

Preparation of carbon ceramic electrodes using different carbon materials

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Abstract – The carbon material is an important factor that can influence the electrochemical response of carbon ceramic electrodes (CCE). In this context, CCE were prepared with graphite (GRCCE) and glassy carbon (GLCCE), using as a silane precursor, the methyltrimethoxysilane. The results in $K_4[Fe(CN)_6]$ indicated that GLCCE ($\Delta E_p=327$ mV) is more resistive than GRCCE ($\Delta E_p=266$ mV). Studies in presence of dopamine (DA) and ascorbic acid (AA), showed higher sensitivity for DA with GRCCE, when compared to GLCCE. In addition, the GRCCE presented lower potential peak separation values and also enables DA determinations in presence of AA (since it is not sensitive for AA).

Carbon ceramic electrodes (CCE) are obtained by the sol-gel method combining the properties of conductive carbon material with mechanical properties of the silica matrix resulting in sensors with renewable surface and high stability [1]. The type of carbon material is an important factor that can influence the electrochemical response of carbon ceramic electrodes, mainly its conductivity. Based on this context, carbon ceramic electrodes were prepared with different carbon materials, in particular graphite (GRCCE) and glassy carbon (GLCCE), using as a silane precursor, the methyltrimethoxysilane (MTMOS).

The CCEs were obtained by mixing 1.8 g of carbon material, 1.2 mL of MTMOS, 15 μ L of HCl (12mol^{-1}) and 1.5 mL of methanol. The electrochemical measurements were performed by cyclic voltammetry (CV) in presence of potassium ferrocyanide $K_4[Fe(CN)_6]$, using a potentiostat OMNI-101 and an electrochemical cell containing three electrodes: Pt as counter electrode, reference electrode Ag / AgCl and working electrode (GRCCE / GLCCE) in $NaCl$ 0.5 molL^{-1} . The morphological characterization was realized by SEM technique (Shimadzu Superscan model SSX-550). The CCE were applied as sensors for dopamine (DA) and ascorbic acid (AA) in the concentration range of 0.1 to 1.0 mmolL^{-1} .

From the morphological analysis of GLCCE, it was found that the glassy carbon spheres are uniformly distributed among the interstices of the silica matrix. In contrast, the image obtained with the GRCCE, resulted in a disorganized lamellar graphite particles on silica matrix. The electrochemical characterization in presence of $[K_4Fe(CN)_6] = 1 \times 10^{-2}\text{ molL}^{-1}$ using GLCCE showed a pair of peaks in $E_{pa}=617$ mV and $E_{pc}=290$ mV and $\Delta E_p=327$ mV. These potential values when compared to the electrode prepared with graphite, GRCCE ($E_{pa}= 378$ mV, $E_{pc}=112$ mV and $\Delta E_p= 266$ mV) are shifted to more positive potentials, indicating that GLCCE is probably more resistive than GRCCE. However, the GLCCE showed higher current values ($I_{pa}=39.8\ \mu\text{A}$) and better definition of the voltammetric peaks in relation to the GRCCE ($I_{pa}=10.1\ \mu\text{A}$). Studies at different scan rates showed a linear relationship between the peak current, I_{pa} and the square root of scan rate, $v^{1/2}$, for both electrodes, indicating a diffusional behavior in presence of $K_4[Fe(CN)_6]$. The CCE were applied for determination of DA and AA. The voltammograms obtained after addition of DA in the electrochemical cell, presented a pair of redox peaks for both GLCCE and GRCCE, while for AA, it was obtained only one peak when using GLCCE which was not observed for GRCCE (in the same concentration range), as shown in Table 1. Studies evaluated at different concentrations in presence of these analytes, resulting in higher sensitivity for DA with GRCCE in relation to GLCCE. In addition, this electrode presented lower potential peak separation values and also enables DA determinations in presence of AA (since it is not sensitive for this analyte). In the case of GLCCE, it can only be applied for separated determination of these analytes (as presented in pharmaceuticals formulations).

Table 1: Voltammetric parameters obtained for the analytes AA and DA.

Parameters		E_{pa} (mV)	E_{pc} (mV)	ΔE_p (mV)	R	Linear range mmolL^{-1}	Sensitivity $\mu\text{A/molL}^{-1}$	Detection Limit molL^{-1}
GLCCE	DA	450	242	208	0.998	0.1 – 1.0	0.020	3.80×10^{-5}
	AA	442	-	-	0.996	0.1 – 1.0	5.95×10^{-4}	1.14×10^{-4}
GRCCE	DA	335	202	133	0.997	0.1 – 1.0	1.58×10^{-4}	3.94×10^{-4}

[1] TSIONSKY, M.; GUN, G.; GIEZER, V.; LEV, O. Analytical Chemistry, v. 66, n. 10., p. 1747-1753, 199.