NREL Transforming ENERGY

ReCell ADVANCED BATTERY RECYCLING

ANODE UPCYCLING

VIA TAILORED SOLVENT TREATMENT

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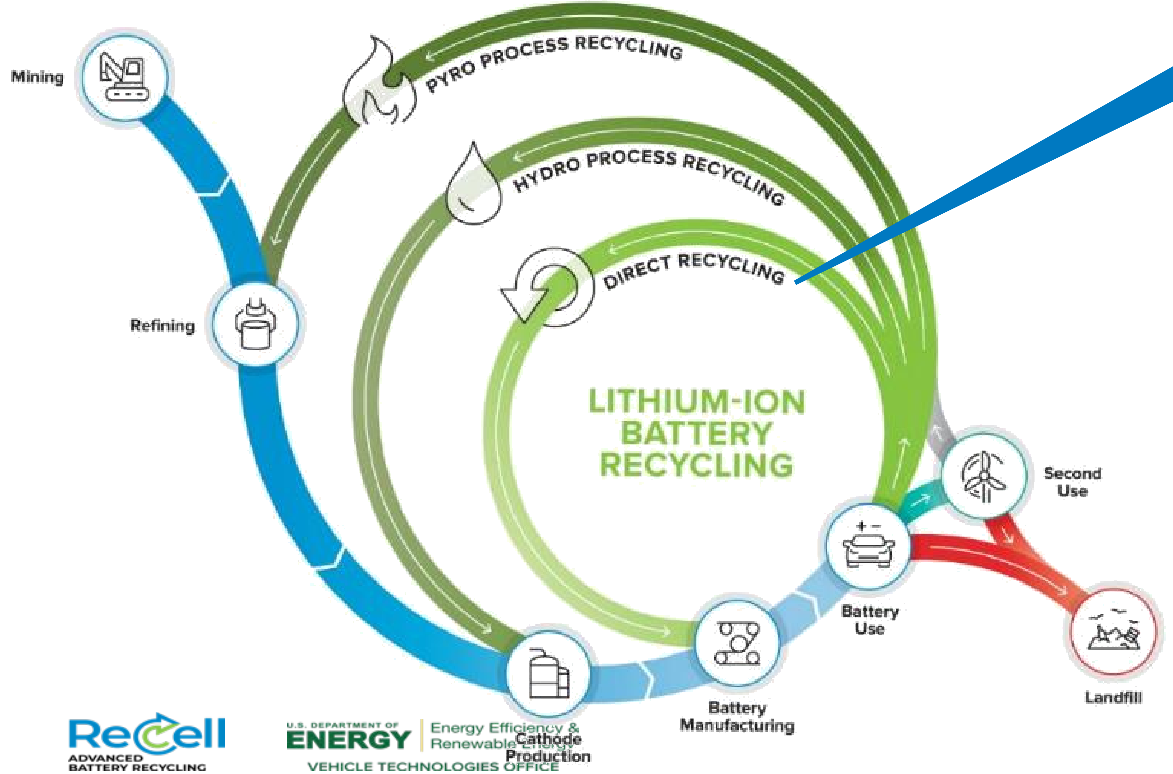
DIRECT RECYCLING OF LI-ION BATTERIES

- Burgeoning demand for Li-ion batteries induces **supply chain instability** and raises concerns regarding **end-of-life disposal**.
- <u>DOE goal:</u> "Reduce the cost of electric vehicle battery packs to <$150/kWh with technologies that significantly reduce or eliminate **dependency on critical materials** and utilize **recycled material feedstocks**"



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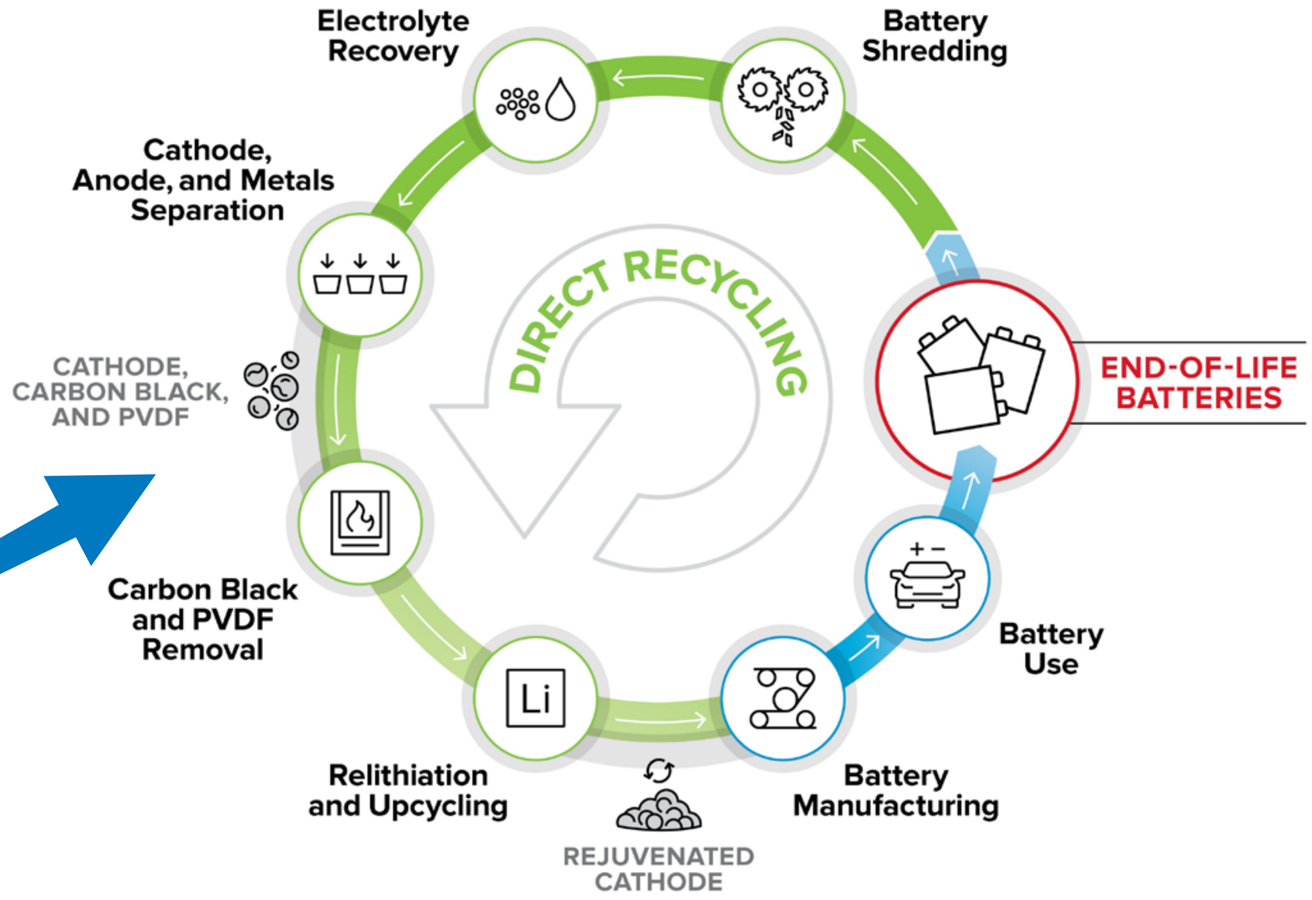


Direct recycling retains the engineered value of battery materials and minimizes processing steps. *To date, direct recycling efforts have primarily focused on the high-value cathode material.*

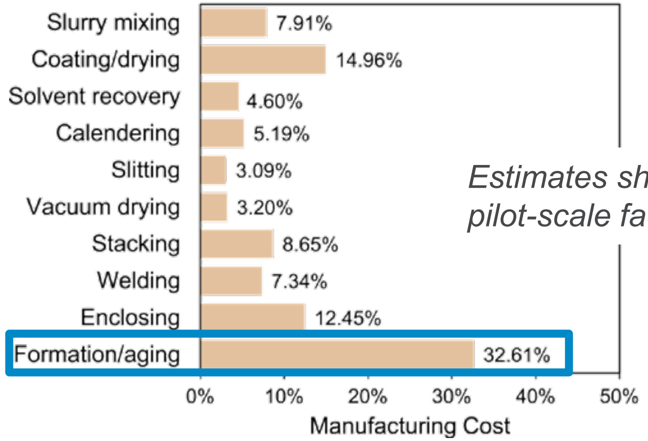
ANODE UPCYCLING: MOTIVATION & GOALS

~10-15% of the cyclable Li in a virgin cell is **irreversibly consumed** during formation (i.e., growing the anode SEI) .

Graphite	Q_{rev} (mAh/g)	**ICL (%)**	ICL (mAh/g)
SL20	370	**9.2**	75
MAG-10	328	**8.6**	62
GDR6	340	**13.3**	108

Reversible capacity (Q_{rev}) and irreversible capacity loss (ICL) during formation for three types of graphite.

Additionally, formation can take days to weeks, necessitating **larger facilities**, consuming **energy**, and producing **GHG emissions**.



Shim, J. et al., *J. Power Sources* 119-121 (2003): 934-937; Liu, Y. et al., *iScience* 24 (2021): 102332

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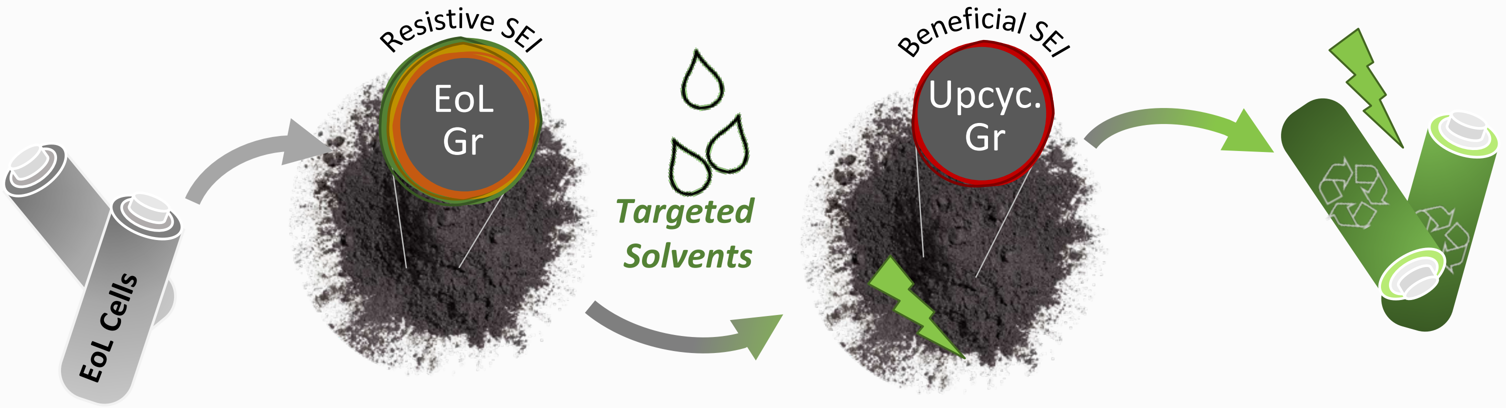
In traditional pyro and hydro recycling processes, Gr is typically pyrolyzed or serves as a simple reductant.

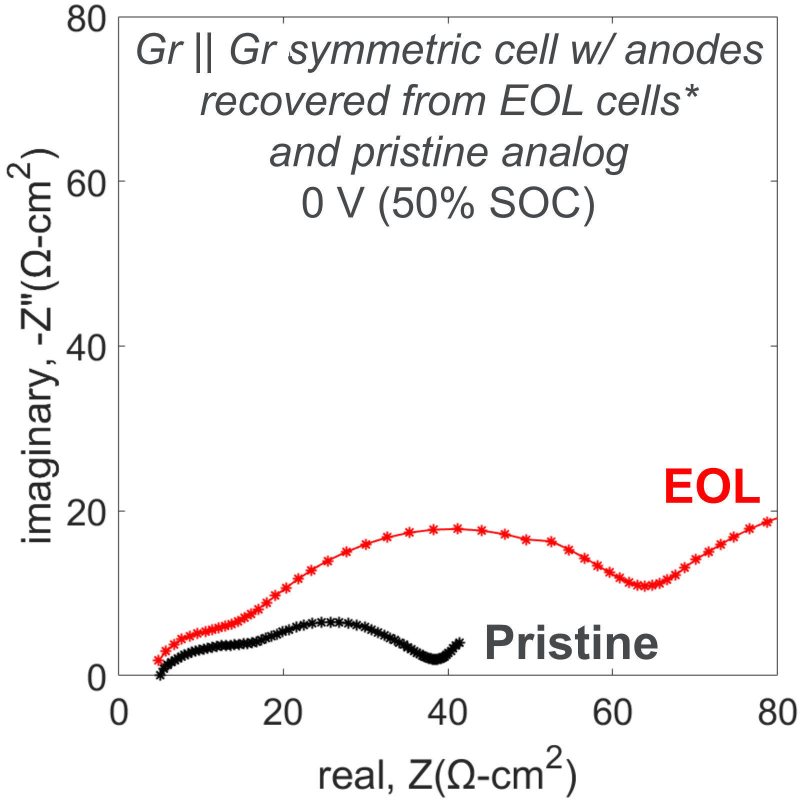
We are developing methods to obtain a high-performing upcycled anode to **reduce/eliminate costly, energy-intensive, and time-intensive formation** protocols and **reduce cathode lithiation requirements**.

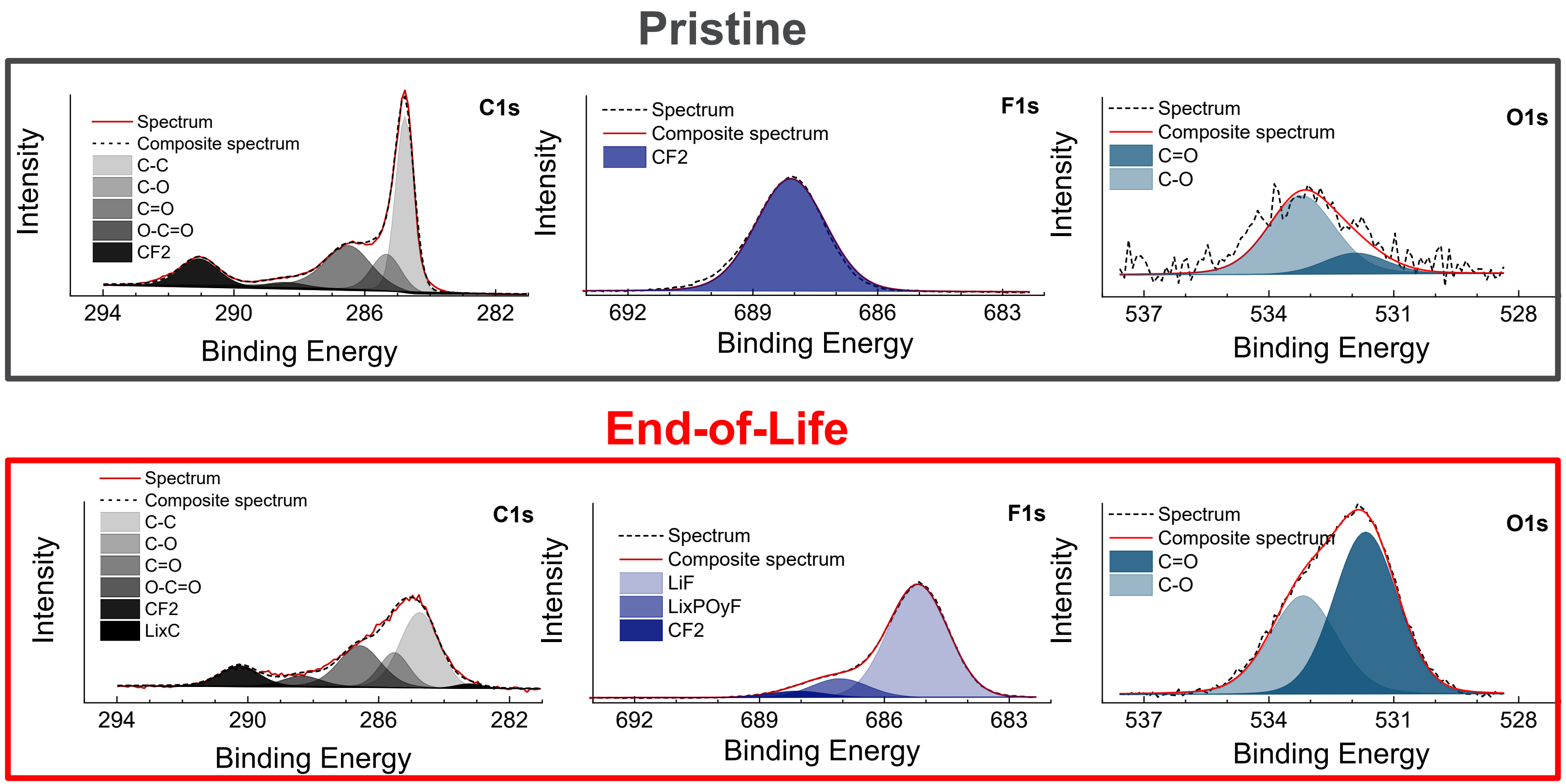
A successfully upcycled anode could offer **major value to cell manufacturers** beyond just the value of pristine graphite.



Goal: Use tailored solvents to selectively surface-purify Gr anode, retaining beneficial SEI species while reducing resistance.

WHY FOCUS ON ANODE SURFACE TREATMENT?





- Bulk graphite structure largely reported to remain intact, even after repeated cycling
- Gr anode recovered at end of life shows drastically higher resistance than pristine Gr, attributable to the evolution of a resistive SEI layer
- This reduces rate performance and reversible capacity by blocking Li intercalation pathways

** EOL graphite anodes recovered from industrial Gr||NMC622 cells (PVDF/NMP) cycled to 80% SOH*

OVERVIEW OF PROCESS WORKFLOW

“Worst-Case” EoL Anodes

Upcycled Anode

“Best-Case” Formation-Only Anodes

Physico-Chemical Characterization

Electrochemical Characterization

“What are we starting with?”

- **Commercial end-of-life anodes***
- Benchmark end-of-life state
  - Chemical composition of SEI
  - (Graphite + SEI) structure
  - Graphite morphology
  - Electrochemical performance

“How does our upcycling treatment affect the system?”

- Process liberated powders in rationally selected solvents
- **Goal: selectively retain a subset of SEI species**
- Evaluate treatment impact

Physico-Chemical Characterization

Electrochemical Characterization

Physico-Chemical Characterization

Electrochemical Characterization

“Where do we need to get to?”

Rationally select subset of analyses

Modeling Efforts:

- Identify electrochemical metrics for “good” vs “bad” SEI
- Inform a pared-down analysis matrix for rapid screening
- Integration with more complex SEI models for predictability

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** EOL graphite anodes recovered from industrial Gr||NMC622 cells (PVDF/NMP) cycled to 80% SOH*

METHODS: SOLVENT TREATMENT AND REPRINTING

EoL Graphite Electrode*

93% graphite
3.5% carbon
3.5% PVdF binder

1. Mechanically liberate graphite (delaminate)

EoL Graphite powder/flake + carbon-binder domain (CBD)

2. Solvent treatment:

- 1g EoL Gr+CBD powder +10 mL solvent(s)
- Bath sonicate for 60 minutes
- Stirred at ambient temp. ~18h
- Centrifuge mixture, decant supernatant
- Rinse 2x more with 10 mL solvent, centrifuge, decant supernatant
- Vacuum dry treated graphite at 80˚C

3. Formulate slurry and print:

- *Add NMP to treated Gr+CBD*
- *Planetary mix*
- *Print onto Cu with blade coater*
- *Vacuum dry at 80˚C, 12h*

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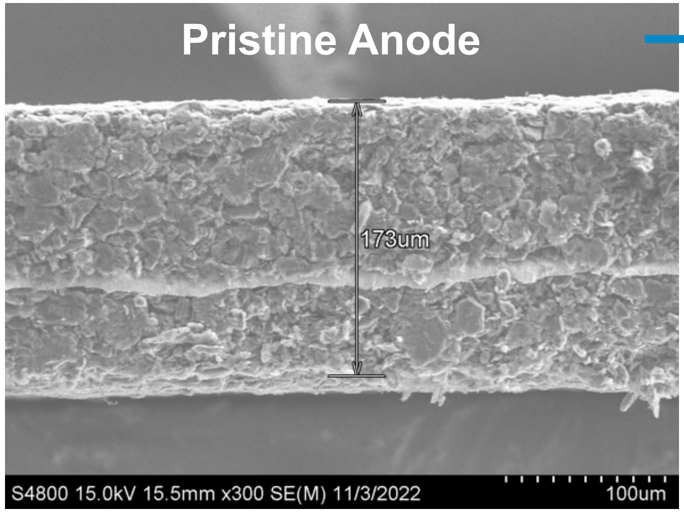
** EOL graphite anodes recovered from industrial Gr||NMC622 cells (PVDF/NMP) cycled to 80% SOH*

CHALLENGES WITH RECASTABILITY IN NMP

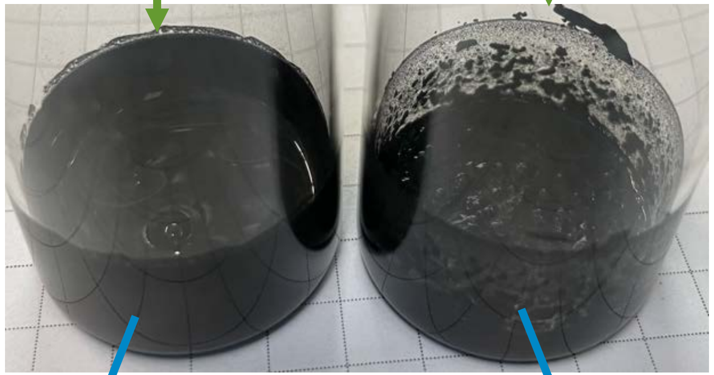
- This upcycling approach requires *at minimum* resuspension in NMP to recast
- Attempt: Re-cast mechanically liberated powders to serve as a "cycled baseline"
- Result: EoL slurry gelled egregiously and was unprintable without supplemental preprocessing (*grinding*)

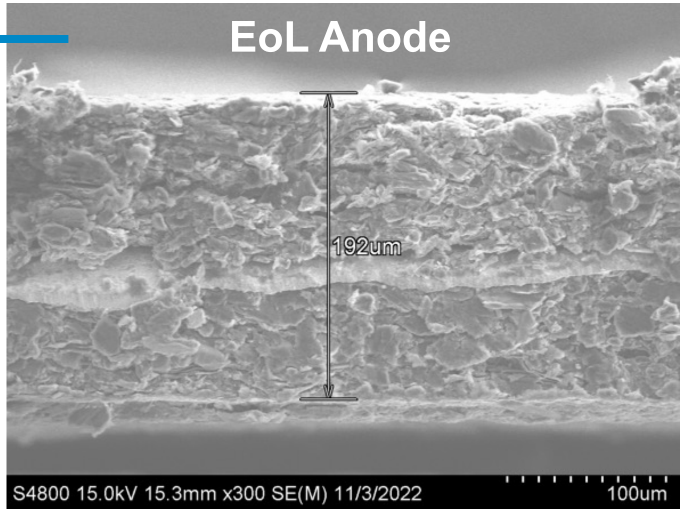


Mechanically delaminate

Add NMP and mix
40% solids loading

Mechanically delaminate





print slurry

Smooth slurry; usable print:

1. Anode has never seen electrolyte;
2. Anode has never experienced reducing potentials

Gelled slurry; chunky/unusable print:

1. SEI or residual electrolyte interacts poorly with NMP; **or**
2. Cycled Gr electrode has irreversibly changed during cycling (e.g., cross-linked PVdF)

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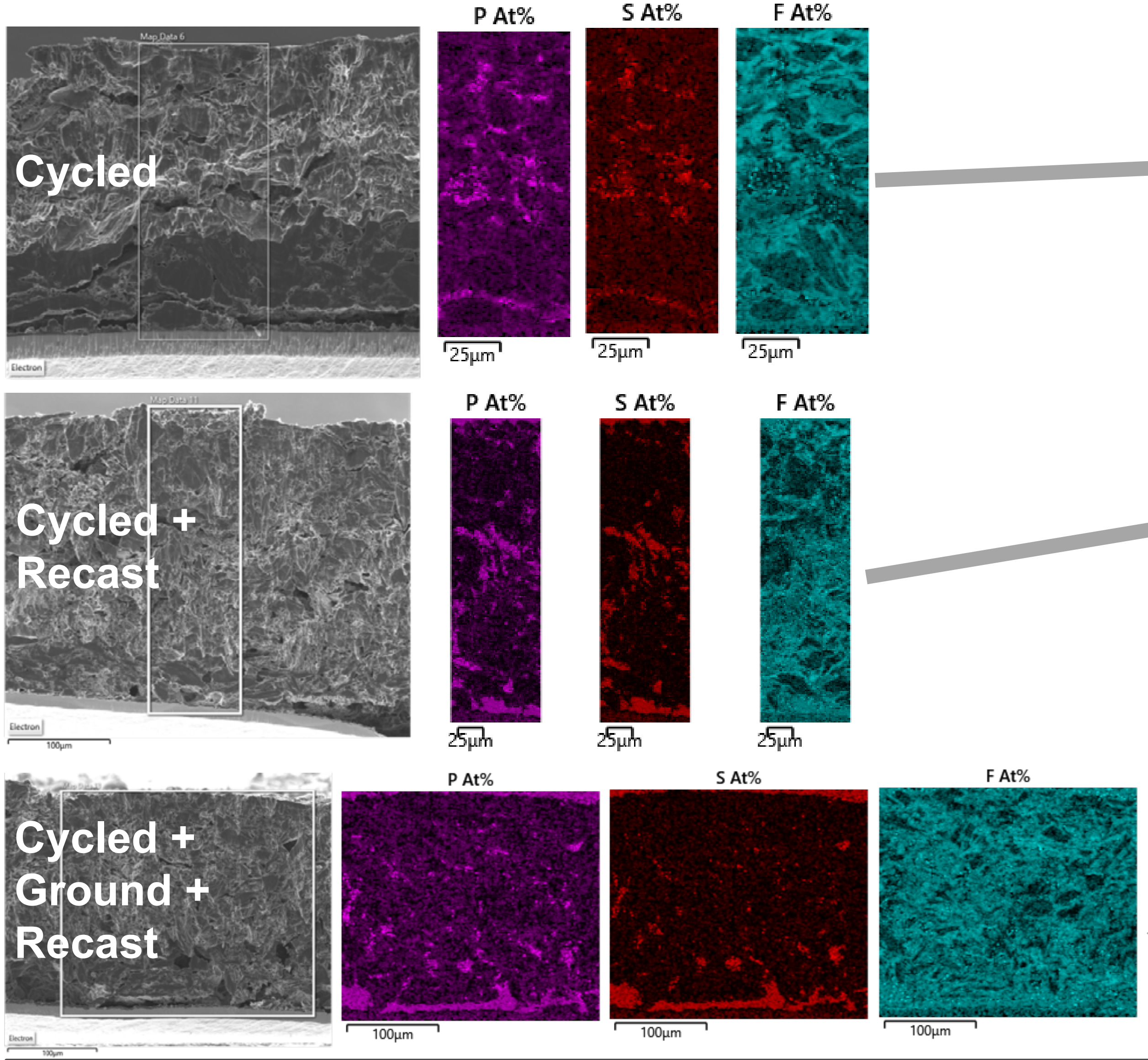
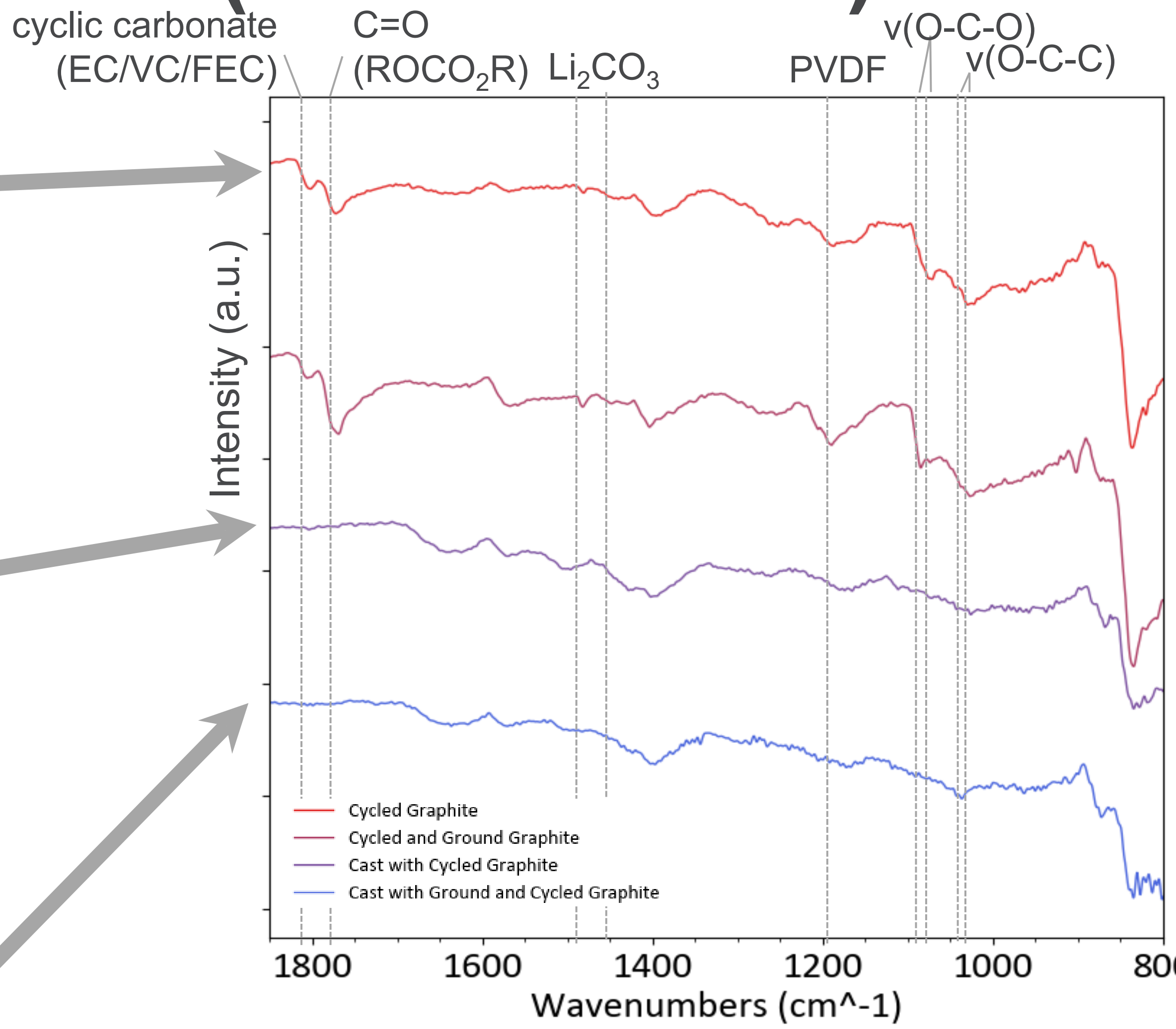
COMPONENT REDISTRIBUTION (+ REACTION)



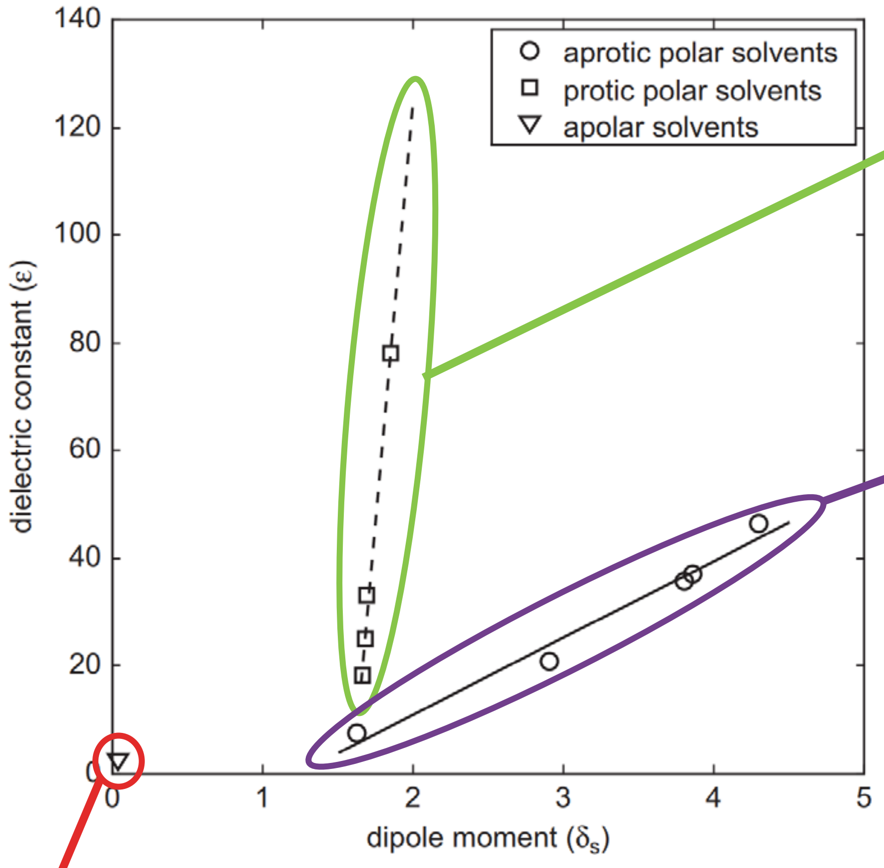


- SEI redistribution confirmed w/ both recasting & grinding
- Grinding may disperse SEI (+ "settling"?)

- Slight peak shifts to higher wavenumber associated w/ carbonate stretches upon grinding
- Loss of cyclic carbonate, polymeric C=O, and Li_2CO_3 signals upon recasting

RATIONAL SOLVENT SELECTION FOR ANODE UPCYCLING

Ideal solvent candidate will retain desirable SEI components while allowing viable electrode printing.



Polar Protic (e.g., water, alcohols)

- Will wash away polar organic SEI species
- Will dissolve inorganic salts in SEI (LiF, Li_2CO_3)
- Will react with reduced SEI species

Polar Aprotic (e.g., ethyl acetate, acetone, THF)

- Will wash away polar organic SEI species
- Minimize reactions with reduced SEI species

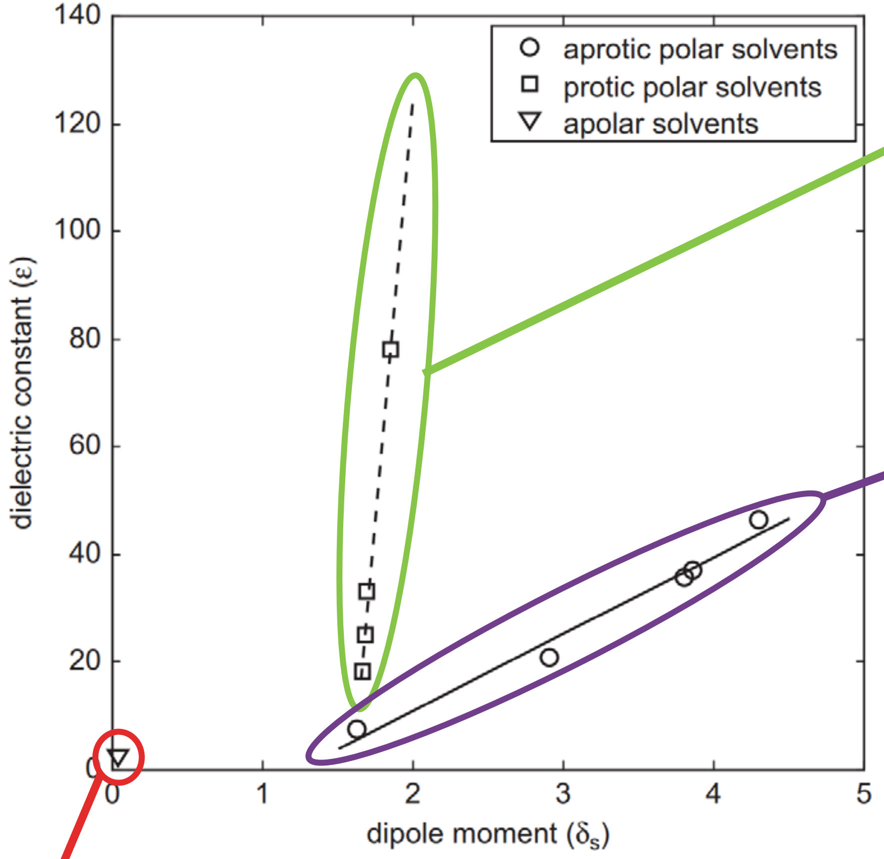
Nonpolar (e.g., hexane)

- Non-polar
- Will wash away highly non-polar SEI species

Figure adapted from P. Marchetti et al., *J. Membrane Sci. 415-416 (2012): 444-458.*

RATIONAL SOLVENT SELECTION FOR ANODE UPCYCLING

Ideal solvent candidate will retain desirable SEI components while allowing viable electrode printing.



Polar Protic (e.g., water, alcohols)

- Will wash away polar organic SEI species
- Will dissolve inorganic salts in SEI (LiF, Li_2CO_3)
- Will react with reduced SEI species

poor initial capacity

Polar Aprotic (e.g., ethyl ... tate, acetone, THF)

- Will wash away ... ganic SEI species
- Minimize rea ... s with reduced SEI species

Solvent Identity	Polarity Index	Dielectric Constant	Solvent pKa
Water (H_2O)	10.2	78.355	14
Methanol (MeOH)	5.1	32.613	15.521
Ethanol (EtOH)	4.3	24.852	15.85
Isopropanol (IPA)	3.9	19.264	16.48

Non... lar (e.g. ... xane)

- Non-p...
- Wi... wash aw... highly non-polar SEI species

casting issues

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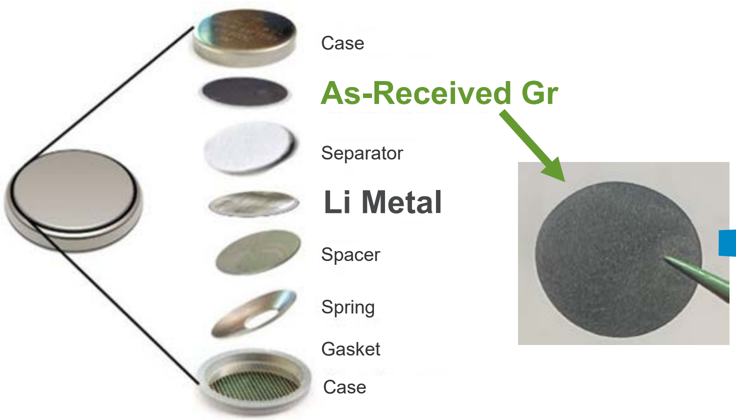
Figure adapted from P. Marchetti et al., *J. Membrane Sci. 415-416 (2012): 444-458.*

METHODS: ELECTROCHEMICAL TESTING

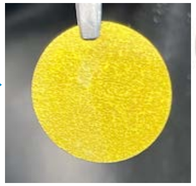
- We have developed a **symmetric cell protocol** to probe electrochemical behavior
- Symmetric cell format minimizes artificial disruptions to the SEI (infinite Li in half cells or cross-talk in full cells)
- Symmetric cell data is used to identify **electrochemical signals** tied to SEI stability

1. Half-Cell Lithiation (one cycle)

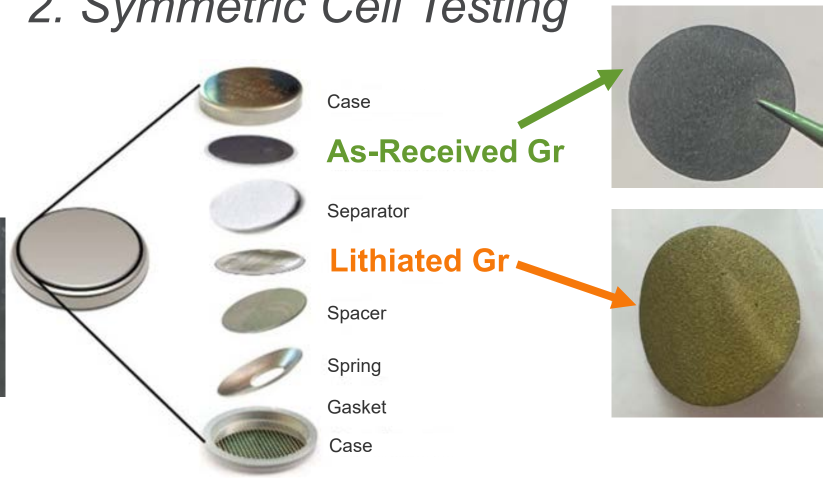


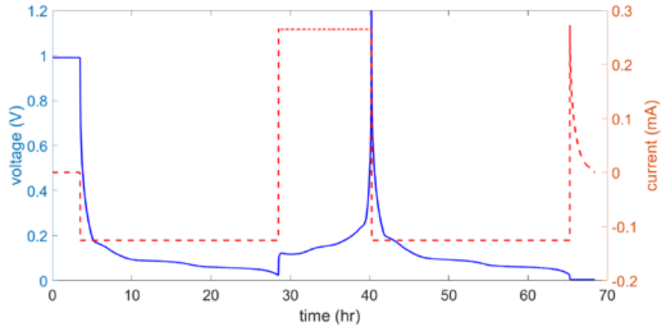
Recover lithiated Gr

Reassemble into symmetric cell



2. Symmetric Cell Testing





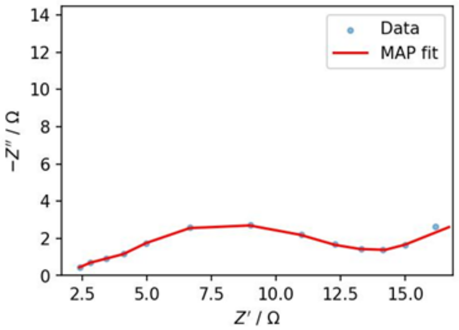
Analysis Protocol:

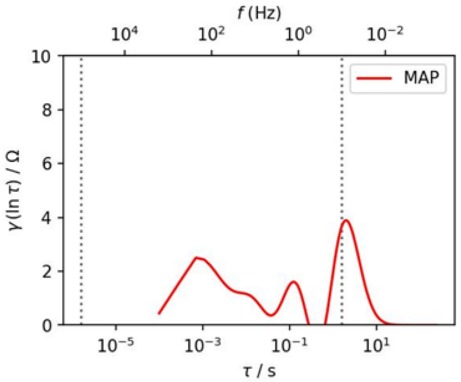
- 10 x C/10 cycling ("break-in")
- SOC-dependent EIS
- Rate performance (C/20 to 3C)
- C/10 cycling (stability)

* Voltage Limits: -0.5 – +0.5 V

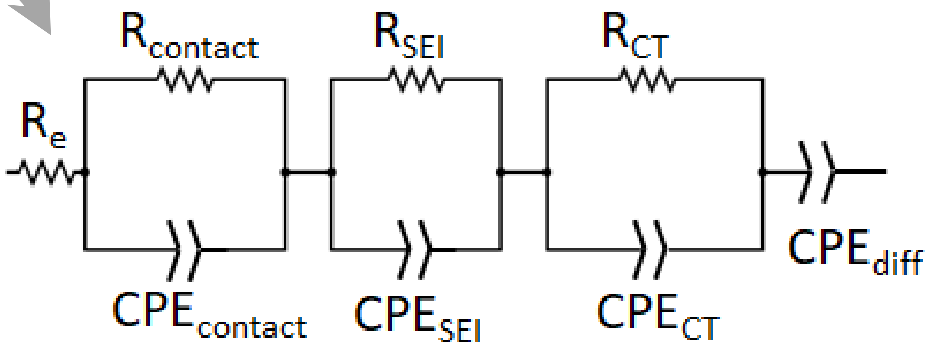
Coin cell schematic adapted from A. Zülke et al., Batteries & Supercaps 4 (2021): 934–947.

ELECTROCHEMICAL PERFORMANCE: IMPEDANCE





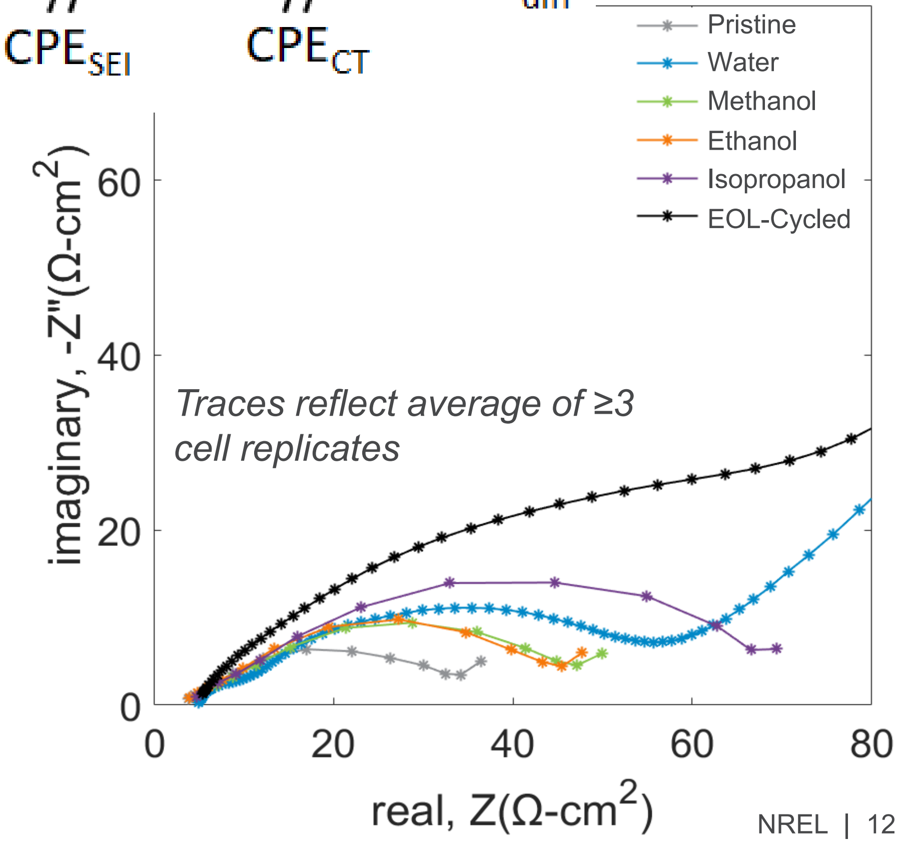
3 R-CPE elements selected for equivalent circuit



Distribution of Relaxation Times (DRT):

- Visualize impedance data in time domain
- Useful to estimate number of R-CPE pairs required for physically meaningful ECM analysis

Sample	$R_{contact}$ (Ω-cm²)	R_{SEI} (Ω-cm²)	R_{CT} (Ω-cm²)
Pristine	1.87 ± 0.16	20.99 ± 7.95	6.66 ± 3.12
Water	3.82 ± 0.75	20.36 ± 1.30	10.00 ± 2.10
MeOH	3.10 ± 0.33	26.68 ± 11.12	14.50 ± 4.14
EtOH	2.82 ± 1.01	36.28 ± 7.60	5.24 ± 1.11
IPA	2.79 ± 0.35	52.75 ± 15.79	10.08 ± 5.18
EOL Gr	-	-	-



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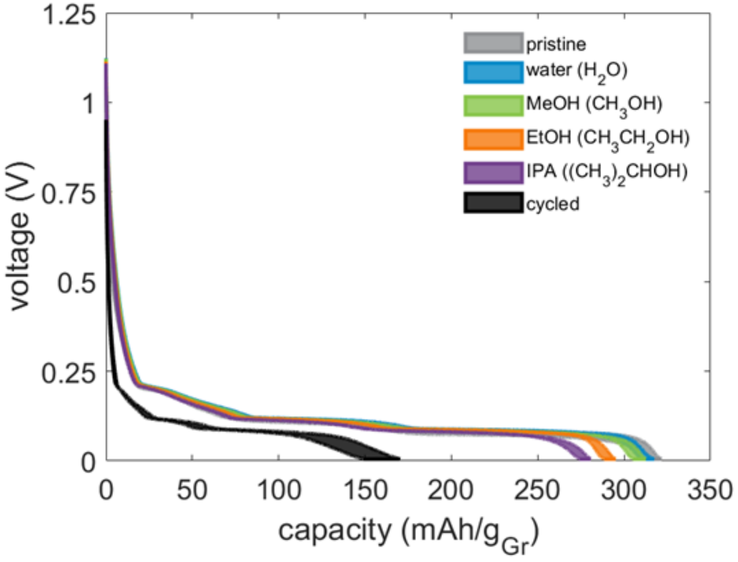
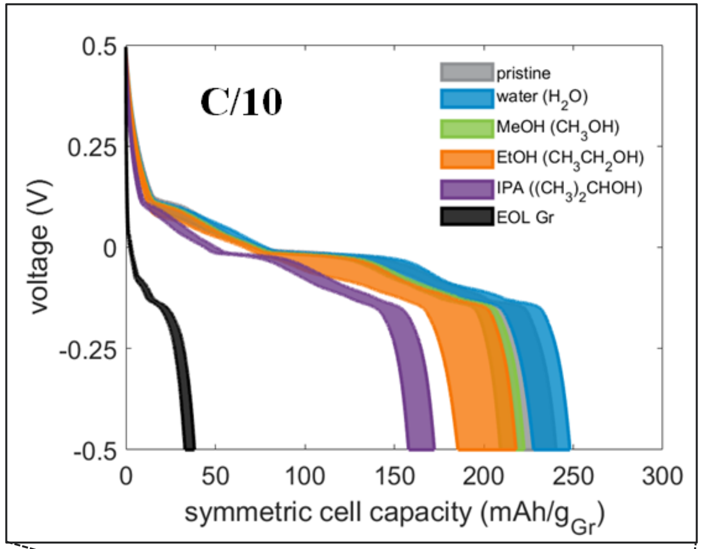
ELECTROCHEMICAL PERFORMANCE: CAPACITY & RATE

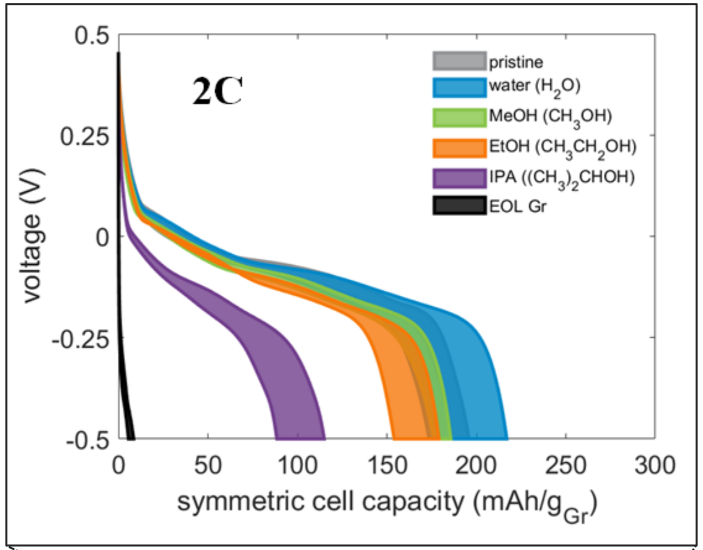
Half Cells



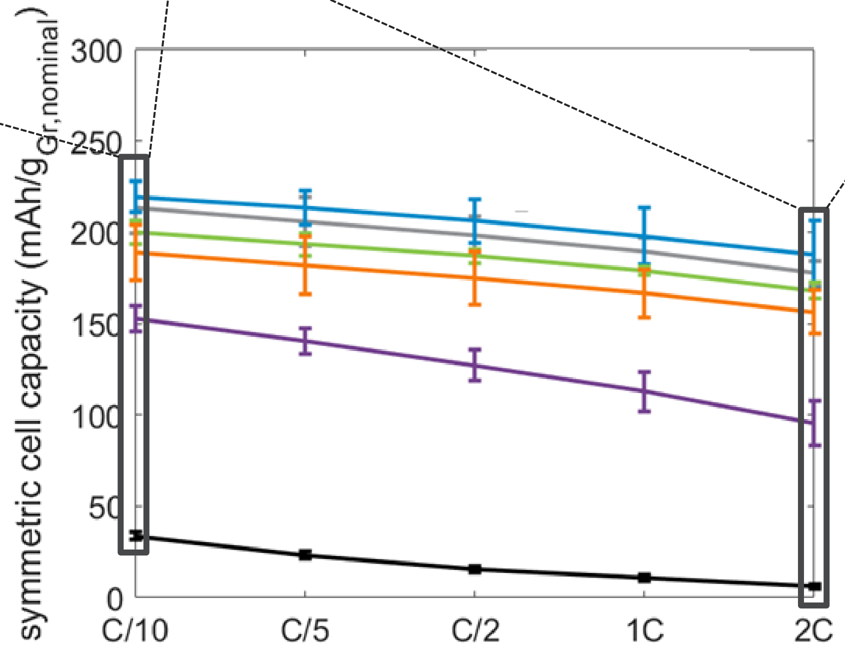
Treatment	Half-Cell CE (Cycle 1; %)
Pristine	89.19
Water	86.58
MeOH	87.67
EtOH	87.62
IPA	88.20
EOL Cycled	74.36

Symmetric Cells

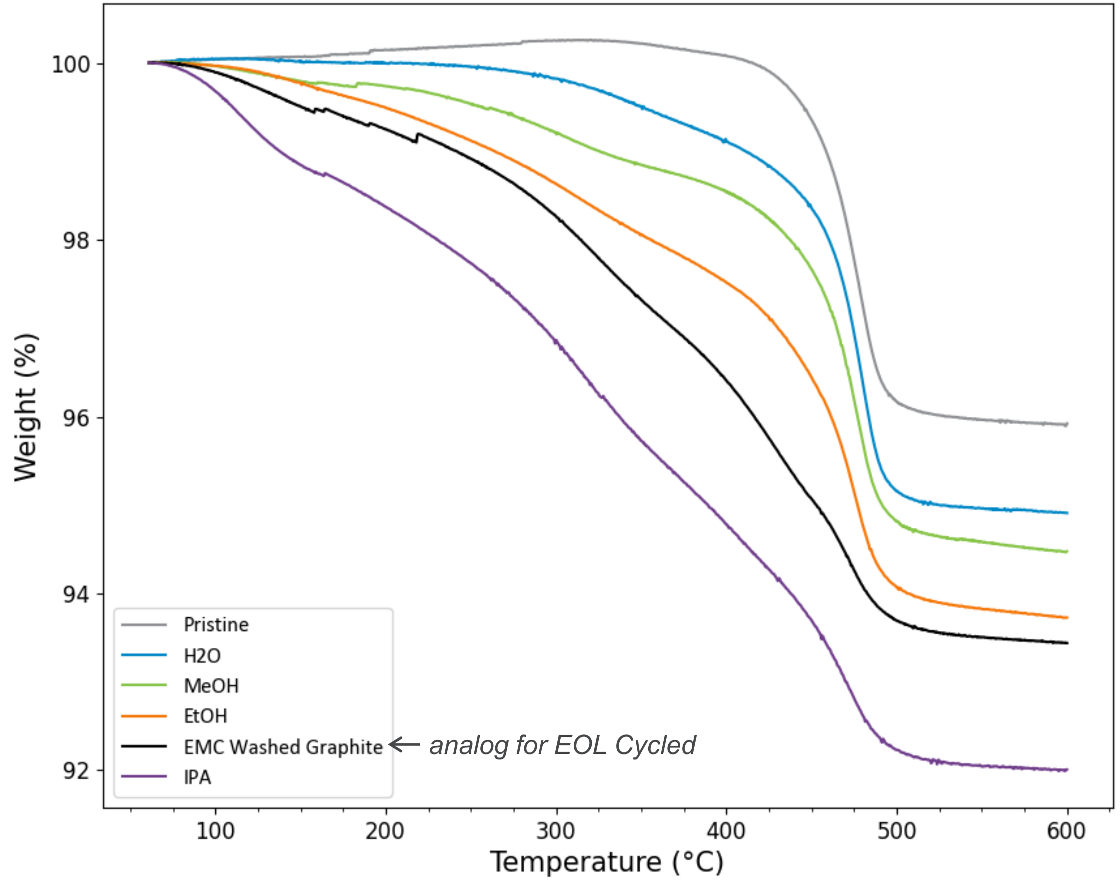




- All washing conditions improve capacity (half & symmetric), CE, and rate performance relative to EOL cycled material
- Stripping SEI = increased accessibility of active Gr sites
- Low CE of EOL cycled material implies incomplete passivation (*result of air/water exposure?)*

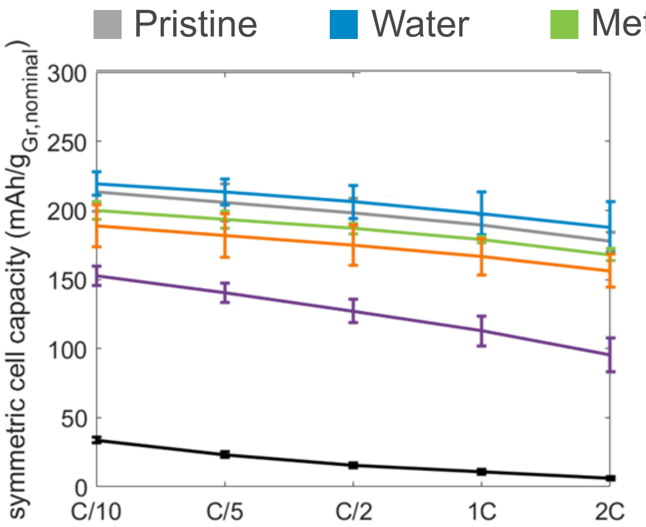


ACCOUNTING FOR A HEAVIER UPCYCLED ANODE

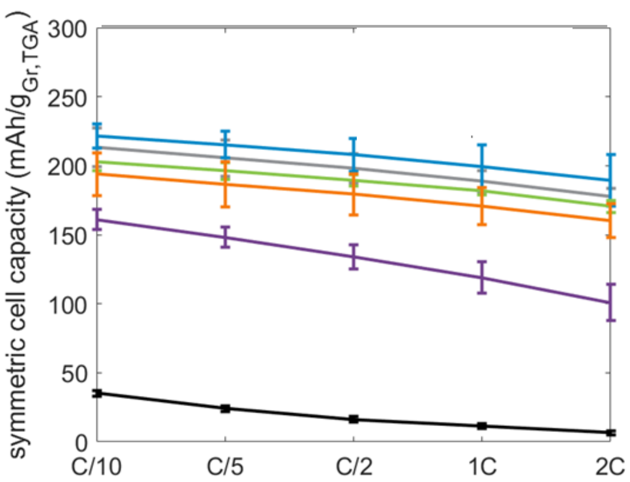


- TGA indicates sample mass loss during controlled temperature ramp due to reaction/offgassing
- Mass loss below PVdF combustion temperature (~475 °C) predominated by reaction of SEI species
- Higher early mass loss = more remaining SEI

- Conventional mass-based capacity accounting penalizes anodes with a pre-formed SEI
- TGA results yield % Gr = more accurate mass value for normalization



Normalization by nominal Gr % (not accounting for SEI mass)



Normalization by TGA-informed Gr % (accounting for SEI mass)

There are no changes in capacity trends simply due to adjusting the normalization approach.

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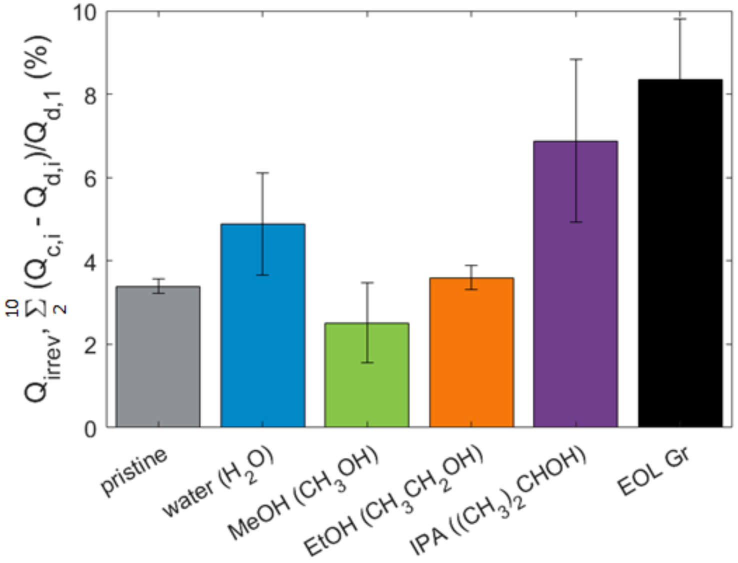
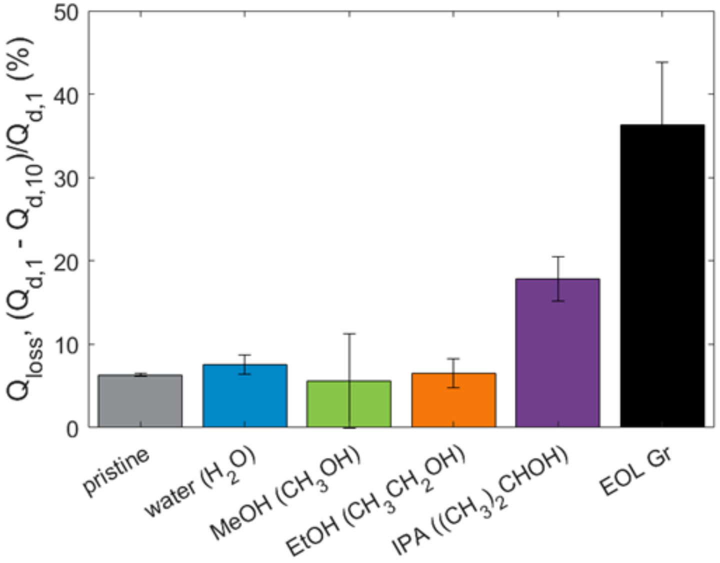
IDENTIFYING RELEVANT MASS-AGNOSTIC METRICS

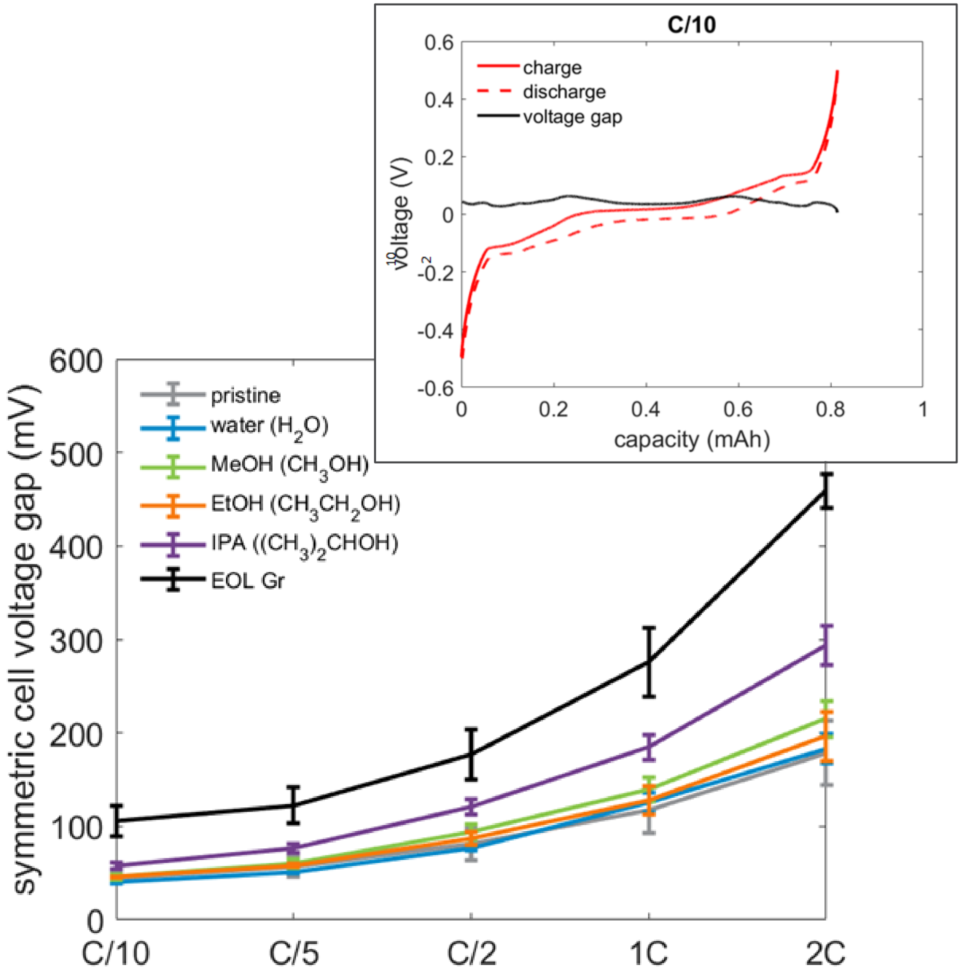


Cumulative irreversible capacity loss during symmetric cell formation (% of initial discharge capacity)



Loss in cyclable discharge capacity during symmetric cell formation (% of initial discharge capacity)

Voltage gap quantifies hysteresis, which reflects kinetic & transport limitations at varying cycling rates



Reported values reflect average of middle half of data

Mass-agnostic metrics reveal nuanced impact of treatment solvent on electrochemical performance.

- Best-performer from capacity perspective (**water**) shows greater formation losses than **MeOH** and **EtOH**
- **IPA** shows both lower capacity and greater formation losses
- **MeOH** shows higher voltage gap but lower formation losses

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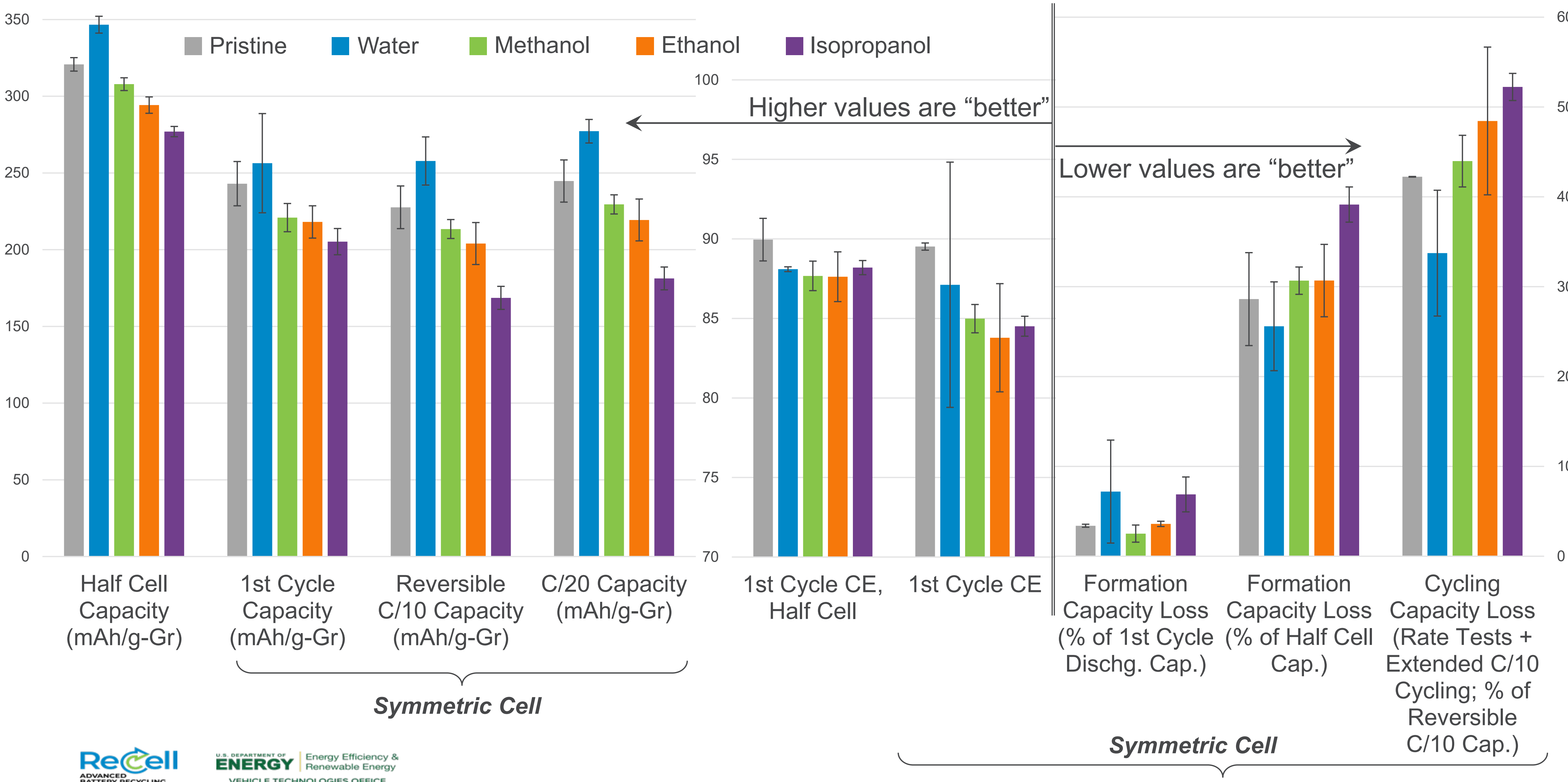
SUMMARY OF ELECTROCHEMICAL METRICS



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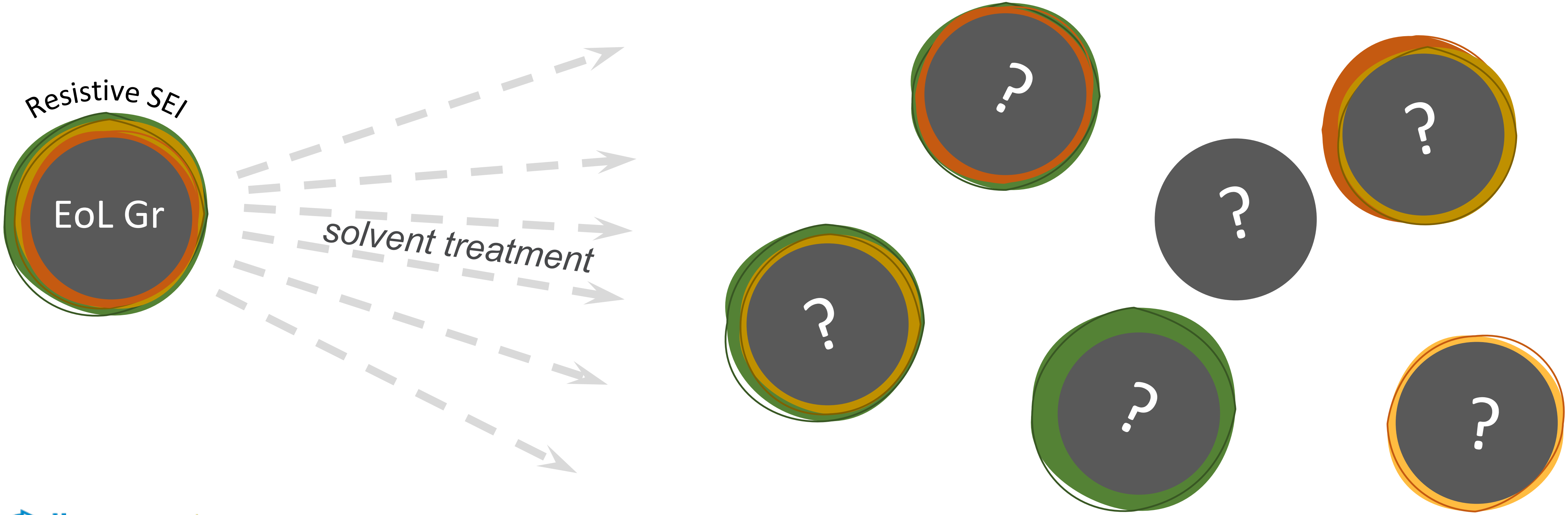
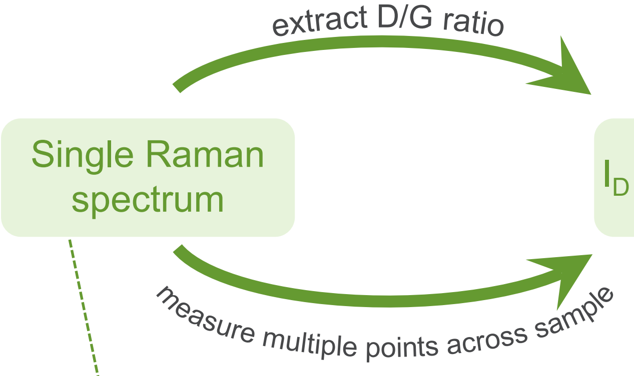
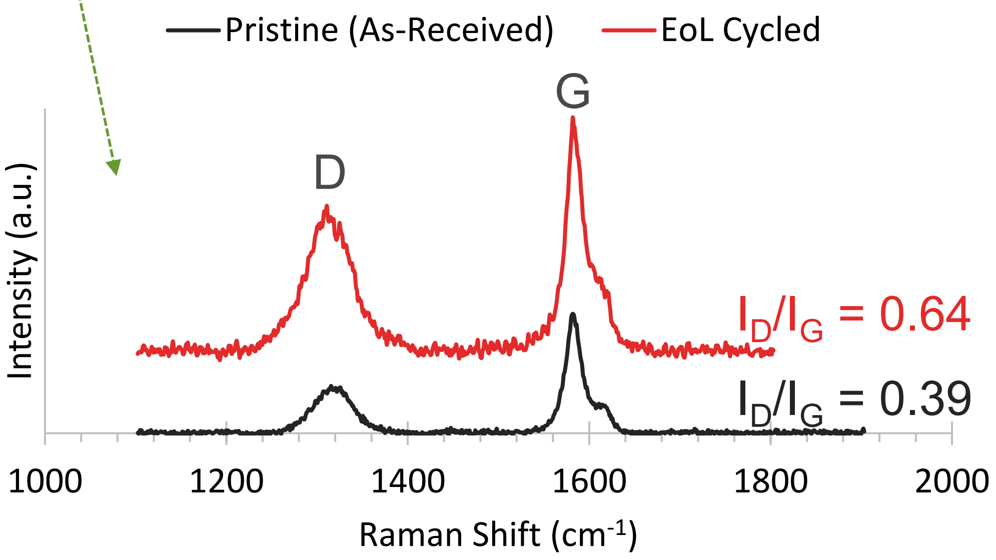
PHYSICO-CHEMICAL ANALYSIS & METRICS

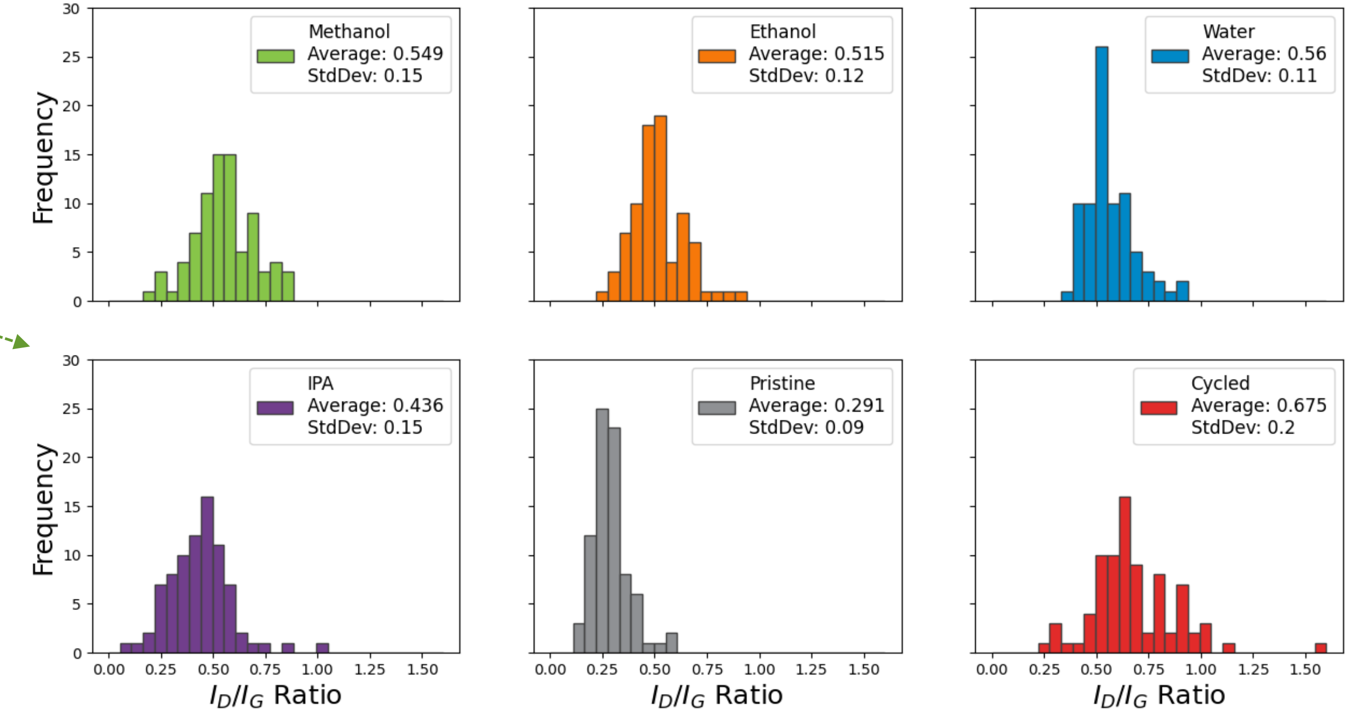
- **Robust physico-chemical analysis is critical to informing *how* solvent treatment alters the EoL graphite**.
  - *What SEI species are removed?*
  - *What SEI species are retained?*
  - *How does the treatment process influence graphite surface chemistry, morphology, and structure?*
  - *What is the optimal outcome from the perspective of electrochemical performance?*
- **Characterization signals offer additional metrics with which to judge upcycling quality & success.**



UPCYCLING EFFECTS: STRUCTURAL ANALYSIS





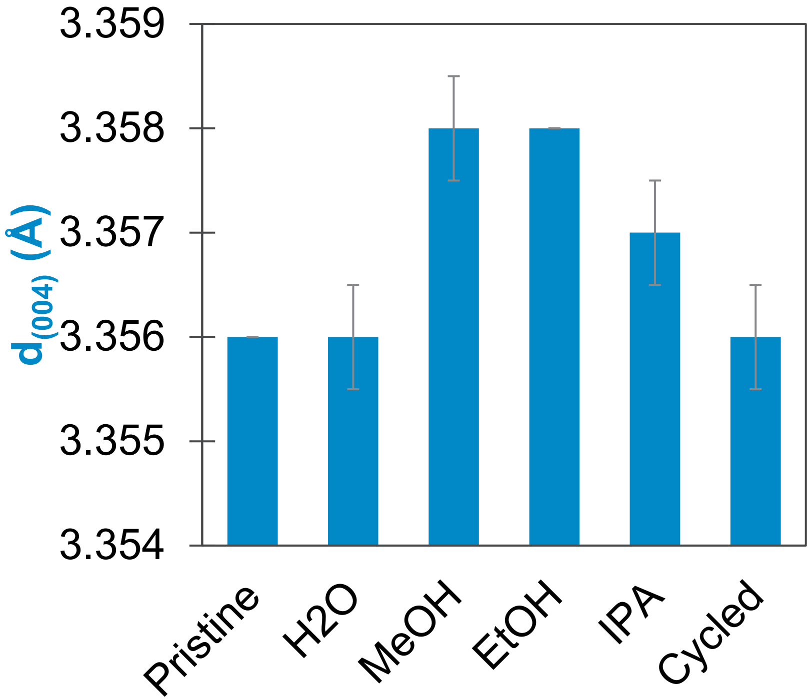


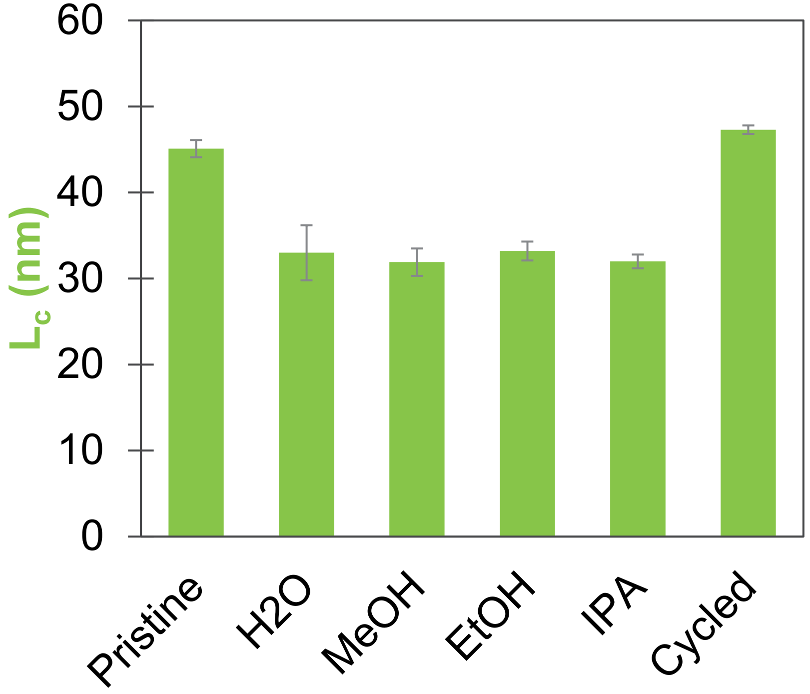
- First-order Raman spectra (~1000-2000 cm^{-1}) consist of *G-band* (E_{2g} mode of sp^2 carbon network) & *D-band* (associated w/ defects)
- Ratio of integrated intensities (I_D/I_G) is standard metric for evaluating defect quantity and degree of disorder in graphitic materials, with **higher I_D/I_G = greater number of defect sites**

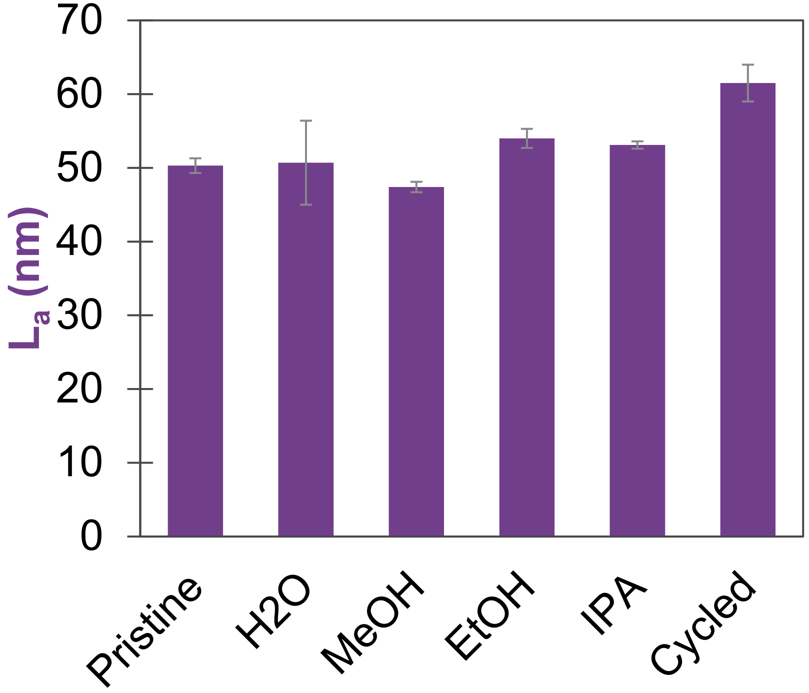
- Raman *mapping* (vs just single point scan) enables construction of I_D/I_G histograms – statistical distribution
- All upcycled samples lie between pristine and EoL cycled samples in terms of both I_D/I_G and distribution width.
- This indicates (1) Solvent treatment cannot remove all surface defects; (2) treatment does not destroy Gr structure

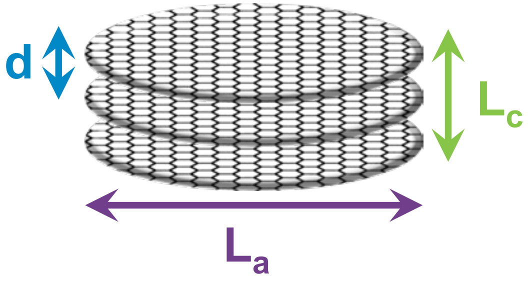
I_D/I_G increases roughly as: IPA < EtOH < MeOH < H_2O

UPCYCLING EFFECTS: STRUCTURAL ANALYSIS









$d_{(004)} = \frac{n\lambda}{2\sin(\theta)}$ (λ = 1.5406 Å; n = 2)

$L_c = \frac{0.9\lambda}{(\beta_{004}\cos(\theta_{004}))}$; $L_a = \frac{0.9\lambda}{(\beta_{100}\cos(\theta_{100}))}$

- No evidence of *d*-spacing increase associated with repeated Li (de)intercalation
- Solvent treatment induces slight interlayer expansion – performance improvement?
- ~25% reduction in L_c relative to pristine following solvent treatment
- Treatment does not substantially impact L_a relative to pristine
- Reduced L_c (but not L_a) implies:
  - Weaker graphene/graphene interactions enables more facile exfoliation; and/or
  - Solvent intercalation during processing driving exfoliation
- Smaller crystallite size may contribute to higher irreversible losses

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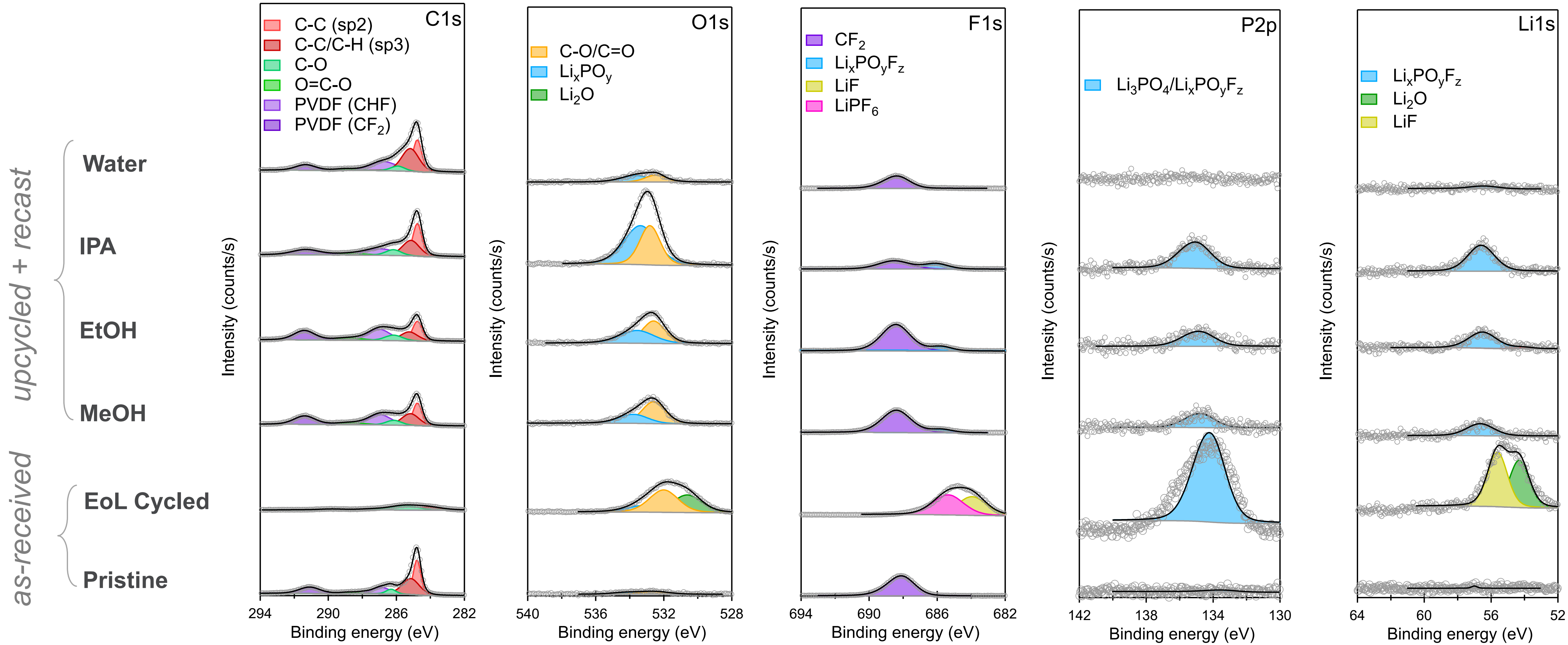
UPCYCLING EFFECTS: CHEMICAL ANALYSIS



- X-ray photoelectron spectroscopy (XPS) suggests composition of SEI species remaining on surface following treatment
- Greater predominance of PVDF-associated peaks (CF_2, CHF) indicates "more" SEI removed (visibility of underlying binder)

All solvents tested remove all LiF and Li_2O

Removal of (fluoro)phosphates increases as IPA < EtOH < MeOH < H_2O

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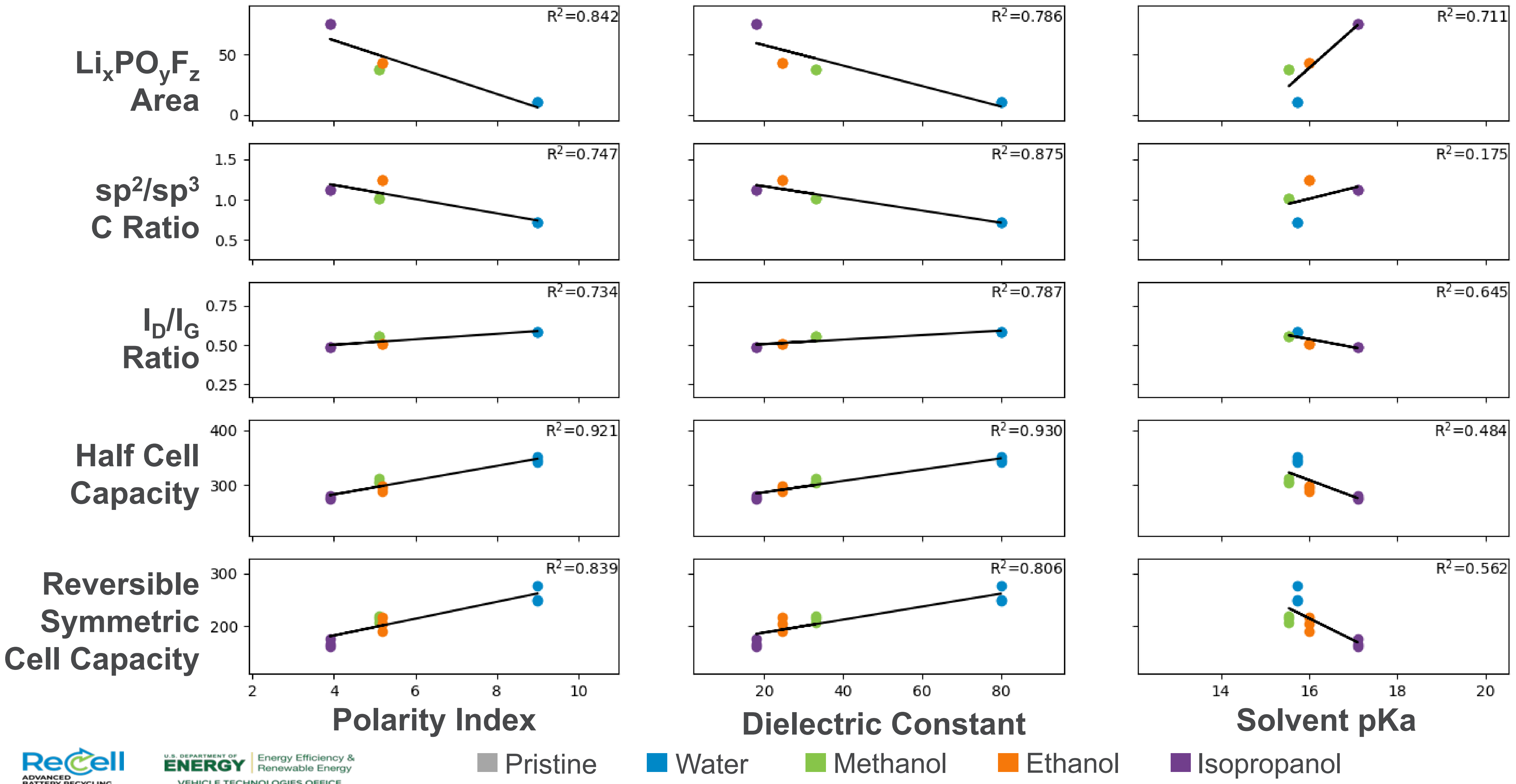
LINEAR CORRELATION ANALYSIS

Solvent properties vs electrochemical & physico-chemical signatures



LINEAR CORRELATION ANALYSIS

Solvent properties vs electrochemical & physico-chemical signatures

STRONGLY POSITIVE CORRELATIONS:

Wash solvent polarity & dielectric constant vs
- Half-cell capacity
- Symmetric cell reversible capacity
- I_D / I_G ratio

Wash solvent pKa vs
- $Li_xPO_yF_z$ area

STRONGLY NEGATIVE CORRELATIONS:

Wash solvent polarity & dielectric constant vs
- sp^2 / sp^3 carbon ratio
- $Li_xPO_yF_z$ area

MODERATELY NEGATIVE CORRELATIONS:

Wash solvent pKa vs
- I_D / I_G ratio
- Half-cell capacity
- Symmetric cell reversible capacity

- **Higher polarity**, **higher dielectric constant**, and **lower pKa** values for wash solvents improve capacity of upcycled material.
- These wash solvents produce upcycled material with **lower sp^2 carbon** content, **reduced $Li_xPO_yF_z$** content, and a **higher I_D / I_G ratio**.
- **Half cell & reversible symmetric cell capacity** appear to correlate most strongly with both solvent properties and physico-chemical metrics for initial set of single-solvent treatments

SUMMARY

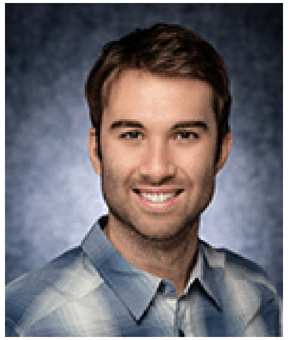
Retaining & "refurbishing" the SEI on EOL anodes offers a unique opportunity to recover a value-added Gr product.

- We demonstrate *selective* removal of classes of SEI species while retaining others by tuning solvent properties
- As-recovered EoL Gr cannot be directly recast
  - Grinding disrupts distribution of SEI species and may alter residual carbonate structure
  - NMP exposure during resuspension appears to induce reactions with cyclic carbonates, polymeric SEI, & Li_2CO_3
- A rational series of alcohol solvents have been evaluated to probe structure-property-performance relationships
- Identified mass-agnostic metrics (formation capacity loss, voltage gap) as critical indicators of performance
- **Higher polarity**, **higher dielectric constant**, & **lower pKa** wash solvents improve capacity of upcycled material
- These treatments produce anodes w/ **lower sp^2 carbon** content, **reduced $Li_xPO_yF_z$** content, and **higher I_D / I_G ratio**

ADDITIONAL ONGOING WORK

- **We are conducting formation, voltage-hold, and cycle-life studies in full cells.**
  - Determine performance of upcycled anode under practically relevant conditions
- **We have expanded beyond single-solvent systems.**
  - Applying learnings from initial alcohol-series study to select additional “active” & background solvents
  - Evaluating concentration & background matrix effects
- **We are implementing a more robust data-driven correlation analysis approach.**
  - Adapting decision-tree model framework
  - Streamline solvent screening & characterization approach
  - Offer predictivity for new solvent systems
- **We are conducting post-mortem analysis to determine “SEI regrowth” behavior.**
  - Performance depends on both what residual SEI remains and what SEI subsequently regrows
- **We have collaboratively conducted technoeconomic & sensitivity analysis.**
  - Quantify added value of upcycled anode (vs recycled Gr)
  - Identify target process parameters for greatest cost reduction opportunities
- **We are evaluating this approach on additional EOL Gr anode materials to determine bounds of applicability.**





Thank you! Questions?

Kae.Fink@nrel.gov

www.nrel.gov **NREL/PR-5700-89971**

US Provisional Patent Application No. 63/561,827 (2024)

M.C. Schulze, N. McKalip, A. Verma, Z. Berquist, S.-B. Son, B. Ingram, A. Colclasure, K. Fink, Facile solvent treatments enable the direct recycling and reuse of graphite from end-of-life Li-ion batteries, *in preparation*

This work was authored by the National Renewable Energy Laboratory, operated by Alliance for Sustainable Energy, LLC, for the U.S. Department of Energy (DOE) under Contract No. DE-AC36-08GO28308. This research is supported by the U.S. Department of Energy's **Vehicle Technologies Office** through the **ReCell Consortium**, overseen by DOE Program Manager **Brian Cunningham** and technology managers **Jake Herb** and **Tina Chen**, ReCell Center Director **Jeffrey Spangenberger**, and ReCell Center Deputy Director **Bryant Polzin**. The views expressed in this presentation do not necessarily represent the views of the DOE or the U.S. Government. The U.S. Government retains a nonexclusive, paid-up, irrevocable, worldwide license to publish or reproduce the published form of this work, or allow others to do so, for U.S. Government purposes.

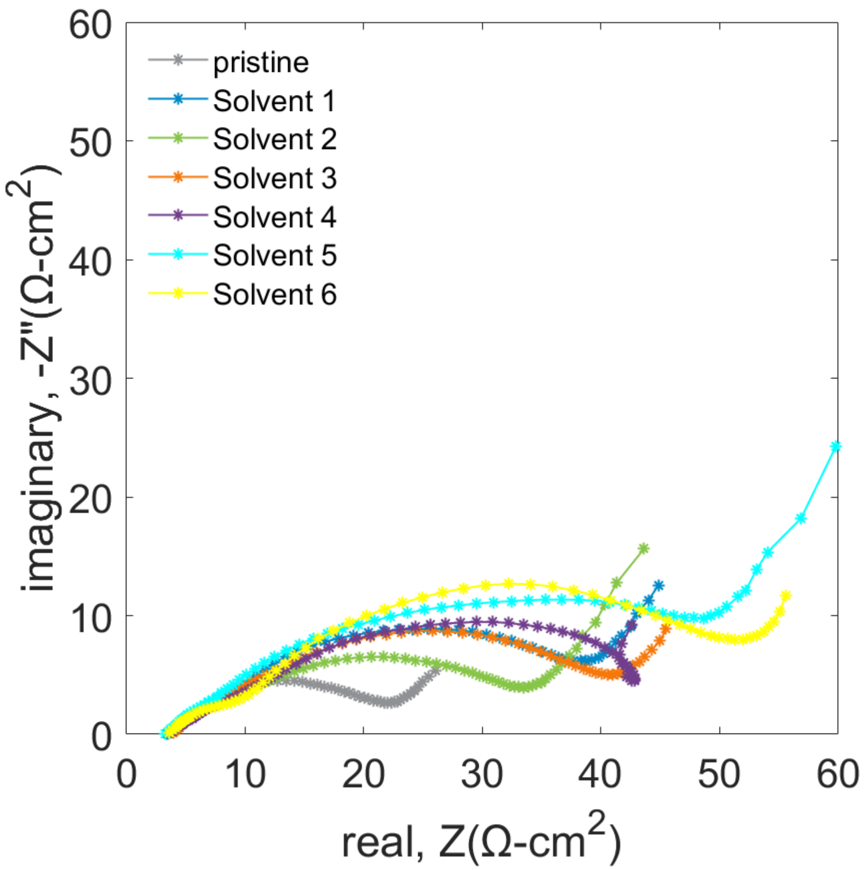
NREL *Transforming* ENERGY

AS-RECEIVED MATERIAL PROPERTIES

Parameter	Cathode	Anode
Chemistry	NMC-622	Artificial Graphite
Binder System	PVDF/NMP	PVDF/NMP
Substrate Areal Mass (mg/cm^2)	4.05	8.96
Active Material Proportion (wt%)	96	93
Areal Loading Single Sided (mg/cm^2)	23.1	14.0
Areal Capacity Single Side(mAh/cm^2)	3.96	4.58
1st Cycle Efficiency (%)	88	94
Typical Half Cell Voltage (V vs. Li/Li^+)	2.5 – 4.35	0.01 – 2.00
Current Collector Thickness	*not available*	11 um

DOWNSELECTION OF TREATMENT SOLVENTS

- A set of 6 solvents spanning a variety of chemical properties (polarity, proticity, molecular size) were initially surveyed.
- Of 6 initial solvents, **4 promising high-polarity protic solvents** (**Solvents 1-4**) were downselected for further detailed study.



Round 1	R_{SEI} (Ω-cm^2)	R_{CT} (Ω-cm^2)
Pristine	1.96 ± 0.87	17.41 ± 2.92
Water	8.77 ± 2.83	31.92 ± 2.29
Methanol	1.54 ±1.07	30.36 ± 2.23
Ethanol	2.12 ± 0.45	37.52 ± 1.03
Isopropanol	4.85 ± 0.34	36.79 ± 2.61

Hexane and acetone showed highest impedance in the initial screen.

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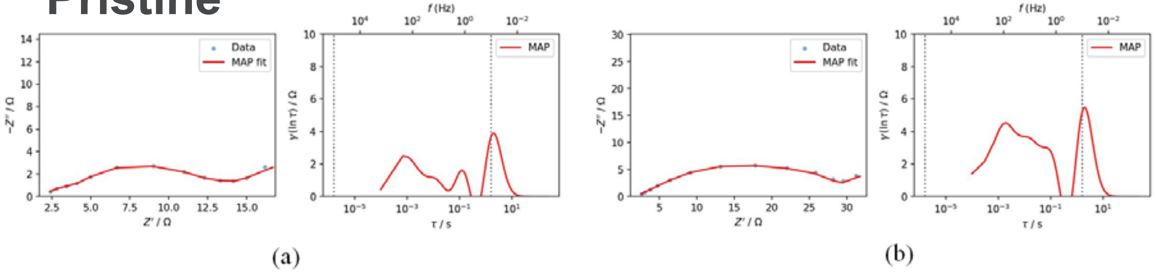
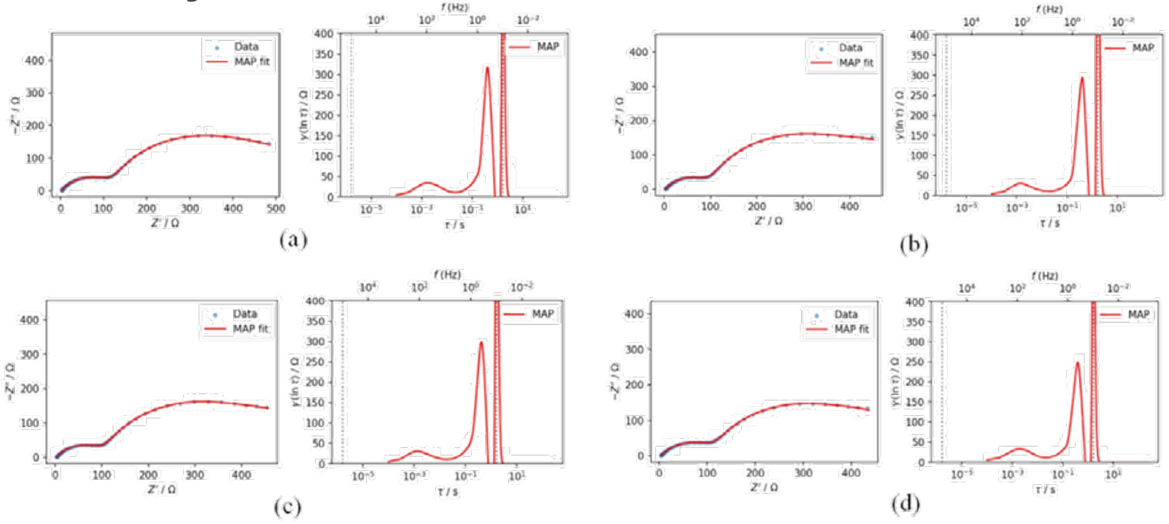
IMPEDANCE: DRT

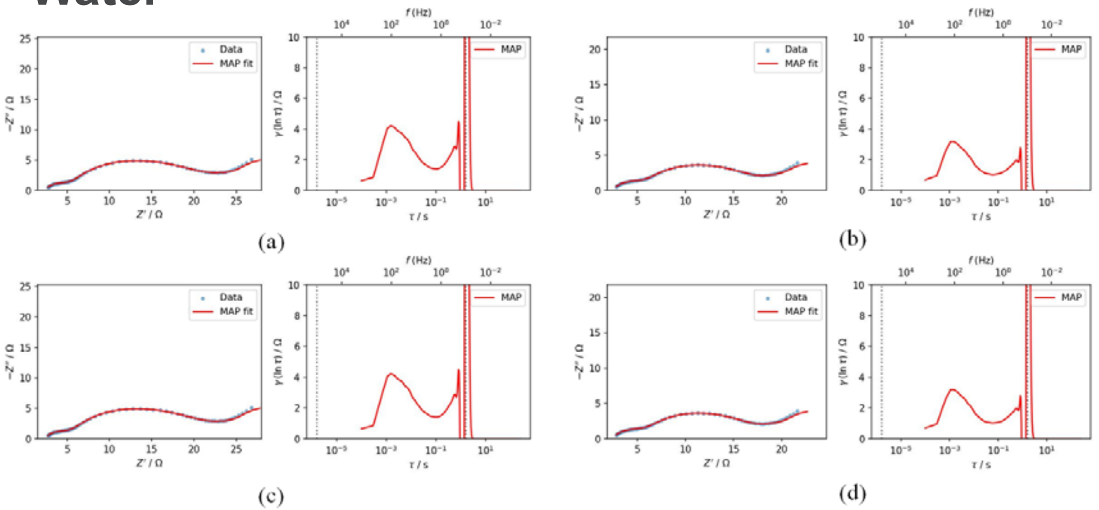
Pristine



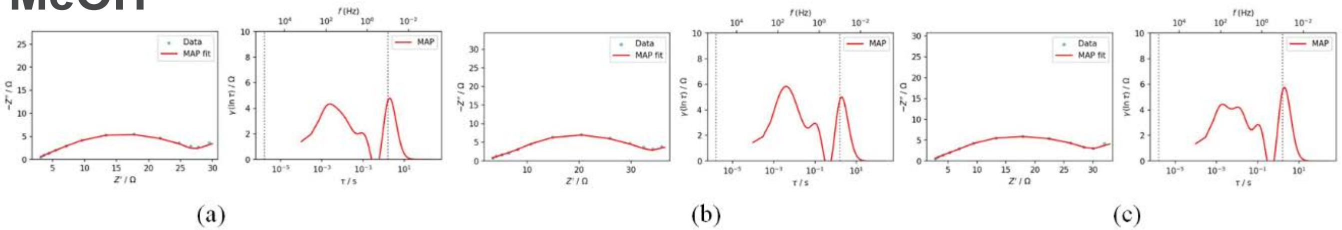
EOL Cycled



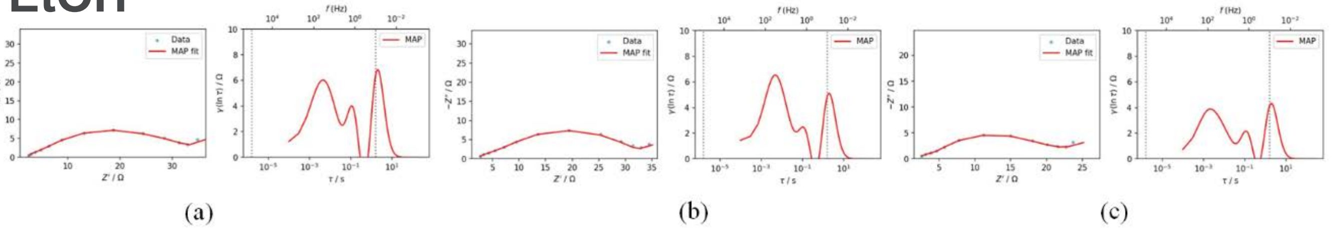
Water



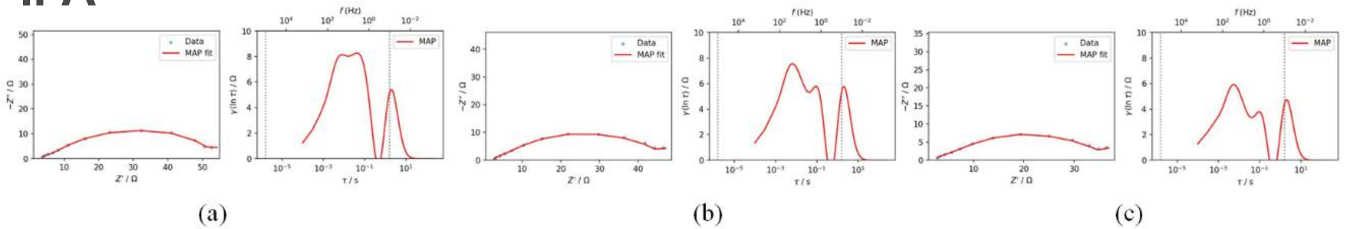
MeOH



EtOH



IPA



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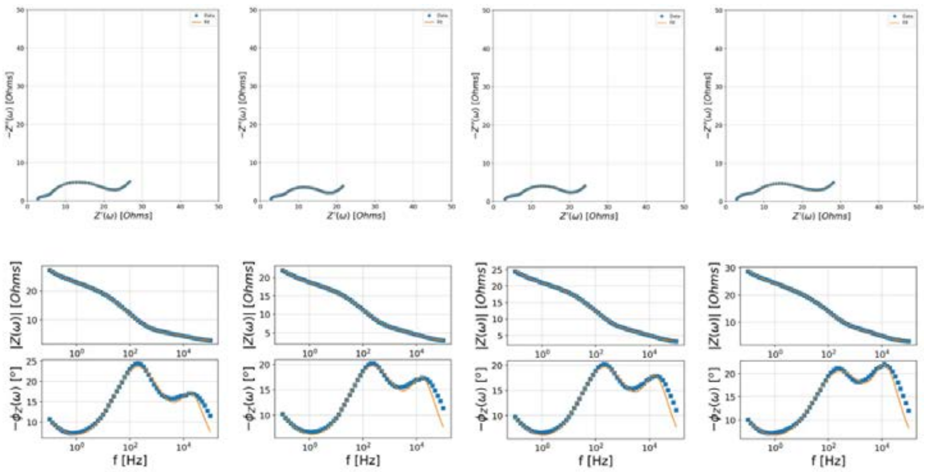
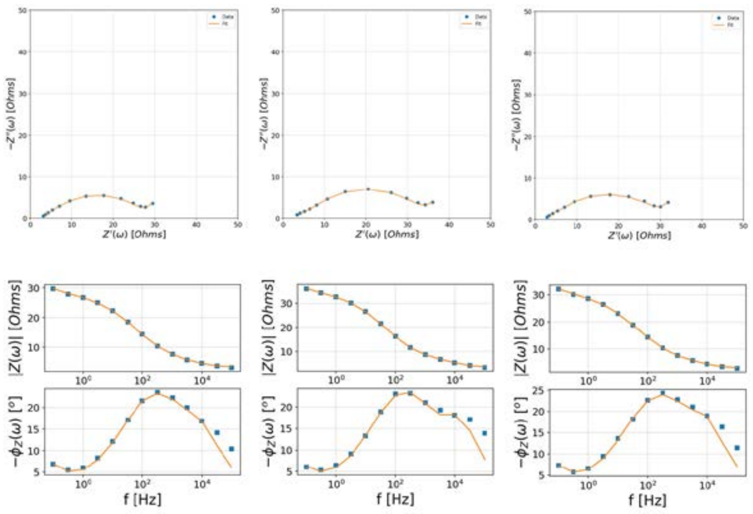
IMPEDANCE: ECM FITS

Water



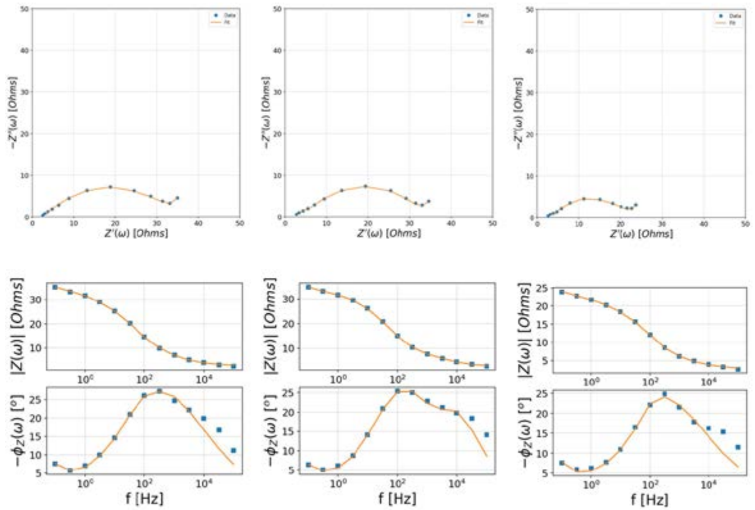
(a) (b) (c) (d)

MeOH



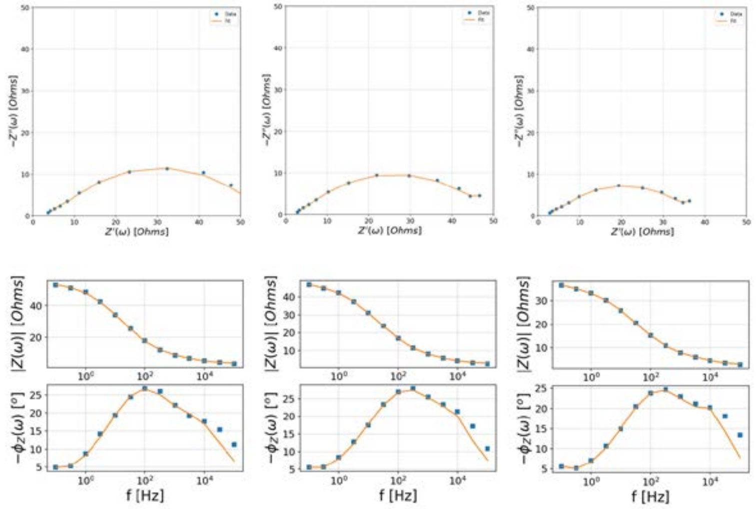
(a) (b) (c)

EtOH



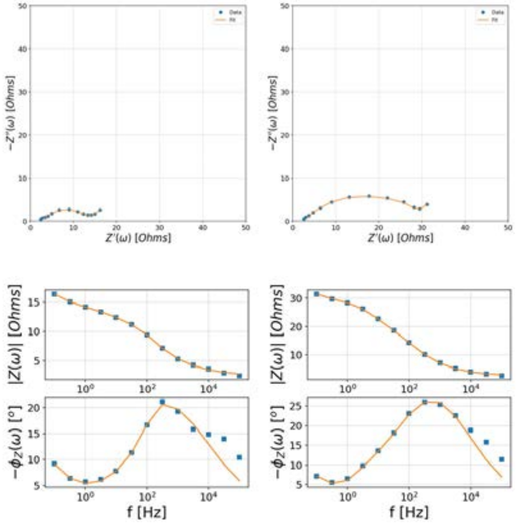
(a) (b) (c)

IPA



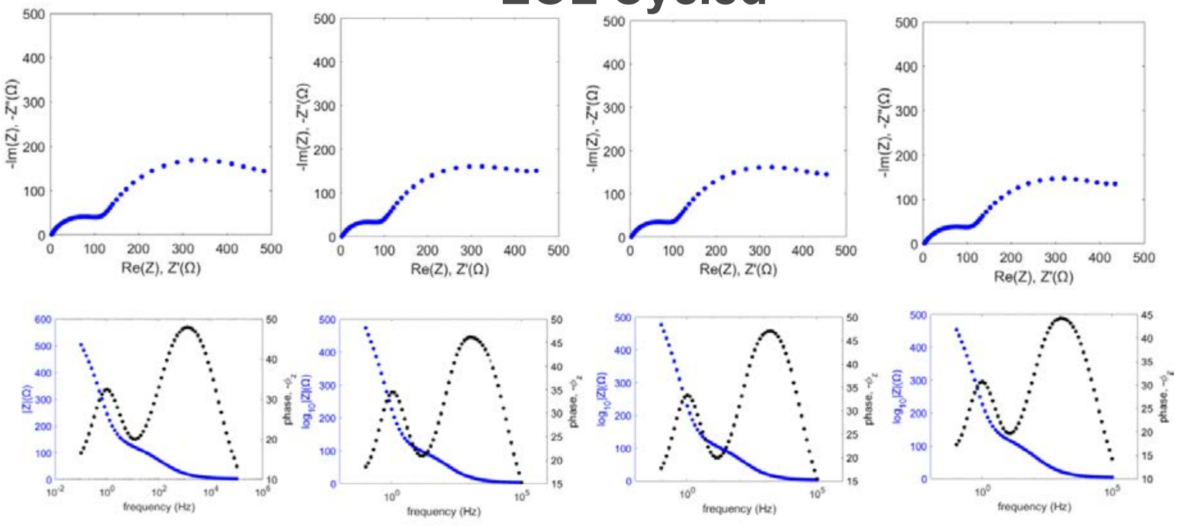
(a) (b) (c)

Pristine



(a) (b)

EOL Cycled

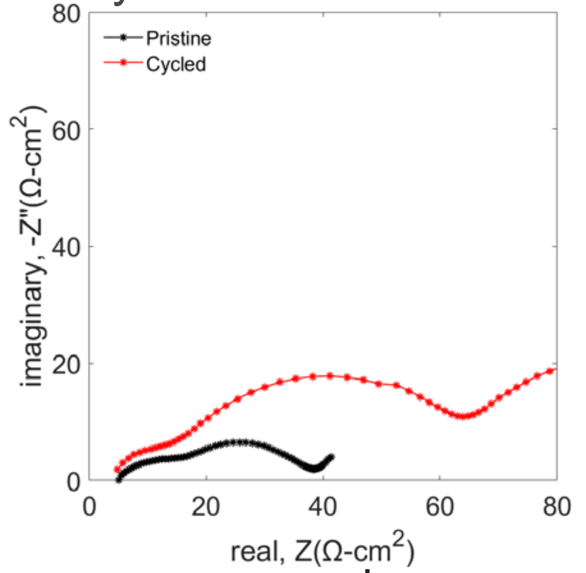


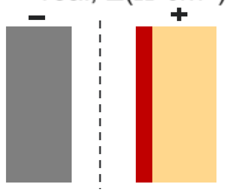
(a) (b) (c) (d)

IMPROVING PHYSICALLY MEANINGFUL EIS DATA

Work in progress to improve usefulness of EIS data & ECM fits

Impedance data collected on symmetric cells to date:

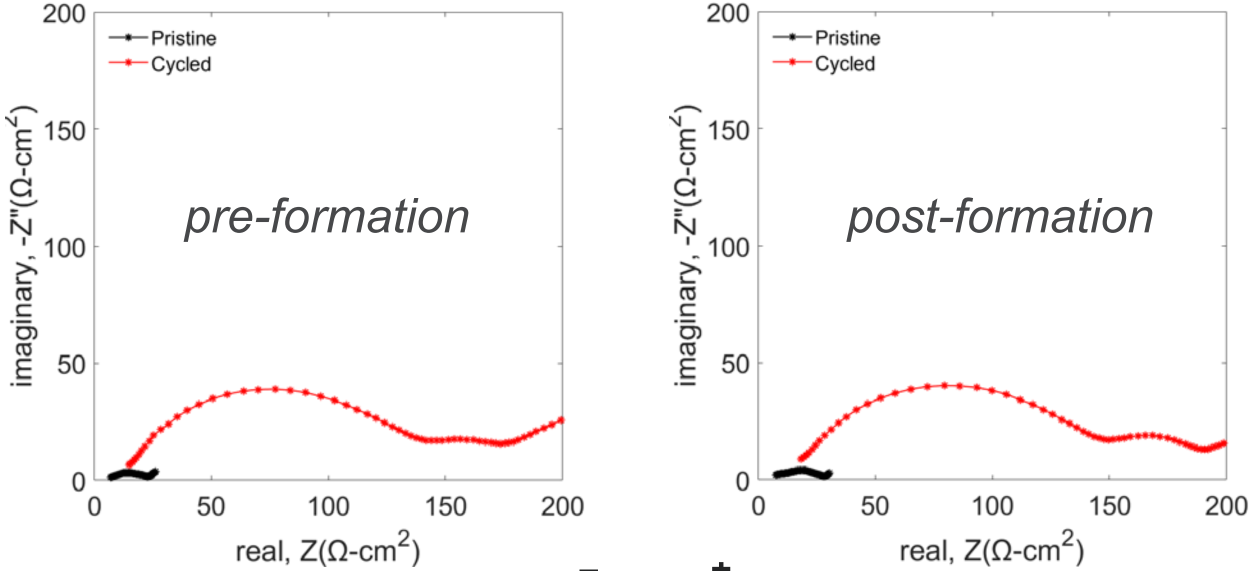


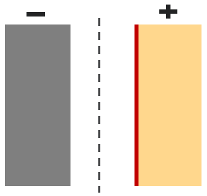


As-received or as-treated Gr

Lithiated Gr:
1 x C/20 cycle + lithiation to 5 mV

Impedance data collected on symmetric cells using newly developed approach:





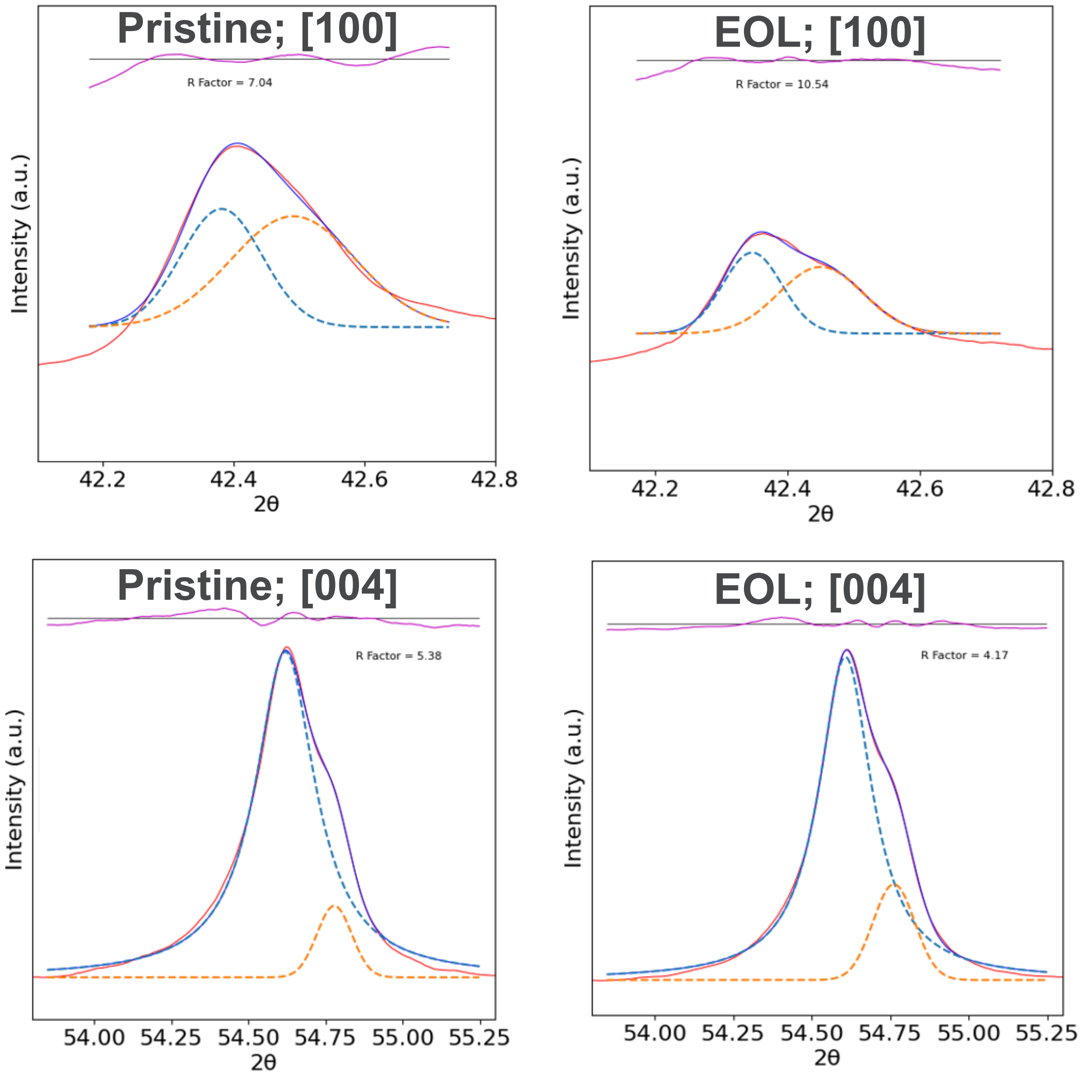
As-received or as-treated Gr

Lithiated Gr:
lithiation to 5 mV *only*

- New approach to collecting impedance data should yield "truer" values of SEI resistance: minimizes convolution introduced by additional SEI grown on the lithiating electrode during the C/20 cycle.
- Impedance will be measured both before and after C/10 formation cycles.
- Separate half cells will be constructed to obtain C/20 data required for P2D model validation.

UPCYCLING EFFECTS: STRUCTURAL ANALYSIS

- Anode powder + 20 wt% Si internal standard (325 mesh; 99% metals basis); triplicate samples
- High-resolution scans collected from 42–48° 2θ and 53.75–55.25° 2θ (0.001° step interval; 0.1° min^{-1})
- 2θ peak positions corrected based on Si standard location



- Pseudo-Voight fits: deconvolution into minimum number of peaks (2-3) for acceptable R-factor fit quality[1]
- Each deconvoluted peak analyzed separately
- $d_{(00l)}$ and crystallite size calculated as weighted average of the based on relative intensities of calculated constituent peaks:[2]

$$d(004) = X_i = \frac{I_i}{\Sigma_{i=1}^{n} I_i}$$

- Interplanar spacing $d_{(004)}$ calculated according to Bragg's Law:

$$d_{(004)} = \frac{n\lambda}{2\sin(\theta)} \quad \text{with } \lambda = 1.5406\ \text{Å and } n = 1$$

- Crystallite height (L_c) calculated by applying Scherrer's equation to the [004] peak:[3]

$$L_c = \frac{0.9\lambda}{(\beta_{004}\cos(\theta_{004}))}$$

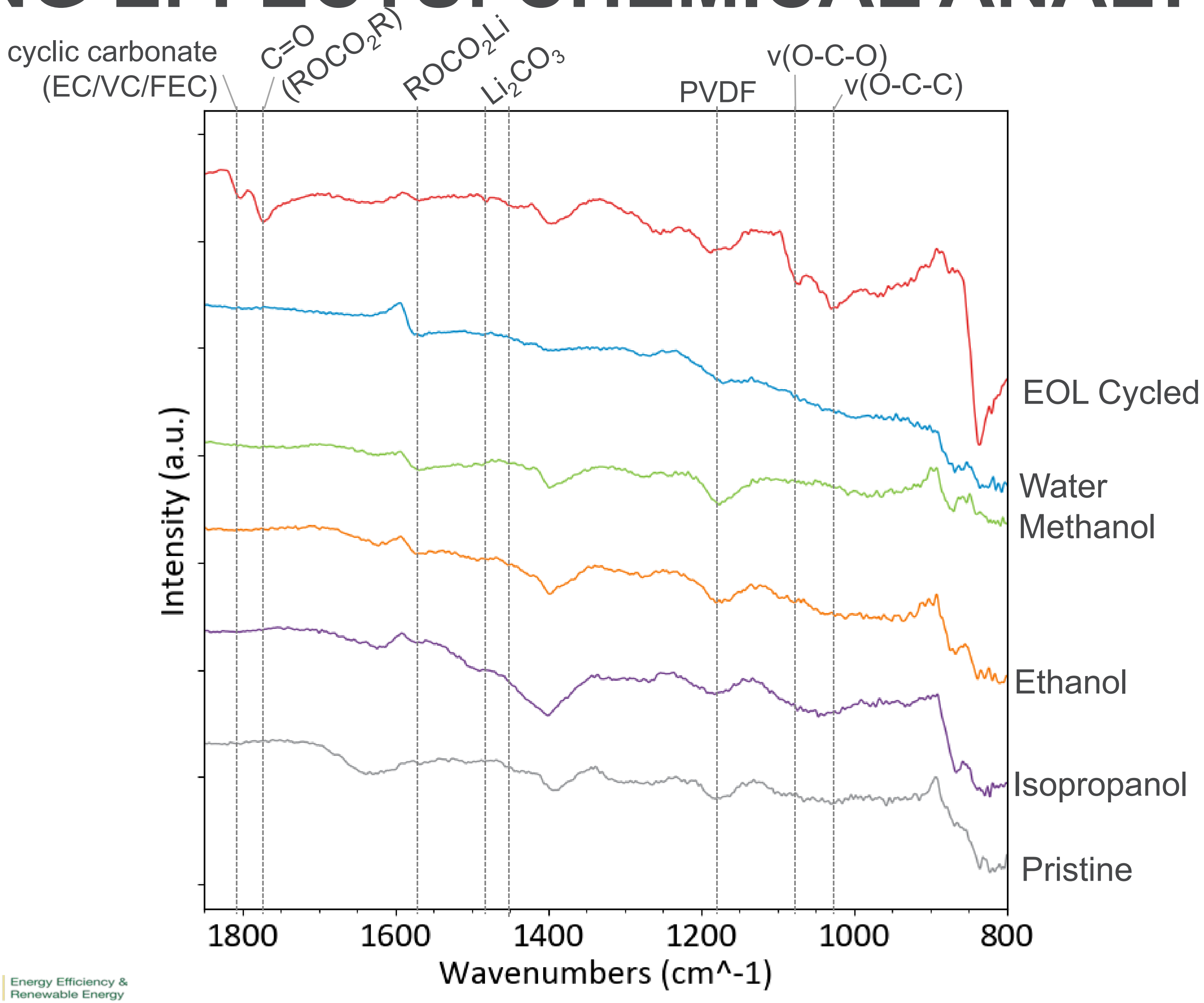
- Crystallite height (L_a) calculated by applying Scherrer's equation to the [100] peak:[3]

$$L_a = \frac{0.9\lambda}{(\beta_{100}\cos(\theta_{100}))}$$

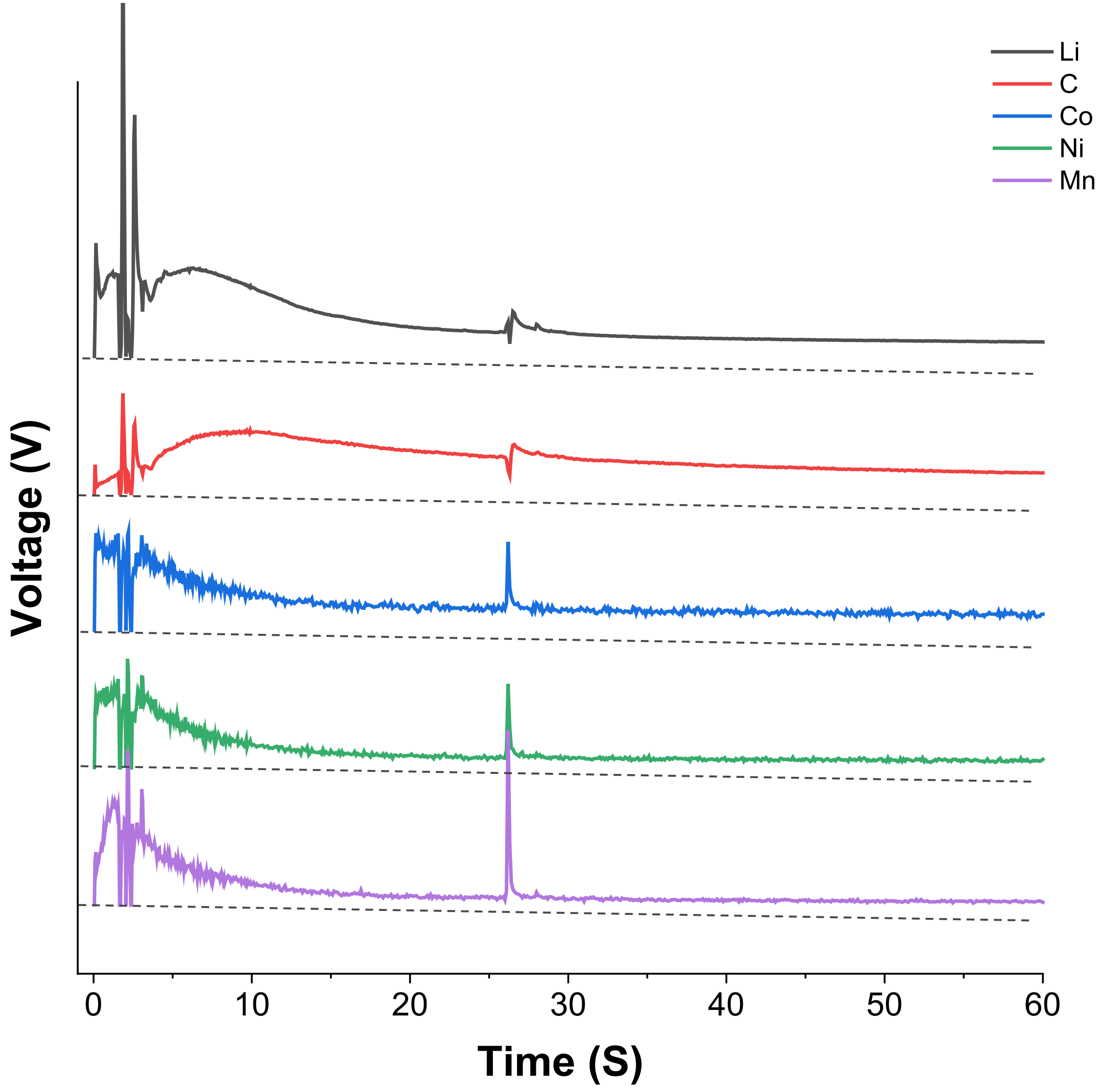
- $\beta_{(hkl)}$ = FWHM of the constituent peak; K (shape factor) = 0.9

(1) Iwashita et al., *Carbon N Y* **42**, 701–714 (2004); (2) Zou et al., *Mater Chem Phys* **82**, 654–662 (2003); (3) Popova et al., *Coke and Chemistry* **60**, 361–365 (2017).

UPCYCLING EFFECTS: CHEMICAL ANALYSIS



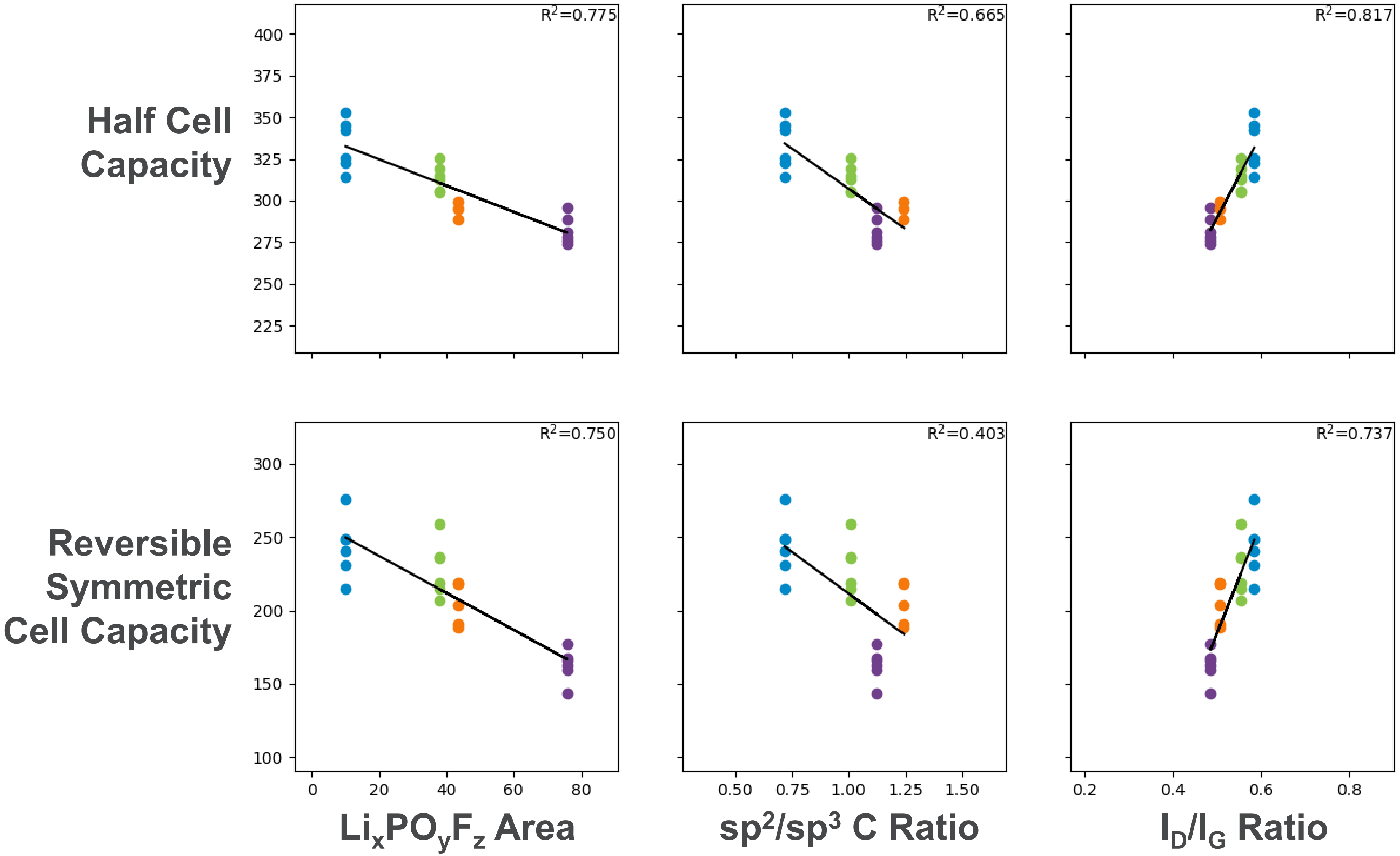
UPCYCLING EFFECTS: CHEMICAL ANALYSIS



- Glow discharge optical emission spectroscopy (**GDOES**) is an analytical tool that provides *elemental analysis* concurrent with *depth profiling* in solid materials, such that depth-resolved composition can be determined.
- The Y-axis value is a normalized signal; quantification requires a standard sample with known elemental ratios
- The observed fluctuations at ~2 mins and ~26 mins are attributed to the abrupt emission of a large number of particles during the electrode sputtering process.
- GDOES analysis suggests that *TM ions* may be present at within the EoL cycled anodes, and that such TM ions are present preferentially at the surface of the graphite. Such TM ions were not observed through elemental analysis in any of the upcycled samples.
- The depth resolution of GDOES offers complementary information to traditional surface-sensitive XPS methods. Further co-development and optimization of GDOES techniques for this material will be pursued with the Post-Test Facility in FY24.

MORE ALTERNATIVE (USEFUL) CORRELATIONS

Physico-chemical signatures vs electrochemical performance



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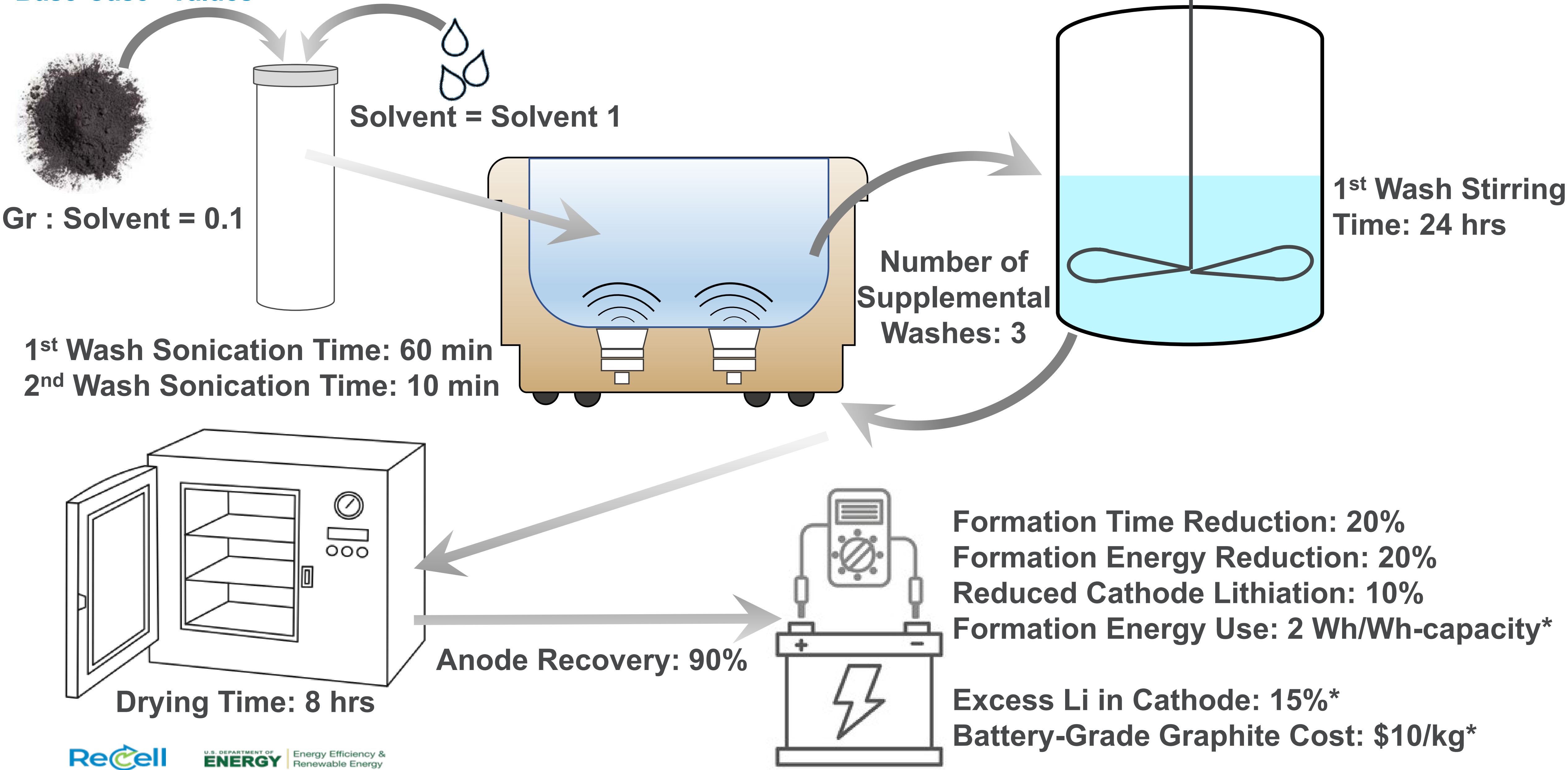
SENSITIVITY ANALYSIS: DEFINING THE “BASE CASE”

“Base-Case” Values



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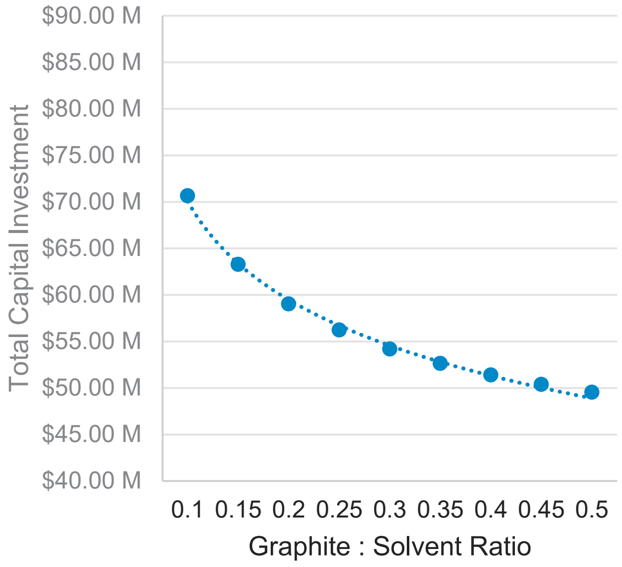
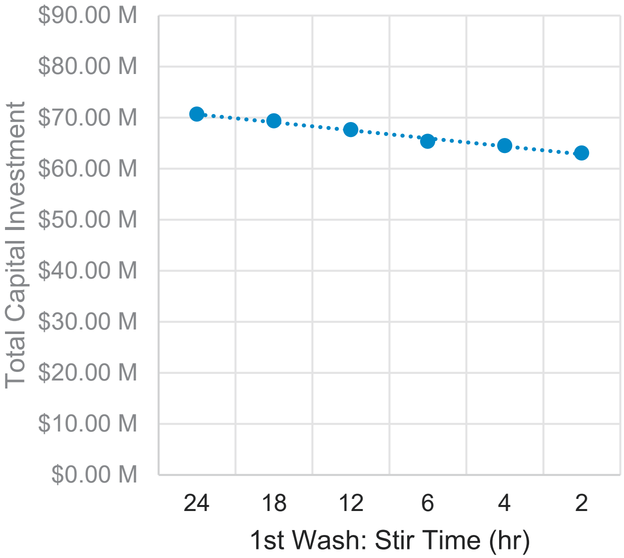
*Sensitivity analysis on Slide 41

SENSITIVITY OF PROCESS COST CONTRIBUTIONS

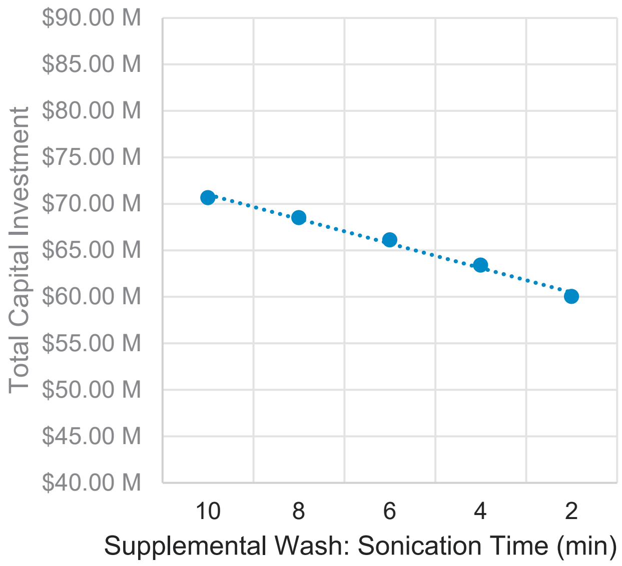
Impacts of Varying Process Parameters on Capital Costs

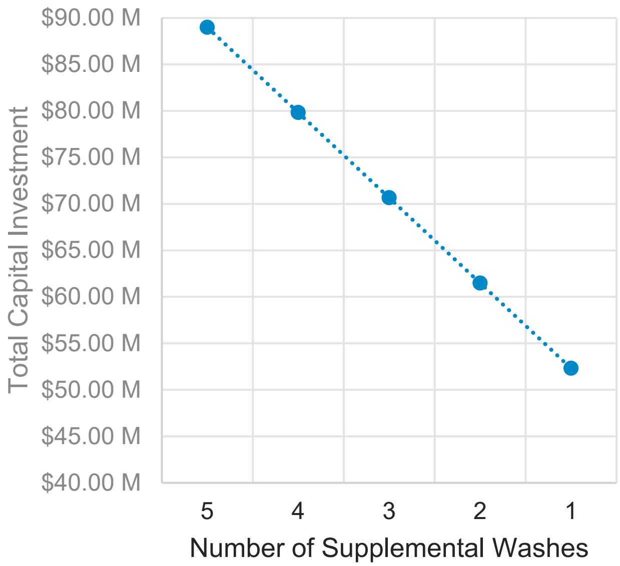
Trendlines shown for visual guide only; note x-axis scales are not always linear.

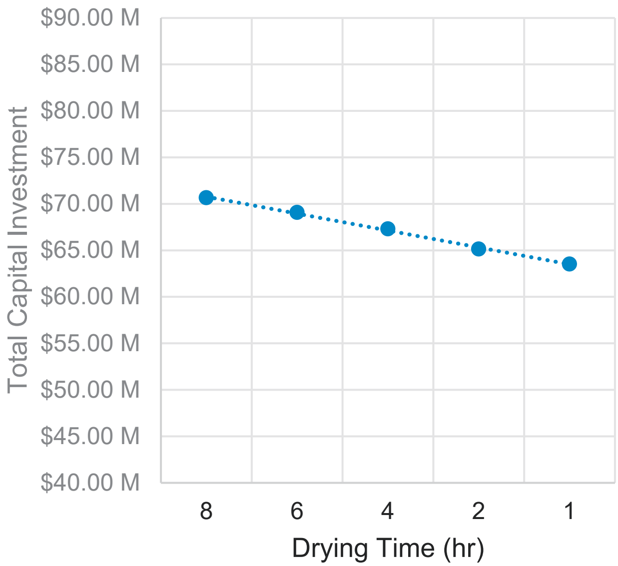




<u>Assumption</u>: Total plant throughput = 10,000 MT/yr *(EverBatt default for modeling ReCell processes)*







Most substantial reduction in capital costs arises from:
1. Reducing # of washes
2. Increasing Gr : solvent ratio
3. Reducing sonication time

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SENSITIVITY OF "ADDED VALUE" FACTOR CONTRIBUTIONS

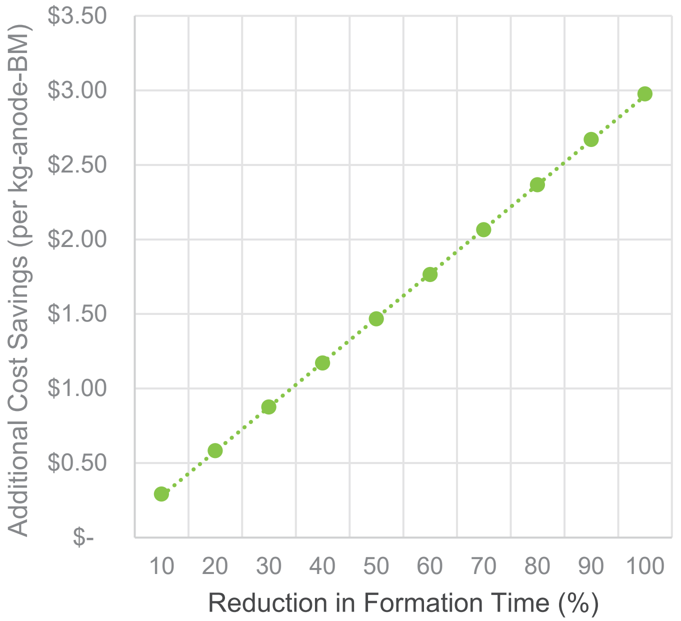
Impacts of Varying Performance/Recovery Metrics on Added Value

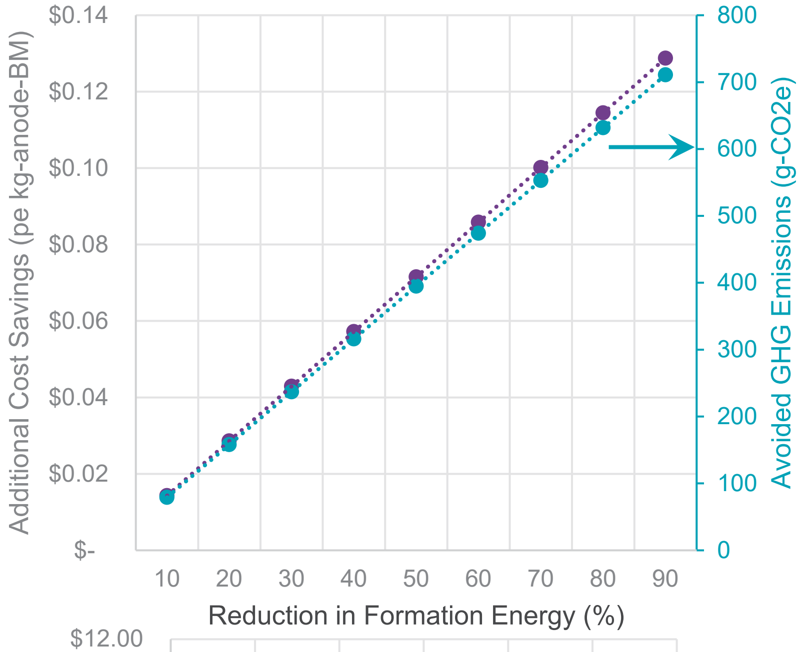
Trendlines shown for visual guide. Note varying y-axis scales.

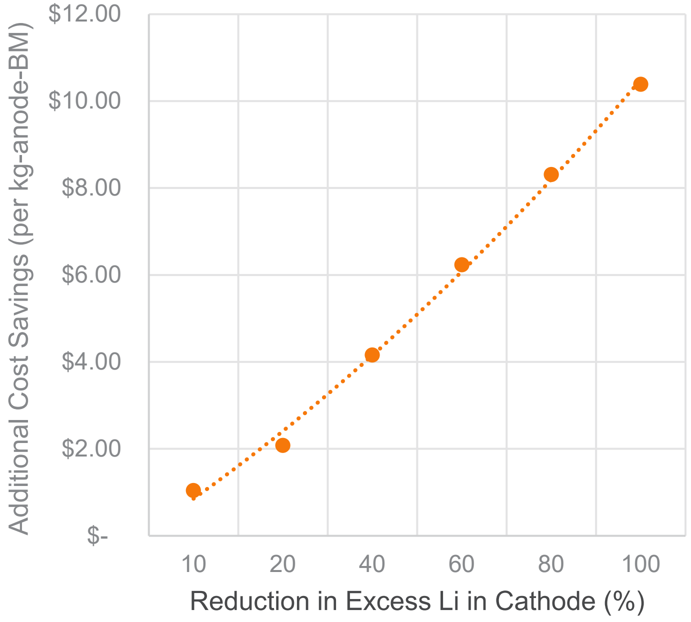
In all cases, additional cost savings reflect the added economic benefit associated with utilizing an upcycled anode product.

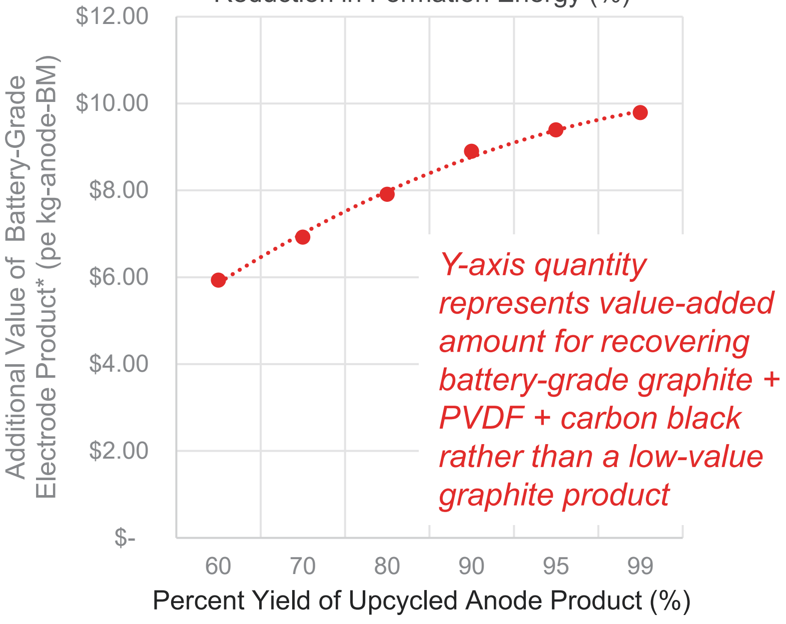
- *Performance metrics* associated with the upcycled anode that add value include reducing formation time, reducing formation energy, and reducing excess Li in the cathode
- The *recovery metric* captures the added value provided by recovering a battery-grade electrode composite material, rather than a low-value graphite product
- The cost savings impact of the four primary "added value" streams associated with the upcycled anode product follows as:

  Recovery of Electrode Product ~
  Reduction in Excess Li >
  Reduction in Formation Time >
  Reduction in Formation Energy
- Formation time reduction only influences capital expenses (equipment, facility size)
- Reducing formation energy offers electricity cost savings + GHG emission reduction benefit









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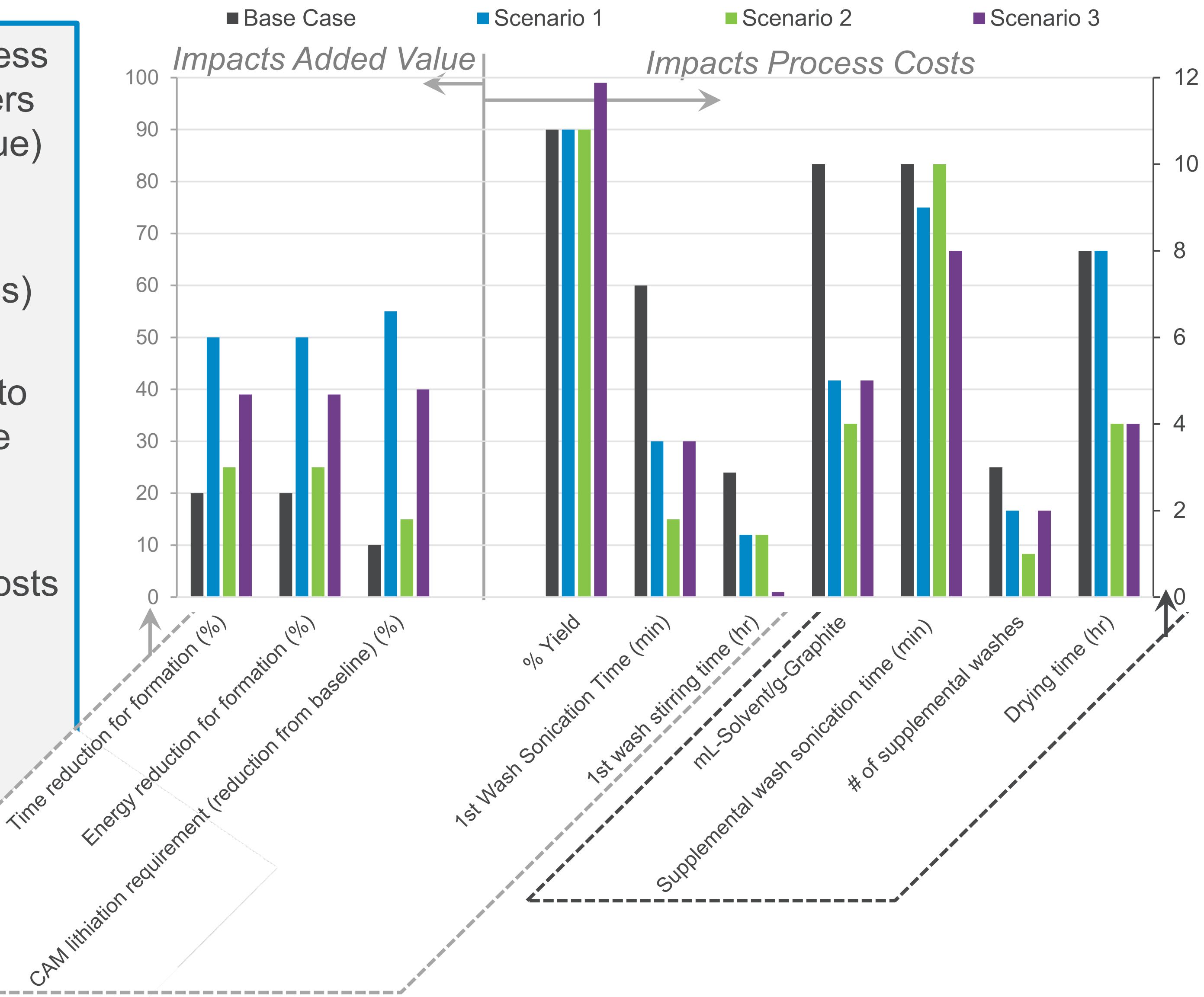
PARAMETER TUNING: “BREAK-EVEN” SCENARIOS

Example Break-even Scenarios (Cost = Added Value) for Solvent 2

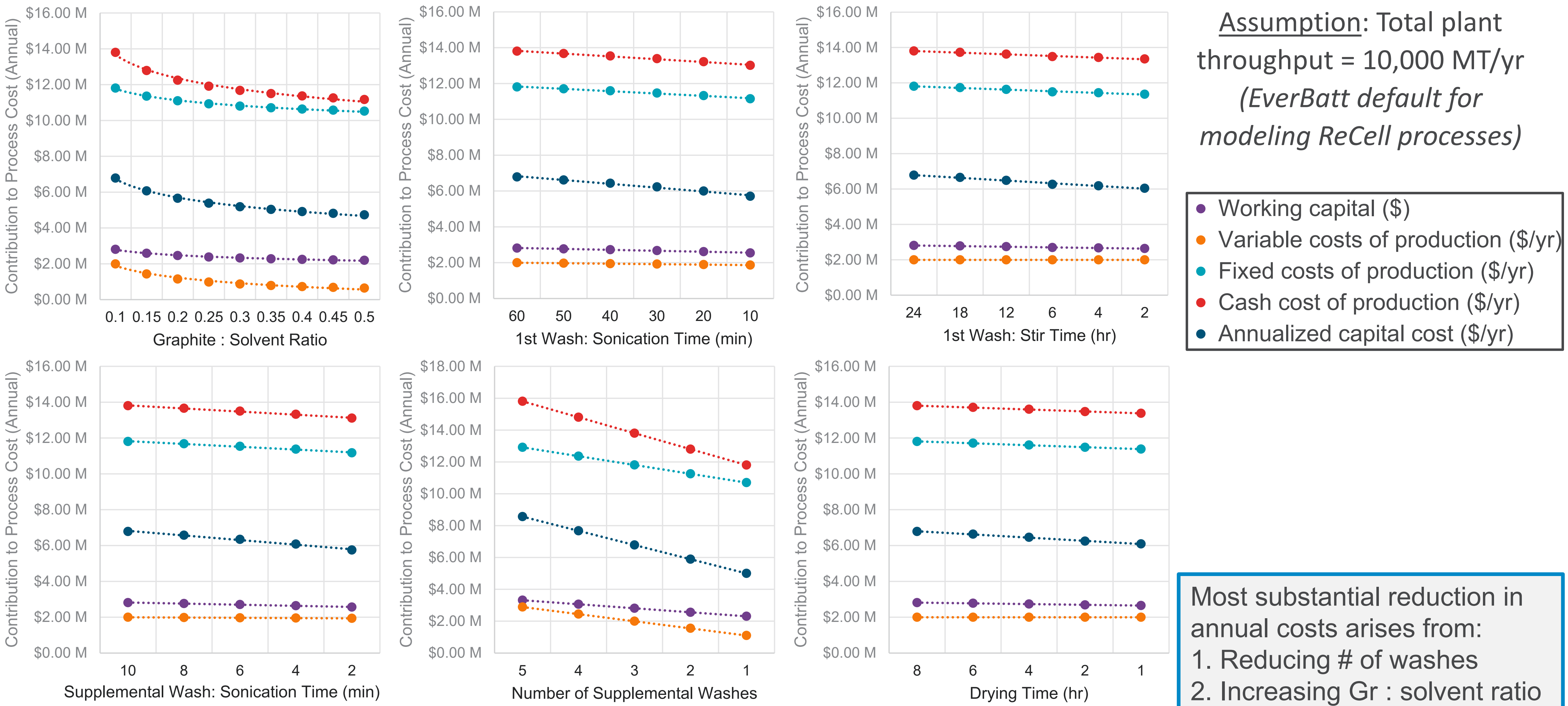
- Break-even scenarios demonstrate combinations of process parameters and added value realized by cell manufacturers that result in a “break-even” point (i.e., costs = added value)
- All break-even scenarios are reported for Solvent 2:
  -Shows promising technical results (reduced resistance, relatively high symmetric cell efficiency = reduced Li loss)
  -More expensive than Solvent 1
- Various combinations of process parameter adjustments to reduce costs + improved product performance to increase value result in the breakeven condition
- Increasing the added value – i.e., improving technical performance of anode – can counteract higher process costs
- In all cases shown, reducing the number of supplemental washes (less than 3) supports achieving breakeven condition
  - Reducing # of sonication steps reduces # of sonicator units purchased, presuming continuous operation
  - Note: Identifying cheaper alternative process intensification methods with lower cost would achieve same outcome



SENSITIVITY OF PROCESS COST CONTRIBUTIONS

Impacts of Varying Process Parameters on Annual Operating Costs

Trendlines shown for visual guide only; note x-axis scales are not always linear.

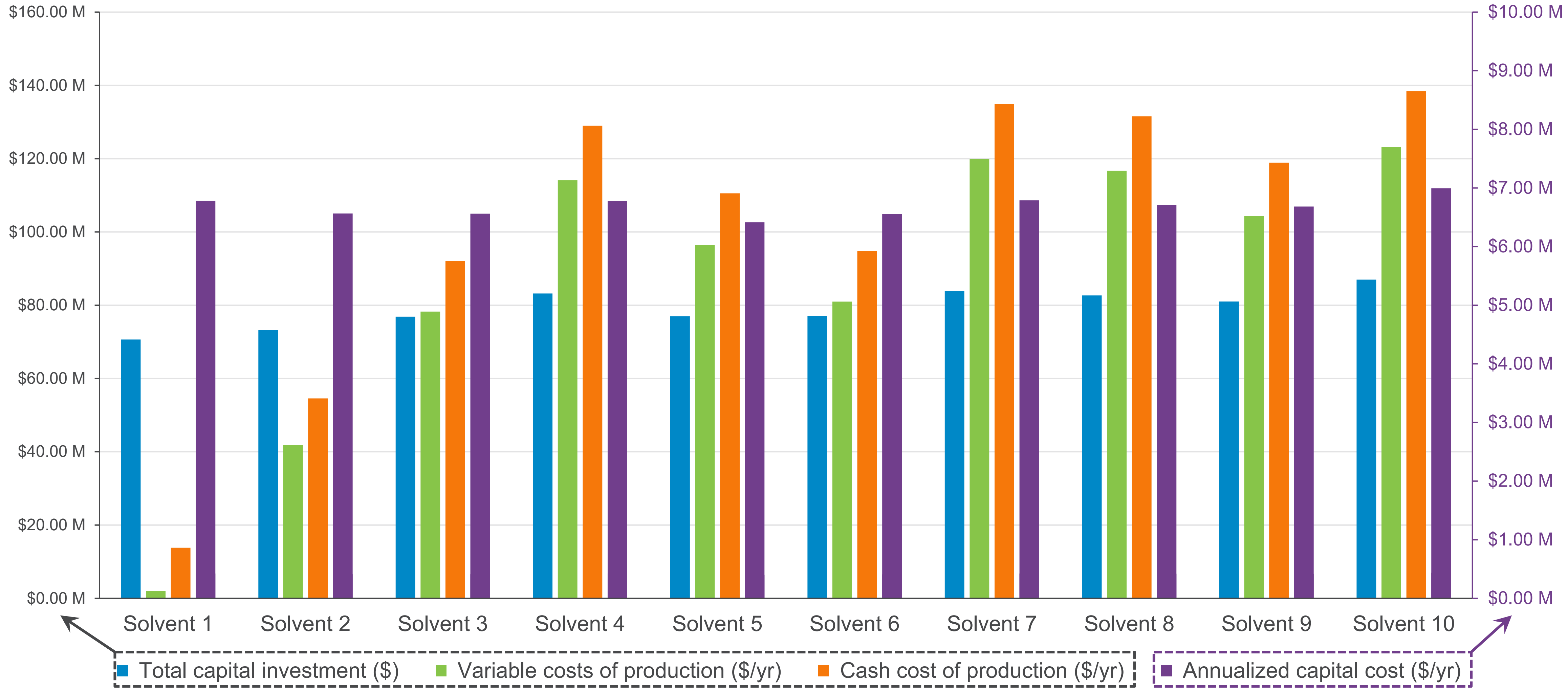


<u>Assumption</u>: Total plant throughput = 10,000 MT/yr *(EverBatt default for modeling ReCell processes)*

Most substantial reduction in annual costs arises from:
1. Reducing # of washes
2. Increasing Gr : solvent ratio

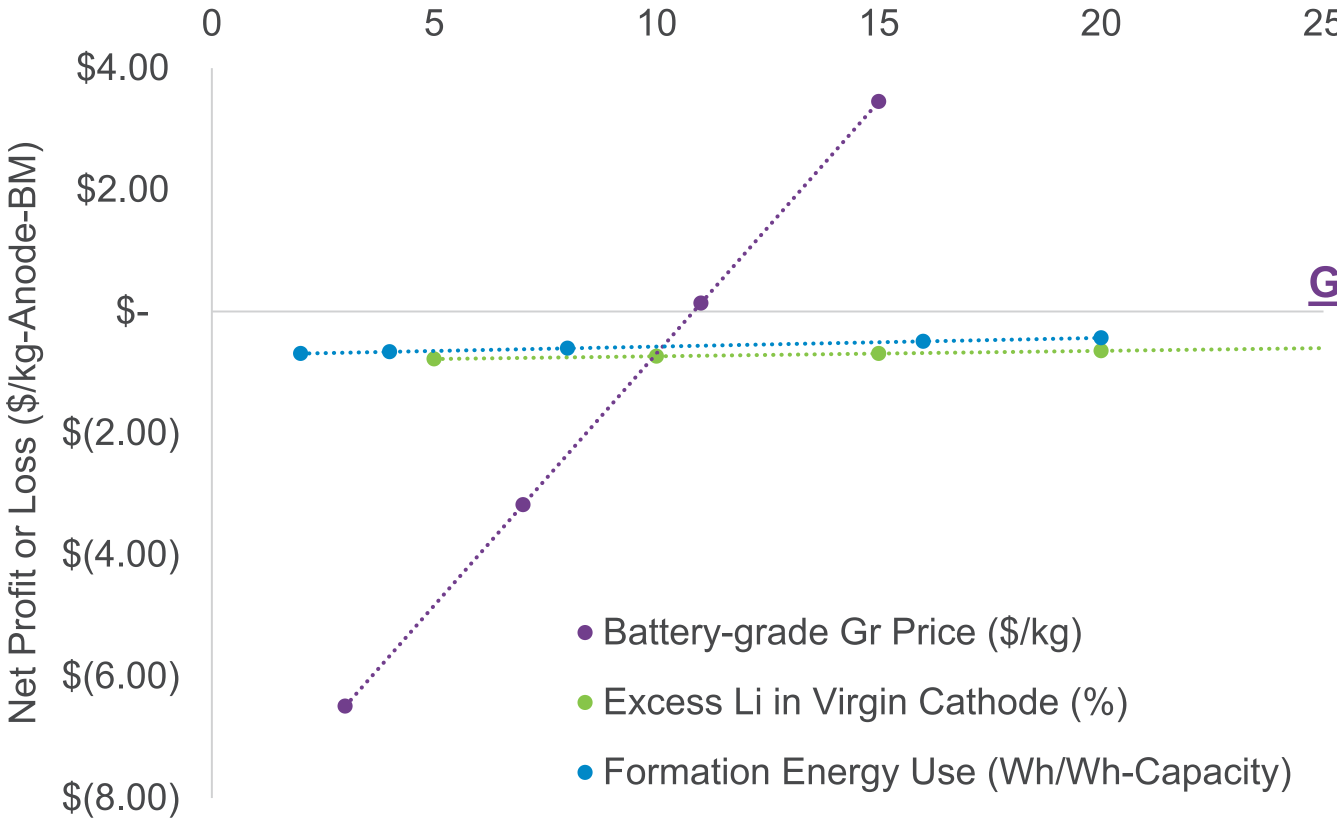
SENSITIVITY OF SINGLE-SOLVENT CHOICE

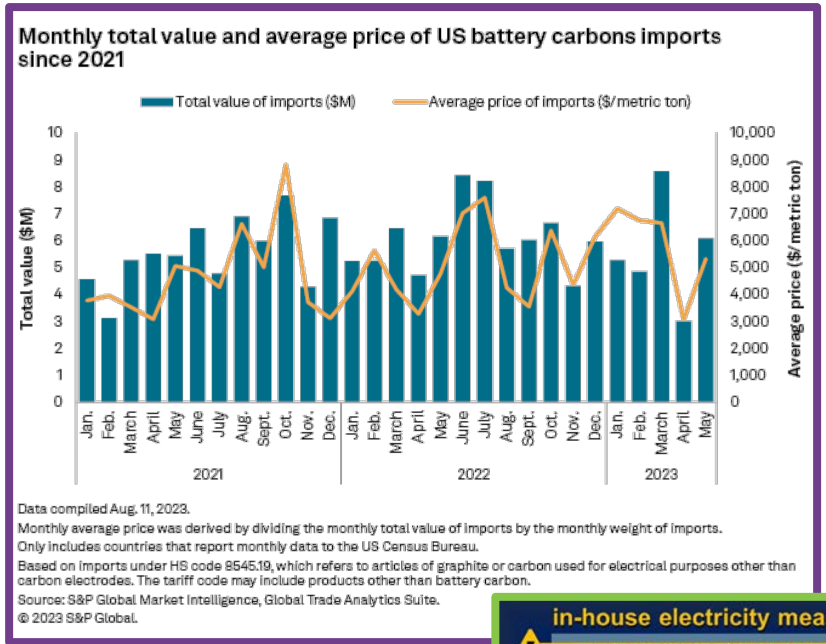
Impacts of Varying Solvent Identity on Process Costs (Base Case Process Parameters)



SENSITIVITY OF SELECTED "BASE CASE" PARAMETERS

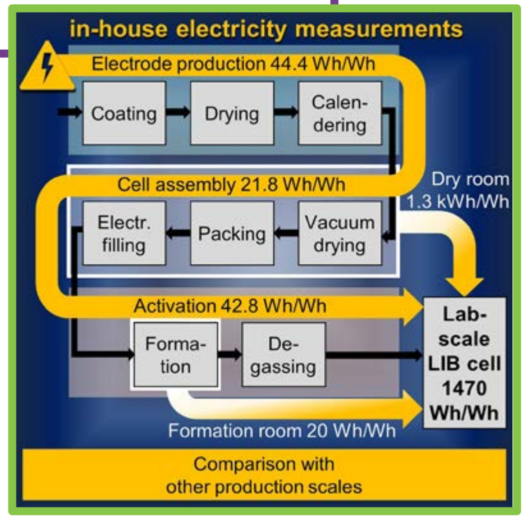
- Sensitivity analysis conducted using "Base-Case" scenario parameters and varying three assumed parameters (see Slide 5)
- Net profit/loss is not highly sensitive to assumed values of formation energy use or excess Li in virgin cathode
- Net profit/loss is strongly sensitive to assumed value of battery-grade graphite price, which is known to be highly volatile (*see inset*)





Graphite Price – Estimate Range:

Based on 2021-2023 average price of graphite imports[1]



Excess Li in Cathode – Estimate Range:

Based on spread around approx. reported loss to SEI (10-15%)[2]

Formation Energy Use – Estimate Range:

2 Wh/Wh = industrial estimate (Q. Dai)

20 Wh/Wh = pilot-scale estimate[3]

ReCell ADVANCED BATTERY RECYCLING

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[1]S&P Global Market Intelligence (2023); [2]Shim, J. et al., *J. Power Sources* 119-121 (2003): 934; [3]Erakca et al., iScience 24 (2021),102437