AN INNOVATIVE APPROACH FOR ELECTROLYTE RECYCLING FROM LITHIUM-ION BATTERIES
- Sub - and Supercritical Carbon Dioxide Extraction -

Nils Zachmann*, Martina Petranikova, Burçak Ebin

Department of Chemistry and Chemical Engineering, Industrial Material Recycling Group

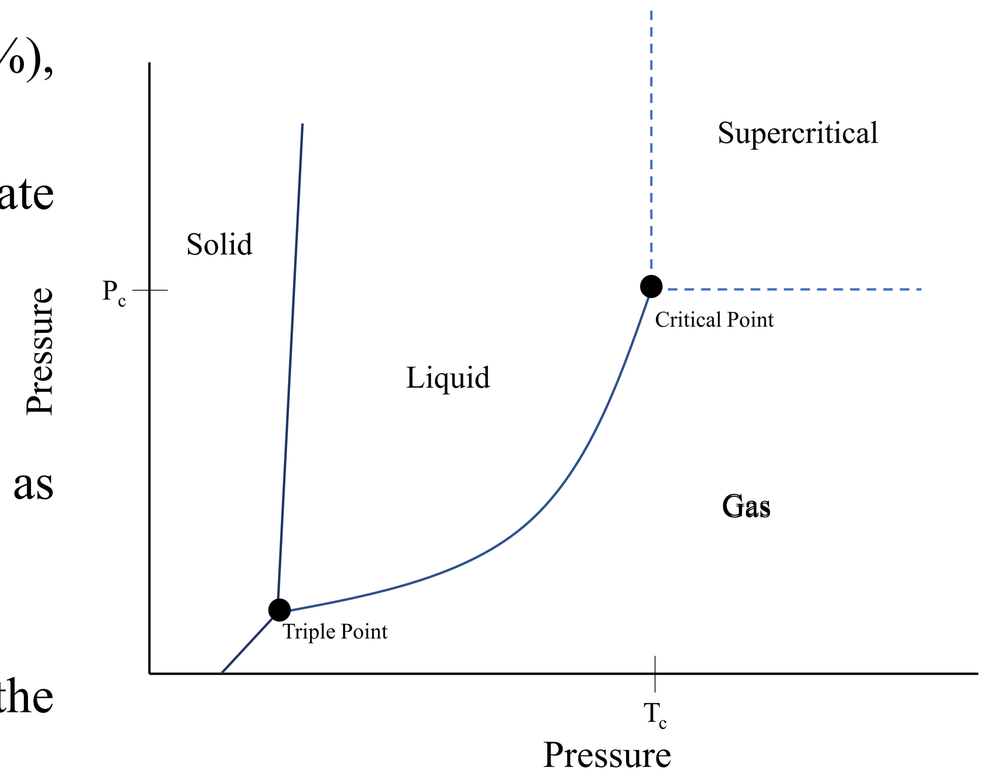
Chalmers University of Technology, SE-41296 Gothenburg, Sweden

**zachmann@chalmers.se*

BACKGROUND

- ❑ **LiB composition:** Cathode (25-35wt%), Anode (15-25wt%), Electrolyte (10-20wt%), Separator (4-10wt%)
- ❑ **Electrolyte composition:** Dimethyl Carbonate (DMC), Ethyl Methyl Carbonate (EMC), Ethylene Carbonate (EC), Lithium Hexafluorophosphate ($LiPF_6$)
- ❑ Recycling strategies focus mainly on valuable cathode active material recovery
- ❑ Electrolyte recovery is seldomly considered
- ❑ Instead, uncontrolled evaporation during common pre-treatment steps as well as degradation at elevated thermal treatment temperatures
- ❑ 10-20wt% loss of potentially recoverable material
- ❑ Controlled and safe recycling of the electrolyte inevitable to minimize the environmental impact of the recycling processes

SUPERCRITICAL CARBON DIOXIDE



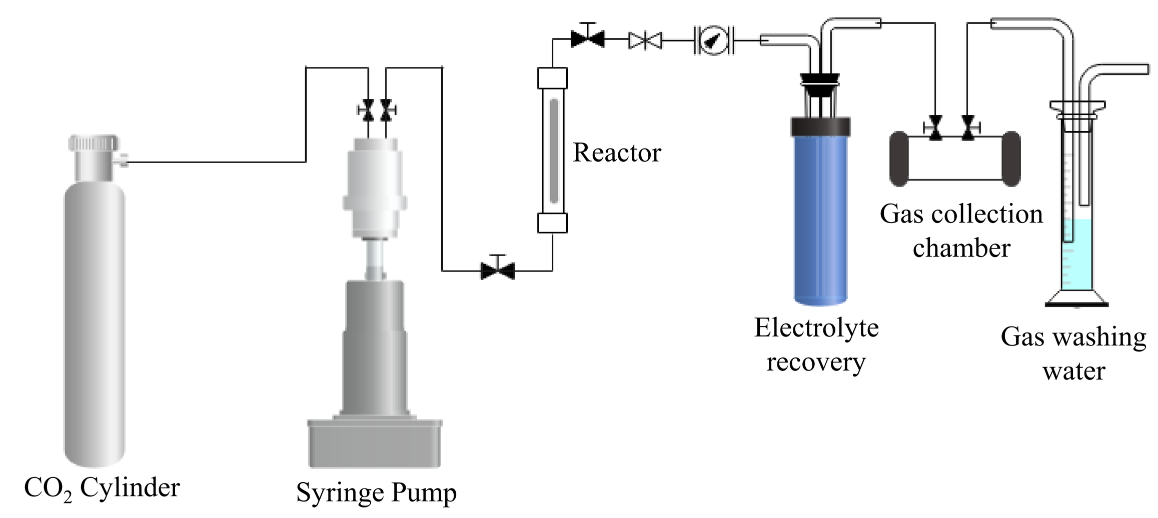
- ❑ **Critical point**: P = 73.8 bar, T = 31°C
- ❑ **Unique Properties:**
  - ➢ Gas-like viscosity
  - ➢ Gas-like diffusion coefficient
  - ➢ Liquid-like density
  - ➢ Neglectable surface tension
- ❑ **Advantages:**
  - ➢ Non-flammable, Non-toxic
  - ➢ Abundant
  - ➢ Mass transfer characteristics
  - ➢ Low reactant and processing cost

METHODS

Critical process parameters:

- ○Pressure
- ○Temperature
- ○Flow-rate
- ○Process time

Experimental set-up:

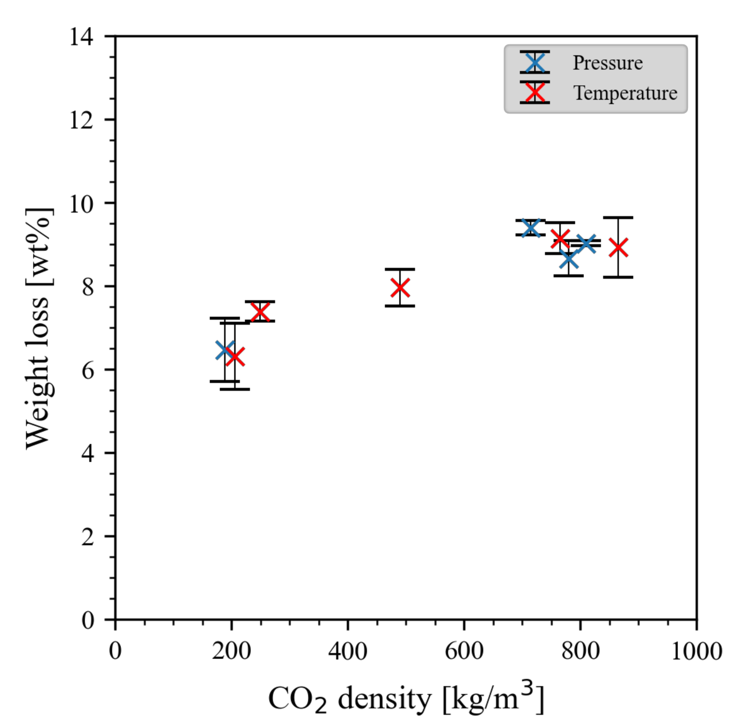


Analysis:

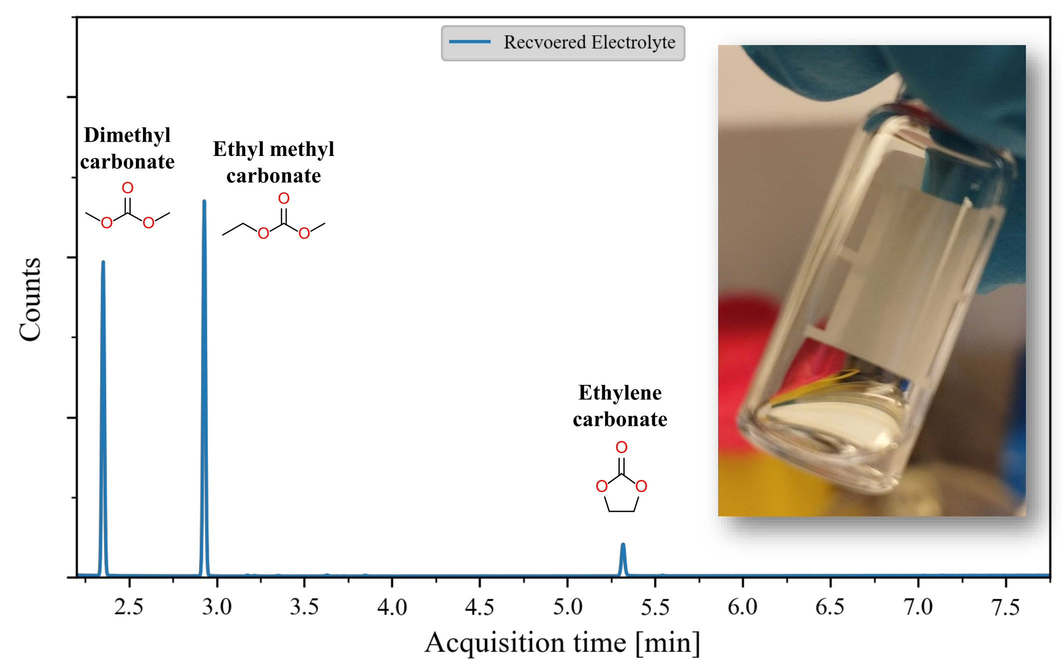
- ✓ Electrolyte recovery rate
  - ➢ *Weight loss of sample*
- ✓ Recovered Electrolyte
  - ➢ *Gas-Chromatography Mass spectroscopy (GC-MS)*
  - ➢ *Fourier Transform Infrared Spectroscopy (FT-IR)*
  - ➢ *Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES)*
- ✓ Process exhaust gas
  - ➢ *Fourier Transform Infrared Spectroscopy (FT-IR)*
- ✓ Impact on cathode material
  - ➢ *X-Ray Diffraction (XRD)*

RESULTS

LiB sample weight loss [wt%] after the extraction process at various densities.

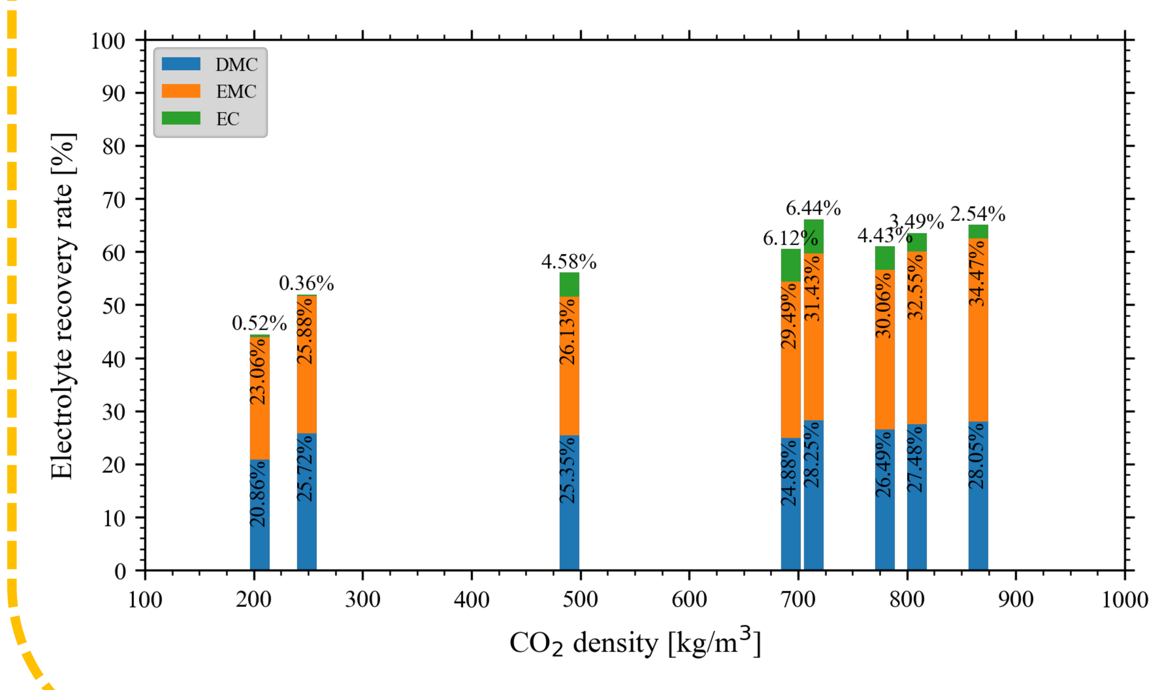


Recovered electrolyte by $scCO_2$ extraction.

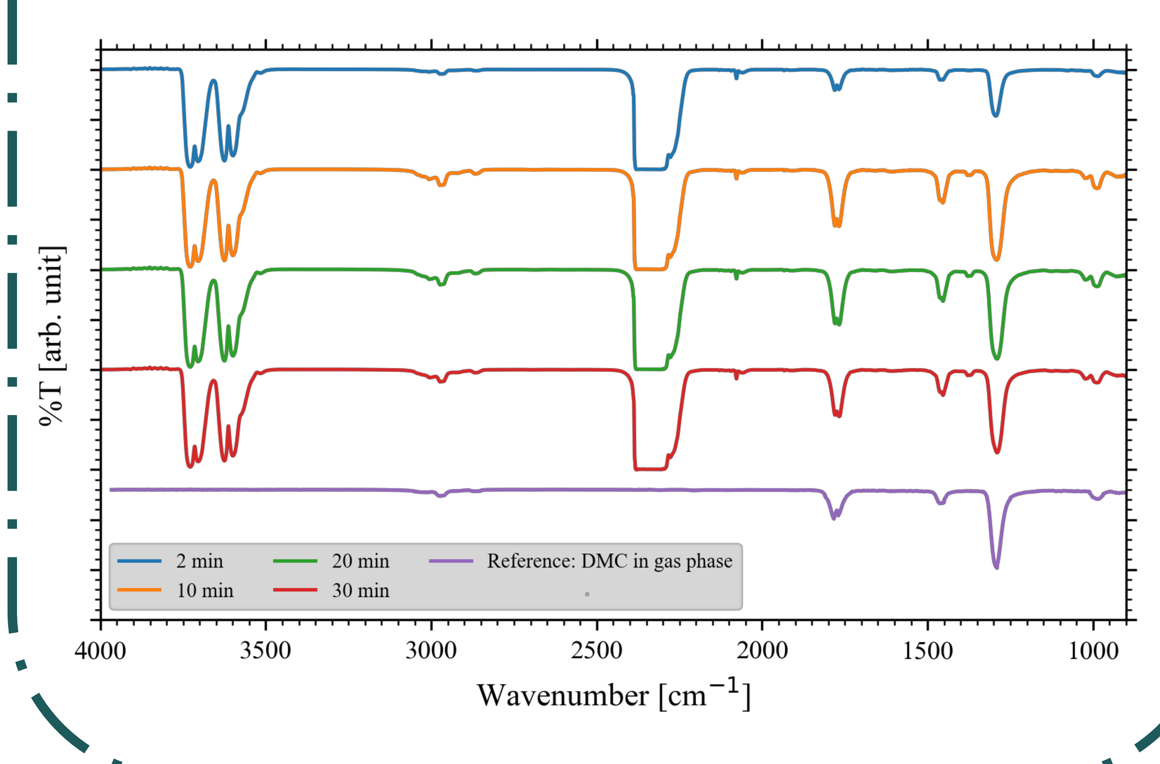


GC-MS chromatograph of the recovered electrolyte after $scCO_2$ extraction with a density of 810 kg/m^3.

Composition of recovered electrolyte at various CO_2 densities.

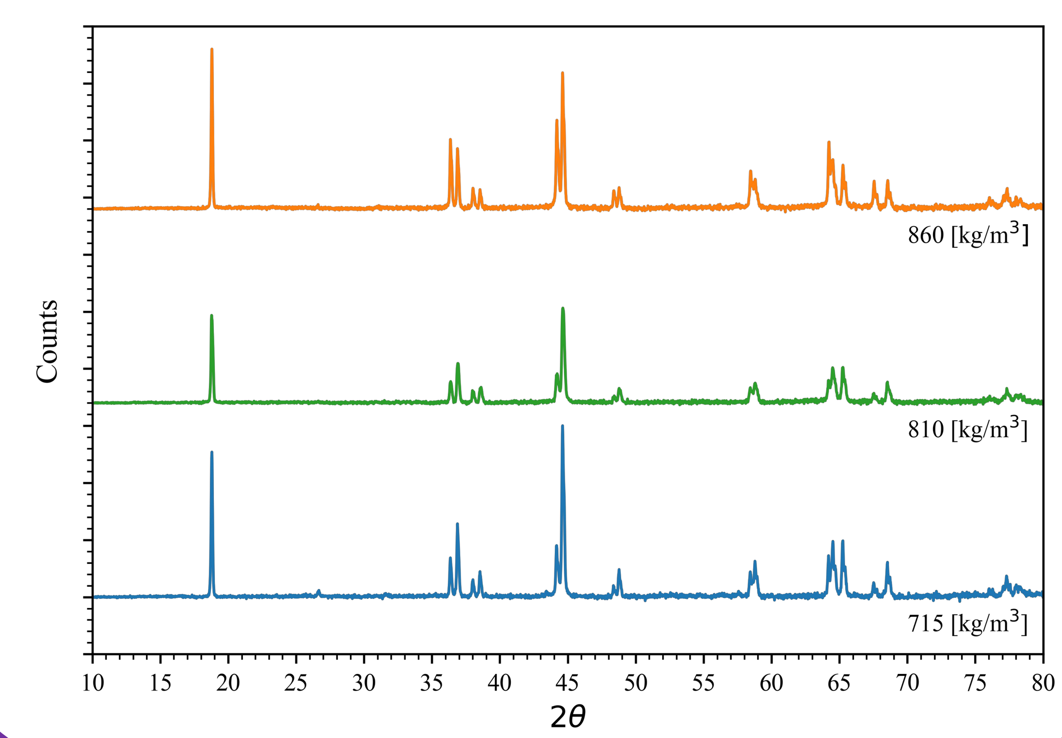


FT-IR spectra of the exhaust gas measured at various times after the start of the $scCO_2$ process.



- ➢**Increase of CO_2 density leads to a higher electrolyte recovery.**
- ➢**DMC, EMC and EC were recovered in the $scCO_2$ process.**
- ➢**Selective electrolyte solvent recovery depended on the CO_2 density. EC poorly recovered given densities.**
- ➢**$LiPF_6$ did not degrade in the process. Toxic-emission free process.**
- ➢**Unchanged cathode active material composition after $scCO_2$ process.**

XRD pattern of the cathode active material after the $scCO_2$ process at various densities.



Funded by: Energimyndigheten

Acknowledgements

This work was supported by the Swedish Energy Agency Battery Fund Program [Project No:50124-1]

In interest of: STENA RECYCLING, VOLVO