



Paper-based potentiometric ion sensors constructed on ink-jet printed gold electrodes

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ARTICLE INFO

Article history:

Received 1 July 2015

Received in revised form

23 September 2015

Accepted 16 October 2015

Available online 19 October 2015

Keywords:

Paper-based sensors

Potentiometric ion sensors

Ink-jet printing

Gold nanoparticles

Conducting polymer

ABSTRACT

Printed electrodes on a recyclable low-cost coated paper were used as a platform for constructing potentiometric ion sensors consisting of an ion-selective electrode (ISE) and a reference electrode (RE). The reference and working electrodes were printed by using a stable suspension of gold nanoparticles (AuNP) as the ink. Sintering turned the printed electrodes conductive. A poly(3,4-ethylenedioxythiophene) (PEDOT) layer, with poly(styrene sulfonate) (PSS) ions as counterions was deposited on the gold electrodes by electropolymerization, drop-casting or ink-jet printing. The reference electrode was prepared by further coating the PEDOT(PSS) layer with a poly(vinyl chloride) (PVC) membrane containing a lipophilic salt, tetrabutylammonium tetrabutylborate (TBA-TBB), thus resulting in a solid-contact reference electrode (SCRE). The working electrode was modified by coating the PEDOT(PSS) layer with a K⁺-selective membrane, to obtain a solid-contact K⁺-ISE. The electrochemical characteristics of the resulting electrode systems were studied by amperometric, potentiometric and electrochemical impedance spectroscopic (EIS) measurements. The potentiometric response toward K⁺ ions was additionally studied. The surface structure of the PEDOT(PSS) layers deposited by the different methods was studied with atomic force microscopic (AFM) measurements. It was shown that a well-functioning planar electrode platform with good electrochemical characteristics could be prepared by ink-jet printing in a repeatable manner. The planar electrode platform presented here offers a user-friendly and ecological alternative to perform chemical analysis from small sample volumes.

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1. Introduction

Potentiometric ion sensors are frequently used for sensing charged species, because they generally possess a large dynamic working range and a short response time. They are attractive for different application areas also because of their small size, relatively low production costs, low power consumption and portability [1,2]. As the need for accessing biochemical and chemical information is continuously increasing, new low-cost on-site measuring possibilities need to be developed further [3]. Different types of sensing setups, with varying deposition methods and substrate materials, have been proposed [4–6]. Paper-based sensors provide a relatively new technological solution for manufacturing simple and disposable sensors for different application areas such as clinical diagnostics and environmental analysis [7]. Furthermore,

printing technologies have attained a great deal of attention as a potential means of fabricating electrodes and different functional devices on paper substrates [8]. Especially low production costs can be obtained by using electrode platforms directly printed on a recyclable low-cost paper substrate [9]. Recyclable paper-based electronics possess flexibility in terms of different physicochemical properties, such as, topography, roughness, stiffness, surface energy and porosity. Paper substrates can even be modified by various surface treatments, which is an additional advantage. Screen-printing technology, adapted from the microelectronics industry, has been one of the most widely used methods for high-volume production of electrodes for electrochemical analysis systems [10,11]. The consumption of materials and the need for a stencil for printing patterns can be considered as the main drawbacks of this technique. Digital ink-jet printing, on the other hand, is a non-contact, additive and high resolution printing method that requires no stencil or printing plates and hence enables printing of versatile thin films with reduced material wastage and low cost. Drop-on-demand digital ink-jet printing is also scalable to

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Fig. 1. A photograph of the ink-jet printed gold electrodes and the hydrophobic SIBS layer. One SIBS pattern was printed also between the gold electrodes to better illustrate the SIBS structure.

large-area manufacturing purposes [12]. Digital ink-jet printing can be used for printing of gold electrodes from gold nanoparticle dispersions on a recyclable coated paper substrate [9] and highly conductive electrodes can be obtained in a roll-to-roll compatible manner by using a fast IR sintering method. Further, the conductivity of the electrode is preserved during bending and twisting of the paper electrode [13–15]. Due to the relatively smooth surface structure of the coated paper combined with the excellent barrier and wetting properties, very thin gold electrodes with low material consumption can be fabricated. The material costs for the ink-jet printed AuNP electrodes in the lab-scale were calculated to be 0.04 €/cm² [9]. In a roll-to-roll type of manufacturing process, the costs would be further reduced. Moreover, good adhesion and scratch resistance of the printed gold nanoparticles are achieved after the sintering procedure [9]. In potentiometry, the potential difference between an ion-selective electrode (ISE) and a reference electrode (RE) in contact with the sample solution is measured [1]. As classical REs with an internal filling solution are difficult to miniaturize and inconvenient to handle in the conjunction with all-solid-state working electrode setups, different configurations of all-solid-state REs have been studied [16–19]. As paper-based potentiometric sensors have recently started to gain attention within the sensor research community [20–24], the development of a paper-based RE is becoming equally important for practical paper-based sensor applications. In addition to functioning as a sensor substrate material, paper has also been used as a platform for microfluidic sampling combined with potentiometric sensing [25].

In this work, a planar electrode system was constructed on a paper substrate. The electrode system consisted of inkjet-printed nanoparticle-based gold working and reference electrodes. The gold electrodes and the paper substrate were partially covered with a hydrophobic styrene–isobutylene–styrene (SIBS) polymer for creating a well-defined electrode area where the PEDOT(PSS) solid-contacts and the subsequent PVC-based membranes could be easily deposited. A K⁺-selective membrane was drop-casted on the working electrode and a non-selective reference membrane, containing a moderately lipophilic salt, on the reference electrode, thus resulting in a solid-contact ion-selective electrode (SC-ISE) and a solid-contact reference electrode (SCRE) [19]. The TBA-TBB reference membrane has earlier been successfully used to complete all-solid-state potentiometric sensor setups like a multielectrode platform [26] and screen-printed carbon electrodes on plastic substrate [27] and on carbon cloth [28].

2. Materials and methods

2.1. Print substrates

An in-house developed multilayer-coated recyclable paper was used as the print substrate [29–33]. In the multilayered paper structure, the thin and porous top coating enables good print

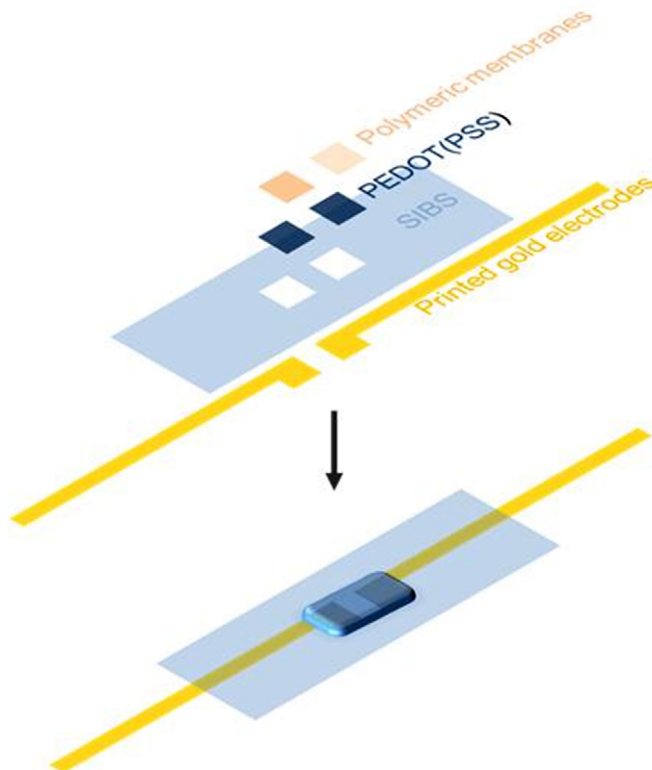


Fig. 2. A schematic representation of the whole electrode construction. The sample solution is deposited on top of the electrodes to complete the circuit.

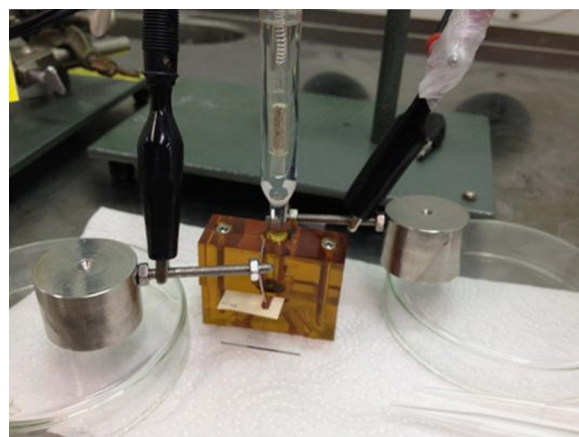


Fig. 3. The electrochemical cell setup used in the electropolymerization and in the characterization of PEDOT(PSS) deposited on printed AuNP-electrodes.

resolution, while the underlying barrier layer consisting of a blend of styrene butadiene latex and platy kaolin pigments provides the essential barrier properties against liquid penetration. Additional smoothing layers under the barrier layer were coated to ensure good horizontal alignment for the platy kaolin pigments. The thickness and the weight of the paper substrate were $130 \pm 30 \mu\text{m}$ and 126 g/m^2 , respectively. The substrate was calendered with a laboratory soft-nip calendar ($\sim 70^\circ\text{C}$, 50 bar) in order to further smooth the surface.

2.2. Chemicals

The synthesis of the dodecanethiol-protected gold nanoparticles (AuNPs) was performed as described previously [14].

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