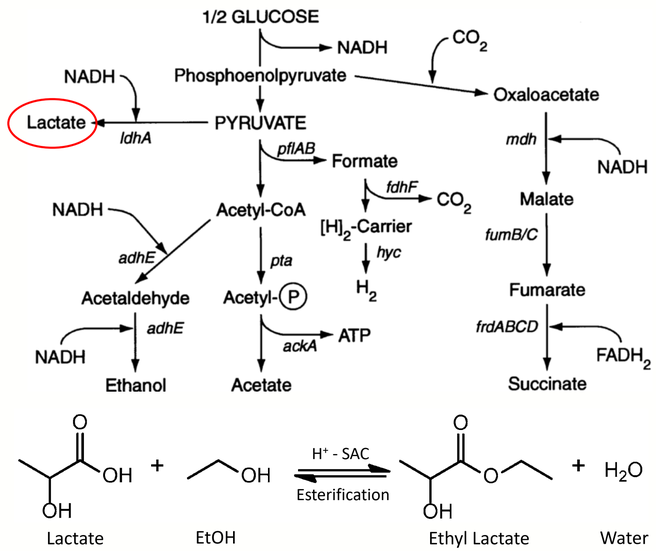
Gas Chromatographic Characterization of Ethyl Lactate from the Reactive Distillation of Post-Fermentation Broth

Pranav Bhavaraju, Collin Travis, Chemical Engineering
Mentor: Dr. David Nielsen, Chemical Engineering
School for Engineering of Matter, Transport, and Energy

D-lactate Production, Esterification via. Reactive Distillation & Gas Chromatography Characterization



- D-lactate is an organic acid used in bioplastics and biodegradable polymers.
- Most notably used to make polylactic acid (PLA).
- Also used in the cosmetics, pharmaceuticals industries as a pH adjuster and additive.
- Can be produced from *Escherichia coli* via D-lactate dehydrogenase under anaerobic conditions.
- A novel bioseparation method is explored for lactate removal from post-fermentation broths.
- D-lactate is esterified to ethyl lactate via addition of EtOH with H^+-SAC ion exchange resin in a reactive distillation apparatus.
- n-hexane (n-hex) and ethyl acetate (EtAc) tested to extract ethyl lactate from water.
- Gas chromatography with flame ionization detector (GC-FID) required for ethyl lactate characterization.

Metabolic Pathway Adapted from Jiang et al.

GC-FID Calibration

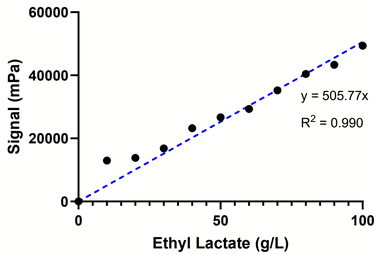


Fig 1. 0-100 g/L Ethyl Lactate in EtAc

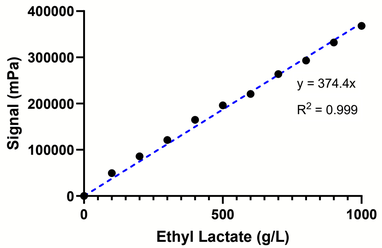


Fig 2. 0-1000 g/L Ethyl Lactate in EtAc

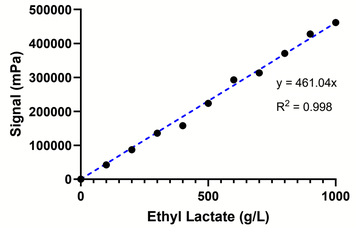


Fig 3. 0-1000 g/L Ethyl Lactate in n-hex

- Calibration curves are needed to correlate GC-FID signal strength with ethyl lactate concentration.
- n-hex presented an immediate challenge of quick evaporation.
- GC-FID Peaks were matched to ensure no overlap. Ethyl lactate residence time avg. 3.66 min.

Solvent Based Extraction of Ethyl Lactate

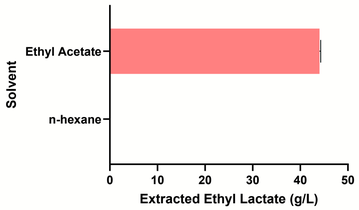


Fig 4. Ethyl Lactate Extraction With EtAc, n-hex

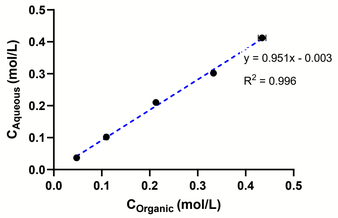


Fig 5. Organic-Aqueous Equilibrium of Ethyl Lactate Extracted in EtAc

- 750 µL of 100 g/L ethyl lactate extracted with 750 µL of EtAc, n-hex.
- EtAc shows superior extraction capabilities.
- Ethyl lactate was varied at different concentration and then extracted with EtAc.
- Ethyl lactate in EtAc Organic-Aqueous equilibrium constant shows ~50% extraction capability.

Batch Experiment Results

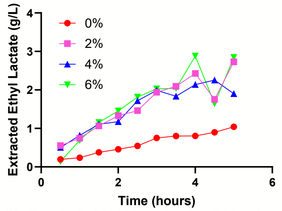


Fig 5a. 4x EtOH With Varying IX Resin wt.%

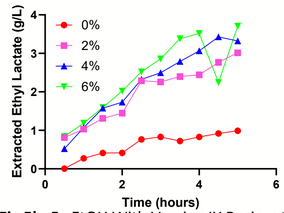


Fig 5b. 5x EtOH With Varying IX Resin wt.%

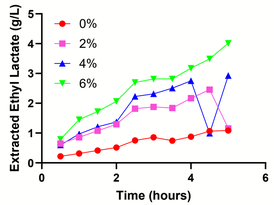


Fig 5c. 6x EtOH with varying IX Resin wt.%

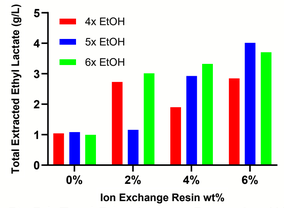


Fig 5d. Total Ethyl Lactate Extraction With Varying EtOH Concentrations, and IX wt.%

Experimental Conditions

- Experiment run at 70 °C .
- 15 mL samples in sealed glass vials.
- DuPont AmberLite IRN77 H^+-SAC resin.
- 77 g/L lactic acid in solution.
- 0,2,4,6 wt.% IX resin tested.
- 4,5,6x Excess EtOH used for esterification reaction.
- 5 hours total run time, with 30 min sample intervals.
- Extraction with EtAc; Analyzed with GC.

- Maximum Ethyl Lactate concentration extracted was 4 g/L with 6 wt.% resin and 6x excess EtOH.
- Ethyl Lactate produced increases with increasing IX resin wt.% and increasing EtOH excess.
- Esterification reversible and can contribute to low yields in batch conditions.
- Reactive distillation apparatus required for maximum ethyl lactate production.

Acknowledgments

- Dr. David Nielsen for his continuous mentorship.
- Collin Travis for his work on ethyl lactate and diethyl malate reactive distillation.
- Isaiah Woodson for his work on lactic acid fermentation via *E. coli*.
- Nandini Kannoju and fellow peers in the Nielsen, Wang, and Varman labs.
- Fulton Undergraduate Research Initiative (FURI) for supporting undergrad research at ASU.
- Amy Trowbridge and Dr. Haolin Zhu for their incredible support.
- Fellow peers in the Grand Challenges Scholars Program.

Grand Challenges Scholars Program

ASU Ira A. Fulton Schools of Engineering
Arizona State University