Cyclic Voltammetry – Investigation of a Gold Surface

PSAPP-006 Electrochemical Experiment

PalmSens
Compact Electrochemical Interfaces

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1 Instructions

The following instructions will guide you to perform the experiment. The theory on which these experiments are based, can easily be checked online at palmsens.com, then search for "Introduction to Cyclic Voltammetry". The material needed for the experiments can be ordered via https://www.palmsens.com/product/educational-kit/#contents

2 Devices and Equipment

- EmStat / EmStat Blue
- sensor cable
- sensor connector
- maybe a USB cable
- computing unit (PC, Laptop, notebook, tablet PC (Android), smartphone (Android))
- potentiostat software (PStrace, PStouch)
- calculation and plotting software (Excel, Origin, MatLab, Mathematica)
- counter electrode
- reference electrode
- working electrode
- ItalSens IS-Au
- retort stand
- retort clamp
- beaker (electrochemical cell)
- stirrer
- gas source (optional)

3 Chemicals

- 0.5 M H_2SO_4

Note: If you use the 50 mL beaker, you will need 40 mL of solution for your measurement. We recommend using a smaller vessel that allows the electrodes to be immersed in 5 mL solution for the enzyme catalysis experiment.

4   Instructions

4.1   Investigation of a Gold Surface

Gold is a special metal. The impact gold had in the history of mankind is well-known, but from a scientific point of view gold has quite a number of special properties. Under the right conditions aurides can be made, that is salts where gold is present as the anion Au^-. Working with gold electrodes is popular due to the fact that organic sulfur compounds like thiols (R-SH) easily bind to a gold surface. Working with gold electrodes has the disadvantage that gold is soft and hydrophobic. As a result a gold surface collects all organic residues in the air and gets dirty rather quickly. A popular method to clean electrodes is electropolishing. In this method high anodic potentials are applied and adsorbed species will desorb. Furthermore gold forms a gold oxide monolayer, that is the gold is covered with a single layer of oxidized gold. The reduction peak is very sharp and in sulfuric acid no other peak overlaps with it. The charge of the reduction peak can therefore be used to calculate the real surface of the gold electrode.

1. Take an ItalSens IS-Au electrode and insert it into the sensor holder. To attach the electrode to the sensor connector take the strip of electrodes and hold it at the end with the single letter (A to D) and a number (1 to 20). Since the electrodes are easily contaminated or destroyed, please take care not to touch it with bare hands. Cut away the access plastic around one electrodes SPE and place it in the connector. You must cut the sensor narrow enough for fitting it into the connector, but please take care not to damage the electrodes nor the lines during cutting.
2. Connect the sensor holder to the potentiostat and fix it in the retort stand.
3. Immerse the three electrodes on the stripe into a 0.5 M sulfuric acid.
4. If a method for performing a CV was already prepared for you, load the method. If not choose *Cyclic Voltammetry* from the drop down menu. Choose the current ranges *1 µA, 10 µA* and *100 µA*. The fields *Sample* and *Sensor* are for your own notes. Since we do not want a pre-treatment of the electrode set *t condition* and *t deposition* to 0. Set the other parameters to:
   a. *t equilibrium* = 8 s
   b. *E start* = 0 mV
   c. *E vertex1* = 0 mV
   d. *E vertex2* = 1.4 V
   e. *E step* = 0.001 V
   f. *Scan rate* = 0.1 V/s
   g. *Number of Cycles* = 5
5. Start the measurement. The electrode should be clean now.
6. Repeat with *Scan rate* = 0.005 V/s, *Number of Cycles* = 1 and save the curve under the menu *Curve*.
7. Repeat the measurement with a *Scan rate* of 0.01 V/s, 0.025 V/s, 0.05 V/s, 0.1 V/s, 0.25 V/s and 0.5 V/s. Save each curve.
8. The peak around 0.65V is the gold reduction peak. The adsorbed species is reduced. Look at the reduction peak and plot $I_{p,c}$ versus ν to proof or falsify.
9. The next steps are used to calculate the real surface. Take the CV with ν = 0.1 V/s to calculate the surface. First take the area of the reduction peak $A_{p,c}$, which is also the integral. To get the charge divide by the scan rate. This is also represented in equation 4.1.

$$Q = \frac{A_{p,c}}{v} \qquad\qquad 4.1$$

10. The real surface is determined by using the charge per surface area for a gold oxide layer q. This value can be found in the literature: 3.84 C/m². [1] The real surface Areal is calculated according to

$$A_{real} = \frac{Q}{q} \qquad\qquad 4.2$$

11. Usually the roughness factor r is used to characterize the gold electrode. It is the ratio between real and geometric area (see equation 4.3). It shows if electrodes are polished in a reproducible way or if they are smooth enough to be used in following experiments.

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[1] V. M. Andoralov, M. R. Tarasevich, O. V. Tripachev, Russ J Electrochem 47 (2011), 12, p. 1327–1336.

$$r = \frac{A_{real}}{A_{geo}}$$

4.3

In this experiment you calculated the gold surface by measuring the charge of the reduction peak when the surface oxidizes.

Please note that teachers can request the answers to the question in the instructions, using
https://www.palmsens.com/contact/

$$r = \frac{A_{real}}{A_{geo}}$$

4.3