

Construction and characterization of carbon paste ultra-microelectrodes

Joshua Oni, Philippe Westbroek, Tebello Nyokong *

Department of Chemistry, Rhodes University, P.O. Box 94, 6140 Grahamstown, South Africa

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Abstract

The construction and characterization of carbon paste ultra-microelectrodes is reported. Besides a disc-shaped electrode, a carbon paste band electrode is studied. It was found that for both electrode configurations steady-state currents were obtained but that with the type of electrode material used for the band electrode, the upper limit of ultra-microelectrode dimensions, which could be used was 100–150 μm . Smaller dimensions for the band electrodes resulted in a high resistance due to the limited conductivity of the carbon paste, when using the optimal paste composition. With cyclic voltammetry, the oxidation of $[\text{Fe}(\text{CN})_6]^{4-}$ was studied at individual constructed electrodes and at ensembles of individual electrodes. Detection limits of less than 10^{-6} mol l^{-1} were obtained. Using the data of the same curves it was possible to test if the electrodes have errors and about 20% of the constructed electrodes did not pass this test. Finally it was found that these electrodes show good stability. © 2001 Published by Elsevier Science B.V.

Keywords: Carbon paste; Ultra-microelectrode; Electrode construction

1. Introduction

Since the late seventies the use of ultra-microelectrodes has increased for several reasons, and the construction of these electrodes became an important area of research [1–4]. Ultra-microelectrodes show interesting advantages over microelectrodes such as lower detection limits [5–8] because of smaller charging and higher faradaic current densities, ability to measure at high scan rates [9–14] with minimal distortion to the voltammogram [15], obtaining time independent (steady-state) currents [16–19] without convection of the electrode and/or in solution and the possibility of measuring in relative high resistive media [5,20–26] because of reduced ohmic drop effects. Ultra-microelectrodes were not popular before the late seventies because of the lack of equipment for the construction of microsized electrodes and measuring accurately the small currents generated from the electrolysis of solution components [7,11,26–30].

A wide variety of construction methods, configurations and materials were used for ultra-microelectrodes.

The difficulty with handling and immobilizing carbon paste into microsized electrode holders is a big challenge to the development of carbon paste ultra-microelectrodes and may be responsible for the limited records in the literature for the construction of carbon paste ultra-microelectrodes [31] and its arrays [32]. On the other hand, there are lots of reports in the literature whereby carbon paste electrodes of conventional dimensions have been used for electroanalysis and particularly impressive applications have been reported for electroanalysis [33,34]. The aim of this project is to develop carbon paste ultra-microelectrodes for use in electroanalysis in order to extend the benefits of ultra-microelectrodes, mentioned above, to carbon paste based.

This paper describes a relatively simple and cheap method for the construction of carbon paste ultra-microdiscs (CPUMDEs) and band electrodes (CPUMBEs).

2. Experimental

The CPUMBEs and CPUMDEs are presented schematically in Figs. 1(a) and (b) and were prepared in the

* Corresponding author. Tel.: +27-46-603-8260; fax: +27-46-622-5109.

E-mail address: t.nyokong@ru.ac.za (T. Nyokong).