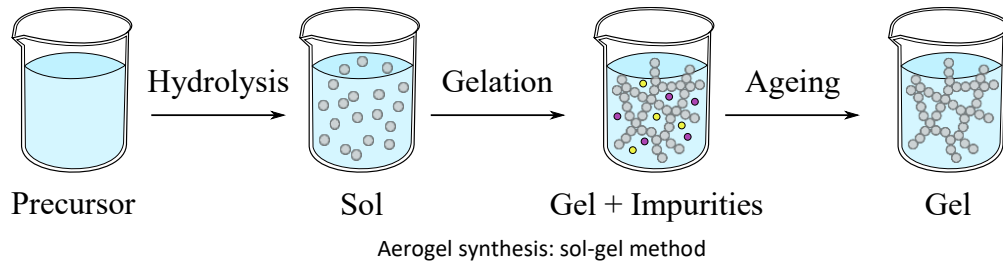
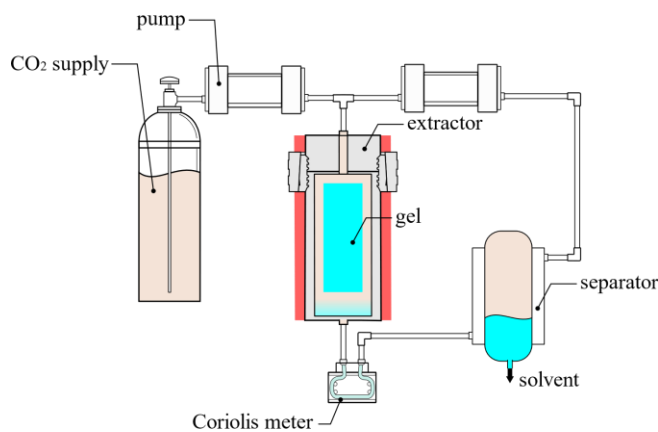
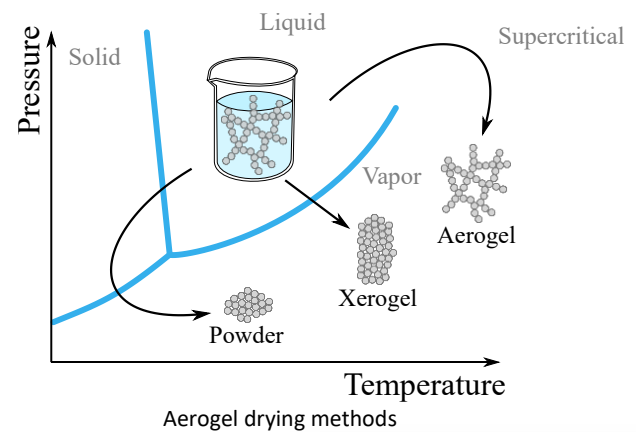


Silica Aerogel

Aerogel is a versatile class of low-density, high surface area, adjustable surface chemistry, nanoporous material attractive for a wide range of applications, including thermal insulation, light-weighting of transportation, catalyst and use as an adsorption medium in environmental remediation.



- **Gelation**: React silicon alkoxide, say, with water to form solid silica skeleton with alcohol-filled pores (alcogel).
- **Aging**: Increase alcogel strength by soaking at elevated temperature – continued hydrolysis and condensation, Ostwald ripening and syneresis.
- **Drying**: Replace pore liquid with gas without damaging the silica structure.



Simplified schematic of a representative SCCO₂ extraction rig



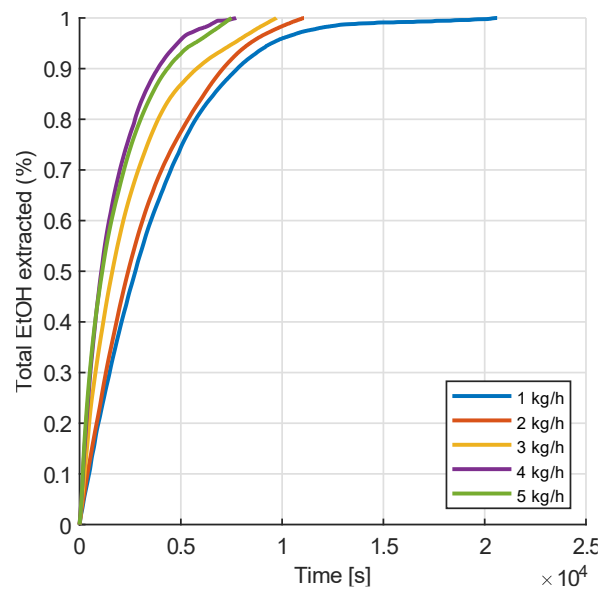
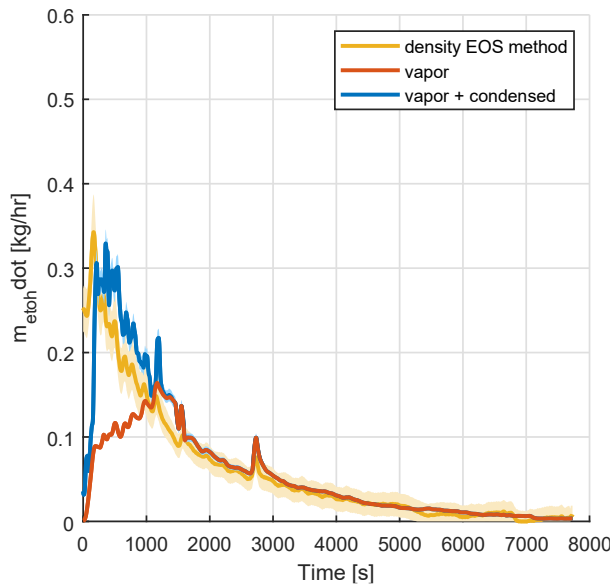
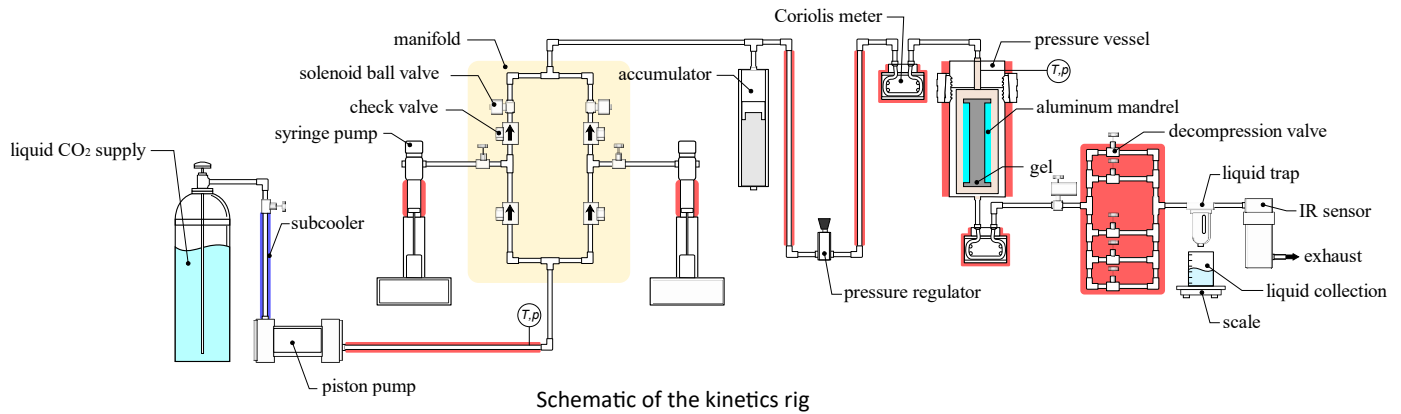
Gel mold and silica aerogels of various thicknesses

During aerogel manufacture, first, an alcogel, i.e., porous nanoskeleton filled with alcohol, is synthesized via a sol-gel process. The alcohol must be removed (i.e., the aerogel must be “dried”) without damaging its delicate porous nanoskeleton via capillary forces to convert it into an aerogel. Slow, diffusion-limited mass transfer during drying is a key barrier to aerogel’s widespread adoption. Development of cost- and energy-efficient aerogel manufacture requires an in-depth understanding of the underlying drying mechanisms.

Supercritical CO2 Drying

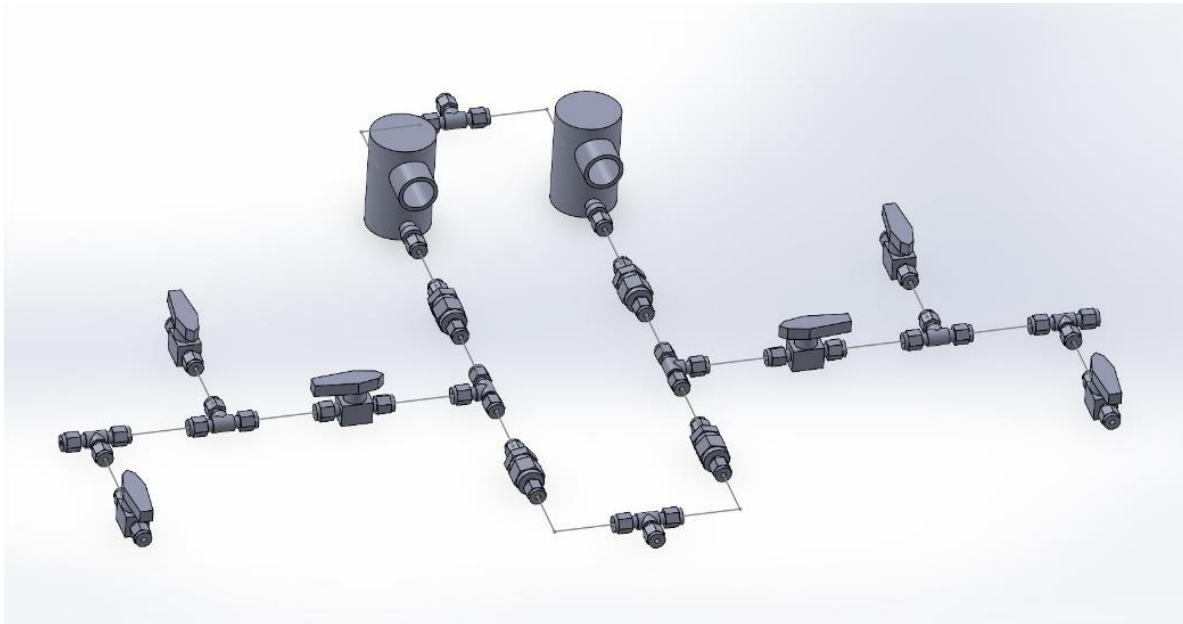
Designed and constructed an experimental apparatus (kinetics rig) to measure continuous rates of alcohol extraction from SCCO2 drying of aerogels using 2 independent methods:

- Total mass flow rate of ($\text{CO}_2 + \text{EtOH}$) effluent (\dot{m}) measured by downstream Coriolis meter and, after decompression, liquid-component of two-phase mixture is continuously weighed on a scale and mass fraction of EtOH in vapor measured by infrared hydrocarbon sensor.
- Measure total effluent mass flow rate and mixture density (ρ) with downstream Coriolis meter and based on known equation-of-state $\rho(m_{\text{EtOH}}, T, p)$ and measured T and p , compute m_{EtOH} in effluent. Then, $\dot{m}_{\text{EtOH}} = m_{\text{EtOH}} \dot{m}$.



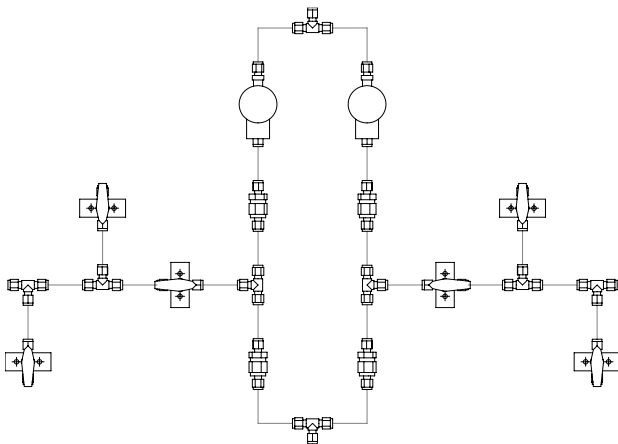
Dual-pump Continuous Flow System

- Built an H-bridge valves manifold to enable continuous flow using 2 Teledyne ISCO syringe pumps
- Designed the manifold consisting of one-way valves, solenoid valves, ball valves, and fittings in SolidWorks



Manifold design in SolidWorks

- Created a layout for the manifold assembly

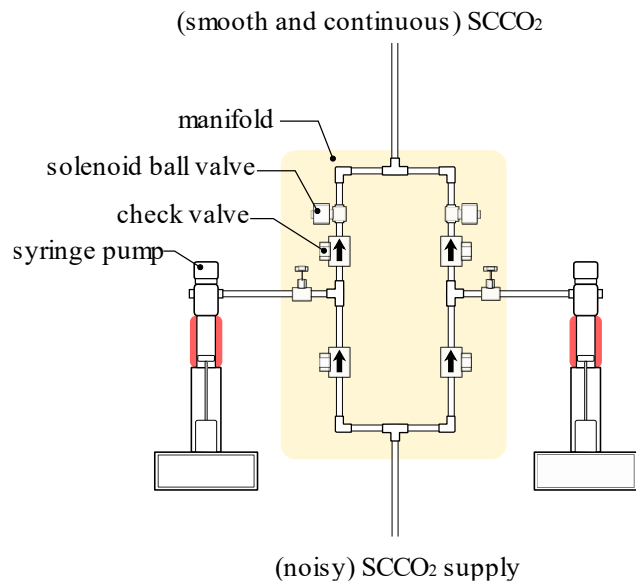


Component layout as designed and pre-assembled

- Cut an aluminum base using a jump shear and machined fixtures for the valves and regulators
- Assembled and connected the manifold to the syringe pumps, finalizing the flow path



Assembled manifold



Schematic of the dual-pump system

- Programed a state machine in LabVIEW to control the pumps and manifold such that when a pump is running at a set pressure/flow rate, the other pump is set to refill and waits on stand-by until the first pump is empty. Then, the second pump switches over to provide a smooth and continuous flow.

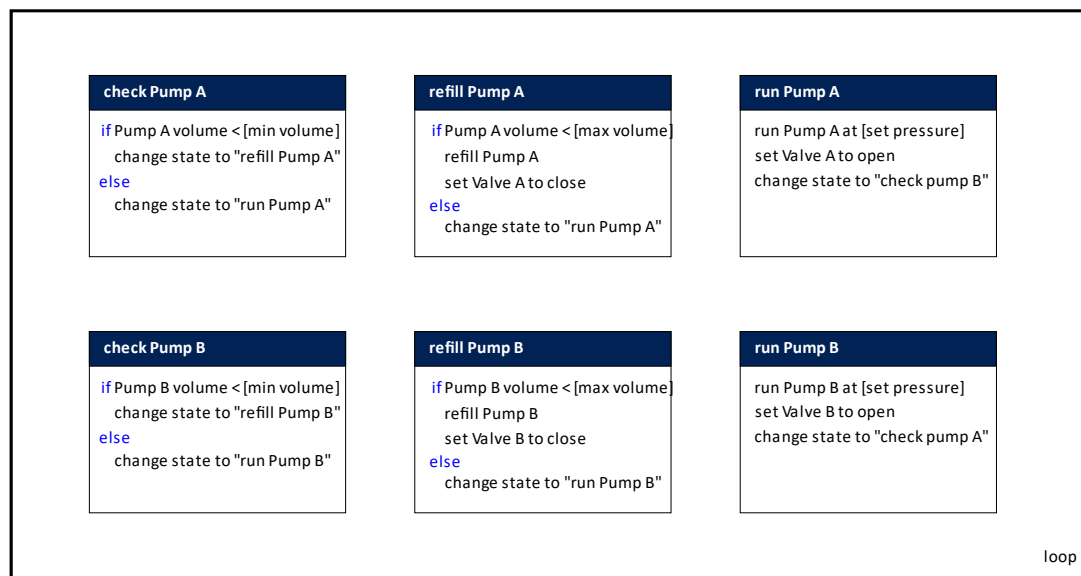
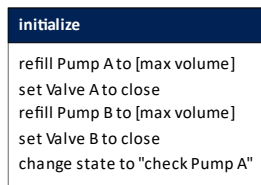
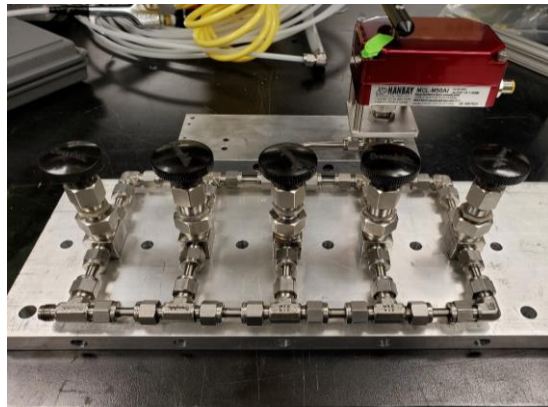
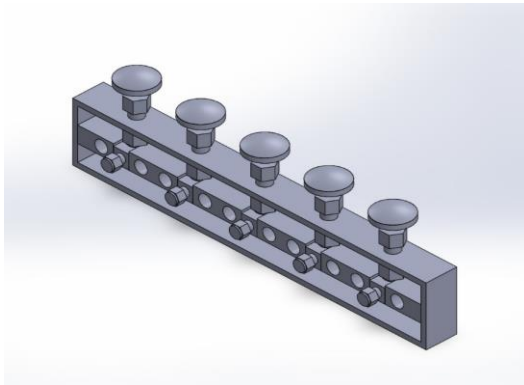


Diagram of the state machine

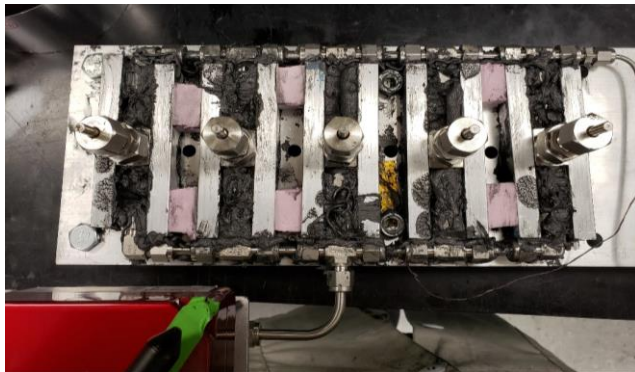
Decompression Valves

- Designed and constructed a heated and automated valve system to prevent decompression valves from freezing due to the Joule-Thomson effect when CO₂-ethanol solutions expand from 1800 psi to ambient pressure

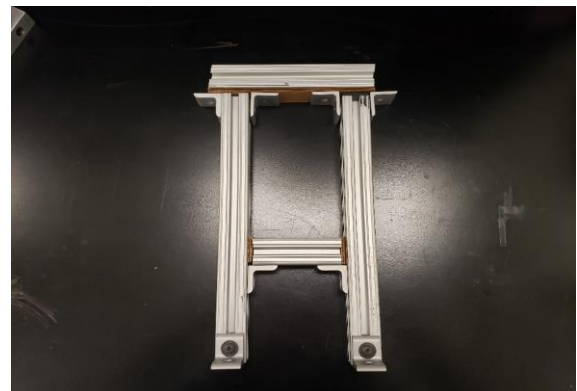


Initial design of the decompression valves in SolidWorks and actual photo

- Completed thermal testing of the initial design to adjust the appropriate thermal mass of the heater block and contact area to increase heat transfer
- Filled the gaps between the valves and heater block with thermal grease and assembled a mounting leg



Revised decompression valve assembly



Mounting leg

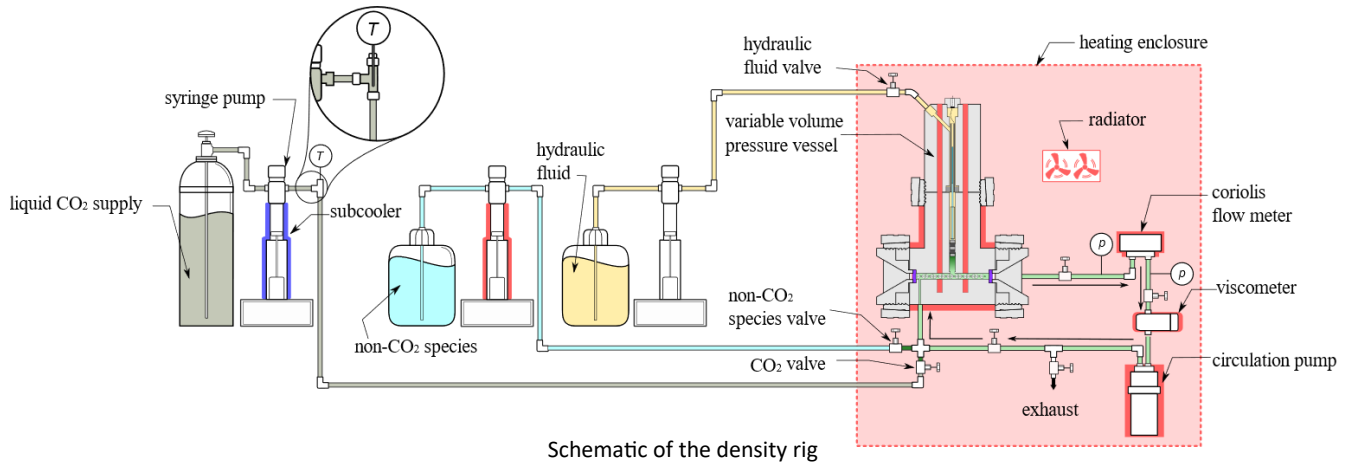
- Insert 5 cartridge heaters (1000 W in total) into the heater block and attached a thermocouple as the sensor of a closed loop temperature control
- Attached an automated (red) needle valve to regulate the CO₂ mass flow rate (measured by a Coriolis meter)
- Eliminated freezing issue at highest operating flow rate (5 kg/h)



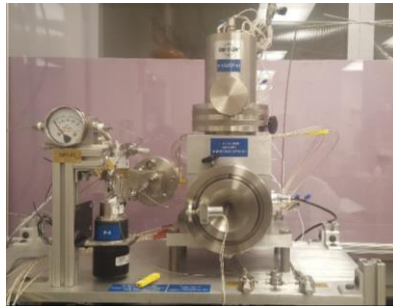
Assembled decompression valve system

Density Rig

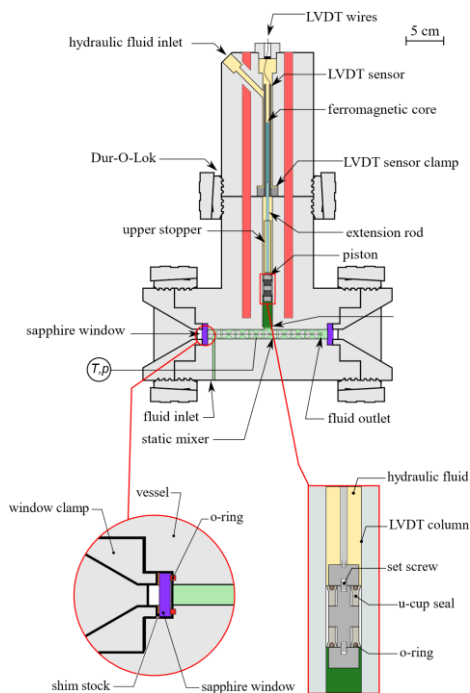
- Designed and constructed an apparatus (density rig) to measure thermophysical properties of binary (and higher order) fluid mixtures at up to 80 °C and 2500 psi.



Schematic of the density rig



Progression of the density rig: fixture + heater blocks (left), assembled vessel with circulation loop (middle), density rig inside heating enclosure (right)

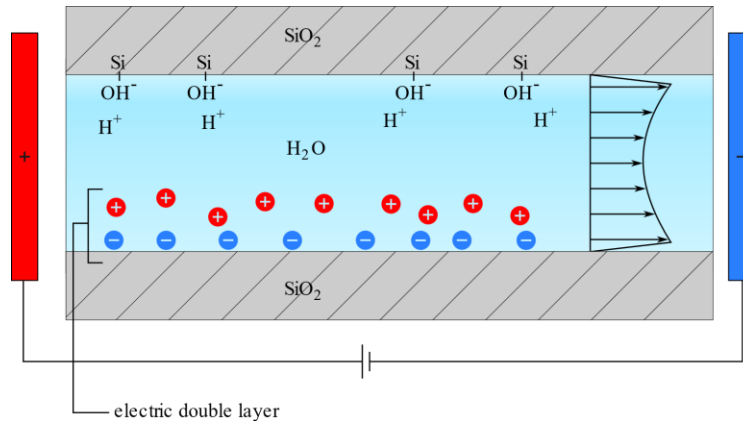


Schematic of the pressure vessel and seals

- The test section is pressure vessel with a piston traversing vertically to vary its volume.
- The piston has Teflon U-cup seals on its upper and lower ends to isolate the CO₂-EtOH mixture from the hydraulic fluid.
- Optical access is enabled by sapphire windows (1 in diameter) at each end of the vessel. O-ring seals between the windows and the fluid side of the vessel are made from buna-N to minimize swelling and blistering observed with other seals upon decompression.

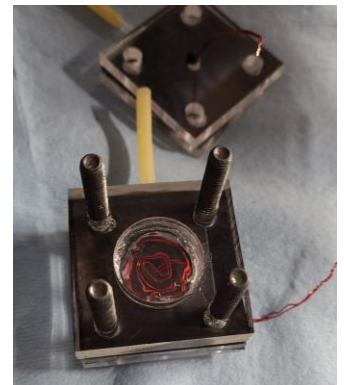
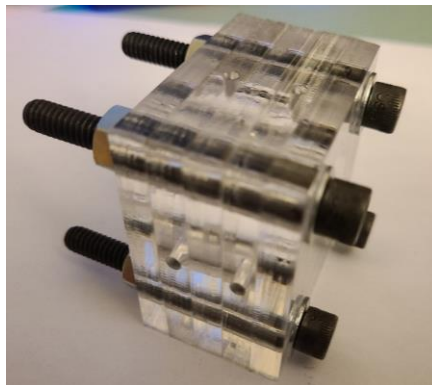
Electroosmosis-enhanced Ageing of Aerogels

- An electric double layer naturally forms along the inner periphery of a polar-liquid-containing tube due to deprotonation of the surface silanol groups. The OH^- ions in the water are pinned to the wall of the tube via interaction with the silica and the corresponding H^+ ions are mobile.
- The application of an electromotive force drags the positive ions in the direction shown. The external potential difference applied across the tube induces a Coulomb force on the mobile H^+ ions. They drag along the neutral water molecules in the middle section of the channel, resulting in a flow with the highest velocity near the wall and, thus, a plug-like velocity profile as opposed to that of a pressure-driven flow, which is parabolic.



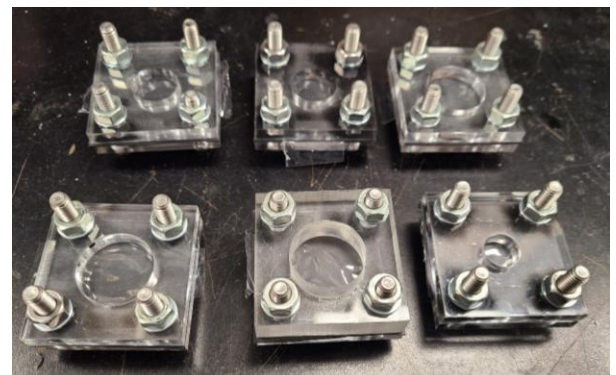
Representative diagram of electroosmotic flow

- An apparatus was designed and constructed to generate EO flow across an alcogel

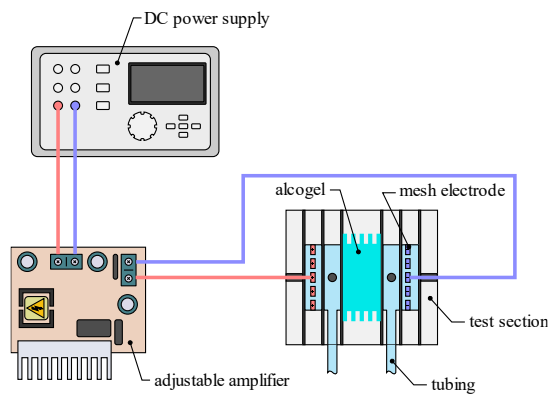


Progression of the apparatus: initial design with separated reservoirs clamped together (left), updated design with laser cut acrylic plates (middle), disassembled apparatus showing the electrode (right)

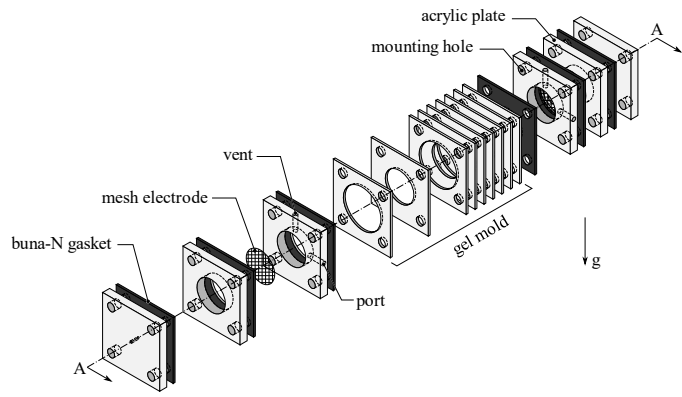
- The gel mold is comprised of nine 1/16 in acrylic plates with 1.3 in and 1 in center holes alternatively stacked and cemented together. When an alcogel is formed using this mold, a lip is created at each layer forming a hermetic seal between the gel and the mold.



Alcogel molds

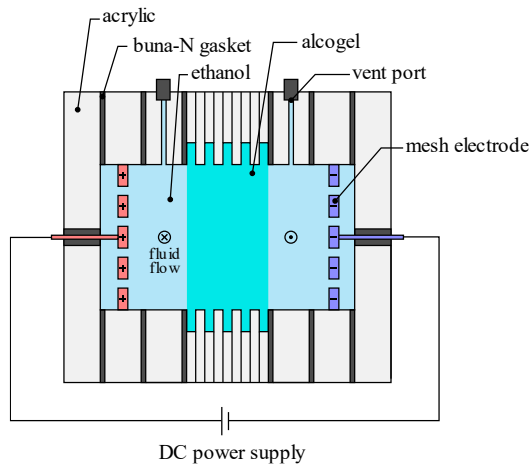


Schematic of the apparatus

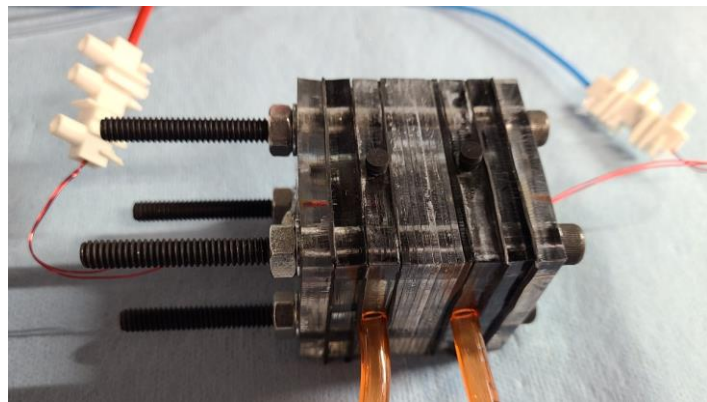


Exploded view of the test section

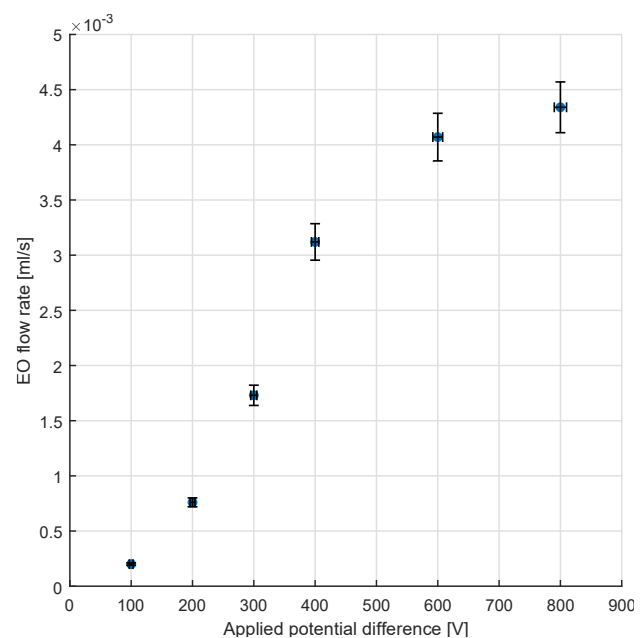
- Ethanol was charged inside the test section and residual air vented through the vent ports.
- Electroosmotic flow was generated at varying potential difference up to 800 V.



Schematic and photo of the test section



- An apparatus was constructed to successfully generate an electroosmotic flow through an alcogel. Experimental results indicate that new founded advection can significantly reduce the time-consuming diffusion-limited solvent exchange process to, potentially, minutes.



Measured EO flow rate vs. applied voltage