



A self-polishing platinum ring voltammetric sensor and its application to complex media



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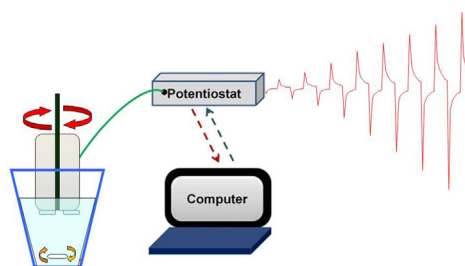
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HIGHLIGHTS

- A developed self-polishing voltammetric sensor eliminates sensor-drift.
- The polishing device of a Pt ring offers a reproducible and clean working electrode.
- Sensor shows a remarkably high capability to make repeatable measurements.
- Sensor was successfully applied to samples of urea, milk and sewage water.

GRAPHICAL ABSTRACT



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ABSTRACT

A self-polishing voltammetric sensor was recently developed and has been applied to samples of urea, milk and sewage water. The polishing device continuously grinds a platinum ring electrode, offering a reproducible and clean electrode surface. Principal component analysis (PCA) and partial least squares (PLS) techniques were applied to interpret the data and to build prediction models. In an evaluation of samples with different urea concentrations, the grinding step allows for repeatable measurements, similar to those after electrochemical cleaning. Furthermore, for the determination of sewage water concentrations in drinking water and for the evaluation of different fat contents in milk samples, the polishing eliminates sensor drift produced by electrode fouling. The results show that the application of a self-polishing unit offers a promising tool for electrochemical studies of difficult analytes and complex media.

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1. Introduction

The use and interest of electronic tongues (ETs) have grown during the last two decades and nowadays ETs are widely used in many applications [1]. Various analytical techniques have been developed for use in ETs, such as potentiometry, amperometry and

voltammetry, and the obtained data are analysed by multivariate data processing [1–3].

Voltammetric electronic tongues (VETs) offer robust measurements and a versatility of information concerning the studied systems [4–6]. In voltammetry different types of potential pulses are applied to the working electrodes and the resulting current response is a measure of ionic species and redox active compounds. When used in complex media the current responses are often very complicated with superposed current peaks. Thus, for their interpretation, multivariate data analysis is valuable.

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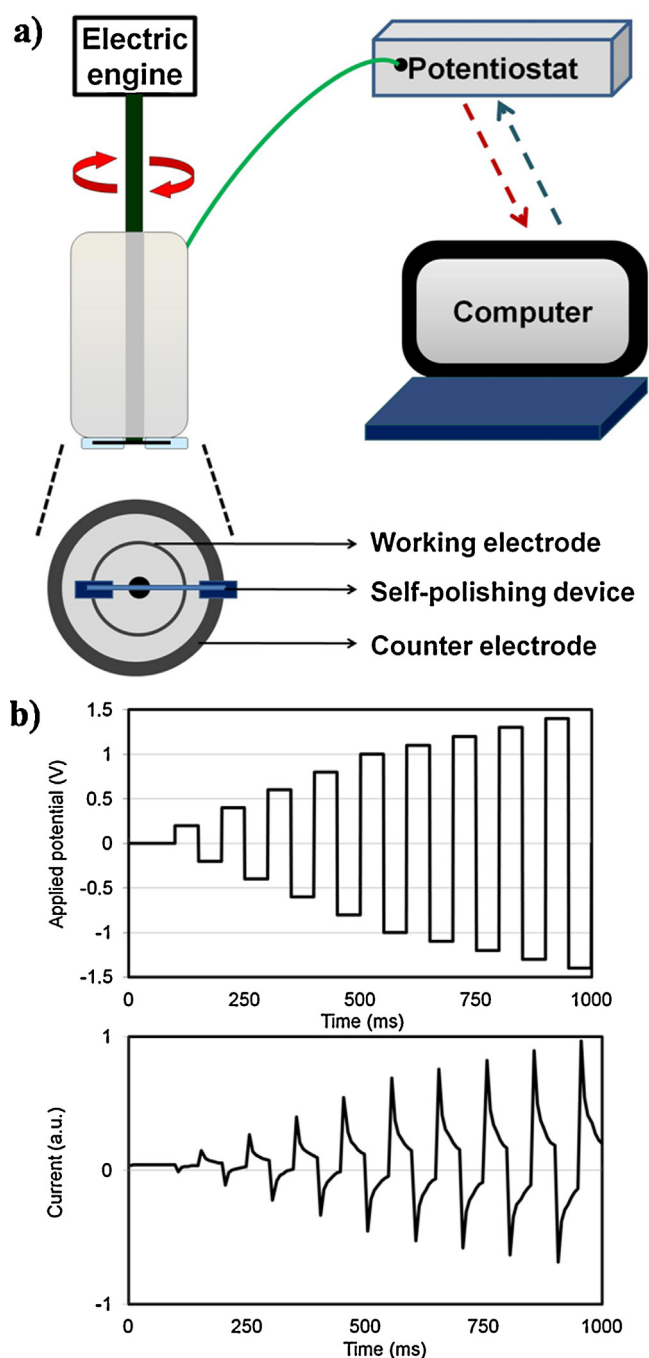


Fig. 1. (a) Scheme of the self-polishing voltammetric sensor. (b) The applied potential scheme (LAPV) with alternating cathodic and anodic potentials from ± 0.2 to ± 1.4 V and a typical current response scan from the voltammetric sensor.

Furthermore, this approach makes the use of a conventional reference electrode unnecessary [4], since the counter electrode works as a pseudo reference electrode. The electrode surface of a VET is of key importance since the material surface, its catalytic properties, its adsorption or chemical sorption properties, homogeneity, and smoothness have a high influence on its behaviour.

A general problem of electronic tongues and noses is electrode fouling which causes a decreased detection limit, a low reproducibility and drift. Fouling typically occurs when the ET is applied to crude and complex media. Especially for the VET, oxidised or reduced layers on the electrode surface and adsorption of various species to the surface will cause fouling of the electrodes [7–10].

There are different strategies to avoid or overcome drift problems [11,12]: (i) using mathematical tools for drift correction, such as normalization or component correction; (ii) recovering the surface with a mechanical polishing step; (iii) applying electrochemical cleaning steps; (iv) using a mechanical self-polishing device which renews the electrode surface. For the last strategy different self-polishing devices have been developed and applied to VETs with disc electrodes, to avoid fouling of the electrode [13]. After polishing the electronic tongue needs some time for equilibration of the electrodes and during this time drift appears in the measurements.

This report describes the use of a self-polishing VET with a platinum ring working electrode, which is continuously grinded by a sharp glass to eradicate these problems. The counter electrode is also continuously polished. This VET does not require a manual polishing step, neither electrochemical pretreatment, since the automatic physical polishing enables a reproducible and clean electrode surface. Since, polishing and measuring occur simultaneously in this system, initial drift due to polishing is minimal.

In this report this VET was applied and evaluated for studies of urea, milk and sewage water. These samples were chosen due to their known complexity and fouling of electrode surfaces.

2. Experimental

2.1. Chemicals and samples

Working solutions of $5 \times 10^{-1} \text{ mol L}^{-1}$ of urea (Merck) were prepared daily. In the urea study $1 \times 10^{-2} \text{ mol L}^{-1}$ phosphate buffer solution (PBS) was used at pH 7.3, prepared from K_2HPO_4 and KH_2PO_4 (Sigma–Aldrich) and diluted in ultrapure filtered water (Milli-Q plus 185 water purification system).

The set of milk and the cream milk samples with different fat contents were bought at a local market. Milk was chosen since it is considered to be a very difficult sample to measure due to its complex composition, strong fouling properties high fat content and some more unwanted properties. All milk samples are from the same Swedish milk manufacturer (Arla, Sweden) and were stored at 4°C .

A stock solution (x50) of synthetic sewage water was prepared diluting the following compounds in sterilized and filtered water: peptone casein pancreatic digest, meat extract, urea, NaCl, CaCl_2 , MgSO_4 , K_2HPO_4 , and NaHCO_3 (Merck) [16], and was stored at 4°C . The standard solutions were prepared daily by dilution of the stock solution. Real filtered sewage water sample were obtained from a local water treatment plant (Tekniska Verken, Linköping, Sweden) and was stored at 4°C . Temperature and pH measurements were made with a Metrel MA 5736 pH meter. All experiments were carried out at room temperature.

2.2. Selection of samples

The choice of samples is important. Urea is a difficult analyte since it has a strong adsorption effect, and during its oxidation a layer is produced on the electrode surface causing a loss in electroactive area. The determination of urea is important e.g. during monitoring of dialysis, demanding a stable sensor signal.

The second selected sample was milk. It is one of the most difficult media for electrochemical measurements since it consists of many different compounds, some of which adsorb strongly to the electrodes, causing fouling of the electrode surfaces, and consequently a dramatic drift in the data. Also, dissolved gases in the milk make it difficult to analyse. Even so, analysis of milk is important for the dairy industry, e.g. for monitoring quality changes of milk, evaluation of fat contents, detection of clinical mastitis, etc.

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