

Quantification of minoxidil in hair products using a glassy carbon electrode modified by gold electrodeposition

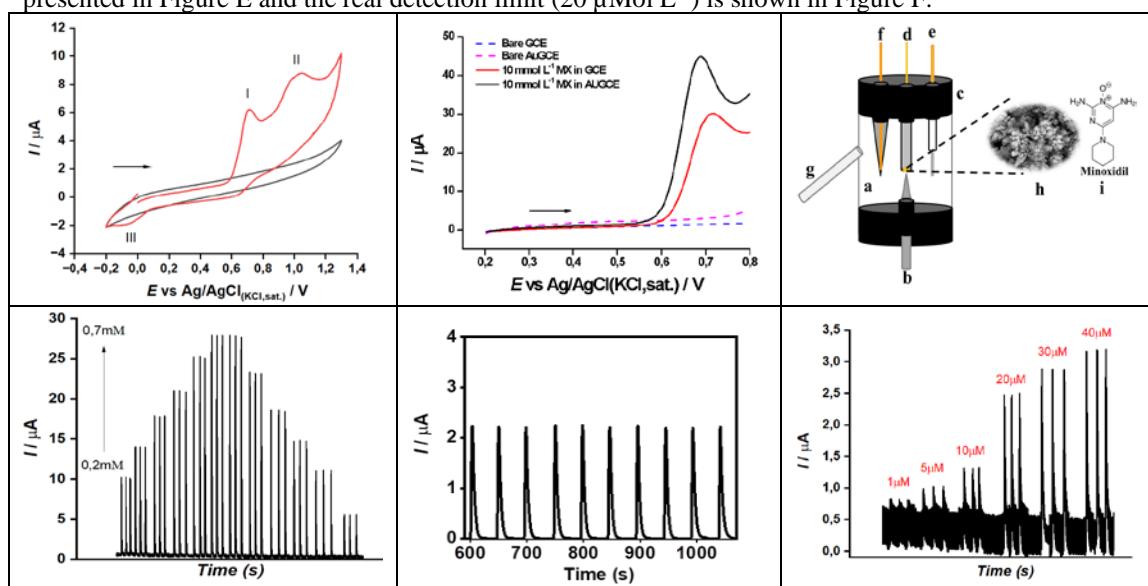
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Minoxidil (MX) is a vasodilator compound that was developed to treat high blood pressure in the 1970s. Researchers noted that some patients taking the drug reported increased hair growth as a side effect. Due to its success in treating androgenetic alopecia, MX has become increasingly popular, especially among men. As demand has increased, counterfeiting cases have also increased. The development of rapid methodologies for monitoring minoxidil is desirable. Electroanalytical techniques are a promising alternative, as they combine speed, sensitivity, selectivity, ease of handling, low cost, and potential for automation. Different electrodes were tested, and the glassy carbon electrode modified by gold electrodeposition showed differentiated performance, allowing the quantification of minoxidil (a relatively poorly soluble compound in water) even in low concentrations. Initial studies were performed by cyclic voltammetry (CV), using a commercial glassy carbon electrode, aiming to verify the redox processes involved. The experiments showed that at pH = 6, three redox processes occur (Figure A). Repetitive voltammograms showed a decrease in signal, an undesirable condition for sequential analyses of this analyte. Gold electrodeposition led to more reproducible results and to an increase in signal, as shown in Figure B. This electrode was then used for flow injection analysis (FIA), which allows analyses with high analytical frequency, precision and great sensitivity, allowing the detection of low concentrations of minoxidil. A cell was constructed where the solution is introduced through the base, with the solution inlet positioned exactly in front of the working electrode, located in the center of the upper cover of the cell. The other two electrodes of the amperometric system were placed next to this electrode, as shown in (Figure C). The calibration curves and the application in commercial samples purchased in pharmacies and barber shops were analyzed and the results were compared with those obtained by chromatography. Typical FIA signals are depicted in Figure D, the elevated reproducibility is presented in Figure E and the real detection limit (20 $\mu\text{Mol L}^{-1}$) is shown in Figure F.



The association of gold electrodes with flow injection analysis utilizing amperometry is a very favorable condition for the quantification of low concentrations of minoxidil. This kind of procedure will also be applied to other analytes.

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Influence of Electrical Contact Resistance Compensation on Paper Based Electrodes

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In a three-electrode system, uncompensated resistance (R_u) between the working and reference electrodes results in an electrostatic potential drop called iR potential drop, where i is a measured current¹. Ohmic drop is the decrease on the effective potential that a potentiostat applies and measures to the working electrode interface as a result of electric connections². Current compensation in electrochemistry is used to correct the voltage loss (i.e., iR drop) caused by the electrical contact between the working electrode and the reference electrode³. In this sense, our goal was to model the working electrode by adding a second working electrode as a resistor in order to obtain a four-electrode configuration. For this purpose, a new paper-based electrode was designed in Autodesk Fusion software and modified with Graphene-induced by CO₂ laser. The four electrodes' response was evaluated by cyclic voltammetry for three different analytes: dopamine, hexaaminruthenium and potassium ferricyanide. For all the three analytes, the compensating Ohmic current with the four-electrode configuration presented better analytical responses than the respective three-electrode configuration system (Figure 1). Based on our knowledge, this is the first work developed for this purpose on a paper-based platform. These results are correlated with those found in our previous publication using instead 3D-printed electrodes³.

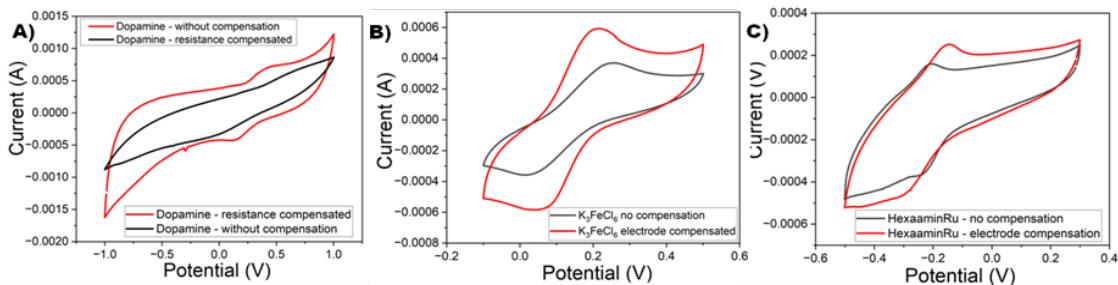


Figure 1. Cyclic Voltammetry performed for the traditional three electrodes configuration in black, and for the proposed four electrodes configuration in red. **(A)** Additions of dopamine 3 mM. **(B)** Additions of potassium ferricyanide. **(C)** Additions of hexaaminruthenium 5 mM.

References:

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