chromatography (HPLC) [8], colorimetry [9], fluorometry [10] and chemiluminescence [11]. However, most existing methods still face common challenges, including laboriousness, slow operation, and the use of large quantities of solvents, reagents, and sophisticated equipment. Consequently, a rapid and low-cost method for efficient Cd(II) analysis is in great demand [12].

Electrochemical detection has become a promising approach to HMIs due to its low cost, fast response, and high sensitivity [13,14]. They offer accurate and convenient means to quantify trace HMIs in complex matrices such as water, soil, and food [15]. The portability and ease of use further enhance the attractiveness of on-site, real-time detection [16]. Among available electrochemical techniques, stripping voltammetry has emerged as a highly efficient method for sensing and quantifying HMIs [17]. Stripping voltammetry involves a preconcentration step followed by an electrochemical determination and has been shown to increase sensitivity and limit of detection (LOD) from nanomolar to subnanomolar, even to picomolar levels [18, 19]. The ongoing growth of it guarantees effective means of environmental monitoring, pollution control and water quality standardization. In fact, stripping voltammetry, with the application of chemically modified electrodes, has demonstrated sensitivity and selectivity for HMIs [20]. Strong and selective binding to target analytes can be achieved by nanomaterials or specific materials deposited on the electrode surface, leading to increased sensitivity and selectivity, particularly in complex matrices such as environmental water samples.

Metal-organic frameworks (MOFs) are a class of porous crystalline materials with ordered, periodic structures [21]. These are formed by the self-assembly of metal ions with organic ligands, resulting in an infinite network. The exceptional and tunable functionalities of MOFs are largely determined by factors such as metal centers, organic linkers, and network topology. Pore size, surface area, chemical functionality and stability are among the key characteristics of MOF to be tailored, which in turn play an important role in the performance of MOFs in applications including

gas storage and separation [22], sensing applications [23], catalysis [24] and environmental treatment processes [25-27]. In terms of sensing, MOFs are considered as attractive materials for the modification of electrode surfaces in electrochemical sensor designs to detect various compounds due to their porosity, high specific surface area and structural versatility [28-30]. In addition, the presence of functional groups in MOF organic ligands confers strong complexing ability toward HMIs [31]. Therefore, it is important to determine HMIs based on amino (NH<sub>2</sub>)-functionalized MOFs as electrode materials. Most MOFs show strong potential for practical use; however, their relatively low electrical conductivity is a limiting factor for further applications, such as electrochemical sensors. Other functional materials, notably carbonaceous nanostructures, can be employed together with MOFs and lead to large improvements in their sensing performance due to the increase of electrical conductivity. Graphene oxide (GO) has attracted particular interest because of its fascinating two-dimensional (2D) nanomaterial structure and remarkable properties, such as a large surface area and high electrical conductivity [32-34]. Due to its attractive properties, GO is a promising candidate for coupling with MOFs to improve electrochemical performance [35,36].

The goal of this study is to create a straightforward, sensitive, and quick electrochemical sensor. This sensor is designed to detect Cd(II) ions in drinking water samples. It's based on NH<sub>2</sub>-functionalized MIL-101(Fe) MOF combined with GO to modify the glassy carbon electrode (GCE). The use of NH<sub>2</sub>-functionalized MIL-101(Fe) MOF along with GO improved the performance of the GCE for determining Cd(II) through differential pulse anodic stripping voltammetry (DPASV) with a LOD of 0.004 μM. Finally, the determination of Cd(II) in the drinking water sample was conducted to confirm the feasibility of the developed method.

## Experimental

### *Reagents and apparatus*

Analytical-grade reagents and chemicals were utilized throughout the study without undergoing further purification. All solutions were prepared in an acetate buffer solution (ABS) 0.1 M, which was obtained from acetic acid and adjusted pH to the defined value by an aqueous solution of sodium hydroxide

DPASV measurements were conducted using a PGSTAT-302N potentiostat/galvanostat (Metrohm-Autolab, Netherlands). A three-electrode system, including a reference electrode (Ag/AgCl, 3 M KCl), a Pt wire counter electrode, and unmodified and modified GCEs as the working electrode, was employed for all electrochemical studies.

### *Synthesis procedure of NH<sub>2</sub>-functionalized MIL-101(Fe) MOF*

To synthesize the NH<sub>2</sub>-functionalized MIL-101(Fe) MOF, 1.34 g of FeCl<sub>3</sub>×6H<sub>2</sub>O and 0.44 g of 2-amino-terephthalic acid (2-ATA) was added to N,N-dimethylformamide (DMF) (40 mL). The mixture was magnetically stirred for approximately 20 min until the reagents were completely dissolved. The homogeneous solution was then transferred into an autoclave and subjected to solvothermal treatment at 110 °C for 24 h. After the solvothermal reaction, the product was separated by centrifugation. The precipitate obtained was repeatedly washed using ethanol and DMF to remove any unreacted reagents or residual impurities. Finally, the purified product was vacuum dried at 65 °C for 12 h.

### Modification of GCE surface using NH<sub>2</sub>-functionalized MIL-101(Fe) MOF and GO

For the preparation of the modified GCE, the cleaned and polished GCE was drop-cast with 5.0  $\mu\text{L}$  of an aqueous suspension (1.0 mL) containing 0.4 mg of NH<sub>2</sub>-functionalized MIL-101(Fe) MOF and 0.6 mg of GO and then air-dried to prepare modified GCE (NH<sub>2</sub>-functionalized MIL-101(Fe)/GO/GCE).

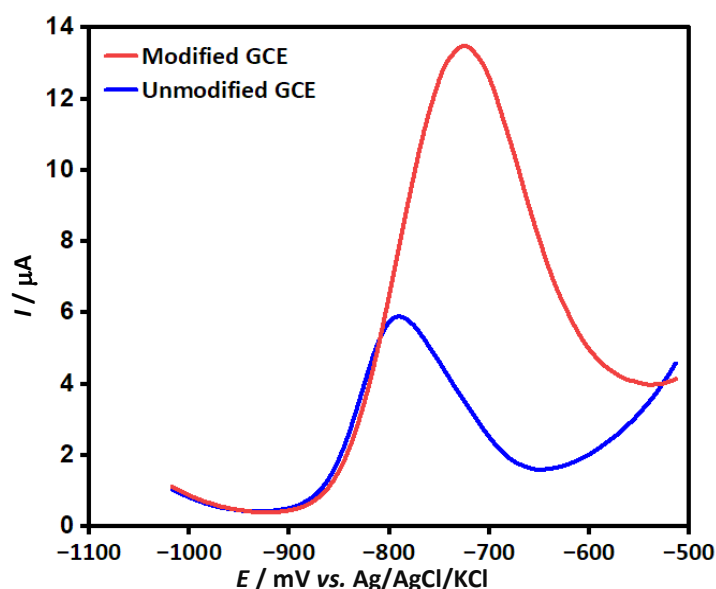
### Procedures for Cd(II) determination using DPASV

The DPASV method was applied for trace analysis of Cd(II). For this purpose, the NH<sub>2</sub>-functionalized MIL-101(Fe)/GO/GCE was placed in ABS (0.1 M, pH 4.0) containing a specific concentration of Cd(II), and Cd(II) was accumulated at the surface of the modified GCE when an accumulation potential ( $E_{\text{Accumulation}}$ ) of -0.9 V vs. Ag/AgCl/KCl was applied for 300 s under continuous stirring of the solution, during which Cd(II) was reduced to Cd(0). Following accumulation, the GCE was washed with distilled water and placed in ABS to record the stripping voltammogram of Cd(II) over the desired potential range.

## Results and discussion

### Comparison of the sensing performance of NH<sub>2</sub>-functionalized MIL-101(Fe)/GO-modified GCE and unmodified GCE for Cd(II) determination

The stripping signal of Cd(II) at the surface of various electrodes can be monitored using DPASV. Figure 1 shows the DPASV voltammograms of the unmodified GCE and the NH<sub>2</sub>-functionalized MIL-101(Fe)/GO-modified GCE after pre-concentration in ABS (0.1 M at pH 4.0) containing 10.0  $\mu\text{M}$  Cd(II). Under the same conditions, the stripping peak of Cd(II) obtained at the unmodified GCE displayed a relatively low peak current (5.87  $\mu\text{A}$ ) within the potential range between -1017 and -513 mV, whereas the modified GCE exhibited a markedly enhanced response to Cd(II), demonstrating a significant increase in the stripping peak current. At the surface of NH<sub>2</sub>-functionalized MIL-101(Fe)/GO-modified GCE, the stripping peak current increased by about 2.3 times, indicating that the high chelating ability of the NH<sub>2</sub>-functional groups in the NH<sub>2</sub>-functionalized MIL-101(Fe) MOF and the high electrical conductivity of GO contributed significantly to the enhancement of the electrocatalytic ability of the modified GCE.



**Figure 1.** DPASV responses of unmodified GCE and NH<sub>2</sub>-functionalized MIL-101(Fe)/GO-modified GCE after pre-concentration in ABS (0.1 M at pH 4.0) containing 10.0  $\mu\text{M}$  Cd(II)

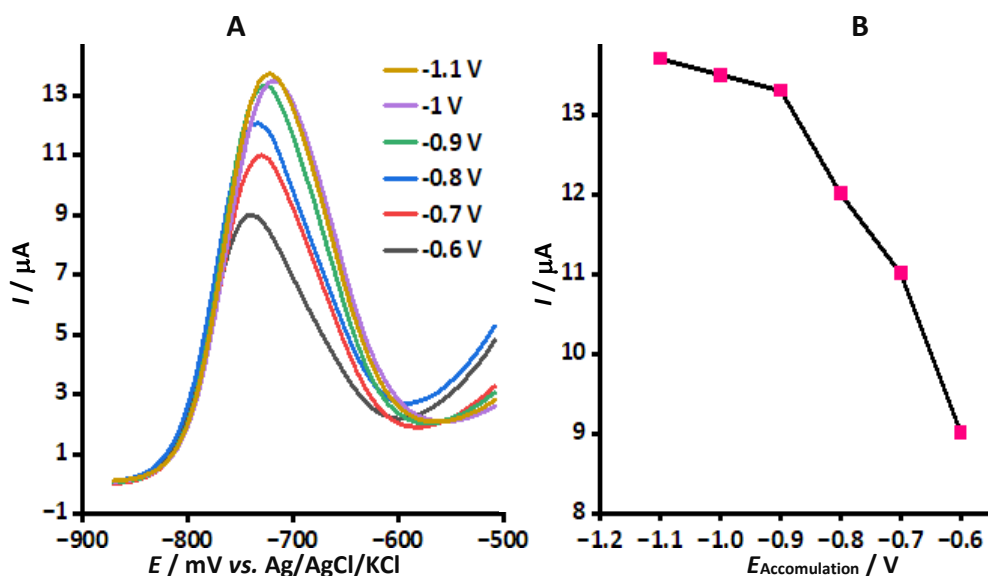
### Optimization of operational conditions and parameters for determination of Cd(II) at the NH<sub>2</sub>-functionalized MIL-101(Fe)/GO-modified GCE

To achieve the highest sensitivity for Cd(II) determination, several experimental conditions and parameters, including the pH of the supporting electrolyte solution (ABS), accumulation potential, and accumulation time, were initially optimized and evaluated.

Initially, the effects of pH on the pre-concentration of Cd using the DPASV method were investigated with 0.1 M ABS. The voltammograms corresponding to the DPASV responses of NH<sub>2</sub>-functionalized MIL-101(Fe)/GO-modified GCE were recorded after pre-concentration in ABSs (0.1 M) at different values of pH from pH 1.0 to 6.0, containing 10.0 μM Cd(II). The dependence of the stripping peak current on solution pH revealed an increase from pH 1.0 to 4.0, followed by a gradual decline at higher pH values. The stripping peak current exhibited a maximum value at pH 4.0. At pH <4.0, protonation of the amino-groups in NH<sub>2</sub>-functionalized MIL-101(Fe) reduces their ability to chelate Cd(II), resulting in low peak currents. The stripping peak current reaches its maximum at pH 4.0 due to optimal chelation, whereas higher pH values decrease current due to partial Cd(II) hydrolysis. Therefore, ABA at 0.1 M was chosen for subsequent experiments at pH 4.0.

Furthermore, accumulation potential and accumulation time are both key parameters in DPASV measurements. The remarkable sensitivity of this technique mainly arises from the accumulation stage, during which the target metal ions are accumulated on the electrode surface.

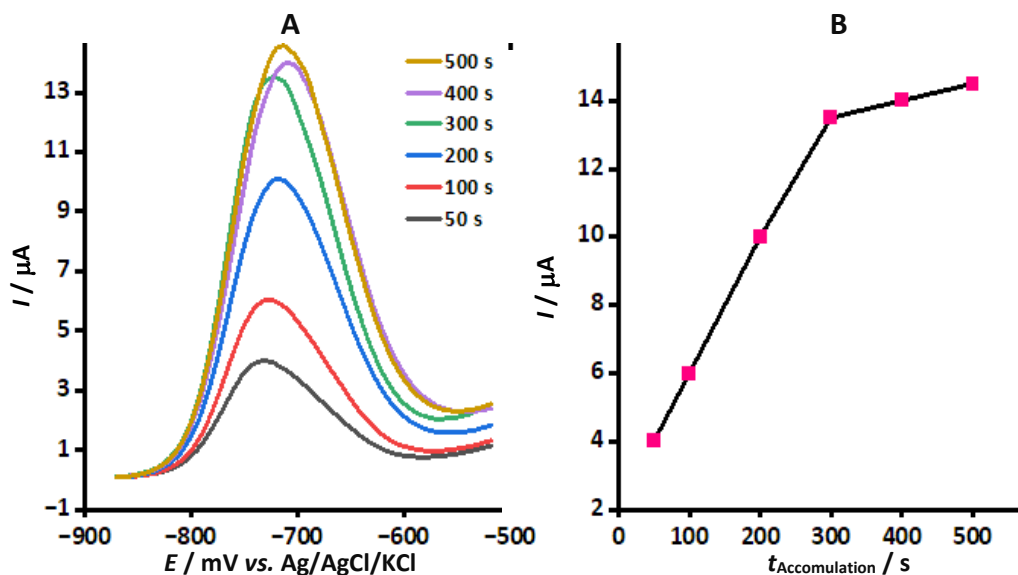
To investigate the influence of accumulation potential, the DPASV responses of NH<sub>2</sub>-functionalized MIL-101(Fe)/GO-modified GCE after pre-concentration in ABS (0.1 M, pH 4.0) containing 10.0 μM Cd(II) were recorded at different accumulation potentials from  $E_{\text{Accumulation}} = -0.6$  to  $-1.1$  V (Figure 2A). As the accumulation potential was raised from  $-0.6$  to  $-0.9$  V, the stripping peak current increased significantly, reaching a maximum of 13.5 μA at  $-0.9$  V (Figure 2B). However, with a further increase in the accumulation potential to  $-1.1$  V, the stripping peak current increased only slightly (Figure 2A). Consequently, for further measurements,  $E_{\text{Accumulation}} = -0.9$  V was selected as the optimized potential for Cd(II) determination.



**Figure 2.** (A) DPASV responses of NH<sub>2</sub>-functionalized MIL-101(Fe)/GO-modified GCE after pre-concentration in ABS (0.1 M at pH 4.0) containing 10.0 μM Cd(II) at various accumulation potentials ( $E_{\text{Accumulation}} = -0.6$  to  $-1.1$  V); (B) Plot of  $I_p$  vs.  $E_{\text{Accumulation}}$ .

To optimize the accumulation time, the DPASV responses of NH<sub>2</sub>-functionalized MIL-101(Fe)/GO-modified GCE were recorded after pre-concentration in ABS (0.1 M, pH: 4.0) containing 10.0 μM

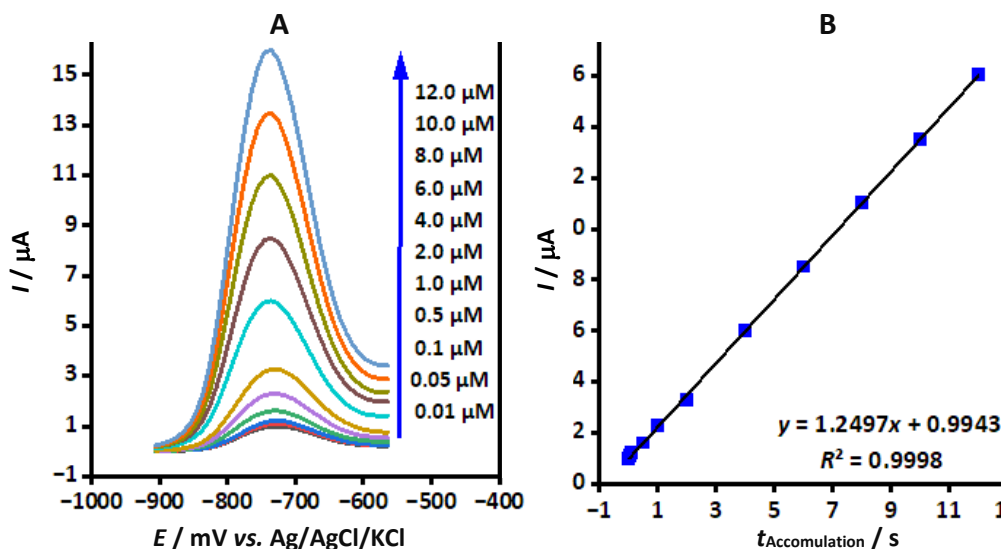
Cd(II) at different accumulation times from  $t_{\text{Accumulation}} = 50$  to 500 s (Figure 3A). From Figure 3B, it was observed that increasing the accumulation time from 50 s to 300 s resulted in a gradual increase in the stripping peak currents of Cd(II), reflecting a higher amount of Cd(II) pre-concentrated on the GCE surface. However, when the accumulation time exceeded 300 s, the stripping peak current increased only slightly with further prolongation, suggesting that the GCE surface had reached saturation (Figure 3B). Therefore, a 300 s accumulation time was found suitable for detecting Cd(II) and was chosen for subsequent measurements.



**Figure 3.** (A) DPASV responses of NH<sub>2</sub>-functionalized MIL-101(Fe)/GO-modified GCE after pre-concentration in ABS (0.1 M at pH 4.0) containing 10.0 μM Cd(II) at various accumulation times (from  $t_{\text{Accumulation}} = 50$  to 500 s); (B) Plot of  $I_p$  vs.  $t_{\text{Accumulation}}$ .

*DPASV measurements of Cd(II) at the NH<sub>2</sub>-functionalized MIL-101(Fe)/GO-modified GCE*

The DPASV method was employed to detect Cd(II) at various concentrations in ABS (0.1 M, pH 4.0) under optimized conditions ( $E_{\text{Accumulation}} = -0.9$  V and  $t_{\text{Accumulation}} = 300$  s). Figure 4A exhibits the DPASV responses of NH<sub>2</sub>-functionalized MIL-101(Fe)/GO-modified GCE after pre-concentration in ABSs (0.1 M, pH 4.0) containing Cd(II) at different concentrations from 0.01 to 12.0 μM.



**Figure 4.** (A) DPASV responses of NH<sub>2</sub>-functionalized MIL-101(Fe)/GO-modified GCE after pre-concentration in ABS (0.1 M at pH: 4.0) containing Cd(II) at different concentrations (from  $C = 0.01$  to 12.0 μM). Conditions:  $E_{\text{Accumulation}} = -0.9$  V and  $t_{\text{Accumulation}} = 300$  s; (B) calibration plot for Cd(II) determination

The obtained voltammograms show that the stripping peak currents of Cd(II) increased proportionally with increasing its concentration. Furthermore, as shown in the calibration plot (Figure 4b), the stripping peak currents of Cd(II) showed a good linear dependence on their corresponding concentrations from 0.01 to 12.0  $\mu\text{M}$ . The LOD was determined to be 0.004  $\mu\text{M}$  for detecting Cd(II).

### Interference study

The potential influence of some metal ions on the stripping voltammetric determination of Cd(II) at the  $\text{NH}_2$ -functionalized MIL-101(Fe)/GO-modified GCE surface was examined. DPASV measurements were carried out using solutions containing 0.5  $\mu\text{M}$  Cd(II) along with a 10-fold excess of Pb(II), Zn(II), and Fe(II). The results indicated that none of these species significantly influenced the Cd(II) signal, as the recorded variations remained within  $\pm 5\%$ .

### Application of $\text{NH}_2$ -functionalized MIL-101(Fe)/GO-modified GCE in the analysis of drinking water

To validate and investigate the efficiency of the  $\text{NH}_2$ -functionalized MIL-101(Fe)/GO-modified GCE for detecting Cd(II) ions in real samples, it was used to analyze a drinking water sample. Initially, the prepared drinking water sample was analysed. Cd(II) was not detected in this sample. Then, recovery tests were performed by spiking known concentrations of Cd(II) into the drinking water sample, and the resulting voltammograms were recorded. Table 1 presents the summarized results from this investigation. It was found that the recovery values for Cd(II) determination in the drinking water sample ranged from 97.8 to 104.3 %. These results demonstrated the practicality of the developed sensing platform for detecting Cd(II) in drinking water samples.

**Table 1.** Determination and recovery of Cd(II) in the drinking water sample using the as-developed sensing platform ( $\text{NH}_2$ -functionalized MIL-101(Fe)/GO-modified GCE) ( $n = 5$ )

$C_{\text{Cd(II)}} / \mu\text{M}$		Recovery, %	RSD, %
Added	Found		
0	-	-	-
5.0	4.0	98.0	2.4
7.0	7.3	104.3	3.6
9.0	8.8	97.8	1.9
11.0	11.1	100.9	2.2

### Conclusion

This study introduced the application of  $\text{NH}_2$ -functionalized MIL-101(Fe) MOF and GO sheets as electrode materials for modifying a GCE into an effective sensing platform for DPASV determination of Cd(II). The  $\text{NH}_2$ -functionalized MIL-101(Fe)/GO/GCE exhibited good performance for Cd(II) determination, resulting from the existence of the  $\text{NH}_2$ -functionalized MIL-101(Fe) and GO with good properties. The  $\text{NH}_2$ -functionalized MIL-101(Fe)/GO-modified GCE was found to have a high sensitivity ( $1.2497 \mu\text{A} \mu\text{M}^{-1}$ ) and a low LOD (0.004  $\mu\text{M}$ ). toward Cd(II) determination was achieved under the optimized parameters and conditions. Furthermore, the  $\text{NH}_2$ -functionalized MIL-101(Fe)/GO-modified GCE was successfully used to determine Cd(II) ions in a drinking water sample, demonstrating significant performance and reliability.

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**Conflicts of Interest:** The authors have no conflict of interest.

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