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Tolerance of glucose electrocatalytic oxidation on NiO\textsubscript{x}/MnO\textsubscript{x}/GC electrode to poisoning by halides

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Awad
M. I.
Cairo University
Department of Chemistry, Faculty of Science
Cairo, Egypt
mawad70@yahoo.com

El-Refaei
S. M.
Cairo University
Department of Chemistry, Faculty of Science
Cairo, Egypt

Saleh
M. M.
Cairo University
Department of Chemistry, Faculty of Science
Cairo, Egypt
mahmoudsaleh90@yahoo.com

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This paper is devoted to the investigation of the tolerance of glucose oxidation on NiO$_x$/MnO$_x$/GC binary catalyst to poisoning by halide ions. NiO$_x$/MnO$_x$/GC electrode was prepared by the consecutive electrodeposition of MnO$_x$ followed by the deposition of NiO$_x$ nanoparticles atop the previously deposited MnO$_x$ nanoparticles. The electrode was characterized electrochemically and morphologically. Interestingly, experimental results showed that the NiO$_x$/MnO$_x$/GC electrode shows a superior electrocatalytic activity compared with the NiO$_x$/GC electrode and a behavior comparable to the NiO$_x$/GC electrode regarding tolerance to poisoning by halides, even in the presence of high concentrations of Cl$^-$ and Br$^-$ up to 0.3 M. On the other hand, it was found that iodide ions have a significant effect on glucose electrooxidation under the prevailing operating conditions and even at concentrations lower than those of the other two halide ions.

Keywords
Glucose - Manganese oxide - Nickel oxide - Halide - Nanoparticles

Foot note information
Tolerance of glucose electrocatalytic oxidation on NiO\textsubscript{x}/MnO\textsubscript{x}/GC electrode to poisoning by halides

S. M. El-Refaei · M. M. Saleh · M. I. Awad

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Abstract This paper is devoted to the investigation of the tolerance of glucose oxidation on NiO\textsubscript{x}/MnO\textsubscript{x}/GC binary catalyst to poisoning by halide ions. NiO\textsubscript{x}/MnO\textsubscript{x}/GC electrode was prepared by the consecutive electrodeposition of MnO\textsubscript{x} followed by the deposition of NiO\textsubscript{x} nanoparticles atop the previously deposited MnO\textsubscript{x} nanoparticles. The electrode was characterized electrochemically and morphologically. Interestingly, experimental results showed that the NiO\textsubscript{x}/MnO\textsubscript{x}/GC electrode shows a superior electrocatalytic activity compared with the NiO\textsubscript{x}/GC electrode and a behavior comparable to the NiO\textsubscript{x}/GC electrode regarding tolerance to poisoning by halides, even in the presence of high concentrations of Cl\textsuperscript{−} and Br\textsuperscript{−} up to 0.3 M. On the other hand, it was found that iodide ions have a significant effect on glucose electrooxidation under the prevailing operating conditions and even at concentrations lower than those of the other two halide ions.

Keywords Glucose · Manganese oxide · Nickel oxide · Halide · Nanoparticles

Introduction

Glucose electrooxidation on non-precious metals, especially Ni and its composites, as an anodic reaction in direct alkaline fuel cells and also as basis for enzymeless detection has been greatly developed in recent years due to their low cost, high oxidation capacity, and high stability [1–5]. One of the most challenging problems of glucose fuel cells and non-enzymatic glucose sensors is poisoning by halides especially chloride ions. So, studying the effect of halide ions on fuel oxidation on the anode is considered a very important topic in electrocatalysis [3, 6–8]. It has been reported that halide ions exhibit a strong poisoning effect on glucose and methanol electrooxidation on noble metal electrodes such as Pt, Au, and their alloys [9–14]. The high poisoning of noble metals by halide ions is attributed to the high specific adsorption of the halide ions on such metals. In this context, a number of articles regarding this issue have been reported [9–14].

On the other hand, metal oxides such as CuO and NiO are susceptible to poisoning albeit to a lower extent compared to their metal counterparts. This is attributed to the difference in the adsorbatibility of halide ions on metal and metal oxides [15, 16]. In a previous report, it has been shown that the binary catalyst NiO\textsubscript{x}/MnO\textsubscript{x}/GC showed extraordinary enhancement of glucose electrooxidation in alkaline medium compared with NiO\textsubscript{x}/GC, MnO\textsubscript{x}/GC, or MnO\textsubscript{x}/NiO\textsubscript{x}/GC [4, 5]. As a continuation of our previous work, studying the effect of halide ion poisoning on our fabricated catalyst is important not only for application systems but also from a theoretical point of view.

Generally, using non-enzymatic sensors in the analysis of glucose often suffers from the drawbacks of low sensitivity and interference of other oxidizable components such as ascorbic acid and uric acid [15]. The adsorption of chlorides, which decrease significantly the operational stability, is another challenge. It has been reported that CuO\textsubscript{x}-modified electrodes are a candidate non-enzymatic catalyst for glucose oxidation in alkaline medium, and thus, poisoning by chloride has been extensively studied [15].

However, to the best of our knowledge, the effect of halide ion poisoning on the electrocatalytic activity of NiO\textsubscript{x} and/or MnO\textsubscript{x} nanoparticle-modified electrodes toward glucose oxidation is considered to be scarce. The purpose of the present work is to study the tolerance of glucose electrooxidation reaction on the proposed catalyst to poisoning by halides. The fabricated catalysts NiO\textsubscript{x}/GC and NiO\textsubscript{x}/MnO\textsubscript{x}/GC will be subjected to different concentrations of halide ions to study their impact on the performance of such electrodes toward glucose oxidation in alkaline medium.
Experimental

All chemicals used in this work were of analytical grade and were purchased from Merck and Sigma-Aldrich, and they were used as received without further purification. All solutions were prepared using second distilled water.

Measurements

Electrochemical characterizations were performed using an EG&G potentiostat (model 273A) operated with E-Chem 270 software. An electrochemical cell with a three-electrode configuration was used in this study. A platinum spiral wire and an Ag/AgCl/KCl (sat.) were employed as counter and reference electrodes, respectively. All potentials are presented with respect to the reference electrode. The working electrode was glassy carbon (diameter = 3.0 mm). It was cleaned by mechanical polishing with aqueous slurries of successively finer alumina powder (down to 0.06 μm) and then washed thoroughly with second distilled water. Scanning electron microscope (SEM) images were taken using a field emission scanning electron microscope (FEI, QUANTA FEG 250).

Electrode modification

NiOx/MnOx/GC was prepared by consecutive electrodeposition of MnOx followed by the deposition of NiOx nanoparticles atop the previously deposited MnOx nanoparticles onto a glassy carbon electrode (GCE). MnOx was electrodeposited by cycling the potential from 0 to 0.4 V at a scan rate of 20 mV/s in 0.1 M Na2SO4 containing 0.1 M Mn(CH3COO)2·5H2O for 60 cycles, and then it was activated by cycling the potential for 5 cycles in 0.5 M NaOH solution in the potential range −0.2 to 0.6 V at a scan rate of 100 mV/s [17]. Next, modification with NiOx was achieved in two sequential steps, i.e., the potentiostatic deposition of metallic nickel on the previously deposited MnOx (i.e., onto MnOx/GCE) from an aqueous solution of 0.1 M acetate buffer solution (pH = 4.0) containing 1 mM Ni(NO3)2·6H2O by applying constant-potential electrolysis at −1.0 V for different time durations. Then the passivation of the thus electrodeposited Ni was achieved in 0.1 M phosphate-buffered solution (pH = 7) by cycling the potential between −0.5 and 1 V for 10 cycles at a scan rate of 200 mV/s. Next, activation for 5 cycles in 0.5 M NaOH solution in the potential range −0.2 to 0.6 V was conducted [18, 19].

Results and discussion

Material characterization

Figure 1 shows the energy-dispersive X-ray (EDX) spectrum of the NiOx/MnOx/GC electrode prepared as mentioned in the "Experimental" section. This figure reveals the peaks of manganese and nickel at 5.9 and 7.5 keV in quantities of 5.4 and 0.5 by weight percent, respectively. This sustains that both NiOx and MnOx are exposed in the same sample and that NiOx does not fully cover the previously deposited MnOx, consistent with the SEM image shown as inset in which the well-known porous texture of MnOx is obscured by the subsequent deposition of NiOx [20]. More discussion regarding the SEM images of the different catalysts can be found elsewhere [4, 5].

Glucose electrooxidation

Glucose electrooxidation reaction on the NiOx and MnOx single and binary catalyst-modified glassy carbon electrode at different orders and loading levels of the two oxides has been previously reported by our group [4, 5]. It has been reported that the NiOx/MnOx/GC electrode (MnOx deposited first) shows an excellent electrocatalytic activity and stability toward glucose oxidation compared with NiOx/GC, MnOx/GC, or MnOx/NiOx/GC electrodes (NiOx deposited first). In those reports, we also found that the optimum loading level is 60 potential cycles of MnOx followed by 10 min of nickel deposition. Thus, in the present study, those conditions were selected for the preparation of the NiOx/MnOx/GC electrode as cited in the "Experimental" section. The extraordinary activity obtained on this electrode was attributed to the compilation of the better adsorption of glucose molecules on the MnOx sites and the increase of conductivity of NiOx due to the increase of Ni3+ content, taking in consideration that both oxides are exposed to the solution as has been reported [4, 5].

In the present work, the poisoning effect of halide ions on glucose electrooxidation on the proposed electrocatalyst (NiOx/MnOx/GC) will be presented. It is noteworthy to mention that NiOx/MnOx/GC has been reported to show extraordinary electrocatalytic glucose oxidation compared with the single catalyst (NiOx/GC) [4, 5]. Thus, the study on halide tolerance is essential for this electrode being applied in fuel cells and in non-enzymatic detection of glucose. As the effect of chloride and bromide ions is expected to be significantly different from that of iodide, results regarding chloride and bromide ions will be presented as one set and then poisoning by iodide ions as another set.

Poisoning effect of chloride and bromide ions

Figure 2 shows the cyclic voltammetry (CV) responses for glucose electrooxidation on the NiOx/MnOx/GC electrode in the presence of different concentrations of (a) chloride and (b) bromide ions, ranging from 0.1 to 0.3 M. Glucose oxidation is revealed at a potential around 0.53 V. It has been reported that NiOOH acts as a mediator of glucose oxidation. The redox transition of nickel species from Ni(II) to Ni(III) occurs, and then in a following step, glucose is oxidized on the modified...
surface, i.e., via EC mechanism. Gluconolactone as well as methanoates and oxalates, as a result of the further oxidation of gluconolactone, has been reported as the possible oxidation product of glucose electrooxidation [5, 21–23].

Interestingly, upon the addition of the corresponding halide ions, chloride or bromide ions, there is no observed significant effect on the forward and backward scans even at high concentrations of the two halide ions. It has been reported [16] that the NiO$_x$/GC electrode has strong tolerance to chloride ions which is consistent with the present results. However, the present electrode has the advantage of higher electrocatalytic activity compared to the NiO$_x$ electrode. This indicates that the proposed binary catalyst exhibits good resistance to surface fouling and is a promising electrocatalyst for the development of enzymeless glucose sensors and alkaline fuel cells at low cost.

The above recorded results are the first run after the direct addition of halide ions. In order to further demonstrate the poisoning effect, various numbers of potential scans (cycles) were recorded in the presence of both the glucose and the halide ions. Similar scans were recorded in halide-free 20 mM glucose in 0.5 M NaOH solution for the sake of comparison. Figure 3 shows consecutive linear sweep voltammetry (LSV) responses for the forward (I) and backward (II) scans obtained on the NiO$_x$/MnO$_x$/GC electrode in 0.5 M NaOH containing 20 mM glucose (halide-free solution) at a scan of rate 100 mV/s. To quantitatively demonstrate the effect of halide ions during potential scan cycling, we can correlate the peak current, which corresponds to glucose oxidation, to the number of potential cycles as it appears in Fig. 4 (the data were taken from Fig. 3).

In Fig. 3 the peak current firstly decreases to a minimum value at run 4, and then it increases again to values close to the initial peak current. This may be attributed to the fact that the electrode surface contains more than one type of active sites with varied activity toward glucose electrooxidation. At the start of the first four runs, glucose oxidation begins to occur at all active sites, but some of the active sites are poisoned by the oxidation products much faster than the other sites. Hence, the peak current decreases reaching the minimum value, albeit it is still high due to glucose oxidation at the other active sites on the electrode surface. The current begins to increase again and continues in a varied manner as the scan number increases. This increase may be attributed to many factors such as the poisoning species on the electrode surface that might be oxidatively recovered after a number of runs due to the regeneration of poisoned active sites. Also, Ni$^{2+}$/Ni$^{3+}$ redox couple transformation increases in the alkaline medium by increasing the potential scan cycles. The last fact is illustrated in Fig. 5 in which further potential cycling of the previously activated NiO$_x$/MnO$_x$/GC electrode in 0.5 M NaOH solution is conducted. Run 1 is recorded after activation of the catalyst in 0.5 M NaOH for 25 cycles to obtain the well-defined redox waves of the Ni$^{2+}$/Ni$^{3+}$ transformation system. The peak current of the Ni$^{2+}$/Ni$^{3+}$ couple increases with increasing potential cycling in glucose-free 0.5 M NaOH. This provides more active sites to glucose oxidation, and thus, the current increases again. Also, Fig. 3 proves that the initial decrease in the peak current is about 10 μA (2 % decrease from the original peak current), and this is considered negligible. Thus, the results reveal that the electrode response toward glucose oxidation is mainly constant within 15 cycles.

Figure 6a, b shows the consecutive LSV responses for the forward scan (I) and backward scan (II) obtained on the NiO$_x$/
MnO_x/GC electrode in 0.5 M NaOH containing 20 mM glucose and 0.1 M Cl^− (a) and 0.1 M Br^− (b), respectively. To quantitatively compare the poisoning effect of Cl^− and Br^− ions, the oxidation peak currents for the forward scan in the halide-free glucose and after adding Cl^− and Br^− are plotted as a function of the number of potential cycles as shown in Fig. 7.

Many features could be extracted from Fig. 7 in comparison with Fig. 6:

1. In the presence of Cl^− or Br^− ions, the oxidation peak current for the first cycle is similar to that in the absence of both species. With potential cycling, the peak current decreases in the presence of any of the two species, i.e., Cl^− or Br^−, albeit to different extent.

2. The decreasing rate of the oxidation peak current follows the sequence Br^− > Cl^− > halide-free (glucose only) solution. This expected behavior is due to the difference between Cl^− and Br^− ions in electronegativity, ionic radii, and solvation ability [24].

3. The peak currents change with the potential cycles with a regime similar to that obtained in the case of the halide-free (glucose only) solution (see Fig. 4). However,
The above results reveal the tolerance of glucose oxidation on the NiOₓ/MnOₓ/GC electrode to poisoning by chloride and bromide ions. This may be attributed to the low adsorbability and high solvation of Cl⁻ and Br⁻ ions. Also, it could be attributed to the high reversible oxidation potentials of Cl₂/Cl⁻ and Br₂/Br⁻ reactions (1.36 and 1.06 V vs. normal hydrogen electrode (NHE), respectively) [25].

Effect of iodide ion

Figure 8 shows the consecutive LSV responses for the forward (I) and backward (II) scans obtained on the NiOₓ/MnOₓ/GC electrode in 0.5 M NaOH containing 20 mM glucose at different iodide ion concentrations: (a) 0.0, (b) 10⁻⁵, (c) 10⁻⁴, (d) 10⁻³, and (e) 10⁻² M. Scan rate, 100 mV/s.
On the surface of Pt and Au anodes, at considerably more positive potentials and before oxygen evolution, the adsorbed zerovalent iodine is oxidized to aqueous iodate (\(I_2\rightarrow IO_3^-\)) along with the oxide formation of the electrode surface. Finally, it is quite probable that the iodates formed on the electrode diffuse back into the solution and react with the iodide, forming iodine again [26–28].

In Fig. 8 upon the addition of the iodide ions, a significant effect appears in the forward and backward scans at each concentration (curves b–e). For \(10^{-3}\) M \(\Gamma\) (curve b), the LSV responses for the forward and backward scans are similar to those for the iodide-free glucose solution (curve a), and there is no significant change in both the forward and backward scans. In general, there are two main reactions (stated above) that contribute to the forward current. The current in the forward and backward directions is considered as a combination of the previously mentioned processes.

For \(10^{-4}\) M \(\Gamma\) (curve c), again the forward scan is similar to that for the iodide-free glucose solution while the backward current is lower than that for the iodide-free glucose solution (curve a). The forward scan current does not decrease as was expected by the poisoning effect of iodide. This can be understood if we consider that the decrease in the current of glucose oxidation due to poisoning by adsorbed iodide is outweighed by the oxidation of the adsorbed \(I_2\) to \(IO_3^-\). Hence, the current in the forward scan is kept almost unchanged. On the other hand, the decrease in the backward current in the presence of iodide ions might be attributed to the adsorption of iodine or iodates formed in the forward scan that block some of the active sites. Another factor is the presence of another reduction reaction of iodide derivatives in parallel to \(Ni^{3+}\) reduction; the combination of these two processes can decrease the overall oxidation current of glucose in the backward scan, so glucose oxidation current in the backward scan decreases in comparison with the iodide-free glucose case.

In the case of \(10^{-2}\) M \(\Gamma\) (curve d), the forward scan current is higher than that in the iodide-free glucose case and the backward scan current is lower than that in the iodide-free glucose case (curve a). The increase in the forward scan may be attributed to the oxidation of the adsorbed zerovalent iodine to aqueous iodine atoms and/or iodates as they are available in this potential range as revealed from Fig. 9 (curve d), in which the CV responses are obtained on \(NiOOH\)/MnO\(_2\)/GC electrode in 0.5 M NaOH containing 20 mM glucose and \(10^{-4}\) (a), \(10^{-3}\) (b), and \(10^{-2}\) (c) M \(\Gamma\), respectively. We can see that with the increase in the number of potential cycles, both the forward and backward currents decrease, and this decrease depends on the iodide concentration. In addition, with the increase in the concentration of \(\Gamma\), a small peak appears in the backward scans (II) in cases (b) and (c) with increasing potential scan cycles especially in case (c). That peak may be attributed to the reduction of the remaining \(NiOOH\), as the surface poisoning by iodates and iodine hinders glucose from interacting with \(Ni^{3+}\).

Figure 9 shows the CV responses obtained on \(NiO_x/MnO_2/GC\) in 0.5 M NaOH at different \(\Gamma\) concentrations: (a) 0.0, (b) \(10^{-2}\), (c) \(10^{-3}\), (d) \(10^{-4}\), and (e) \(10^{-5}\) M. Scan rate, 100 mV/s.
current for the first run in cases (c) and (d) is higher than the blank itself (curve a). This behavior was attributed to adsorbed iodine oxidation as it is available in this potential range. In the following potential cycles, the peak current decreases with the potential scan number to values lower than that of the blank. The fact that the peak current decreases again after the first run, although two reactions occur (glucose oxidation and adsorbed iodine oxidation), could be explained by considering the cross effect of both glucose and I\textsuperscript{−} oxidation on each other. That is to say, the glucose oxidation reaction is retarded by the adsorbed products of iodine oxidation, and the iodide oxidation reaction may retard further oxidation of I\textsuperscript{−} to I\textsubscript{2}.

In Fig. 11 the three curves in the presence of I\textsuperscript{−} ions exhibit two decay stages, an initial rapid stage which depends on the concentration of I\textsuperscript{−} ions followed by a slow one. During the initial decay, the glucose oxidation peak current ($I_p$) rapidly falls to ca. 90.0, 84.4, and 65.4 % of its original value for cases (b), (c), and (d), respectively, suggesting a quick poisoning process especially with the high I\textsuperscript{−} concentration. This rapid stage is followed by a slow one in which the decrease in the peak current is negligible. For instance, for case (d), the peak current decreases from 343 to 332 \(\mu\text{A}\) (about 11 \(\mu\text{A}\)) that represents about 2 % of the whole current value.

It becomes clear that the poisoning effect of chloride and bromide ions differs greatly from the poisoning effect of the iodide ion, and this difference may be attributed to the difference in their adsorbability and reactivity. It is likely that the radii and the electronegativity of the halide ions have a profound influence on their adsorption process. Electronegativity increases from I\textsuperscript{−} to Cl\textsuperscript{−} (I\textsuperscript{−}=2.5, Br\textsuperscript{−}=2.8, Cl\textsuperscript{−}=3.0), while atomic radius decreases from I\textsuperscript{−} to Cl\textsuperscript{−} (I\textsuperscript{−}=135 pm, Br\textsuperscript{−}=114 pm, Cl\textsuperscript{−}=90 pm). So, the iodide ion is more predisposed to adsorption than bromide and chloride, and this is the reason behind their difference in poisoning effect [24].

In parallel to these and from a thermodynamic point of view, potentials for reversible Cl\textsubscript{2}/Cl\textsuperscript{−}, Br\textsubscript{2}/Br\textsuperscript{−}, and I\textsubscript{2}/I\textsuperscript{−} reactions are 1.36, 1.06, and 0.54 V vs. NHE, respectively. This also
explains why chloride and bromide ions do not affect the glucose oxidation reaction even at high concentrations. While for the iodide ion, the state is very different as iodide can be oxidized easily to iodine or iodates that are also possible in the present experimental conditions [25–28].

Conclusions

Halide ions have a significant poisoning effect on the electrocatalytic activity of the NiO$_x$/MnO$_y$/GC electrode toward glucose oxidation in alkaline medium. The poisoning degree of halide ions largely depends on their chemical nature and concentration in solution. For chloride and bromide ions, our fabricated electrode has high tolerance toward them even at high concentrations up to 0.3 M. While for the iodide ion, it was found that it has a significant effect on glucose oxidation in our operating conditions at very low concentrations in comparison with the concentrations of the other two halide ions. The present catalyst could serve as a catalyst for glucose oxidation and non-enzymatic sensor for glucose even in the presence of a large concentration of CT.

References

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