



Mechanical polishing as an improved surface treatment for platinum screen-printed electrodes



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ABSTRACT

The viability of mechanical polishing as a surface pre-treatment method for commercially available platinum screen-printed electrodes (SPEs) was investigated and compared to a range of other pre-treatment methods (UV-Ozone treatment, soaking in *N,N*-dimethylformamide, soaking and anodizing in aqueous NaOH solution, and ultrasonication in tetrahydrofuran). Conventional electrochemical activation of platinum SPEs in 0.5 M H₂SO₄ solution was ineffective for the removal of contaminants found to be passivating the screen-printed surfaces. However, mechanical polishing showed a significant improvement in hydrogen adsorption and in electrochemically active surface areas (probed by two different redox couples) due to the effective removal of surface contaminants. Results are also presented that suggest that SPEs are highly susceptible to degradation by strong acidic or caustic solutions, and could potentially lead to instability in long-term applications due to continual etching of the binding materials. The ability of SPEs to be polished effectively extends the reusability of these traditionally “single-use” devices.

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1. Introduction

Although various commercial brands of screen-printed electrodes (SPEs) are now available, the specific compositions of the materials used in their manufacture are often not disclosed. Screen-printing inks typically comprise of a blend of conductive material, polymeric binders and suitable solvents [1,2]. These “electrochemically-inert” binding materials can potentially block the electrochemically active surfaces, resulting in slower kinetics for heterogeneous reactions [2,3]. As a result, the development of surface treatment methods to enhance the electrochemical properties of SPEs has been of great interest [2]. In our previous work, the compatibility of room temperature ionic liquids (RTILs) with various commercial SPEs has been investigated for the sensing of ammonia [4], chlorine [5], methylamine and hydrogen chloride [6] and oxygen [7] gases. It was discovered that SPEs gave a poorer response for oxygen reduction (compared to conventional disk electrodes), and that a cross-over of the forward and reverse scans was present in cyclic voltammograms recorded in imidazolium based RTILs [7]. It was proposed that this was due to a chemical reaction of the highly reactive superoxide with the binding materials present on these electrodes [7]. This demonstrates that the binding materials in SPEs could in fact undesirably interfere with the electrochemistry of certain target species.

Investigations into pre-treatment methods for SPEs have been largely focused on carbon (C) based SPEs [2,3,8–10], mainly due to their low cost and wide use in a range of sensing strategies. However results from our earlier work [7] showed that C-SPEs do not perform as well as metal-based SPEs (e.g. silver (Ag), gold (Au), and platinum (Pt)) when used with RTIL solvents. Platinum SPEs (Pt-SPEs), on the other hand, showed the best response out of the four SPEs investigated. This has motivated us to investigate alternative pre-treatment methods for Pt-SPEs in order to improve their long-term stability for gas-sensing applications [7].

This paper explores the viability of mechanical polishing as an alternative pre-treatment for Pt-SPEs. To the best of our knowledge, there are only two reports of polishing of SPEs (both carbon). Office paper was used to polish C-SPEs, showing improved responses for IgG antibody adsorption and sensing [10], and alumina slurry or diamond spray was used to polish C-SPEs on polishing cloths, showing an improved analytical performance for the sensing of nitrite [11]. These techniques can be considered more “gentle” compared to the mechanical polishing with a machine polisher used in the present work. Four additional pre-treatment methods were explored as a comparison, two of which were reported to be ideal for C-SPEs, namely soaking in *N,N*-dimethylformamide (DMF) [8], and soaking followed by anodizing in aqueous NaOH solution [2], one (UV-Ozone treatment) was chosen as a similar method to oxygen plasma treatment (previously suggested for C-SPEs) [9], and one (ultrasonication in THF) to serve as a control to the polishing procedure investigated. It is to be noted that these treatments when applied to SPEs will not necessarily lead to a “cleaner”

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surface ideal for electrochemical experiments. The goal is to assess the viability of the different pre-treatment methods for Pt-SPEs and the possibility of using mechanical polishing of SPEs in a similar way to the well-known polishing of metal disk electrodes.

2. Materials and methods

2.1. Chemicals and materials

Ultrapure water (resistivity of 18.2 M Ω cm) prepared by an ultrapure laboratory water purification system (Millipore Pty Ltd., North Ryde, NSW, Australia), and ethanol (EtOH 99%, Sigma-Aldrich Pty Ltd., NSW, Australia) were used as solvents to rinse the Pt-SPEs (DropSens DRP-550, Oviedo, Spain) before and after experiments. Tetrahydrofuran (THF > 99%, Sigma-Aldrich Pty Ltd., NSW, Australia) and *N,N*-Dimethylformamide (DMF 99.8%, Sigma-Aldrich Pty Ltd., NSW, Australia) were used as received without purification. 0.5 M and 3 M Sodium hydroxide (NaOH, $\geq 98\%$, Sigma-Aldrich Pty Ltd., NSW, Australia), 0.5 M sulfuric acid (H₂SO_{4(aq)}, 95–98 wt.%, Ajax Finechem, WA, Australia), and 0.1 M potassium chloride (KCl, >99.5%, Fluka, Buchs, Switzerland) aqueous (aq) solutions were prepared using ultrapure water. 1.1 mM hexaammineruthenium(III) chloride ([Ru(NH₃)₆]Cl₃, 98%, Sigma-Aldrich Pty Ltd., NSW, Australia), and 1.0 mM potassium hexacyanoferrate(III) (K₃[Fe(CN)₆], 99 + %, Strem Chemicals, Massachusetts, USA) in electrolyte solution of 0.1 M KCl were used to characterize the electrochemically active surface areas of the Pt-SPEs.

2.2. Treatment methods for Pt-SPEs

2.2.1. Mechanical polishing

The Pt-SPEs used in this study contain a blue polymer mask to cover the underlying Ag traces (Fig. 1 (top)). The blue mask is made of a synthetic polymeric dielectric material that is soluble in THF. Accordingly, the electrode was sonicated in THF then polished with a mechanical polisher (Dap-V, Struers, Copenhagen, Denmark) at a rotation speed of 600 rpm, using 1 μ m alumina powder (Kemet Australia Pty Ltd., Marayong, NSW) on a soft lapping pad (Buehler, Illinois, USA) until a glossy finish was obtained (see Fig. 1 (bottom)). The planar electrode was pressed down firmly on the rotating polishing pad for approximately 1 min while moving the electrode in a circular motion, in order to ensure even polishing of electrodes. This is believed to be a much “harsher” method of polishing compared to that used previously for a C-SPE [10]. It was noted that for C-SPEs, polishing with emery paper easily destroyed the SPE [10], but the destruction of Pt-SPEs was not observed in the present

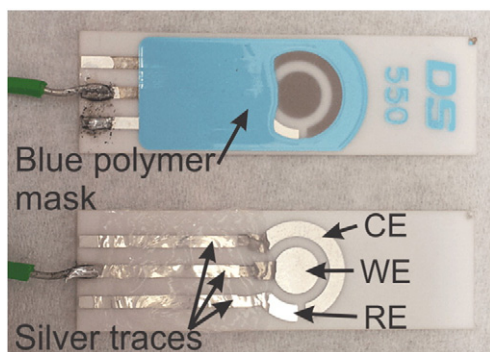


Fig. 1. (Top) fresh-out-of-the-box (untreated), and (bottom) mechanically polished Pt-SPEs (ca. 1 min duration on a mechanical polisher at a rotation speed of 600 rpm). A layer of silicone mask was applied to cover the exposed screen-printed silver traces. Wires were soldered at the end of the working electrode trace to connect the working electrode with the potentiostat.

work, despite the harsh polishing method employed. The exposed screen-printed Ag electrical traces were then re-masked with SELLEYS® Silicone Sealant (100% Silicone, purchased from local hardware store) to leave only the three electrode surfaces uncovered. Photographs of unpolished and silicone-masked polished Pt-SPEs with connecting wires soldered on are shown in Fig. 1. A more detailed account of the polishing protocol followed can be found in Section A of the Supporting information (SI).

2.2.2. Other treatment methods

Due to the unavailability of a plasma cleaner, a 50 W UV-ozone cleaner (BIOFORCE Nanosciences, Inc., Model: UV.TC.220 USA) was employed (Section B in SI), and Pt-SPEs were exposed for 30 mins. NaOH treated Pt-SPEs were prepared based on a method used for C-SPEs [2]; Pt-SPEs were soaked in 3 M NaOH_(aq) for 1 h, then anodized at 1.2 V vs. Ag/AgCl in 0.5 NaOH_(aq) for 20 s. The NaOH treated Pt-SPEs were then thoroughly rinsed in ultrapure water, and dried under a stream of nitrogen gas. DMF treated Pt-SPEs were prepared based on the pre-treatment method suggested for C-SPEs [8]. Pt-SPEs were briefly soaked in DMF in a small glass vial for 5 mins, before being cured in an oven for 20 mins at 100 °C in a covered glass Petri dish. Pt-SPEs designated as “THF treated” were subjected to the same pre-washing and post-washing methods (by sonication in THF and other solvents) as polished Pt-SPEs, but with the mechanical polishing step omitted. The THF treated Pt-SPE serves as a control for the polished Pt-SPEs, and also to explore its viability as a “stand-alone” alternative pre-treatment method for Pt-SPEs.

2.3. Electrochemical experiments

Electrochemical experiments were performed using a μ -Autolab Type III potentiostat (Eco-Chemie, Netherlands) interfaced to a PC with NOVA 1.8 software. All experiments were carried out inside a custom made aluminum Faraday cage and at a temperature of 293 \pm 2 K. A copper wire was soldered at the end of the middle Ag trace to allow the working electrode of the Pt-SPE to be connected to the potentiostat (see Fig. 1). The Pt-SPE has a nominal working electrode diameter of 4 mm, with the working (WE) and counter (CE) electrodes made of screen-printed Pt, and with a screen-printed Ag quasi-reference electrode (RE). The CE and RE on the SPE were not used in the experiments; instead, an external reference electrode (Ag|AgCl|KCl (1.0 M)) (BASi, Indiana, USA), and CE (Pt wire, \varnothing = 0.50 mm, Goodfellow Cambridge Ltd., UK) coil were used. This was to ensure that any differences observed in the voltammetric response were purely attributed to differences in the condition of the WEs. The Pt wire CE was cleaned by rinsing in EtOH, flaming in a Bunsen burner, sonicating in EtOH and then in ultrapure water.

Before measurements, solutions were purged with nitrogen gas by bubbling for 15 mins. Cyclic voltammograms (CVs) were collected at scan rates of 100 mV s⁻¹ or 1 V s⁻¹, and a step potential of 3 mV. For the [Ru(NH₃)₆]^{3+/2+} and [Fe(CN)₆]^{3-/4-} redox experiments, background corrections of the peak currents, *I*_p, were performed. This was carried out by subtracting the linearly extrapolated current (from the non-Faradaic part of the CV just before onset of the peak) from *I*_p. Potential-step chronoamperometry (PSCA) measurements were performed for 20 s (with 60 s pre-biasing at 0 V) with suitable over-potentials to ensure fast kinetics for convergence to a Cottrellian type response. PSCA in 0.1 M KCl_(aq) was also performed to allow for background-subtraction of the PSCA data. Finally, electrochemical impedance spectroscopy (EIS) of the unpolished and polished Pt-SPEs was conducted in 0.1 M KCl_(aq) electrolyte solution. EIS data was measured in the frequency range 1 mHz–1.5 MHz, operating in the single-sine-wave mode, with an RMS AC voltage of 5 mV oscillating about the open-circuit-potential (OCP) on the WE. For EIS experiments, to ensure that the RE was as close to the WE as possible, the Ag-RE on the integrated Pt-SPE device was used. A Pt coil CE was employed.

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