Electrochemical Fabrication of a GO-NiO/AISI 316L Electrode and Its Evaluation for Glucose Detection

Elsa Nadia Aguilera González[1,], Diana Sofía Leija Puente[1], Francisco Mherande Cuevas Muñiz[2], Sofía Estrada Flores[1], Antonia Martínez-Luévanos[1]*

[1] Departamento de Materiales Cerámicos Avanzados y Energía, Facultad de Ciencias Químicas, Universidad Autónoma de Coahuila, Saltillo, 25280 Coahuila, México,
[2] Centro de Investigación y Desarrollo Tecnológico en Electroquímica, S.C. Parque Tecnológico Querétaro Sanfandila, Pedro Escobedo, 76703 Querétaro, Mexico.
[*]E-mail: elsaaguilera@uadec.edu.mx

This paper describes the fabrication of a GO-NiO/AISI 316L electrode and its subsequent evaluation for enhanced glucose detection. The GO-NiO/AISI 316 L electrode was fabricated *in situ* by the electrodeposition of a GO-NiO nanocomposite generated by cyclic voltammetry (CV) on an AISI 316L stainless steel substrate, with a subsequent thermal treatment at 400 °C. The obtained electrode was characterized by scanning electron microscopy (SEM) and Fourier transform infrared (FTIR) spectroscopy. The effectiveness of the electrode to detect glucose was evaluated by both cyclic voltammetry and chronoamperometry (CA). SEM and FTIR results showed that GO-NiO nanocomposite was realized on the surface of the stainless steel substrate confirming the successful fabrication of the GO-NiO/AISI 316L electrode. Electrochemical experiments showed that the electrode catalyzed the electrochemical oxidation of glucose in an alkaline medium. Voltammetric results suggested that the glucose oxidation rate was controlled by glucose diffusion on the electrode surface. The GO-NiO/AISI 316L electrode could detect glucose in an alkaline medium in a linear range of 0.01 mM to 0.7 mM with a response time of less than 5 s, a detection limit of 0.05 mM and a sensitivity of 744.68 $\mu A\ mM^{-1}\ cm^{-2}$.

1. INTRODUCTION

Recently, graphene-based nanomaterials have been increasingly studied due to their fascinating properties, including highly their specific surface area, superior mechanical properties, improved thermal conductivity, and remarkable electronic conductivity. In particular, graphene has been used as a promising two-dimensional (2D) support to load polymers or nanoparticles for applications in

electrochemical devices, such as supercapacitors[1–3], rechargeable lithium-ion batteries [4–6], catalysts[7–9] and electrochemical sensors [10–12].

Among the different graphene-based nanomaterials, graphene-metal oxide-based nanocomposites have increasingly studied as recognition elements in the development of nonenzymatic glucose biosensors, because they exhibit excellent catalytic activity, selectivity, and sensitivity for electrochemical glucose detection [12]. Several graphene oxide-based nanocomposites have been synthesized and immobilized on different electronic conductive materials to develop nonenzymatic glucose electrodes. Among these nanocomposites, graphene-based nanocomposites of metal oxides such as copper oxides [13–15], nickel oxides [16,17] and cobalt oxides [18] have been widely investigated due to their electrocatalytic properties for glucose oxidation; in particular, nanocomposite-based graphene and Ni in any of its forms (Ni, NiO, Ni(OH)$_2$). The electrocatalytic properties of graphene-Ni based nanocomposites to oxidize glucose are related to the formation of the Ni (III)/Ni(II) redox pair on an electrode surface which electrocatalyzes the oxidation reaction of glucose [12].

Some of the works in which graphene-based nanocomposites with Ni compounds have been used as recognition elements for the manufacture of nonenzymatic glucose electrodes are cited promptly. Su-Juan Li et al. (2014) [7] synthesized G/NiO nanocomposites on the surface of a glassy carbon electrode (GCE) using a one-step electrochemical method, and then investigated the electrocatalytic properties of the G/NiO nanocomposites for glucose and methanol oxidation reactions in alkalinemedia. The obtained electrodes showed good electrocatalytic performance, as well as both fast and highly sensitive responses for glucose and methanol detection.

Su-Juan Li et al. (2016) [19] prepared NiO/CuO/rGO, NiO/rGO and CuO/rGO nanocomposites on the surface of a GCE using an easy and simple electrochemical method, and they evaluated the electrocatalytic properties of modified electrodes for methanol oxidation in alkaline media. The modified NiO/CuO/rGO electrode exhibited much better electrocatalytic performance for glucose oxidation than their NiO/rGO and CuO/rGO counterparts under optimal conditions. The GCE NiO/CuO/rGO amperometric sensor demonstrated a wide linear range (5 μM-4.85 mM), high sensitivity (1046 μA mM^{-1} cm^{-2}), low detection limit (0.5 μM) and good selectivity for the glucose detection.

Xiaohui Zhang et al. (2016) [20] introduced modified Ni/NiO nanoflowers on reduced graphene oxide nanocomposites by a screen-printed electrode for the construction of a nonenzymatic glucose biosensor. The Ni/NiO-rGO nanocomposite was synthesized by an *in-situ* reduction process. The resulting sensor was able to quantify glucose over a wide linear range from 29.9 μM to 6.44 mM (R = 0.9937) with a low detection limit of 1.8 M (S/N = 3) and a high sensitivity of 1997 μA mM^{-1} cm^{-2}. This sensor also exhibited good reproducibility as well as high selectivity.

Kurt Urhan et al. (2020) [21] fabricated an electrochemically reduced graphene oxide (ERGO) electrode coupled with Ni nanoparticles (NiNPs) for amperometric glucose determination in an alkaline medium. The electrode was obtained by the electrochemical coreduction and electrodeposition in a single step of a NiNP/ERGO composite on an indium-doped tin oxide (ITO) electrode. The glucose biosensor had the following behavior: linear range of 0.5–244 μM, and a detection limit of 40 nM with a signal-to-noise ratio of 3 (S/N = 3).

It can be seen in the cited research works above; that different electronic conductive materials have been used to immobilize graphene/Ni nanocomposites to prepare nonenzymatic glucose electrodes.

Among the electronic conductive materials used for this purpose are glass carbon electrodes (GCEs) [7,19], screen-printed electrodes [20], indium doped tin oxide (ITO) [21], fluorine doped tin oxide (FTO), gold electrodes, Ti sheets and Ni foams [12].

Stainless steels are another promising material that can be used as substrates to immobilize graphene-based nanocomposites to fabricate electrodes for electrochemical sensing. Stainless steels have interesting characteristics, such as their high corrosion resistance, excellent biocompatibility and low cost, which make them suitable for use in biomedical applications. Additionally, due to their high electrical conductivity and relatively low cost, stainless steels (SS) are promising candidates to constitute the transducer substrate of devices for electrochemical sensing [22,23].

To continue the study of graphene-Ni nanocomposites and their application as recognition elements in glucose detection, in this work, the fabrication of a GO-NiO/AISI 316L electrode, and an evaluation on its the effectiveness to catalyze the electrochemical oxidation of glucose are reported. GO-NiO/AISI 316L is prepared through the electrochemical synthesis of Ni-GO nanocomposites on AISI 316L stainless steel substrates and subsequent thermal treatment at 400 °C. The prepared GO-NiO/AISI 316L electrode is characterized by Fourier transform infrared (FTIR) spectroscopy and scanning electron microscopy (SEM). The effectiveness of the electrode to catalyze the electrochemical oxidation of glucose is evaluated by cyclic voltammetry (CV) and chronoamperometry (CA). The GO-NiO/AISI 316L electrode performance was compared with other graphene-Ni-based composite electrodes for amperometric glucose detection.

2. EXPERIMENTAL

2.1 Materials

Nickel acetate ($Ni(CH_3COO)_2$ $4H_2O$, 98%), and graphene oxide (sheets 4-10% oxidized-edge) from Sigma-Aldrich were used as precursors for the electrochemical synthesis of GO-NiO nanocomposites. An AISI 316L stainless steel plate with dimensions of 1.2 cm × 2.0 cm was used as the substrate. Electrochemical synthesis and electrochemical evaluation of the electrochemical oxidation of glucose were performed with an Autolab PGSTAT204 potentiostat/galvanostat with a conventional three-electrode system. Pt rod and Ag/AgCl electrodes acted as the counter and reference electrodes, respectively, while the AISI 316L stainless steel plate acted as the working electrode. Anhydrous sodium sulfate (Na_2SO_4, Jalmek, 99%) was used as the supporting electrolyte. A BRANSON-1800 ultrasonic bath was used to clean the steel plate and prepare the GO dispersion. The nanocomposite synthesized on a stainless steel substrate was thermally treated in a FELISSA FE-340 muffle furnace.

Glucose was used as the model molecule for glucose sensing. Amperometric and voltammetric measurements were performed with an Autolab PGSTAT204 potentiostat/galvanostat station with a conventional three-electrode system. Potassium hydroxide (KOH) was used as the supporting electrolyte.

2.2 Fabrication of the GO—NiO/AISI 316 L electrode

The GO-NiO/AISI 316 L electrode was fabricated by the *in situ* electrochemical synthesis and electrodeposition of a GO-NiO nanocomposite generated on an AISI 316L stainless steel substrate. The GO-NiO nanocomposite was electrosynthesized by cyclic voltammetry for 40 cathodic potential scan cycles in the voltage range of -1.1 to 1.2 V at a scan rate of 50 mV s^{-1}. The electrochemical cell consisted of a stainless-steel plate of 1.2 cm x 2.5 cm as the working electrode, a platinum rod as the counter electrode, and Ag/AgCl as the reference electrode. The stainless-steel plate was polished mechanically, sonicated in distilled water, and dried at room temperature. Prior to electrosynthesis, 6 mg of GO was dispersed in 6 mL of distilled water and ultrasonication treatment was performed for 0.5 h. The *in situ* electrodeposition process was performed in an aqueous solution containing 6 mL of the GO aqueous dispersion and 32 mL of the 0.01 M nickel acetate aqueous solution in 0.5 M sodium sulfate. The obtained electrode was dried at room temperature and then subjected to thermal treatment at 400 °C for 3 h.

2.3 Morphological and structural characterization

The morphology of the GO-NiO/AISI 316 L electrode was characterized by SEM, while the chemical and structural characterization was carried out by FTIR spectroscopy. SEM images were captured using JEOL electronic microscope, and FTIR spectra were obtained with a Thermo Scientific spectrometer using a KBr disk.

*2.4 Electrochemical study and evaluation of oxidation of glucose on GO-NiO/AISI 316L electrode*

The electrochemical study and effectiveness to oxidize glucose were evaluated by CV using an Autolab PGSTAT204 potentiostat/galvanostat with a conventional three-electrode system. The amperometric and voltammetric measurements of glucose were performed on an as-prepared GO-NiO/AISI 316L electrode in a 0.1 M KOH solution. Cyclic voltammetry (CV) measurements were studied in the potential range of -0.1 to +0.65 V. A GO-NiO/AISI 316L electrode was used as the working electrode, and a Ag/AgCl electrode and a platinum rod were used as the reference and counter electrodes, respectively. The steady-state current response (chronoamperometry) was obtained by applying a constant potential of +0.6 V vs the Ag/AgCl electrode for 30 s.

3. RESULTS AND DISCUSSION

3.1 Characterization of the electrode

Fig. 1 shows an image of the physical aspect and micrographs of the obtained electrode at 20,000, 50,000 and 350,000x. After thermal treatment, the presence of a dark brown iridescent coating on both sides of the stainless-steel plate is observed. The iridescence property suggests that the coating is a thin

film. As shown in Fig.1 the micrograph of the electrode consists of micrometer-sized GO sheets and a few agglomerates of spherical-shaped NiO nanoparticles with a size around of 50 nm.
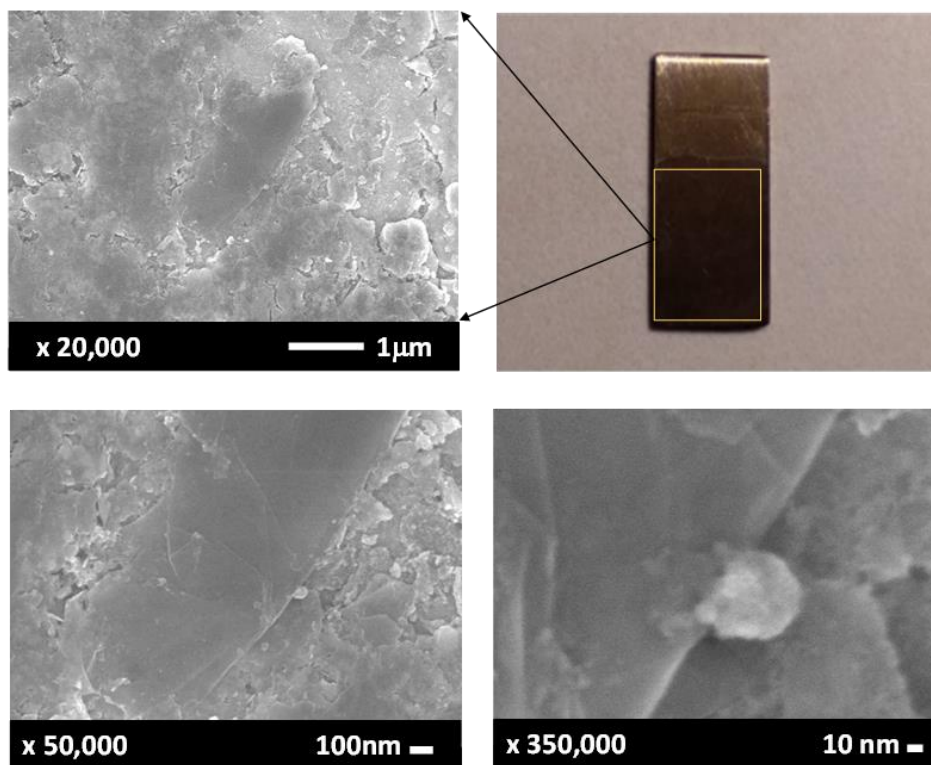
Figure 1. SEM morphology images and physical aspect of obtained electrode.

Fig. 2 displays the FTIR spectra of KBr disks of scraped-off coating powder (Fig. 2a) and GO, which is used as the precursor (Fig.2b). In the FTIR spectrum of electrode (Fig.2a) , the characteristic peaks of GO are observed: the broad peak at 3406 cm^{-1} corresponds to the stretching vibration of C—OH of carboxylic groups or water molecules absorbed onto GO. Additionally, a small sharp peak at 1728 cm^{-1} can be seen, which is attributed to the C=O stretching vibration. Additionally, the absorption peak at 1614 cm^{-1} of the aromatic C=C stretching vibration of the graphite domain of GO, epoxy C—O stretching vibrations at 1132 cm^{-1}, and alkoxy C—O stretching vibrations at 1111 cm^{-1} and 1047 cm^{-1} are observed. These peaks are also in the FTIR spectrum of pristine GO (Fig. 2b). Compared to GO, the FTIR spectrum of the electrode shows new peaks at wavenumbers less than 700 cm^{-1}, which are associated with Ni—O vibration bonds. The absorption band at 430 cm^{-1} is associated to Ni—O vibration bonds, while the absorption band at 601 cm^{-1} is assigned to Ni—OH stretching bonds.[24] These results confirm the presence of GO and NiO in the chemical structure of the electrode and therefore the successful fabrication of the GO-NiO/AISI 316L electrode. The absorption peak at 1383 cm^{-1} in Fig.2a is related to the stretching vibrations of the C-OH bond which indicates that the electrode can absorb water from atmosphere.

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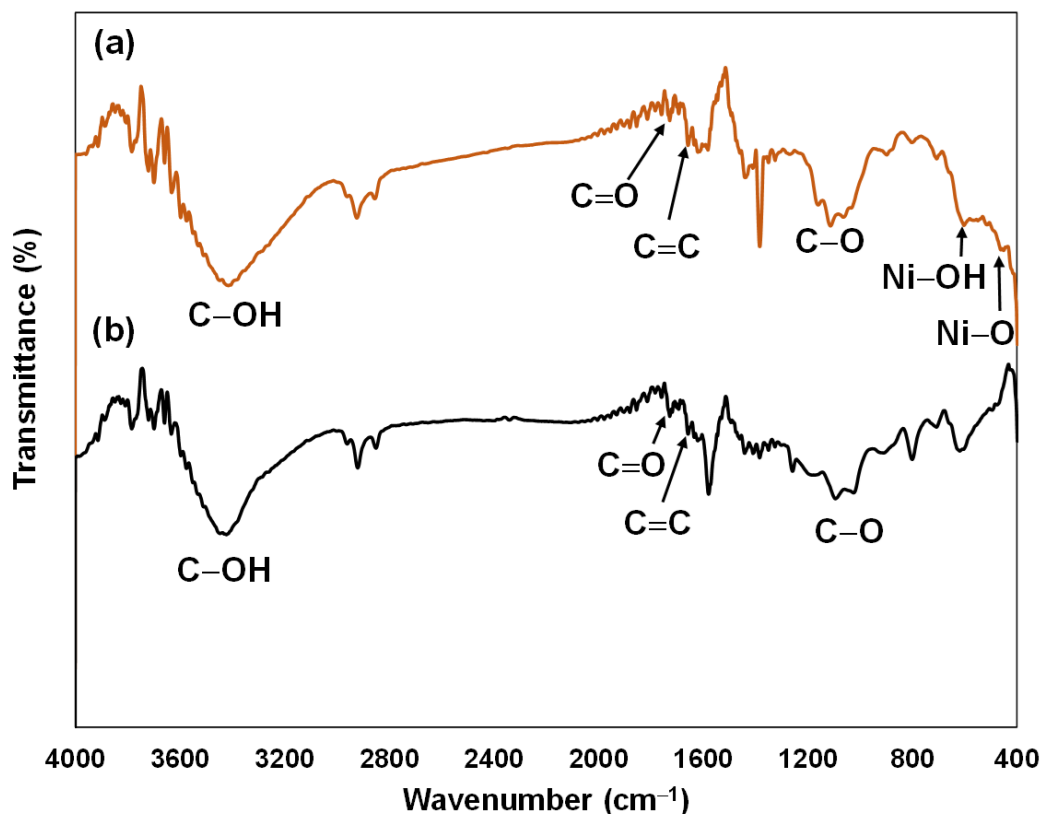
Figure 2. FTIR spectra of the KBr disks containing the (a) scraped-off coating powder of the obtained electrode and (b) GO, which was used as the precursor.

3.2 Electrochemical study and evaluation of the oxidation of glucose on the GO-NiO/AISI 316L electrode

Cyclic voltammetry was used to study the electrochemical behavior of the GO-NiO/AISI 316L electrode. The study was performed in 0.1 M aqueous KOH solution at positive potential values, where glucose oxidation takes place. Fig.3 shows the cyclic voltammograms obtained in 0.1 M KOH at 50 mV s^{-1} of stainless-steel plate treated at 400 °C (denoted AISI 316L) and GO-NiO/AISI 316L electrode.

The cathodic peak (denoted as I) that is observed in the CV curve of the GO-NiO/AISI 316L electrode between 0.4 V and 0.5 V is attributed to the transition of the Ni (III) to Ni (II) of Ni (III)/Ni (II) redox pair. This result is consistent with those previously reported in the literature for Ni based materials [7,20,21] confirming the presence of NiO on the electrode surface. In Fig.3, the anodic peak of the transition from Ni (II) to Ni (III) of the oxidation reaction of NiO to NiOOH is not observed; however, the presence of a cathodic peak (peak I) suggests the formation of NiOOH from the oxidation reaction of NiO to NiOOH. In the CV curve of GO-NiO/AISI 316L, water-splitting starts at approximately 0.55 V. Therefore, it is possible that the anodic peak corresponding to the transition from Ni (II) to Ni (III) is covered by the increased current of the water-splitting reaction.

The electrocatalytic performance toward glucose oxidation on the GO-NiO/AISI 316L electrode was first investigated by CV. Fig.4 presents the CV response of the GO-NiO/AISI 316L electrode in 0.1 M KOH with different glucose concentrations and without glucose.
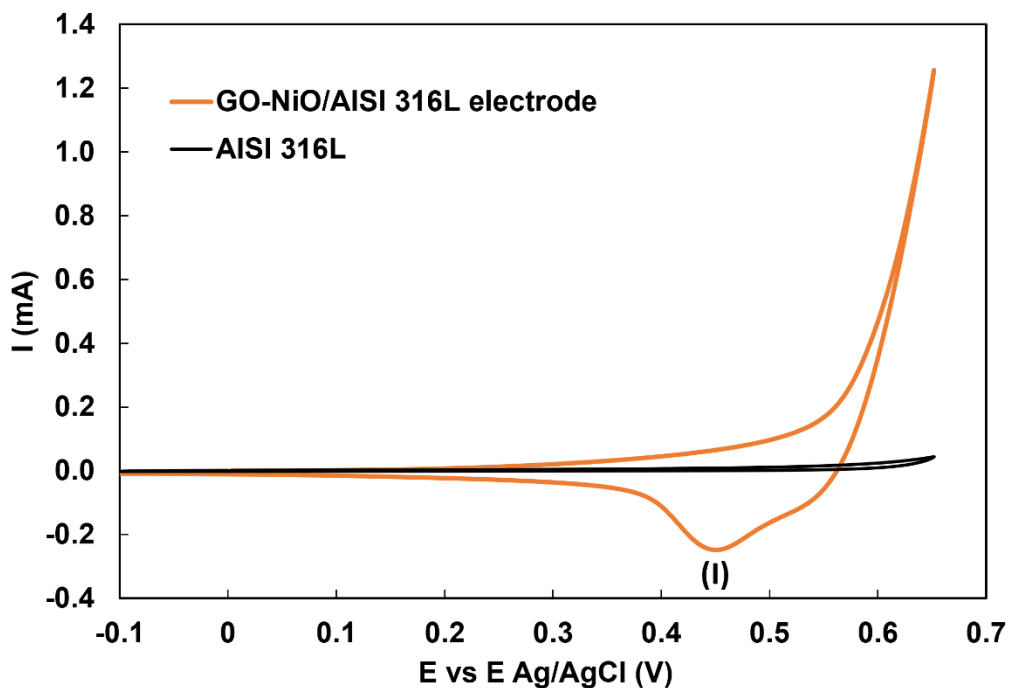
Figure 3. CV curves obtained in 0.1 M KOH at 50 mV s^{-1} on an AISI 316L stainless steel plate and the GO-NiO/AISI 316 L electrode.
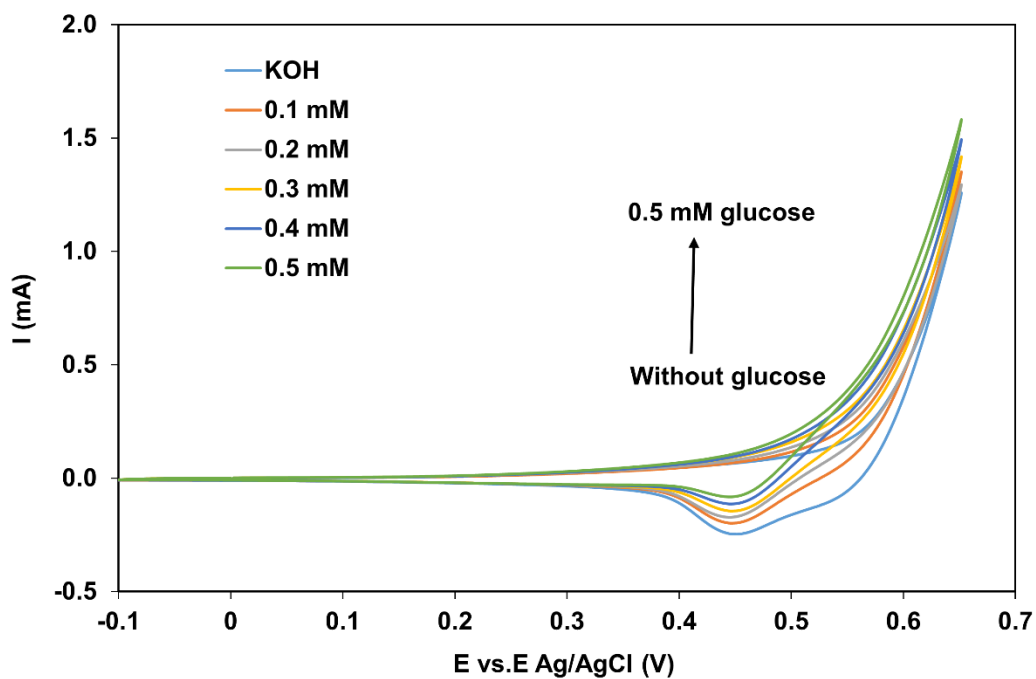

Figure 4. CV response on the GO-NiO/AISI 316L electrode in 0.1 M KOH using different glucose concentrations and without glucose.

As shown in Fig.4 the addition of glucose produces a negative shift in the oxidation current

potential of the electrode and a decrease in the cathodic current peak. The enhancement of the oxidation current potential and the decrease in the cathodic peak with the addition of glucose suggest the electrocatalytic activity of the GO-NiO/AISI 316L electrode in the glucose oxidation process. The decrease in the reduction current peaks can be ascribed to the consumption of Ni^{3+} (NiOOH), which induces glucose oxidation and yields $Ni(OH)_2$ and gluconolactone, according to the following reactions [7,12,20,21]

$$NiO_2 + OH^- \rightarrow NiOOH + H_2O + e^-$$

$$NiOOH + glucose \rightarrow Ni(OH)_2 + gluconolactone$$
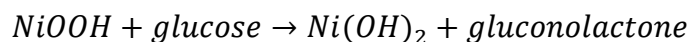
To investigate the electron transfer process of glucose oxidation on the GO-NiO/AISI 316L electrode, VC curves of the electrode were obtained in the presence of 1 mM glucose and 0.1 M KOH using different scan rates. The results are shown in Fig. 5.
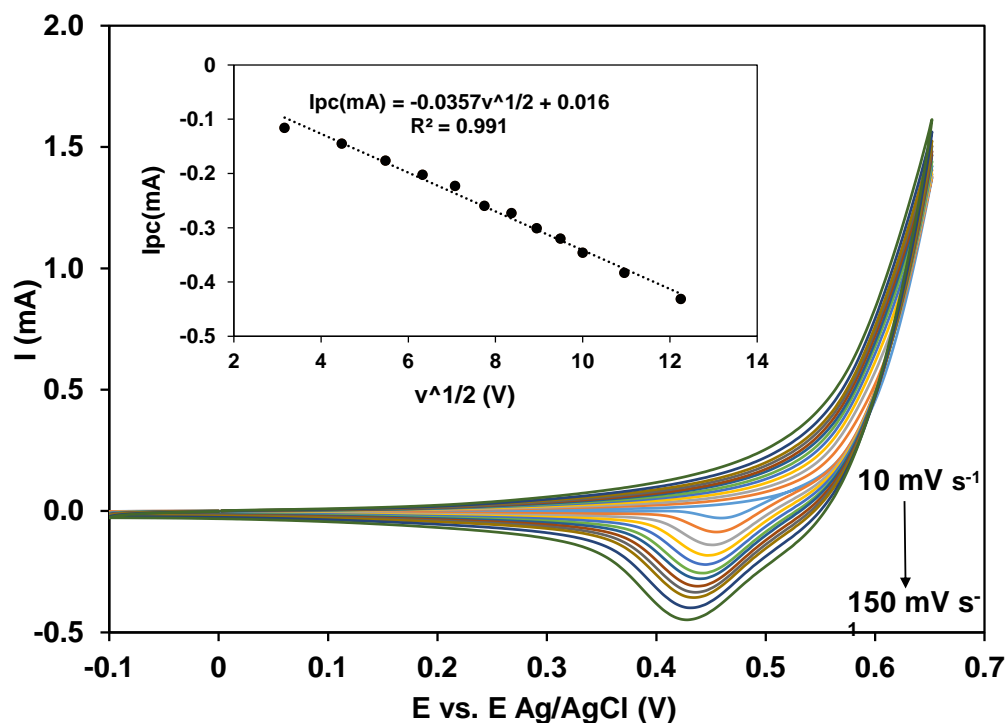
Figure 5. GO-NiO/AISI 316L electrode in the presence of 1 mM glucose and 0.01 M KOH at different scan rates. The inset shows the plot of the cathodic current peak versus scan rate.

The obtained reduction current peaks are proportional to the square root of the scan rate. This result indicates that electrochemical glucose oxidation by NiOOH is a diffusion-dominated electron transfer process. These results are consistent with those previously reported for other Ni/NiO-graphene nanocomposite-based electrodes [7,20,21].

The real surface area of the GO-NiO/AISI 316L electrode was estimated using the Randles-Sevcik equation from electrochemical measurements in a solution containing 0.04 mM $Fe(CN)_6^{3-}$/$Fe(CN)_6^{4-}$ and 0.5 M KOH. The obtained result shows a real surface area of 1.1415 cm^2 for the GO-NiO/AISI 316L electrode.

3.3 Glucose amperometric detection on the GO-NiO/AISI 316L electrode

The amperometric response of the GO-NiO/AISI 316L electrode for the detection of glucose in an alkaline medium was proven using an applied potential of +0.6 V. As shown in Fig. 6 the amperometric response is proportional to the concentration of glucose over the range from 0.01 mM to 0.7 mM, with a correlation coefficient of 0.9967. The required time for achieving a 90% steady current upon the addition of glucose is less than 5 s, demonstrating fast and sensitive glucose oxidation on the GO-NiO/AISi 316L electrode. The detection limit for the GO-NiO/AISI 316L electrode is 0.01 mM and thecalculated sensitivity is 744.68 $\mu A\ mM^{-1}\ cm^{-2}$.
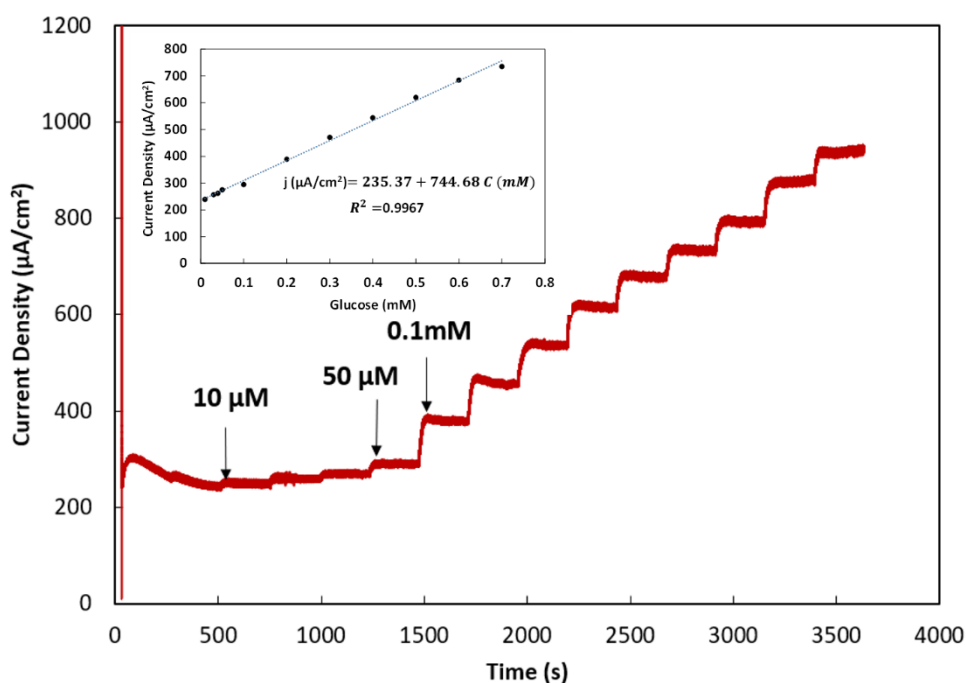
Figure 6. Amperometric detection of glucose by successive addition of glucose on the GO-NiO/AISI 316L electrode using 0.6 V in 0.1 M KOH. Inset: amperometric response with an increasing concentration of glucose from 0.01 mM to 0.7 mM.

The performance of the GO-NiO/AISI 316L electrode was compared with other nonenzymatic glucose sensor-based graphene and NiO. As shown in Table 1, the GO-NiO/AISI 316L electrode has a faster response time and better sensitivity than other reported electrodes (for example, NiO-NPs@FTO and NiO hollow sphere/rGO/GCE electrodes). However, the linear range for the glucose concentration of GO-NiO/AISI 316L is lower than those reported in Table 1. In addition, the detection limit of the GO-NiO / AISI 316L electrode is higher than that of the other electrodes.

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Table 1. Comparison of the performance of the GO-NiO/AISI 316L electrode with other electrode-based graphene and NiO compounds for glucose amperometric detection. GR, GP: graphene, NPs: nanoparticles; GCE: Glassy Carbon Electrode SPE: Screen Printed Electrode; NFI: nanofibers.

Electrode	Response time (s)	Potential (V)	Sensitivity (μA mM^{-1} cm^{-2})	Linear range	LOD	Reference
GP/NiO/GCE	3	+0.4	1571	5 μm-4.8 mM	1 μM	[7]
Ni/NiO-rGO /SPE	<2	+0.55	1997	29.9 μM - 6.44 mM	1.8 μM	[20]
NiO-GR/GCE	<3	+0.35	--	20 μm-4.5 mM	5 μM	[25]
NiO-NPs@FTO	<1	+0.5	3.9	0.1 mM-1.1 mM	1 μM	[26]
NiONPs/GO/GCE	---	---	1082	3.13 μM-3.05 mM	1 μM	[27]
NiO hollow sphere/rGO/GCE	<5	+0.58	2.04	0.009 mM -1.129 mM	82 nM	[28]
GO-NiO/AISI 316L	<5	+0.6	744.68	0.01 mM-0.7 mM	0.05 mM	This work

To investigate the repeatability of the measurements of the GO-NiO/AISI 316L electrode toward glucose sensing, five amperometric measurements of 0.2 mM glucose at +0.6 V using the same electrode was performed. The results showed a relative standard deviation (RSD) of only 0.83%, indicating excellent electrode repeatability.

4. CONCLUSIONS

A GO-NiO/AISI 316L electrode was prepared by the electrochemical synthesis of a GO-NiO nanocomposite on an AISI 316L stainless steel substrate with a subsequent thermal treatment. The GO-NiO/AISI 316L electrode showed activity for the electrochemical oxidation of glucose in an alkaline medium and could detect glucose over the range of 0.01 mM to 0.7 mM with a correlation coefficient of 0.9967 and with a detection limit of 0.01 mM. The calculated sensitivity of the GO-NiO/AISI 316L electrode was 744.68 μA mM^{-1} cm^{-2}. This material is a promising candidate for glucose detection in several body fluids.

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