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Voltammetric behavior of a Chemically Modified Carbon Paste Electrode with Cadmium Nitroprusside Prepared in Different Water to Formamide Ratios

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This paper describes a voltammetric study of cadmium nitroprusside synthesized from different compositions of water/formamide in your reaction of Cd^{2+} with $[Fe^{III}(CN)_5NO]^{2-}$ (CdNP). After infrared and transmission electron microscopy characterization, the CdNP was incorporated into a carbon paste electrode and a systematic voltammetric study was performed for four systems (CdNP-1, CdNP-2, CdNP-3 and CdNP-4). The spectroscopic studies reveals that all preparations was conducted with success. The cyclic voltammogram of all CdNP particles obtained from a carbon paste electrode modified (10% m/m) exhibited a redox couple with formal potential ($E^{0'}$) = 0.63 ± 0.02 V (10 % (m/m); v=20 mV s⁻¹; KNO₃ 1.0 M)) attributed to the redox process [Fe^{II}(CN)₅NO]/[Fe^{III}(CN)₅NO] in the presence of Cd²⁺.

Keywords: Cadmium nitroprusside, voltammetry, formamide, eletro-oxidation, isoniazid

1. INTRODUCTION

In the electrochemistry area it is shown that chemically modified carbon paste electrodes have some advantages over conventional electrodes, contributing to the development of numerous electroanalytical applications, including electrocatalysis and electrochemical sensors [1-4]. Carbon paste electrode (CPE) is a heterogeneous mixture between carbon powder and a proper water-immiscible and non-conducting binder. When this powder is properly mixed with electroactive substance [5-7] or inert platforms [8-14] of high surface area containing electroactive substances it becomes capability in catalyzing the reduction–oxidation reactions through by enhancing the electron transfer rate. Besides, their versatility allows the addition of other component during the paste preparation, such as metal nanoparticles that improve the conductivity and electroactive area [15-19].

The properties of the modified electrodes depend on the formation and characteristics of the film formed on the electrode surface. One of the main advantages of chemically modified electrodes is their ability to catalyze the oxidation of some species that exhibit high potential in unmodified electrodes, thus increasing their sensitivity and selectivity [4, 20-26].

One important group of inorganic compound used for modification of carbon is transition metal cyanoferrates in view of the well known catalytic activity [10-17].

Metal nitroprusside formed by d-block metals such as Zn (II) Cu (II), Ni (II), Co (II) is a class of bimetallic compounds that has been employed for the preparation of thin films on the surface of solid electrodes in recent years. The preparation of these films is not an easy task and they are usually difficult to reproduce. The lack of reproducibility explains the small number of works in the literature and the different points of view between the authors [18].

Many problems arise in its use, such as low electrochemical stability and mainly lack of reproducibility in its formation, the latter subject being the source of great controversy [22].

Aiming to establish an alternative methodology, this work suggests the exploration of these insoluble complexes using carbon paste electrodes, because it has a greater reproducibility, ease and rapidity of synthesis without altering the electrocatalytic properties of this interesting class of compounds. Insoluble metal nitroprussides are normally prepared by mixing solutions of soluble nitroprussides and the pertinent cations, but

As a continuity of this study we describe in this work a preliminary study about the electrocatalytic abilities of the carbon paste electrode modified with cadmium nitroprusside as a mediator in the oxidation of isoniazid. To our knowledge, there is only one report in the literature [24] on the preparation and electrochemical behavior of a glassy carbon (GC) electrode modified with a thin film of cadmium nitroprusside. In this above mentioned work (Ref.24) the modified electrode were studied in the presence of different supporting electrolytes and at different pHs, but was not reported at moment any mediator activity of analytical interest.

2. EXPERIMENTAL

2.1. Reagents and Solutions

The reagents and solvents used in the laboratory preparations and procedures were of high analytical purity and were used as received. Sodium Nitroprussiate and Cadmium Chloride were purchased from Sigma-Aldrich; Formamide, Carbon Powder and Nujol Mineral Oil were purchased from Vetec, and deionized water with resistivity no less than 18.2 M Ω cm was used to prepare all solutions. The solutions of isoniazid were prepared immediately before use.

2.2. Preparation of Nanoparticles of Cadmium Nitroprusside

The cadmium nitroprusside was prepared according to the procedure described in the literature with some adaptions [27,5] described as follows: two solutions, A and B, were prepared. Solution A consisted of $6,72\times10^{-3}$ mol of Na[Fe(CN)₅NO] dissolved in a solution of 30 mL of water and formamide mixture. Solution B was prepared by dissolving $8,4\times10^{-3}$ mol of CdCl₂.H₂O in 20 mL of water and formamide. Next, the solution A was poured into solution B and stirred for 3 hours at 50 °C.

After this procedure, the solution was sonicated for 1 h and then the solid phase was isolated by centrifugation (2000 rpm). Then the solid phase were carefully gravity filtered and washed thoroughly with deionized water. The product was then dried at 50°C in a vacuum oven and then stored in a vacuum desiccator and protected from light.

With the intention of investigating the effect of formamide and also its composition with water in the voltammetric properties, four samples, CdNP-1, CdNP-2, CdNP-3 and CdNP-4, were prepared in four reaction media with different water and formamide ratios (v/v), 10:0; 8:2; 4:6 and 0:10, respectively. Table 1 summarizes the procedure performed for the preparation of the four materials. The yield of the four prepared materials was 95.96%; 96.42%; 95.57% and 54.16% for, CdNP-1, CdNP-2, CdNP-3 and CdNP-4 respectively.

	SOLUTI	ION A (30mL)		SOLUTION B (20mL)				
SYSTEM	Water	Formamide	Na[Fe(CN)5NO]	Water	Formamide	CdCl ₂ .H ₂ O		
	(mL)	(mL)	(mol)	(mL)	(mL)	(mol)		
CdNP-1	30	0	6.72 x 10 ⁻³	20	0	8.4 x 10 ⁻³		
CdNP-2	24	6	6.72 x 10 ⁻³	16	4	8.4 x 10 ⁻³		
CdNP-3	12	18	6.72 x 10 ⁻³	8	12	8.4 x 10 ⁻³		
CdNP-4	0	30	6.72 x 10 ⁻³	0	20	8.4 x 10 ⁻³		

Table 1. Reagents used for the preparation of the systems being studied.

For the electrochemical measurements a Potentiostat PalmSens3 from OS Trace was used. A cell with three electrodes was used to compose the electrochemical system, with a working electrode modified with carbon paste, a reference electrode (Ag / AgCl / KCl (sat)) and a platinum auxiliary electrode. Preliminarily, three proportions (m / m) of carbon paste and modifier (CdNP), such as 10, 20 and 40% were tested for the four systems in order to choose the proportion that presents better voltammetric performance. The modified paste electrode was prepared by mixing carbon powder with material (CdNP) in the proportions indicated, totaling a mass of 0.1 g and 20 μ l of mineral oil (Nujol) as a binder. The electrode body was produced from a glass tube of 3 mm i.d. and 14 cm height, containing carbon paste. A copper wire was inserted through the opposite end to establish electrical contac (do Carmo et al., 2015). It was found that the best paste / modifier composition was 10% and this was employed in all electrochemical studies.

2.3. Fourier transform infrared measurements

All infrared spectra of the materials were performed on a Nicolet 5DXB FTIR 300 spectrometer (Nicolet Instruments, Madison, WI). Approximately 150 mg of KBr was grund in a mortar/pestle, and sufficient solid sample was ground with KBr to produce a 1% (w/w).

2.4. Transmission Electronic Microscopy (TEM)

The transmission electronic microscopy was performed using the microscope Philips brand - CM200, equipped with pole piece that allows to obtain high resolution images. It is operated at an accelerating voltage of 200 kV.

2.5 Electrochemical measurements

Electrochemical experiments were carried out using a potentiostat model MQPG1. A conventional cell with three electrodes was used: working electrode (carbon paste electrode modified with CdNP), the a reference electrode (Ag/AgCl_(s) (KCl, 3.0 mol L⁻¹) and an auxiliary electrode being (platinum wire (3.0 cm x 0.1 cm). All experiments were performed at ambient temperature. At most experiments, a 1.0 mol L⁻¹ of KNO₃ (pH 7.0) support electrolyte was employed at a scanning rate of 20 mV s⁻¹.

3. RESULTS AND DISCUSSION

3.1. Infrared Region Spectroscopy (FTIR)



Figure 1. Infrared spectra of cadmium nitroprusside (III) nanoparticles and sodium nitroprusside compound: (A) sodium nitroprusside; (B) CdNP-1; (C) CdNP-2; (D) CdNP-3 and (E) CdNP-4.

Vibrational spectra (FTIR) provide valuable information on the water of hydration and the coordination metal to ligand. Infrared spectra of the cadmium nitroprusside particles prepared in the four proportions, and the precursor compound are shown in Figure 1.

As illustrated for Figure 1 (A, B, C, D, E), the main characteristic absorptions of the precursor compound (sodium nitroprusside (A)) were observed for all systems (Figure B, C, D, E). The main ones being related to the symmetrical stretching NO (υ N-O) in 1945 cm⁻¹ and the symmetrical C=N (υ C=N) stretch at 2192 cm⁻¹.

The (ν C=N) stretch was shifted to a region of higher frequency about 5 cm⁻¹ for all systems (CdNP) relative to the precursor. The displacement of (ν C=N) being observed for the four systems in relation to sodium nitroprusside suggest the coordination of the Cd (cadmium) to the N (nitrogen) of the five cyanide groups of the Fe metallic center (Fe^{III}-C=N-Cd^{II}) [28]. A further observation is that the absorptions of NO ν (N-O) stretches of the CdNP particles have been narrower, relative to sodium nitroprusside (III) [29]. In addition It was observed that as the proportion of formamide increases, the range at 1620 cm⁻¹ corresponds to the vibration of flexion, δ (HOH) attributed to weakly bound water molecules, decreases and another band appears in, this phenomenon was attributed to the possible formation of hydrogen bonding of water with formamide. The water molecules are mainly in this structure as uncoordinated water filling the cavities due to the vacancies of the nitroprusside anion.

3.2. Trasmission Electron Microscopy (TEM)



Figure 2. Transmission electron microscopy images of systems: (A) CdNP-1 (B) CdNP-2 (C) CdNP-3 and (D) CdNP-4

Transmission electron microscopy was concomitantly used to verify the morphology of the prepared particles. As shown in Figure 1, the presence of cubic form particles of dimensions ranging from 200 to 1300 nm is observed. TEM showed cubic particles for the CdNP-1 system, as well as evidence of smaller particles. For the other systems, well-organized cubic particles were observed too, and their size ranged from 681 to 1330 nm following the following order: CdNP-2> CdNP-3> CdNP-4

3.3. Electrochemical characterization of CdNP nanoparticles

A rigorous study was performed on the voltammetric behavior of all prepared cadmium nitroprusside (III) particles using a carbon paste electrode as the working electrode. The carbon paste prepared in the proportion of 10% (m/m), as previously described, was chosen to carry out the subsequent studies because it presented the best voltammetric performance for future electroanalytical applications. The cyclic voltammogram of the four CdNP systems, as shown in Figure 3, exhibit a well defined redox process with formal potential (E^{θ_1}) of 0.63 ± 0.02 V, attributed to the Fe^(II) (CN)₅NO/Fe^(III) (CN)₅NO redox couple of the complex formed in the presence of Cd ^{(II).}

3.3.1 Study on the effect of different supporting electrolytes



Figure 3. Cyclic voltammograms of carbon paste modified electrode with CdNP-1 (A); CdNP-2 (B); CdNP-3 (C); CdNP-4 (D) in different electrolytes based on nitrates (NO₃⁻) (m/m), v = 20mV s⁻¹, 1.0 mol L⁻¹)

Figures 3 illustrate the study on the influence of cations and anions from the supporting electrolytes on the voltammetric behavior of the four CdNP systems. The process of oxidation and reduction of the complexes formed from cadmium nitroprusside occurs initially, by the equilibrium of the cation present in the support electrolyte with the surface of the electrode containing the material [30]. In this experiment, different support electrolytes based on alkali metal salts such as KCl, NaCl, KNO₃, NaNO₃ and also the ammonium salts NH₄Cl and NH₄NO₃ (1.0 mol L⁻¹ and v = 20 mV s⁻¹) were tested, through these studies it was observed that the nature of cations and anions affected not only the formal potential (E^{θ}) but also current intensities. The cyclic voltammograms of the CdNP systems in the formal potential (E^{θ}) was observed for more positive potentials, following the order NH₄⁺> K⁺> Na⁺, not only for nitrates but also for chlorides. The NH₄⁺ and K⁺ cations can penetrate more easily into the crystalline lattice of the mixed valence complex because they have lower diameter of hydrated cation radii.

Tables 2 and 3 list the values of the formal potentials (E^{θ}) and the main electrochemical parameters obtained from the cyclic voltammograms of the modified carbon paste for the four systems mentioned above, in different electrolytes.

Table 2. Electrochemical parameters of CdNP-1 modified carbon paste electrode in different electrolytes (10% (m/m); $v = 20 \text{ mV s}^{-1}$; 1.0 mol L⁻¹).

Electrolytes	Ipa (µA)	Ірс (µА)	Ipa/Ipc	Epa (V)	Epc (V)	Ε ^θ ' (V)	ΔEp (V)	Diameter of hydrated cation (nm)[31]
KNO ₃	119.72	-107.57	1.11	0.69	0.57	0.63	0.12	0.24
NaNO ₃	133.70	-112.72	1.18	0.56	0.43	0.49	0.13	0.36
NH4NO ₃	106.50	-99.04	1.07	0.71	0.61	0.66	0.10	0.24
KCl	113.43	-110.76	1.02	0.69	0.56	0.62	0.13	0.24
NaCl	111.83	-109.36	1.02	0.56	0.41	0.48	0.15	0.36
NH ₄ Cl	86.01	-86.84	0.99	0.73	0.60	0.66	0.13	0.24

Table 3. Electrochemical parameters of CdNP-2 modified carbon paste electrode in different electrolytes (10% (m/m); $v = 20 \text{ mV s}^{-1}$; 1.0 mol L⁻¹).

Electrolytes	Ipa (µA)	Ipc (µA)	Ipa/Ipc	Epa (V)	Epc (V)	Ε ^{θ'} (V)	ΔEp (V)	Diameter of hydrated cation (nm)[31]
KNO ₃	142.05	-176.87	0.80	0.67	0.56	0.61	0.11	0.24
NaNO ₃	165.81	-201.86	0.82	0.57	0.45	0.51	0.12	0.36
NH4NO ₃	135.18	-145.00	0.93	0.69	0.59	0.64	0.10	0.24
KCl	111.83	-127.80	0.87	0.69	0.55	0.62	0.14	0.24
NaCl	147.46	-179.90	0,81	0.57	0.41	0.49	0.16	0.36
NH ₄ Cl	84.71	-93.72	0,90	0.70	0.59	0.64	0.11	0.24

According to the influence study of the electrolytes, KNO₃ was chosen for the later studies because it presented good chemical stability and good voltammetric performance for the four systems containing the particles, besides presenting electrochemical parameters close to the reversibility.

3.3.2 Influence of supporting electrolyte concentrations



Figure 4. Cyclic voltammograms of carbon paste modified electrode with CdNP-1 (A); CdNP-2 (B); CdNP-3 (C); CdNP-4 (D) in different concentrations of KNO₃ ($v = 20 \text{ mVs}^{-1}$).

Figure 4 shows the cyclic voltammograms of CdNP-modified carbon paste at different concentrations of KNO₃ (0.1 to 2.0 mol L⁻¹), where it was observed that the performance becomes different and there is an increase in the intensity current as the concentration of the supporting electrolyte increases. This behavior indicates the participation of the K⁺ ion in the redox process in addition; it is notable that the redox process occurs faster and better defined in the higher concentrations. Because cadmium (II) is nonelectroactive in the paste, the redox reaction of CdNPs involves only one-electron transfer between nitroprusside (III/II) ions in presence of cadmium, as following (Eq 1):

$$Cd^{(II)}[Fe^{(III)}(CN)_5NO] + K^+ + e^- \longrightarrow KCd^{(II)}[Fe^{(II)}(CN)_5NO]$$
 Eq. 1

A linear relationship between the mean potential (E^{θ}) and the log (log) K⁺ concentration ffour systems was found, indicating that the redox process is highly dependent on the K⁺ concentration, as shown in Figure 4. A slope of the line was around 30 mV per decade of concentration of K⁺ ions, indicating a "quasi" Nernstian process [26], for all systems.

3.3.3 Study on the effect of different hydrogen concentrations

The pH effect of supporting electrolyte on the electrochemical behavior of the CdNP was studied. Cadmium hexacyanoferrates and other metal pentacyanoferrate (MPCFs) or metal hexacyanoferrate (MHCFs) films can maintain stable electrochemical responses only in neutral or weakly acidic solution [31,32], while in alkaline solution the hydrolysis of metal cations in the film can cause deterioration of the framework of MHCFs [32,33].



Figure 5. Cyclic voltammogram of carbon paste modified with: CdNP-1(A), CdNP-2 (B), CdNP-3 (C) and CdNP-4 (4) at different pH values pH (3-9); (10% (m/m); KNO₃ 1,0 mol L⁻¹, v = 20 mV s⁻¹).

However, in performed study for the four CdNP systems with different hydrogen concentrations (pH 3.0 to 8.0) performed for the four CdNP systems, as shown in Figure 5, where from the

voltammograms it was verified that by varying the hydrogen ion concentration there are no significant changes not only in the intensity of the anodic and cathodic peaks as well as other electrochemical parameters. In this case it can be stated that the hydrogen ion concentration does not affect the electrochemical process and the framework of CdNP is not deteriored and this demonstrates that the material is chemically and electrochemically stable The pH value chosen for the subsequent studies was 7.0 due to the possibility of carrying out further studies for the electrocatalytic determination of drugs at near-biological pH.

3.3.4 Influences of scan rate

The Figure 6 shows the cyclic voltammograms obtained from the study of the voltammetric behavior of CdNPs modified carbon paste for the four systems at different scan rates. It was observed that with the increase of the scan rate, the peak currents of the voltammogram are linearly proportional to the square root of the scan rate between 10 - 100 mV s⁻¹ are and the E^{θ_1} practically remained around 0.64 V, which characterizes a diffusional process [34].



Figure 6. Cyclic voltammogram of the carbon paste modified with CdNP-1 (A), CdNP-2 (B), CdNP-3 (C) and CdNP-4 (D) at different scanning rates (10-100 mV s⁻¹).

From the voltammograms, a linear dependence between the anodic and cathodic peaks current and the square root of the scanning rate is observed for the four systems as shown in Figure 7, which characterizes a diffusional process [34].



Figure 7. Dependence of peak current intensity (anodic and cathodic) as a function of square root of scan rate: CdNP-1(A), CdNP-2 (B), CdNP-3 (C) e CdNP-4 (D).

3.2.6. Preliminary study of electrochemical of CdNP-3 and CdNP-4 systems in the electro-oxidation of isoniazid by cyclic voltammetry

The Figure 8 shows the voltammetric behavior of the two systems that presented electrocatalytic activity for the detection of isoniazid, namely: CdNP-3 (I) and CdNP-4 (II), where (a) is the unmodified carbon paste electrode, (b) with modifier, (c) carbon paste in the presence of isoniazid and (d) with modifier in the presence of isoniazid.

These systems (a) and (c) did not present any redox process in the window of potential studied (-0.2 to 1.0 V), but when adding millimolar amount of isoniazid the intensity of the anodic current increases to only the electrode carbon paste with modifier (d). This increase in current intensity (d) is proportional to the increase in isoniazid concentration. The increase in the intensity of the anodic current (Figure 8) occurs due to the electro-oxidation of isoniazid by electron mediator particles. Fe (III)

produced during the anode scan "chemically" oxidizes isoniazid, while Fe (III) is reduced to Fe (II), which should be oxidized to Fe (III) again on the surface of the electrode [35].



Figure 8. Voltammetric behavior of CdNP-3 (I): (a) carbon paste electrode without modification, (b) with modifier; $(9.0 \times 10^{-3} \text{ mol } \text{L}^{-1})$ and (d) with modifier in the presence of isoniazid $(9.0 \times 10^{-3} \text{ mol } \text{L}^{-1})$ and CdNP-4 (II): (a) unmodified carbon paste electrode, (b) with modifier; $(2.0 \times 10^{-2} \text{ mol } \text{L}^{-1})$ and (d) with isoniazid modifier $(2.0 \times 10^{-2} \text{ mol } \text{L}^{-1})$ (10% (m/m), KNO₃ 1.0 mol L ⁻¹, v = 20 mV s ⁻¹).

The electrochemical oxidation of Isoniazid for the two systems studied can be described and presented by equations 2 and 3 below:

 $4\text{KCd}[\text{Fe}^{(\text{II})}(\text{CN})_5\text{NO}] \longleftarrow 4\text{Cd}[\text{Fe}^{(\text{III})}(\text{CN})_5\text{NO}] + 4\text{K}^+ + 4\text{ e}^- \qquad \text{Eq. (2)}$

 $4Cd[Fe^{(III)}(CN)_5NO] + 4K^+ + H_2O + IZDR \implies AISO + N_2 + 4 H^+ + KCd[Fe^{(II)}(CN)_5NO]$ Eq.(3)

Where IZDR = Isoniazid and AISO = Isoniazid oxidation product (isonicotinic acid).

4. CONCLUSION

The Vibrational spectrum studies allowed us to conclude that the synthesis of cadmium nitroprusside particles was successfully performed.

The carbon paste electrodes modified with the aforementioned particles showed good electrochemical stability and excellent reproducibility. The voltammetric behavior of the CdNP particles in the proportions of water and formamide studied showed a well defined redox pair with $E^{\theta} = 0.63 \pm 0.02 \text{ V} (10\% \text{ (m/m)}, \text{KNO}_3 1.0 \text{ mol } \text{L}^{-1}; \text{ v} = 20 \text{ mV s}^{-1})$, assigned to Fe^{II}(CN)₅NO / Fe^{III} (CN)₅NO in the presence of Cd²⁺.

Of the four systems studied, only two were sensitive to concentrations of isoniazid, namely CdNP-3 and CdNP-4. The CdNP particles can be tested for other types of analytical applications that are surface area dependent and morphological, but the carbon paste electrodes modified with CdNP particles in certain proportions of water and formamide are potential candidates in the manufacture of electrochemical sensors for determination of isoniazid in real samples.

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