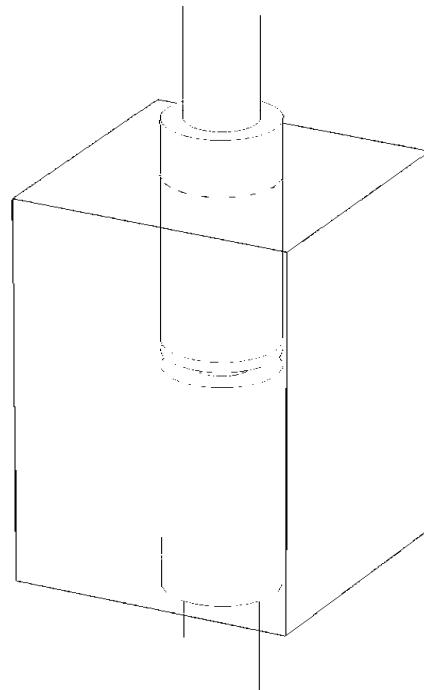


# Evaluation of Composite Electronic Materials Based on Poly (3, 4 – propylenedioxythiophene/Poly – (p – Naptheleneethynylene) Wrapped Single Wall Carbon Nanotubes for Supercapacitors



Johary Rivera Melendez  
University of Puerto Rico  
Rio Piedras Campus  
Chemistry  
SUNFEST 2010

SUNFEST 2010  
NSF REU Site

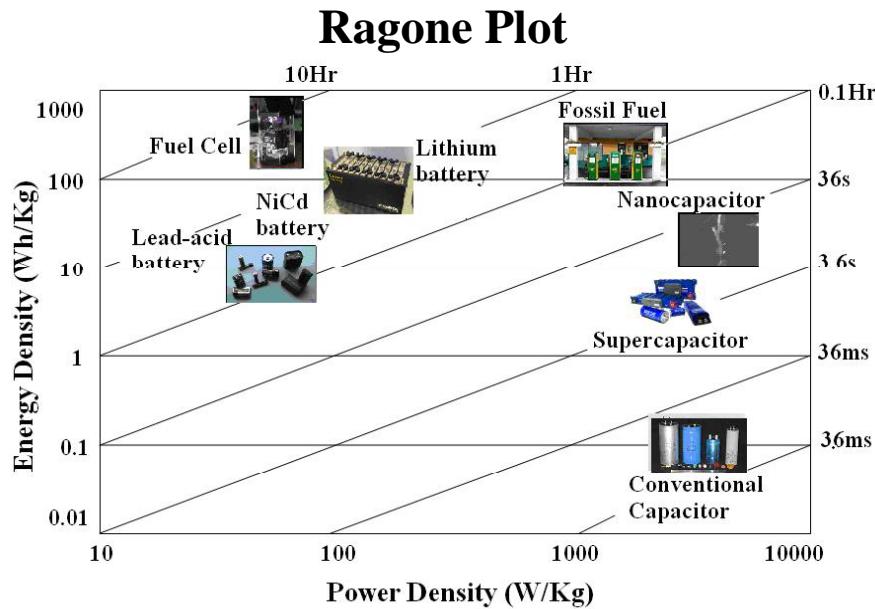


# **SUPER CAPACITOR**



$$C = \frac{Q}{V}$$

# Kinds of Energy Storage



## Advantages of Supercapacitors

- ❖ Power density
- ❖ Recycle density
- ❖ Environmental friendly
- ❖ Safe
- ❖ Light weight

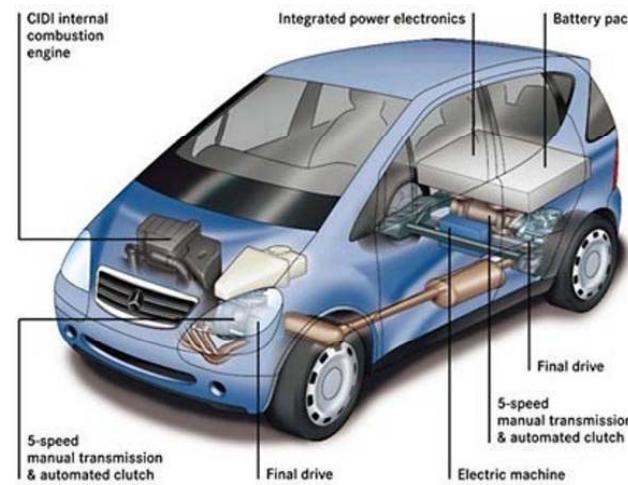
## Types of Supercapacitors

**Electrical Double Layer (EDLC)  
Redox (Pseudo capacitor)  
Hybrid**



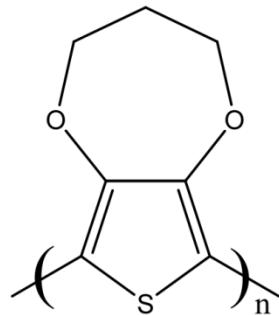
# Application for Supercapacitors

- ❖ Power back-up for memory functions in a wide range of consumer products such as mobile phones, laptops and radio tuners.
- ❖ To prolong battery life in products or devices using mechanical actuators such as digital cameras.
- ❖ Also used for energy storage for solar panels, and motor starters.
- ❖ Load Leveling



# Redox Capacitors

**Active materials:** metal oxides or electronically conducting polymers (ECPs)



**PProDOT**

## Advantages of ECPs

- ❖ Storage capacity
- ❖ Chemical stability
- ❖ Reduced cost relative to noble metal oxides
- ❖ ECPs can provide higher capacitance and/or higher power capability than other electrodes

Symmetric	Asymmetric
Type I: same p-doping polymer	Type II: different p-doping polymers
Type III: same polymer as the p- and n-dopable material	Type IV: different polymers as the p- and n-dopable material



# Experimental Procedure

---

- ❖ Solutions
- ❖ Electrochemical Polymerization
- ❖ Scan Rate Tests
- ❖ Oxidizing Tests
- ❖ Doping
- ❖ Morphological Characterization
- ❖ Construction of Device
- ❖ Single Wall Carbon Nanotubes



## Solutions

Electrochemical Polymerization

Scan Rate Tests

Oxidizing Tests

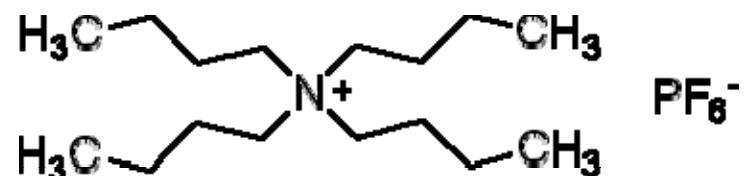
Doping

Morphological Characterization

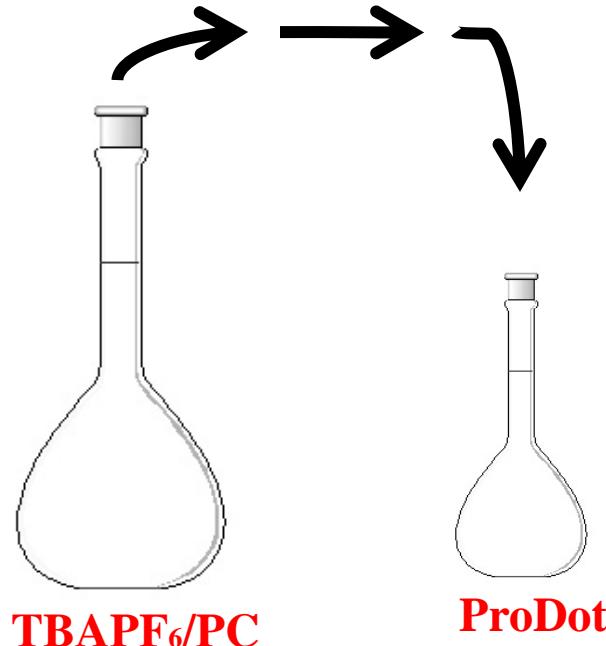
Construction of Device

Single Wall Carbon Nanotubes

❖ 25 mL TBAPF<sub>6</sub>/PC solution ~ 0.1 M



❖ 10 mL ProDot/TBAPF<sub>6</sub>/PC

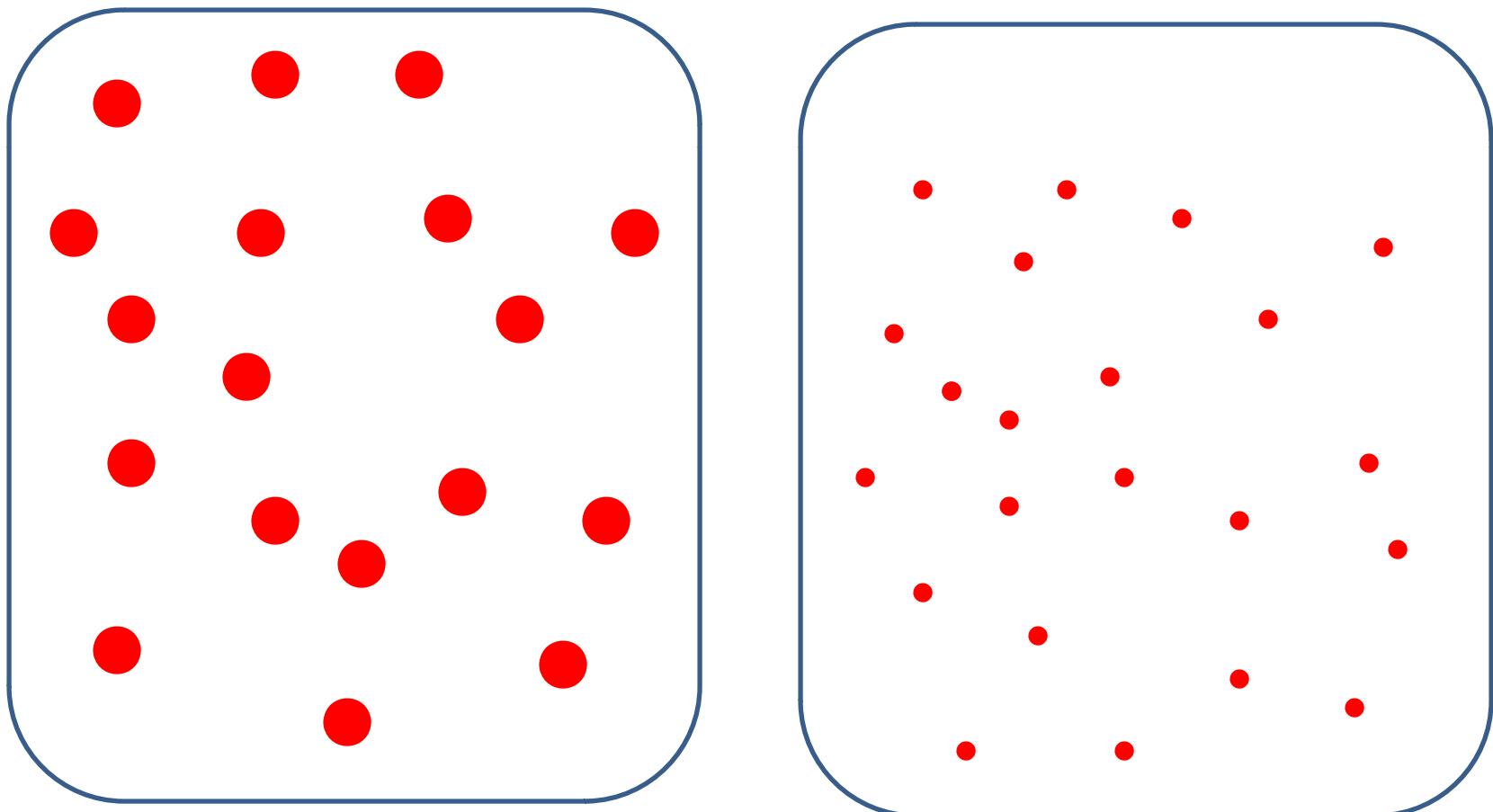


<http://www.sigmaaldrich.com>

<http://www.epa.gov/region3/ee/chesapeake/images/volum.gif>



# Why TBAPF<sub>6</sub>?



Solutions

Electrochemical Polymerization

Scan Rate Tests

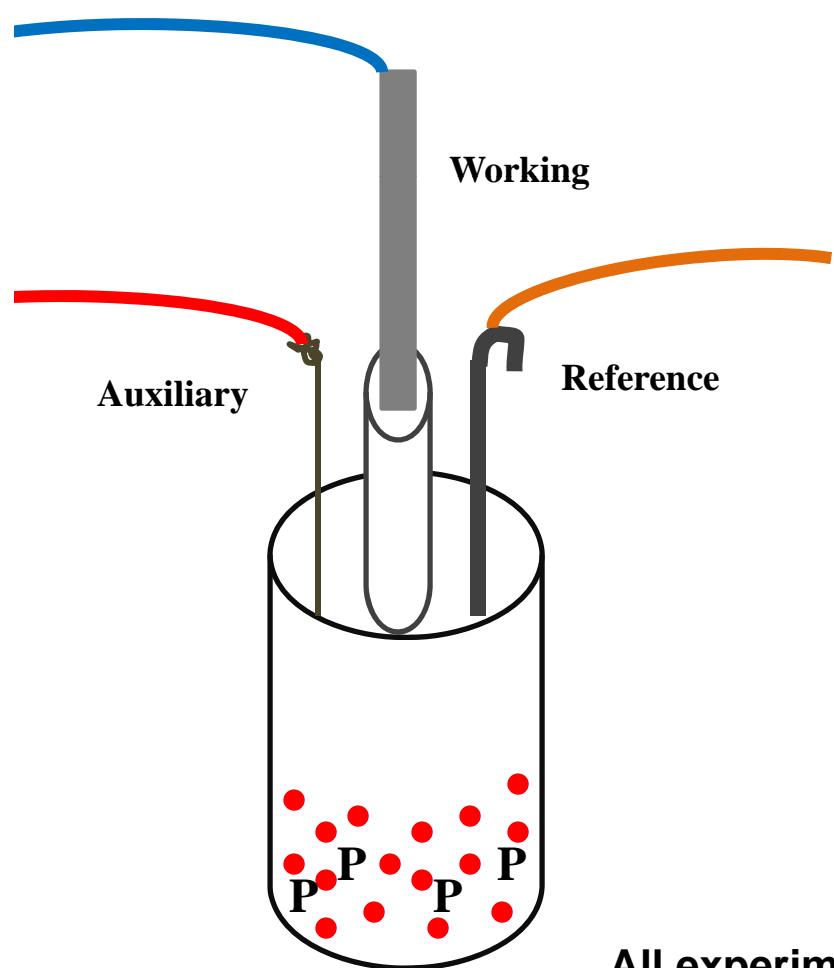
Oxidizing Tests

Doping

Morphological Characterization

Construction of Device

Single Wall Carbon Nanotubes



### Three electrode setup

- ❖ **Working electrode:** Platinum (5 mm disc OD)
- ❖ **Reference electrode:** Silver wire
- ❖ **Auxiliary /Counter electrode:** Platinum wire

### Parameters used

Sweep rate = 100 mV/s

Total sweep = 24

Sweep potentials = -1000 mV - 1600 mV

Color of deposition = deep purple

**All experiments were done in a glove box in N<sub>2</sub> atmosphere**

Solutions

Electrochemical Polymerization

Scan Rate Tests

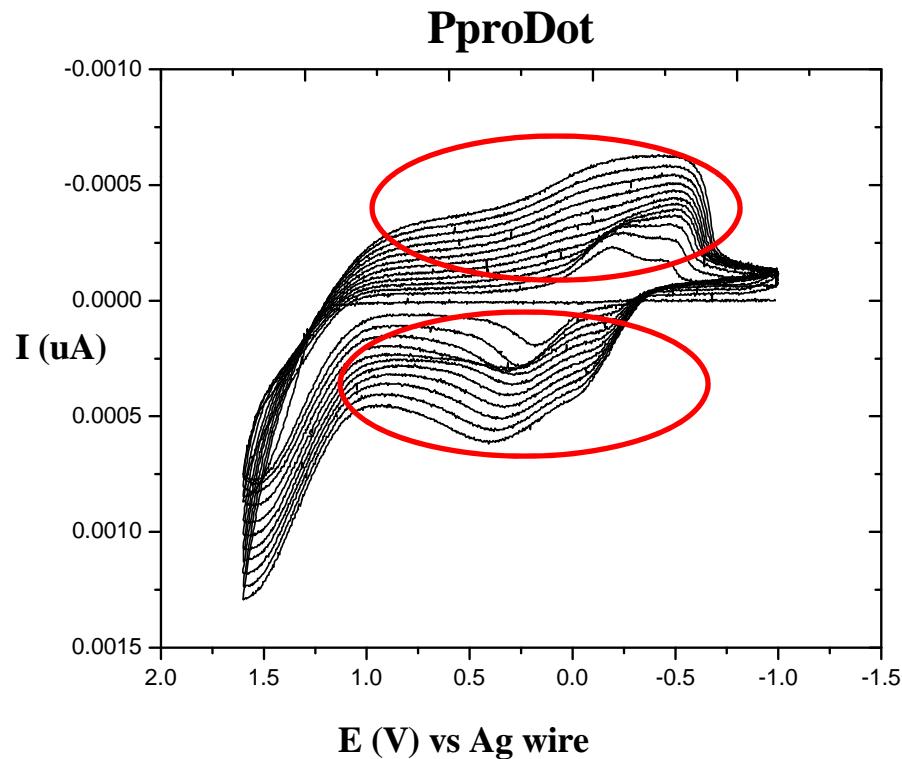
Oxidizing Tests

Doping

Morphological Characterization

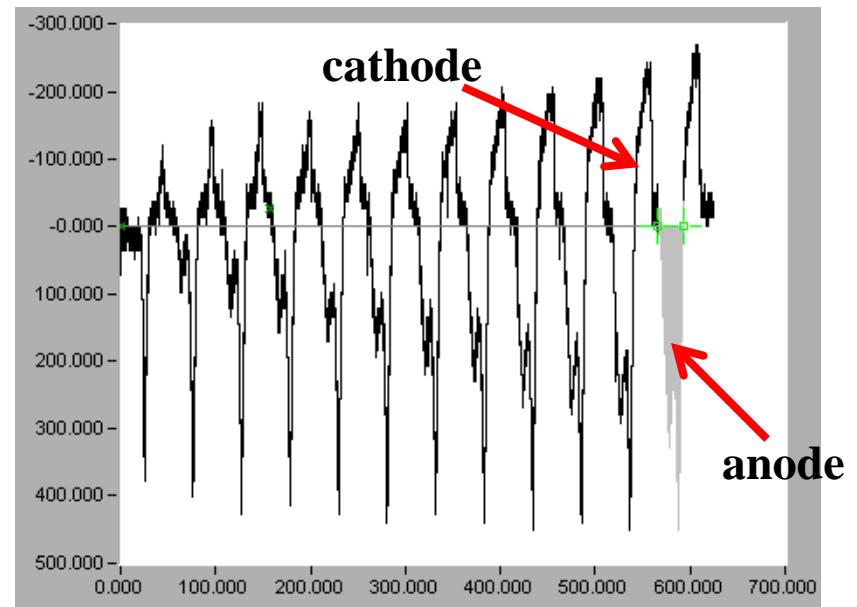
Construction of Device

Single Wall Carbon Nanotubes



CV of electropolymerization

Potential charges (mC) were determined for the anode and cathode.



Solutions

Electrochemical Polymerization

Scan Rate Tests

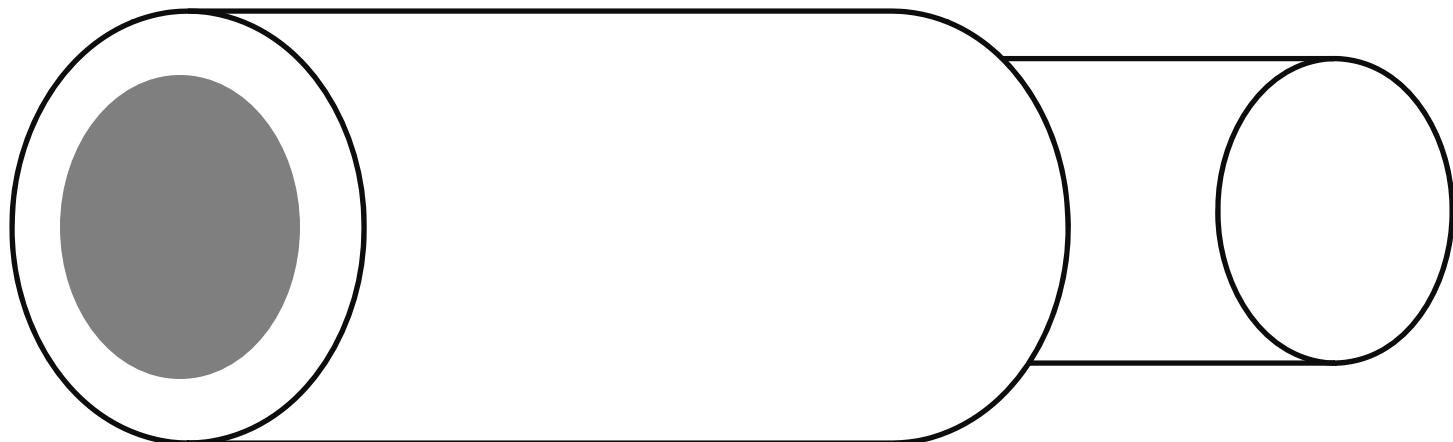
Oxidizing Tests

Doping

Morphological Characterization

Construction of Device

Single Wall Carbon Nanotubes



**Electropolymerization was done to two electrodes. If the difference in potential charges exceeded the 30% limit the electropolymerization process was repeated.**

Solutions  
Electrochemical Polymerization  
Scan Rate Tests  
Oxidizing Tests  
Doping  
Morphological Characterization  
Construction of Device  
Single Wall Carbon Nanotubes

## Scan rate tests are done primarily to know how fast the device will work

### Parameters used

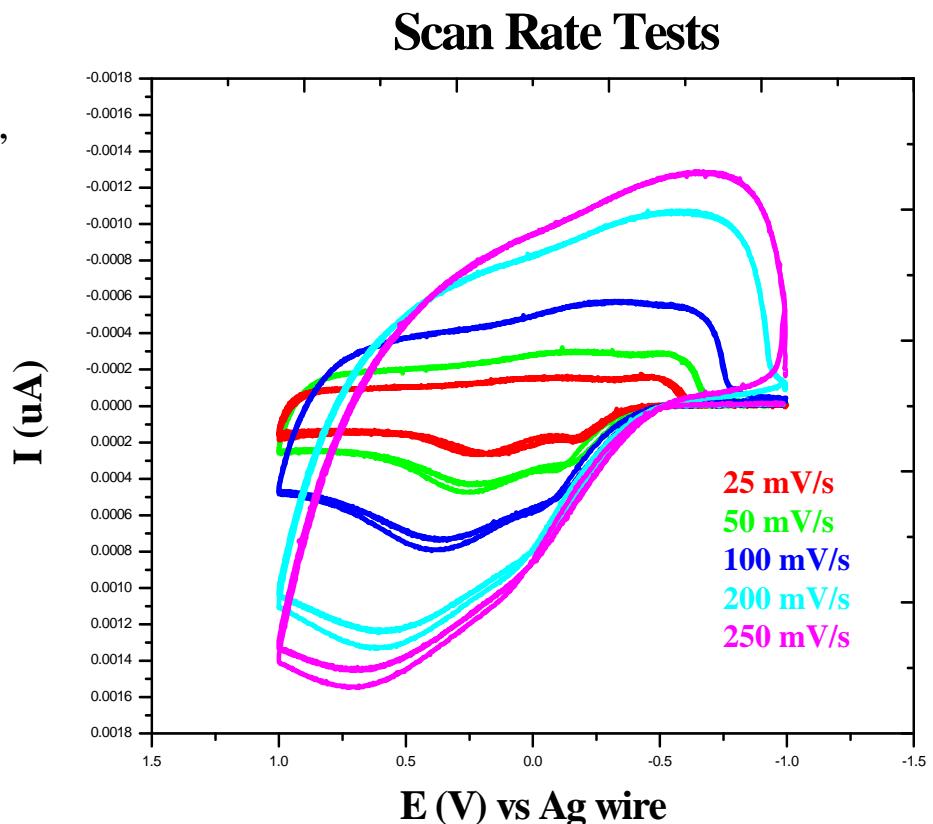
Sweep rates = 25, 50, 100, 200, 250, 300, 400, 500,  
750, 1000, 2500, 5000, 10000 mV/s

Total sweeps = 6

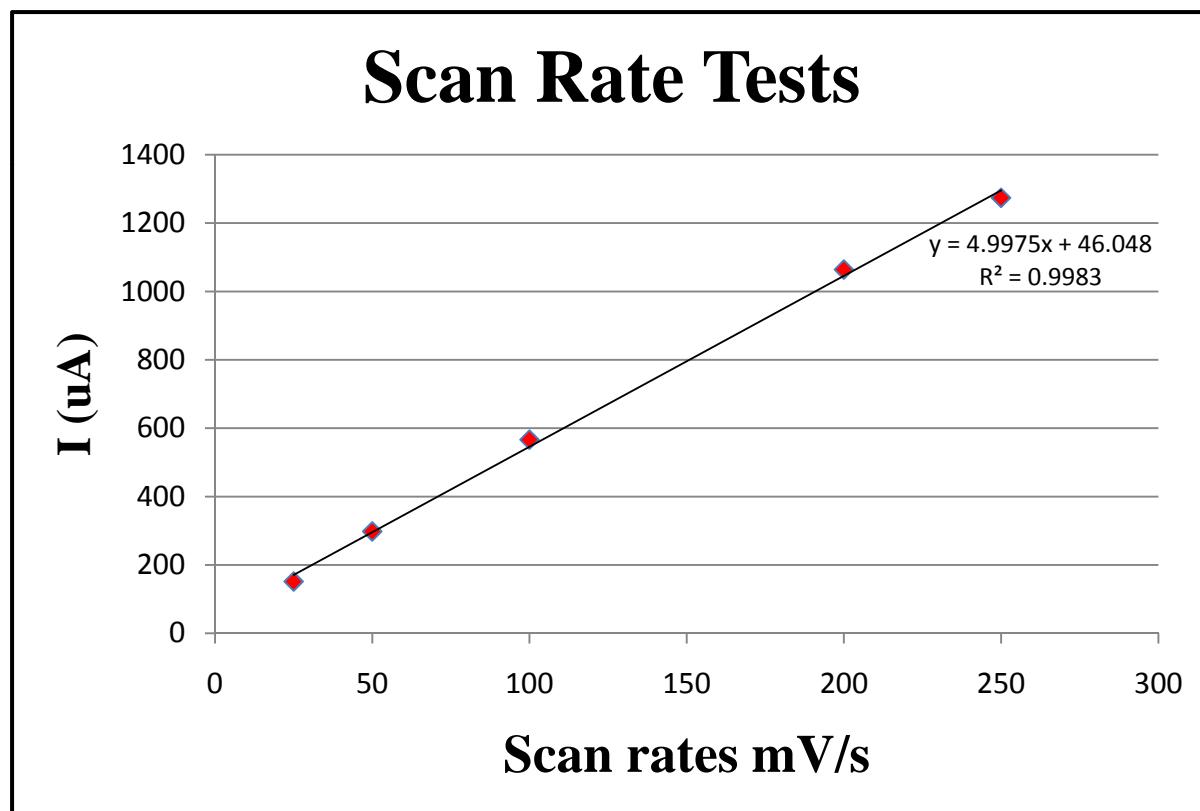
Sweep potentials = -1000 mV - 1000 mV

**Solution used: TBAPF<sub>6</sub>/PC  
monomer free solution**

Columbic efficiency = (cathode/anode x 100)%



Solutions  
Electrochemical Polymerization  
Scan Rate Tests  
Oxidizing Tests  
Doping  
Morphological Characterization  
Construction of Device  
Single Wall Carbon Nanotubes



Linear relation evinces a surface-immobilized film



Solutions  
Electrochemical Polymerization  
Scan Rate Tests  
**Oxidizing Tests**  
Doping  
Morphological Characterization  
Construction of Device  
Single Wall Carbon Nanotubes

### Parameters used

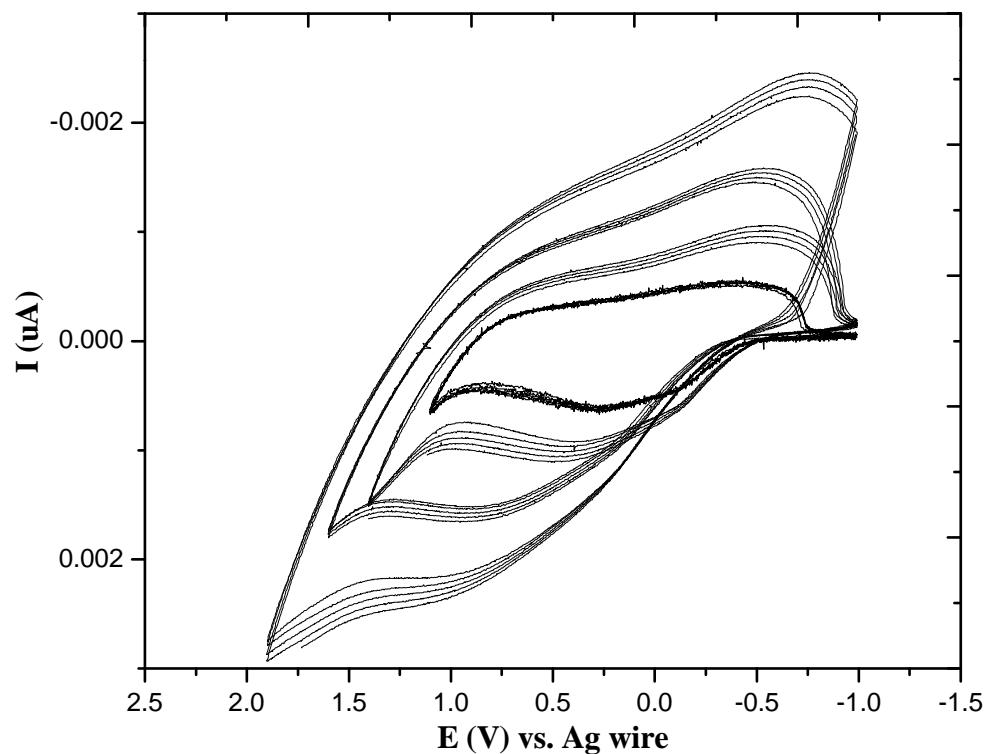
Sweep rate = 100 mV/s

Total sweeps = 9

Sweep Potentials = Various potentials

**Solution used:**  
**PProDot/TBAPF<sub>6</sub>/PC**

### Oxidizing Voltage Window Tests



Solutions

Electrochemical Polymerization

Scan Rate Tests

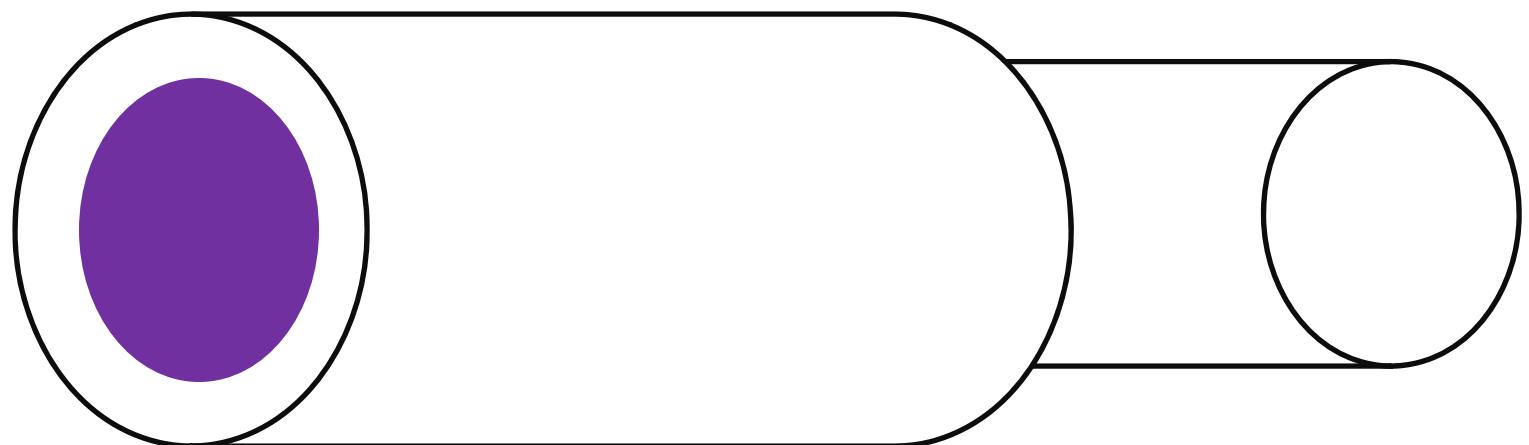
**Oxidizing Tests**

Doping

Morphological Characterization

Construction of Device

Single Wall Carbon Nanotubes



Solutions  
Electrochemical Polymerization  
Scan Rate Tests  
Oxidizing Tests  
**Doping**  
Morphological Characterization  
Construction of Device  
Single Wall Carbon Nanotubes

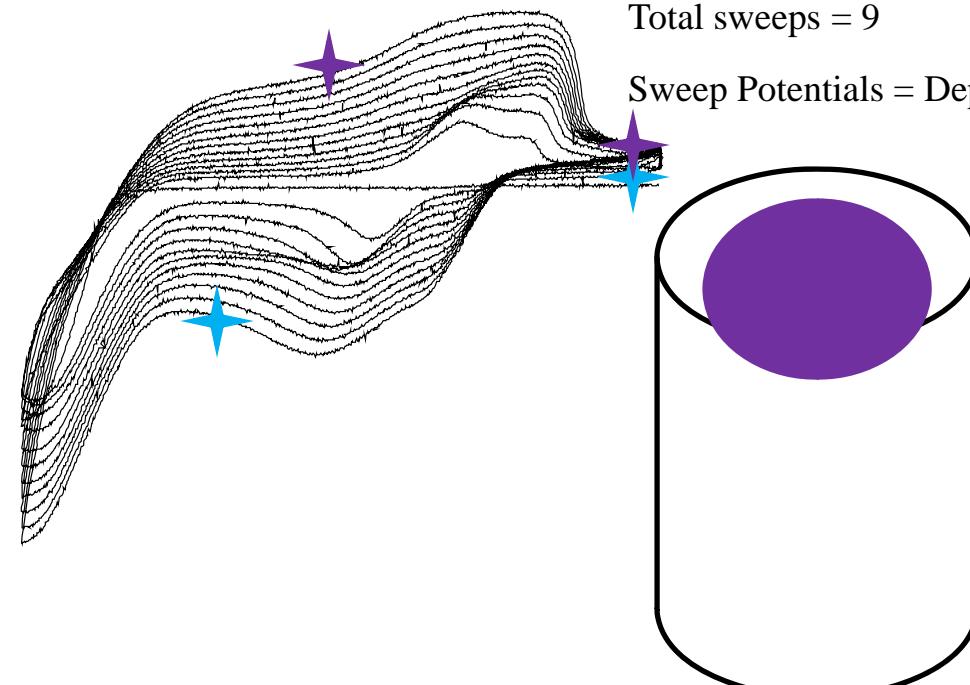
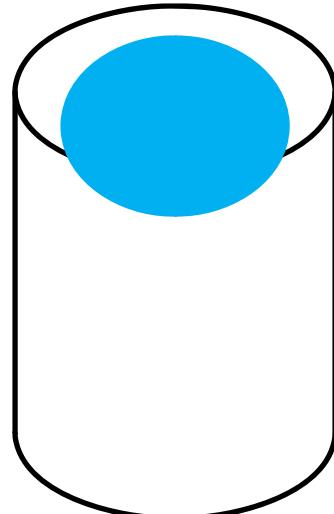
## Doping polymer to oxidized state

### Parameters

Sweep rate = 100 mV/s

Total sweeps = 9

Sweep Potentials = Depends



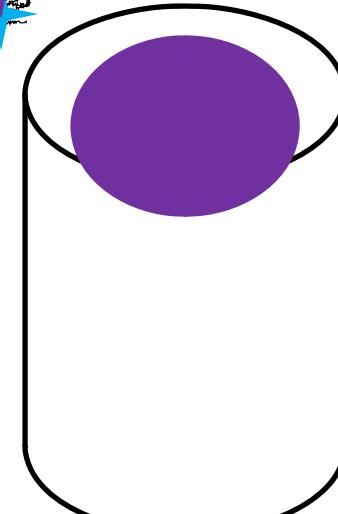
## Doping polymer to neutral state

### Parameters

Sweep rate = 100 mV/s

Total sweeps = 9

Sweep Potentials = Depends



Solutions  
Electrochemical Polymerization

Scan Rate Tests

Oxidizing Tests

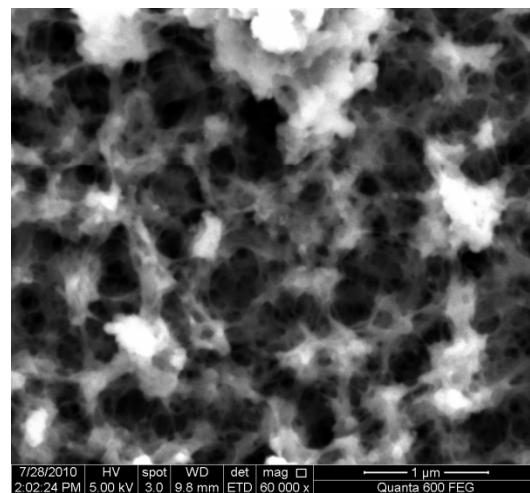
Doping

Morphological Characterization

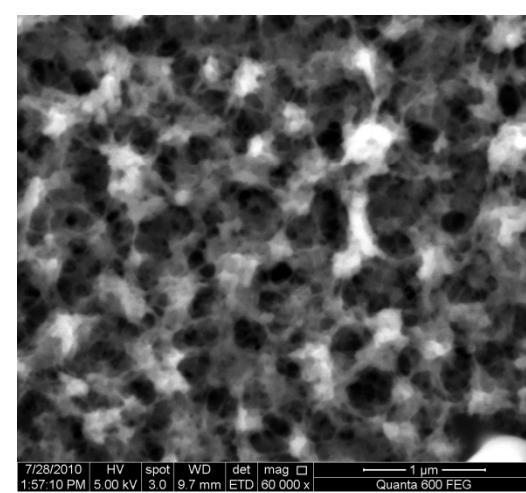
Construction of Device

Single Wall Carbon Nanotubes

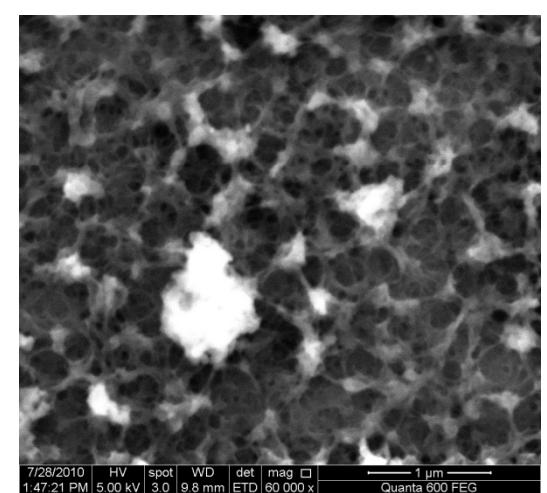
## Morphological Characterization of PProDot Films



SEM of PProDot Film



SEM of PProDot Oxidized Film



SEM of PProDot Neutral Film

Solutions

Electrochemical Polymerization

Scan Rate Tests

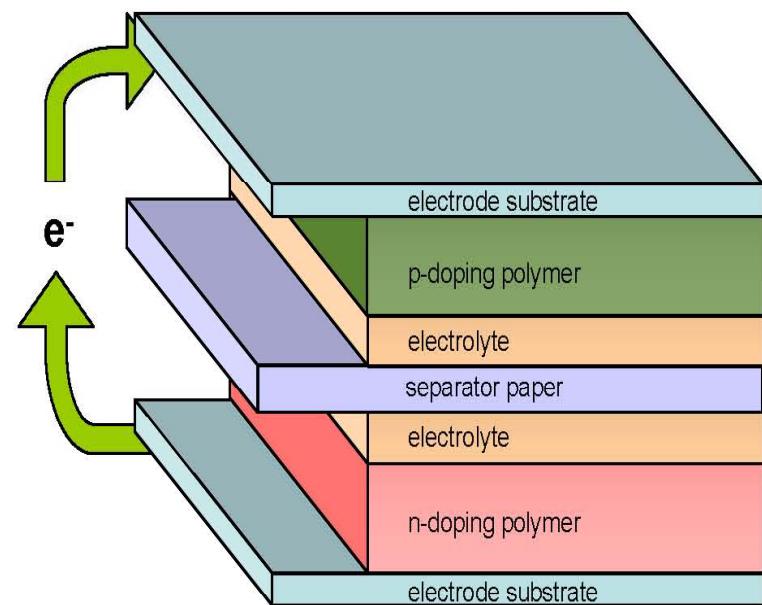
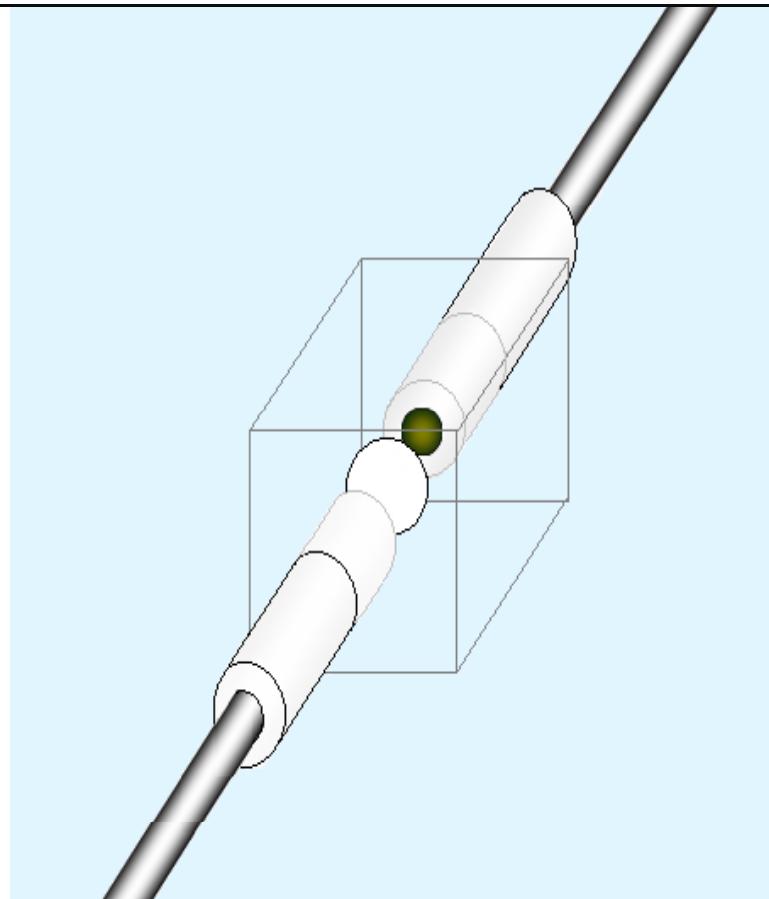
Oxidizing Tests

Doping

Morphological Characterization

**Construction of Device**

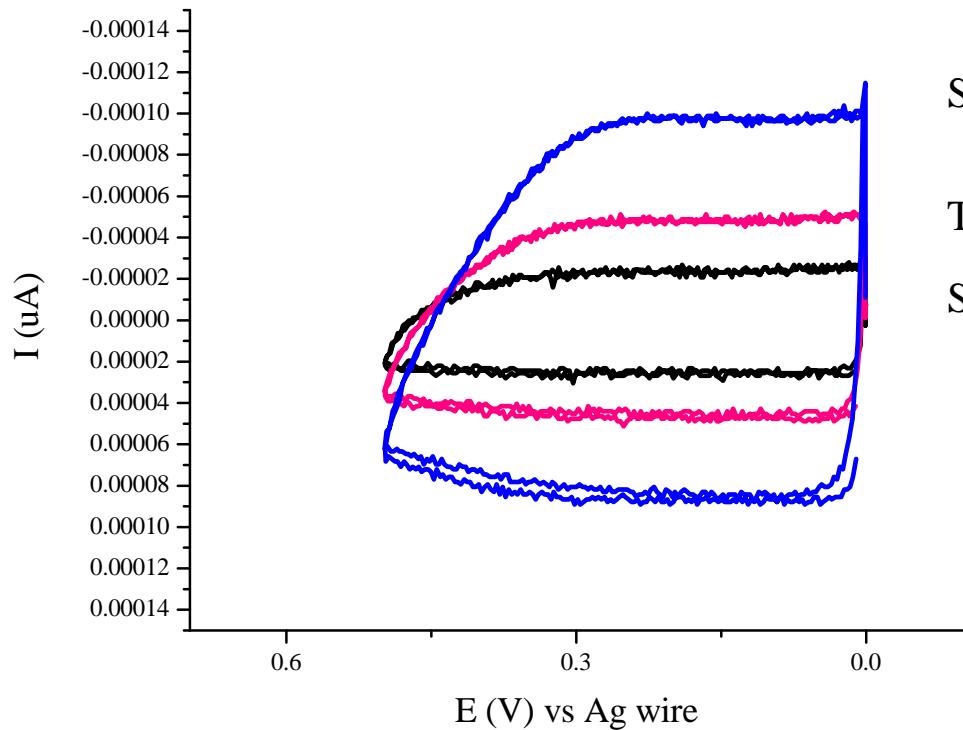
Single Wall Carbon Nanotubes



**Electrolyte = TBAPF<sub>6</sub>/PC**

Solutions  
Electrochemical Polymerization  
Scan Rate Tests  
Oxidizing Tests  
Doping  
Morphological Characterization  
**Construction of Device**  
Single Wall Carbon Nanotubes

### Scan Rate Tests on Device



### Scan Rate Tests on Device

#### Parameters used

Sweep rates = 25, 50, 100, 250, 500, 1000,  
2500, 5000, 10000 mV/s

Total sweeps = 6

Sweep potentials = 0 mV - 500 mV

Solutions

Electrochemical Polymerization

Scan Rate Tests

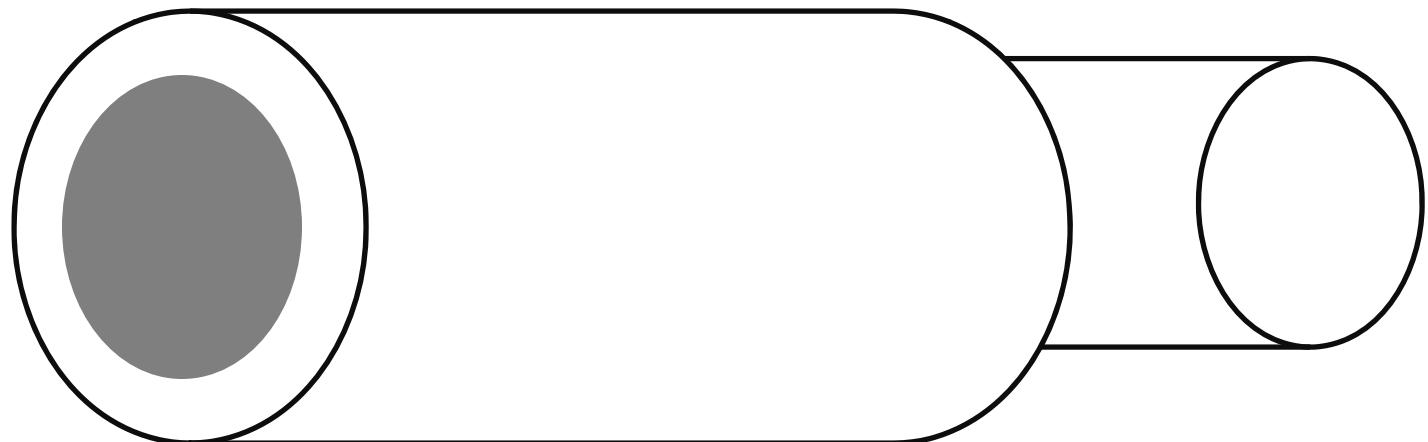
Oxidizing Tests

Doping

Morphological Characterization

Construction of Device

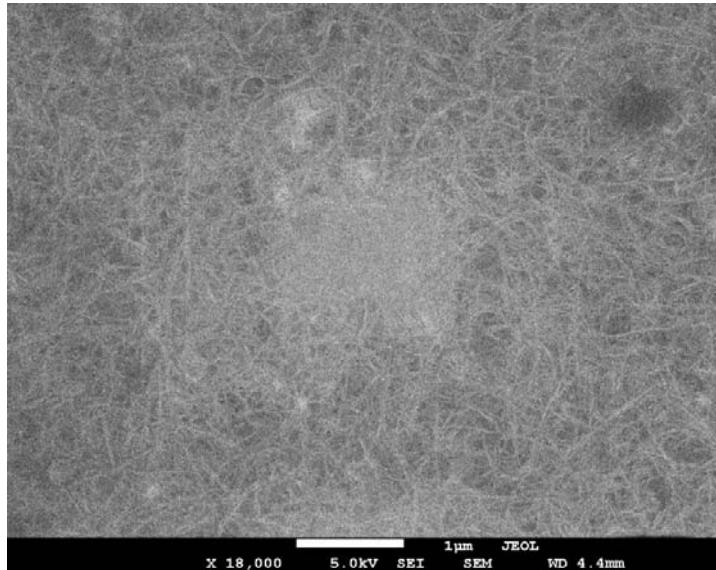
Single Wall Carbon Nanotubes



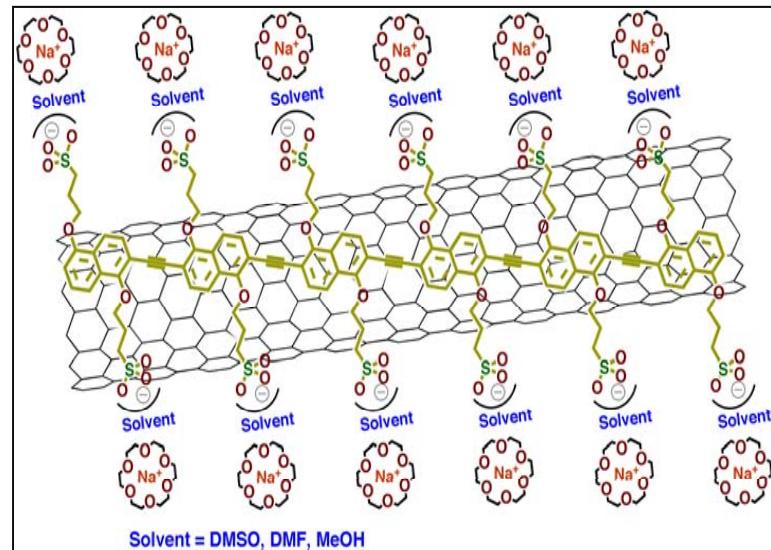
**Drop cast 8  $\mu\text{L}$  of PNES/SWNT**

Solutions  
Electrochemical Polymerization  
Scan Rate Tests  
Oxidizing Tests  
Doping  
Morphological Characterization  
Construction of Device  
Single Wall Carbon Nanotubes

## Morphological Characterization of PNES/SWNT by SEM



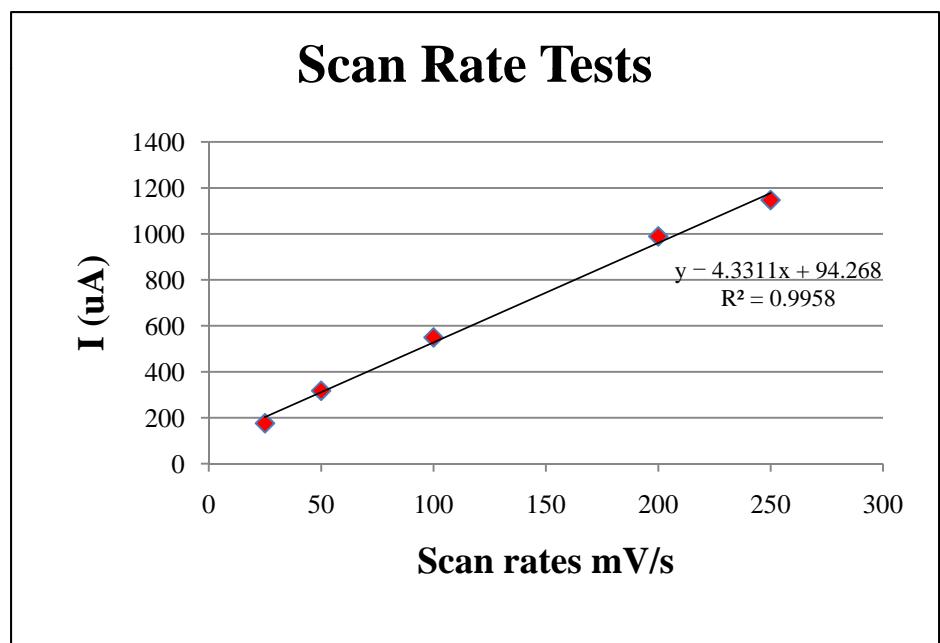
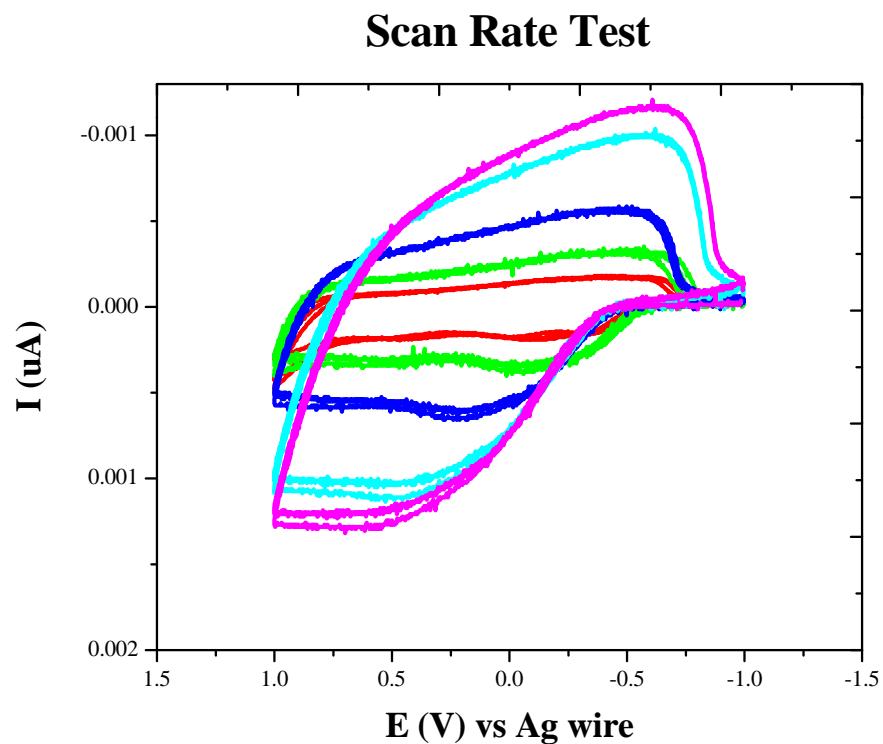
M. Rosario-Canales et al., Manuscript in Preparation



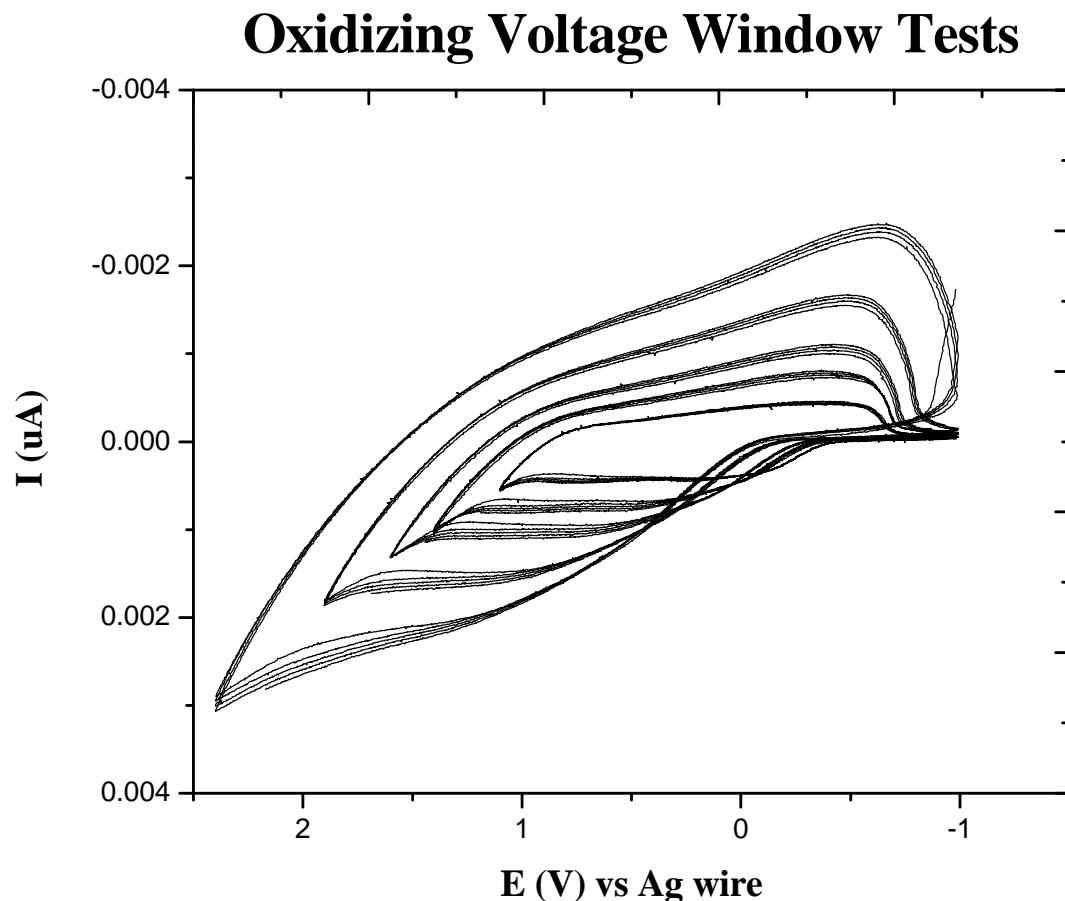
P. Deria et al., Manuscript Submitted to JACS

**PNES: poly[2,6-{1,5-bis(3-propoxysulfonic acidsodiumsalt)}naphthylene]ethynylene**

Solutions  
Electrochemical Polymerization  
Scan Rate Tests  
Oxidizing Tests  
Doping  
Morphological Characterization  
Construction of Device  
Single Wall Carbon Nanotubes

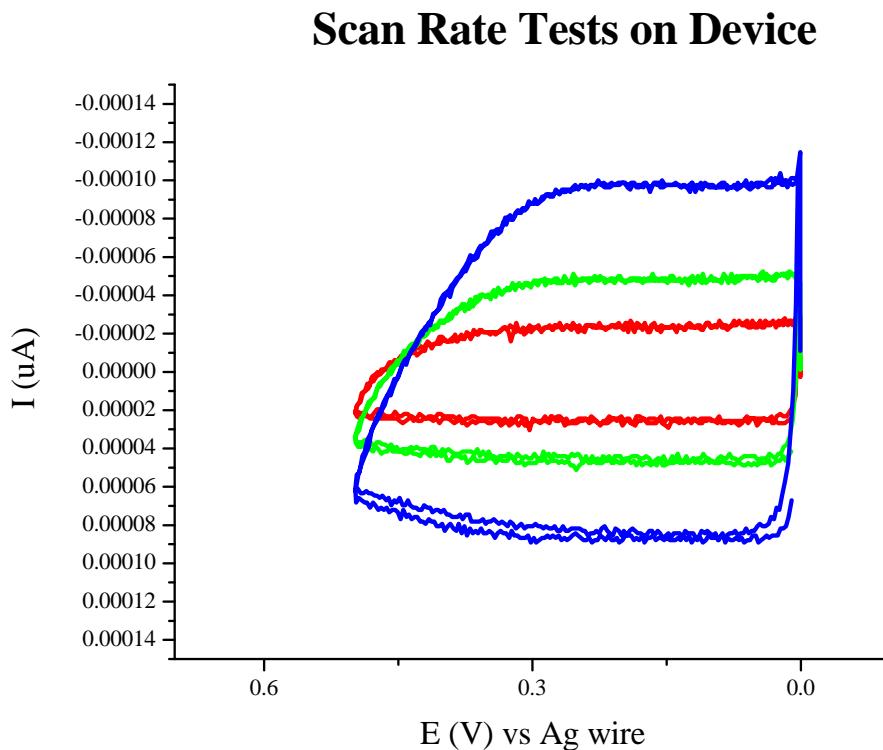


Solutions  
Electrochemical Polymerization  
Scan Rate Tests  
Oxidizing Tests  
Doping  
Morphological Characterization  
Construction of Device  
Single Wall Carbon Nanotubes



Solutions  
Electrochemical Polymerization  
Scan Rate Tests  
Oxidizing Tests  
Doping  
Morphological Characterization  
Construction of Device  
Single Wall Carbon Nanotubes

## Scan Rate Tests on Device



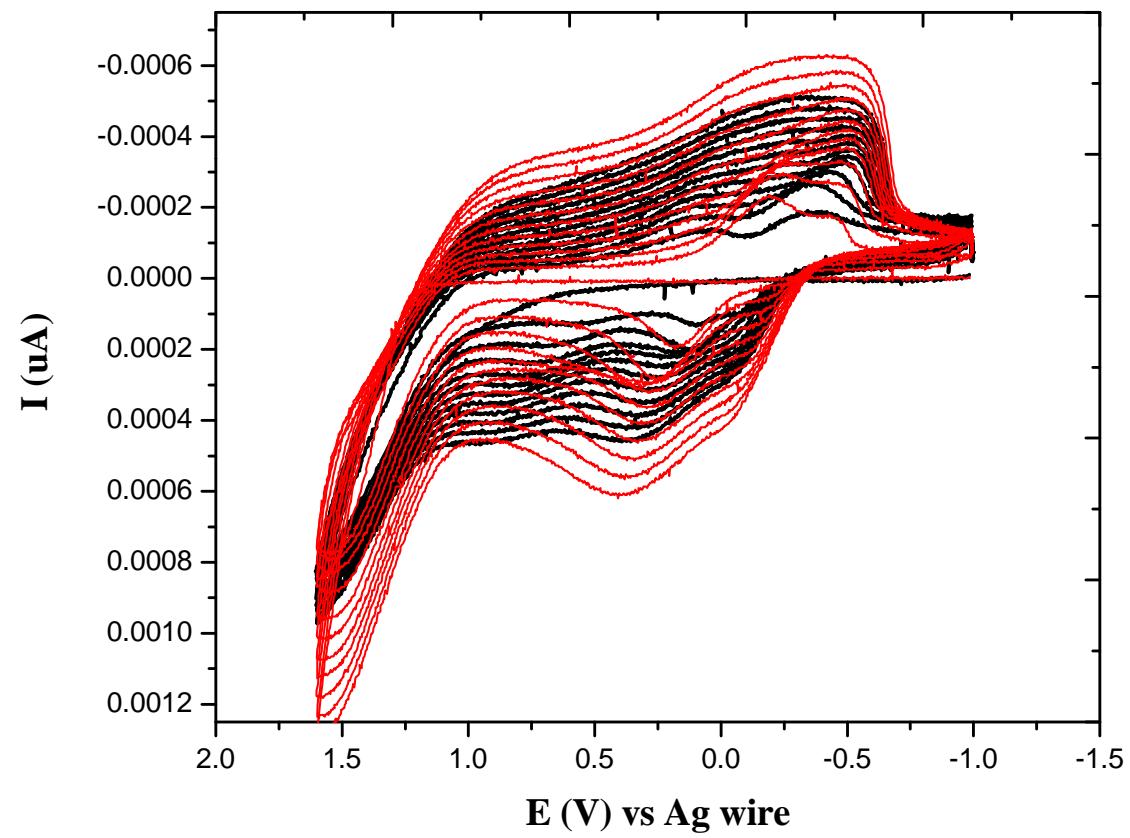
### Parameters used

Sweep rates = 25, 50, 100, 250, 500, 1000,  
2500, 5000, 10000 mV/s

Total sweeps = 6

Sweep potentials = 0 mV - 500 mV

# Results



# Acknowledgements

**Professor Jorge Santiago-Avilés**

University of Pennsylvania, Department of  
Electrical and Systems Engineering

**Jan Van der Spiegal & SUNFEST**

University of Pennsylvannia

**Preethi Gopu**

University of Pennsylvannia

**Xenia Barbosa**

University of Puerto Rico, Humacao

**Donniece Brownlee**

Philadelphia Military Academy

