



Application Note 5 Using Combined Electrochemistry and SPR to Monitor the Electropolymerization

Conducting polymer thin films have been the focus of increased investigation because of their importance to a variety of applications including displays, batteries and sensors. Polyaniline (PAn) is a conducting polymer that has been widely studied because of its unique properties. These features include its relative ease of synthesis, remarkable environmental chemical stability, and the drastic change in its electrical conductivity upon simple oxidation and reduction.¹ Owing to the SR7000DC's open architecture, Reichert offers specialized flow cells/wells for performing other measurements in combination with surface plasmon resonance (SPR). This application note presents the combination of electrochemistry and SPR to monitor the growth of a PAn film on the surface of bare gold with the Reichert SR7000DC system. Specifically, cyclic voltammetry (CV) is used as the deposition method while SPR simultaneously monitors the growth of the PAn film in real time.

Experimental

The CV/SPR experiments were carried out utilizing a three-electrode system with the gold surface as the working electrode, a platinum wire as the counter electrode and a Ag/AgCl reference electrode. The potential was cycled between -0.2 and 0.8 V 10 times and the SPR baseline was established for at least 4 min before the next set of potential cycles. 6 total sets of cycles were carried out including 2 blank cycle sets before the aniline injection. The experimental conditions for this study are summarized in the following Table:

Aniline Concentration	Running Solution	CV Scan Rate	CV Scan Potential Range
0.1 M	0.5 M H ₂ SO ₄	100 mV/s	-0.2 to 0.8 V

Results

Figure 1 presents the cyclic voltammograms during the electropolymerization of aniline after potential sweeps. The results reflect the smooth growth of the PAn film with each successive potential cycle. The set of redox peaks occurring at ~ 0.2 V in the positive scan and ~ 0.05 V in the negative scan correspond to the electron transfer from/to the deposited PAn film. The large current change at > 0.75 V has been attributed to the oxidation of the aniline monomer generating a precursor for the PAn film.²

Figure 2 presents the change in SPR response during the electropolymerization of aniline. Sequential oscillations of the SPR signal correspond to the cycling potential at the gold surface. After the aniline is injected, the SPR response increases, indicating growth of the PAn film on the gold surface. The inset in Figure 2 displays the reflectivity data (reflectivity intensity vs. angle) taken after each set of 10 potential cycles. The data indicates that the SPR minimum shifts to higher angles due to an increase in PAn film thickness.

¹ Guiseppi-Ellie, A.; Pradham, S.R.; Wilson, A.M. *Chem. Mater.* **1993**, 5, 1474.

² Baba, A. et al. *J. Electroanal. Chem.* **2004**, 562, 95.

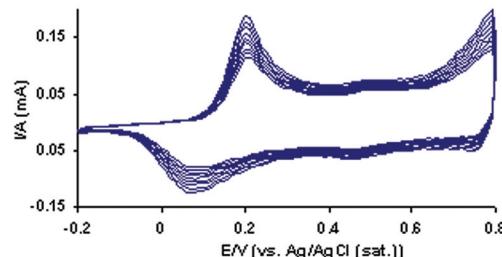


Figure 1: Cyclic voltammograms of the electropolymerization of aniline in 0.5 M H₂SO₄ on the gold electrode.

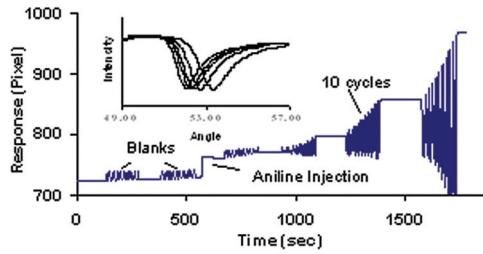


Figure 2: SPR response during the electropolymerization of aniline. The inset shows the SPR reflectivity data taken from each set of 10 potential cycles.

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