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Formation of Alkane-Thiolates on Gold Electrode

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Abstract

Since the self-assembled monolayer (SAM) is the most typical organic assembly used to create alkane-thiolates monolayers on gold electrodes, the modified SAM on gold has a wide variety of applications in many different study fields. In order to determine the most effective method of SAM removal so the ICE can be reused, this work investigated the desorption of a SAM that was formed on the gold surface of an interdigitated chain-shaped electrode (the ICE, a novel electrode design, was created by our lab). Using a sodium borohydride solution and a short-term treatment, a straightforward and effective solution-based cleaning approach was used to remove a SAM from the gold surface of the ICE. This procedure was successful in recovering 90.3% of the ICE's original electrochemical properties. The successful re-deposition of a SAM onto the electrode surface following the removal process demonstrated that the ICE could be used again, yielding a high efficiency percentage of 90.1% for the reusability of ICE with the SAM modification. When removing SAM and recycling electrodes, the electrode interface alterations were examined using electrochemical impedance spectroscopy (EIS) and cyclic voltammetry (CV). The bonding structure and chemical state of the unmodified ICE and the changed ICE at each treatment step were characterised using the potent spectral techniques of X-ray photoelectron and Fourier-transform infrared spectroscopy. We confirmed and demonstrated the efficacy of this promising method for the removal of a SAM from the ICE and the re-use of ICE in the field of material deposition, with the goals of cost savings, improved experimental handling and environmental protection, based on the thorough discussion of analytical chemistry from the obtained EIS and CV data in this study.

Keywords: Interdigitated chain-shaped electrode • Self-assembled monolayer • SAM removal • Electrochemical impedance spectroscopy

Introduction

The deposition of materials, thin films, or composite coatings on a substrate is the focus of deposition procedures. They have a wide range of uses in the semiconductor, electronics, energy and biosensor industries [1-3]. There are several deposition methods, including vacuum thermal evaporation [1,4], electron beam evaporation, laser beam evaporation, nickel-fullerene C60 composition coatings [5], functionally graded Zn-Ni-Al_2O_3 coatings [5] and nickel-fullerene C60 composition coatings [6]. Electrochemical deposition is another method used to create thin layers, graphene and nanoparticles.

Self-assembled monolayers are organic assemblies created when molecules from a solution adsorb onto a solid surface (SAMs). This is illustrated by a SAM with a head group, a tail group and a functional end group. Numerous self-assembling systems have been investigated. Since the SH molecules of the head groups are firmly attached to the gold surface, monolayers of alkane-thiolates on gold are perhaps the SAMs that have been investigated the most. The easiest way to produce ultrathin, repeatable, oriented and ordered monolayers that can maintain the activity of functionalized macro- or micro-molecules is to use a SAM. This is because a SAM's functional end group has a carboxylic acid (-COOH) terminal, which is where functionalized macro- or micro-molecules attach. As a result, SAM has been employed in numerous types of study, such as studies on electrochemical biosensors, interface phenomena, biological and biochemical processes, electrochemistry and molecular interactions. As examples, gold electrode surfaces were modified with SAMs for binding different probe sequences to capture DNA target in order to establish a DNA biosensor for the diagnosis and treatment of an infectious disease; a gold substrate was treated with a SAM by dip-coating and by patterning with a benchtop microdropper to explore neuronal adhesion through the precise and exclusive positioning of the neural cell bodies onto modified electrodes and inhibits and at the same time, the effects of these modifications on the neural cell adhe, In order to immobilise bacteriophages for real-time monitoring of methicillin-resistant Staphylococcus aureus using surface plasmon resonance, a gold surface was functionalized with SAMs. SAMs were also used to develop a variety of electrochemical biosensors for the sensitive detection of protein biomarkers for Alzheimer's disease.

The usual method to ascertain the adsorption of SAMs onto a gold surface is the quartz crystal microbalance (QCM). The resonant frequency of the QCM chip was measured in three stages using a model QCM device: the bare chip, the chip immobilised with SAMs and the chip after removing SAMs. Recent QCM studies have reported the formation of SAMs from stirred ferrocenylundecanethiol in hexane at room temperature and the total frequency shift suggested SAMs formation. They have also reported the adsorption of two SAMs, namely octadecanethiol and octanethiol, in stirred hexane and cyclohexane solutions at room temperature over a broad range Then there is electrochemical impedance spectroscopy (EIS), By observing changes in the impedance at the electrode-electrolyte interface, a potent electrochemical approach has been used to characterise the production of SAMs on gold electrode surfaces [7,8].

Two linear interdigitated electrodes with two connection tracks make up the usual rectangular interdigitated electrodes (IDEs). Using IDEs has many advantages, including working with small amounts of sample and skipping the tedious polishing of solid electrodes. Through the use of electrochemical impedance spectroscopy to quantify changes in charge-transfer resistance and double-layer capacitance at the electrode interface, these two IDE configurations allowed for the widespread use of IDEs (EIS). However, a side consequence of IDEs is a concentrated electric field at the edge, which degrades the signal quality during EIS measurements. Particularly, it is well known that the working area is not homogenous in the standard rectangular IDE because the electric fields on the IDE are heterogeneous in our earlier study for monitoring the EIS of the cells on the IDE (the electric field is highly concentrated at the edge of the rectangular electrode). This implies that the cells positioned at the electrode corners have a significant impact on the EIS

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measurements of the cell or material layers on the IDE, lowering the sensitivity of the cell's EIS measurement across the entire electrode region.

Therefore, in our recent studies, the influence of the electrode edge on the distribution of the electric field has been avoided by properly designing the electrode shape, by controlling the width or spacing of the electrode, leading to improved sensor area homogeneity on electrodes and precise EIS measurements of the electrode surface modifications. We created an interdigitated chain-shaped electrode (ICE), a distinctive design, for earlier experiments. To counteract the impact of the concentrated electric field at the edge, the interdigitated electrodes of ICE are fashioned into a chain with numerous gold fingers each measuring 5 m in length. According to a COMSOL simulation, the distribution of electric fields in the spaces between the ICE fingers is largely uniform, which improves the homogeneity of the electrode working area and the ability to modify the electrode surface. Furthermore, the use of ICE has totally reduced the Warburg diffusion impedance in EIS.

Discussion

Cleaning the ICE is a crucial step in preparing it for chemical alteration by getting rid of various impurities from the electrode surface. The bare ICE was therefore cleaned with a 0.5 M $NaBH_4$ solution to get rid of any dust or other surface contaminants. The ICE's EIS and CV, both before and after cleaning. In an electrolyte of 1 mM K[Fe(CN)6]3/4 containing 0.1 M KCl, EIS results are expressed as changes in the interfacial electrode surface in a Nyquist plot of the image impedance (Im(Z)) vs. the real impedance (Re(Z)). With the help of the Randle's equivalent circuit model (REC), which had three parameters, the implications of Nyquist plots were elucidated. Rs, corresponding to the solution resistance, which can be calculated by looking at the real axis (Re(Z)) value at the high frequency intercept; Rct, which stands for the interfacial charge-transfer resistance and corresponds to the semicircular diameter of the Nyquist plot; and Cdl, which stands for the double-layer capacitance that is parallel to the Rct.

The electrode and the modified electrode's bonding groups were identified using FT-IR. the ICE, ICE/SAM and re-ICE FT-IR spectra. The spectra of ICE/SAM demonstrate the deposition of the SAM on the Au surface of the bare ICE by showing the formation of the covalent bonding of Au-S, the stretch mode of the C-S groups of the SAM and the O-H deformation mode, respectively, in the band ranges (500 to 850) cm^{-1} and (950 to 1400) cm^{-1}. After the SAM was removed by NaBH4, the re-ICE displayed FT-IR spectra in all wavenumber areas that matched the spectra of the bare ICE. This outcome demonstrated that the ICE had successfully been reorganised following the deposition and removal of SAM, respectively.

Conclusion

In order to assess the viability of reusing the electrode, this work set out to investigate the most efficient way to remove changed SAM from an ICE's gold surface. The goal of this work was to reuse ICE in order to save money, protect the environment and handle experiments more quickly. As a result, a practical, straightforward and effective technique to eliminate SAM on the gold surface of ICE was investigated. By examining electrochemical tests like EIS and CV as well as microscopy techniques like XPS and FT-IR, researchers were able to assess and validate the complete removal of the SAM and the good reusability of ICE with re-addition of the SAM. The high-efficiency percentages of 90.3% and 90.1% for the re-deposition of the SAM on the ICE surface and recovery of the electrochemical properties of the ICE were also calculated. This indicates a well-founded method for the regeneration of the gold surface of the ICE in material deposition applications, offering benefits in terms of cost savings, experimental control and environmental protection. This publication also provided a novel and clear procedure for cleaning the bare electrode surface using a $NaBH_4$ solution.

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