Thin Films of Polypyrrole on Particulate Aluminum

1

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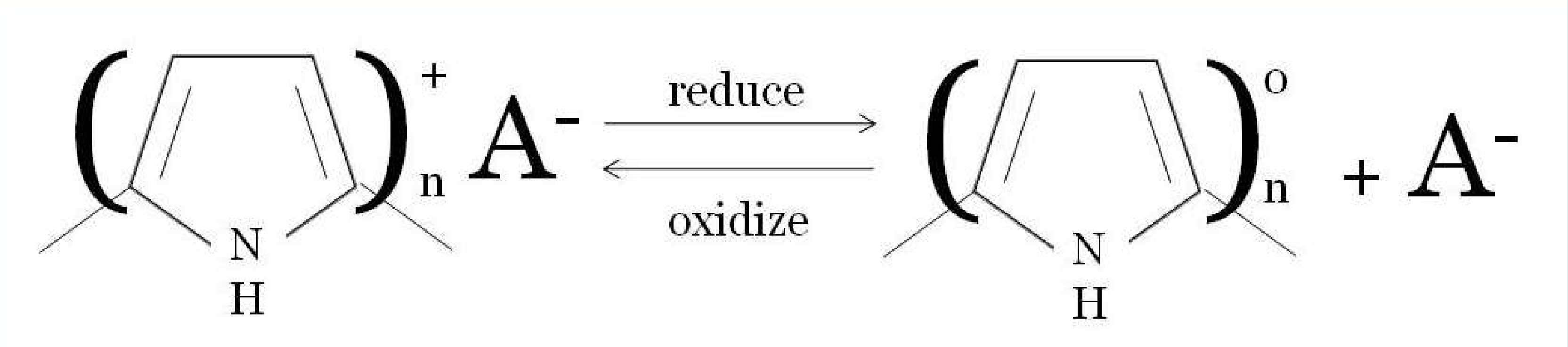
Why Conducting Polymers?

- Perhaps act as “Smart Coating”
  - Release of Corrosion Inhibiting Anions
- Mixed Potential between surface and ECP
- Perhaps acts as an oxidant to form passive layer



Why Polypyrrole/Flake?

Polypyrrole

- Poor mechanical properties
- Poor adhesion
- Solubility issues
- Continuous layer needed

Polypyrrole Coated Flake

- Easy coating incorporation
- Less quantity of conducting polymer required
- Solubility is not an issue

Synthesis

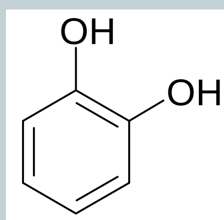
Synthesis Details

Ex #	Al Flake (g)	Pyrrole (ml)	Catechol (g)	$(NH_4)_2S_2O_8$ (g)	H_2O (ml)
1	50	11.5	18.2	37.7	1650
2	50	11.5		37.7	1650

Al flake paste combined with:

- DI water
- Catechol ($C_6H_6O_2$)
- $(NH_4)_2S_2O_8$
- Pyrrole monomer

Vacuum filtration & paste dried overnight, then ground using mortar and pestle and passed through a sieve with 150μm pore size



Catechol

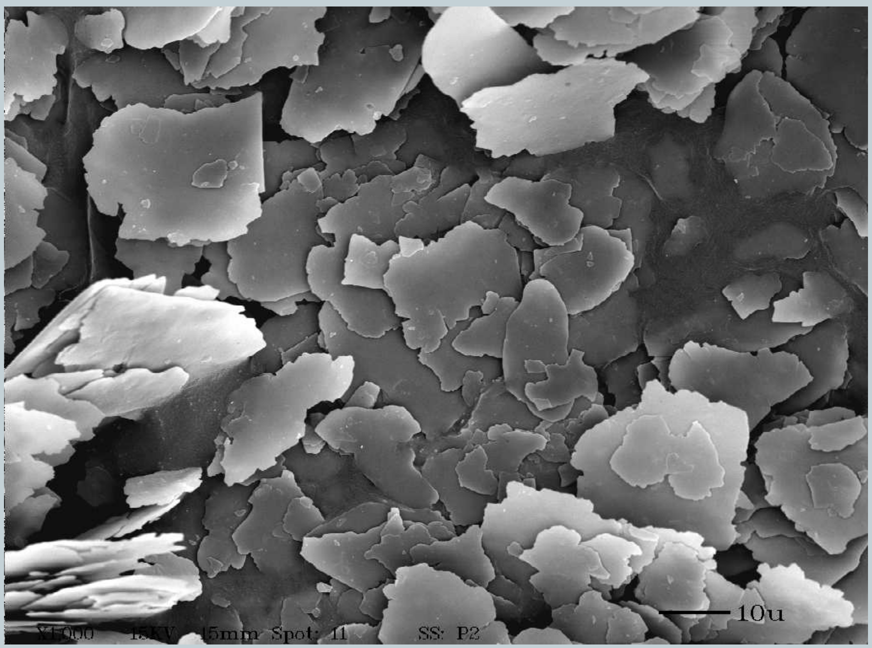
Density Test

As Received Ex 1 Ex 2

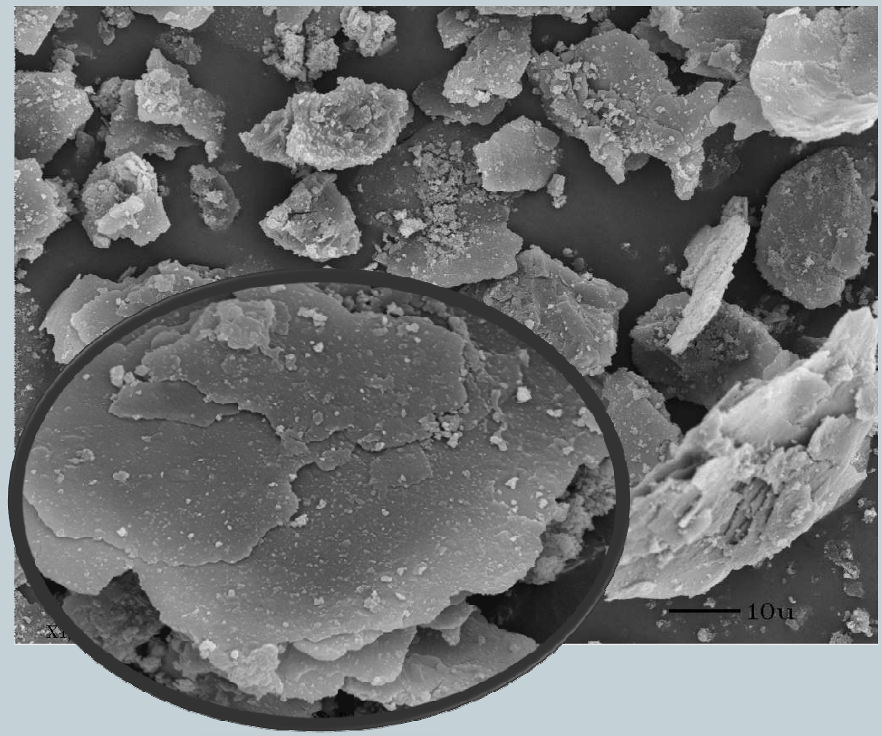
Tetrachloroethylene (1.622 g/cm^3)
Plain Aluminum Flake (2.70 g/cm^3)
Polypyrrole (0.967 g/cm^3)

SEM

As Received Aluminum Flake



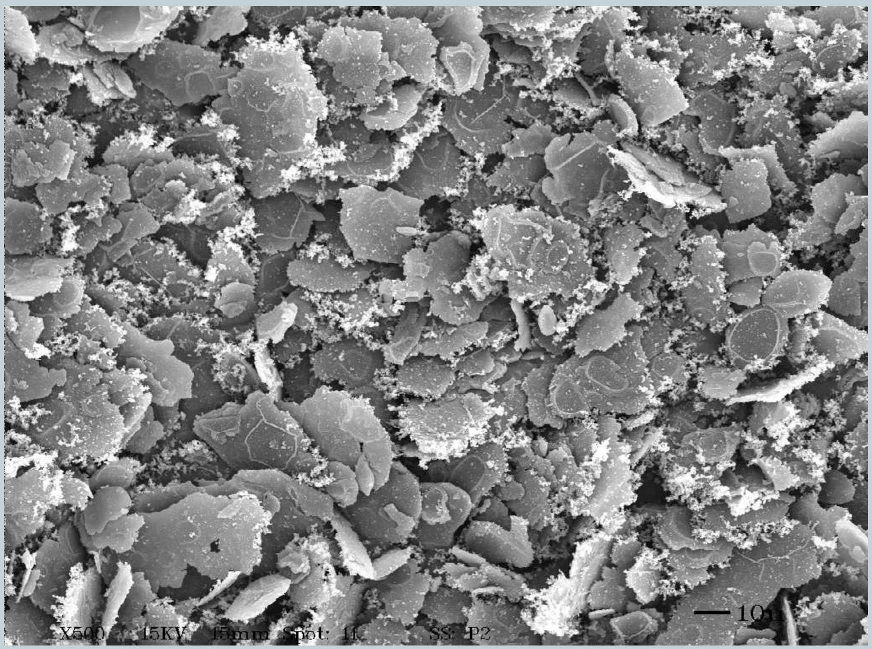
PPy (Catechol) Ex #1

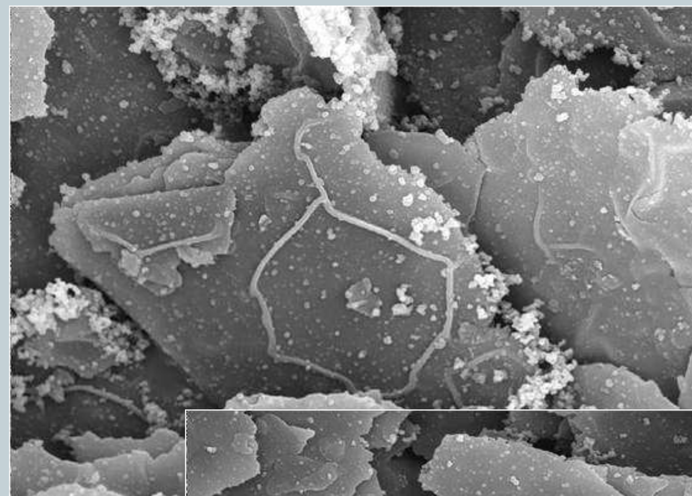


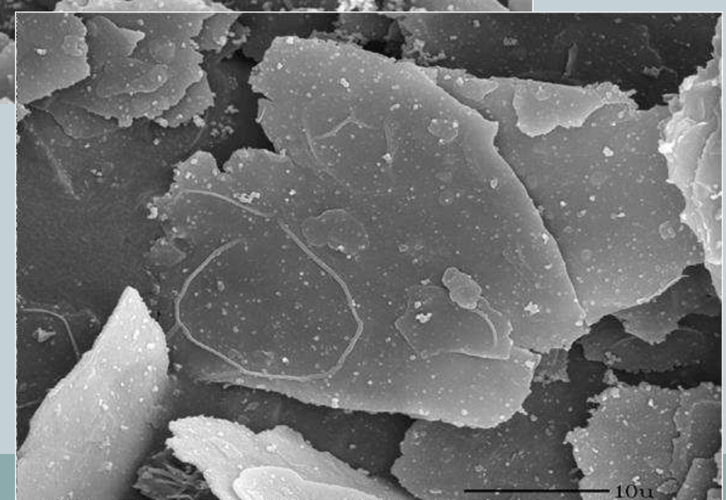
SEM Continued

PPy (no Catechol)

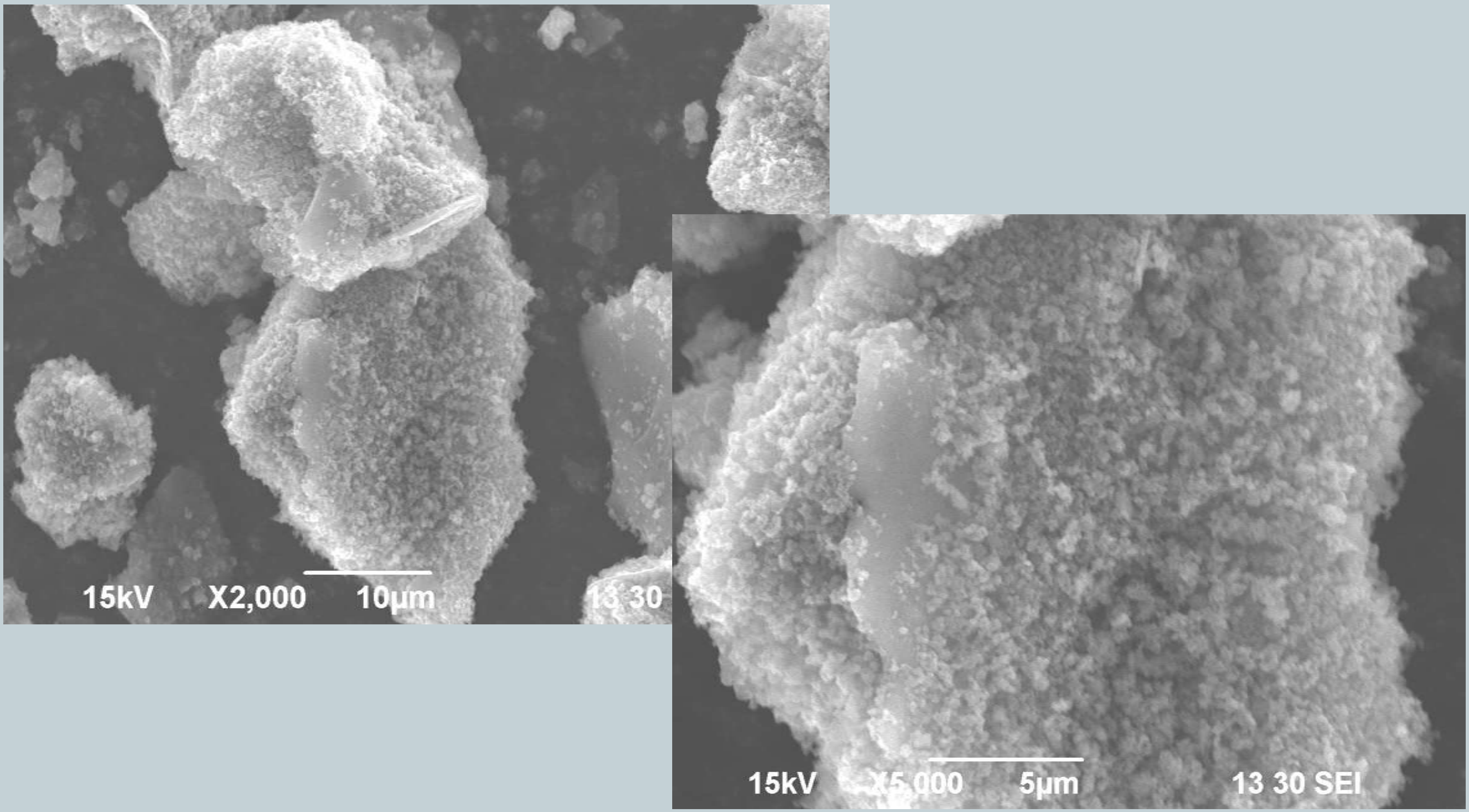
Ex #2



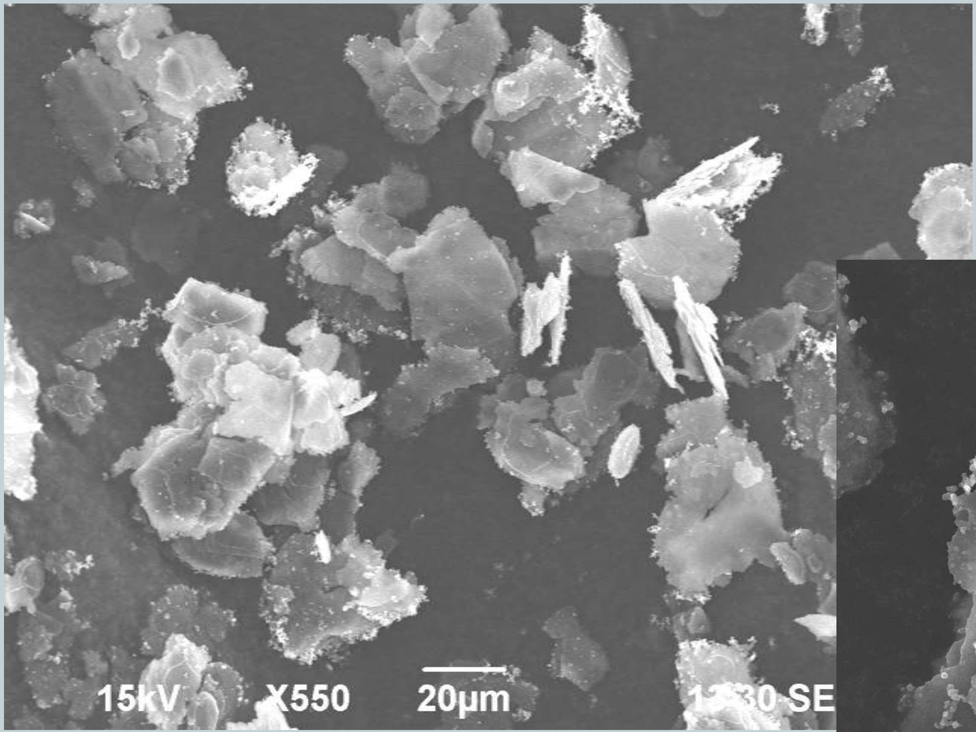


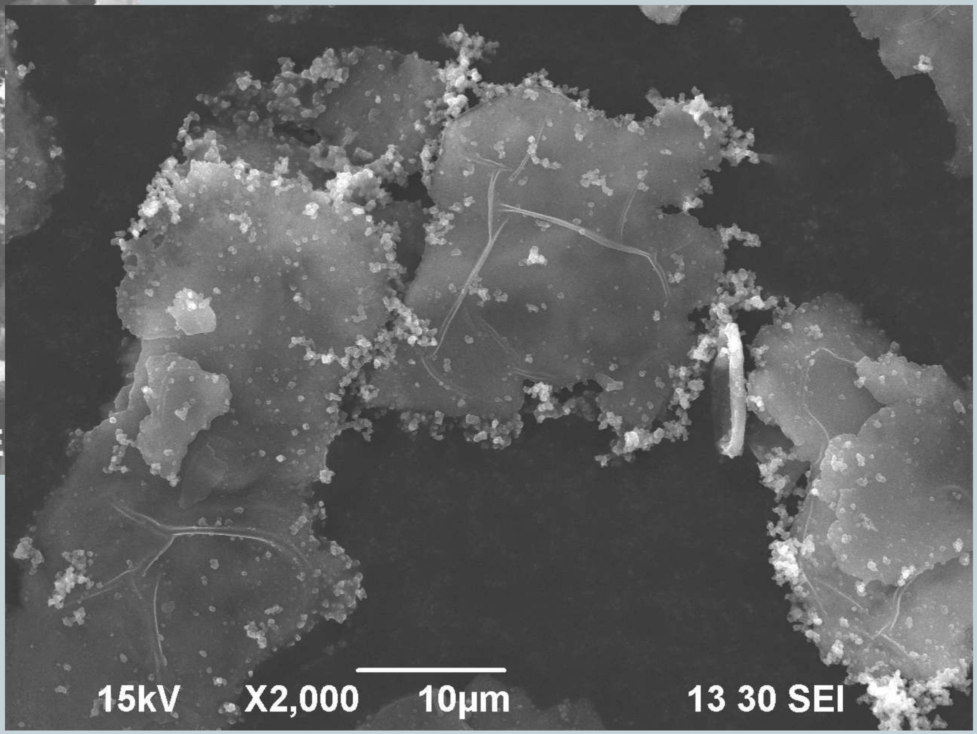


SEM (No Gold)
Ex #1



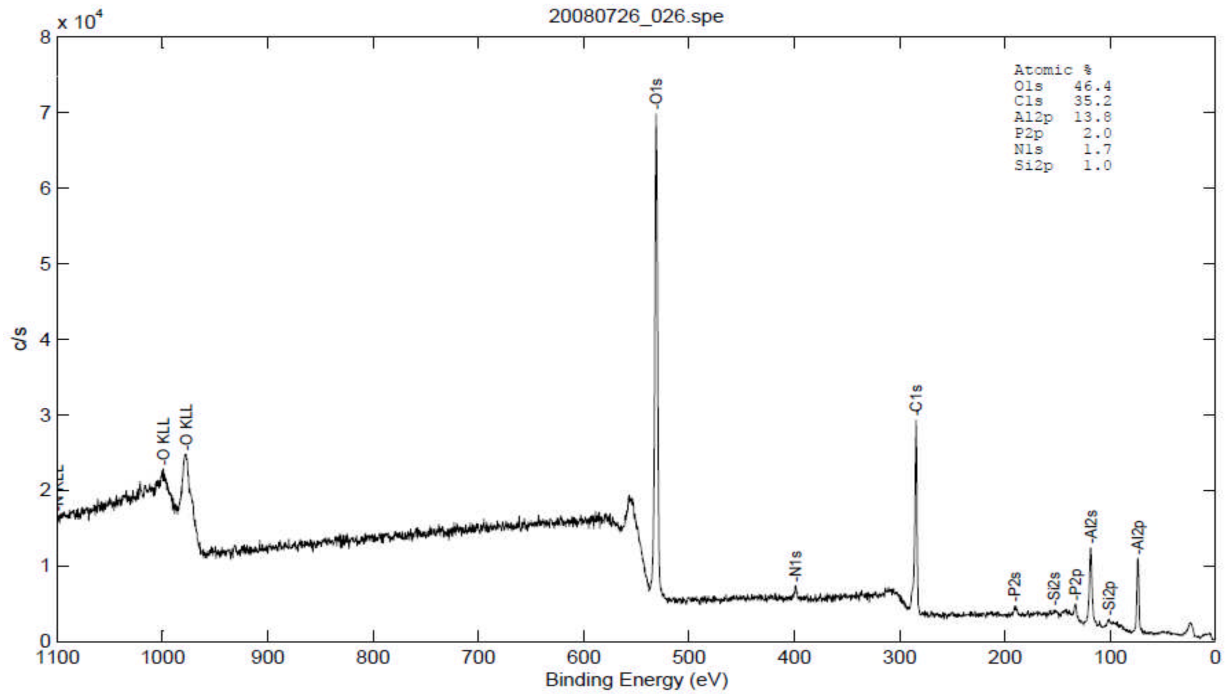
SEM (No Gold)
Ex #2





XPS

As-Received Al Flake



Atomic	%
O1s	46.4
C1s	35.2
Al2p	13.8
P2p	2.0
N1s	1.7
Si2p	1.0

<u>From MSDS of Flake</u>

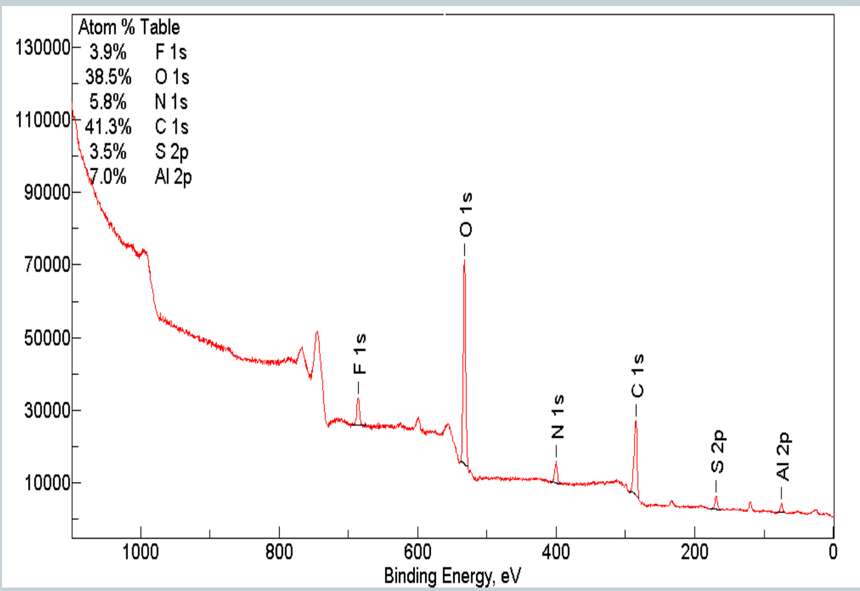
39.00 % aluminum

26.00% aluminum oxide

35.00% 1-methoxy-2-propanol

From XPS, it appears the coatings are ~10-20 nm thick

Polypyrrole/Al Flake from Experiment #1

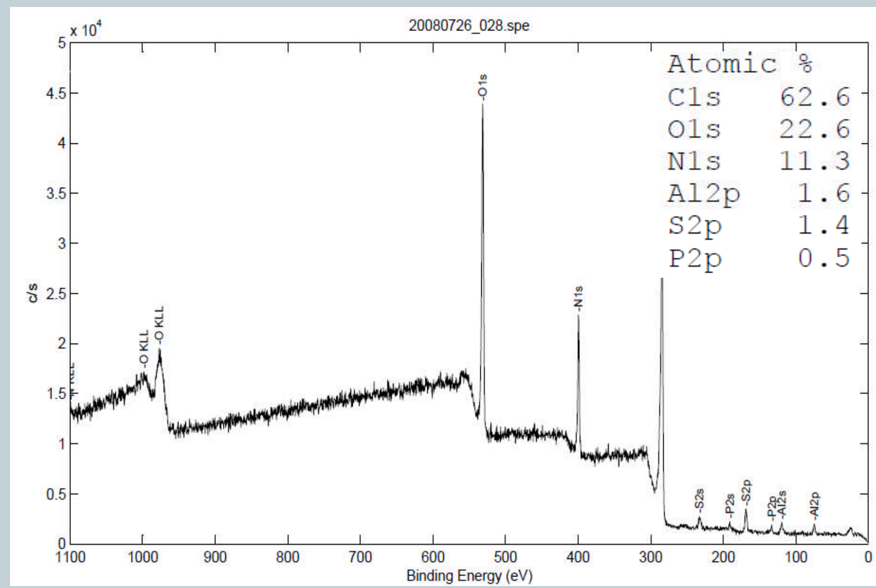


S/N (Dopant Level)
S/N = ~3/5
3 dopant ions per 5 pyrrole units

C/N (Polymer)
C/N = ~7/1

4/1 if polypyrrole

Polypyrrole/Al Flake from Experiment #2



S/N (Dopant Level)
S/N = ~1/11
1 dopant ions per 11 pyrrole units

C/N (Polymer)
C/N = ~5/1

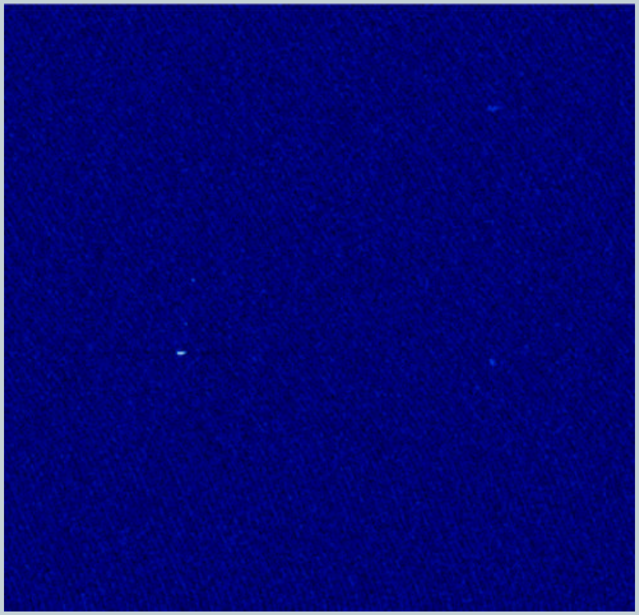
4/1 if polypyrrole

Conductive AFM

As-Received Flake

Polypyrrole/AL Flake Ex #1

Current Image



σ_{ave}= 1.6 *S*/cm

Scan Size 2.5 x 2.5 µm

Formulations of Polypyrrole/Flakes

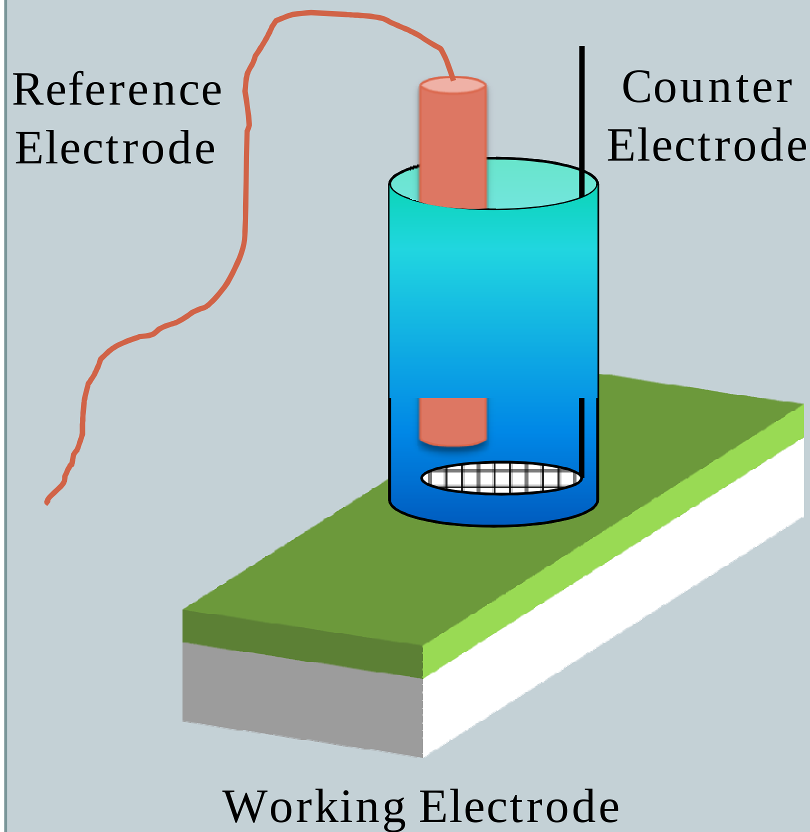
- Formulations prepared at various PVCs of coated and uncoated flakes and were combined with:
  - Epikure 3175 (aluminum) or Epikure 3115 (mica)
  - Epon Resin 828
  - Methyl Isobutyl Ketone (MiBK)

Al 2024-T3 panel application of formulation

- Coatings were applied to sanded and degreased 3” x 6” Al 2024-T3 panels using a 3” drawdown bar at 8 mils

- Panels were allowed to flash off and were then placed in an oven to fully cure

Electrochemical Impedance Spectroscopy



V=IR (DC)

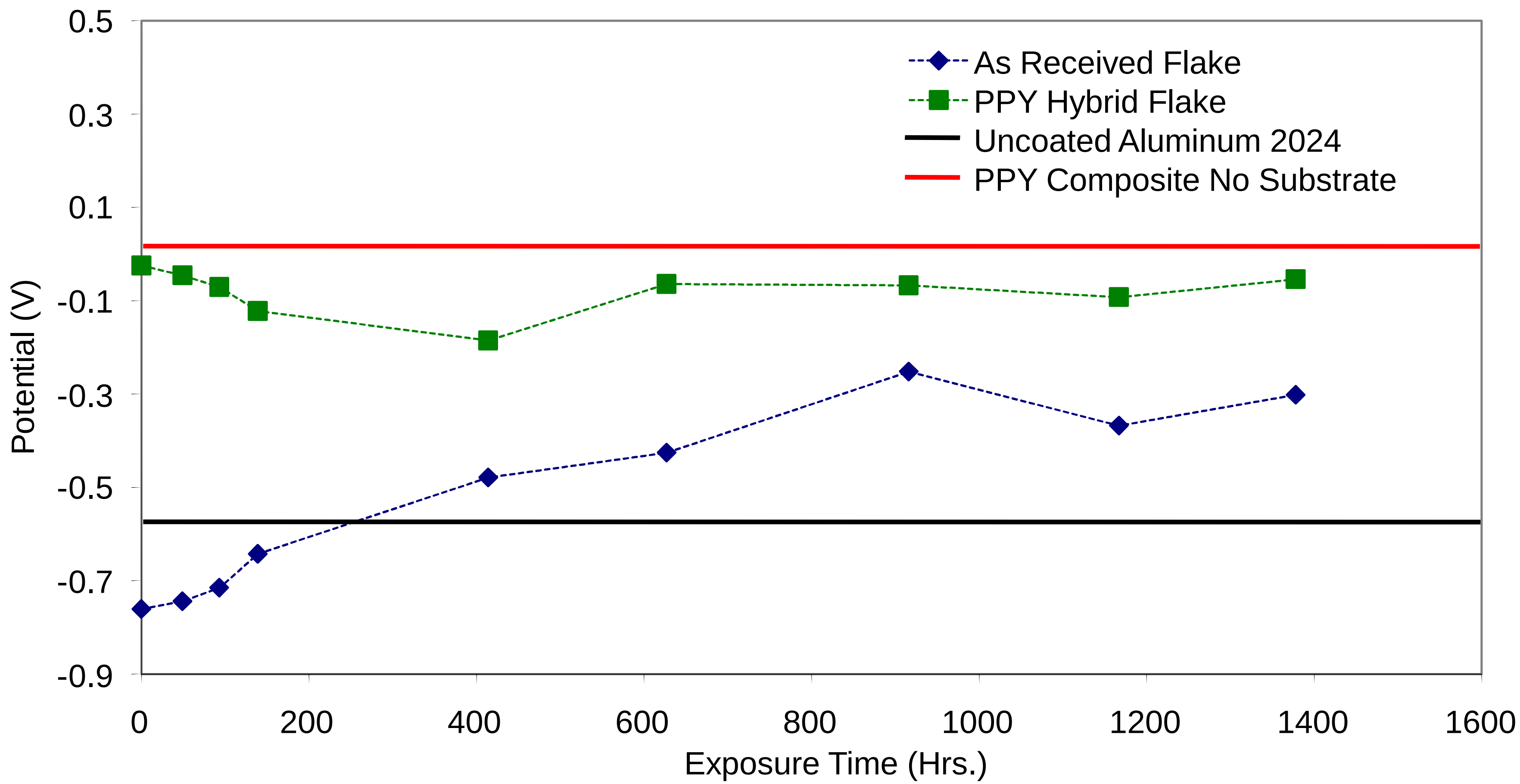
V=IZ (AC)

Apply a small sinusoidal potential (~5 to 10 mV) to the open circuit potential at varying frequencies

Measure phase lag (V- I) and current for varying applied frequencies

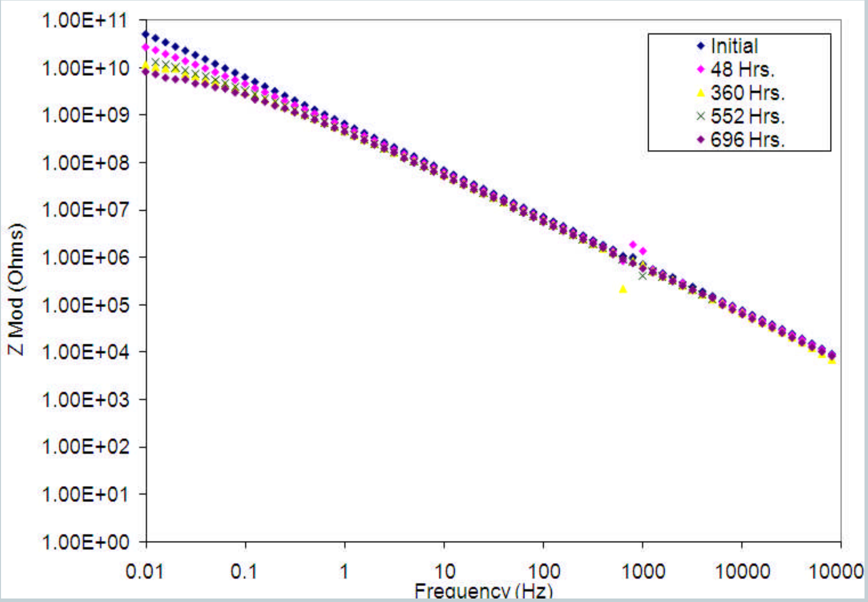
Presented usually as either a Bode (log modulus vs. log frequency) or a Nyquist plot (Z' vs. Z")

Open Circuit Potentials
Al 2024-T3

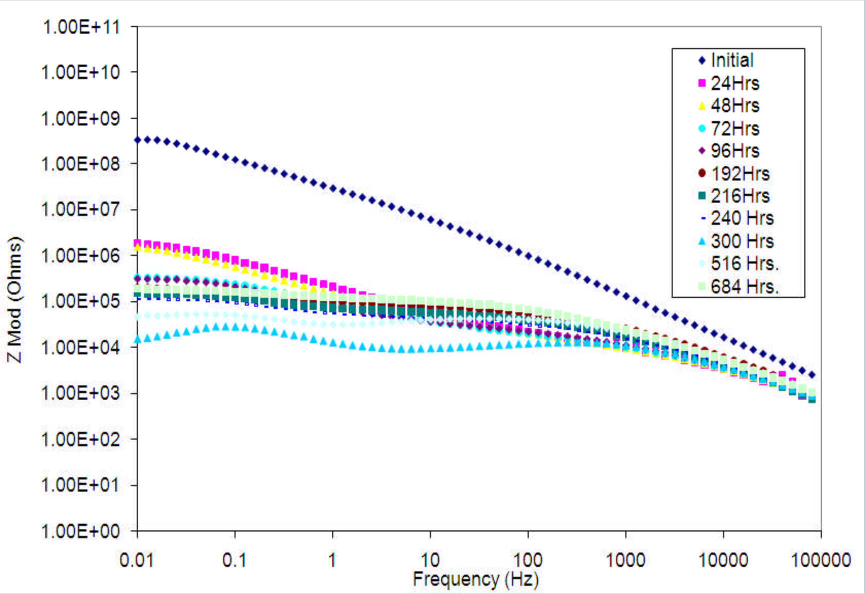


25% PVC—Aluminum

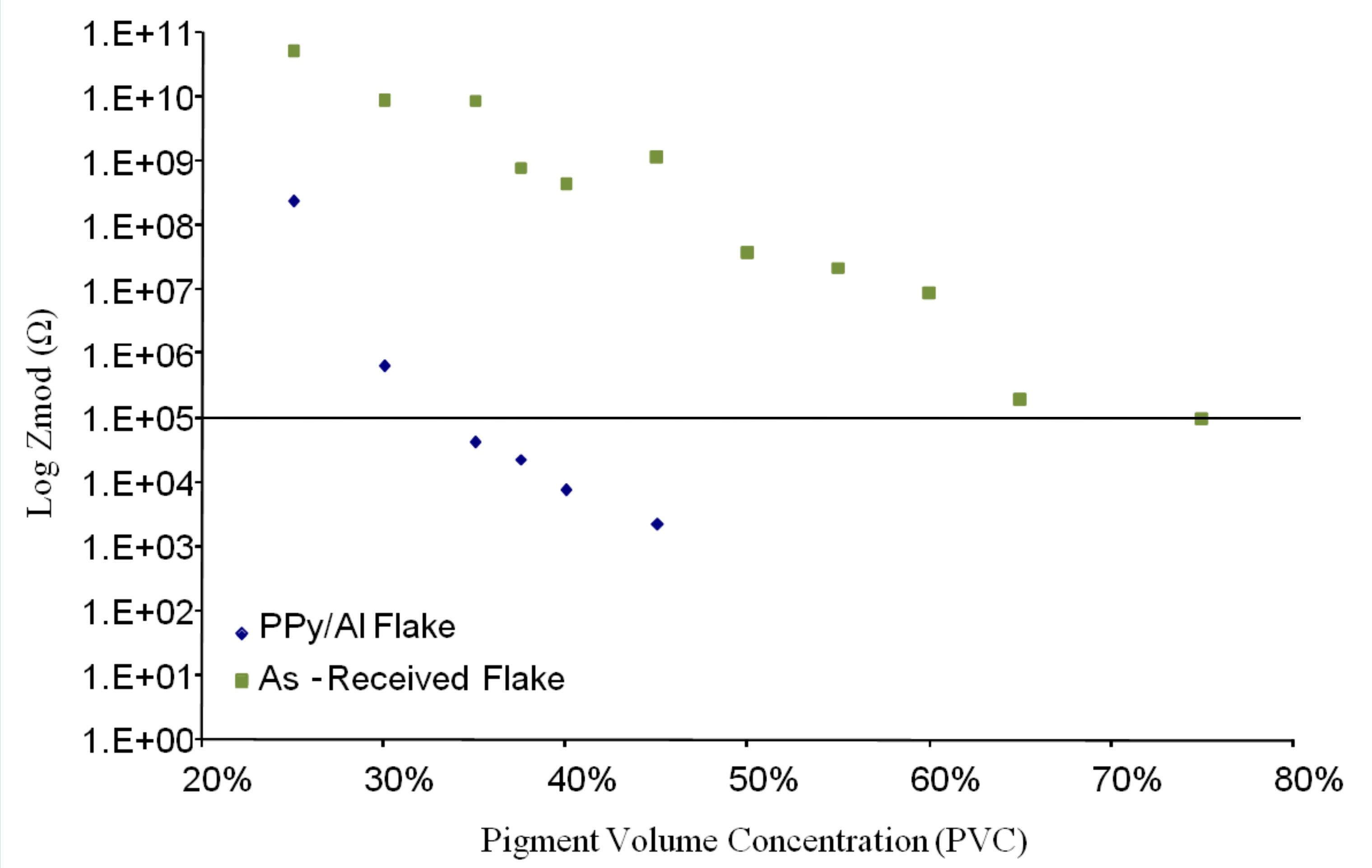
As-Received Flake



Polypyrrole Aluminum Flake

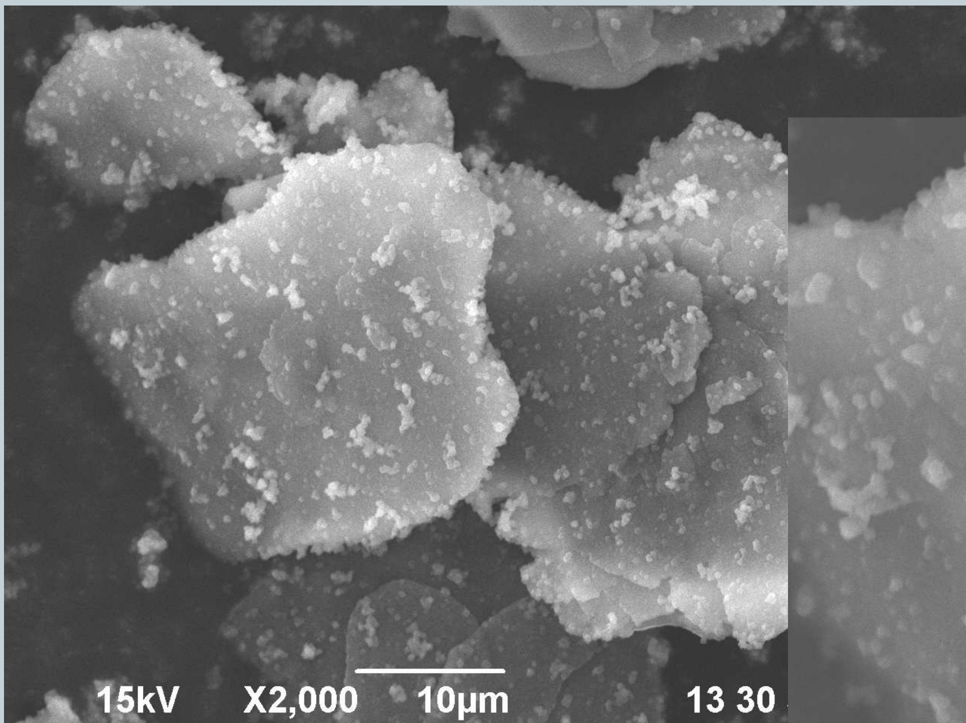


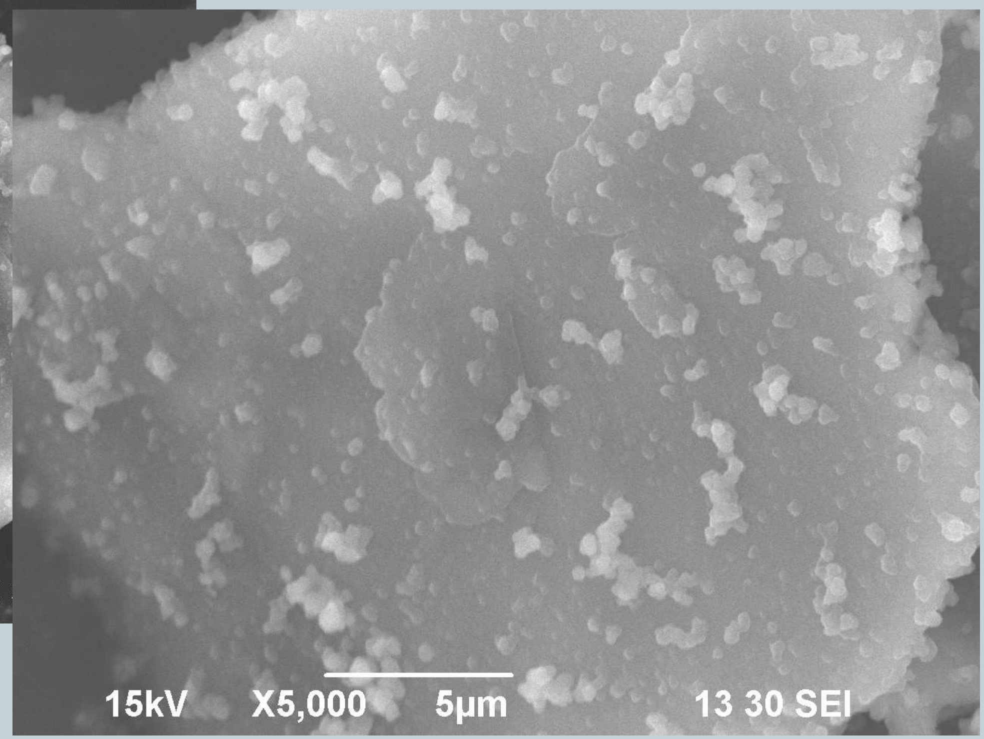
Impedance and PVC---Aluminum Flake



New Directions

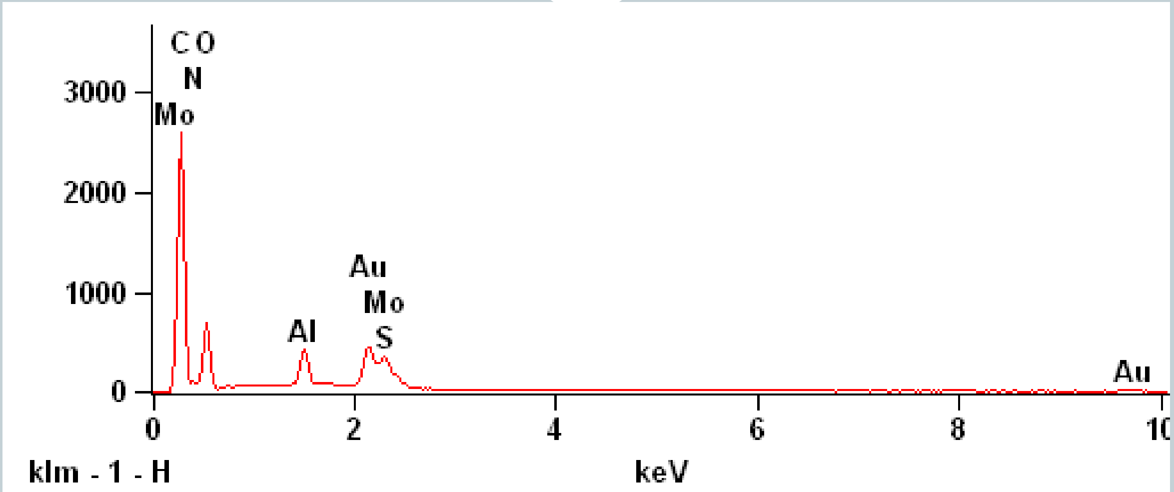
- Incorporation of corrosion inhibiting anions.





Molybdate

From EDX



Atom %

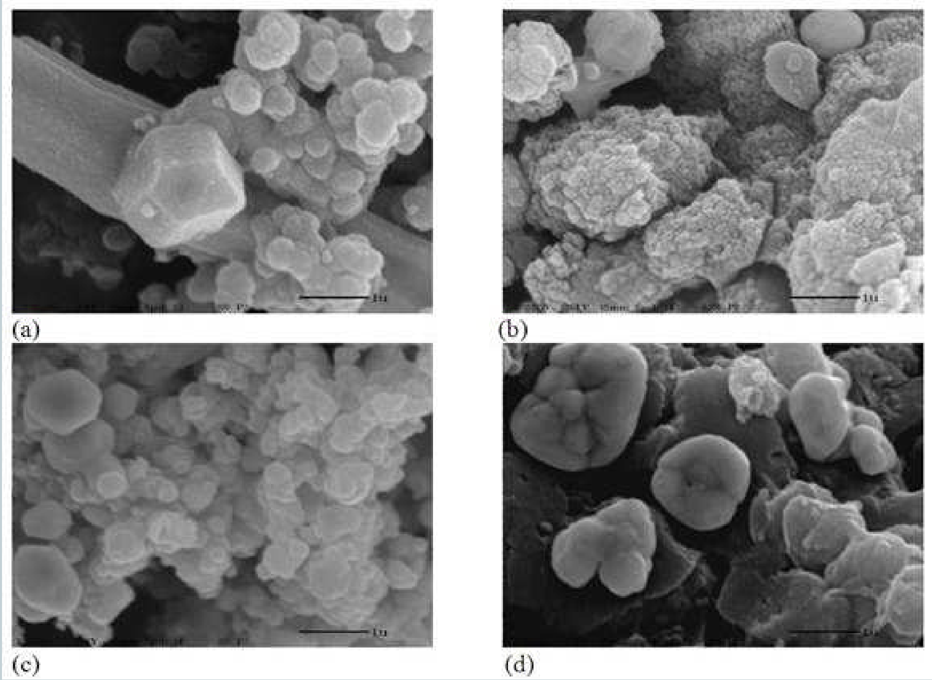
C	N	O	Al	S	Mo
76.04	8.42	14.19	0.76	0.05	0.54

Atom % Error (± 3σ)

C	N	O	Al	S	Mo
1.67	7.10	1.46	0.07	0.20	.16

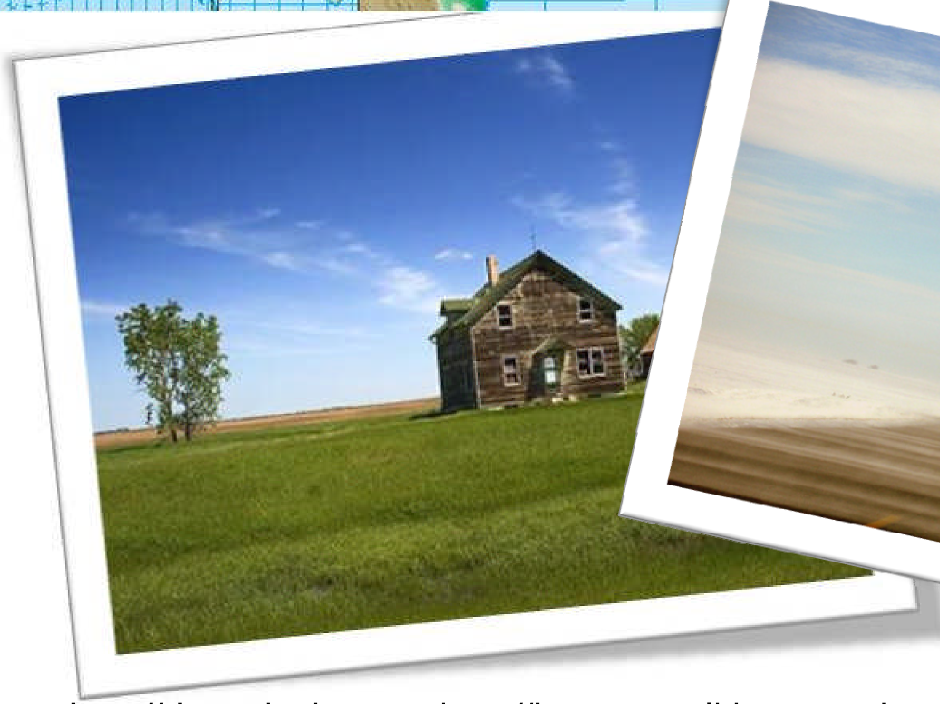
Photosynthesis of Polypyrrole

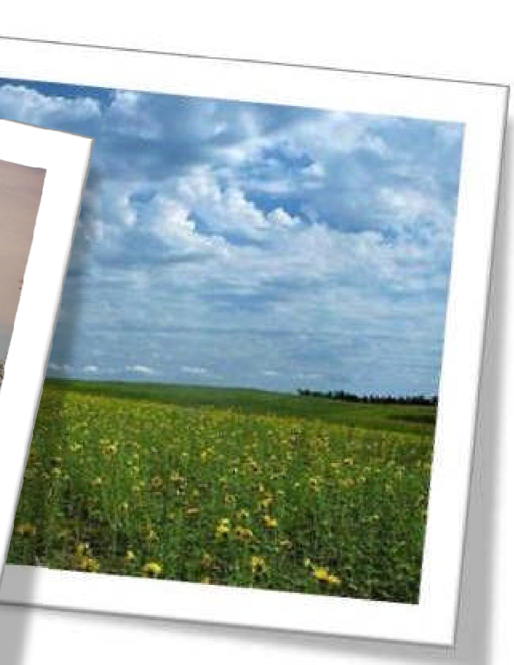
Part 2: Photo-chemical polymerization reactions (UV light)					
Label	Monomer	Oxidant	Pyrrole:Oxidant	Pyrrole:SDS	Pyrrole:pTSA
PPy-5	Pyrrole	$AgNO_3$	1:1		
PPy-6	Pyrrole	$AgNO_3$	1:1	4:1	
PPy-7	Pyrrole	$AgNO_3$	1:1		4:1
PPy-8	Pyrrole	$AgNO_3$	1:1	4:1	4:1



Polypyrrole Photo- chemically synthesized (a) without surfactant (PPy-5), (b) SDS as surfactant (PPy-6), (c) pTSA as surfactant (PPy-7), and (d) both SDS and pTSA (PPy-8)

Questions?





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