Synthesis and characterization of gold nanoparticles electrodeposited onto a boron-doped diamond electrode for sensors development

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Introduction

Next to gold, carbon surfaces have been extensively researched as support substrates in electrocatalytic systems\(^1\). Specifically, boron-doped diamond (BDD) films are quite attractive as electrode materials due to properties such as a wide electrochemical potential window.

Metal nanoparticles, particularly those of gold, have electronic, optical, and catalytic properties that are very different from those of bulk materials due to their scale dimensions. In recent years, these nanoparticles have been widely investigated and applied in molecular catalysis and as multifunctional reagents and biosensors\(^2\).

In this work the obtention and characterization of gold nanoparticles electrodeposited onto a BDD electrode for biosensor development is reported.

Results and Discussion

A stock solution of hydrogen tetrachloroaurate(III) trihydrate (Aldrich) was diluted in water. The preparation of the gold nanoparticles was carried out in a 0.1 mol L\(^{-1}\) HCl solution, using a fixed concentration of HAuCl\(_4\) (1.0 mmol L\(^{-1}\)). The electrochemical deposition of gold nanoparticles on the BDD surface was carried out amperometrically by applying a potential to the BDD electrode\(^3\). Prior to use, this electrode was cathodically pretreated in a 0.5 mol L\(^{-1}\) H\(_2\)SO\(_4\) solution by applying –0.5 A cm\(^{-2}\) during 250 s (preliminary studies with anodic pretreatments did not yield good electrodepositions).

The electrodeposition was optimized using a two-level factorial design, in which the variables were the reduction potential [–0.2 V or –0.4 V vs. Ag/AgCl (3 mol L\(^{-1}\) KCl)] and duration (20 s or 40 s) in the obtention process of the gold nanoparticles. Nanoparticles of diameters ranging from 30 nm to 100 nm were obtained. Applying the reduction potential of –0.4 V for 40 s, 30-nm gold nanoparticles were obtained for further experiments.

![Figure 1. SEM image of gold nanoparticles deposited at –0.4 V vs. Ag/AgCl (3 mol L\(^{-1}\) KCl) for 40 s using a 1.0 mmol L\(^{-1}\) HAuCl\(_4\) in 0.1 mol L\(^{-1}\) HCl solution.](image)

The electroactive areas of the BDD and BDD with electrodeposited gold (Au\(_{nano}\)/BDD) electrodes were estimated in 0.10 mol L\(^{-1}\) KCl in the presence of 1 mmol L\(^{-1}\) [Fe(CN)\(_6\)]\(^{3-}\) using the Randles-Sevck equation. Assuming a diffusion coefficient value of 6.2 x 10\(^{-6}\) cm\(^2\) s\(^{-1}\) for this redox species, the electroactive areas of the BDD and Au\(_{nano}\)/BDD electrodes were calculated to be 0.26 cm\(^2\) and 0.28 cm\(^2\), respectively. The transfer coefficient (\(\alpha\)) and heterogeneous electron transfer rate constant (\(k\)) on Au\(_{nano}\)/BDD were then determined from the slopes of the anodic and cathodic processes. Values of 0.48 and 0.032 s\(^{-1}\) were thus obtained for \(\alpha\) and \(k\), respectively\(^4\).

Conclusion

A factorial design method was successfully applied in the investigation of gold nanoparticle electrodeposition on cathodically pretreated BDD electrodes. A BDD electrode with 30-nm gold nanoparticles was obtained and characterized for further use for sensors development.

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