



Characterisation of embroidered 3D electrodes by use of anthraquinone-1,5-disulfonic acid as probe system



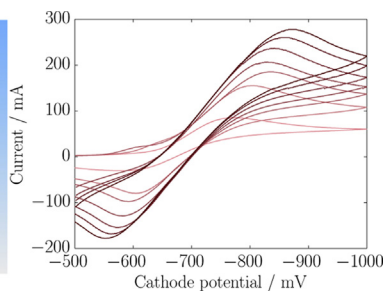
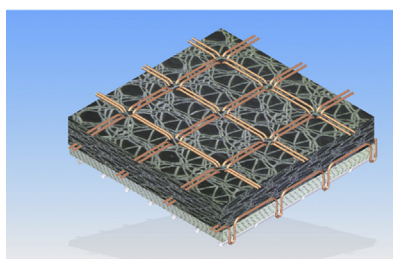
Noemí Aguiló-Aguayo, Thomas Bechtold*

Institute for Textile Chemistry and Textile Physics, Leopold-Franzens-University Innsbruck, Hoechststrasse 73, A-6850 Dornbirn, Austria

HIGHLIGHTS

- 100 cm² embroidered 3D porous electrode design can be used for electrochemical applications.
- AQDS²⁻ exhibits quasi-reversible consecutive two electron transfer reactions.
- 3D electrode shows current densities similar to micro cell and 100 cm² plane Cu-plate electrode.
- 3D electrode presents higher peak-to-peak separation due to its porous nature.

GRAPHICAL ABSTRACT



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ABSTRACT

New electrode designs are required for electrochemical applications such as batteries or fuel cells. Embroidered 3D Cu porous electrodes with a geometric surface of 100 cm² are presented and characterised by means of the anthraquinone-1,5-disulfonic acid (AQDS²⁻) redox system in alkaline solution. The electrochemical behaviour of the 3D electrode is established by the comparison of cyclic voltammetry responses using a micro cell and a 100 cm² plane Cu-plate electrode. Dependencies of the peak currents and peak-to-peak potential separation on scan rate and AQDS²⁻ concentration are studied. The AQDS²⁻ characterisation is also performed by means of spectroelectrochemical experiments.

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1. Introduction

For many applications of electrochemical energy storage the maximum cell current and energy efficiency are limited by the current density at the electrode. As the composition of the electrolyte and the type of electrode reaction are defined by the type of electrochemical system to be used, an increase in cell current per geometric area requires the use of electrodes with high specific surface.

In particular for application in energy storage different approaches have been chosen to realise a 3D-structured electrodes.

Besides incorporation of conductive fibres into the active electrode coating, three-dimensional structures formed by fibre webs, fabric from conductive material and metal foam have been proposed to increase electrode thickness. The use of a third dimension in the electrodes provides a foundation for the design of different cell configurations and provides enhanced energy efficiency essential in storage energy devices, such as batteries [1].

Embroidery techniques can be useful to prepare conductive 3D-structures for electrodes with high flexibility in geometry and choice of conductive material e.g. Cu-wire, stainless steel fibre yarn, aluminium wires. This allows design of new 3D-current collectors

* Corresponding author.

E-mail addresses: textilchemie@uibk.ac.at, noeaguilo@gmail.com (T. Bechtold).

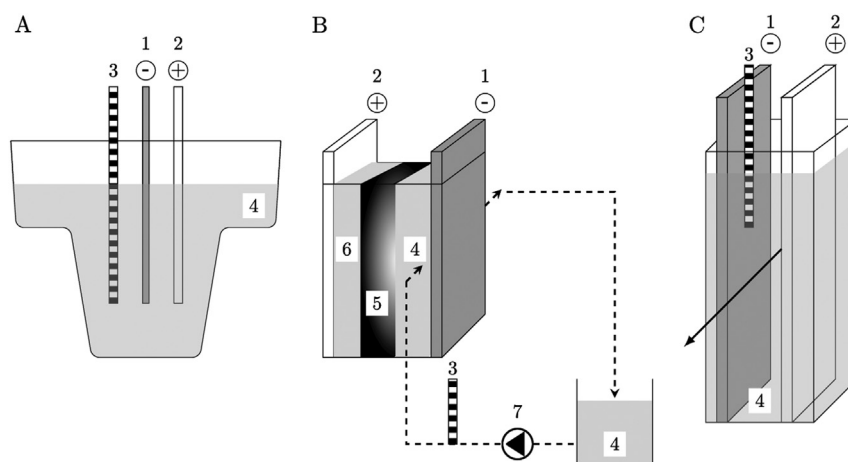


Fig. 1. Schematic drawing of the different cell configurations used in the experiments: 1 cathode, 2 anode, 3 reference electrode, 4 AQDS²⁻ solution. (A) Small cell using circular plane ($\phi = 1.5$ mm in diameter) Cu electrode. (B) 100 cm² cell assemblies for the Cu-plate and 3D embroidered Cu porous electrodes: 5 membrane, 6 anolyte, 7 peristaltic pump (arrow indicates the flow direction). (C) Cell for the spectroelectrochemical measurements (arrow indicates the direction of the light beam).

with high conductivity and adjustable porosity. Furthermore when using this technique the electrodes are manufactured in the dimensions of the electrochemical cell and thus no cutting is required. This helps to minimise the risk of short-circuits and hot-spots at the sides of the electrodes.

The electrochemistry of anthraquinoid compounds is of growing interest as the properties of these chemicals makes them interesting for many applications, e.g. oxygen reduction [2–4] hybrid capacitors [5], batteries [6], liquid crystalline applications [7].

The cathodic reduction of anthraquinoid compounds to the dihydroxy form requires the transfer of two electrons. In aprotic solvents radical intermediates can be observed, while in alkaline aqueous solutions a rapid two electron transfer is observed. Depending on the solution's pH value the reduction also involves protonation steps. The anthraquinone-1,5-disulfonate (AQDS²⁻) is a water soluble molecule which exhibits a reversible redox behaviour in alkaline solutions as both the anthraquinone and the reduced leuco form are chemically stable. Thus this compound can serve as soluble redox probe, which can be used to assess performance of electrodes. In addition yellow anthraquinone changes its colour during reduction from yellow to dark red, which allows optical monitoring of the reduction.

In the present work alkaline solutions of AQDS²⁻ were used to characterise the performance of embroidered 3D-electrodes manufactured from Cu-wire. The electrodes were tested by voltammetry in a stopped-flow cell. For comparison CV-experiments with AQDS²⁻ were performed on small diameter plane Cu electrodes, on a 100 cm² plane Cu metal electrode and on embroidered 3D-electrodes both mounted in a stopped-flow cell. For further characterisation of the AQDS²⁻ system spectroelectrochemical experiments were performed.

2. Experimental

The redox system under study was a set up of different concentrations (0.005 M, 0.010 M and 0.015 M) of a solution of 9,10-anthraquinone-1,5-disulphonic acid (AQDS²⁻, 95%, Sigma–Aldrich Chemie GmbH, Steinheim, Germany) in 0.1 M NaOH. The electrochemical reduction of AQDS²⁻ was investigated by cyclic voltammetry using three different cell configuration (Fig. 1, Table 1). Cyclic voltammograms (CV) were performed using a potentiostat (EG&G 274A Princeton Applied Research). All potential values were taken related to (Ag/AgCl/3 M KCl) reference electrode (RE).

The micro cell configuration consisted of a small cell (EG&G Mirco Cell, 10 mL catholyte volume), using a circular plane copper working electrode (WE) with 1.5 mm in diameter. The counter electrode (CE) was a Pt-wire electrode placed in a separated electrolyte bridged by a porous diaphragm (vycor). The WE was polished before an experiment by use of a 0.5 μ m alumina dispersion (EG&G polishing kit). CV were recorded in the potential range between -500 mV and -850 mV at different scan rates from 5 to 100 mV s⁻¹. Solutions were degassed with argon for 3 min before each CV measurement. Experiments were repeated 5 times for each condition.

The 100 cm² cell assemblies were built as parallel plate cell, with anolyte and catholyte separated by a cation-exchange membrane (Nafion type). A plane copper plate with an active area of about 100 cm² (mass around 50.14 g/100 cm²) or an embroidered three-dimensional copper based porous electrode (mass around 4.33 g/100 cm²) were used as WE [8]. For comparison a 0.1 mm Cu foil with an area of 100 cm² would have a mass of 8.61 g/100 cm². The schematic presentation of the cross section is given in Fig. 2. The 3D electrodes (Fig. 3) were prepared by technical embroidery (Tegra 71

Table 1

Characteristics of the different cell configurations used. Electrodes present same geometric (Area_{geom}) and electrode (Area_{electr}) area unless otherwise specified.

Cell configuration	Cathode (WE)	WE dimensions	Anode (RE)	Electrolyte V mL ⁻¹
A	Circular Cu-plane	ϕ : 1.5 mm Area: ~ 1.8 mm ²	Pt wire	10
B	Plane Cu-plate	Area: 100 \times 100 mm ²	Stainless steel plate	Anolyte: 300 Catholyte: 800
B	Embroidered 3D Cu porous	Cu-wire ϕ : 0.08 mm Cu-wire length (l): 100 mm Number of wires (N): 400 Area _{geom} : 100 \times 100 mm ² Area _{electr} : $2\pi(l/2)N = 10^4$ mm ²	Stainless steel plate	Anolyte: 300 Catholyte: 800
C	Plane Cu	Area: 30 \times 10 mm ²	Pt foil	2.70

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