

Office of ENERGY EFFICIENCY & RENEWABLE ENERGY

AMMTO & IEDO JOINT PEER REVIEW

May 16th-18th, 2023

Washington, D.C.

Scale-up Production of Graphene Monoxide for Next-Generation LIB Anodes | AMMTO

Trevor Dzwiniel, Argonne National Lab; Carol Hirschmugl, COnovate WBS# 2.1.0.442 AMR Review

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- Scale eCOphite[™] anode material , a novel product discovered by COnovate with better performance than graphite in lithium-ion batteries.
- This fits AMMTO roles with a focus on *applied* R&D of an innovative new anode material for LIB's working towards the broader DOE goal of U.S. energy storage manufacturing.
- **Barriers:** Scaling patented eCOphite materials from milligram lab bench to 10's kg scale using continuous methods for ton scale production while retaining battery performance.
- **Impact:** eCOphite material is a drop-in replacement for graphite anodes made from domestic bio-sourced feedstocks, delivering low-cost, low-carbon footprint solution with higher capacity and faster charging.

Project Outline

Innovation: Scale up of eCOphite material to multi-kilo scale **Project Lead:** Trevor Dzwiniel, Argonne National Laboratory **Project Partners:** Carol Hirschmugl, COnovate, Inc.

Timeline: 6/1/21 to 7/31/23, 90% complete **Budget:** \$600K total (ANL), 50% cost share (COnovate)

	FY21 Costs	FY22 Costs	FY23 Costs	Total Planned Funding
DOE Funded	\$101K	\$192K	\$307K	\$600K
Project Cost Share	\$73K	\$429K	\$98K	\$600K

End Project Goal: Successfully demonstrate eCOphite material production at pilot scale with 10 kg volume production and enabled enhanced LIB performance.

Background & Strategic Approach

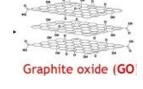
Graphene Monoxide Versus Other Carbon-oxygen Structures

Graphite

Reduced graphene

oxide (RG-O)

suspension





Graphene Monoxide Layers in **COphite** Material

Potts et al., Composites Sci. Tech. 74 (2013)166

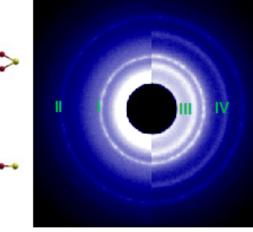
Graphene Monoxide is distinct from Graphite Oxide, Graphene Oxide and reduced Graphene Oxide.

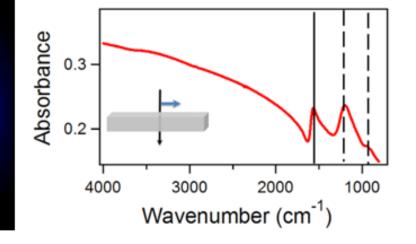
Graphene Monoxide discovered at nanograms and scaled to milligrams before 2020.

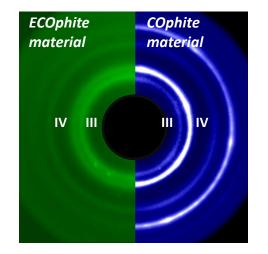
Graphene Monoxide confirmed with diffraction and spectroscopy.

Mattson, E.C., et. Al., ACS Nano 5(12), 9710-9717 (2011). Hirschmugl, C., et. al. U.A. Pat Appln Nos. PCT/US21/13274 & USPTO17148010 (2021).

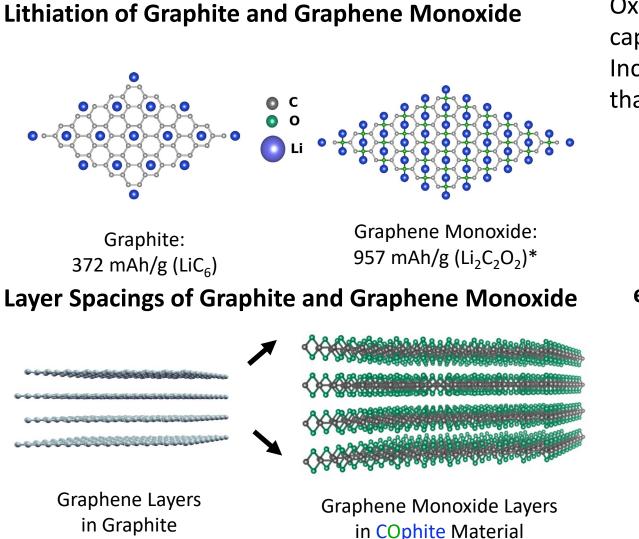
Evidence for Graphene Monoxide in COphite[™] and eCOphite Materials







Background & Strategic Approach

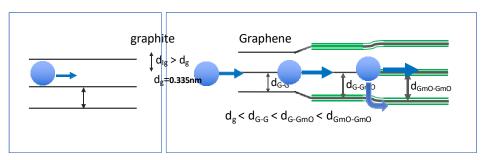


Oxygen Functional groups lead to higher lithium capacity than graphite.

Increased Interlayer spacing enables faster charging than graphite.



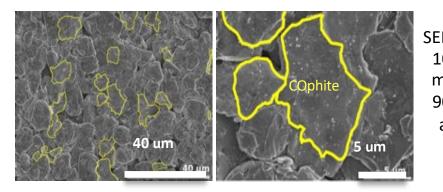
eCOphite and COphite Material Composites



*D Radevych, M Gajdardziska-Josifovska, C Hirschmugl, M Weinert, J. Phys. Chem. C 125(2021)11820, Patent No. 9,236,633/B2 (2016).

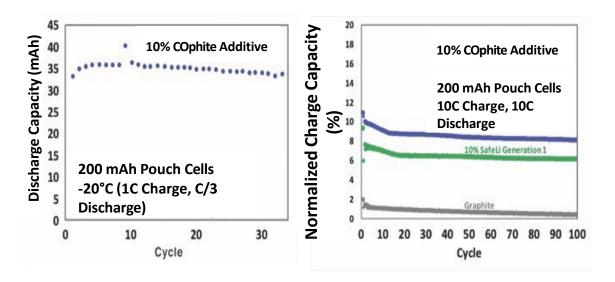
Background & Strategic Approach

COphite Drop-in Additive in Electrodes



SEM images of 10% COphite material with 90% Graphite active anode material

Enhanced Lithium-Ion Battery Performance



• COphite material compatible with existing conventional anodes.

- COphite material drop-in additive.
- TRL: 4 (Demonstrated Minimum Viable Product).
- Anodes of 90% Graphite & 10% COphite Materials Demonstrated 2X Capacity, 6X Faster Charging.

Low Temperature Performance and Safety



Graphite w/Li deposition

graphite + 10% COphite Material w/ minimal Li deposition



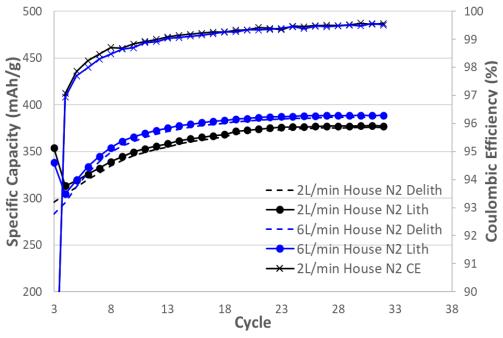
Separators after cycling 200 mAh pouch cells at 20°C (10 cycles) followed by 0°C (100 cycles)

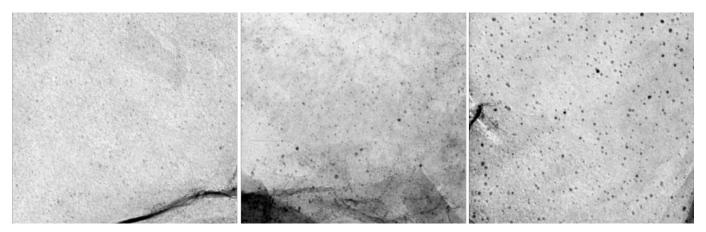
Results and Achievements

Initial research to assess calcination process variable effects on material.

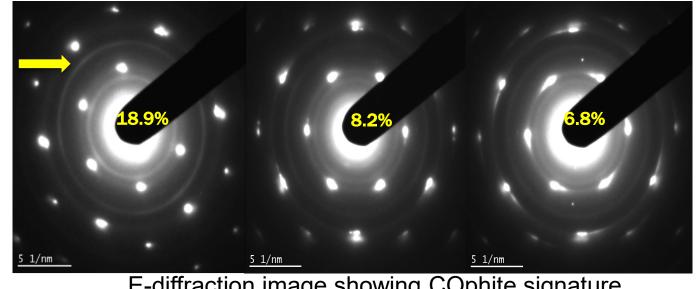
- Run using 1" tube furnace. •
- Flow rate and temperature • predominant factors.

House Nitrogen Flow Rate Comparison C rate: 1C





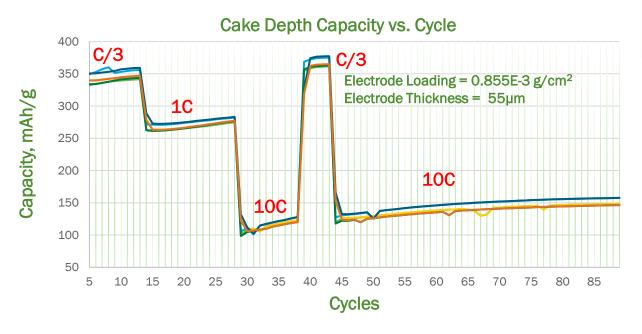
SEM image for flow rate of 6, 4, 2 L/min effect



E-diffraction image showing COphite signature

Results and Achievements

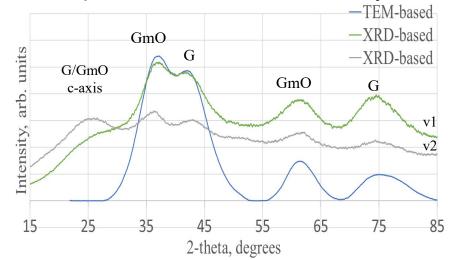
- Scale-up to 3" and 6" tube furnace smooth.
 - Met mid-project goal of 100g scale.
 - No cake depth issues (XRD, cycling).
 - Tube furnace outperformed box furnace.
- Work on electrodes/ match industry std.



COphite blend vs Li half cell. Cycled at: $3 \times C/20$; $10 \times C/3$; $15 \times 1C$; $10 \times 10C$; $5 \times C/3$; $100 \times 10C$ from 10mV-2V.

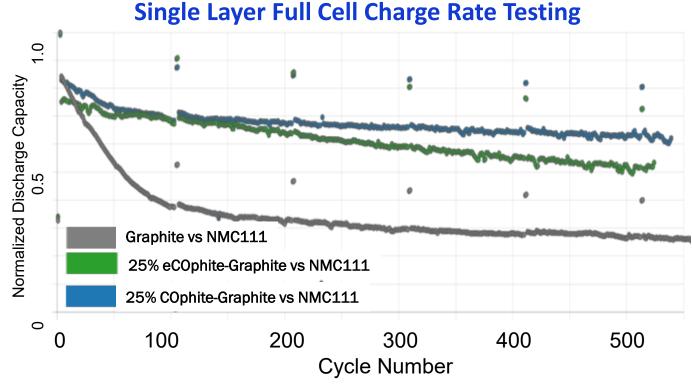


COphite- Based Materials Analysis



Results and Achievements

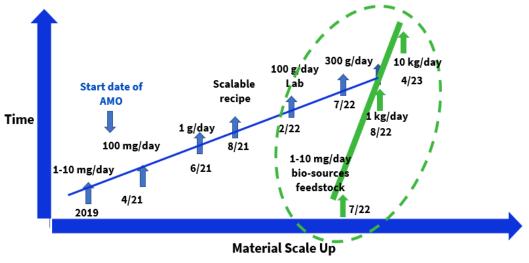
• Final scale goals realized with continuous processing.



Charge Rate testing: Graphite/ (e)-COphite material blend electrodes vs NMC111 2C charge, C/2 Discharge. 2.0V to 4.3V for (e)-COphite based cells, 3.0-4.3V for Graphite Cells @ 21°C.



GREAT PROGRESS ON DE-RISKING SCALE UP



3rd party battery validation: Material Quality HIGH

Future Work, Technology Transfer, & Impact

Future Work:

10kg continuous processing at MERF and Harper and 2Ah batteries.

Technology Transfer:

COnovate's Commercialization Plan is outlined to the right.

Impact:

This project supported the scale-up of eCOphite material manufacturing from mgs to multi-kilos using proven scalable

- 2023 2024 2025 2026 2027-2032 2022 Scaling to 10 kg/day Commercialization Scaling to 1 KTA Expanded Adoption 100 KTA plant \$5M in funding \$25M+ funding \$370M+ funding 10 kg/day of eCOphite • 3 T/day of eCOphite 100 KTA eCOphite material - continuous material production material for 175 GWh scalable process Validating battery of LIBs Validating 2Ah+ cells packs
- processes. The project transitioned to domestic feedstocks that can be leveraged into a new domestic manufacturing capability. This fits AMMTO roles with a focus on *applied* R&D of an innovative new anode material for LIB's working towards the broader DOE goal of U.S. energy storage manufacturing.

CONOVATE'S COMMERCIALIZATION PLAN

Questions?

Scale-up Production of Graphene Monoxide for Next-Generation LIB Anodes | AMMTO Trevor Dzwiniel, Argonne National Laboratory <u>tdzwiniel@anl.gov</u> Carol Hirschmugl, COnovate, Inc. <u>dr.carol.h@conovateinc.com</u>



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