Electrochemical Coal to 2-Dimensional Materials (e-Coal2D) Process to Enable Renewable Energy Storage

DE-FE0032275

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Ohio University

U.S. Department of Energy
National Energy Technology Laboratory
Resource Sustainability Project Review Meeting
April 2-4, 2024

Disclaimer

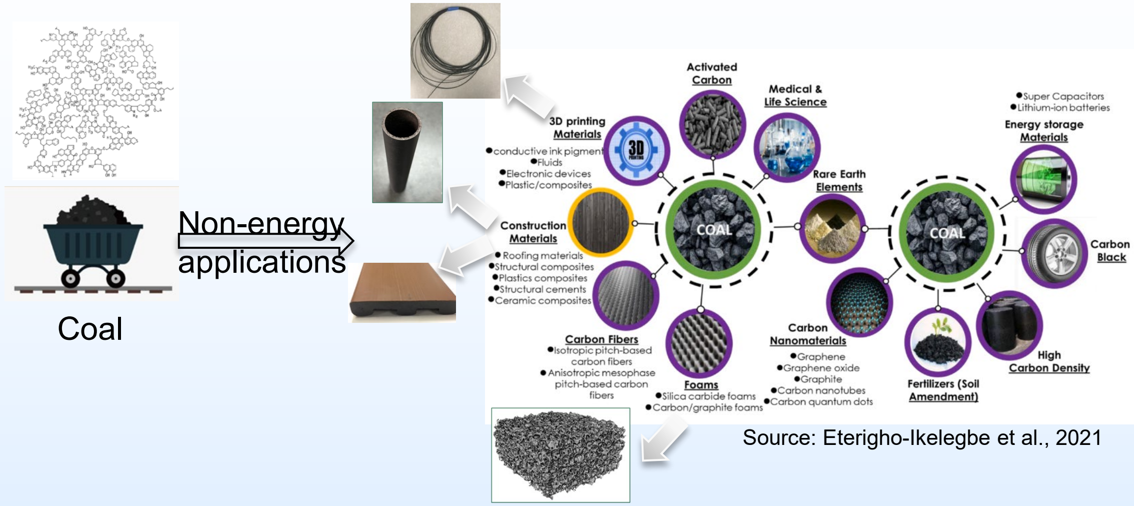
ISEE Overview

Institute Facts

- Faculty: 3
- Staff: 3 (Engineers and scientists)
- Students: 10 GS; 20 UG
- Space: 14,000 ft^2
- Over $20M in external research since 2008

Research Capabilities

- Electrochemical Engineering
- Process Engineering & Design
- Process Modeling & Simulation

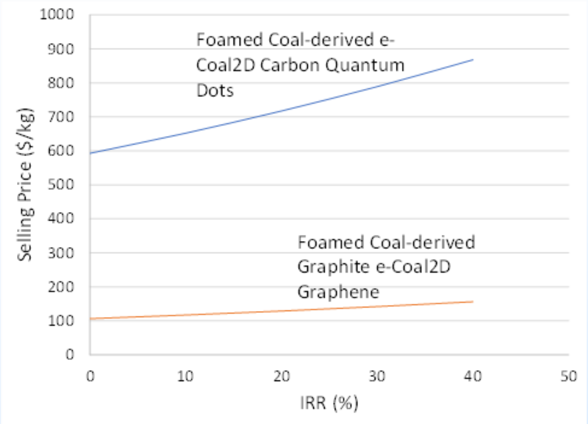


Source: Eterigho-Ikelegbe et al., 2021

Electrochemical Coal to 2-Dimensional Materials (e-Coal2D) Process to Enable Renewable Energy Storage

Overview

- Supercapacitor market is large and growing
- >$470 million in North America, >14% CAGR[1]
- Activated or high surface area carbon is typical electrode material
- Nanoscale materials could enhance capacity



Preliminary Estimated Graphite Selling Prices vs. Internal Rate of Return.

Advantages of Proposed Technology

- Conversion of coal and waste coal into nanoscale materials to stand up a domestic supply chain
- Utilizing nation's coal resources as energy storage materials for supercapacitors
- Diversifying and securing U.S. battery/energy storage supply chains
- Lower graphene prices while further valorizing coal
- Converting rural mining brownfields into energy storage material manufacturing hubs with prevailing wage jobs

[1]Supercapacitor Market by Type (Double Layer Capacitors, Pseudocapacitors, Hybrid Capacitors), Electrode Material (Carbon, Metal Oxide, Conducting Polymers, Composites), Application (Automotive, Energy, Consumer Electronics) – Global Forecast to 2027, https://www.marketsandmarkets.com/Market-Reports/supercapacitor-market-37140453.html

Project Overview

DOE Funding: $998,648

Cost Share: $249,628

Total Project Cost: $1,248,276

Cost Share: 20%

DOE Program Manager: Christian Robinson

Project Performance Dates:

August 1, 2023-July 31, 2025

Project Overview

Project Participants

- Ohio University (Prime Recipient)
  - PI Staser, co-PI Jason Trembly
  - Research Engineers Kody Wolfe and Omar Movil
  - Graduate and undergraduate students
- CONSOL Innovations: Rudy Olson and Natasha Smith
- Capacitech Energy: Joe Sleppy, Isaiah Oladeji, Alisa Silverstein

Overall Project Objectives

- Characterizing electrochemical methods to generate nanoscale carbon materials from coal and waste coal
- Characterizing resulting nanoscale carbon materials
- Evaluating performance of energy storage devices using nanoscale carbon materials
- Meaningful community benefits activities

Project Overview

Budget Period 1 Goals

- Initiate Community Benefits Plan activities
- Develop electrochemical exfoliation process
- Analysis of parameter effects on exfoliation
- Characterize commercial and coal-derived materials

Budget Period 2 Goals

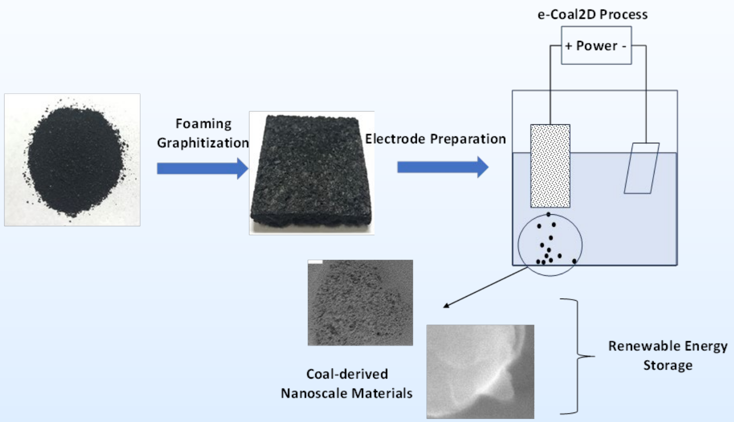
- Evaluate coal-derived materials in supercapacitors
- Complete techno-economic analyses

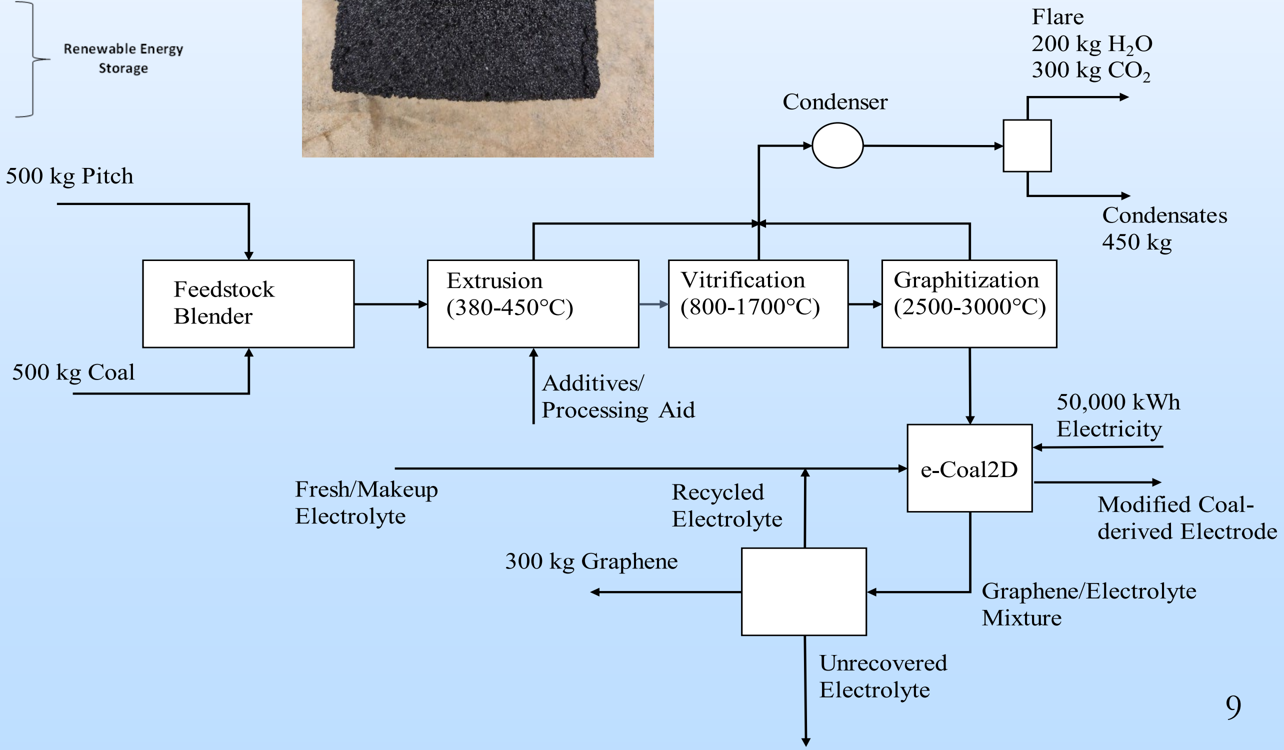
Project Overview

Community Benefits Metrics

	Community Engagement	Educational Outreach	Undergraduate Research Opportunities
Specific	Engages stakeholders from Appalachia in discussions on the environment and ways university/community partnerships can benefit the local communities	Seeks to increase number of Appalachia students enrolled in STEM fields through student engagement	Undergraduate students have the opportunity to benefit this project and gain research experience
Measurable	Number of community members engaged	Number of local area students engaged	Number of undergraduate student researchers
Achievable	Work with local nonprofit organizations and outreach programs to target community engagement.	Work with local school districts. OHIO has already established relationships with local schools.	PI and co-PIs work with undergraduate researchers. Paths are in place for undergraduate student research.
Relevant	Aligns with the broader goal including rural communities in environmental and economic issues	Aligns with the broader goal of increasing low-income, rural, first-generation college student matriculation in STEM fields	Provides undergraduate students, many of whom are rural, first-year students, with research opportunities
Timely	Engage 10 community members in BP 1; 20 by BP 2	Engage 10 area students in BP 1; 25 by BP 2	2-3 student researchers in BP 1; 5 by BP 2
Inclusive	Solicit feedback from community members. Feedback will be used to develop future DEIA plans that are most impactful	Students provide information on barriers to college. Students engage with OHIO representatives to achieve college enrollment.	Undergraduate researchers fully participate in research, provide feedback and ideas for project success
Equitable	Targets marginalized community members from Appalachia and rural areas	Targets underserved students in rural Appalachia	Provides research opportunities for undergraduate students; provides work-study programs for low-income students.

Technology Background





Technology Background

a. Technology Operation
   - Electrochemical exfoliation of coal-derived graphite to nanoscale materials
   - Coal-derived graphite is the electrode
   - Nanoscale materials used for energy storage (capacitors)
   - Electrochemical exfoliation
     - Anodic: coal-derived graphite is positive electrode
     - Cathodic: coal-derived graphite is negative electrode
     - Operation dictates electrolyte (large negative ions for anodic exfoliation, large positive ions for cathodic exfoliation)

b. Fundamental Science
   - Intercalation of large ions between graphene sheets in the graphite electrode
   - Exfoliates sheets to nanoscale materials by electrochemical process

c. Previous Efforts
   - Many approaches to synthesize graphene
   - Exfoliation (chemical, electrochemical, etc.) has good potential for commercial production
   - Has not been done on coal-derived graphite

Technology Background

d. Technical/Economic Advantages
   - Precludes the use of strong acids (i.e., Hummer's method)
   - Electrification of graphene production
   - Can be scaled

e. Technical/Economic Challenges
   - Potential high energy use (depends on graphite quality)
   - Collection/purification may be difficult
   - Yield may be low
   - Coal may introduce toxic species

Technical Approach/Project Scope

a. Experimental Design

- Factors: voltage, electrolyte composition and concentration, exfoliation time, coal rank
- Voltage: +/- 10 V, 5 V, 2.5 V
- Exfoliation Time:< 4 hours, 4 hours, 8 hours 24 hours, 48 hours
  - Use of condenser
- Collection/Purification
  - Centrifugation
  - Vacuum filtration
  - Dialysis
- Characterization
  - Raman spectroscopy
  - XRD
  - Imaging
- Use in Energy Storage Systems (capacitors)

Technical Approach/Project Scope

Milestones

Task	Description	Planned Completion Date	Actual Completion Date	Verification Method
1	Updated Project Management Plan	August 31, 2023	August 22, 2023	Submission of updated PMP to NETL FPM
1	Project Kick-Off meeting held	October 30, 2023	September 28, 2023	Presentation file
1	Preliminary Technology Maturation Plan	October 30, 2023	October 19, 2023	Quarterly report
2	Commercial Graphitic Materials Analysis	January 31, 2024	January 20, 2024	Quarterly report
2	e-Coal2D Product Analysis	April 30, 2024		Quarterly report
GO/NO-GO DECISION		July 31, 2024		Quarterly report
3	Nanoscale Carbon in Small Capacitor Devices	October 30, 2024		Quarterly report
4	Characterization of Modified Foamed Coal	January 31, 2025		Quarterly Report
3	Nanoscale Carbon in Flexible Capacitor Devices	April 30, 2025		Quarterly Report
5	TEA, LCA and Market Analyses	July 31, 2025		Final report

Technical Approach/Project Scope

Project Success Criteria

- Demonstrate graphene and carbon quantum dots with similar properties to other graphene and carbon quantum dots (those produced by, for example, Hummers' method or hydrothermal procedures) can be manufactured from foamed coal materials by an electrochemical process,
- Establish that e-Coal2D products can enhance the capacity of supercapacitor devices over the business-as-usual case,
- Demonstrate high-value potential for e-Coal2D products with a significant cost reduction over business-as-usual prices for these materials produced by other methods,
- Verify e-Coal2D products generated from foamed coal materials are occupationally and environmentally safe, and
- Track common contaminants in coal through the life of these materials

Technical Approach/Project Scope

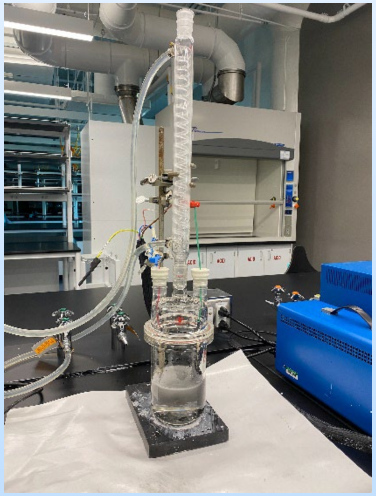
Project risks and mitigation strategies

Perceived Risk	Risk Rating (Low, Med, High) Probability	Impact	Overall	Mitigation/Response Strategy
Financial Risks:				
Insufficient cost share	Low	Low	Low	OHIO and project partners have committed appropriate cost share for this project. Commitment letters have been provided.
Cost/Schedule Risks:				
Project Overspending	Low	Low	Low	ISEE's administrative associate continuously tracks project spending via OHIO's Oracle system. Further, project spending is reviewed on a monthly basis by OHIO Grants Accounting.
Schedule Delays	Low	Low	Low	OHIO has all necessary facilities/equipment/staff available to execute project.
Technical/Scope Risks:				
e-Coal2D Operating Costs	Med	Med	Med	Utilize readily available coal materials and low-cost electricity for production of nanoscale carbon materials
e-Coal2D Product Quality	Med	Med	Med	Preliminary results indicate that e-Coal2D products possess properties that are consistent with graphene and carbon quantum dots. Operating parameters can be controlled to influence product properties.
Supercapacitor Device Performance	Med	High	High	Producing supercapacitor devices using e-Coal2D materials without the required energy storage capabilities would significantly hinder demand. Supercapacitor devices will be evaluated using the e-Coal2D materials and the process will be modified to target desired energy storage properties.
Product Cost	Low	Med	Med	Graphene and carbon quantum dots are currently too expensive for high industrial impact. Coal-based materials synthesized by low-cost electrochemical methods will be evaluated for improved process economics.

Progress and Current Status of Project

Synthesis Equipment

- Batch electrochemical reactor
- Coal-derived graphite positive electrode
- Pt or platinized Ti mesh counter electrode
- Reactor voltage controlled by potentiostat with a power booster
- Condenser prevents evaporation of electrolyte



Collection/Purification

- Centrifugation
- Filtration
- Dialysis

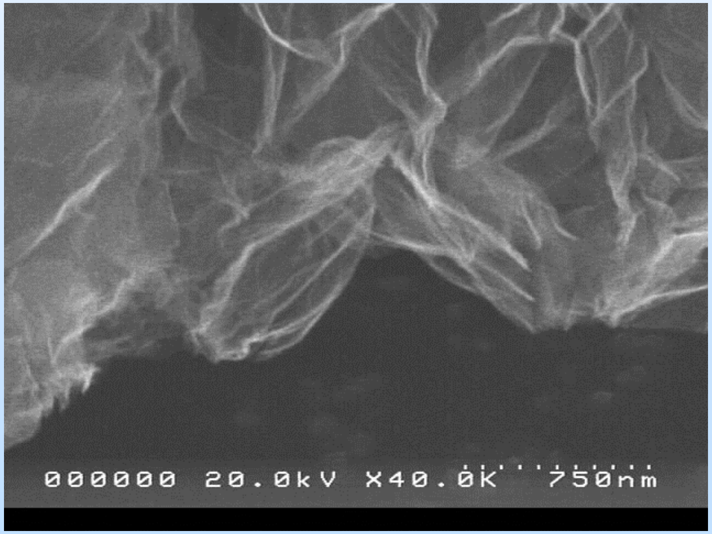
Operation

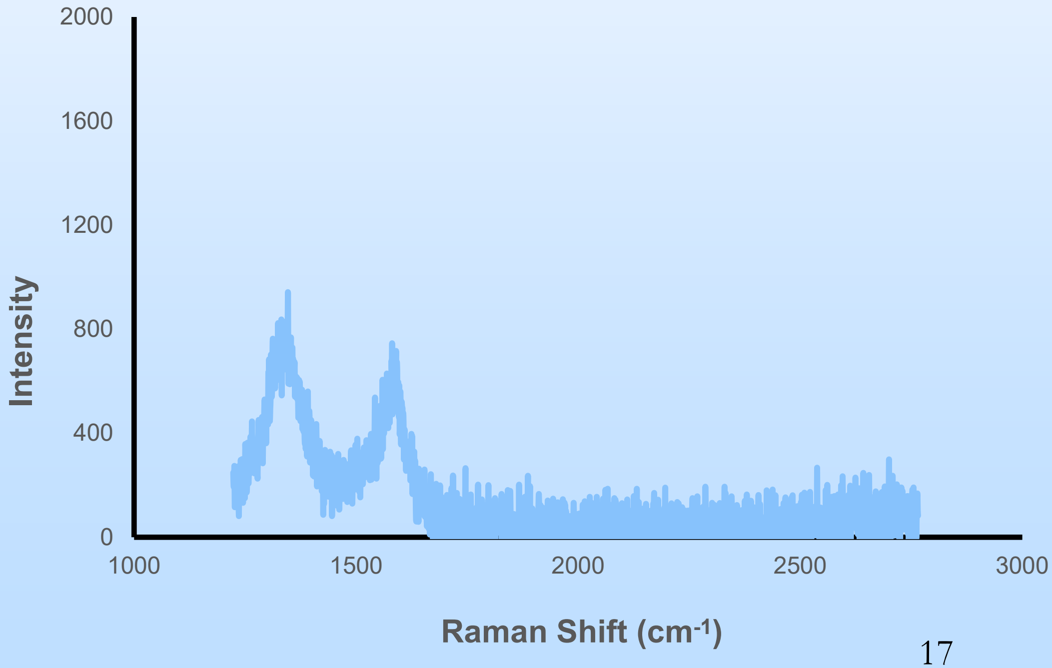
- Original focus was anodic exfoliation in Na_2SO_4
- Have expanded to cathodic exfoliation in KCl

Progress and Current Status of Project

Commercial Nanoscale Materials: MSE Supplies

- FESEM imaging
- Raman spectroscopy

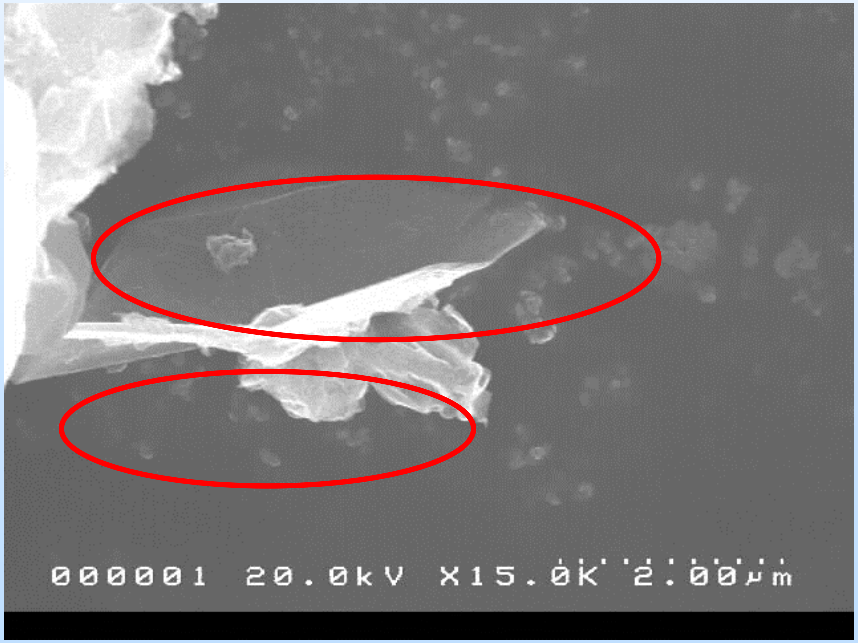


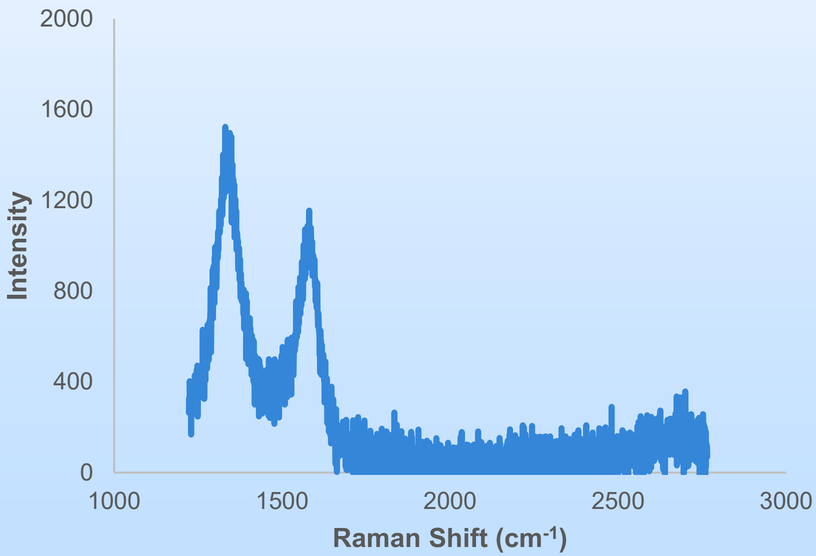


Progress and Current Status of Project

Coal-derived Nanoscale Materials: Anodic Exfoliation

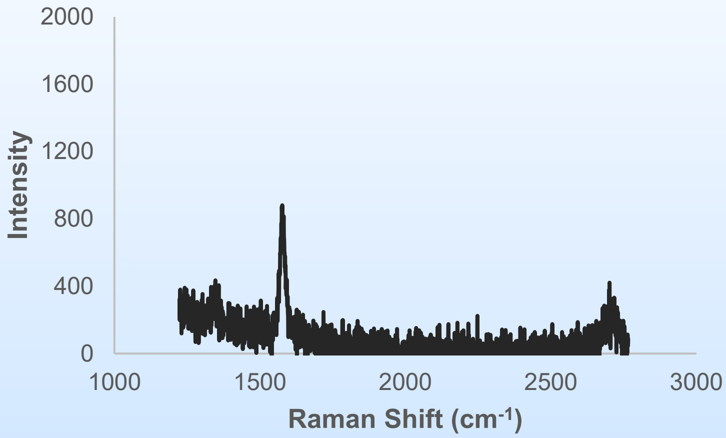
- FESEM imaging
- Raman spectroscopy

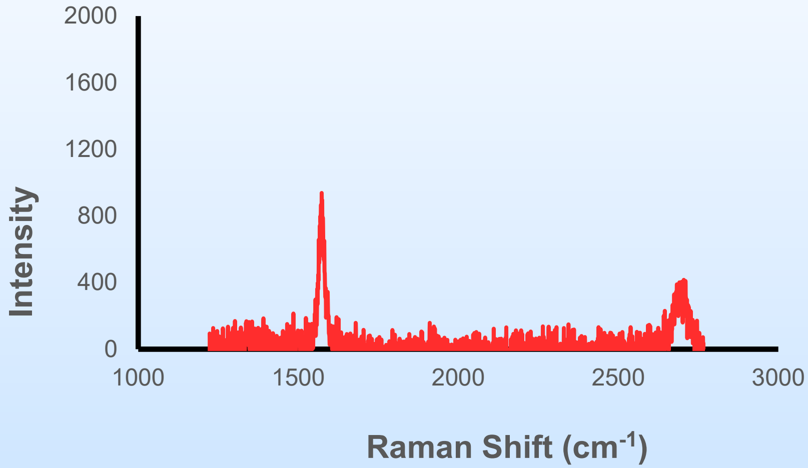




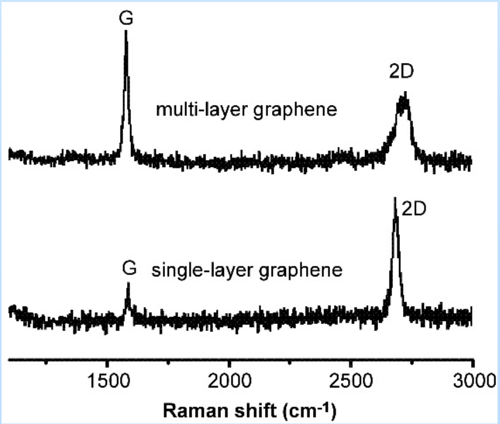
Progress and Current Status of Project

Commercial Nanoscale Materials: Graphene Supermarket





Coal-derived Nanoscale Materials: Cathodic Exfoliation



LibreTexts Chemistry: https://chem.libretexts.org/Bookshelves/Analytical_Chemistry/Physical_Methods_in_Chemistry_and_Nano_Science_%28Barron%29/08%3A_Structure_at_the_Nano_Scale/8.07%3A_Characterization_of_Graphene_by_Raman_Spectroscopy

Progress and Current Status of Project

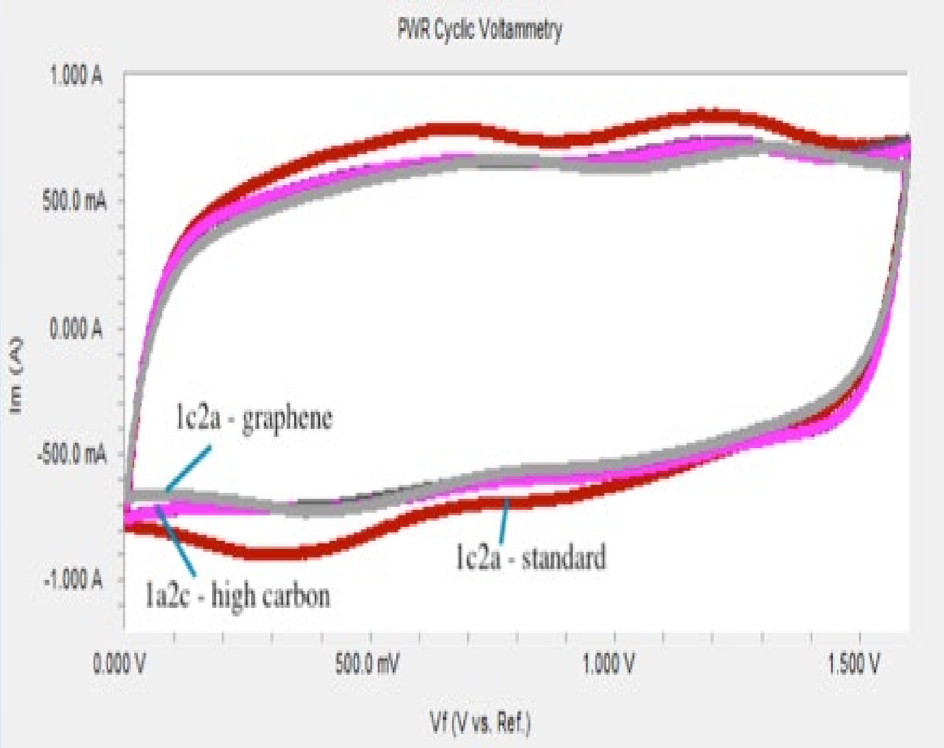
Synthesis and Characterization

- Commercial graphene from MSE Supply and Graphene Supermarket have been characterized by FESEM, TEM, XRD, Raman
- Anodic exfoliation of coal-derived graphite has been carried out in 0.1 and 1.0 M Na_2SO_4 at 5 V and 10 V for 1 hour, 4 hours, 8 hours, 12 hours, 24 hours, 48 hours
  - Likely forms a multi-layer graphene due to absence of 2D peak at Raman shift of ~2700 → similar to commercial graphene purchased from MSE Supply
- Cathodic exfoliation of coal-derived graphite has been carried out in 2 M KCl at 5 V for 1 hour
  - Raman spectrum similar to few-layer graphene

Progress and Current Status of Project

Supercapacitor Accomplishments

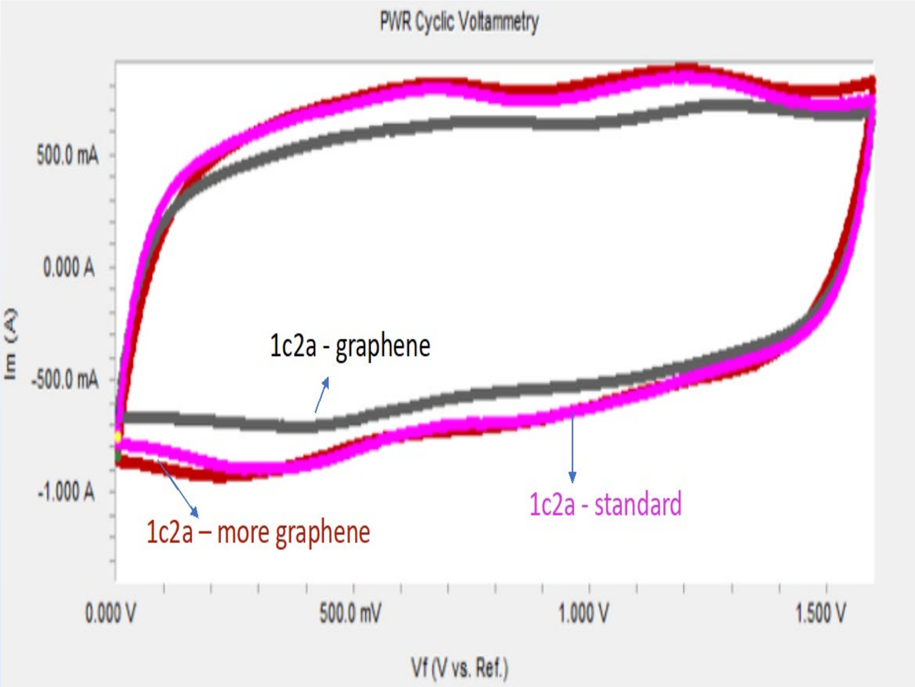
- Baseline established with commercial graphene
- Subawardee Capacitech's standard evaluation system



	1c2a -standard		1c2a -high carbon		1c2a -graphene	
	Capacitance (F)	ESR (Ω)	Capacitance (F)	ESR (Ω)	Capacitance (F)	ESR (Ω)
10mA	23.06	0.244	20.552	0.077	18.86	0.036
50mA	18.627	0.089	16.629	0.041	15.035	0.032
100mA	16.928	0.071	15.339	0.038	13.876	0.035
250mA	15.457	0.06	13.891	0.035	12.699	0.031
500mA	14.842	0.055	13.11	0.033	12.118	0.032
750mA	14.513	0.053	12.674	0.032	11.825	0.032
1000mA	14.257	0.05	12.366	0.03	11.636	0.033

"graphene" = 2.6 wt%

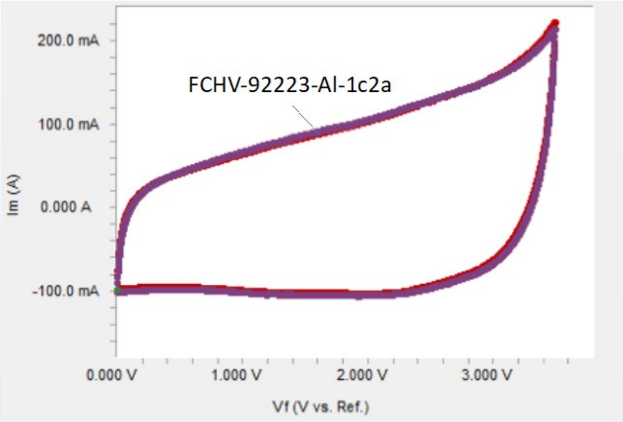
Progress and Current Status of Project

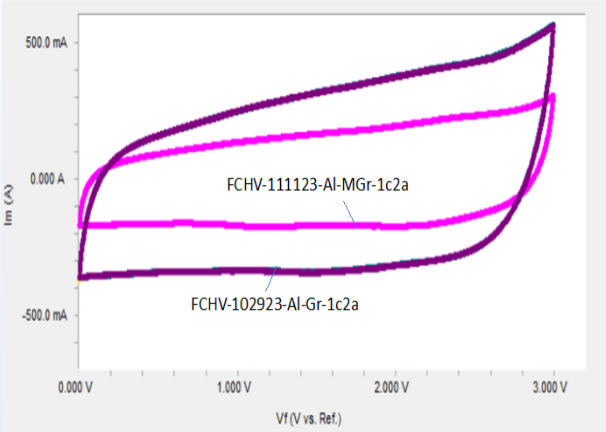


"graphene" = 2.6 wt%
"more graphene" = 4%

	1c2a -standard		1c2a -graphene		1c-2a - more graphene	
	Capacitance (F)	ESR (Ω)	Capacitance (F)	ESR (Ω)	Capacitance (F)	ESR (Ω)
10mA	23.06	0.244	18.86	0.036	24.538	0.256
50mA	18.627	0.089	15.035	0.032	20.165	0.096
100mA	16.928	0.071	13.876	0.035	18.645	0.075
250mA	15.457	0.06	12.699	0.031	17.016	0.061
500mA	14.842	0.055	12.118	0.032	16.186	0.057
750mA	14.513	0.053	11.825	0.032	15.7	0.055
1000mA	14.257	0.05	11.636	0.033	15.353	0.053

Progress and Current Status of Project





"graphene" = 2.6 wt%
"more graphene" = 4%

	FCHV-92223-AL-1c2a no graphene		FCHV-102923-AL-GR-1c2a graphene		FCHV-111123-AL-MGR-1c2a more graphene	
	Capacitance (F)	ESR (Ω)	Capacitance (F)	ESR (Ω)	Capacitance (F)	ESR (Ω)
10mA					4.104	0.48
50mA	2.017	0.433	7.701	0.213	2.789	0.428
100mA	1.707	0.409	6.382	0.267	2.249	0.407
250mA	1.342	0.359	4.974	0.262	1.609	0.361
500mA	1.144	0.333	4.035	0.244	1.256	0.352
750mA	1.052	0.305	3.467	0.231	1.087	0.334
1000mA	1.007	0.302	3.077	0.227	0.989	0.309

Progress and Current Status of Project

Synergistic Opportunities

- Utilizes coal and waste coal as energy storage materials
- Brings together company focused on valorizing coal with a supercapacitor company and a university developing advanced energy storage materials
- Connects supplier to end-user for technology development
- Work with local schools and community organizations to advance Community Benefits Plan initiatives

Plans for future testing/development/ commercialization

a. In this project
   - Continued characterization of coal-derived materials (April 30)
   - Evaluation of coal-derived materials in capacitors (BP 2)
   - Determine yield and energy requirements (BP 2)

b. After this project (i.e., next project)
   - Evaluate in other applications (batteries, etc.)

c. Scale-up potential
   - Exfoliation is a top-down approach
   - Greater scale-up potential than bottom-up synthesis
   - Large coal-derived electrodes can be used to generate large quantities

Outreach and Workforce Development Efforts or Achievements

Outreach

- Presentation at Trimble High School
  - School district in rural Athens County
  - Many students would be first-generation if they attended college
  - Plans in April for students to engage in hands-on electrochemistry activities
- Presentation at Regeneration
  - Engaged local community stakeholders

Workforce Development

- Graduate students
- Undergraduate students

Summary Slide

Key Findings

- Exfoliation dependent on operating conditions (potential, electrolyte, time) and on operating mode (anodic vs. cathodic exfoliation)
- Both modes result in nanoscale materials
- Anodic exfoliation likely results in multi-layer graphene while cathodic exfoliation results in few-layer graphene
- Commercial graphene can improve capacitor performance

"Take-away" Message

Electrochemical exfoliation of coal-derived graphite results in nanoscale materials with characteristics similar to commercial materials.

Acknowledgements

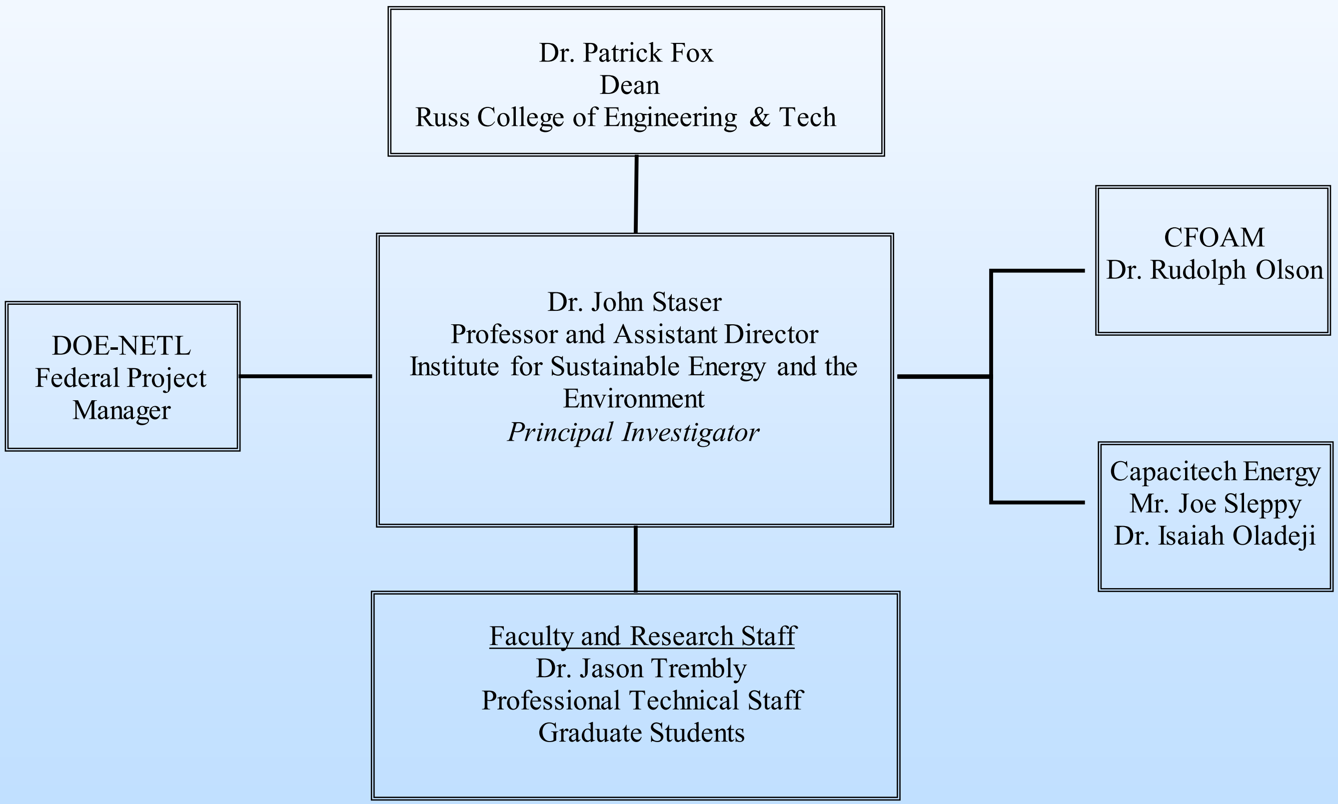
Research Team at Ohio University, CONSOL Innovations, Capacitech

- Jason Trembly, Kody Wolfe, Omar Movil, Rudy Olson, Natasha Smith, Isaiah Oladeji, Alisa Silverstein
- Undergraduate and graduate students
- Martin Kordesch (Ohio University Dept. of Physics and Astronomy)
- David Ingram (Ohio University Dept. of Physics and Astronomy)

Department of Energy Support

Appendix

Organization Chart

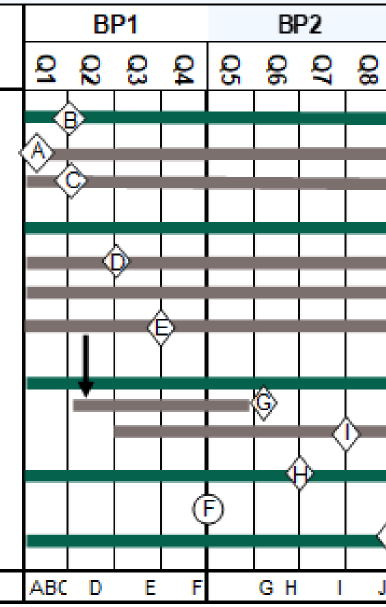


Organization Chart

- Ohio University (Prime Recipient)
  - Oversees project management and planning
  - Develops the electrochemical exfoliation process
  - Lead in material characterization
  - Constructs small capacitors for fast screening of materials
- CONSOL Innovations
  - Develops coal-based electrodes
  - Leads in potential toxic chemical tracking
- Capacitech Energy
  - Evaluates materials in commercial capacitors
  - Will develop a product sheet

Gantt Chart

Task	Responsible Organizations
Task 1.0 – Project Management and Planning	OHIO
Subtask 1.1 - Project Management Plan	OHIO
Subtask 1.2 - Technology Maturation Plan	OHIO
Task 2.0 – Conversion of Coal to Graphitic Materials	OHIO, CF
Subtask 2.1 - Commercial Graphitic Materials Analysis	OHIO, CF
Subtask 2.2 - e-Coal2D Operational Variables Analysis	OHIO
Subtask 2.3 - e-Coal2D Product Analysis	OHIO
Task 3.0 – Characterization of Nanoscale Carbon Products	OHIO, CE
Subtask 3.1 - Use of Nanoscale Carbon Products in Small Capacitor Devices	OHIO
Subtask 3.2 - Use of Nanoscale Carbon Products in Flexible Capacitor Devices	CE
Task 4.0 – Characterization of Modified Foamed Coal	OHIO, CF
Task 5.0 – Techno-economic (TEA) and Life-Cycle Analyses (LCA) of e-Coal2D Process	OHIO, CF, CE
Milestone Log	



Ohio University (OHIO), CFOAM (CF), Capacitech Energy (CE)

Milestones: A: Updated Project Management Plan; B: Project Kickoff Meeting; C: Preliminary Technology Maturation Plan; D: Commercial Graphitic Materials Analysis; E: Initial e-Coal2D Product Analysis; F: GO/NO-GO DECISION POINT; G: Evaluation of e-Coal2D Products in Small Capacitor Devices; H: Analysis of Modified Foamed Coal Electrodes; I: Comparison of e-Coal2D Modified Flexible Capacitors to Business-As-Usual Baseline; J: TEA, LCA and Market Analyses