ENVIRONMENT SAFETY HEALTH

Electrospun PVA/PEI Nanofibers for Rare Earth Element Extraction

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CHRES Technical Forum at NETL
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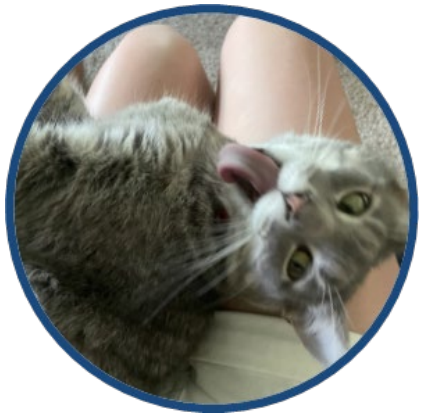
Intro (Stacie)

MICKEY LELAND ENERGY FELLOWSHIP

Cornell Engineering



Intro (Jalyn-Rose)

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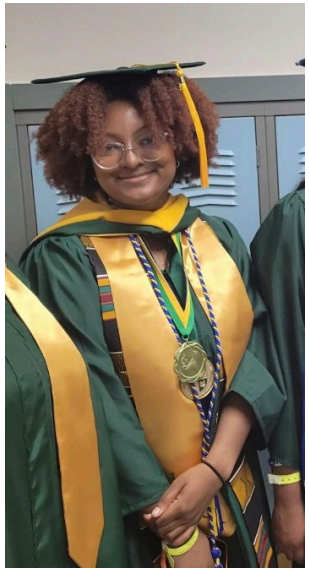

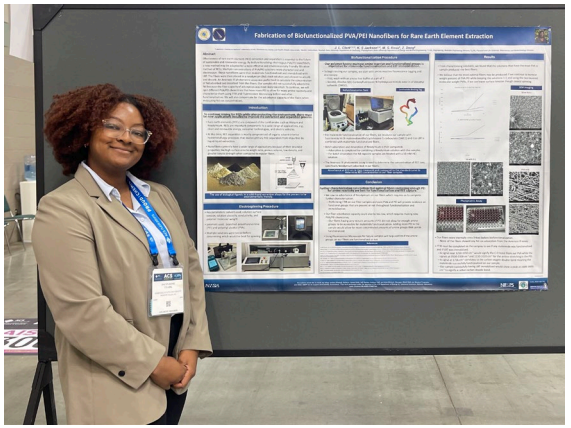

Fabricating a Solid-Liquid, Environmentally Friendly Extraction Process for Rare Earth Elements

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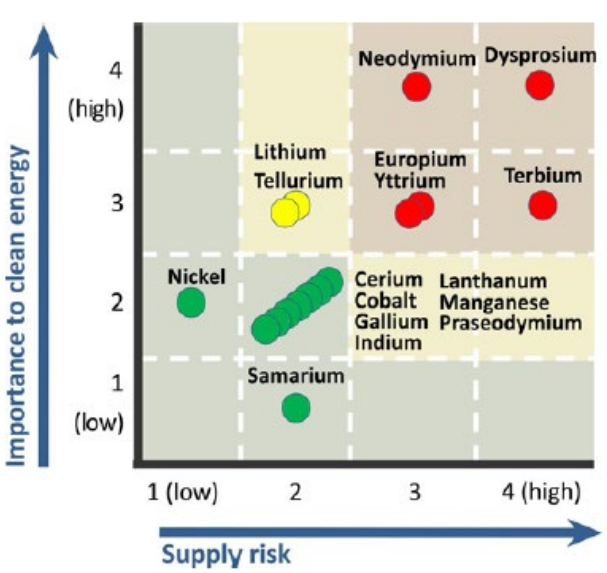
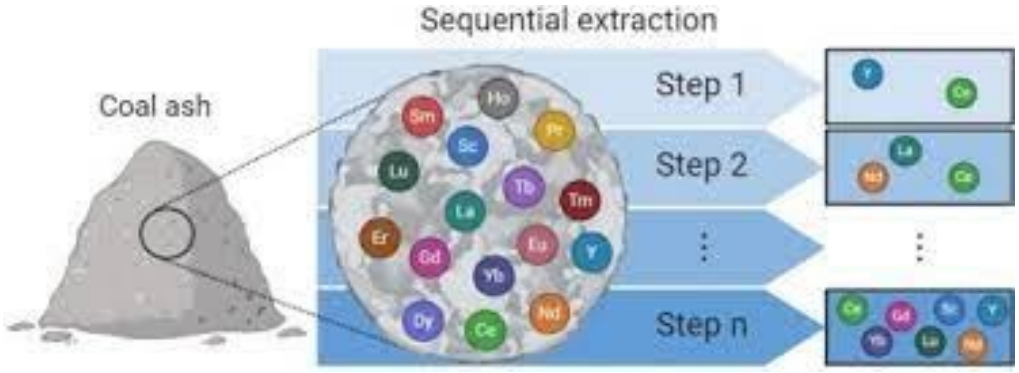
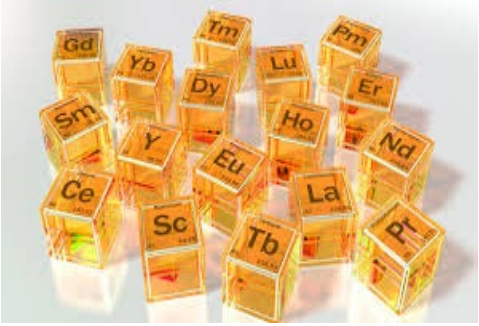


Critical
Near-Critical
Not Critical

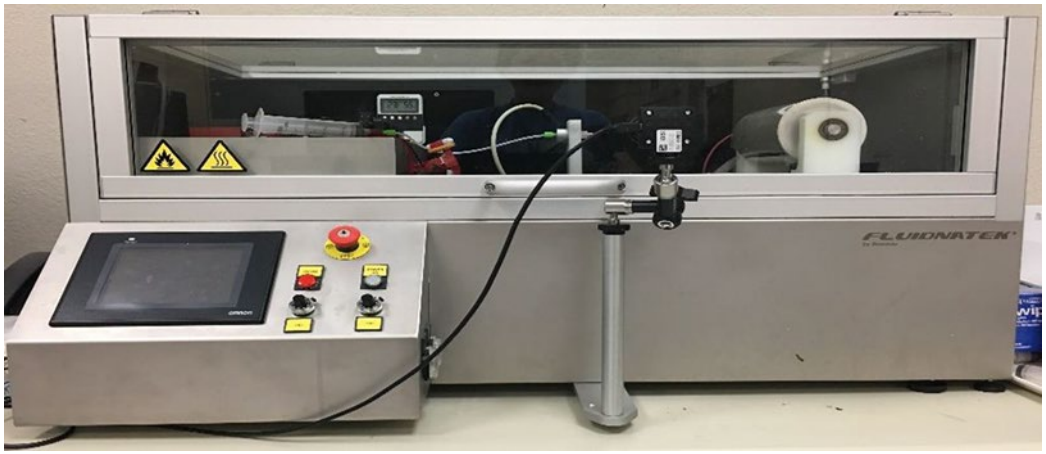


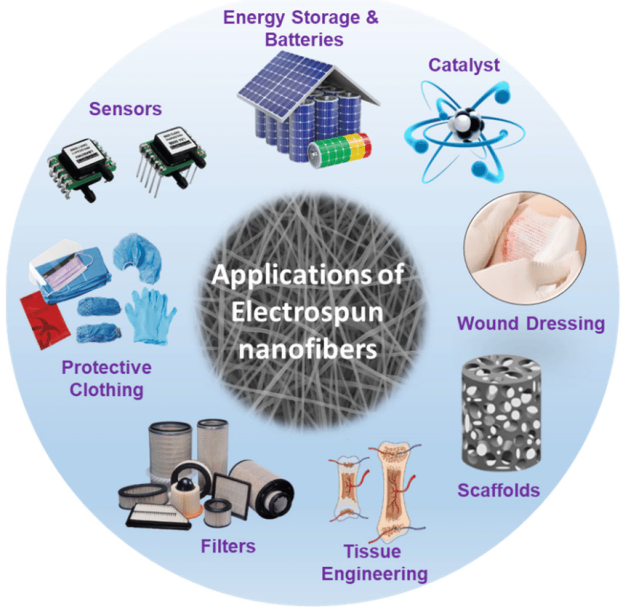


Nanofibers

can retain physical, chemical, and biological properties

- Nanofibers currently have a wide range of applications because of their high surface area to weight ratio, porous volume, low density, and greater tensile strength when compared to regular fibers

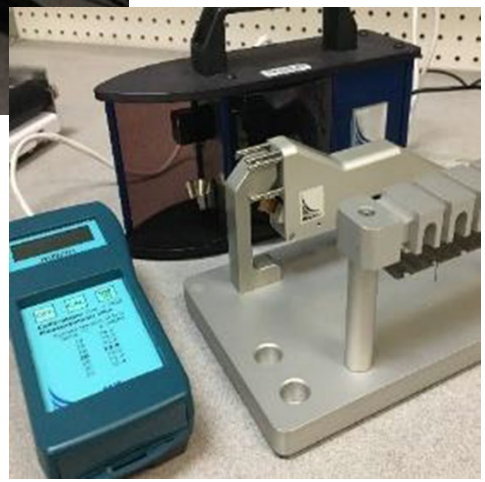




We believe that incorporating biological-ligands with our electrospun nanofibers will allow for a more effective filtration method of REEs while also allowing the method to be environmentally friendly

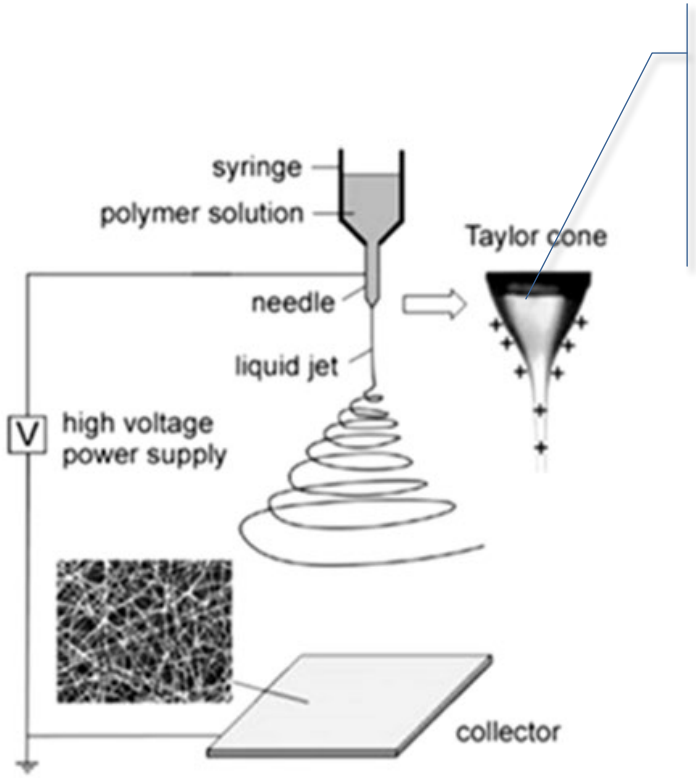
PVA/PEI Solution Characterization

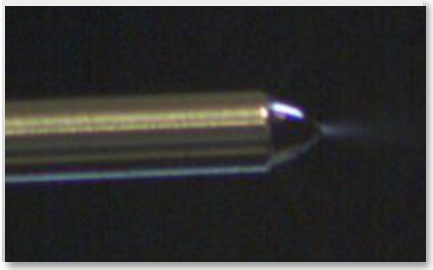
Characterization Instruments

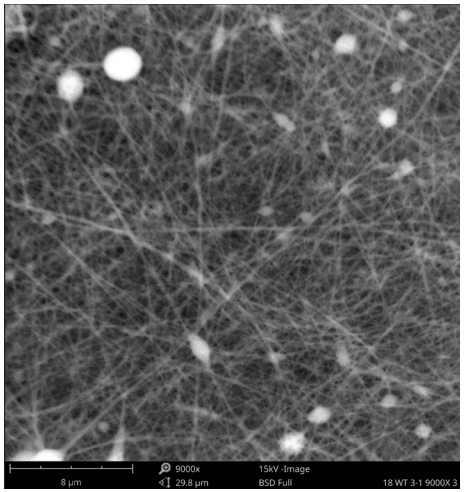


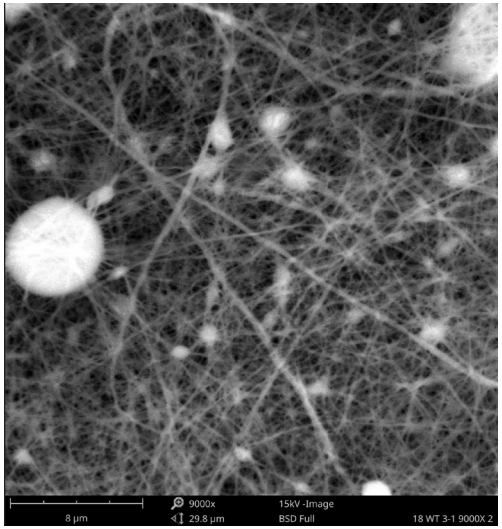
- Polymers we are using are branched polyethyleneimine (PEI) and polyvinyl alcohol (PVA) mixed in water
- Characterization included viscosity, surface tension, and conductivity
  - Range of viscosity desired in our solutions is 100-500 cP while our surface tension should be 35-50 mN/m
- After conclusive testing, we found the solutions suitable for spinning are 16, 18, and 20 weight percent for the ratios 3:1, 2:1, and 1:1

Electrospinning Procedure





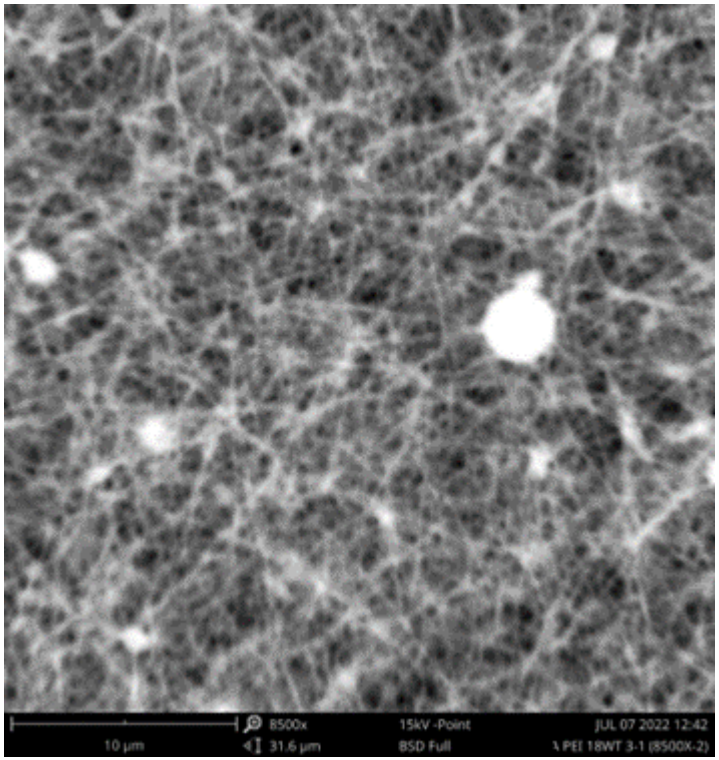


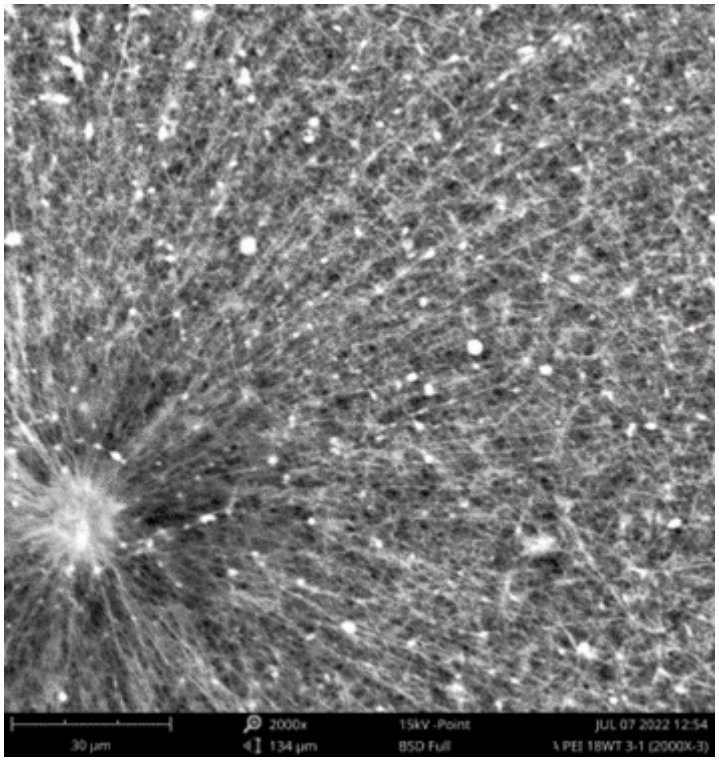


- Once the fibers are spun, they are thermally crosslinked
  - Cross-linking occurs in the chain segments of polymer to form a stable three-dimensional network structure

Under the SEM, we immediately observed that all our samples had many beads

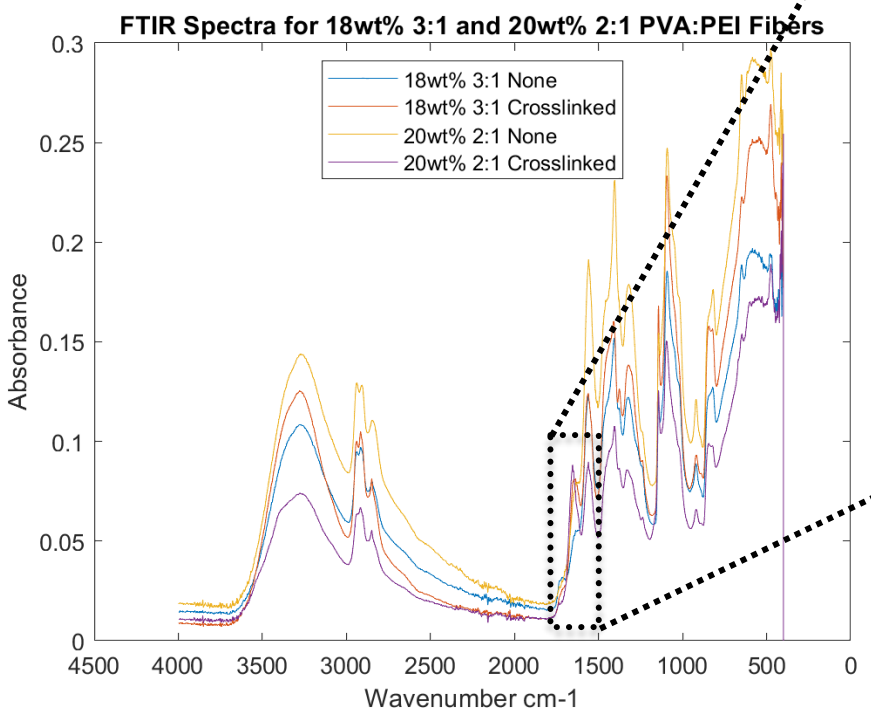
- Our 3:1 samples had the most visible fibers ranging from 180-500 nm
  - They have multiple beads in various sizes on them, but the fibers are clear
  - Beaded fibers are usually not desirable, but they are still fibers and have binding capabilities with the peptides

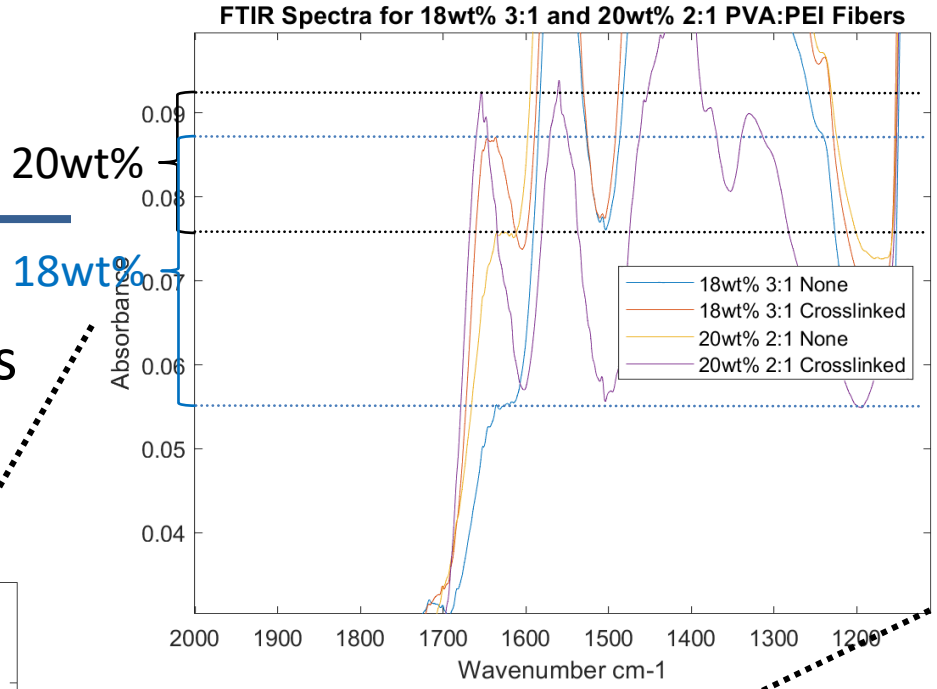




Crosslinking the Fibers

- At least 110°C, at least 10 hrs

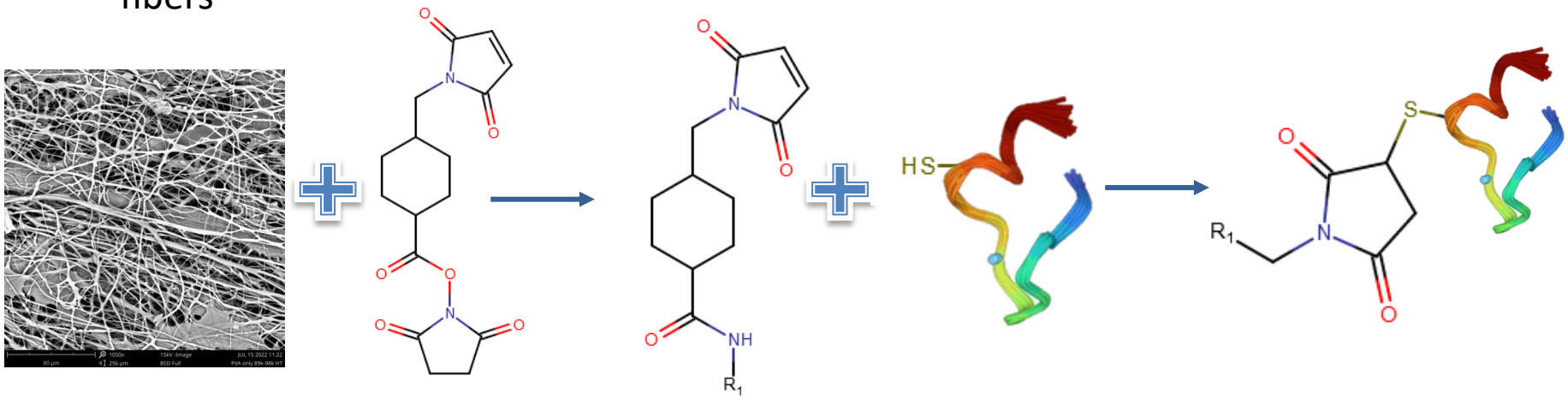




Crosslinking ensures the fibers won't dissolve in water or other solvents

Lanthanide Binding Tag (LBT) Immobilization

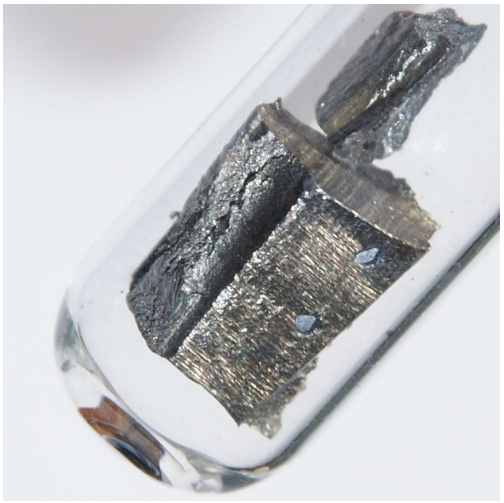
- LBT immobilization carried out using a thiol-maleimide conjugation reaction
  - Short peptides created for REE selectivity and affinity; have 1 reusable binding site
  - For maleimide functionalization of our fibers, we wash our samples with PBS at a pH of 7. Succinimidyl 4-(N-maleimidomethyl)cyclohexane-1-carboxylate (SMCC) is then dissolved in DMSO and incubated with the fibers
  - Sample is then washed three times with a coupling buffer to remove DMSO at pH 8
  - Cys-LBT is incubated with TCEP beads then combined with maleimide functionalized fibers

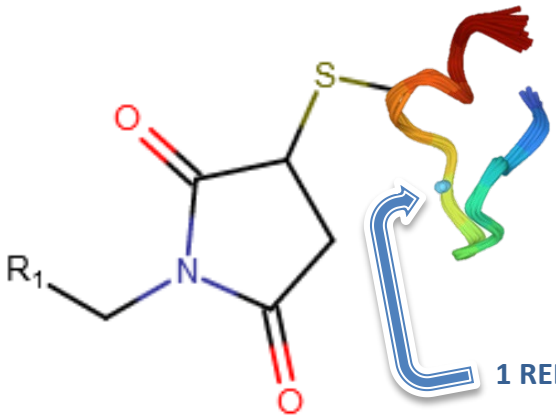


Amine Functionalized Fibers (R1) SMCC Maleimide Functionalized Fibers Cys-LBT LBT Immobilized Fibers

Adsorption and Desorption of Neodymium

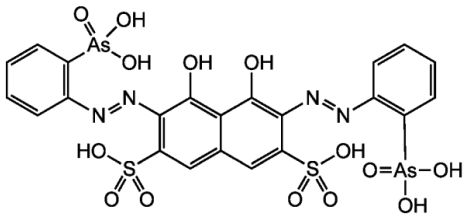
- Batch **adsorption** is completed by preparing a Nd stock solution with a final concentration of 0.1 mM with a pH of 6.
  - Batch adsorption is then initiated by combining the Nd solution with each LBT immobilized sample
- For batch **desorption,** the Nd exposed samples are washed with a MES or HEPES buffer to get rid of any unadsorbed metal ions
  - They are then treated with a 25 mM HCl solution
- From the treated solution, the adsorbed metal ions will desorb and the solution ion concentration can be measured

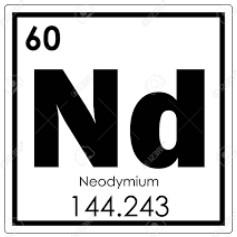


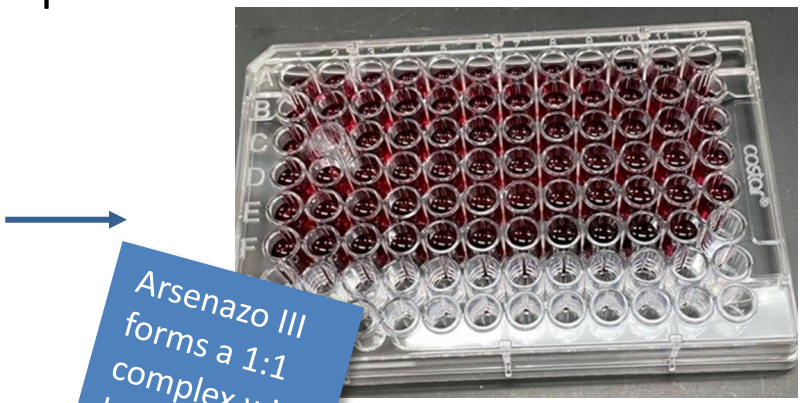


Spectrophotometric Determination

- Arsenazo III photometric assay is used to determine the concentration of metal ions in unknown samples
  - First, 120 µl of filtered 0.1 wt% Arsenazo in 6.25 wt% TCA is then combined to the solution
  - Second, 40 µl of 12.5 wt% trichloroacetic acid (TCA) is pipetted into well plates.
  - Third, 40 µl of solution is combined in the well.
- Assay is completed after adsorption and desorption
- Absorbance at 654 nm is then measured and compared to the standard curve to determine REE concentration in our fiber samples.







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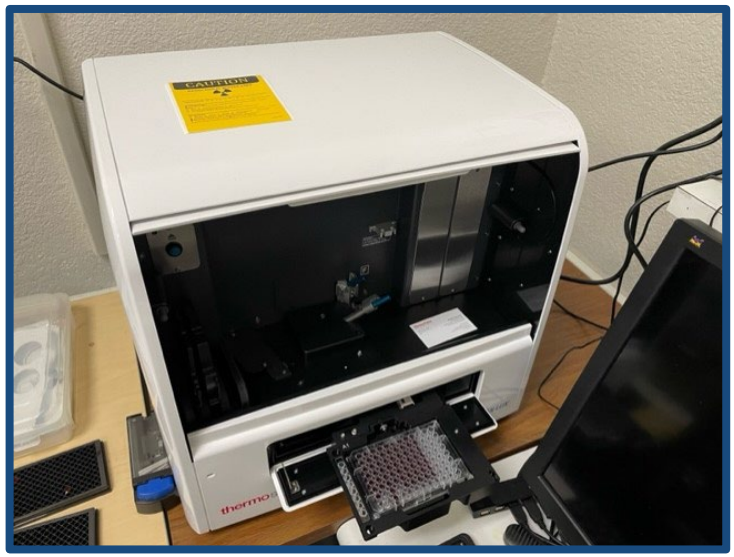
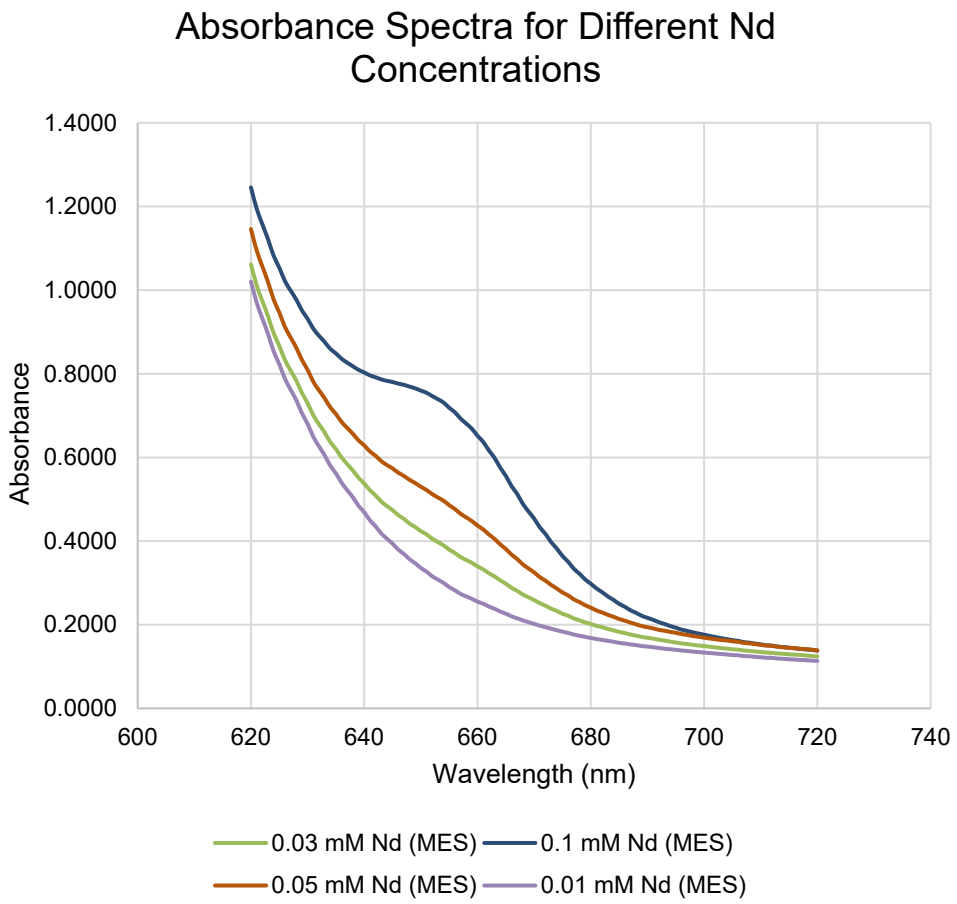
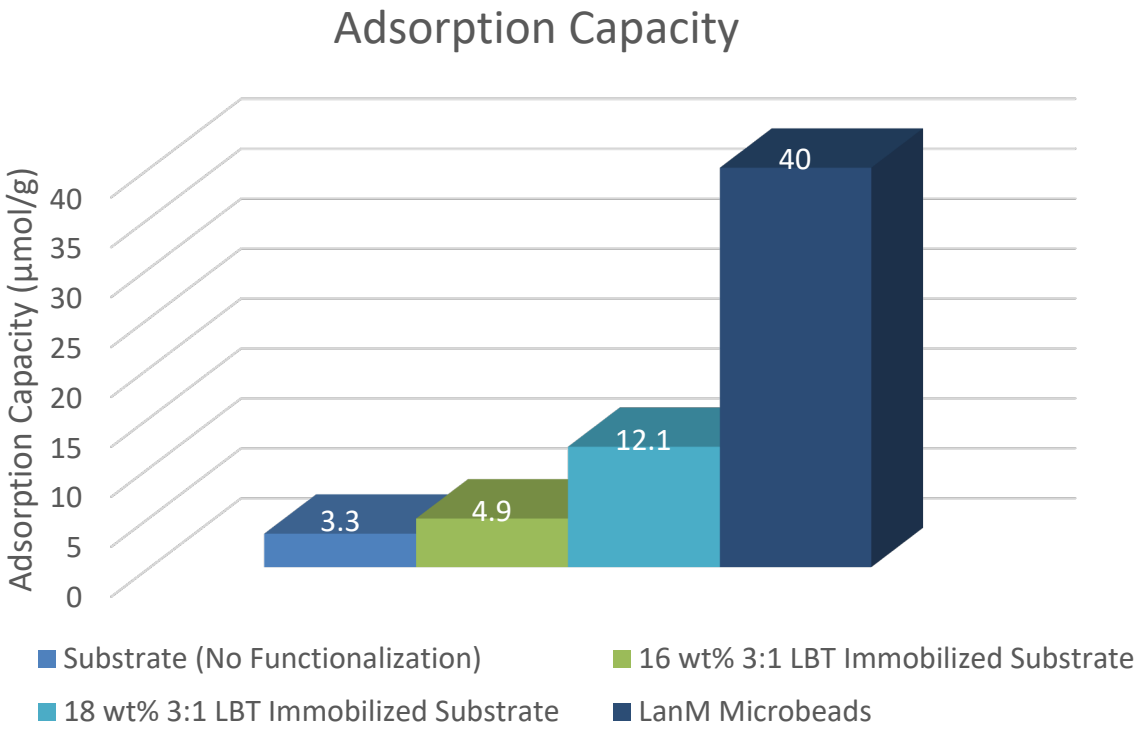




Initial Neodymium Extraction

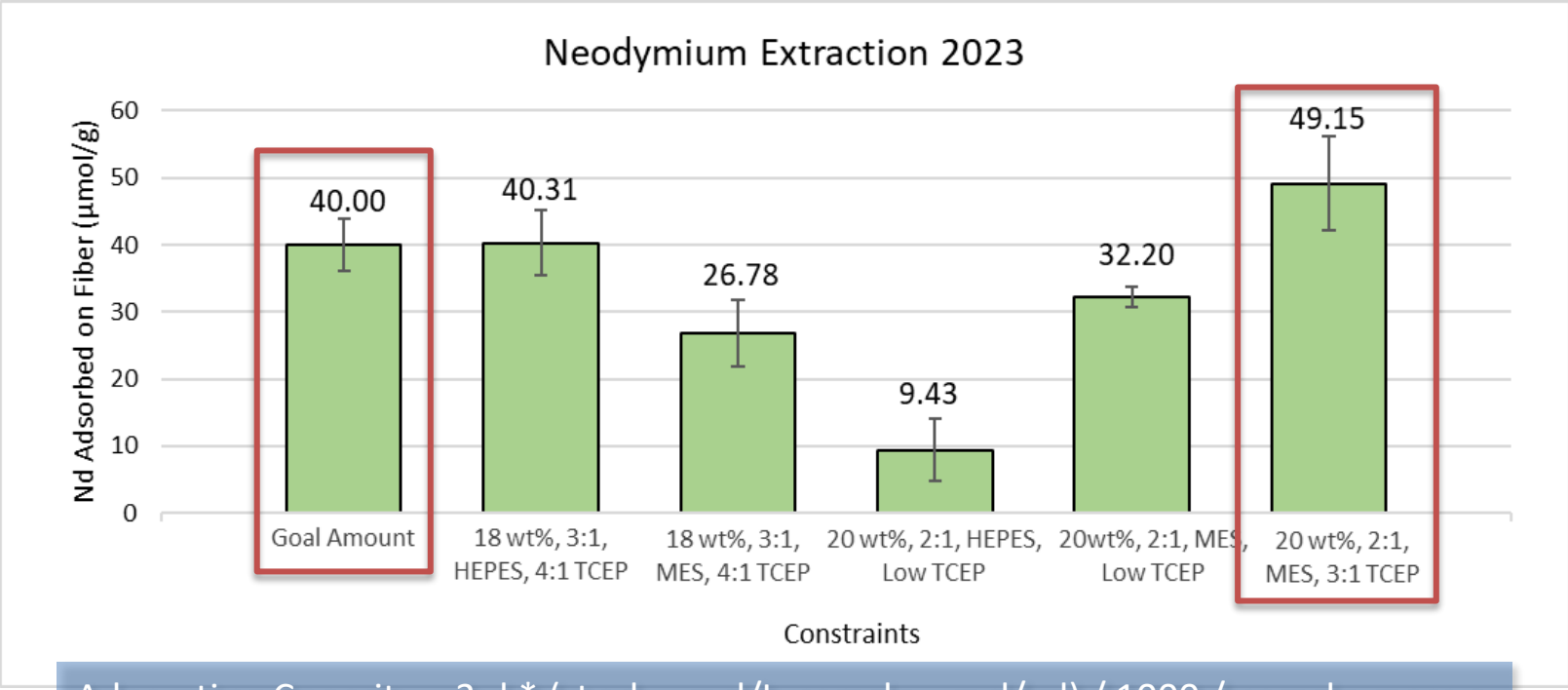
- We placed 3 ml of 200 µM Neodymium in each sample
  - Samples were allowed to adsorb, and the remaining solution was measured for Nd concentration



Adsorption Capacity = 3 mL* (stock – sample [mmol/L])/ sample mass [g]

Current Neodymium Extraction

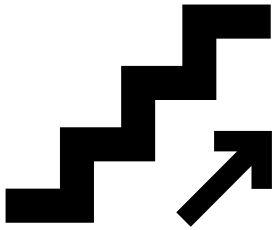
- We placed 3 ml of 100 µM Neodymium in each sample
  - Samples were allowed to adsorb, and the remaining solution was measured for Nd concentration

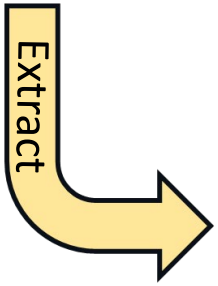


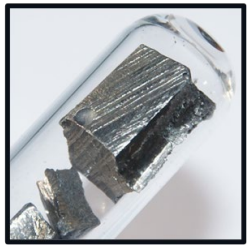
Adsorption Capacity = 3µl * (stock µmol/L-sample µmol/ml) / 1000 / sample mass g

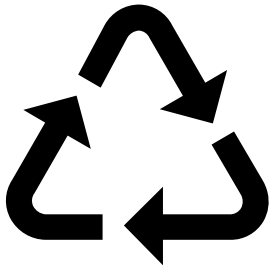
Next steps

- Next steps
  - Quantifying exact amounts of SMCC and LBT to use with fibers
  - Looking at different fiber compositions
  - Testing the reusability of the fibers
- Future work
  - Upscaling the process
  - Testing filter capabilities









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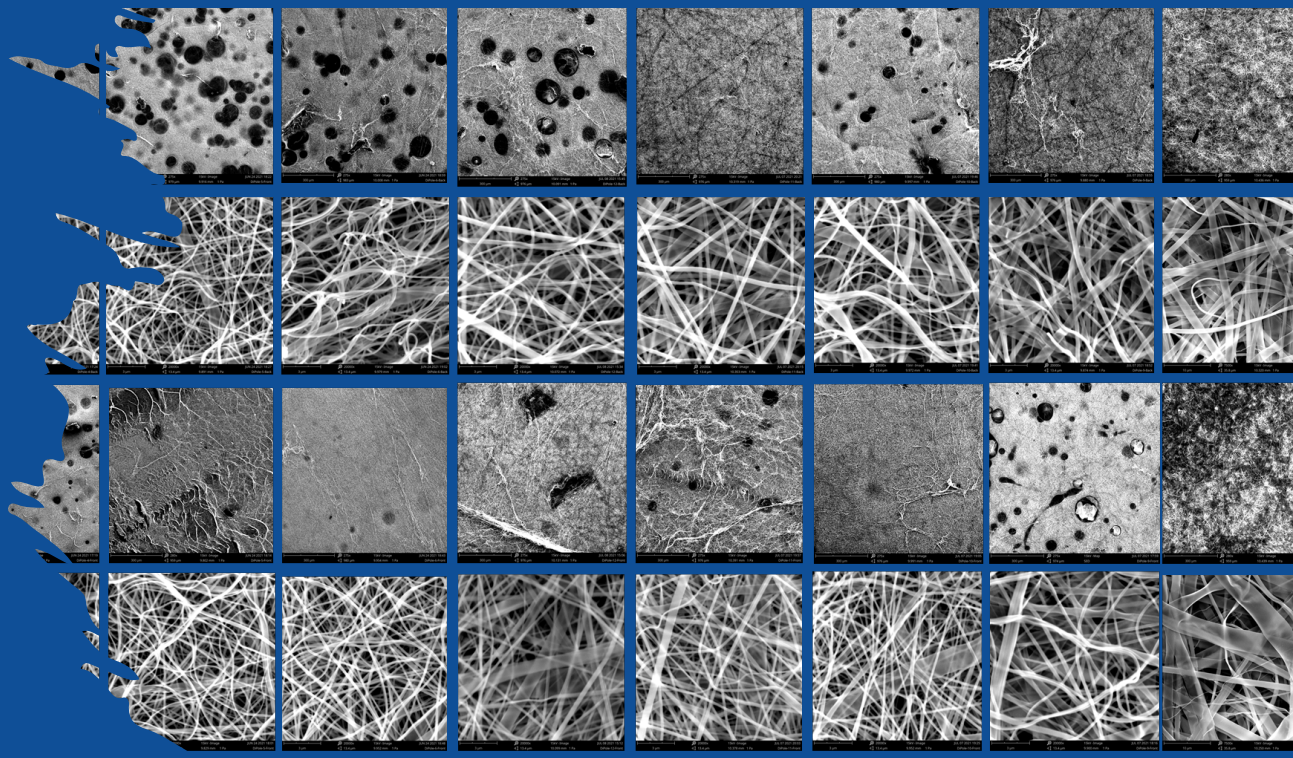

Auspices
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Adsorption of Nd vs. Mass