

# Electrode Passivation

Subjects: Electrochemistry

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The main reason for electrode passivation is adsorption/deposition of a passivating compound on working electrode surface. The most pronounced consequences are the decrease of the rate of electrode reaction resulting in the shift of half-wave or peak potential to more negative (in the case of cathodic reaction) or to more positive (in the case of anodic reaction) potentials and in the decrease of peak current, which is especially in the case of pulsed technique influenced by this rate.

Keywords: electrode passivation ; electrode surface renewal ; electrode pretreatment ; electrode modification ; measurement in flowing systems ; electrode protection ; novel electrode materials resistant to passivation

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## 1. Introduction

The immense potential of modern electroanalytical methods in monitoring of organic compounds is generally recognized and appreciated <sup>[1][2][3][4][5][6][7]</sup> because of their low investment and running costs, reasonable and in many cases “fit for the purpose” sensitivity and selectivity, easy automatization, easy miniaturization resulting in portability of corresponding instrumentation, user friendliness, and environmental friendliness (“green electroanalytical chemistry”). For the same reason, electrochemical sensors are frequently used nowadays. Recent advances in electrochemical biosensing with special emphasis on universal point-of-care systems can be found in minireview <sup>[8]</sup>. The heart of any electrochemical sensors is a working electrode on which the electrical signal is generated via an electrochemical process and which serves as a transduction element. In more complex and sophisticated biosensors, it can be covered with a sensing and biorecognition layer. Advances in the development of electrochemical sensors for clinically relevant biomolecules, including small molecules, nucleic acids, and proteins are reviewed in <sup>[9]</sup>. Excellent review <sup>[10]</sup> is focused on advancing the speed, sensitivity, and accuracy of biomolecular detection using multi-length-scale engineering from more general view not limited to electrochemical sensing. Modular construction of an autonomous and programmable multi-function biochemical network that can identify, transform, translate, and amplify biological signals into physiochemical signals based on logic design principle <sup>[11]</sup> is another possible approach. Similarly, a scalable system of reporter enzymes cleaves specific DNA sequences in solution, which results in an electrochemical signal when these newly liberated strands are captured at the surface of a nanostructured microelectrode <sup>[12]</sup>. These sensors utilized complex chemistry to achieve high-accuracy sensing of large biomolecules, but the electrochemical interface is actually very simple. All above mentioned papers <sup>[8][9][10][11][12]</sup> are focused mainly on large molecular analytes and comprehensively describe the construction of various types of electrochemical sensors, which can be beneficial to readers of this review, too. Nevertheless, this review pays attention mainly to smaller organic molecules.

Here, the big problem connected with the use of modern electrochemical techniques in practical analysis remains the passivation of working electrodes by products/intermediates of electrochemical reactions or by components of complex samples, which can adsorb on the electrode surface, thus fouling the electrode and complicating the determination or even making it impossible. Strategies to combat passivation must be tailored to the type of sensors, electrode materials, mechanism of passivation, used electroanalytical method, and type of matrix. It is obvious that surface chemistry plays a decisive role in this process. Generally, the problems with passivation can be addressed (eliminated, diminished, or minimised) by the following approaches:

- To renew the surface of the working electrode after each measurement. An ideal example is dropping mercury electrode, the surface of which is renewed spontaneously every few seconds, thus minimising problems with passivation <sup>[1][2][3][4][5][6][7]</sup>. However, problems with mercury toxicity prevent widespread use of this excellent tool. Similar surface renewal is used in the case of dropping gallium-based electrode <sup>[13]</sup>, so far not too frequently used. The same holds for renewable liquid Cd-Ga electrode <sup>[14]</sup> and In-Ga electrode <sup>[15]</sup>. Another possibility is to use carbon paste electrodes with easily mechanically renewed surface <sup>[16][17]</sup>, solid amalgams electrodes with mechanically or electrochemically renewed surface <sup>[18][19][20]</sup>, or mechanical, chemical, or electrochemical cleaning of the working electrode after each measurement <sup>[1][2][3][4][5][6][7]</sup>. However, these approaches (with notable exception of

electrochemical cleaning) require human involvement and complicate desirable automatization. This complication can be at least partially eliminated by using retractable-pen-based renewable silver amalgam film electrode [21]. Another interesting possibility currently under investigation in our laboratory is to use analogously the renewable gallium film. Carbon electrodes electrochemical pretreatment can play a very useful role in this field [22] due to its simplicity, efficiency, and low cost and due to resulting undistorted, well-defined, and reproducible signals.

- To use disposable electrodes just for one measurement. This approach is frequently used in medical diagnosis, where exchange of working electrodes is imperative for each sample to eliminate any risk of cross-contamination [23]. Increased price of the determination and generation of waste of used disposable electrodes (a not too “green” situation) makes this approach not well suited for large scale monitoring. However, recently proposed electrodes based on aluminium wrapping foils [24], gold on the surface of used CD [25], or carbon rods from used batteries [26] present an inexpensive alternative to commercial disposable electrodes. The same holds for carbon film electrodes where only the film is disposable [27][28], resulting in decreased price and more “green” attitude. A novel approach can be demonstrated by the use of disposable pencil graphite electrodes for detection of SNP (single nucleotide polymorphism) in human mitochondrial DNA [29].
- To use surface modifications preventing passivation [5][6][7]. Many papers on modified electrodes confirm increasing interest in this approach. However, this approach still remains a combination of art and science, with limited robustness and shelf-life of modified electrodes [30][31][32] complicating to a certain degree routine applications of modified electrodes in large scale monitoring.
- To use measurements in flowing systems minimizing passivation [33][34][35] or to use RDE (rotating disc electrode) [36][37][38][39][40][41][42][43][44][45][46] or hydrodynamic electrodes [37]. In all these cases, products/intermediates of electrochemical reaction are washed away from the electrode surface thus minimizing their deposition on working electrode. Here, HPLC-AD (HPLC with amperometric detection), FIA-AD (flow injection analysis with amperometric detection), and BIA-AD (batch injection analysis with amperometric detection) are frequently used for large scale monitoring [47][48]. This approach is useful only if electrode products/intermediates are responsible for passivation. Combination of BIA with passivation resistant BDDE (boron doped diamond electrode) [48] can further amplify antifouling effect.
- To use novel separation methods preventing access of passivating components of environmental matrices towards the working electrode surface (membrane separation, HFME (hollow fibre membrane microextraction) [49][50][51][52][53]. This approach is useful only if matrix components are responsible for passivation.
- To use novel electrode materials more resistant toward passivation. This approach is generally most promising and acceptable for practical laboratories. This is the most attractive and appealing approach and most welcome one from the point of view of large scale and high throughput monitoring. Moreover, it is well compatible with continuous monitoring. Probably the best known “passivation resistant” electrode material is boron doped diamond [54][55][56] especially if its surface is –H terminated. Another more recently developed “antifouling” material is tetrahedral amorphous carbon with incorporated nitrogen (ta-C:N) [57] and sp<sup>3</sup>-carbon dominated hydrogenated surface [58][59]. However, in all cases, passivation depends on conditions, analysed solutions, potential program, electrode surface, and on many other parameters.
- To use various heated electrodes (metallic [60] or carbon based [61]) to remove deposited substances from the electrode surface. This approach can also improve S/N ratio, mass transport, and reaction kinetics resulting in improved figures of merits. However, it is experimentally more demanding and so far not too widely used.
- To use various enzymes converting organic analytes prone to electrode fouling into simple inorganic products with negligible danger of electrode fouling [62]. Enzyme based electrodes are very frequently used for their selectivity and sensitivity so that antifouling possibilities are not too frequently accentuated.
- To use intentional electrode fouling by adding strongly adsorbable organic compounds (e.g., mercapto-hepta(ethyleneglycol) forming SAM (self-assembled monolayer) on electrode surface), thus minimising further electrode fouling by analytes/electrode reaction products. This “fighting fire with fire” approach results in the decreased signal of an analyte after formation of SAM but then the signal remains stable after multiple repetition of voltammetric scans, thus enabling standard addition of calibration curve evaluation. The above mentioned SAM exhibits strong antifouling properties against proteins adsorption, and thus it can be useful for biosensors construction [63].

It can be summarized that in spite of a number of papers dealing with electrode passivation, a non-systematic and more or less random approach still prevails in this field. Therefore, it is desirable to investigate the relationship between structure of the electrode surface, structure of passivating substance, and electrochemical behaviour of passivating

substances to clarify the nature and mechanism of these processes and to find ways to diminish, suppress, or completely eliminate passivation and connected negative effects complicating electroanalysis of real samples.

## 2. Importance of Electrode Passivation—Reasons and Consequences

It is obvious that the main reason for electrode passivation is adsorption/deposition of a passivating compound on working electrode surface. The most pronounced consequences are the decrease of the rate of electrode reaction resulting in the shift of half-wave or peak potential to more negative (in the case of cathodic reaction) or to more positive (in the case of anodic reaction) potentials and in the decrease of peak current, which is especially in the case of pulsed technique influenced by this rate. Decreased active surface of the working electrode naturally results in the decrease of limiting currents observed in CV (cyclic voltammetry), LSV (linear scan voltammetry), DCV (direct current voltammetry), and in amperometric methods. The most usual symptom of electrode passivation is the decrease of an electrochemical signal with repeated scans in voltammetric and with time in amperometric techniques. It is clear that electroanalytical methods can provide sufficient evidence of electrode passivation and valuable information about its mechanism and are most frequently used for these purposes. However, a number of other techniques used for surface characterization in material sciences should be exploited in this field more frequently to monitor changes in the course of electrode passivation and opening new pathways for its minimisation/elimination. Moreover, the obtained knowledge regarding characterization of chemical composition and electronic structure of electrode surfaces may eventually lead to rational design of new (smart) electrode materials and measuring protocols resistant to electrode passivation for monitoring of organic compounds. Last but not least, this approach could close the gap between quickly growing number of electroanalytical methods for organic substances and their practical applications for large scale monitoring still somewhat hindered by problems with electrode passivation. It is worth noting that the same phenomenon (adsorption) that helps to increase the sensitivity of adsorptive stripping techniques in other cases complicates and even prevents the application of electroanalytical methods in large scale monitoring, making it even more important topic for basic research. Moreover, understanding the mechanism of passivation will pave the way for its minimisation/elimination.

## 3. How to Minimise Electrode Passivation

The following different approaches to eliminate unfavourable influence of electrode passivation are pursued most frequently, the first one being the most challenging and most promising, as documented by selected papers from our UNESCO Laboratory of Environmental Electrochemistry <sup>[1][16][17][18][19][20][21][27][28][47][50][51][54][55][56][64][65][66][67][68][69][70][71]</sup>.

- Search for materials resistant to passivation and investigation of the relationship between surface properties and proclivity to passivation. This general strategy can be successful, whether the passivation is caused by some matrix components or by products/intermediates of electrode reaction. Moreover, the detailed structural investigation of the electrode surface before and after passivation can reveal the mechanism of passivation and contribute to the elimination of its unfavourable effects. Passivation resistant electrodes would be definitely the most useful electroanalytical tool. This research is scientifically the most complicated, and it requires intensive cooperation of experts in surface analysis (both in pre-passivation and “post-mortem” (after passivation) electrode surface analysis), in new electrode materials and in the development of novel electroanalytical instrumentation/measuring protocols. Without this synergy, it would be impossible to reach this very ambitious goal.
- Investigation of electrochemical polishing/cleaning/pre-treatment of electrode surface between successive measurements to eliminate unfavourable effects of electrode passivation together with using less common potentials programs and currents response treatments. The possibility of electrochemical activation in situ will be again applicable irrespective of the mechanism of passivation; this is the most simple and user-friendly approach from the point of view of an electroanalytical chemist if the antifouling electrode is not available for the given analyte and matrix. Many inspiring details on the application of electrochemically pretreated electrodes can be found in an excellent review <sup>[72]</sup>. The electrochemical assisted antifouling strategy using continuous CV was used, e.g., for voltammetric determination of clenbuterol and ractopamine at phosphorene nanocomposite modified electrode <sup>[72]</sup>. Antibiofouling strategy for optical sensors by chlorine generation using low-cost, transparent and highly efficient electrodes based on platinum nanoparticles coated oxide <sup>[73]</sup> can be inspiring for electrochemical removal of organic compound fouling the electrode surface.
- The use of disposable electrodes with a low number of repeated measurements limiting the influence of passivation on obtained electrochemical signal. This approach is most frequently used in medical laboratories because of low risk of cross contamination. However, the electrode passivation should not be too pronounced to enable at least a few

measurements on one electrode necessary for standard addition method. However, the large piles of disposed electrodes are not too compatible with the idea of “green electroanalytical chemistry”. Electrodes with renewable surface, either bare or modified, are more acceptable from this point of view.

- Elimination of electrode passivation by measurements in flowing systems resulting in removal of the products of electrochemical reactions, which can cause passivation by dimerization/polymerization and deposition on electrode surface. This approach can be useful only in the case where electrode passivation is undoubtedly caused by electrode reaction products/intermediates, which can be revealed by detailed surface investigation of the electrode before and after passivation. Moreover, the instrumentation is slightly more complicated than for batch analysis. On the other side, determination is faster. Here, most promising areas are HPLC-AD (HPLC with amperometric detection), FIA-AD (flow injection analysis with amperometric detection) [74], and BIA-AD (batch injection analysis with amperometric detection) [47][48][75][76], the last one being the most interesting because of its so far untapped potential in the field of large-scale monitoring.
- Prevention of passivating molecules from matrix to access the electrode surface via preliminary separation of the analyte from those molecules, e.g., using membrane filtration or HF (hollow fibre) microfiltration [49][50][51][52][53]. This approach is used quite frequently in the analysis of biological liquids but not so frequently in environmental and food analysis. Obviously, it can be used more frequently, but it will have some limitations for high throughput large scale monitoring of organic compounds. Here, the development of miniaturised solid electrodes compatible with this approach is an imperative.
- Application of electrodes with easily renewable surface. This approach mirrors a fascinating property of dropping mercury electrode, the surface of which is automatically renewed with each new drop. This is a very promising approach both from the point of view of the fundamental research and its practical applications. Renewable amalgam, carbon paste, solid carbon-based, bismuth, gallium, and antimony electrodes will play decisive role in this field. An interesting possibility is to use the renewal of the PGE (pyrolytic graphite electrode) surface by using the adhesive tape, which is more laborious than renewing the mercury drop but still much simpler than many typical polishing procedures required for most solid electrodes [77]. Another undoubtedly very useful fouling resistant electrode with easily renewable surface can be commercially available bismuth drop electrode [75].
- Modification of the electrode surface by fouling resistant polymers, films, or nanoparticles (either metallic or carbon-based). This is a most “high-risk–high-gain” research with sometimes unpredictable results. However, it can open new frontiers in the fight against passivation. Therefore, this kind of fundamental research should be pursued more frequently, because it can bring really new knowledge, combining the successful elimination of problems with passivation with increased sensitivity and selectivity connected with the application of nanoparticles. Silver nanoparticles on the black phosphorene surface and its amino-functionalized multi-walled carbon nanotubes nanocomposite and the application of this nanohybride nanozyme sensor for voltammetric monitoring of uric acid, xanthine, and hypoxanthine [78] is an interesting example of this approach. Similarly, Pt nanoparticles and other metal nanoparticles can be used for these purposes.
- The use of different solvents that can help to minimize electrode fouling is well documented by a detailed study of electrooxidation of phenol on platinum electrode in different solvents [79]. This simple and straightforward approach definitely deserves further detailed investigation.
- The fascinating development in 3D printed sensors has led to new generation of sensors with good passivation characteristics, wearable sensors being just one of many examples [80]. Undoubtedly, further fast development of fouling resistant electrodes/sensors can be envisaged.

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