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Original scientific paper

Electrochemical detection of gliclazide and glibenclamide on metal organic framework/multi-walled carbon nanotubes nanocomposite modified screen printed electrode

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Abstract

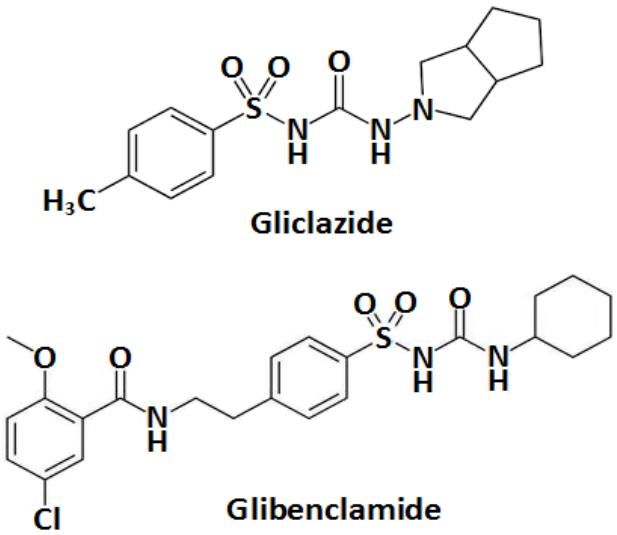
This study presents a novel SPE modification using a Zn-Ni-based 1,4-benzenedicarbon-xylate (BDC) metal-organic framework (MOF)/multi-walled carbon nanotubes (MWCNTs) nanocomposite (Zn-Ni-BDC MOF/MWCNTs nanocomposite) for the detection of gliclazide and glibenclamide. Several methods, including cyclic voltammetry (CV), differential pulse voltammetry (DPV) and chronoamperometry, were used to characterize the modified electrode (MOF/MWCNT/SPE). On the modified electrode surface, the electrochemical characteristics of gliclazide were investigated. The MOF/MWCNT/SPE sensor's ability to electrochemically detect gliclazide and glibenclamide in aqueous solution was shown to be greatly enhanced. The improved sensor's electrocatalytic properties were further utilized to detect gliclazide using DPV. The sensor effectively resolved the overlap in the corresponding voltammetric responses and displayed two well-resolved voltammetric peaks for the two targets. According to DPV, the peak currents from gliclazide increased linearly at concentrations between 0.75 and 500 μM. Gliclazide showed a detection limit of 0.12 μM, according to the modified electrode. The provided approach was also effective in identifying two analytes in urine samples and medications.

Keywords

Diabetes treatment; sulfonylureas; electrochemical determination; chemically modified electrode; urine analysis, pharmaceutical tablets analysis

Introduction

Gliclazide (Scheme 1) is a sulphonylurea used to treat Type 2 diabetes. It acts like most other sulphonylureas but also offers some advantages over them, such as a shorter half-life and a lower risk of hypoglycemia and cardiovascular complications [1]. On top of that, gliclazide has shown vascular plus antioxidant advantages, at least in rats [1]. Nowadays, gliclazide shows up in bulk materials, in biological fluids, and in pharmaceutical products, *via* a bunch of methods like UV and visible spectrophotometry [2], high-performance liquid chromatography [3], capillary electrophoresis (CE) with an amperometric detector [4], gas chromatography [5], and radioimmunoassay [6]. Even so, there are rather few investigations focused on the electrochemical behaviour of gliclazide [7,8].



Scheme 1. *Chemical structure of gliclazide and glibenclamide*

Patients with Type 2 diabetes usually end up needing more than one oral antidiabetic medication so they can reach the desired blood sugar level, because they often cannot get enough of a glycaemic index with only one drug. In this kind of situation, gliclazide and glibenclamide (Scheme 1) are two sulfonylurea-based drugs that are given [9]. Each of these medicines can be used alone, but in some formulations, both drugs are combined. Measuring antidiabetic medication concentrations in plasma is important for many reasons [10].

Because of their distinctive physicochemical properties, which can substantially enhance material behaviour, nanostructured materials have influenced many scientific fields [11]. Using nano-materials to tune or modify electrodes during electrochemical measurements has been particularly useful in electroanalysis [12].

A fascinating class of crystalline materials known as MOFs comprises coordinated metal ions and organic linkers [13]. These materials have distinctive physicochemical and structural characteristics, such as a notably large surface area, an extremely porous framework, and an adaptable architecture with a variety of pore sizes. They also provide a large number of active sites and may be generated in various shapes and dimensions with adjustable surface properties. Due to their distinctive characteristics, MOFs are considered a significant development in materials science, offering flexible and effective solutions to pressing problems in industries such as chemical sensing and medicine [14]. MOFs' inherent poor electrical conductivity, which often hinders effective electron transfer at the electrode surface, is frequently the main reason for the limited practical use of these materials in electrochemical sensors. Usually, better sensing performance is achieved when MOFs are deliberately combined with conductive nanostructures, such as thick carbon nanostructures, to

address this issue [15]. Carbon nanotubes (CNTs) exhibit remarkable electrical and mechanical behaviour, what is usually linked to their distinct sp^2-hybridized carbon framework, arranged in a hexagonal pattern with a very large specific surface area. Because their nanostructured architecture is so fine-tuned, CNTs offer high electrical conductivity, a large surface area and strong chemical stability, so they are often used to modify electrode surfaces and form efficient electrochemical sensors [16]. Moreover, when CNTs and MOFs are used together, they exhibit synergistic effects that enhance overall electrochemical sensing efficiency [17].

The disposable, inexpensive screen-printed electrode (SPE) has been rapidly developed [18]. Because of thit, the memory effect is basically gone, and there is no longer need for the complex pre-treatment usual for traditional electrodes [19]. It also helps address several drawbacks of classic electrode systems, such as the need for frequent recalibration, low stability, and limited suitability for on-site analysis. As a result, SPE-based sensors offer greater capability, portability, and versatility [19].

So far, the bioengineering, pharmaceutical detection, food safety, clinical diagnosis, and environmental analysis communities have all made extensive use of SPE sensors. In particular, the successful commercialization of SPE really shows how promising its introduction into the commercial market is [20].

Considering all of this, our work describes the synthesis and use of a Zn-Ni-based 1,4-benzenedicarboxylate (BDC) metal organic framework (MOF)/multi-walled carbon nanotubes (MWCNTs) nanocomposite (Zn-Ni-BDC MOF/MWCNTs) for the surface modification of SPE, enabling the development of a sensor for gliclazide detection even when glibenclamide is present. The use of a Zn-Ni-BDC MOF/MWCNT nanocomposite to change the surface of a SPE as a new sensing platform for the simultaneous measurement of gliclazide and glibenclamide is what makes this work novel. Zn-Ni-BDC MOF and MWCNTs significantly improve SPE performance, which in turn improves gliclazide's voltammetric response. It seems to keep working even when gliclazide is in the same medium as glibenclamide. When DPV was used, the MOF/MWCNT/SPE showed accurate measurements of gliclazide and glibenclamide in real samples, with decent recoveries.

Experimental

Chemicals and solutions

Gliclazide, glibenclamide, and other highly pure analytical-grade compounds were employed in the current investigation as supplied, without additional processing. Deionized water was used to create the fresh aqueous solutions. An aqueous sodium hydroxide solution was added to a 0.1 M phosphoric acid solution to prepare phosphate buffer (PB) solutions within the appropriate pH range.

Instruments and characterizations

All electrochemical tests were conducted using the PGSTAT 302N Autolab and run by GPES software. A pH meter (Metrohm model 713) was used to measure the pH values of buffer solutions.

Synthesis of Zn-Ni-based 1,4-benzenedicarboxylate metal-organic framework/multi-walled carbon nanotubes nanocomposite

First, 1 mmol (0.290 g) $Ni(NO_3)_2{\cdot}6H_2O$, 1 mmol (0.297 g) $Zn(NO_3)_2{\cdot}6H_2O$ and 1 mmol (0.332 g) BDC ligand were dissolved in 35 mL DMF, 2.5 mL deionized water, and 2.5 mL ethanol while being magnetically stirred for 10 minutes to create the precursors of Zn-Ni-BDC MOF. To distribute MWCNTs in the suspension, 0.012 g of MWCNTs-COOH was added to the precursors and

ultrasonicated for 30 minutes at room temperature. The resulting suspension was then put into a 100 mL high-pressure reactor. For 15 hours, the solvo/hydrothermal process was allowed to proceed at 125 °C. The precipitates were collected by centrifugation after cooling to room temperature and repeatedly cleaned with ethanol and deionized water. Ultimately, the Zn-Ni-BDC MOF/MWCNTs nanocomposite was produced following an 18-hour drying process at 65 °C.

The same process was used to create Ni-BDC MOF (without MWCNTs-COOH and $Zn(NO_3)_2{\cdot}6H_2O$) and Zn-Ni-BDC MOF (without MWCNTs-COOH). Furthermore, the synthesis of the Ni-BDC and Zn-Ni-BDC MOFs was performed using the previously outlined methods [21].

Preparing real samples

Urine samples in 10 mL tubes were centrifuged at 2,000 rpm for 15 minutes. After that, different volumes of the solution were transferred into a 20-milliliter volumetric flask and diluted with phosphate-buffered to the appropriate volume. Gliclazide and glibenclamide in varying doses were used to anesthetize these diluted urine samples. The recommended process used the conventional addition method to analyse the content of gliclazide and glibenclamide.

Five tablets containing 100 mg of gliclazide each have been crushed. The pill solution was then made by using ultrasonication to dissolve 500 mg of powder in 20 ml of water. After that, different volumes of the diluted solution were transferred to a 20-milliliter volumetric flask and made up to volume with PB solution. The conventional addition method has been used to analyse the gliclazide content.

Five glibenclamide tablets (labelled 100 mg each) have been crushed. The pill solution was then made by using ultrasonication to dissolve 500 mg of powder in 20 milliliters of water. After that, different volumes of the diluted solution were transferred to a 20-milliliter volumetric flask and made up to volume with PB solution. The conventional addition method has been used to analyse the glibenclamide content.

SPE modification

To modify the SPE with the Zn-Ni-BDC MOF/MWCNTs nanocomposite, 3.0 µL of a suspension containing 1 mg of Zn-Ni-BDC MOF/MWCNTs, dispersed in 1.0 mL of water, was applied to the working electrode. After that, Zn-Ni-BDC MOF/MWCNTs was used when the solvent had evaporated in about 20 minutes.

For the CV measurements, the CVs of the unmodified SPE and the MOF/MWCNT/SPE were recorded using a redox probe (1.0 mM $K_3[Fe(CN)_6]$) in 0.1 M KCl solution. This was done at several scan rates, primarily to estimate the electroactive surface area of both the unmodified and modified SPEs. The electroactive surface areas were calculated based on the Randles-Ševčik Equation (1):

$$I_p = 2.69\times10^5 n^{3/2} A C_0 D^{1/2} v^{1/2} \quad (1)$$

Here, I_p is the peak current; n is the number of electrons, A is the electrode's electroactive surface area, C_0 is the analyte concentration, D is the diffusion coefficient and v is the scan rate. From the calculation, the MOF/MWCNT/SPE had an electroactive surface area of 0.129 cm^2, whereas the unmodified SPE had 0.033 cm^2. Overall, modifying the SPE with the Zn-Ni-BDC MOF/MWCNTs nanocomposite clearly and noticeably boosted the electroactive surface area.

Electrochemical experiments

To examine the electrochemical behaviour of gliclazide, CV was performed in 0.1M PB solution containing 300.0 µM gliclazide at a scan rate of approximately 50 mV s^{-1}. To assess how the scan rate influences the response, cyclic voltammograms were collected at different scan rates in PB

solution containing 100.0 µM gliclazide, with a potential window of 265-1120 mV. The working electrode was then held at a fixed step potential of 800 mV for 30 s, allowing chronoamperometry to be performed in PB solution containing gliclazide at different concentrations.

Using the given settings scan rate 50 mV s^{-1}, step potential 10 mV, and pulse amplitude 25 mV, the differential pulse voltammetry (DPV) measurements in PBS solution with gliclazide across a range of concentrations were recorded to enable quantitative determinations of gliclazide from -560 to 940 mV. DPV runs were also performed in buffer solutions containing both gliclazide and glibenclamide at multiple concentrations, covering potentials from 590 to 1060 mV, primarily to verify the presence of gliclazide.

Results and discussion

Electrochemical behaviour of gliclazide on the surface of unmodified SPE and MOF/MWCNT/SPE

One key factor in assessing the electrochemical investigations and gliclazide measurements is the pH of PB solution. Thus, DPV was used to examine the impact of pH values (5.0, 6.0, 7.0, 8.0 and 9.0) on the oxidation of gliclazide. The best gliclazide signal was observed at pH 7.0, as indicated by the corresponding voltammograms at different pH values. Therefore, the ideal pH for detecting gliclazide was determined to be 7.0.

The electrochemical behaviour of gliclazide on unmodified SPE and on MOF/MWCNT/SPE was investigated using CV to evaluate the performance of the built MOF/MWCNT/SPE sensor (Figure 1). The oxidation peaks observed in the voltammograms confirm that the electrochemical reaction of gliclazide is irreversible.

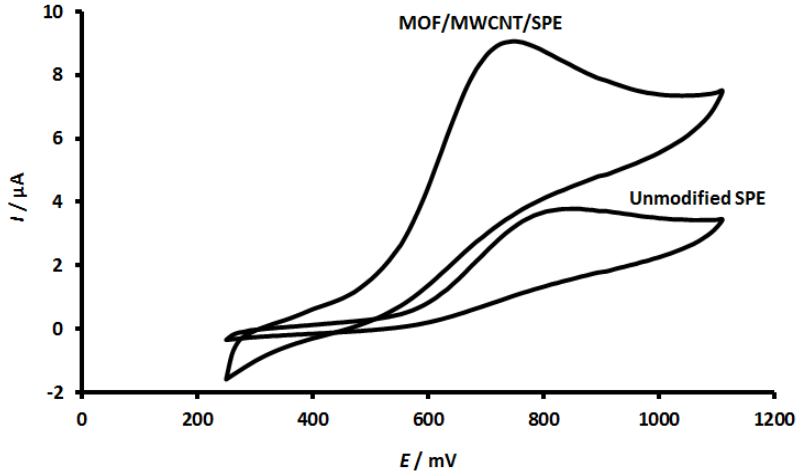


Figure 1. *CV responses of unmodified SPE and MOF/MWCNT/SPE in PB solution containing 100.0 µM gliclazide*

Cyclic voltammograms of gliclazide at bare SPE show only a small oxidation peak, which suggests a sluggish electron transfer. On the other hand, the oxidation peak current becomes notably higher for gliclazide, and the peak potentials shift to lower values after the SPE is modified with the Zn-Ni-BDC MOF/MWCNTs nanocomposite.

Effect of scan rate

The impact of scan rate was also investigated to assess the mechanism of gliclazide reaction on the MOF/MWCNT/SPE surface. The CVs at different scan rates in 100.0 µM gliclazide on the MOF/MWCNT/ /SPE are shown in Figure 2A. It is evident that as the scan rate increases, the oxidation peak current increases. In addition, a linear relationship between the anodic (I_{pa}) peak currents *vs.* the $v^{1/2}$ suggests that the oxidation of gliclazide on MOF/MWCNT/SPE is a diffusion-controlled process (Figure 2B).

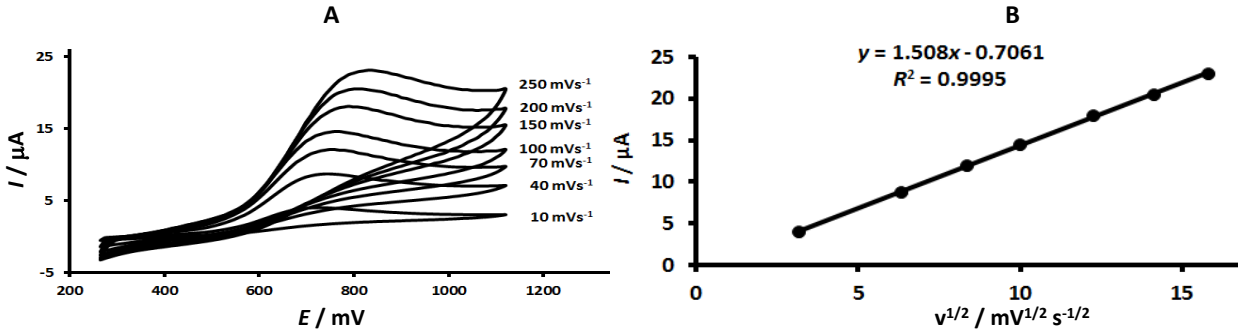


Figure 2. *A - CV responses of MOF/MWCNT/SPE in 100.0 µM gliclazide at various scan rates and B -linear response between the* I_p *and the* $v^{1/2}$

Chronoamperometric studies

The diffusion coefficient (*D*) of electroactive substances is frequently estimated using the chronoamperometry approach. The chronoamperograms in PB solution at different gliclazide concentrations on the MOF/MWCNT/SPE surface are shown in Figure 3. As observed, the current response steadily diminishes over time because the concentration gradient is decreasing. Cottrell's equation is used to estimate the *D* of gliclazide from the chronoamperometric investigations. Plots of *I vs.* $t^{-1/2}$ for different gliclazide concentrations are shown in Figure 4A. Then, the concentration of gliclazide was plotted against slopes of the lines shown in Figure 4A (Figure 4B). By applying Cottrell's equation and using the slope from the resulting curve, the *D* for gliclazide was found to be 8.3×10^{-6} cm^2 s^{-1}.

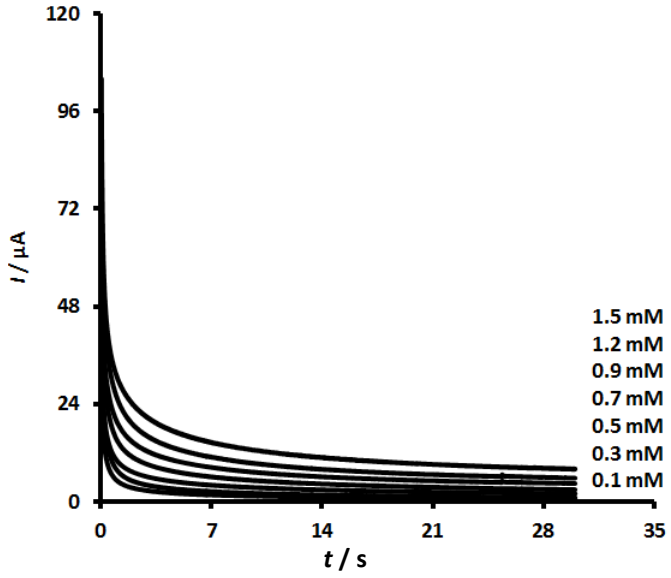


Figure 3. *Chronoamperometric responses of MOF/MWCNT/SP at different concentrations sensor's chronoamperometric responses for the addition of gliclazide in 0.1 M PB*

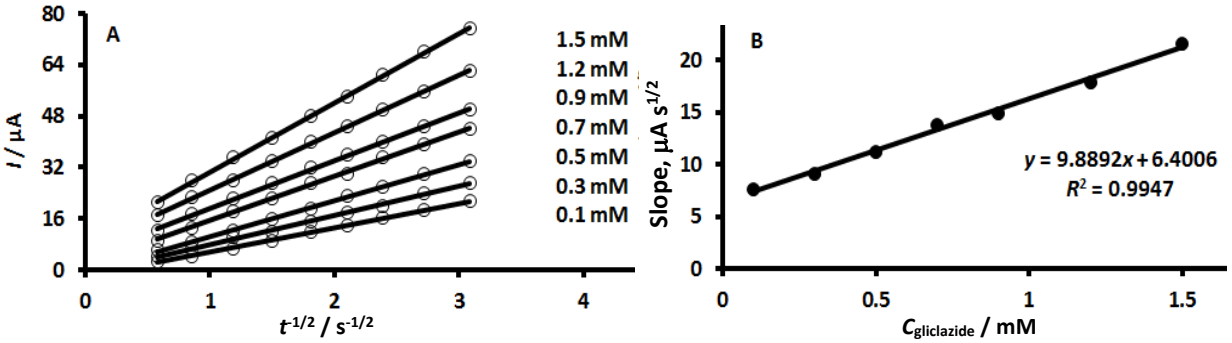


Figure 4*. (A) Plots of the* I vs.$t^{-1/2}$ *and (B) acquired slopes from (A) vs. concentration of gliclazide*

Quantification of gliclazide by using MOF/MWCNT/SPE

The quantification of gliclazide in PB solution was investigated using MOF/MWCNT/SPE over the concentration range of 0.75 to 500.0 µM by DPV. The resulting voltammograms are displayed in Figure 5. The currents of gliclazide increase with the increase in gliclazide concentration. Additionally, the currents are linear with gliclazide concentration over the range 0.75 to 500.0 µM, as shown in Figure 5 (Inset). The LOD was determined to be 0.12 µM (*S*/*N* = 3).

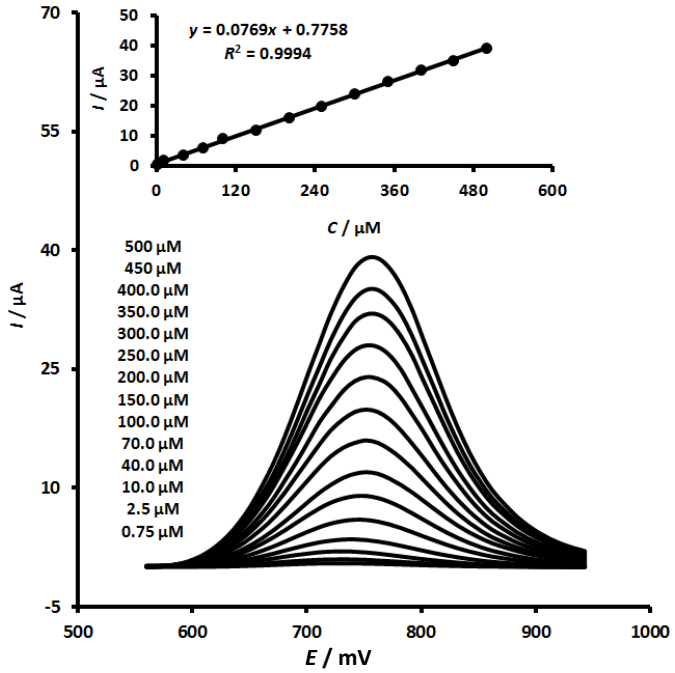


Figure 5. *DPV responses of the MOF/MWCNT/SPE sensor withdifferent concentrations of gliclazide. The calibration curve (current response* vs. *gliclazide concentration) is shown in the inset*

Quantification of gliclazide in the presence of glibenclamide by using metal organic framework/multi-walled carbon nanotubes nanocomposite modified screen printed electrode

DPV further examined the effectiveness of MOF/MWCNT/SPE for gliclazide determination in the presence of glibenclamide. Gliclazide and glibenclamide concentrations were simultaneously changed to record the voltammograms on MOF/MWCNT/SPE (Figure 6).

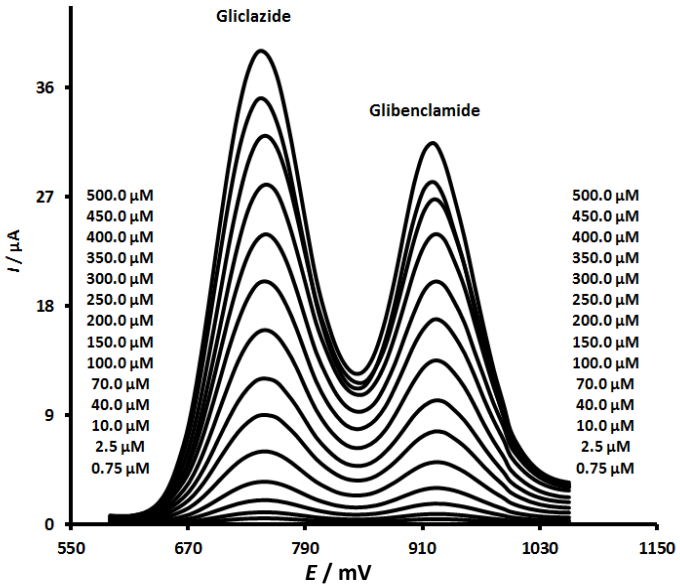


Figure 6*. Voltammetric responses of the MOF/MWCNT/SPE sensor with different concentrations of gliclazide and glibenclamide*

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It is evident that there are two distinct oxidation peaks for glibenclamide and gliclazide, respectively. The corresponding response currents also increased and showed good linear relationships as the concentrations of gliclazide and glibenclamide increased from 0.75 to 500.0 µM (Figure 7).

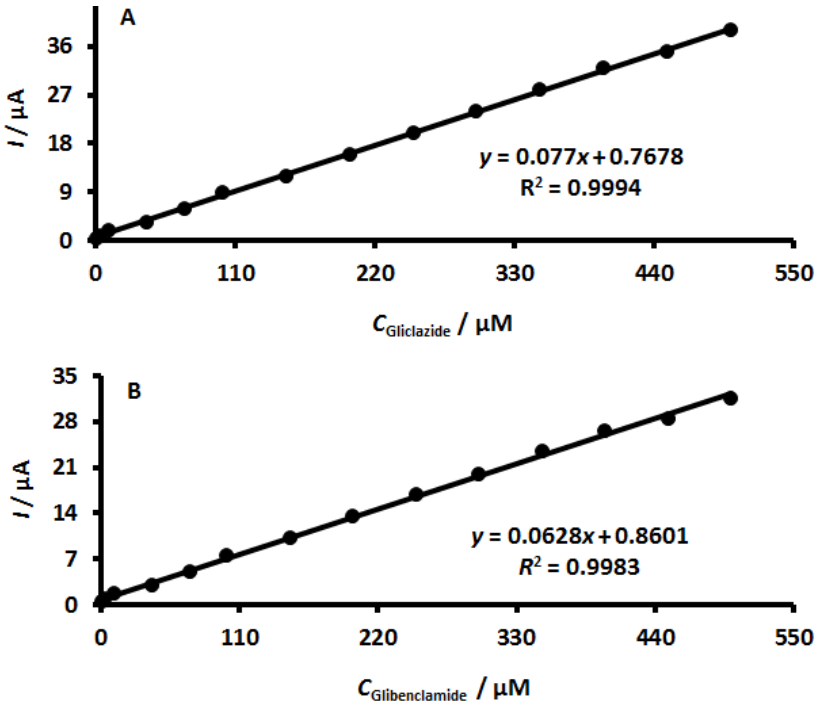

Figure 7*. The calibration plots for: A - gliclazide concentration and B- glibenclamide concentration*

Reproducibility and repeatability studies

Seven different MOF/MWCNT/SPEs were independently prepared using the same method and used to measure 40.0 µM gliclazide in buffer solution to evaluate the reproducibility of the developed sensor. The responses showed no significant variation, with a relative standard deviation (RSD) of 4.1 %. Another crucial factor in an electrochemical sensor's effectiveness is measurement reproducibility. In this study, a buffer solution containing 40.0 µM gliclazide was measured six times using the same sensor (MOF/MWCNT/SPE). The MOF/MWCNT/SPE sensor has good repeatability, maintaining 97.7 % of its initial current response under identical conditions.

Interference study

Under ideal conditions with 50.0 µM gliclazide, the effects of various substances, including potential interferents in gliclazide determination, were investigated. The compounds frequently co-present with gliclazide in medications and biological fluids were selected as interferents. The greatest interferent concentration that results in a gliclazide determination error of less than ±5 % is known as the tolerance limit. The results showed that the determination of gliclazide was unaffected by citric acid, glucose, methanol, fructose, sucrose, phenylalanine, Mg^{2+}, Cl^{-}, lactose, ethanol, SO_4^{2-}, alanine, F^{-}, Al^{3+}, epinephrine, methionine, norepinephrine, saturated starch solution, NH_4^{+}, glibenclamide, uric acid, CO_3^{2-}, ascorbic acid, dopamine and urea.

Analytical application of MOF/MWCNT/SPE

The constructed sensor was used to measure two analytes in tablets and urine samples to verify the MOF/MWCNT/SPE sensor's suitability for real-world applications. Table 1 displays the results obtained. The ability to identify gliclazide and glibenclamide in water samples using MOF/MWCNT/ /SPE was demonstrated by recoveries ranging from 96.2 to 104.0 % and RSD values ≤3.6 %.

Table 1. *Estimation of two analytes in tablets and urine samples using MOF/MWCNT/SPE (n=5)*

Sample	C / µM				Recovery, %		RSD, %	
	Spiked		Found					
	Gliclazide	Glibenclamide	Gliclazide	Glibenclamide	Gliclazide	Glibenclamide	Gliclazide	Glibenclamide
Gliclazide tablets	0	0	3.9	-	-	-	3.4	-
	2.0	5.0	6.0	4.9	101.7	98.0	2.1	3.0
	4.0	7.0	7.7	7.3	97.5	104.3	1.9	2.5
	6.0	9.0	10.2	8.9	103.0	98.9	2.9	2.8
	8.0	11.0	11.8	11.1	99.2	100.9	2.3	3.1
Glibenclamide tablets	0	0	-	4.1	-	-	-	2.7
	5.5	2.0	5.4	6.2	98.2	101.6	3.2	1.8
	7.5	4.0	7.6	8.0	101.3	98.7	2.0	3.6
	9.5	6.0	9.3	10.5	97.9	104.0	2.9	2.6
	11.5	8.0	11.8	11.8	102.6	97.5	3.1	2.7
Urine	0	0	-	-	-	-	-	-
	4.0	4.5	3.9	4.6	97.5	102.2	2.8	3.5
	6.0	6.5	6.2	6.4	103.3	98.5	3.7	2.5
	8.0	8.5	7.7	8.6	96.2	101.2	2.1	2.9
	10.0	10.5	10.1	10.3	101.0	98.1	2.4	2.0

Conclusion

In conclusion, a straightforward method (hydrothermal) was used to create the Zn-Ni-BDC MOF/MWCNTs nanocomposite, which was then utilized to alter the SPE to create an electrochemical sensor for gliclazide detection. The Zn-Ni-BDC MOF/MWCNTs nanocomposite significantly increased gliclazide's peak currents and reduced its overpotential, according to electrochemical measurements. Gliclazide was found using DPV, which has a linear range of 0.75 to 500.0 µM with a LOD of 0.12 µM. Additionally, when gliclazide was measured in the presence of glibenclamide using the MOF/MWCNT/SPE, two distinct peaks were observed for each species. This made it possible to identify both species simultaneously. Furthermore, the constructed sensor showed a consistent and repeatable response to the determination of gliclazide. Lastly, the results for measuring glibenclamide and gliclazide in real samples demonstrated the accuracy and precision of the developed electrochemical sensor.

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