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CASE STUDY

CAPTURING A FULL SPECTROELECTRO-CHEMISTRY EXPERIMENT IN A SINGLE MEASUREMENT

INTRO

CAPTURING A FULL SPECTROELECTROCHEMISTRY EXPERIMENT IN A SINGLE MEASUREMENT

A team in the Department of Chemistry and Biochemistry at Auburn University, including Caitlyn Clark and Dr. Christopher Grieco, built an automated absorbance spectroelectrochemistry setup that cuts a slow, manual experiment down to a single run. At the core of the system: two Avantes spectrometers, the AvaSpec-ULS2048XL-EVO and the AvaSpec-NIR256-2.5-HSC.

THE CHALLENGE: WATCHING MATERIALS CHANGE COLOR IN REAL TIME

Spectroelectrochemistry combines electrochemistry and optical spectroscopy to reveal how materials change, based off their absorbance spectrum, in response to an applied voltage. Researchers use this technique to study a wide range of materials, including battery electrodes, electrochromic coatings, conductive polymers, metal complexes, and electrocatalysts. By monitoring changes in light absorption as these materials gain or lose electrons, researchers can track chemical reactions in real time and gain insight into their electrochemical properties.

It's especially useful for electronic materials like mixed ionic-electronic conducting polymers, which are a big focus in areas like organic electronics and energy storage.

The standard chronoamperometry approach works like this:

- Hold the sample at one fixed voltage
- Wait until it settles into equilibrium
- Record a spectrum
- Pick the next voltage and repeat, across the whole electrochemical window

This method works, but it’s slow and labor intensive. And each spectrum is typically recorded after the sample stabilizes, so changes that occur in between measurements can be missed. There’s no time-resolved view of the material in motion. The result? Researchers get snapshots of the material at different voltages rather than a continuous, time-resolved view of how it changes.

THE SOLUTION: AUTOMATED ABSORBANCE SPECTROELECTROCHEMISTRY SYSTEM

The team set out to automate the whole thing. They paired a high-speed, broadband optical absorbance instrument with normal pulse voltammetry, so the voltage steps and the spectral measurements happen together in one automated run.

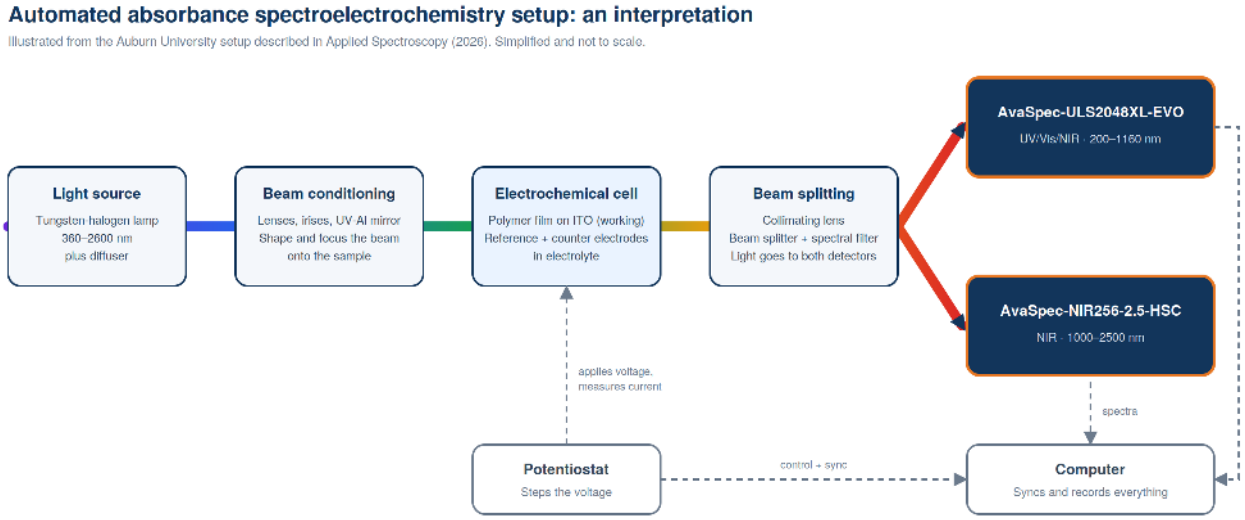
The setup covers 400 to 2500 nm, visible through near-infrared, and is capable of up to 200 spectra per second (a 5 ms response time). This demo ran at 1 spectrum per second, so there's plenty of headroom.

That range matters. Conducting polymers show important features across both the visible and the near-infrared, and the near-IR region is where charge carrier signatures often show up. Covering all of it in one shot means nothing gets missed.

Two Avantes spectrometers do the work: the AvaSpec-ULS2048XL-EVO (since replaced by the newer AvaSpec-ULS2048XL+) and the AvaSpec-NIR256-2.5-HSC. Between them they cover the full 400 to 2500 nm window

The rest of the setup:

- **Light source:** a stabilized tungsten-halogen lamp
- **Optics:** free space, with lenses routing the beams. Fiber optics would work too, and Dr. Grieco says the measurement should come out the same.
- **Cell:** a custom-assembled cell with a 2 mm path length. Standard 1 cm electrochemical cells should also work, though near-infrared transmission will be lower.



THE TEST CASE: A POLYTHIOPHENE ELECTRODE

To prove the method, the team used it to study the electrochemical charging of a P3HT polymer film, a well-known polythiophene. Polythiophenes are a well-known family of conducting polymers, so they make a good benchmark for a new technique.

Instead of building the data set one voltage at a time, the system automatically collected:

- A full set of potential-dependent absorbance spectra
- Absorbance transients, showing how the signal changes over time at each step

In this demo, the measurements were time-resolved at 1 spectrum per second.
The details: a P3HT film on an ITO electrode, sitting in a lithium perchlorate electrolyte (0.5 M in propylene carbonate). The team stepped the voltage up in pulses and recorded spectra the whole way.

What they saw as the voltage went up:

- The absorption peak for neutral polymer chains (around 2.35 eV) shrank as the film was oxidized, a process called electrochemical doping.
- Two new bands appeared in the near-infrared, around 0.7 eV and 1.5 eV. Those come from charge carriers (polarons) building up in the film.
- Nothing changed below 0.3 V, so the action happened between 0.3 V and 0.95 V (vs Ag/AgCl).

WHY THE SPECTROMETER MATTERED

This kind of experiment only works if the detector can keep up. The voltage is changing, the material is responding, and the instrument has to capture full spectra fast enough to follow along, without giving up spectral range or quality. Fast acquisition and broadband coverage are the whole point here.

> *"The ability to synchronize measurements between both Avantes spectrometers covering such a wide spectral range, combined with their sensitivity and rapid spectral acquisition rates, were crucial for capturing the full absorption spectra of our polymer films during electrochemical charging and discharging."*
>
> **-Dr. Christopher Grieco, Department of Chemistry and Biochemistry, Auburn University**

THE RESULTS: MORE DATA, LESS HANDS-ON TIME

The big win is that one automated measurement captures what used to take a whole series of separate experiments. In practice, that means:

- **Faster data collection.** The full potential sweep happens in a single run.
- **Less manual work.** No stepping through voltages by hand and waiting for each one to settle.
- **Time-resolved information.** The transients show how the material responds as it charges, not just where it ends up.

Beyond speed, the data told a richer story:

- **Built-in proof of equilibrium.** Spectra are captured continuously, so the team could check that the signal flattened out at the end of every voltage step before trusting that spectrum. If it hadn't, they'd rerun with longer step times.
- **Charging vs. discharging.** The film took longer to settle when charging than when discharging, which you can't see with equilibrium-only measurements.
- **Bipolarons.** Taking derivatives of the spectra with respect to voltage sharpened the picture. At the highest voltages (above roughly 0.775 V), a new signal near 1 eV showed up. That's evidence of bipolarons (doubly charged species) that are hard to spot in standard spectra.
- **A clean-up trick.** The polymer-on-ITO interface created an oscillating baseline in the near-IR. Subtracting the baseline spectrum removed it and made the absorption bands easier to read.

WHO SHOULD CARE

The authors note the method could also work on polyelectrolytes and photoelectrodes, and could be adapted to capture fluorescence instead of absorbance. It's relevant for anyone studying how materials respond to an applied voltage:

- Conducting and mixed ionic-electronic polymers
- Organic electronics and electrochromic materials
- Battery and energy storage materials
- Anyone looking to speed up electrochemical characterization

LEARN MORE

Read the Full Paper: "Automating Absorbance Spectroelectrochemistry Using Normal Pulse Voltammetry", Applied Spectroscopy, 2026. Read it here: https://journals.sagepub.com/doi/10.1177/00037028261479001

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