Chem 402
Effect of Heat Treatment on the Performance of Nanoporous Carbon Scaffold as a Supercapacitor

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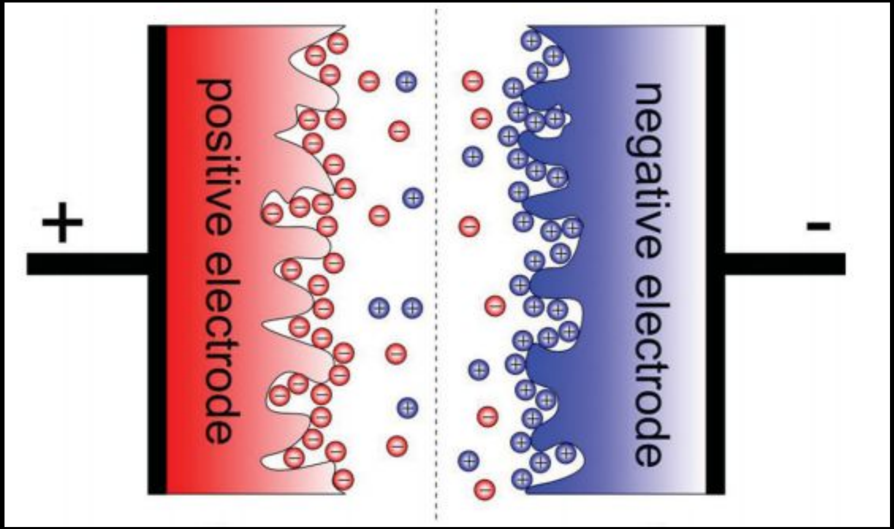
Monica Nguyen Supervisors: Dr. Viola Birss, Chengying (Arlene) Ai

Background

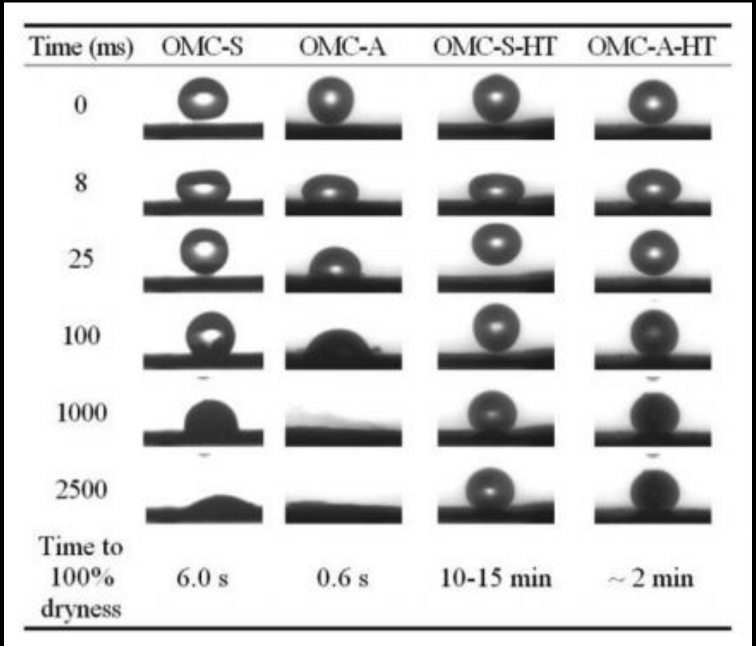
- Global warming crisis requires clean energy alternatives
- Nanoporous carbon scaffold (NCS)
  - Controlled pore structure
  - High surface area
- Battery and fuel cells
- Supercapacitor
  - High power density



Béguin, F. , Presser, V., Balducci, A., Frackowiak, E. (2014). Carbons and Electrolytes for Advanced Supercapacitors. Advanced Materials, 26, 2219–2251. DOI: 10.1002/adma.201304137.

Connection to Birss Research

- Nanoporous carbon materials
- Heat treatment resulted in hydrophobicity
- Wettability impacts materials being developed by the Birss Group



Li, X., Forouzandeh, F., Fürstenhaupt, T., Banham, D., Feng, F., Ye, S., … Birss, V. (2018). New insights into the surface properties of hard-templated ordered mesoporous carbons. *Carbon*, *127*, 707–717. doi: 10.1016/j.carbon.2017.11.049

Research Goals

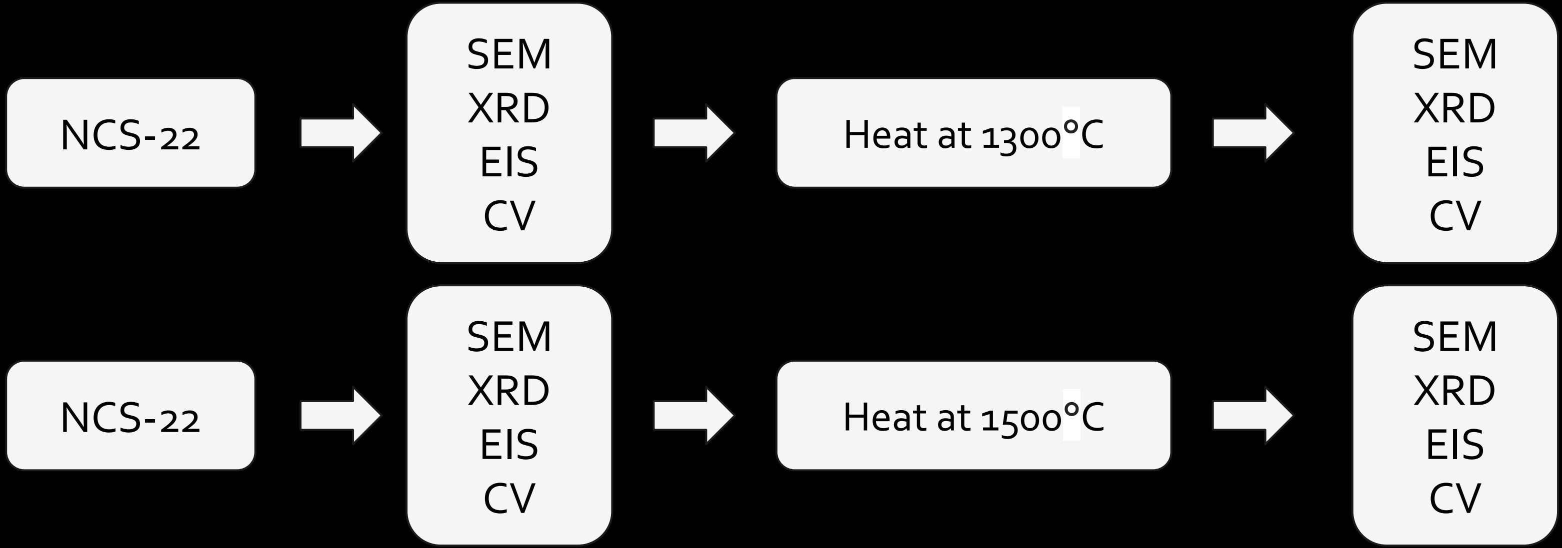
- Determine the effect of heat treatment on the conductivity and electrochemical behavior of NCS-22
- Assemble a supercapacitor and test its performance using the heat-treated NCS-22 electrodes

Overall Plan: NCS-22

- Pore size 22nm

Characterization
- Scanning Electron Microscopy (SEM)
- X-Ray Diffraction (XRD)

Determine Electrochemical Nature
- Electrical Impedance Spectroscopy (EIS)
- Cyclic Voltammetry (CV)

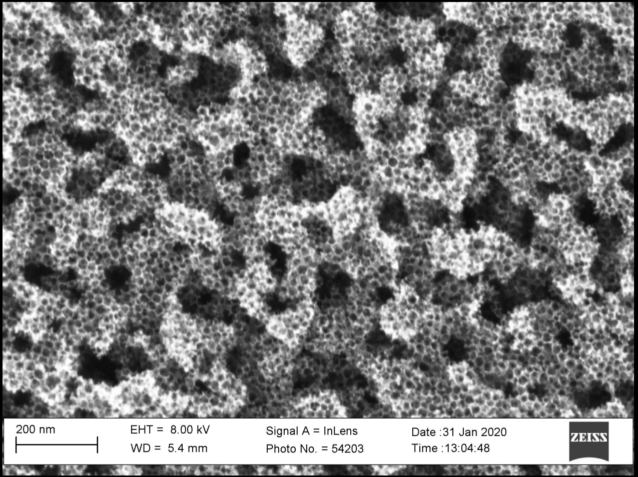


Current Results with NCS-22 Before Heat Treatment

SEM of NCS-22

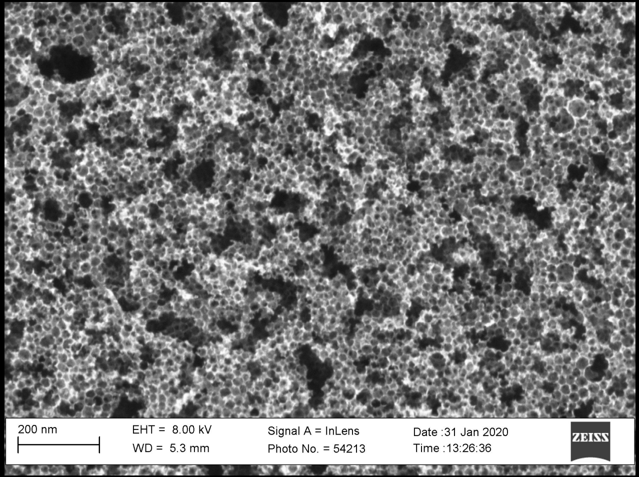
- Under vacuum: electrons can reach sample
- Signal: InLens

200 000x - Top



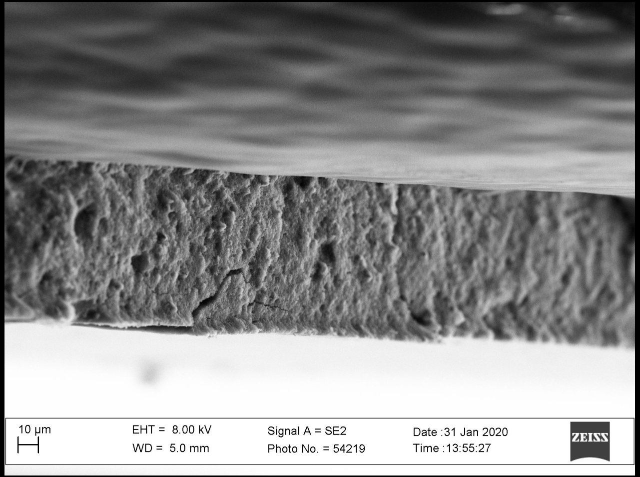
- More rough - gases escaped
- Larger holes ~100nm

200 000x - Bottom



- More smooth - face on glass
- Smaller holes ~50nm

1000x - Side



- NCS-22 ~60µm thick
- Further magnification impeded
- Improper carbon tape adhesion
- Not flat on platform

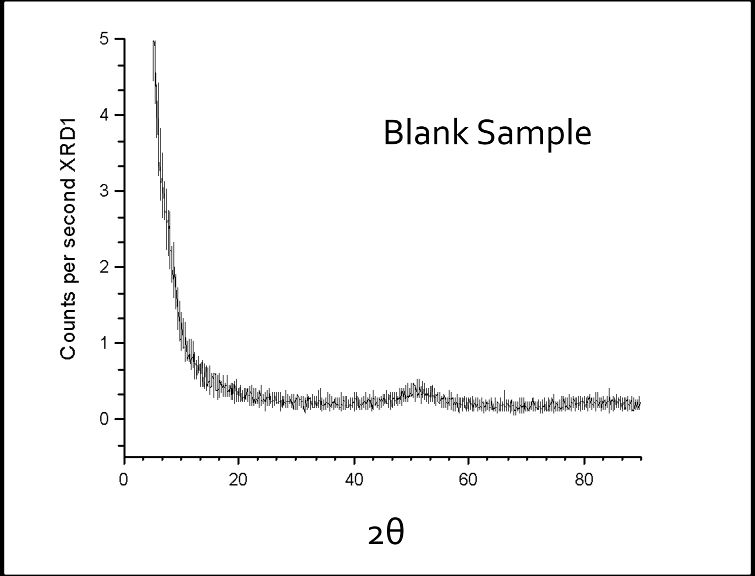
Sources of Holes:

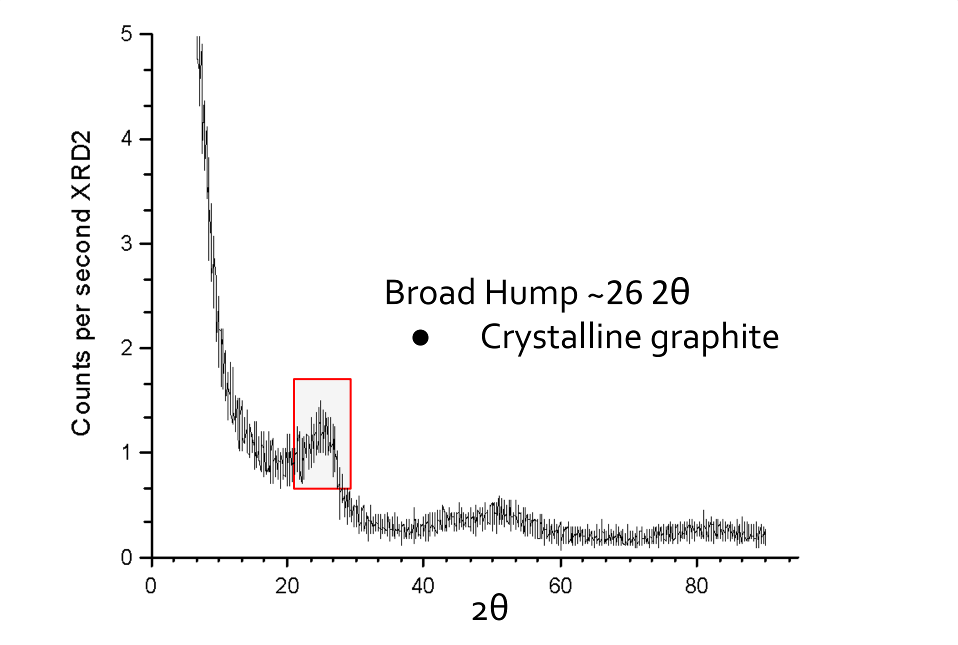
- Trapped hydrogen, oxygen, or organics escaped during heating
- Mesophase pitch melted
- Holes in carbon framework from silica particles that were washed away with NaOH

XRD of NCS-22

Anode: Cu
Scantype: Coupled TwoTheta/Theta
Time Per Step: 96

NCS-22 ground into powder
Vial + acetone

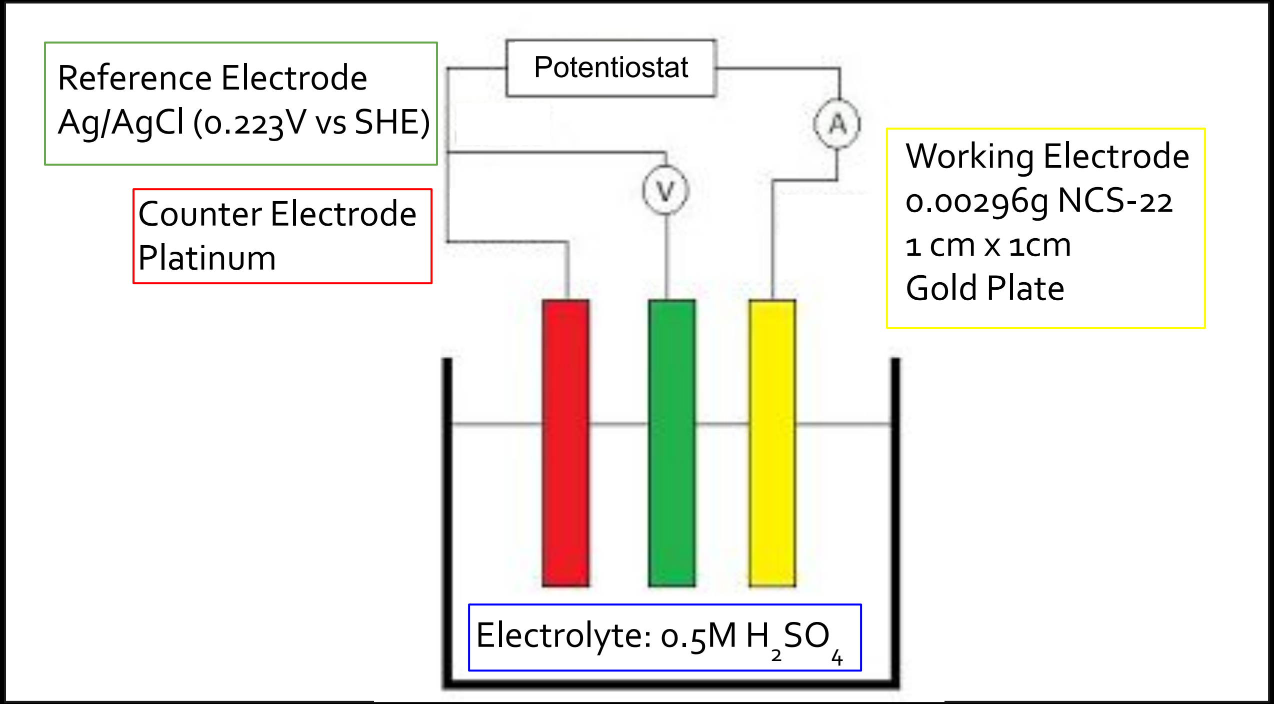




- XRD only detects crystalline substances
- Broad hump shows presence of crystalline graphite
- Amorphous silica may exist but it is not crystalline to produce any peaks

- Heating noncrystalline carbon at high temperatures produces crystals lower in energy

Electrochemical Cell



EIS

Mode = Single sine

E(V) = 0.1770V vs RE

A_m = 10mV

F_i = 100.000 kHz

F_f = 10.000 mHz

CV

E_i (V) = 0.177V

E_1(V) = -0.173V

E_2(V) = 0.877V

E_f (V) = 0.177V

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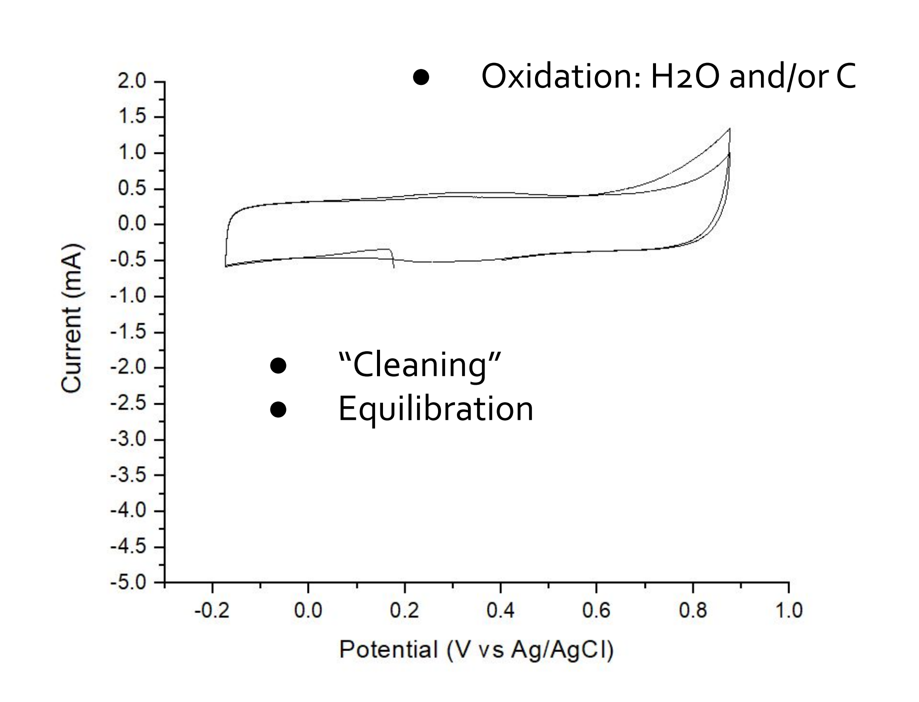
CV

- WE = 1cm² NCS-22 ~60μm thick on gold plate
- RE = Ag/AgCl (0.223V vs SHE)
- CE = Pt
- Potential window = -0.173V to 0.877V



Scan Rate 2mV/s || Capacitance = 57 F

Sample Calculation

C = Q / (ΔV * υ * m)

C = ∫ (I * dE) / (ΔV * υ * m)

Q from integration of curve ⟶ Origin Software

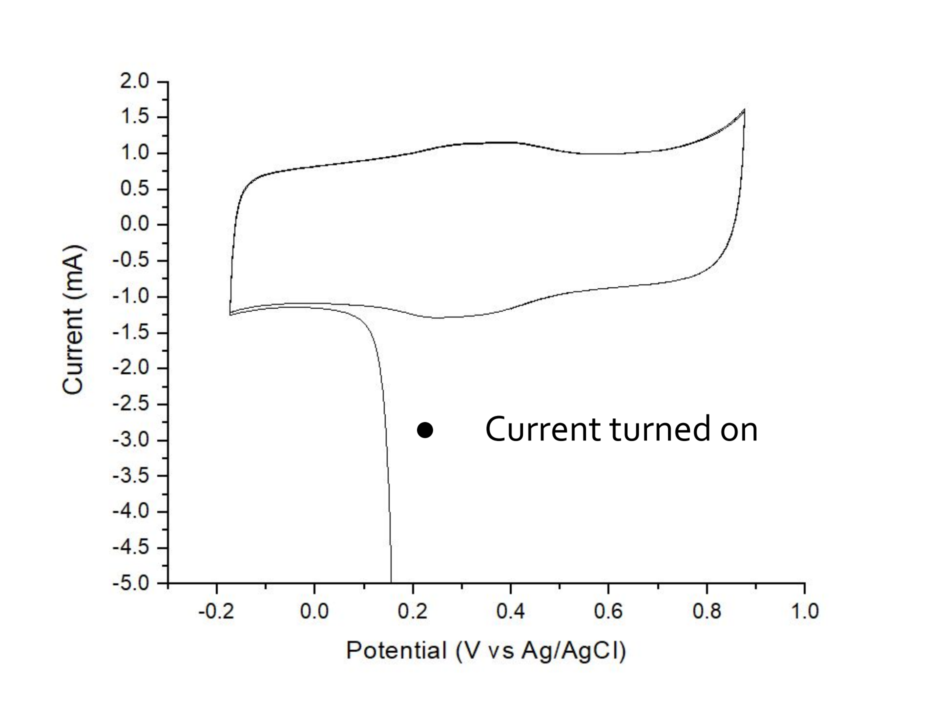
C = 0.26 mA*V / ((0.600V+0.173V) *2mV/s * 0.00296g)

C = 57F

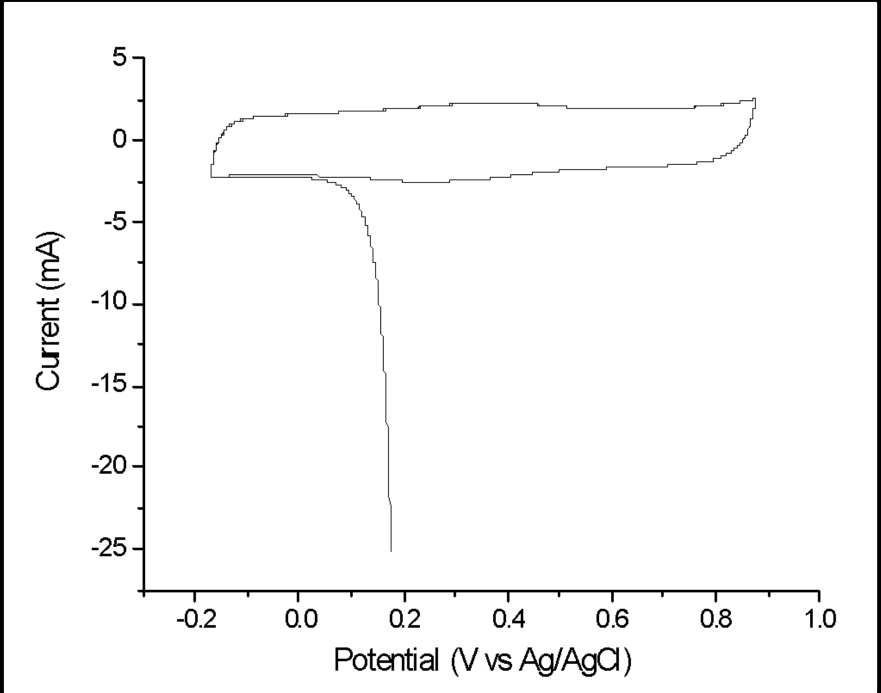
Anodic: -0.173 V to 0.600V
Cathodic: -0.173V to 0.177V

CV

- WE = 1cm² NCS-22 ~60µm thick on gold plate
- RE = Ag/AgCl
- CE = Pt
- Potential window = -0.173V to 0.877V



Scan Rate 5mV/s || Capacitance = 68 F

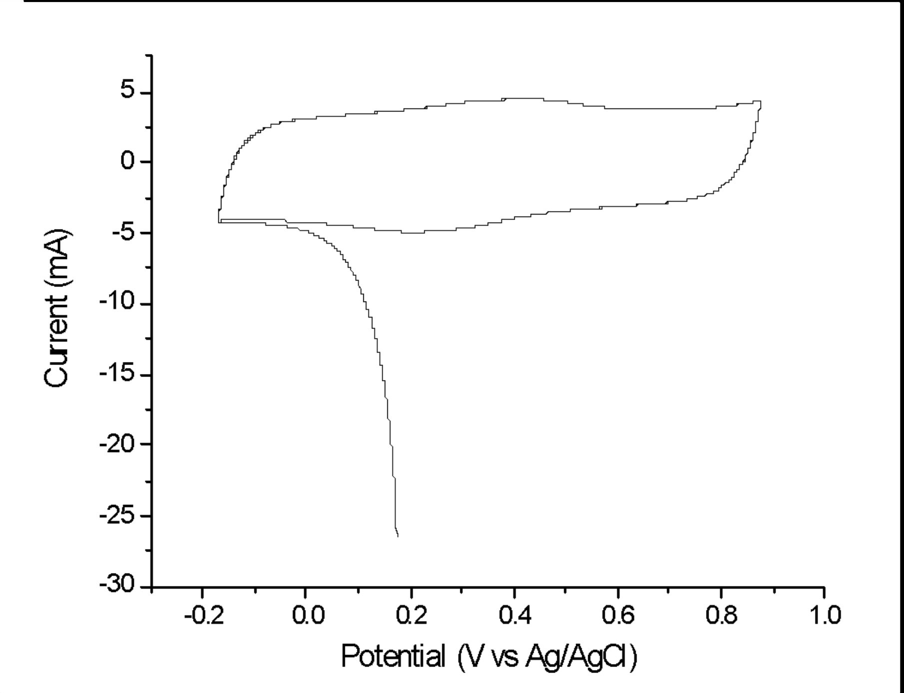


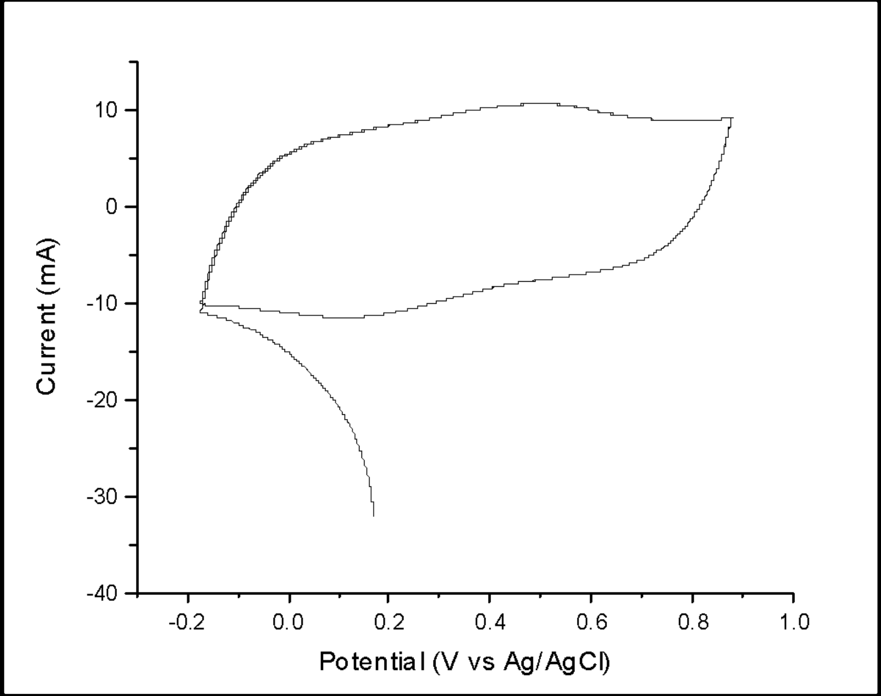
Scan Rate 10mV/s || Capacitance = 65 F

Anodic: -0.173V to 0.877V || Cathodic: 0.4V to 0.6V

CV

- WE = 1cm² NCS-22 ~60μm thick on gold plate
- RE = Ag/AgCl
- CE = Pt
- Potential window = -0.173V to 0.877V





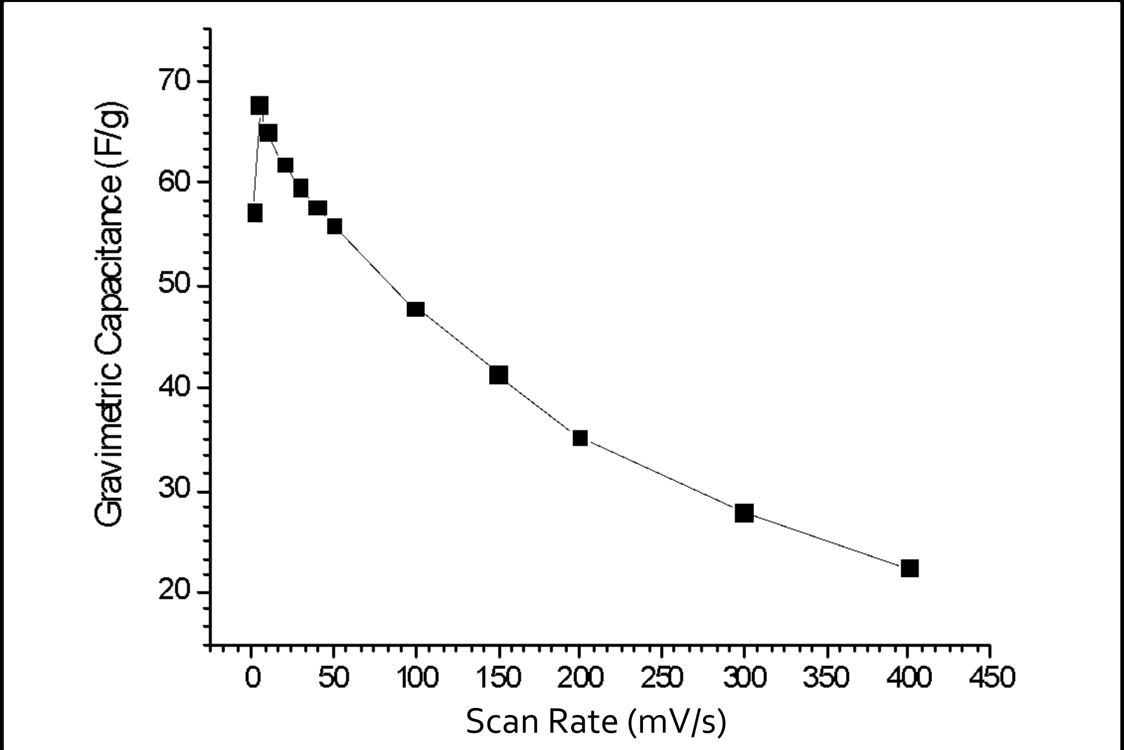
Scan Rate 20mV/s || Capacitance = 62F

Scan Rate 50mV/s || Capacitance = 56F

Anodic: -0.173V to 0.877V || Cathodic: 0.4V to 0.6V

CV

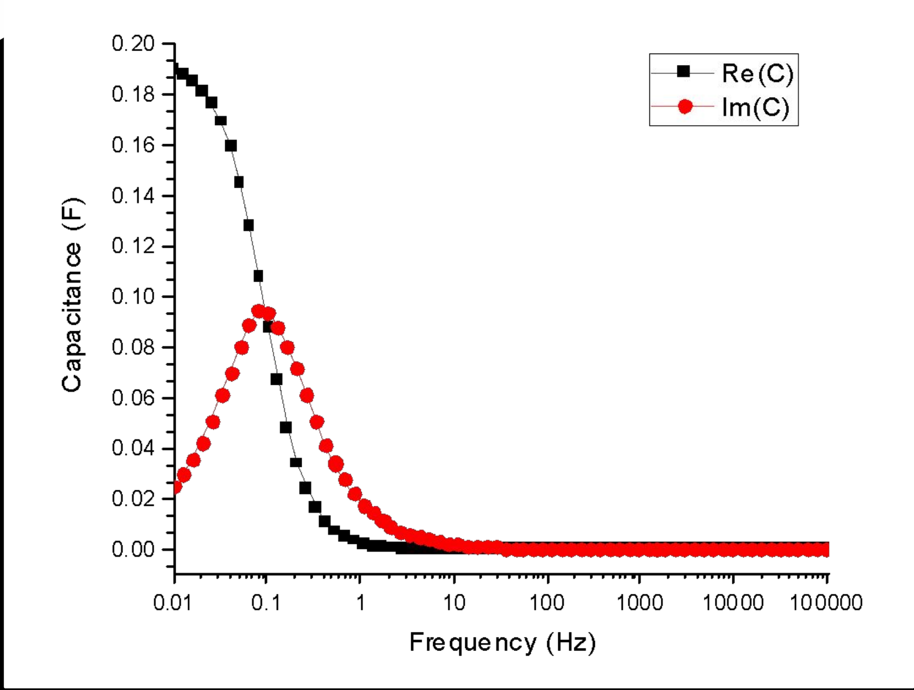
- WE = 1cm² NCS-22 ~60μm thick on gold plate
- RE = Ag/AgCl
- CE = Pt
- Potential window = -0.173V to 0.877V



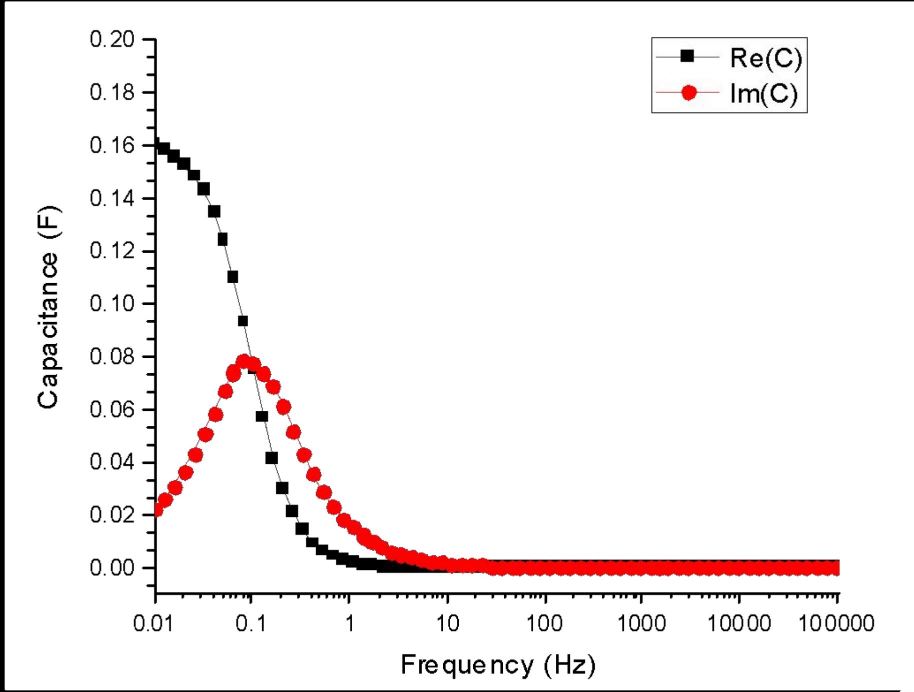
- At low scan rates, there is more time for ions to penetrate pores to produce higher capacitance.

EIS

- WE = 1cm² NCS-22 ~60μm thick on gold plate
- RE = Ag/AgCl
- CE = Pt
- Potential window = -0.173V to 0.877V



EIS before CV



EIS after CV

T = 1 / 0.08192 Hz = 12.2 s

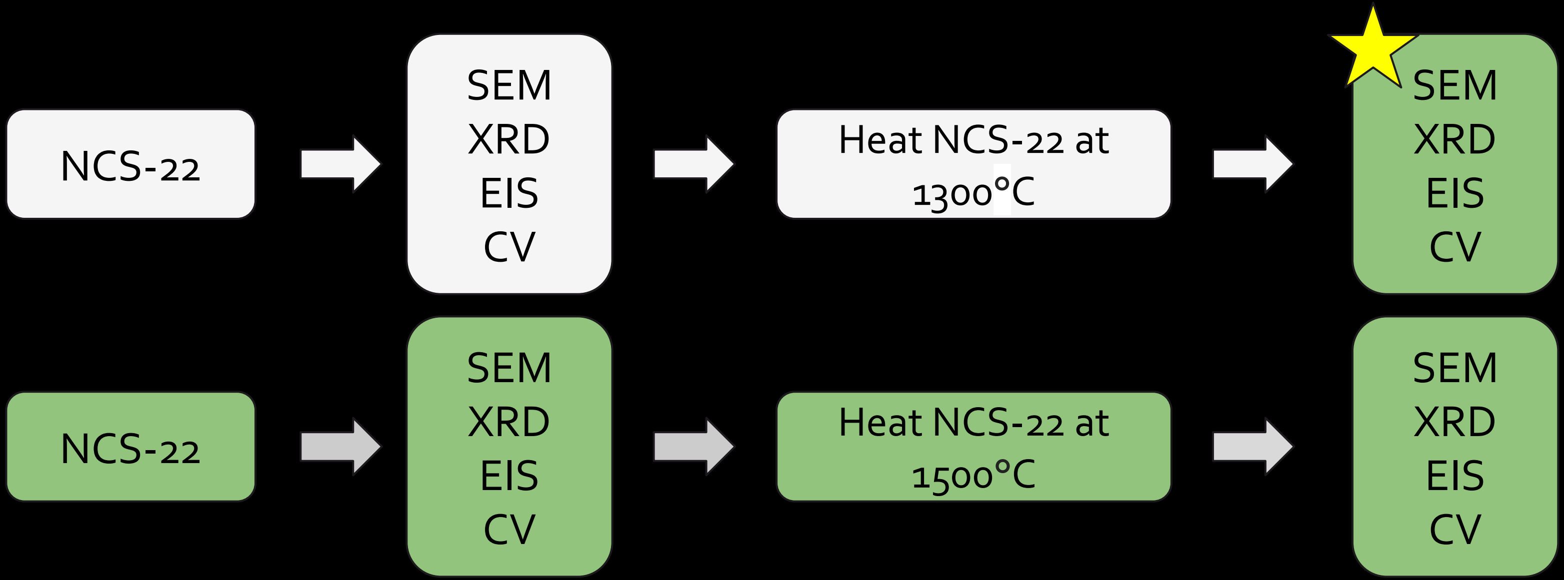
At low frequency, ions can sufficiently penetrate pores. At high frequency, ions 'sweep' past pores.

Conclusion

- SEM assesses quality of material by showing high resolution images at high magnification to show defects and structure
- XRD data allows comparison to literature values to identify crystalline materials
- CV and EIS show that the capacitance of the material is dependant on the time given for ions to penetrate pores

Further research can focus on adjusting the pore size of NCS because this impacts the surface area which may produce higher or lower capacitance values.

Term Plan



- Compare physical and electrochemical characteristics before and after heating NCS-22
- Time permitting, construct a supercapacitor and test its performance using heat-treated NCS electrodes

Acknowledgements

Special thanks to:

- Dr. Viola Birss for supervising this project and giving me the opportunity to learn about current clean energy research
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