

## Glucose electrooxidation at nickel nanoparticles modified glassy carbon electrode: tolerance to poisoning

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The electrooxidation of glucose in alkaline media was studied on glassy carbon (GC) electrodes modified with nickel oxide nanoparticles, which were prepared by taking advantage of the control in the nickel bath deposition medium in addition to controlling the interfacial properties of the underlying GC electrode on which the deposition was done. The Electrodeposition was achieved from a medium containing a certain concentration of nickel and glucose ions, typically 5 mM glucose, for modifying the interfacial properties, which reflected on how the nickel was deposited. The properties of the glassy carbon electrode were also controlled by oxidizing it first before nickel was deposited on it. The combination of these reasons led to the preparation of a highly efficient electrode, designated as GC<sub>ox</sub>/NiO<sub>x</sub>(glu), in the electrochemical oxidation of glucose. Given the importance of electrode resistance to poisoning agents. It may achieve high efficiency, but it may lose it in the presence of potential toxins. The oxidation of glucose has been studied in alkaline media that contain halide ions with different concentrations depending on the type of halide. The peak current of glucose oxidation increases five folds at GC<sub>ox</sub>/NiO<sub>x</sub>(glu) compared to the GC/NiO<sub>x</sub> (Glu) electrode in which nickel was deposited similarly to the case of GC<sub>ox</sub>/NiO<sub>x</sub>(glu) but onto unmodified glassy carbon electrode. Also at GC<sub>ox</sub>/NiO<sub>x</sub>(glu) the potential for glucose oxidation was shifted to more negative potential values. Interestingly, tolerance to poisoning by halides is significant. Susceptibility to poisoning increases in the following way: iodide > bromide > chloride. Upon cycling the potential of the modified electrode in chloride solution containing glucose, the peak current continuously decreases. However, under the experimental condition, the peak current for glucose oxidation after cycling of potential is not less than the value of the current corresponding to the absence of glucose. Thus, the present modified electrode not only enhances the glucose oxidation, but also characterized by high immunity to electrode poisoning by halides.

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**Keywords:** Glucose - Nickel oxide-poisoning - Halide – Glassy Carbon Electrode – Nanoparticles.

## 1. INTRODUCTION

Electrochemical oxidation of glucose has generated much interest over the years. It has been extensively studied for applications in glucose–oxygen fuel cells [1], wastewater treatment [2], biotechnology [3] and also, sensing the glucose in medical applications [4]. Although the oxidation of glucose is thermodynamically favorable, the reaction kinetics is very slow at unmodified conventional electrodes, and this hinders its use for analytical purposes, especially in the presence of low concentrations [5]. Moreover, a large overvoltage of this reaction requires a very high applied potential, which leads to reduced selectivity, in addition, the products of the oxidation reaction usually poison the electrode surface [6]. A great deal of interest still exists in developing materials capable of electrocatalyzing glucose in order to reduce the typical overvoltages that are encountered in direct reduction at most electrode surfaces. Therefore, the electrooxidation of glucose on non-precious metals, especially nickel and its compounds, has been greatly developed in recent years, which is characterized by its low cost, high oxidation capacity and high stability. Glucose oxidation is an anodic reaction in a direct alkaline fuel cell and is used as a basis for enzyme-less detection [7-12].

A large number of transition metal oxides, including both bulk and nanostructures, such as NiO<sub>x</sub>, FeOOH, CuO, MnO<sub>2</sub>, RuO<sub>2</sub>, CuO<sub>x</sub>, ZnO and Co<sub>3</sub>O<sub>4</sub> have been reported as non-enzymatic electrocatalysts for glucose oxidation as an alternative to expensive noble metals. [13-17]. The catalytic ability of transition metal oxides such as CuO and NiO of nonenzymatic direct electrooxidation of glucose is one of the attractive properties in glucose detection allowing minimum fabrication cost, stable glucose sensors and environmental safety [18-22].

The oxidation of glucose, which is used in two basic applications, the first is in fuel cells, which is a hope that researchers seek to use as one of the alternatives to traditional energy sources, and the second is in the use of modified electrodes in the determination of glucose, suffers from glucose oxidation poisoning in the presence of potential toxins, especially the chloride ion. Therefore, it is imperative for researchers working in this regard to study the effect of halide ions on the electrochemical oxidation of glucose, and this is what researchers always do when studying the electrochemical oxidation of glucose [23, 24-26]. The effect of the halide ion does not stop at the poisoning of glucose oxidation, but extends to the oxidation of small organic molecules. Therefore, the effect of halide ions on the oxidation of these molecules at the noble electrodes such as gold and platinum has been studied. It is remarkable that nickel oxides have a high resistance to halide poisoning through their resistance to adsorption. Metal oxides such as CuO<sub>x</sub> and NiO<sub>x</sub>, although they are also subject to poisoning, occur to a lesser extent compared to their metal counterparts, as the susceptibility to halide ions changes on these oxides [33, 34]. In this study, an electrochemically oxidized glass carbon electrode modified with nickel oxide nanoparticles was prepared by a specific method from precipitation media containing additives that enhance the preparation of the electrode, catalyze the electrochemical oxidation of glucose and enhance its resistance to poisoning by halide ions.

## 2. EXPERIMENTAL

### 2.1. Chemicals.

The chemicals used in this research were prepared from high purity materials purchased from Sigma Aldrich and Merck and used as is. The solutions used were prepared using deionized water.

### 2.2. Electrochemical Measurements.

Electrochemical measurements were accomplished in a three-electrode electrochemical cell using a Gamry Instruments (Potentiostat / galvanostat / ZRA model Reference 600™) software, where platinum spiral and the silver/silver chloride (Ag/AgCl/ KCl (sat.)) were served as the counter and reference electrodes, respectively. All potentials will be presented with respect to this reference electrode. Glassy carbon (GC) electrode (3 mm in diameter) was employed as the working electrode. Prior to the loading of the modifier, the surface of the GC electrode was mechanically polished with aqueous slurries of successively finer alumina powder with the help of a microcloth, then was washed thoroughly with deionized water followed by sonication in distilled water for 5 minutes. The measurements were conducted at room temperature (25 °C).

### 2.3. Preparation of $GC_{ox}/NiO_x$ (Glu)Electrode.

GC-modified nickel oxide (GC/NiO<sub>x</sub>) nanoparticles were prepared by activating the GC electrode in 0.5 M H<sub>2</sub>SO<sub>4</sub> as cited in literature [12], designated as GC<sub>ox</sub>. The activation of the GC electrode was using cyclic voltammetry in 0.5 M H<sub>2</sub>SO<sub>4</sub> for certain number of cycles in the potential range – 0.2 to 2 V vs. Ag/AgCl/ KCl (sat.). Next, the GC<sub>ox</sub> was subjected to 5 potential cycles, n the range of 0.0 to -1.0 V, in a solution containing a nickel bath (0.02 M NiSO<sub>4</sub>•6H<sub>2</sub>O + 0.03M NiCl<sub>2</sub>•6H<sub>2</sub>O + 0.03M H<sub>3</sub>BO<sub>3</sub>), in the presence of 5 mM glucose as an additive that is expected to improve nickel deposition. Then, manipulation of the electrocatalytic activity of the modified electrode was performed at different concentrations of glucose, and different loading of nano-NiO<sub>x</sub>, both in halide free and halide containing glucose solutions. To demonstrate the reproducibility of the results, the CVs were repeated several times.

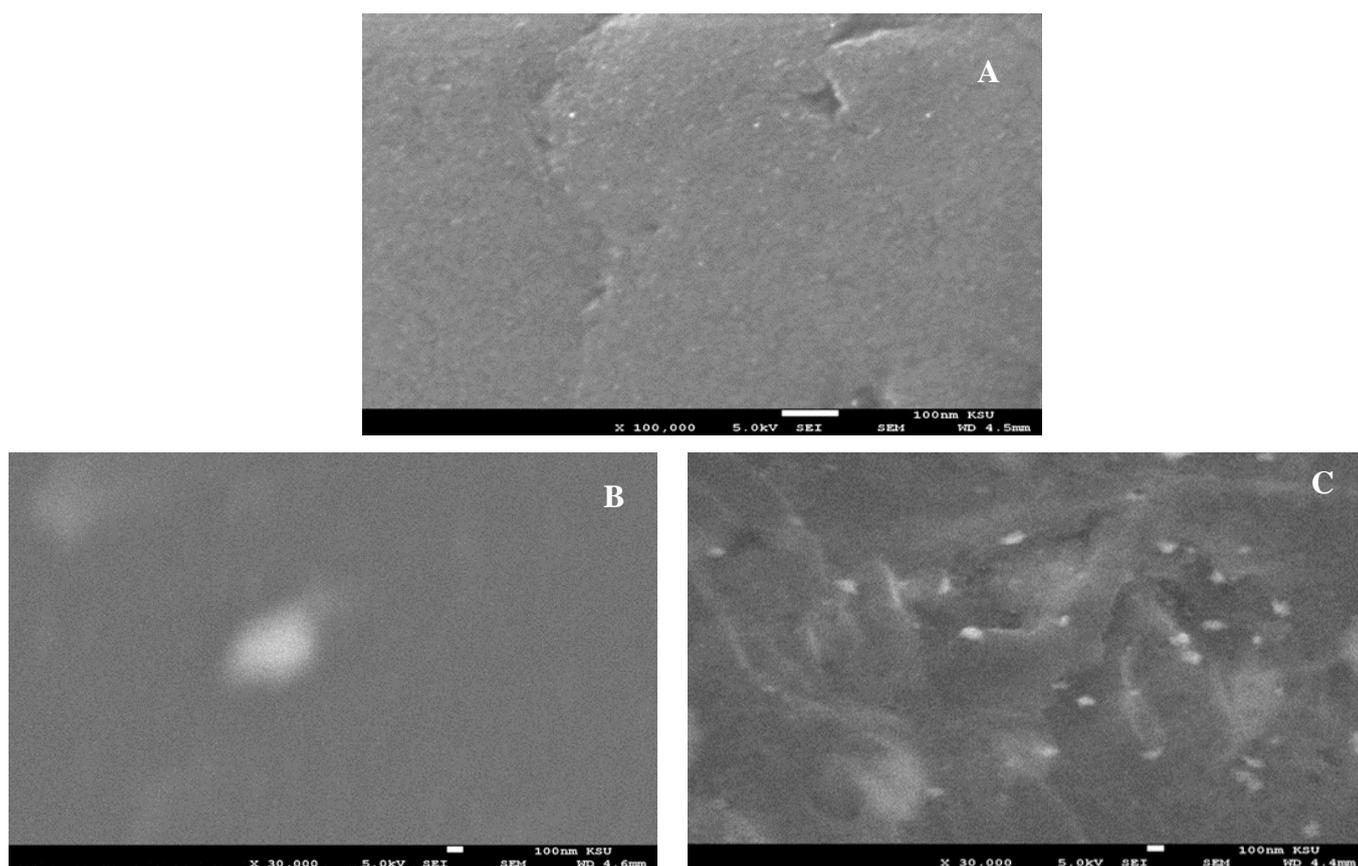
### 2.4. Characterization.

The morphology of the catalyst layer formed on the modified GC electrodes was studied using a field emission scanning electron microscope, FE-SEM, (QUANTA FEG 250).

### 3. RESULTS AND DISCUSSION

#### 3.1. Morphological characterizations.

Fig. 1 shows SEM images of Bare GC, GC/NiO<sub>x</sub> (Glu) and GC<sub>ox</sub>/NiO<sub>x</sub> (Glu) electrodes prepared by a cyclic voltammetry technique described in the experimental section. In image (A) a smooth surface is shown for bare GC electrode. In case of GC/NiO<sub>x</sub> (Glu) electrode, i.e., at nickel oxide deposited onto GCE in the presence of glucose in the deposition bath, a deposition of NiO<sub>x</sub> is not clearly observed, image (B). At GC<sub>ox</sub>/NiO<sub>x</sub> (Glu) electrode which is prepared in the same manner as GC/NiO<sub>x</sub> (Glu) but from a deposition bath containing glucose (image (C)), nickel nanoparticles of size ca. 50 nm are revealed. The chemical composition and the crystallographic orientation is reported elsewhere [35].



**Figure 1.** SEM of (A) GC, (B) GC/NiO<sub>x</sub>(glu) and (C) GC<sub>ox</sub>/NiO<sub>x</sub> (glu) electrodes.

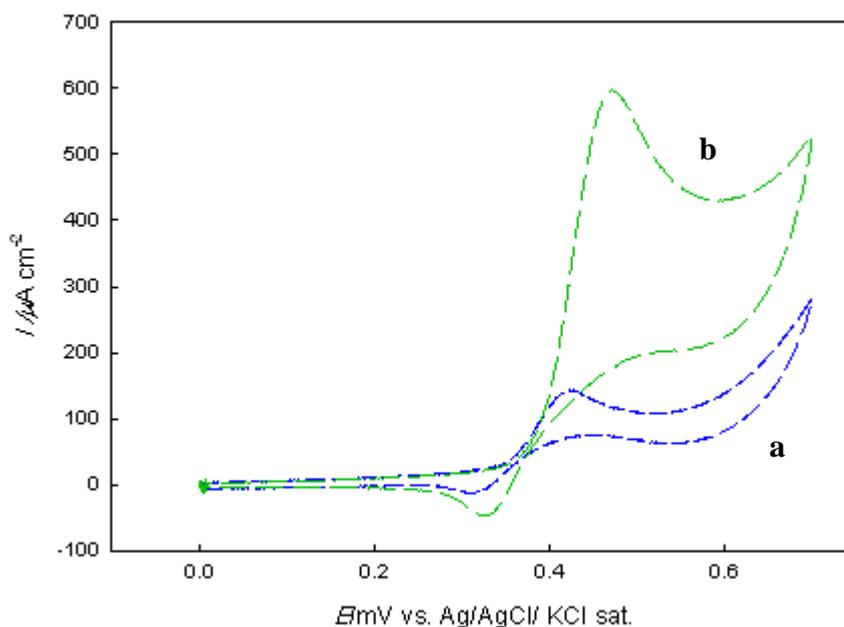
#### 3.2. Electrochemical characterizations.

Fig. 2 shows CVs responses obtained (a) GC/NiO<sub>x</sub>(Glu) and (b) GC<sub>ox</sub>/NiO<sub>x</sub> (Glu). Inspection of this figure reveals several interesting points.

- i. At GC/NiO<sub>x</sub> (Glu) (curve a), a well-defined oxidation response for the oxidation of glucose is acquired.

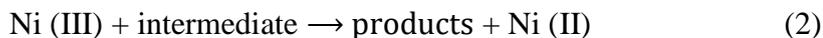
ii. At  $\text{GC}_{\text{ox}}/\text{NiO}_x$  (Glu) (curve b), the oxidation of glucose is significantly enhanced, with the one at the latter is larger indicating the significant effect of the underlying substrate as well as the controlling of the deposition bath composition. The prominent role of the substrate surface was demonstrated by comparing curves (a) and (b) in which nickel was deposited from a glucose-containing bath, the only difference being the state of the substrate. It has been documented that the oxidation of glassy carbon strongly affects the behavior of the electrode and what is deposited on this electrode, and this is the result of the different functional groups that are formed on the surface and that can be controlled using electrochemical methods. Electrochemically oxidized glass carbon affects the electrochemical reactions according to its richness in different functional groups. For example, the electrode rich in hydroxyl groups was found to stimulate the oxidation of glucose [36] and other small organic molecules [37].

iii. At the electrode,  $\text{GC}_{\text{ox}}/\text{NiO}_x$  (Glu), that was prepared from media containing glucose, it was found that its effect on the oxidation of glucose in alkaline media doubled compared to what if it was made from media that did not contain glucose.. In previous studies, the stimulation of electrodes modified with nickel oxides was explained that the oxidation takes place through two pathways: one of them is oxidation on the surface of the nickel oxides and the other is through mediation by  $\text{NiOOH}$  [38].



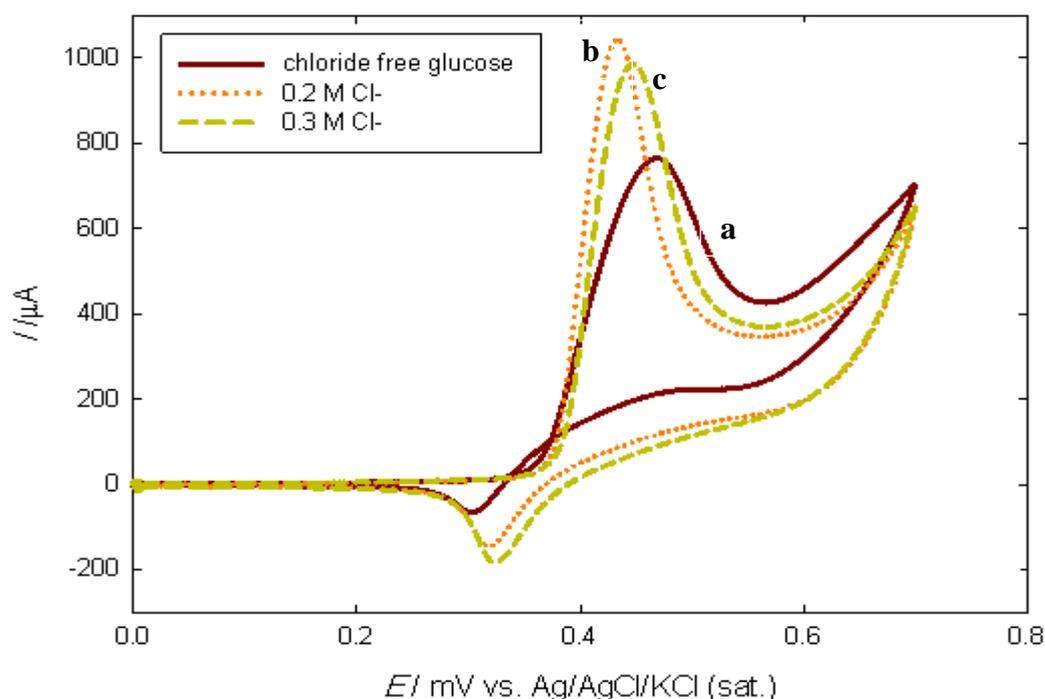
**Figure 2.** CV obtained at (a)  $\text{GC}/\text{NiO}_x$  (Glu) and (b)  $\text{GC}_{\text{ox}}/\text{NiO}_x$  (Glu) electrodes in 0.5 M NaOH containing 2.5 mM glucose at  $\text{SR} = 100$  mV/s.

It has been documented that the oxidation of glucose takes place as shown in the following equations, in which the nickel(II) is oxidized to the nickel (III), then followed by the oxidation of glucose on the resulting surface, which is produced with each cycle:



where nickel(III) is produced using the energy source, as well as through direct oxidation of nickel(II). [39,40]:

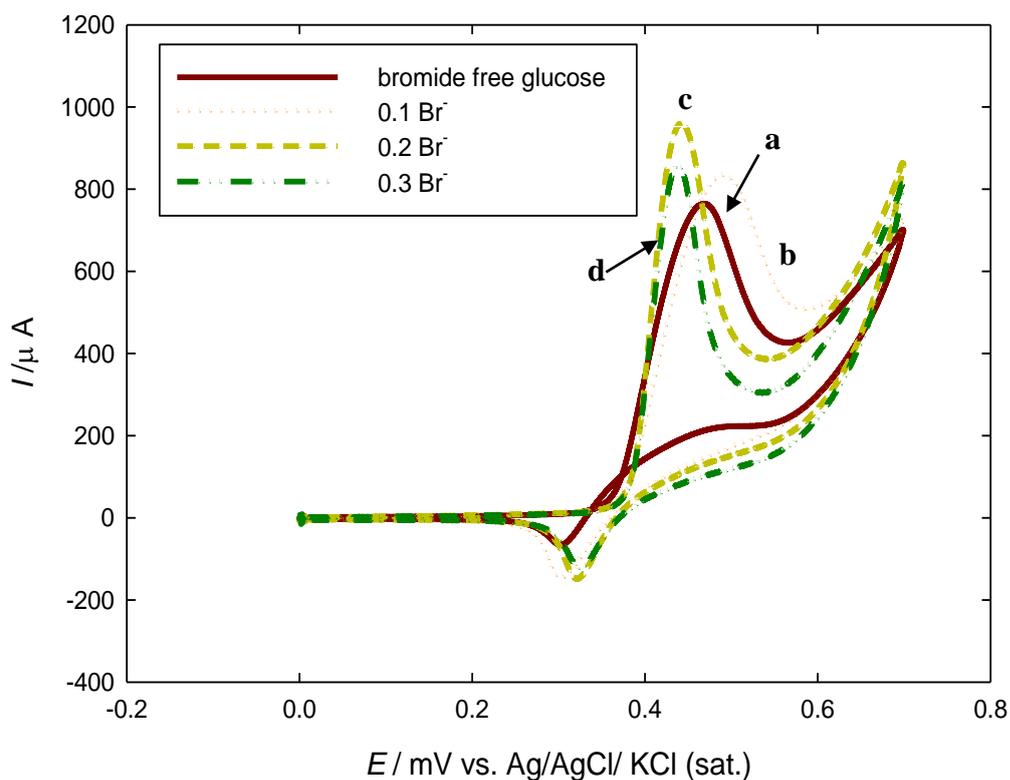
In this section the oxidation susceptibility of glucose in electrodes modified for halide poisoning is studied.



**Figure 3.** CV obtained at  $\text{GC}_{\text{ox}}/\text{NiO}_x$  (Glu) electrodes in 0.5 M NaOH containing 2.5 mM glucose in the presence of different concentrations of the halide ions  $[\text{Cl}^-]$  (a) chloride free glucose solution, (b) 0.2M, and (c) 0.3M, at scan rate of 100 mV/s.

Figures 3-5 show the responses of cyclic voltammetry (CV) to the electrooxidation of glucose on a  $\text{GC}_{\text{ox}}/\text{NiO}_x$  (Glu) electrode in alkaline media contains a fixed concentration of glucose and different concentrations of halide ions. Glucose oxidation was detected at 0.5 V versus Ag/AgCl (KCl sat.). As mentioned above,  $\text{NiOOH}$  acts as a mediator of glucose oxidation, i.e., Ni (II) is converted to Ni (III), and subsequently glucose is oxidized on  $\text{GC}_{\text{ox}}/\text{NiO}_x$  (Glu) electrode, that is, via the EC mechanism [41-43]. Halide ions affect the electrochemical behavior of the modified electrodes, and this effect depends on the type and concentration of the halide. The shift in both cases is halide

concentration dependent. For example, in the presence of a chloride ion whose concentration 0.2 M increases in the forward scan by a percentage of ca. 25 %, and in the presence of a concentration of 0.3 M it increases by 23 %. In the case of bromide, it is similar to chloride in behavior, but the increase in the peak current in this case is smaller. In case of iodide a much lower concentration is used, 10  $\mu\text{M}$   $\text{I}^-$  is used. In this case, even though the concentration of iodide is around four order of magnitude lower than chloride and bromide, the increase in the peak current is larger. Probably iodide is oxidized at the same potential of glucose. The effect of halide, increase in the order  $\text{I}^- > \text{Cl}^- > \text{Br}^-$ , even though the concentration of iodide is of different order compared with chloride and bromide. This is attributed to the high adsorbability of iodide ions, which increases its concentration to completely blocking of the electrode surface towards the oxidation of glucose.

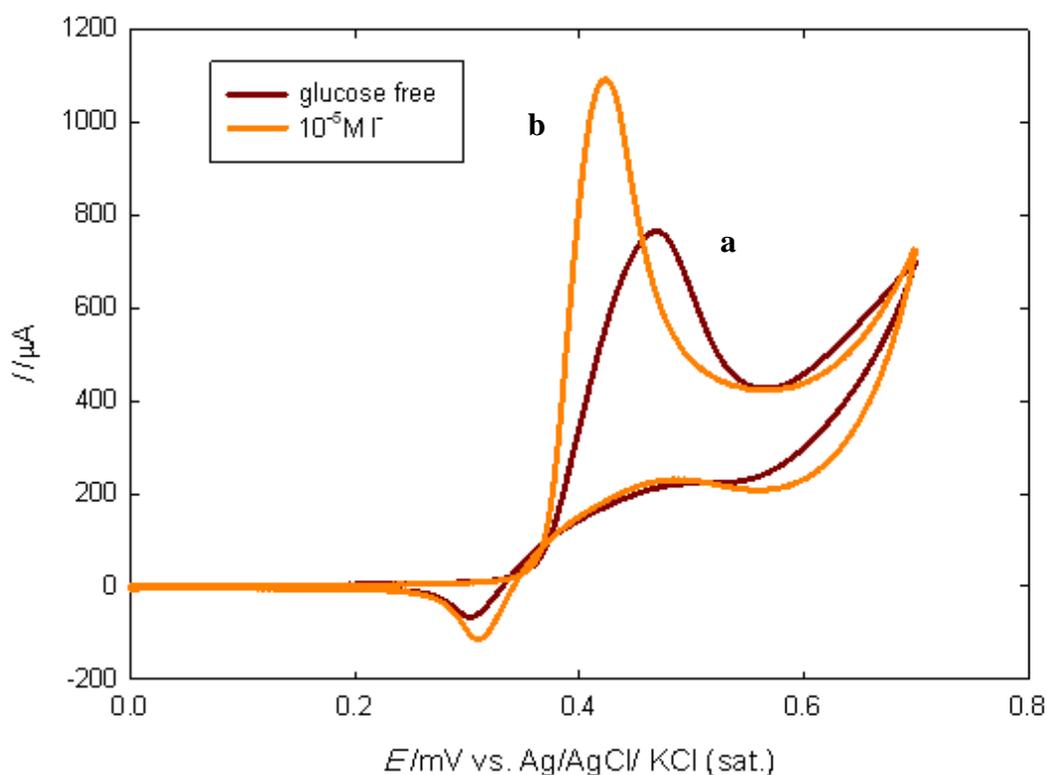


**Figure 4.** CV obtained at  $\text{GC}_{\text{ox}}/\text{NiO}_x$  (Glu) electrodes in 0.5 M NaOH containing 2.5 mM glucose in the presence of different concentrations of the halide ions (a) bromide free glucose, (b) 0.1M, and (c) 0.2M, and (d) 0.3M at scan rate of 100 mV/s.

The literature indicated that iodide adsorption depends on the potential difference on the electrode surface. So, it is adsorbed in the form of atomic state in the voltage between  $-0.4$  and  $+0.4$  V (Ag/AgCl reference), and that the adsorption increases with increasing voltage to values greater than  $+0.4$  V, which results in more adsorption of iodine on the limited electrode surface area, which is also limited by Van der Waals forces. Excess of iodine on the surface results in the formation of molecular iodine, which turns into an aqueous solution.  $\text{I}_2$  ( $2\text{I}^- \leftrightarrow \text{I}_2 + 2\text{e}^-$ ). At high positive potential values,

zerovalent iodine is converted to aqueous iodate ( $I_2 \leftrightarrow IO_3^-$ ), this is done before the oxygen evolution. The  $IO_3^-$  formed at the electrode surface diffuses into the solution and reacts with the iodide forming iodine again [44-46]. As it is clear, the forward current in the absence of the halide is less than in its presence. This is explained by the coincidence of the oxidation of glucose with the oxidation of the adsorbed zerovalent iodine to iodine or aqueous iodine molecules, and these are the possible oxidation process within the studied potential range.

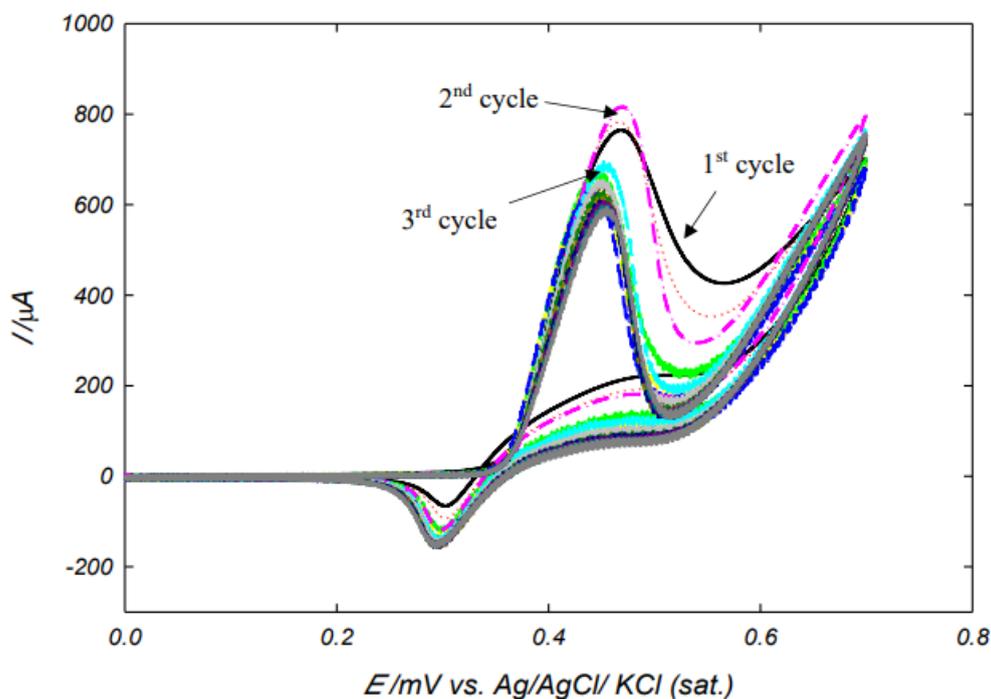
The literature has indicated that the GC/NiO<sub>x</sub> electrode has a tolerance to halide ions poisoning, and this means that the results presented in this research agree with the literature [34]. Moreover, the current electrode, in addition to its resistance to halide poisoning, gives stability to the current, and that the current is not less than that measured in the absence of halide ions even after several potential cycles. This makes it a promising electrode for glucose analysis and for fuel cells.



**Figure 5.** CV obtained at GC<sub>ox</sub>/NiO<sub>x</sub> (Glu) electrodes in 0.5 M NaOH containing 2.5 mM of glucose in the presence of the halide ions [I<sup>-</sup>] (a) iodide free glucose, and (b) 10<sup>-5</sup> M at a scan rate of 100 mV/s.

It should be clarified that the above results (Figures 3-5) was taken immediately after adding the halide ion concentration to the alkali solution containing 20 mM of glucose. To explain the effect of adding the halide ion on the oxidation of glucose, the cyclic voltammetry were conducted ring Xs 20 times in the presence of the halide (Fig. 6), and the values of the anodic peak current were recorded and plotted against the number of cycles as shown in a figure 7. As shown, the current decreases

continuously; the peak current after 15 cycles is around 0.8 of the initial runs. This may point to the poisoning of the electrode by the oxidation product.



**Figure 6.** CV obtained at  $\text{GC}_{\text{ox}}/\text{NiO}_x$  (Glu) electrodes in 0.5 M NaOH containing 2.5 mM of glucose (halide-free) at a scan rate of 100 mV/s for 15 cycles.

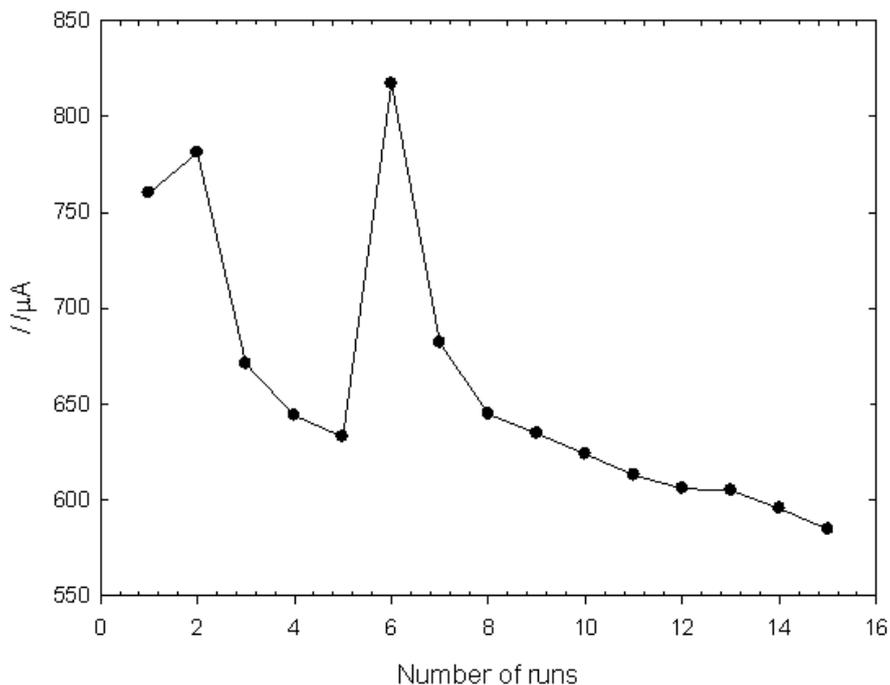
Fig. 8 and 9 shows the continuous cycling of potential in glucose solution containing  $\text{Cl}^-$  and  $\text{Br}^-$ , respectively. To compare the effect of chloride and bromide poisoning, what happened with chloride was repeated, that is, the measurement of the cyclic voltammetric behavior in the presence of bromide ions were repeated 20 times, then the anodic peak current was recorded, and then the plotted against the number of cycles, as shown in the figure 10.

The following points are extracted from Fig. 10.

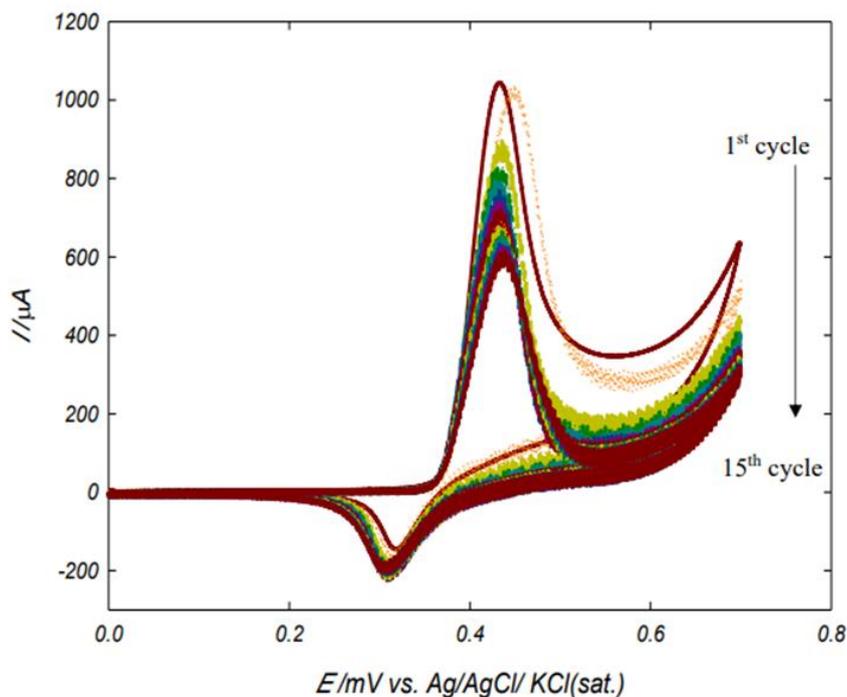
i. It was found that the oxidation current peak in the absence of chloride and bromide is lower than in their presence, as is evident by comparing the results in Figure 10 with that in Fig. 7 By repeating the potential cycles, it was found that the oxidation current peak decreases, but the current value in the last measured cycle remains higher in the presence of the halide ion than in the absence of it.

ii. The rate in the decrease of oxidation in the presence of halide does not occur by the same amount, it is in the presence of halide, and if it is approximately equal, its decrease in the absence of it is larger.

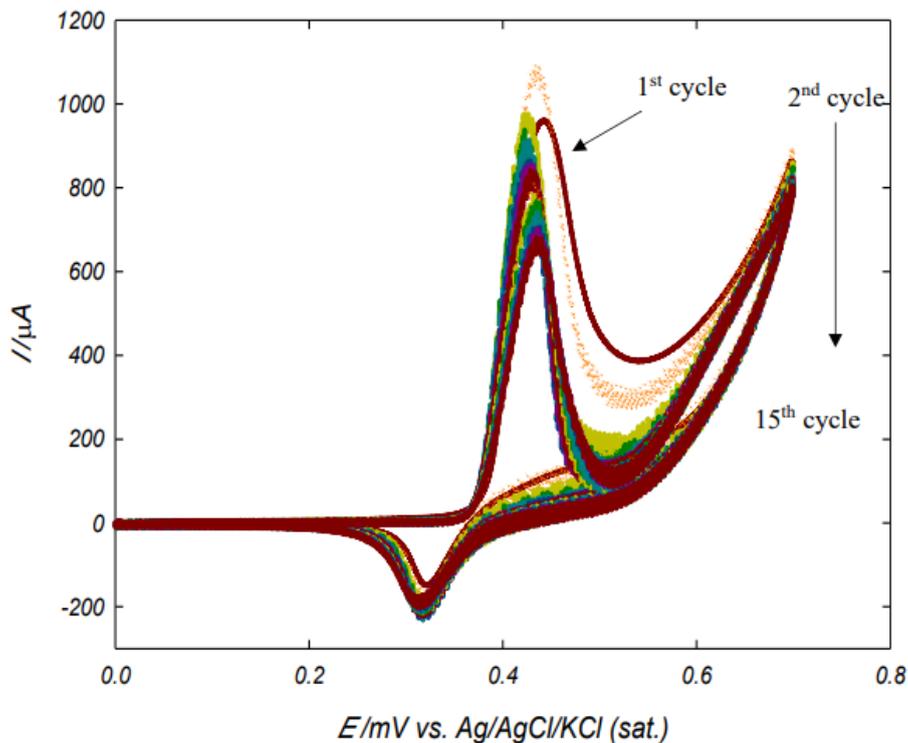
iii. Although the value of the anodic peak current varies in the presence of halide ions and the absence of it, the method of decreasing in anodic peak current remains almost the same.



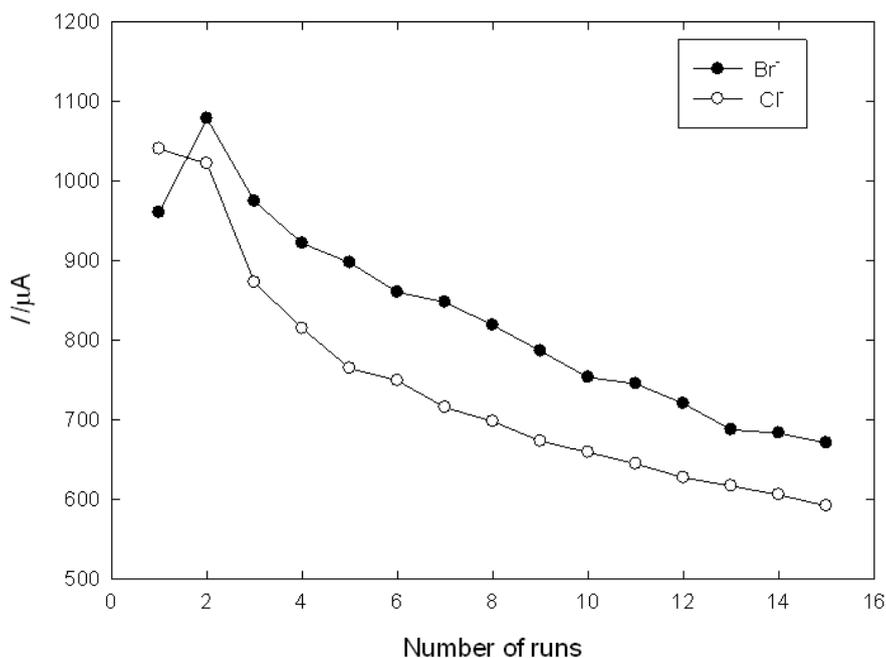
**Figure 7.** Relation between peak current for CV's scan at each run and number of runs.



**Figure 8.** CV obtained at GC<sub>ox</sub>/NiO<sub>x</sub> (Glu) electrodes in 0.5 M NaOH containing 2.5 mM glucose in the presence of Cl<sup>-</sup> (0.2M) at a scan rate of 100 mV/s for 15 cycles.



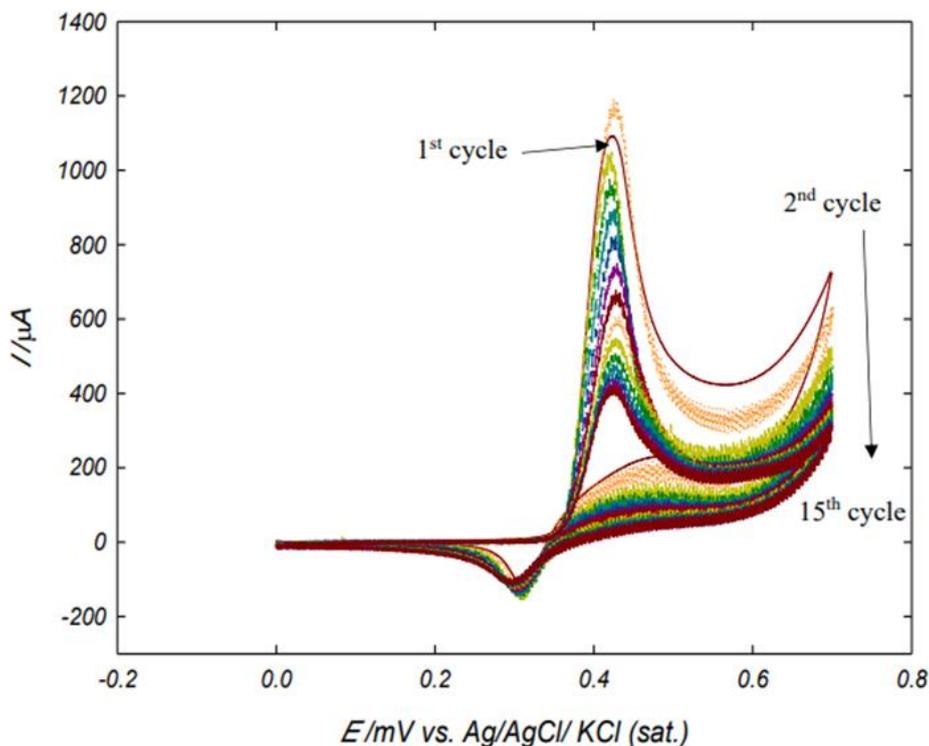
**Figure 9.** CV obtained at  $GC_{ox}/NiO_x$  (Glu) electrodes in 0.5 M NaOH containing 2.5 mM of glucose in the presence of the  $Br^-$  (0.2M) at a scan rate of 100 mV/s for 15 cycles.



**Figure 10.** Relation between peak current for CV's scan at each run and number of runs data were extracted from figures 6, 8, and 9.

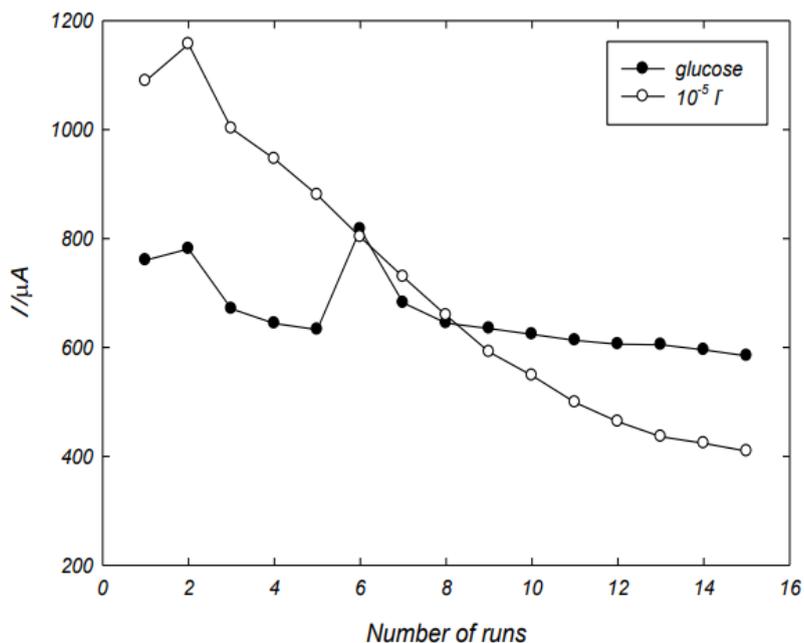
These results show the ability of the  $\text{GC}_{\text{ox}}/\text{NiO}_x$  (Glu) electrode in this research to be highly resistant to poisoning by chloride and bromide ions, and this is due to their low adsorption capacity in addition to their high oxidation potential, for  $\text{Cl}_2/\text{Cl}^-$  and  $\text{Br}_2/\text{Br}^-$  reactions (1.36 and 1.06 V vs. normal hydrogen electrode (NHE), respectively) [47].

And as was done in the case of chloride and bromide, it was also done in the case of iodide under the same conditions, but in the presence of very low concentrations of iodide (v). As shown in this figure, with the increase in the number of potential cycles, the forward current decreases.



**Figure 11.** CV obtained at  $\text{GC}_{\text{ox}}/\text{NiO}_x$  (Glu) electrodes in 0.5 M NaOH containing 2.5 mM of glucose in the presence of the halide ions,  $\text{I}^-$  ( $10^{-5}$  M) at scan rate of 100 mV/s for 15 cycles.

In Figure 12, oxidation current was also plotted with the number of cycles, and the behavior obtained in this case is different from the previous ones, as it was found that the oxidation current increases with the first cycle and then gradually decreases as the previous one. This can be explained by the presence of two reactions, i.e. the oxidation of glucose and iodide, and that both affect the other, and in the end, the oxidation current is the result of these two reactions and their effect on each other.



**Figure 12.** Relation between peak current for CV's scan at each run and number of runs. Data were extracted from figure 11.

#### 4. CONSLUSIONS

The electro-oxidation of glucose has been examined in an alkaline medium using the GC<sub>ox</sub>/NiO<sub>x</sub>(glu) electrode in the presence of halide ions (Cl<sup>-</sup>, Br<sup>-</sup>, I<sup>-</sup>). The electrochemical measurements demonstrate the enhanced electrocatalytic performance of modified electrode for the electro-oxidation of glucose prepared by the NiO<sub>x</sub> coupled with different halide ions as compared to those synthesized using a NiO<sub>x</sub> modified atop bare glassy carbon electrode. Although the presence of halide ions in a high concentration leads to poisoning of the modified electrode towards the oxidation of glucose, its presence in a low concentration leads to the stimulation of glucose oxidation. Also it leads to a shift in the glucose electrooxidation potential to more negative potential values compared to those without added halide. This demonstrates the improved electrocatalytic activity of the GC<sub>ox</sub>/NiO<sub>x</sub>(glu) in the presence of halide additive for the electro-oxidation of glucose in an alkaline medium.

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