Cyclic Voltammetry – Ferrocene-carboxylic Acid as a Mediator

PSAPP-007 Electrochemical Experiment

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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
- retort stand
- retort clamp
- beaker (electrochemical cell)
- stirrer
- gas source (optional)

3 Chemicals

- 5 mM Ferrocenecarboxylic acid in 0,1 M K_2HPO_4 (for easier dissolving use an ultrasonic bath or heat)
- 1 M glucose solution (has to be prepared one day earlier)
- 4 mg/l GOx in 0,1 M K_2HPO_4
- 0,1 M K_2HPO_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 Ferrocenecarboxylic Acid as Mediator for Glucose Oxidase

This part of the experiment will show that ferrocenecarboxylic acid is a mediator for glucose oxidase. The natural electron acceptor for glucose oxidase is oxygen. Because of this a removal of oxygen from the measuring solution usually improves the result. It is recommended to gently bubble argon or nitrogen gas through the measuring solution for 15 min after assembly of the cell. Usually Sufficient results are also acquired without oxygen removal. A schematic representation of the electron transfer from glucose via the enzyme glucose oxidase and the redox mediator ferrocene carboxylic acid to an electrode is shown in Figure 4.1.

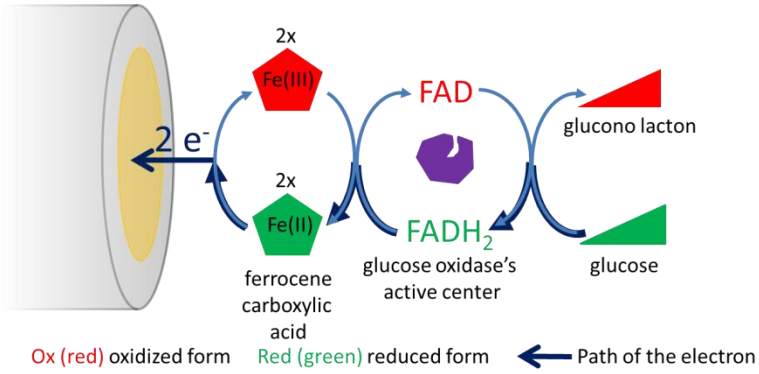

Figure 4.1: schematic representation of the electron transfer from glucose via the enzyme glucose oxidase and the redox mediator ferrocene carboxylic acid to an electrode

1. This time a cell with a lower volume can save money, because enzymes are quite expensive. We recommend using a smaller vessel than the usual 50 mL beaker. The recommended low volumes are used during this instruction and in brackets you will find the volumes for a 50 mL beaker. Set up the cell. Fill the cell with 5 mL 0.1 M K_2HPO_4 (20 mL) solution and immerse all three rod electrodes.
2. 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* = 0.7V
    e. *E step* = 0.001 V
    f. *Scan rate* = 0.05 V/s
    g. Number of Cycles = 3
3. Start the measurement. Save the curve under the menu *Curve*.
4. Add 1.25 mL (5 mL) of 5 mM ferrocene carboxylic acid solution to the cell and pump the solution a few times with a pipette or stir the solution until it is homogeneous. Set the dropdown menu next to *Run* button to *Overlay*. Repeat the CV from point 2.
5. Add 1 mL (5 mL) of 4 mg/L GOx solution and pump or stir until the solution is homogeneous. The removal of oxygen is recommended. Repeat the CV.
6. Add 120 µL (600 µL) of 1 M glucose solution and repeat the CV.
7. Do your CVs show that ferrocene carboxylic acid is a good mediator? Explain your conclusion.

In this experiment you detected the amount of glucose using standard addition.

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