



Short communication

## A new strategy for eliminating interference from EC' mechanism during analytical measurements based on plane-band-recessed microdisk array electrodes



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## ABSTRACT

A new type of three-electrode generator–collector sensor is described for the selective detection of any target analyte whose CV is chemically reversible in the presence of any interferent whose CV is chemically irreversible. The device consists of a layer of metallic material (Au in this work) poked by an array of cylindrical pores containing a ring-band electrode (Au in this work) placed in their middle and a disk electrode at their bottom (Au in this work). Operating the array of recessed disk electrodes to monitor the product of the stable analyte electronation while the top plane and the ring-band electrodes are poised at a potential located on the analyte and on the interferent waves allows suppressing entirely any contamination of the analyte concentration measurement by direct or indirect (EC') involvement of the interferent. The efficiency of such devices was successfully demonstrated based on the detection of dopamine in the presence of ascorbic acid in PBS electrolyte.

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## 1. Introduction

Electrochemical methods have been widely used for detecting electroactive species due to their advantages of simplicity and sensitivity [1–6]. A ubiquitous problem is that the coexistence of undesired electroactive species may seriously interfere with electrochemical signal of target analyte [7–10]. Modifying electrode surfaces has been an active area of research in electrochemistry and is an important way to eliminate signal of interferent [11–16]. However, this cannot eliminate the possible effect of redox recycling through solution electron transfer (ET) between the electronated analyte and the interferent (EC' mechanism) with the consequence that the analyte concentration appears larger than actual as was recently evidenced by Gros group [17]. Therefore, unless when ET rate is small enough or when the analyte detection produce a non-electroactive follow-up species, hence is unable to propagate an EC' sequence, this is a serious obstacle for quantifying a target concentration.

Dopamine (DA) is an important neurotransmitter, and abnormal DA concentrations cause physiological diseases [18,19]. Electrochemistry is

an important method for the detection of DA [20]. However, the detection of DA is generally affected by ascorbic acid (AA). This leads to a typical EC' mechanism [17]. The rather stable oxidation product of dopamine (DA<sub>ox</sub>) is reduced by AA, thus regenerating dopamine, enhancing the apparent electrochemical response of DA and affecting the meaning of measurements vs. independent calibrations. For this reason we wish to report a method for radical elimination of direct and indirect (EC') interferences.

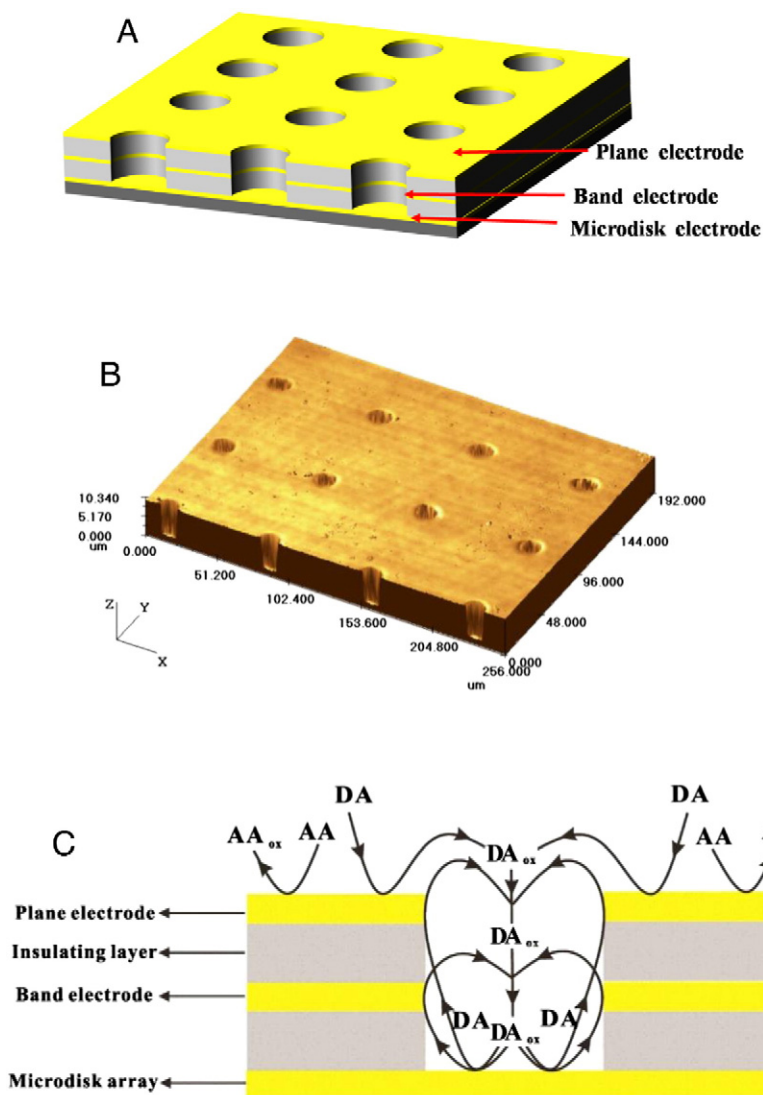
In our previous work, the microfabrication of plane-recessed microdisk array electrodes composed of a planar film electrode over a recessed microdisk array electrode by lithographic technology was demonstrated [3]. Simulations were performed to examine in full detail the origin of the successful performance of such devices and proposed a few theoretical possibilities for its further optimization [21]. Among these possibilities, one consisted in poisoning the plane electrode potential so that both DA and AA are oxidized under steady state, while the microelectrode recessed disk array is swept negatively to detect DA<sub>ox</sub>. Because AA exhibits a chemically irreversible electrochemical behavior due to the fast hydration of the tri-ketone primary oxidation product, interference signal from AA is avoided while DA can be detected.

In this paper, we wish to examine the experimental benefit of such operating mode, namely, inverted generator–collector mode [21], for the selective detection of DA in the presence of AA through avoiding direct and indirect interference arising from EC' mechanism.

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**Fig. 1.** (A) Sketch of the plane-band-recessed microdisk array electrodes assembly microfabricated and used in this work; (B) laser confocal image of the device; (C) sketch of the diffusion-electrochemical paths followed by the various species when the assembly is operated as described in the text in the presence of a sample containing dopamine (DA) and ascorbic acid (AA); DA<sub>ox</sub> represents the quinonic form of oxidized dopamine and AA<sub>ox</sub> the non-electroactive hydrated oxidation product of AA.

## 2. Experimental

### 2.1. Reagents

Aqueous solutions were prepared using fresh Milli-Q water with resistivity of 18.2 MΩ·cm. Dopamine was purchased from Sigma. Ascorbic acid, Na<sub>2</sub>HPO<sub>4</sub>·12H<sub>2</sub>O, KH<sub>2</sub>PO<sub>4</sub>, and H<sub>3</sub>PO<sub>4</sub> were purchased from Shanghai Chemical Reagent Co. All chemicals were of analytical grade and used as received. A phosphate buffer solution (pH 3.0) was used as electrolyte.

### 2.2. Plane-recessed microdisk array electrodes

The microfabrication of plane-recessed 12 × 12 microdisk array electrodes has been documented in detail elsewhere [3,22–25]. Different strategies for fabricating microelectrode arrays have been reviewed by Compton [24]. For screen-printed technique, the operation is simple and electrodes are usually fabricated by a special printer. The method is suitable for making planar or recessed microdisk electrode arrays, microband electrode arrays, interdigitated microelectrode arrays and linear microelectrode arrays [24,26–28]. Thermal lamination is an inexpensive method for construction of microelectrode arrays and suitable

for preparing electrodes with simple structures [29–31]. Lithography is an extensively used technique to fabricate various types of microelectrode arrays [24]. It benefits building complex structures through patterning of thin film. Based on the fact that our electrode configuration is composed of three working electrodes, lithography technique is selected to fabricate the electrode arrays. Compared with plane-recessed microdisk array electrodes [3,21], the only change consisted in the additional implantation through the same technique of a conducting Au layer located in between the recessed disks and the top plane and separated from these two elements by a polyimide insulating layer. This intermediate layer was perforated so that it appeared as an array of ring band electrodes located in the wells (Fig. 1A). For achieving the perforation, a layer of polyimide was coated, which was followed by photolithography, lift-off of Cr/Au layer, and etching polyimide by inductively coupled plasma. The vertical height distance between top plane and recessed disk electrodes was measured by profilometer (Dektak3 Series).

### 2.3. Instrumentation

All electrochemical measurements were carried out on a CHI814A potentiostat (Chenhua Corp., China). A standard polypropylene pipette tip was used as the electrochemical cell. A saturated calomel electrode

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