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A versatile in-situ EPR spectroelectrochemical approach for
electrocatalyst research

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Dedication

Abstract: Empirical electrocatalyst research generally consists of the
synthesis and experimental characterization of catalysts and the
analysis of electrolysis products by conventional analytical techniques. In-situ EPR-spectroelectrochemistry provides an evidence based in-
depth understanding of the formed intermediates and the reaction
mechanism enabling the desired tuning of electrocatalysts. The use
of this technique has been underexploited because of the opposite
requirements they impose on the conventional setup. In this work, a
versatile electrode with commercially available indium tin oxide on
polyethylene terephthalate (PET) was constructed for the first time
which can fit inside commonly used EPR flat cells. It allows
reproducible electrodeposition of catalytic material combined with
sensitive radical detection, due to its large surface area and minimal
disruption to the resonator’s Q-factor. Moreover, with a resistivity of
8–10 Ω sq⁻¹, the surface potential of the thin semiconductor electrode
within the resonator was well-controlled, allowing targeted radical
production.

Introduction

Electron paramagnetic resonance (EPR) can provide valuable
information about the oxidation state of an atom or atoms within a
(macro)molecule and its environment by using unpaired electrons
as probes. The technique has been used to study various
paramagnetic species[7,8] such as organic radicals[9,10], proteins[11–
13], polymers[6,14] and transition-metal complexes[11,12]. Although
EPR spectrometers operating at up to 263 GHz are now
commercially available, X-band (~ 9 GHz) equipment are still the
most widely used. Most X-band EPR spectrometers use
resonators to enhance the sensitivity for measuring weak
magnetic dipole transitions. These resonators (or cavities when a
metal box is used) store the microwave energy so no microwaves
are reflected back and a standing wave is attained with the electric
and magnetic component exactly out of phase. The efficiency of
this storage is represented by the Q-factor and acts as an
indication of sensitivity. In X-band continuous-wave EPR
erspectrometers rectangular cavities are most widespread and
applicable for a wide range of samples whilst cylindrical ones are
more suitable for aqueous and high-dielectric samples[13]. The
choice of cavity thus depending on the nature of the sample.
Analysis of aqueous samples at room temperature in a
rectangular cavity is preferably done in a flat cell with sample layer
thickness of approximately 0.3 mm because the electric dipole of
water interacts with the electric field of the microwaves resulting
in a large amount of electromagnetic radiation being absorbed by
the sample and consequently a lower Q-factor[14]. The sample
has to be located at the position in the cavity with minimum of the
electric field component and the maximum of magnetic field
component of the microwave which is facilitated by the use of a
flat cell for a rectangular cavity operating in the TE102
mode[15]. In cylindrical cavities, a capillary needs to be used for the
measurements of lossy samples.
Combining electrochemical generation of radicals with EPR spectroscopy was first established by Ingram et al. in 1958 for ex-situ generated aromatic radicals which were measured at low temperature by taking aliquots and subsequent freezing[19]. Simultaneous Electrochemical and Electron Spin Resonance (SEESR), comprising of in-situ generation of paramagnetic species in solution, was first exploited around 1960 by Geske and Maki[20-23]. This simultaneous approach allowed for the detection of shorter-living radicals. The working electrode was positioned in a narrow tube in the center of the cylindrical cavity while the reference electrode sat in a reservoir on top. Ohmic resistance resulting from such positioning reached thousands of ohms which is undesirable. The Adams group published for the first time the use of similar electrodes for aqueous solutions in a flat cell suitable for rectangular cavities[19] which was then adapted by Goldberg et al. who positioned all three electrodes in the flat part of the cell[24] solving most of the resistance issues inherently present in the use of narrow channels[25]. However, positioning of the counter electrode this way resulted in the detection of undesired counter reaction radicals, complicating the spectrum[26]. Even when the counter electrode was placed in the bulk section of the flat cell these counter reaction radicals were able to diffuse into the flat part.

The use of metal wire pseudo reference electrodes which fits the flat part, should be weighed of against the use of a real one at the entrance of the flat part. The main advantage of a pseudo reference in a flat cell is that it can be placed in the flat part close to the working electrode surface where detection will occur, thus minimizing the ohmic drop. However, its potential can vary over time. In contrast a normal reference electrode is stable over time but only controls the potential in the bulk section of the flat cell. The working electrodes vary from wires to mesh/gauze structures to metal rods[23,24]. In analytical electrochemistry flat and uniformly accessible working electrodes are preferable to derive kinetic parameters or the diffusion coefficient. When doing combined experiments for qualitative intermediate detection, it is desirable that the working electrode has the highest possible electroactive surface to produce the desired radical instantly. Sensitivity can be increased by introduction of electrolyte flow in different configurations like channeles[25,26], tubular[27,28] and wall-jet[29] electrodes. Bond et al. constructed a small volume cell with wire electrodes which provides possibilities for the use of lessy solvents at temperatures as low as 203K[30]. Most of the SEESR setups, which are not commercially available, utilize a solid metal working electrode to generate radicals in-situ. The bulk metal electrode always results in deterioration of the Q-factor by absorption of microwaves.

In recent electrochemical research the used catalytic materials are mostly nanostructured and require special deposition procedures. To obtain uniform catalyst distribution and morphology, the surface of the substrate material should be flat and reproducible which is not the case in any of the above setups. On top of that, electrodeposition requires semi-infinite linear diffusion, sufficient surface conductivity and a homogeneous potential distribution over the working electrode. The material of the working electrode should also be inert in the applied reaction conditions and in EPR preferably non-susceptible for the incident microwave radiation, in the field interval investigated, nor for the applied field inducing undesired currents.

One of the promising materials possessing properties suitable for spectroscopic analysis techniques is Indium Tin Oxide (ITO) or other Transparent Conductive Oxides (TCO) like Aluminum Zinc Oxide (AZO). Multiple papers use glass-supported ITO as a substrate because of its optical transparency and others deposited metallic particles on the surface[31-34]. It is being used in photovoltaic devices and thus has been already incorporated into EPR setups for Electrically Detected Magnetic Resonance[35]. However, the dimensions of commercial glass-supported electrodes do not allow their utilization in EPR flat cell applications. The combination of in-situ UV-Vis/EPR-spectroelectrochemistry was exploited by Neudeck and Dunsch in several publications using wire working electrodes[36], laminated Au and Pt meshes[37–39], LIGA electrodes[40] and laminated ITO glass electrodes[41,42]. The latter focusing on the behavior of a polypyrurole layer on the surface and other hole-transporting organic substrates. Recently the use of mesoporous layers of ITO particles was reported for water oxidation and alcohol oxidation[43,45] and applied for the investigation of redox reactions in biomolecules. This electrode consisted of a Ti wire decorated with 50 nm ITO particles with the subsequent immobilization of proteins on the surface and was used in combination with an EPR tube[46].

In this work, we demonstrate the construction of a promising ITO-on-PET-based EPR electrochemical setup fitting the commercial Wilmad flat cell for aqueous samples in a rectangular cavity in an X-band EPR spectrometer. The ITO working electrode has a flat surface allowing reproducible integration of (electro)deposited nano-scale catalysts. The construction is inexpensive and straightforward so experiments can easily be repeated multiple times. The setup is applicable in a broad range of reactions in both aqueous and non-aqueous environments and demonstrates reasonable electrochemical behavior in the EPR flat cell. Consequently, targeted in-situ radical generation is possible. It is optically transparent and very thin which is an amelioration compared to the bulk metal electrodes because the material does not significantly interfere with the Q-factor of the resonator. The electrode surface area approaches the maximal available space in the flat cell producing the maximum radical concentration possible. As a proof of concept the electrodeposition of Ag nanoparticles and NIO layer was performed with consecutive in-situ use for the reduction of methyl viologen in aqueous solutions and benzoquinone in acetonitrile with detection of their radical oxidation and alcohol oxidation[31–34]. It is being used in photovoltaic devices and thus has been already incorporated into EPR setups. The bulk metal electrode always results in deterioration of the Q-factor by absorption of microwaves.

Results and Discussion

1. Setup and procedure

Combined EPR-spectroelectrochemical measurements were performed in a Wilmad Supracil WG-810-A-Q quartz flat cell assembled as shown in figure 1. Custom-made ITO working electrodes were made using ITO-on-PET microscope slides (8-10 Ω sq⁻¹, 200 μm thickness with a 700 nm ITO layer) obtained from SPI supplies and (60 Ω sq⁻¹, 178 μm thickness with a 100 nm ITO layer) from Sigma-Aldrich. This allowed it to fit inside the 500 μm thin gap of the quartz flat cell. The ITO layer of the SPI product had a homogeneous surface promoting reproducible electrodeposition of particles (figure 2A). This particular product
was chosen because of its low resistivity (Bode and Nyquist plot of both in supp. info.) and high transmittance. Further increases in conductivity with ITO layer thickness reaches a plateau as does transmittance at higher wavelengths\textsuperscript{47,48}. The paper of Do-Hyoung Kim even suggests that electromagnetic wave shielding occurs with increasing film thickness\textsuperscript{49}. The slides were cut to rectangles of 37.5 x 3.5 mm. The tip of the ITO working electrode reached the center of the flat part which coincided with the most sensitive section of the resonator. The junction with the connecting Cu wire was kept in the bulk section on top and was shielded by a polypropylene (PP) encapsulation using a heat gun whilst being pressed against the semiconductor surface by carbon tape for maximum conductivity. The PP covered 10 mm of the ITO surface resulting in 0.96 cm\textsuperscript{2} exposed working electrode surface area. A Teflon covered 75 µm Ag wire pseudo reference electrode was used (stripped of the Teflon at both ends) and positioned as close to the tip of the ITO electrode without causing a short circuit (<1 mm). The Pt auxiliary electrode was placed in the bulk section on top as close to the entry of the flat part as possible. The cell was filled with 1 ml solution submerging all electrodes while leaving the exposed Cu connection wire dry. This setup provided a cheap, straight-forward and versatile method for combined in-situ EPR and electrochemical measurements of electrodeposited catalytic materials. The setup was placed in a TE\textsubscript{102} rectangular cavity in a Bruker E580 Elexsys spectrometer.

2. Electrodeposition

In order to do in-situ electrocatalyst research, a catalytic material should first be deposited on the ITO surface. It is possible to chemically synthesize particles and coat them on the surface of any material. Since the substrate is conductive, we chose to electrodeposit the catalytic material directly on the ITO layer (figure 2A) after the electrode was assembled as mentioned in §1. Deposition experiments were performed on the ITO obtained from SPI as the resistance of the Sigma-Aldrich product was very high (2.25 ± 0.23 x 10\textsuperscript{4} Ω) when electrochemical impedance spectroscopy was used for its determination. Every reference to ITO from hereon will be concerning the first one unless stated otherwise. Impedance values for the fitted Nyquist and Bode plots by R(RQ) and R(RQW) circuits for the Sigma and SPI obtained ITO respectively, can be found in the supporting information. (Table S1). Ag and Ni are active electrode materials for a number of reactions\textsuperscript{50–55} and were therefore selected as catalyst material. Ag was deposited by reduction of Ag\textsuperscript{+} from the solution with varying double pulse conditions. The nucleation potential was varied from -0.6 to -1 V vs Ag/AgCl for 1 to 2 s and the growth potential between -0.05 and -0.15 V vs Ag/AgCl for 50 to 150 s. The optimal parameters were found to be similar to what we published on a glassy carbon substrate resulting in the smallest and best distributed particles\textsuperscript{56} namely 1.5 s at -0.8 V and 100s at -0.15 V (figure 2B). The catalyst deposited onto the ITO layer did not show any discrimination between locations on the electrode in terms of particle size and morphology even though the surface resistance should theoretically vary from 62.9 to 94.3 Ω from connection to tip. It was imperative to create the desired morphology at the tip of the electrode as it was positioned in the most sensitive part of the resonator in our setup. Deposition of a Ag layer by one pulse deposition at the nucleation potential was attempted for 5 min resulting in the formation and agglomeration of bigger clusters (figure 2C) giving the electrode a matt appearance (figure 3A). Ni was deposited by applying -1.3 V vs Ag/AgCl for 10 minutes and showed a tendency of depositing in 30 nm particles which clustered and formed a layer (figure 2D). As the layer was not electrodeposited under inert atmosphere, NiO was rapidly formed. Total coverage of the ITO substrate was already visual evidenced by a shiny grey layer on the ITO semiconductor (figure 3B).
3. Electrochemical validation

Cyclic voltammetry (CV) was used to investigate the behavior of the electrode assembly. The reduction of benzoquinone (BQ) in acetonitrile and that of methyl viologen (MV) in water were selected as test cases because they allow the assessment of the set up in both organic and aqueous solutions as well as starting from neutral or positively charged reagents. Reducing BQ with one electron yields the semiquinone radical anion in aprotic environment (Eq. 1). In the flat part of the EPR flat cell this could be observed visually because of the change in color of the solution from pale yellow to darker yellow and even orange in higher concentrations (figure 3C). MV$^{2+}$ also undergoes a one-electron reduction with the formation of the methyl viologen.
smaller volume and thus less analyte to reach the surface of the position in the flat cell. This could be explained by the non-

The bulk experiment showed a bigger reduction current than that data for the electrode with Ag

(red

radical cation (Eq. 2). This radical has a very distinct blue color (figure 3D) and the 2-electron reduced species is again colorless (Eq. 3). These visual properties make them particularly helpful in designing electrodes dealing with a resistive surface or for testing of the electrical connection encapsulation. CVs of the bare ITO on PET, and the Ag- or Ni-deposited electrodes were done in an undivided setup and in the EPR flat cell. Figure 4 shows the response of the bare ITO electrode in both cases (BQ and MV).

The splitting between oxidation and reduction peak of BQ was similar for both the bulk cell as the flat cell (0.6 V). The potential shift of 0.9 V between the cells was caused by the use of a pseudo reference in the flat cell. The reduction reaction of BQ in the flat cell did not show a notable reagent depletion fallback after the reduction potential was reached. It should be noted that this reduction on a regular glassy carbon cathode displays normal reversibility[57]. Methyl viologen was scanned to the second
electron reduced species is again colorless

For BQ reduction the electrode with a NiO layer deposited on ITO (NiO/ITO) behaved similarly to its Ag analogue (figure 6). There was a gradual separation of oxidation and reduction peaks in both bulk and flat cell with the latter showing less separation than any other BQ experiment (220 mV). The CV of MV in bulk is stretched out in both cells indicating that there is a background current with the peaks of MV superimposed. Hydrogen evolution onset

\[
\begin{align*}
\text{BQ} + \text{e}^- & \rightarrow \text{BO}^+ \\
\text{MV}^{2+} + \text{e}^- & \rightarrow \text{MV}^+ \\
\text{MV}^+ + \text{e}^- & \rightarrow \text{MV}^0 \\
\text{MV}^0 & \rightarrow \text{MV}^{2+} + \text{e}^- \\
\text{MV}^{2+}_{\text{ads}} & \rightarrow \text{MV}^{+\text{aq}} + \text{e}^-
\end{align*}
\]
potential could already be reached considering the steep increase in current. The CV vertex potential for the flat cell measurement was taken at -1.1 V. Scanning to more negatively potentials had the consequence of producing noisy current variations. Presumably the thin ITO layer and its junction with the metallic particle deposits begins to degrade at these negative potentials and associated current. This degradation could be the reduction of the ITO which we had suspected when scanning over a wider potential range with the bare ITO electrode. The initially transparent surface produced a pinkish color on the surface when scanning negatively that faded in the back scan. This effect was undesirable because it may have influenced the electronic structure (conductivity) of the ITO and the transparence of the electrode by reducing the oxides in the semiconductor which may have led to inactive regions on the surface.

Figure 6. CV of 5 mM BQ (top) and 5 mM MV (bottom) on Ni/ITO on PET electrode in bulk vs (sat.) Ag/AgCl and in EPR flat cell vs Ag wire at 25 mV s⁻¹.

4. In-situ EPR spectroelectrochemical validation

The Q-factor for the empty flat cell in the cavity was 2600 ± 100 which was in accordance with the specifications of the spectrometer for an empty cavity. Introduction of acetonitrile + 0.1 M TBAP lead to a Q-factor of 700 ± 100, while H₂O + 0.2 M Na₂SO₄ reduced it even more to 300 ± 100. Introduction of a non-lossy solvent (toluene) lead to a Q-factor of 2000 ± 100. These Q-factors did not change when the ITO electrode was introduced into the electrolyte filled flat cell which is beneficial for the sensitivity. In-situ electrolysis experiments were performed as described in §1. The employed electrodes were bare ITO, Ag/ITO and the NiO/ITO. Potentials in the kinetic region of the corresponding CVs were applied. Figure 7 displays the spectra that were obtained for BQ and MV reductions. The EPR spectra were simulated using the EasySpin-5.1.11 module running in Matlab®.[60] The following isotropic $g$ and hyperfine values were used for the simulation of the spectra of the BQ radical anion: $g_BQ = 2.0050$ with $A_H = 6.75$ (4), while for the MV radical cation $g_{MV} = 2.0024$, $A_H = 11.85$ (2), $A_{NI} = 11.18$ (6), $A_{NI} = 3.40$ (4), $A_{NI} = 11.18$ (4). These are in accordance with the literature values.[18,61]

The spectra showed that for the three electrodes that were utilized, the formed radicals for each reduction reaction are the same, indicating the formed radical was independent of the deposited electrocatalyst. This was as expected considering the mechanism of the reduction reaction being analogous on each of the catalytic materials and the substrate. The peak intensities differ depending on the electrode due to the different active surface area resulting from the electrodeposition. Comparison of the double integral of the acquired spectra with that of a TEMPO (2,2,6,6-tetramethyl-1-piperidinyl)oxyl) spin-counting calibration gave radical concentrations of approximately 83 μM BQ and 66 μM for MV reduction reactions (Tables S2, S3). When the concentration of BQ was increased its reduction at the Ag/ITO electrode gave a broadened spectrum (figure 8). Using the same EPR parameters as above, the broadened spectrum can be simulated by simply increasing the Voigt linewidth with $\sigma = 0.02$ mT and $\gamma = 0.034$ mT, to $\sigma = 0.144$ mT and $\gamma = 0.095$ mT, where $\sigma$ and $\gamma$ are the Gaussian and Lorentian components, respectively (figure 8). Broadening could be due to adsorption on the electrode surface and/or viscosity of the solution resulting in restricted mobility of the radicals. However, these phenomena are unlikely as broadening was not observed in the lower concentration experiments. A more probable reason for the spectral broadening is that a high concentration of the BQ radical anion is being generated, leading to dipolar and exchange interaction between the radicals. The high radical concentration may also result in the formation of aggregates which reduces the mobility of the radicals, further broadening the spectra.

The combination of favorable conditions for electrodeposition of catalytic material on the commercial ITO-on-PET substrate with generation of paramagnetic species or radicals in the sensitive part of the resonator on a high surface area electrode are critical for the detection of short living radicals (lifetime shorter than the timescale of 1 scan) in electrocatalytic research.

Conclusion

Combining electrochemical catalyst research with in-situ EPR detection provides some challenges but also opportunities for the electrochemist. These challenges consist of the choice of cell, electrode materials and their configuration. A working electrode material that is thin, inert, conductive and spectroscopically transparent can be found in an ITO layer deposited on PET as presented in this work. This commercially available substrate provides a reproducible surface area for electrocatalyst deposition and fits in the flat part of the measuring cell, reaching the most sensitive part of the cavity. The electrode is thin and optically transparent and does not disturb the cavity tuning resulting in a good Q-factor. Both electrodeposited Ag nanoparticles and NiO layer were electrochemically and spectroscopically evaluated as electrodes for the BQ and MV...
Figure 7. EPR spectra obtained from in-situ electrolyzing 0.5 mM BQ at 9.655 ± GHz and 0.2 mW (left) and 5 mM MV at 9.661 ± 0.001 GHz and 0.8 mW (right) on bare ITO on PET (A,B) electrode, Ag/ITO on PET (B,C) and NiO/ITO on PET (E,F). The spectra are a mean of 10 scans over ~ 105 s. Red: experimental, black: simulation.

Figure 8. EPR spectra obtained from in-situ electrolyzing 5 mM BQ at 9.661 ± 0.001 GHz and 0.2 mW on Ag/ITO on PET electrode with simulation of the broadened spectrum. The spectra are a mean of 10 scans over ~ 105 s.

reduction. Particles were evenly distributed across the total surface and could be tuned in size. This proof of concept set-up shows the possibility of advanced electrocatalyst synthesis being tuned to fit the desired reaction pathway and final chemical products through in-situ monitoring. It can give an understanding of why an electrode material favors certain reaction pathways. The resistivity of the ITO layer is low enough to avoid a potential gradient from an order that would be detrimental for radical generation at the tip of the electrode. In general, the amount of radicals formed can be increased by raising the concentration of analyte or enlarging the active surface area. Especially the latter is of primordial importance for detecting short living radicals, as an increase in the bulk concentration is not always feasible or desirable. Our setup maximally uses the available space in terms of active surface area for commercial flat cells. It opens the door to study more reactive radicals in a reproducible and affordable manner.
Experimental Section

Electrochemical measurements were performed in acetonitrile (Chem-Lab, HPLC Grade) and MilliQ water (18.2 MQ cm⁻¹) with respectively 0.1 M tetrabutylammonium perchlorate (TBAP, Sigma-Aldrich, 99.0 %) and sodium sulphate (Acros Organics, 99.0%) as supporting electrolyte. Benzoquinone (Sigma-Aldrich, ≥99.5%) solutions were prepared in acetonitrile and methyl violet (Sigma-Aldrich, 98%) in water. Depositions of Ag were performed using 1 mM AgNO₃ (Sigma-Aldrich, ≥ 99.0%) in acetonitrile with 0.1 M LiClO₄. Ni depositions were performed in an aqueous solution with 0.01 M Ni (SO₄)₂-Fluka, ≥ 99.0%), 0.227 mM NiCl₂ (Merck, ≥ 97.0%) and 0.025 M H₂BO₃ (≥ 99.0%). Toluene was purchased from Merck (Uvasol for spectroscopy). All chemicals were used without any further purification. All electrochemical experiments were conducted with a PAR VersaSTAT 3 potentiosat. The experiments performed in bulk and the depositions were recorded versus a saturated Ag|AgCl reference electrode with a Pt auxiliary electrode in a conventional undivided cell. The EPR spectra were recorded at X-band (Frequencies specified in captions) in continuous wave mode at ambient temperature with a 0.1 mT modulation amplitude and 100 kHz modulation frequency, 5.12 ms time constant, sweep time of 10.49 s, and a 0.2 mW and 0.8 mW power for benzoquinone and methyl violet, respectively. The Q-factors reported were determined by the built-in Q indicator of the Xepr programme. TEMPO (2,2,6,6-tetramethyl-1-piperidinyloxy, Sigma-Aldrich, 98%) spin-count calibration was performed in the flat cell for concentrations ranging from 0.5 mM to 0.025 mM. At a power of 0.2 mW, sweep time of 10.49 s, and with a time constant of 5.12 s. The integral of the absorption curve was calculated considering that every molecule of TEMPO is EPR active. The same method and settings were used to determine the spin concentration of BQ and MV radicals generated on the custom made ITO electrode.

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Keywords: Electrocatalyst screening • EPR spectroscopy • Detection of intermediates • Transparent conductive oxide electrode • Cyclic Voltammetry

References

ARTICLE


A novel electrode design for combined in-situ electrochemical electron paramagnetic resonance experiments is presented. The use of a transparent Indium Tin Oxide on PET semiconductor as a substrate for electrocatalyst deposition and in-situ investigation of radical intermediates provides unique opportunities in catalyst research. The electrode approaches the maximum available surface area in the electrolytic flat cell resulting in superior sensitivity.

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