Synthesis of a Sonogel-Carbon Modified Sensor Electrode with Titanium Oxide (TiO_2) to Detect Catechol in the Presence of Common Interferent by Voltammetric Studies

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Abstract: This experiment was part of an Analytical/Instrumental Analysis course. It requires the synthesis of a sonogel-carbon electrode (SGC) modified with a TiO_2 sol-gel and heated at high temperatures. The electrochemical response of the synthesized SGC/TiO_2 electrode was compared to that of an unmodified sonogel-carbon electrode to detect catechol (catecholamines). The design of the experiment encourages some choices to be made by the student, although the sol-gel syntheses are recipe driven. The students were required to determine if the modified electrode showed marked enhanced detection of catechol in the presence of ascorbic acid, compared to an electrochemically prepared conducting poly (3-methylthiophene) electrode in the detection of catechol over several scans. The students gain electrochemical instrumentation skills and implement them by studying reduction-oxidation states of catechol in the presence of ascorbic acid.

Introduction

One of the first reactions one learns in general chemistry, inorganic chemistry, and organic chemistry are the oxidation and reduction reactions. These types of reactions are very important to biological molecules such as catecholamines. Catecholamines are a class of neurotransmitters (1,2- dihydroxybenzenes) that are involved in a wide variety of physiological processes [1-6]. These classes of neurotransmitters are secreted in the brain and altered levels have been associated with mental and behavioral disorders such as schizophrenia, attention deficient disorder, Alzheimer's disease, Parkinson's disease, eating disorders, epilepsy, amphetamine addiction, and cocaine addiction [4]. Voltammetric detection of catecholamines is affected by the presence of interferents such as ascorbic acid [7]. The ability to enhance catecholamine selectivity is of notable interest within electroanalytical research. Various techniques have been used previously [8-18], with modified electrodes becoming the most prevalent technique [19-22]. The rate and selectivity of an electrode can be controlled by deliberately modifying its surface chemically. The concept of electrode modification originated over three decades ago with work of several research groups [1-2, 22].

Chemical modification of the electrode surface with electroactive polymeric films, has gained wide popularity in the past due to the simplicity of altering the electrode surface [3]. Further, polymer films introduce additional active sites allowing electrochemical processes at their surfaces to be more pronounced than the electrochemical processes at unmodified surfaces. Polythiophenes stand out among the numerous research projects done on electrochemically conducting polymers. This attention has been focused on polythiophenes due to their process ability, environmental stability, thermal stability, and ease of fabrication [23]. In spite of this, during the course of developing polymeric films as chemical sensors, there have been hurdles

impeding successful analysis of clinical and environmental samples. Within the developmental stages of the electrode lie the need to improve its stability and selectivity of clinical and environmental samples [3]. Vandaveer et al. have utilized redox cycling (cyclic voltgammetry) measurements of neurotransmitters in ultrasmall volumes with a self-contained microcavity device that showed improved response and sensitivity [24-25]. However, optimization needs to occur of the microcavity device to make it applicable to clinical practices to achieve the desirable detection limits of neurotransmitter (dopamine) in the presence of ascorbic acid without the complications encountered by Niwa et al. [22].

This lab experiment creates a challenging opportunity for the student. It requires the synthesis of a sonogel-carbon electrode (SGC, SG=Sonogel, C=Carbon) modified with nanostructured titanium oxide, and comparison to a bare SGC electrode (no titanium oxide on the bare sonogel carbon electrode) and a poly (3-methylthiophene) (P3MT) modified electrode. Their responses to detect catechol in the presence of common interferents were studied by cyclic voltammetry [26-27]. This experiment was designed to fit into three lab sessions where each session was four hours long. This lab was created for Project REEL, R=Research, E= Experiences, E=Enhance, and L= Learning (Research Experiences to Enhance Learning) and a pre-test and post-test were given to evaluate the students' gain in content from this lab experiment. All the figures shared in this article are student data obtained from Project REEL.

Experimental

Safety/Hazards

All solution preparations and syntheses were carried out under a fume hood. Catechol is harmful upon contact with tissue or if ingested. Acetonitrile is toxic or fatal by inhalation, ingestion or skin absorption. Tetrabuytlammonium tetrafloroborate is an irritant to eyes, the

respiratory system and the skin. The compound 3-methylthiophene is highly flammable, and harmful through inhalation.

Upon mixing concentrated sulfuric acid with water (Corning Mega Pure system) and hydrochloric acid, heat evolves so precaution was taken in this mixing step. All waste was disposed of in the proper waste container with labels. Protective garment and gloves were worn at all times. For preparation of the modified sonogel-carbon titanium oxide electrode, all steps were carried out under a hood and care was taken not to inhale the methyltrimethoxysilane (MTMOS) or graphite powder. All MSDS information can be found at www.sigmaaldrich.com for all products.

Construction of the modified electrodes

The sonogel-carbon electrode was prepared by first creating a carbon mixture to be packed in a small glass capillary tube. The carbon material was manually packed into the glass tube by pushing the glass tube into the thick carbon layer. The methyltrimethoxysilane (MTMOS, Fluka) and 0.2 M HCl (A.C.S. Reagent by Fisher) were combined in a vial, and the mixture was ultrasonicated (2510R-DH, Bransonic) in a 40 mL borosilicate glass vial with cap for 15 seconds. Graphite powder (Alfa Aesar 99.9%, 2-15 micrometer) was added and homogenously dispersed. Prior to placing the mixture of 500 μL MTMOS, 100 μL 0.2 M HCl and graphite, into a capillary tube (Sutter Instrument, 0.69 mm I.D., 1.2 mm O.D., 10 cm length, borosilicate glass both ends open), a 0.25 mm copper wire was inserted to serve as an electrical contact, the copper wire (Alfa Aesar) was coated with thin insulator, but removal is not necessary since the insulator layer was removed during the 500^{o}C heating process in air. The titania precursor titanium tetraisopropyloxide (TTIP, Aldrich) material was prepared as follows: Tween 80 (Aldrich) 2.62 grams was dissolved in isopropanol (99%, Fisher), followed by the addition 0.68

mL of concentrated acetic acid (Aldrich) and 0.61 mL of titanium tetraisopropyloxide under vigorous stirring. The capillary tube containing the carbon material was dip coated with the titanium oxide (Aldrich) material. After coating, the capillary tubes were heated to 500^{o}C for 20 minutes [5], and cooled naturally.

Electropolymerization was carried out in a one compartment cell, illustrated in Figure 1, that contained 0.05 M 3-methylthiophene (P3MT, Aldrich), 0.1 M tetrabutylammonium tetrafluoroborate (Fisher Scientific), and acetonitrile (HPLC grade, Aldrich). The P3MT was grown on a sonogel-carbon electrode at a potential range of -0.2 V to+1.7 V for 5 cycles. All cyclic voltammograms were carried out in 10 mM sulfuric acid (A.C.S. Reagent by Fisher Scientific).

Voltammetric Studies

All electrochemical measurements were carried out on a Bioanalytical Systems Epsilon, in a single compartment cell as displayed in Figure 1, at room temperature. The auxiliary electrode was a platinum wire, and an Ag/AgCl/3 M NaCl electrode was used as the reference electrode, and the composited filled capillary tubes dipped in TiO_2 sol-gel and electrodeposited with P3MT were used as the working electrodes. The Catechol (99% HPLC grade) and ascorbic acid (99% HPLC grade) were all purchased from Aldrich. All solutions such as catechol and ascorbic acid were prepared with Corning Mega Pure Water. Cyclic voltammetry at a scan rate of 100 mV/s was the electrochemical technique applied to study the behavior of the modified electrodes.

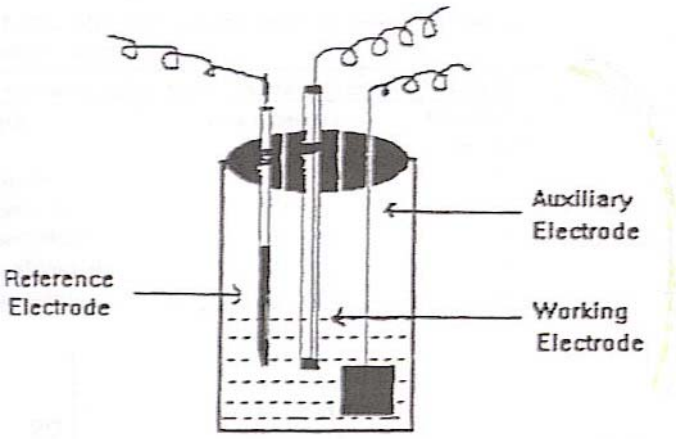

Figure 1. Schematic diagram of three-electrode compartment single compartment cell.

Results and Discussion

The cyclic voltammograms in Figures 2(a), illustrate the selectivity principle in the electrochemical detection of catechol in the presence of ascorbic acid. Ascorbic acid illustrates irreversible behavior and catechol illustrates reversible behavior. As mentioned previously, the detection of catechol can be effected by the presence of ascorbic acid. The tailor-designed porous structure facilitated the electrochemical response to catechol in the presence of ascorbic acid. Figure 2(a), (Blue Line) shows clearly that the SGC/TiO_2 electrode was able to electrocatalyze the oxidation of catechol. The oxidation peaks of catechol and ascorbic acid are being resolved at different potentials and the reduction peak of the catechol oxidized in the forward sweep is detected. However, the cyclic voltammogram at the bare sonogel-carbon electrode with no titanium oxide as designated in Figure 2(a), (Red Line) illustrates that the oxidation peaks of catechol and ascorbic acid are not resolved, and the reduction and oxidation peaks for catechol are not detectable.

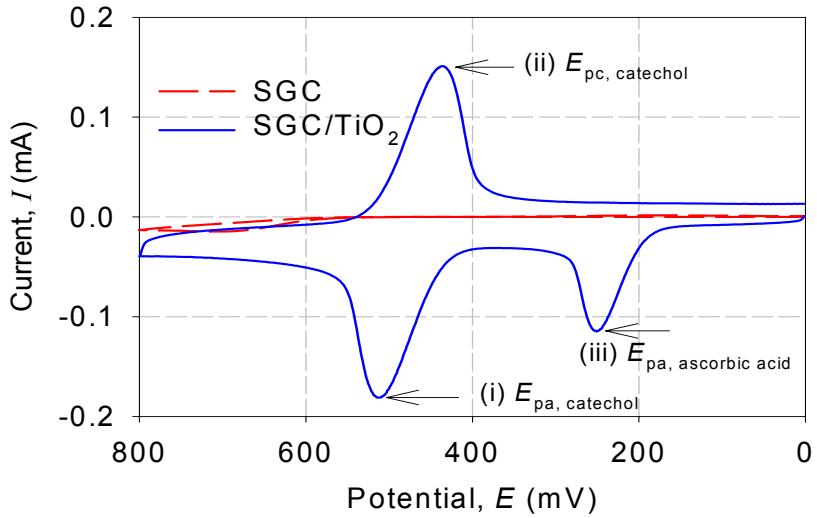

Figure 2 (a): Cyclic voltammogram of 5mM catechol +5mM ascorbic acid in 10 mM H_2SO_4 (100 mV/s) vs Ag/AgCl /3 M NaCl, on bare sonogel-carbon electrode (Bare SGC, Red Line) and compare to (SGC-TiO_2, Blue Line) modified electrode [current (mA) vs. potential (mV)].

A similar study by coworkers has been conducted in the past for the detection of catechol in the presence of ascorbic acid with a modified poly (3-methylthiophene) electrode (P3MT) [4]. Like the SGC-TiO_2 electrode, the P3MT electrode has indicated the reversibility of catechol oxidation is significantly improved compared with the bare sonogel-carbon (SGC) electrode. Studies have shown that catechol and ascorbic acid can be difficult to distinguish in a cyclic voltammogram (refer to lab appendix for example of student data) with a P3MT electrode and there is deterioration of the P3MT electrode over time [4, 26]. The problem found with the P3MT modified electrode is poor stability and electrode fouling. The SGC-TiO_2 electrode was found to be more stable than the P3MT modified electrode after repeated sweeping cycles. In Figure 2(b), the stability of the SGC/TiO_2 electrode is compared to the P3MT modified electrode in the detection of catechol over 20 cycles. As shown in Figure 2(b), the SGC/TiO_2 electrode did not show any change when it was swept repetitively, indicating that the TiO_2 sol-gel has a good adherence to the sonogel-carbon electrode surface and this electrode has a good stability

over 20 cycles. Figure 2(b), the P3MT modified electrode (Red Line) shows instability over 20 cycles. The cyclic voltammogram for the P3MT modified electrode shows that as the number of cycles increase, the anodic and cathodic peak currents vary. This indicates that the P3MT adherence to the sonogel-carbon electrode surface weakens as the number cycles increases, exhibiting instability. Another sign that the P3MT lacked stability was the result of black P3MT particles falling off the electrode into the sulfuric acid solution during the 20 cycles. There was a problem with adherence of the P3MT (polymer) over several scans.

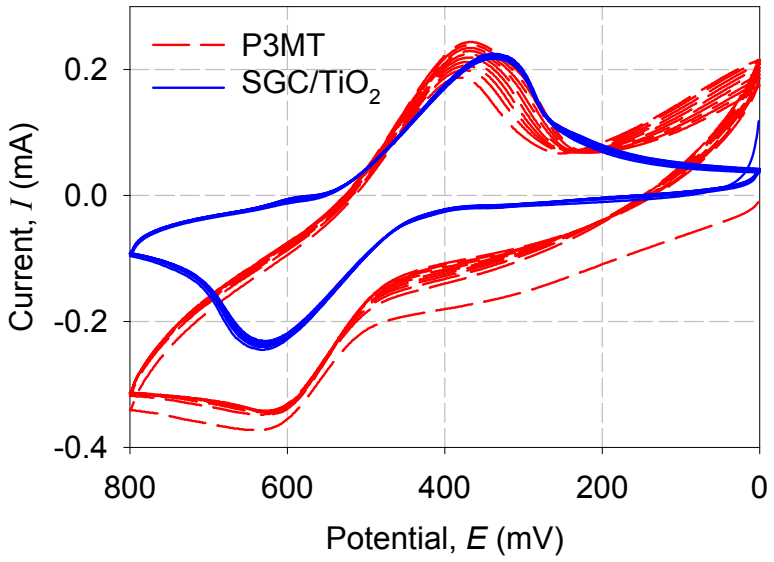

Figure 2(b): Comparison of the stability of SGC/TiO_2 electrode (Blue Line) and P3MT modified electrode (Red Line) in the detection of 5 mM catechol in 10 mM H_2SO_4 (100 mV/S) over 20 scans [current (mA) vs. potential (mV)].

The modified SGC/TiO_2 electrode shows improved electrocatalysis and improved selectivity towards catechol compared to the bare SGC electrode and the modified P3MT electrode. Profitable features that students learn about the advantages of the SGC/TiO_2 synthesized electrode include relative chemical inertness, good mechanical properties, physical rigidity, stability, and enhanced catalytic properties for the nanostructured modified titanium

oxide electrode. {Note the results shown in Figure 2(a), and Figure 2(b), are examples of actual student data and illustrate the inconsistent peak splitting for catechol can be due to not obtaining reproducible sol-gel synthesis results of modified electrodes}

Although the syntheses of the modified electrode surfaces are given to the students in recipe format, the investigation of the oxidation and reduction reactions of catechol in the presence of ascorbic acid is left open. The student must determine from the electroanalytical results which sensor would be the best sensor to detect catecholamines in the presence of interferents. A justification of this choice is an expectation in the lab report. The emphasis on grading of this lab can be placed on the student's analysis of the oxidation and reduction reactions for catechol (reversible behavior, oxidation and reduction peaks) and ascorbic acid (irreversible behavior, oxidation peak).

The cyclic voltammogram data presented are student results. Our experience has shown that a degree of student independence is welcomed at the level that this lab is presented. The experiment can be expanded to require students to create an optimized sensor that is applicable for real world analysis at pH of 7.4. However, due to the extra time needed to prepare phosphate buffer and the pH adjustments that are needed to prepare each solution to keep a physiological pH of 7.4, sulfuric acid was employed in this experiment due to the limited lab time at our university. Students typically have difficulty making solutions at the undergraduate level and adjusting pH can be tough in a limited time period. Additional interferents that may be studied to expand the lab could be acetaminophen, ascorbic acid and uric acid in the presence of catechol and other catecholamines (L-Dopa, dopamine, serotonin, etc.) by cyclic voltammetry. Salimi et al. discuss the need for simultaneous determination of ascorbic acid, acetaminophen, uric acid and neurotransmitters with a carbon electrode prepared by the sol-gel technique [28].

Simultaneous detection of neurotransmitters, ascorbic acid, and uric acid are oxidized at nearly the same potential with poor sensitivity at solid electrodes, resulting in overlapped voltammetric responses. Simultaneous detection is a problem of critical importance not only in the field of biomedical chemistry and neurochemistry but also diagnostic and pathological research. For the instructor, the possibilities for modification are abundant, and our hope is that ideas presented here are the seeds for many more. See the Lab Appendix for pre- or post-test questions and suggested readings to assist students with the experiment.

Acknowledgements

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Lab Appendix

Introduction/Purpose: Cyclic voltammetry is one of the most effective electroanalytical methods for the elucidation of the mechanism of the electrode processes. Catechol is an important neurotransmitter secreted in the brain and controls locomotion. Many mental and behavioral disorders such as Alzheimer's disease, Parkinson's disease, eating disorders, epilepsy, amphetamine addiction and cocaine addiction have been associated with altered levels of these neurotransmitters such as catechol in the brain. The goal of this lab is to develop two different types of electrochemical sensors; 1) a poly (3-methylthiophene) sonogel-carbon electrode and, 2) a sonogel-carbon titanium oxide (SGC/TiO_2) to detect catechol by cyclic voltammetry in the presence of a common interferent ascorbic acid. Determine which modified sensor electrode developed would be the best to detect neurotransmitters from the cyclic voltammogram results.

References/Supplemental Readings Suggested:

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Apparatus/Equipment: Instrument for cyclic voltammetry such as (Bioanalytical Systems, Epsilon model), sonogel-carbon electrode (SGC-TiO_2, P3MT) , platinum auxillary electrode, Ag/AgCl/3 M NaCl reference electrode, volumetric flasks (10 mL, 50 mL, 100 mL, 1000mL), disposable pipets, pipet bulbs, analytical balances to weigh samples. Ultrasonicator instrument

such as Bransonic. Copper wire 0.25 mm, capillary glass tube (0.69mm inner diameter) for the SGC electrode. Oven/furnace (Thermcraft) to heat sonogel-carbon TiO_2 electrode.

Chemicals: Catechol, ascorbic acid, tetrabutylammonium tetrafluoroborate, acetonitirle, sulfuric acid, hydrochloric acid, 3-methylthiophene, acetone(clean glassware), methyltrimethoxysilane, graphite carbon powder (99%), polyoxyethylenesorbitan monooleate (Tween 80), isopropanol, acetic acid, titanium tetraisopropoxide. Refer to MSDS sheets in the laboratory.

Experimental:

Sonogel-Carbon Electrode

For the fabrication of sonogel-carbon electrode, 1.5 mL of methytrimethyoxysilane (MTMOS), should be added into 0.3 mL of 0.2 M HCl solution. The mixture should be ultrasonicated for 15 seconds. Then, add 3 grams of graphite carbon powder into the MTMOS solution and mix thoroughly for 10 minutes (condensing the SGC). Install a 0.25 mm copper wire inside of the 0.69 mm inner diameter glass tube to use as the body of the SGC electrode. The glass tube should then be filled with the SGC material.

Modification With the TiO_2

The TiO_2 should be synthesized via sol-gel method. Add the molar ratio of Tween-80: isopropanol:acetic acid:titanium tetraisopropoxide=1:45:6:1. The tip of the SGC should be dipped in the TiO_2 sol-gel for 3 seconds and then heated in an oven for 20 minutes at 500^{o}C. To have a control must make ten SGC electrode without TiO_2 (called a bare electrode) and SGC electrode modified with P3MT (based on the articles of suggested readings on P3MT).

Once made, there should be ten modified sonogel-carbon TiO_2 and ten bare sonogel-carbon electrodes. Prepare the 1 mM- 5 mM catechol diluted in 10 mM sulfuric acid and 1mM – 5mM ascorbic acid diluted in 10 mM sulfuric acid and an equal mixture of catechol and ascorbic acid (5 mM catechol + 5mM ascorbic acid). Also, prepare the 0.05 M 3-methythiophene solution diluted with 0.1 M tetrabutylammonium tetrafluoroborate diluted in acetonitrile. Read the Epsilon handout on cyclic voltammogram instrumentation before proceeding. Run all cyclic voltammograms at 100 mV/s. Check with instructor to make sure the data that will be carried out are correct to assist with the pre-post-test questions and if the range that the cyclic voltammograms are in a logical range as determined from the suggested readings (utilize suggested readings to assist with how to prepare a P3MT electrode). To compare stability of electrodes run the cyclic voltammograms (CV) for 20 scans. Run three trials to obtain reproducible results for each CV (catechol SGC/TiO_2, ascorbic acid SGC/TiO_2, catechol + ascorbic acid SGC/TiO_2, catechol + ascorbic acid bare electrode, catechol P3MT, ascorbic acid P3MT, catechol + ascorbic acid P3MT).

Pre- or Post-test Questions:

There are several concepts that students need to grasp in this experiment as outlined below, (all the suggested readings above will help students answer these questions below):

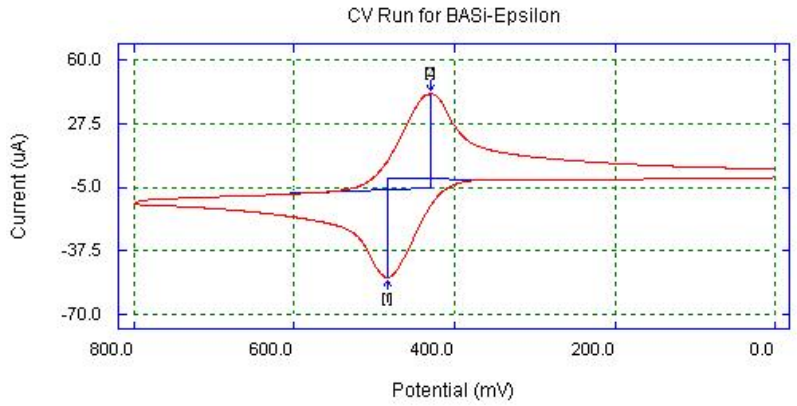
I. What is a cyclic voltammogram and how does one identify the reversibility of an electron transfer reaction by cyclic voltammetry? Students are required to read the references on cyclic voltammetry as noted above, (Kissinger et al., discuss the how to measure peak potential differences, and it illustrates examples of reversible, irreversible and quasireversible cyclic voltammograms). Kissinger et al. shows that

the irreversible cyclic voltammogram only has an anodic peak observed. For the reversible cyclic voltammogram shows the cathodic and an anodic peak observed. The peak potential difference between the anodic and cathodic peak relates to the supporting electrolyte effect on the cyclic voltammogram as described by Zhang et al. The P3MT electrode was shown by Zhang et al., that the smallest peak potential difference response to catechol was in sulfuric acid compared to a bare electrode. This assists the students with understanding electrocatalytic ability of P3MT electrode compared to a bare electrode and this helps the student with answering part II.

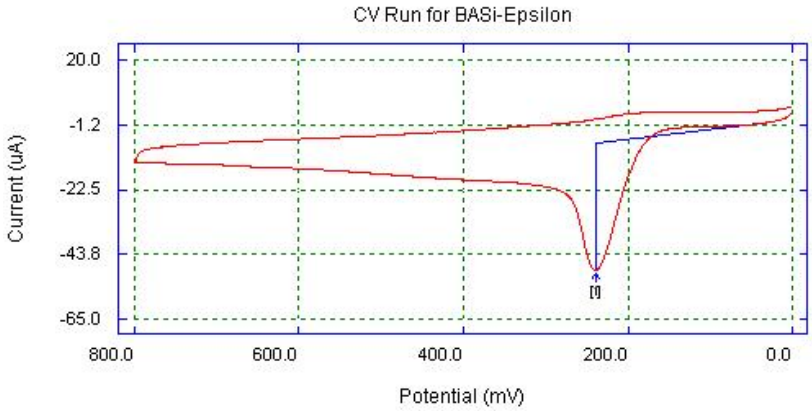
II. Why use the modified polymer electrode or the sonogel-carbon modified electrode with titanium oxide to detect catechol compared to the use of a bare carbon electrode? Catechol and ascorbic acid is detected at the modified electrode and no response of catechol and ascorbic acid at the bare sonogel-carbon electrode. The electron faster is easier and better at the modified electrodes P3MT and SGC/TiO_2 compared to the bare electrode.

III. Which electrode has the greatest stability the P3MT sonogel-carbon electrode or the SGC/TiO_2 electrode over 20 cyclic voltammogram scans to detect catechol in the presence of ascorbic acid? Students typically get results as cyclic voltammograms displayed by Figure 2(b)., above that illustrate the problems of poor stability over 20 scans. The P3MT black polymer falls into the sulfuric acid over several scans where the SGC/TiO_2 electrode can be used over and over without breaking down over time.

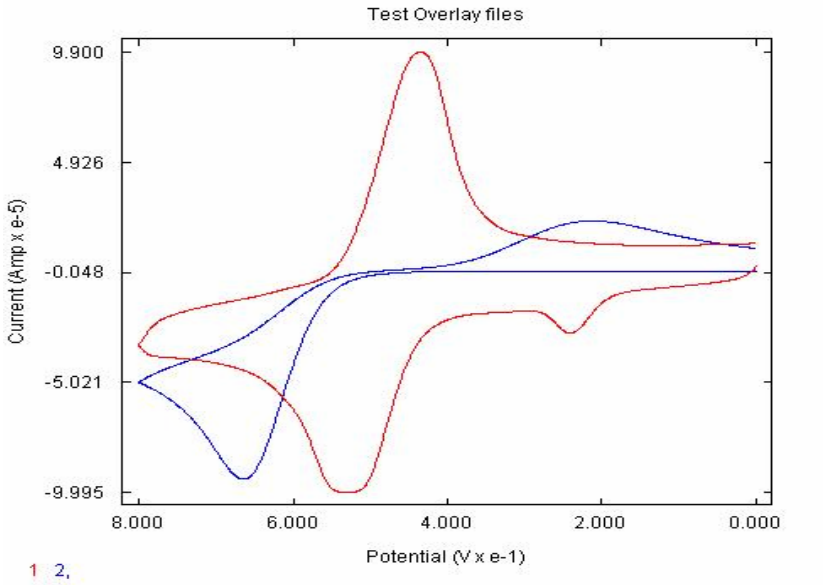
More examples of data compiled by students are shown below:



Cyclic voltammogram of 5mM catechol in 10 mM H_2SO_4 (100 mV/s) vs. Ag/AgCl /3 M NaCl, on SGC/TiO_2 modified electrode (Reversible catechol).



Cyclic voltammogram of 5 mM ascorbic acid in 10 mM H_2SO_4 (100 mV) vs. Ag/AgCl/3M NaCl on SGC/TiO_2 modified electrode (Irreversible ascorbic acid).



1 SGC/TiO_2 and 2 P3MT: cyclic voltammogram of 5 mM catechol + 5 mM ascorbic acid in 10 mM H_2SO_4 (100 mV/s). This data allows the student to see that the SGC/TiO_2 can distinguish there is ascorbic acid and catechol present where with the P3MT electrode there is no peak for ascorbic acid and the peak difference (cathode –anode) is much greater for catechol detection.