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Assessment of carbon nanotubes-epoxy composite electrode for in-field detection applications

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Abstract

Carbon nanotubes-epoxy (CNT) composite electrode consisted of 20%, wt. CNT was electrochemically characterized envisaging its application for the *in-field* detection. CNT electrode was prepared by two-roll mill procedure and detailed description of preparation and morphostructural characterization was reported by our group [1]. The main peculiarity of an electrode for *in-field* detection application is to be able to respond without deliberately adding the supporting electrolyte that is required for conventional macroelectrode and as consequence, limits this kind of application.

It is well-known that carbon based composite electrode can be regarded either ordered (array) or randomized micro/nanoelectrode ensembles [2]. Two characteristics are followed to characterize the ordered or randomized micro/nanoelectrode ensembles, *i.e.*, the dependence of diffusion flux towards on the experimental timescale and the direct detection of the target analyte without supporting electrolyte adding.

In the preparation of carbon-based composites with microelectrode array behaviour, certain important requirements should be met. The main requirement is that the gaps between individual conductive carbon microzones should be much larger than its radius, when spherical diffusion dominates mass transport. The closely spaced carbon microzones array will behave similar to a macroelectrode (linear diffusion controlled mass transport) because of the diffusion layer overlap [3]. Another requirement is subjected to the insulating matrix to prevent current leakage, which resulted in the distortion of the cyclic voltammetry [4]. Microelectrode arrays exhibit the advantages of single microelectrodes, *e.g.*, reduced ohmic drop and charging current, the lower detection limit and better sensitivity.

Based on the above-presented aspects, the electrochemical behavior of the CNT composite electrode was characterized by ferri/ferrocyanide classical method, and it was found that linear diffusion controlled the mass transport that is characteristics to the macroelectrode behaviour, with a substantial hysteresis. However, the ability to deliver the current responses in the absence of any supporting electrolyte, which is an attractive feature for practical *in-field* detection application, was tested using salicylic acid (SA) as target analyte. Two reasons subjected to SA selection as target analyte were taken into account, the first referred to direct analysis of pharmaceutical compounds in surface water as emerging pollutants envisaging indirect aspirin detection and the second issue referred to the certification of the micro/nanoelectrode array behavior of CNT composite electrode by SA detection in surface/tap water, without supporting electrolyte.

Cyclic voltammetry was used for the electrochemical characterization of CNT composite electrode and differential-pulsed voltammetry (DPV) and square-wave voltammetry (SWV) for the detection experiments. The comparative responses obtained at CNT composite electrode using DPV technique in the absence/presence of 0.1 M Na₂SO₄ supporting electrolyte are presented in Figure 1 a and b, and it can be noticed that similar results were achieved. All electroanalytical parameters determined for SA detection under three variants of aqueous media (0.1 M Na₂SO₄ supporting electrolyte, surface water and tap water) are similar for each electrochemical technique. Based on these above-presented results, it can be concluded that MWCNT-EP composite electrode manifested the microelectrode array behavior in relation with the direct detection of SA in tap water, without supporting electrolyte. Moreover, the results of the SA detection direct in surface water, also without the supporting electrolyte adding show for this electrode a real potential for *in-field* detection application.

Table 1. The comparative electroanalytical performance for SA detection using CNT electrode

Aqueous media	Peak potential	Technique Used/operation conditions	Concentration range (mM)	Sensitivity (mA /mM ⁻¹)	Correlation coefficient (R ²)	LOD (mM)	LQ (mM)	RSD [**] (%)
0.1 M Na ₂ SO ₄ supporting electrolyte	+0.52V	DPV Sp=0.01V Ma=0.1V	0.2-1.2	0.017	0.994	0.002	0.04	1.315
	+0.46V	SWV Sp=0.005V Ma=0.05V F=50 Hz	0.2-1.2	0.060	0.986	0.005	0.016	0.250
Bega River	+0.52V	DPV Sp=0.01V Ma=0.1V	0.2-1.2	0.026	0.991	0.005	0.019	0.980
	+0.56V	SWV Sp=0.005V Ma=0.05 V F=50 Hz	0.2-1.2	0.073	0.999	0.004	0.013	0.186
Tap Water	+0.65V	DPV Sp=0.01V Ma=0.1V	0.06-0.2	0.018	0.993	0.008	0.028	2.702
	+0.52V	DPV Sp=0.01V Ma=0.1V	0.2-1.2	0.029	0.999	0.010	0.034	3.030

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Figures

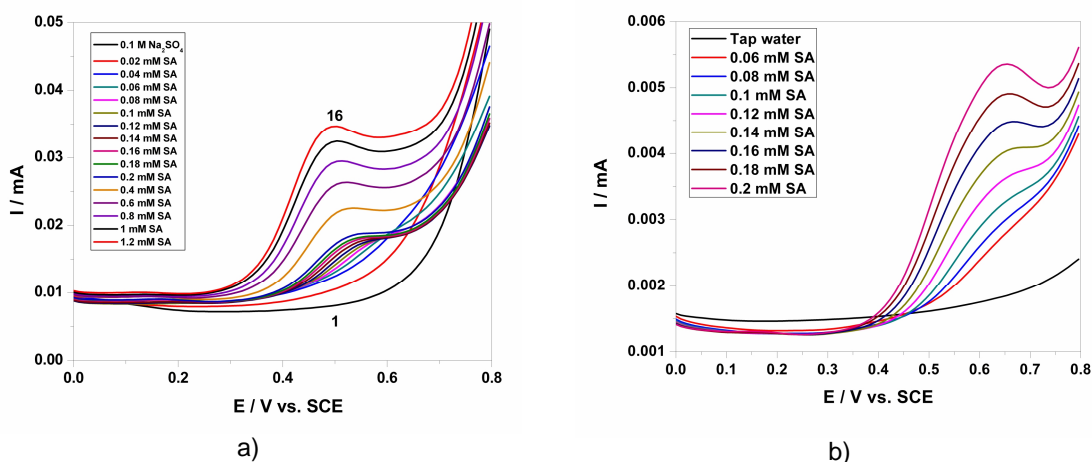


Fig. 1. DPV recorded at CNT composite electrode in the presence of various SA concentrations in: a) 0.1 M Na₂SO₄ supporting electrolyte and b) tap water