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Adsorption of Neodymium Experiment Design to Simulate Nuclear Fuel Reprocessing

Brian C. Froese

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Adsorption of Neodymium Experiment Design to Simulate Nuclear Fuel Reprocessing

A Capstone Project Submitted in Partial Fulfillment of the Requirements of the Renée Crown University Honors Program at Syracuse University

Brian C. Froese
Candidate for B.S. Degree and Renée Crown University Honors
May, 2011

Honors Capstone Project in Chemical Engineering

Capstone Project Advisor: [Signature]
Honors Reader: [Signature]
Honors Director: [Signature]

Date: April 25, 2011
Abstract:

The purpose of this project was to develop a laboratory experiment to be conducted by future Nuclear Engineering Track (NET) students that adequately demonstrates adsorption of the lanthanide neodymium, an extraction technique used in nuclear fuel reprocessing. Nuclear fuel reprocessing is the chemical separation of various elemental components in spent nuclear fuel. These components, once isolated, can be recycled or reused and ultimately minimize the volume of radioactive waste. There were several steps taken to accomplish this task. First, an independent study of Dr. Hyung Jun Park’s work on phosphorus based adsorbents was conducted. Research was also done on current nuclear fuel reprocessing techniques and how it related to Dr. Park’s work. The adsorbent that was the major outcome of his work was used as the basis of the experiment design. Materials needed for the experiment such as a Masterflex peristaltic pump, columns, micropipettes, test vials, graduated cylinders, chemicals and other supporting equipment were purchased. As well, the department’s inductively couple plasma mass spectrometer (ICP-MS) was repaired to use in this laboratory experiment.

Two separate procedures were created for future students and laboratory supervisors. The first was an experiment preparation procedure which specified in detail what solutions needed to be made prior to conducting the experiment, as well as how and when to reload the packed bed column. The second procedure made was the experimental procedure. This specifies step-by-step how students should conduct the batch and column portion of the experiment. It also describes how dilutions should be made prior to running them through the ICP-MS and what calculations are required from the results.

The final part of the project was to run the experiment according to the specified procedure using the purchased laboratory equipment to test its efficacy. All samples were run through the ICP-MS to determine concentration. The results of the experiment are in a supplementary laboratory report. This includes a theory section, experimental results, calculations, conclusions, and recommendations to the procedure. The procedures in this project include these recommendations.

Excellent results were achieved in the experiment. The breakthrough curve is evident and a high percentage of the neodymium was stripped. The experiment is currently located in Link 024 and ready for use by the first group of students who take the NET laboratory. The goal of this lab is to teach students about nuclear fuel reprocessing, as well as common extraction techniques which are used over a variety of majors and industries. This is one of three experiments that students will partake in. This experiment will adequately display the process of adsorption, a technique common in industry and may become more prevalent in the up-and-coming field of nuclear fuel reprocessing.
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Acknowledgements:

I would like to thank Dr. Hyung Jun Park for his research on phosphorus based adsorbents. My capstone project would not have been successful without his developments. I would also like to give a special thank you to my professor, research advisor, academic advisor, and honors advisor Dr. Lawrence Tavlarides. His high expectations and demand for perfection have forced me to grow into a more technical and qualified student in the field of chemical engineering. He has been there for advice when my future hit a crossroad and I would not be the same person today without his direction. Finally I would like to thank my honors reader Dr. James Pommersheim for taking the time to give input on my paper.

Advice to Future Honors Students:

From my experience, the honors program is challenging to complete as an engineer. If you are an engineering major looking to graduate with honors, know that you will have to jump through several hoops to avoid taking extra classes. Also, finding a suitable Capstone Project can be very difficult. With that in mind, know that it is very possible to complete the program. Hard work and the ability to plan ahead are essential traits. My sophomore year I took twenty credits, four of which came from a Chinese class which I used to complete a global awareness requirement. Unique challenges such as this class along with the rigors of chemical engineering proved as a challenging path. However, there have been several courses I would never have been able to take without the program. Three that come to mind are: Gang Violence in Syracuse, Introduction to Zoo
Management, and General Chemistry II Honors Lab which focused on cancer research. The greatest benefit of all, although it may not seem like it in the process, is the Honors Capstone Project. This is a project that can propel you to the next level after graduation, whether it’s graduate school or full-time employment.

Like many other honors students, there are times when you want to quit and drop the program. It’s a commitment which needs to be incorporated every semester; however the end result is worth the effort. You’ll graduate not just with a rare distinction, but with a project that employers and graduate programs will take great interest in. This is a stepping stone into the next level.
Introduction:

The purpose of this project was to design an experiment to be conducted by students enrolled in the Nuclear Engineering Track (NET) supplementary laboratory starting in the spring, 2012 semester. This experiment demonstrates the extraction of the element Neodymium from solution using a process called adsorption. This is a technique currently being researched for its applications to nuclear fuel reprocessing. Nuclear fuel reprocessing is the separation of spent nuclear fuel into individual components. These components include radioactive materials, non-radioactive materials, and unused fuel. The ultimate goal of nuclear fuel reprocessing is to separate out usable components of spent fuel so the waste volume is minimized. Neodymium is an element which is found in spent nuclear fuel that is nonradioactive and has practical applications if it can be isolated.

This project was separated into three parts. The first is a paper I wrote titled A Discussion of Separation of Lanthanides over Actinides which analyzed Dr. Hyung Jun Park’s dissertation. It describes the characteristics of the adsorbent used, ideal conditions for an experiment, and contains some of his results on the adsorption of Neodymium. The adsorbent used in my experiment was the product of Dr. Hyung Jun Park’s doctoral work and my Capstone Project is based on one of the experiments he performed.

The second part of the project was to write a procedure to conduct the adsorption lab experiment. This was divided into two parts. The first, titled
Adsorption Experiment Preparation is a detailed procedure I wrote of the experimental setup and the chemical solutions which need to be made prior to conducting the experiment. This is specifically for the TA or professor who will be running the lab. The second, titled Adsorption Experimental Procedure is a step-by-step procedure written for students to conduct the experiment. It also contains the calculations which will be done once results are obtained.

The final part of the project was to test the experimental procedure written by conducting the actual experiment. Equipment was purchased specifically for this laboratory. Below is a snapshot of the experimental setup. More detail is given in the procedure sections.
The section titled Adsorption Experiment Proper is the lab report I wrote based on the results obtained from conducting the experiment myself and will be comparable to what future students will be expected to complete in the lab. Excellent results were achieved and showed that the experiment adequately demonstrates the adsorption of neodymium. The preparation and experimental procedures were tailored based on improvements identified while conducting the experiment.

The purpose of this lab experiment design is to help students interested in nuclear engineering better understand nuclear fuel reprocessing, a technique which can ultimately minimize the volume of spent nuclear fuel to around 5% of the original amount. It is a process utilized by countries such as France and Great Britain and hopefully will be done in America in the near future.
A Discussion of Separation of Lanthanides over Actinides Using Phosphorus Based Adsorbent
Abstract:

In current nuclear fuel reprocessing, the TALSPEAK (Trivalent Actinide - Lanthanide Separation by Phosphorus reagent Extraction from Aqueous Komplexes) process utilizes liquid-liquid extraction to separate lanthanides over actinides. This paper will contain an analysis of the organo-phosphorus functional silica adsorbent HDEHP’s ability to adsorb lanthanides. After further testing, this may result as an alternative to liquid-liquid extraction in the TALSPEAK process.

The first part of this study is an examination of the aqueous phase speciation of a standard TALSPEAK solution. It was found that a pH of 3 is ideal [2]. The second part is assessing how the distribution coefficient of the solution depends on pH. The distribution coefficient refers to the concentration of lanthanide present in the adsorbent phase relative to the concentration of lanthanide in the aqueous phase at equilibrium. It was found that as pH increased, the distribution coefficient decreased. The third part of the study is examining the effect DTPA (diethylenetriaminepentaacetic acid) concentration has on the distribution ratio of the adsorbent. The distribution ratio refers to the concentration of lanthanide in the adsorbent phase compared to the concentration of the lanthanide-DTPA complex in the aqueous phase. It was determined that as the concentration of DTPA increased, the distribution ratio decreased. The final part of this study investigated the maximum capacity of the adsorbent for each lanthanide. It was determined that europium and yttrium adsorb better than neodymium and erbium.
1. Introduction:

Rare Earth Elements (REEs) are defined as the fifteen lanthanides and yttrium. The purpose of the TALSPEAK process is to separate REEs from actinides. This approach is done for several reasons. First, REEs are not radioactive in nuclear waste; however they are grouped together with actinides because they have similar properties. REE’s are also more abundant than actinides in nuclear waste, so by separating them out, the amount of waste is reduced. REEs also have many technological applications. According to USGS (U.S. Geological Study), Rare Earth Elements are used in the creation of fiber-optics, cathode-ray tubes, and liquid-crystal displays [1]. In the experiments observed, yttrium, neodymium, erbium, and europium were used to represent the sixteen rare earth elements. This selection is made because they are about evenly spaced throughout the series.

The UREX +1a process consists of four sequential solvent extraction processes to separate dissolved spent fuel [2]. The first step is UREX, which is short for uranium extraction. The second part is called CCD-PEG, which removes strontium and cesium from the dissolved spent fuel. The third step is called the TRUEX, which is short for TransUranic extraction. The purpose of this step is to remove all fission products remaining other than the rare earth elements and actinides. The final step is the TALSPEAK process, which is the separation of lanthanides from actinides. To study the TALSPEAK process, a standard TALSPEAK solution is used. This contains 1mM of selected lanthanides, 0.01M DTPA, and 0.25M lactic acid.
Current practice utilizes liquid-liquid extraction (LLE) for the TALSPEAK process. However, extraction is not the only separation process which can be used. Adsorption is currently being researched as an alternative to LLE. The reason for this is because LLE produces a significant amount of waste. The adsorption process on the other hand produces much less waste because adsorbents can be regenerated and used multiple times.

2. Experimental:

2.1 Adsorbent Making Procedure

The adsorbent studied in this paper is a 2-ethylhexyl phosphonate based adsorbent. It is known to be excellent for extracting heavy metal ions like the lanthanides. The following figure shows the reaction to create this adsorbent.

![Synthetic procedure for making 2-ethylhexyl phosphonate based adsorbent](image_url)

Figure 1: Synthetic procedure for making 2-ethylhexyl phosphonate based adsorbent [2]
2.2 Equilibrium Isotherm

It is known that the metal ion creates three bonds with three oxygen atoms along the adsorbent [2]. In order to facilitate bonding with lanthanides over actinides, diethylenetriaminepentaacetic acid (DTPA) is introduced into the solution. When the DTPA molecule complexes with each of the metal ions (REEs and actinides), it impedes the extraction of the metal, more so for actinides than lanthanides.

The equilibrium isotherm experiment was conducted with a 0.01M DTPA and 0.25M lactic acid solution with varying concentrations of the four selected lanthanides ranging between 0 and 1 mM each. The distribution ratio, which is an indicator of how well extracted the species is, was measured for each of the concentrations. The purpose of this experiment is to calculate the maximum capacity and equilibrium constants of the adsorbent from each of the lanthanides. Details of the equilibrium constants are shown elsewhere [2].

2.3 DTPA Variation

The DTPA Concentration Variation experiment was with a 1mM lanthanides and 0.25M lactic acid solution with varying concentrations of DTPA ranging between 0 and 0.1M. The distribution ratio was calculated for each lanthanide at each concentration of DTPA.
3. Results/Discussion:

3.1 Overall Stability Constants and Aqueous Phase Speciation

The following equations show the overall stability constants ($\beta_n$) for each of the complexes shown in the aqueous phase speciation. Note that $L^-$ refers to the lactic acid ion and that $R^{5-}$ refers to the DTPA ion.

$$M^{+3} + L^- \leftrightarrow ML^2^+$$

$$\beta_1 = \frac{[ML^2^+]}{[M^{+3}][L^-]}$$

$\beta_1$ is the ratio of products over reactants. The expression assumes an elementary reaction, meaning each concentration is raised to its respective stoichiometric coefficient.

$$M^{+3} + 2L^- \leftrightarrow ML^3^+$$

$$\beta_2 = \frac{[ML^3^+]}{[M^{+3}][L^-]^2}$$

$$M^{+3} + 3L^- \leftrightarrow ML_3^-$$

$$\beta_3 = \frac{[ML_3^-]}{[M^{+3}][L^-]^3}$$

$$M^{3+} + R^{5-} \leftrightarrow MR^2^-$$

$$\beta_{11} = \frac{[MR^2^-]}{[M^{3+}][R^{5-}]}$$

$$M^{3+} + R^{5-} + H^+ \leftrightarrow MRH^-$$

$$\beta_{12} = \frac{[MRH^-]}{[M^{3+}][R^{5-}][H^+]}$$

The following table shows the stability constant for each of the selected REEs. These experiments were done at a temperature of 250°C and an ionic strength of 0.1M [2].
Table 1: Stability Constants [3]

<table>
<thead>
<tr>
<th></th>
<th>DTPA</th>
<th>Lactate</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>log $\beta_{11}$</td>
<td>log $\beta_{12}$</td>
</tr>
<tr>
<td>Nd$^{3+}$</td>
<td>21.62</td>
<td>24.01</td>
</tr>
<tr>
<td>Eu$^{3+}$</td>
<td>22.39</td>
<td>24.54</td>
</tr>
<tr>
<td>Er$^{3+}$</td>
<td>22.75</td>
<td>24.75</td>
</tr>
<tr>
<td>Y$^{3+}$</td>
<td>22.05</td>
<td>23.96</td>
</tr>
</tbody>
</table>

The following graph is the aqueous phase speciation in a standard TALSPEAK solution (1mM selected Lanthanides, 0.01M DTPA, 0.25M lactic acid) [2].

Figure 1: Aqueous Phase Speciation. By permission of Mr. Hyung-Jun Park [2].
Where $\text{MR}^{2-}$ and $\text{MRH}^{-}$ are the two forms of the DTPA/lanthanide complex, $M^{3+}$ is the lanthanide ion and $\text{ML}_2^{-}$ is the lactic acid/lanthanide complex. As the graph shows, the higher the pH is, the more prevalent the desired $\text{MR}^{2-}$ complex is. The TALSPEAK solution is held at pH 3. This is because if the pH goes any higher, it will bypass the acceptable ionic strength.

3.2 Effect of pH on Adsorption of Lanthanides in the presence of DTPA and Lactic Acid:

To understand the possibility of applying an organo-phosphorus based adsorbent in the reprocessing to spent nuclear fuel, the effect pH has on the adsorption of the specified lanthanides in a standard TALSPEAK solution was investigated [2]. The following figure shows the dependence of pH in adsorption onto the organo-phosphorus adsorbent.

Figure 2: Distribution coefficients of neodymium, europium, erbium, and lanthanide as a function of pH: total REE concentration 1mM; DTPA =0.01M; lactic acid = 0.25M. By permission of Mr. Hyung-Jun Park [2].
As seen on the graph, as the pH increases from 2 to 5, the distribution coefficient of the four rare earth elements decreases. The distribution coefficient is the amount of a species present in the aqueous phase (cm$^3$) relative to the amount present in the adsorbent phase (grams) at equilibrium. The pH dependence is associated with the dissociation of DTPA. The increase in extraction as the pH decreases indicates a lessening in the degree of complexation between the metal ion and complexing agents DTPA and lactic acid. In this case, the complexation ability of DTPA predominates the extractability, which increases as pH increases.

3.3 Effect of DTPA Concentration on Distribution Ratio

The following graph shows the experimental data for the DTPA concentration effect on the distribution ratio.

Figure 3: Dependence of adsorption of Nd(III), Eu(III), Er(III), and Y(III) on concentration of DTPA in an aqueous solution: Total concentration of lanthanide = 1mM; lactic acid = 0.25M. By permission of Mr. Hyung-Jun Park [2].
As shown from the graph, the distribution ratio of the adsorbent decreases as the concentration of DTPA increases. A high distribution ratio is desired because the goal is to keep as much lanthanide in the aqueous phase as possible. A DTPA concentration of 0.01M (1E-2) is used in the standard TALSPEAK solution, which is the minimum amount required for all of the lanthanide ions to bond with a DTPA molecule. It was also noted that europium had the highest distribution ratio, followed by yttrium, neodymium, and finally erbium.

3.4 Equilibrium Isotherm Experiment:

The following graph shows the experimental results of the equilibrium isotherm experiment [2].

![Equilibrium Isotherm Experiment](image)

*Figure 4: Equilibrium Isotherm Experiment. Each REE concentration 1mM; DTPA = 0.01M; lactic acid = 0.25M, pH = 3. By permission of Mr. Hyung-Jun Park [2].*
Where $q$ is the uptake capacity of the adsorbent and conc refers to the concentration of each lanthanide. This graph can be used to determine the maximum capacity of the adsorbent for each of the Lanthanides. A concentration of 1mM is used for the standard TALSPEAK solution. This concentration is used because as shown from the graph, the distribution ratio levels off near 1mM.

4. Concluding Remarks:

An organo-phosphorous functional silica adsorbent was studied as an alternative to traditional liquid-liquid extraction in the TALSPEAK portion of the UREX+1a process. From the experiments, it is evident that the adsorbent exhibits the potential for separation of lanthanides using the TALSPEAK process.

The next step in the process is to test the adsorbent’s ability to separate lanthanides over actinides. Due to the fact that the actinides in nuclear waste are radioactive the adsorbent was tested in a nuclear fuel reprocessing center.

5. Acknowledgements:

I acknowledge that the data shown in Figures 1 through 4 were produced and developed by Mr. Hyung-Jun Park as part of his doctoral dissertation work. I was given the opportunity to observe and learn the procedures for obtaining some of the data shown in the report.
Adsorption Experiment Preparation
Introduction:

The experiments described in *A Discussion of Separation of Lanthanides over Actinides Using Phosphorus Based Adsorbent* were used as a basis to develop a lab module for students enrolled in the NET supplementary lab to conduct. In this lab module, the adsorption and stripping of Neodymium was studied. No DTPA or lactic acid was used in order to keep the experimental variables limited and so it was easier to create a clear breakthrough curve. Instead, Neodymium Trioxide was dissolved in a mixture of water and nitric acid and run through a packed bed column. Afterwards, a concentrated nitric acid solution was used to strip the adsorbent. Samples were collected accordingly.

The following is the procedure intended for the professor/TA prior to students conducting the lab module. This procedure contains direction on how to prepare each required solution, how to replace the packed bed column/batch, and a list of all equipment needed.
Preparation Procedure: For TA One Day Prior to Lab

Stock Solutions and Materials

- HNO₃ 70% (Aldrich)
- NaOH 1M (Aldrich)
- Nd₂O₃ (Aldrich)
- 4, 7 pH buffer solution
- Adsorbent

Solution Preparation

Nitric Acid (Stripping) Solution – Makes 50 ml 1M HNO₃: **Repeat Twice**

- Use 1mL pipette to add 3.125 ml of 70% nitric acid in a 50 ml neck flask
- Fill to the 50 ml line with distilled water
- Cover with Parafilm
- Shake mixture gently to homogenize
- Repeat to make two Nitric Acid Solutions.

Neodymium Stock Solution - Makes 200 ml of 10 mM Nd(III)

- Add approximately 0.841-0.842 g of Nd₂O₃ on weigh paper and funnel into 200ml neck flask (Use metric balance in 421 Link)
- Add 20 ml of 1M HNO₃ (Make a Nitric Acid Stripping Solution if needed)
- Fill to the 200 ml line with distilled water

NaOH Solution(for pH adjustment) - Makes 50 ml of 0.01M from 1M NaOH Stock Solution (Aldrich)

- Pipette 0.5 ml of 1M NaOH in a 50 ml neck flask
- Fill to the 50 ml line with distilled water
- Cover with Parafilm and gently mix

pH Meter Calibration –Do prior to making Neodymium Adsorption Solution (below)

- Pour 50 ml of pH standard 7 and 4 into separate 50 ml beakers
- Insert pH meter into 7 solution. Adjust “standard” knob to match 7.0
- Insert pH meter into 4 standard solution. Adjust “slope” knob to match 4.0.
- Insert pH meter back into 7 solution. Adjust “standard” knob to match 7.0.

Neodymium Adsorption Solution- Makes 200 ml of 2.5 mM Nd(III) at pH 3.5

Note: **Repeat twice**

- Add 50 ml of neodymium stock solution in a 200 ml neck flask
- Add 100 ml of distilled water
- Gently stir solution
- Check pH with pre-calibrated pH meter
- Pour 10 ml of NaOH standard solution into a 50 ml beaker
- Pipette NaOH into Nd solution as needed to adjust pH of solution to 3.5. Start slowly (i.e. 1 drop) and add accordingly from there. 
  Note: if too much base is added, do not add acid because it will increase the ionic strength too much. Rather, start over again with stock solution.
- Fill to 200 ml line with distilled water
- Cover with Parafilm
- Gently shake mixture to homogenize
- Repeat Neodymium Adsorption Solution procedure to create two 200 ml solution of 2.5 mM Nd(III). One is for the batch experiment, the other is for the column experiment.
**Adsorption Column Experiment: Use if Logbook Indicates Replacement**

**Equipment Needed:**
- Adsorption column
- 50+70 mesh sand (Fisher)
- Adsorbent
- 1 L beaker
- 50 ml beaker
- Distilled water
- Ruler
- Scale

**Procedure:**

1) **Adsorbent Preparation: Wetting**
   a. Weigh approximately 0.5g of adsorbent in 50 ml beaker. Record amount used.
   b. Add 5mL of distilled water, submerging the adsorbent.
   c. Cover with Parafilm.
   d. Allow mixture to sit for 24 hours before using it.

2) **Constructing the column**
   a. Assemble column according to Apparatus Diagram (attached). Wait to attach column top until adsorbent and sand have been loaded.
   b. Place a 1L beaker under column and open the valve.
   c. Measure two 0.6g samples of 50+70 mesh sand
   d. Mark the adsorption column with a marker 10 cm from the bottom as reference. **Keep the column filled with DI water at least 10 cm above the bottom of the column throughout the process of loading.**
   e. Fill the column with DI water. The 1L beaker will collect drainage.
   f. While the column is draining, add one of the 0.6g samples of sand into the column and wash down with distilled water. The best technique for this is to measure the sand in a 50 ml beaker, then squirt water into the beaker while pouring sand into the column. If necessary after loading sand, tap column after to make a flat line of sand.
g. Add the adsorbent/water mixture in the column and rinse down with DI water. Allow the adsorbent to settle to the bottom (on top of bottom sand layer). Note, add adsorbent slowly so a uniform layer forms on the bottom sand layer with parallel lines. Squirt DI water in beaker to help pour into column.

h. Add the second 0.6g of mesh sand to the column after the adsorbent has settled to the bottom and formed a layer. Note, also add the last layer of sand slowly so the layers form parallel lines. Once completed, the column should resemble the close-up on the apparatus diagram (See page 24).

i. Once all three layers are added, stop refilling with DI water and allow the column to drain. When the water is 10 cm above the top layer of sand, close the valve.

j. Cover the top of the adsorption column with Parafilm. Column cap still should not be attached.

Note: column should always have solution inside.
Adsorbent Capacity Determination Experiment: Use if Logbook Indicates Replacement

Equipment Needed

• Adsorbent
• 200 ml beaker
• Distilled water
• Scale

Procedure:

• Weigh 0.5g of adsorbent in 200 ml beaker. Record amount used.
• Fill beaker to 100 ml line with distilled water, submersing adsorbent.
• Cover with Parafilm.
• Allow mixture to sit for 24 hours before use.
Apparatus Diagram

- Peristaltic Pump
- Tubing
- Column
- Stand
- Adsorption Column
- Collection Cylinder
- Solution
- Column Top
- See Zoom-In
- Adsorbent
- Top Sand Layer
- Bottom Sand Layer
- Column Release Valve

Adsorption Column Close-up

<table>
<thead>
<tr>
<th>Layer</th>
<th>Thickness</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sand</td>
<td>0.5 cm</td>
</tr>
<tr>
<td>Adsorbent</td>
<td></td>
</tr>
<tr>
<td>Sand</td>
<td>0.5 cm</td>
</tr>
</tbody>
</table>
**Logbook**

When stripping efficiency gets below 80% or the adsorbent is more than 10 months old, replace the adsorbent according to Adsorption Column Replacement Procedure or Adsorption Batch Capacity Replacement Procedure.

<table>
<thead>
<tr>
<th>Date of Experiment</th>
<th>Batch Stripping Efficiency</th>
<th>Column Stripping Efficiency</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
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</table>

**Stripping Efficiency is:**

\[
\text{stripping \ efficiency} = \frac{\text{stripped amount}}{\text{sorbed amount}} \times 100
\]

**Example:**

\[
\text{stripping \ efficiency} = \frac{30.23g}{30.57g} \times 100 = 98.9\%
\]
Adsorption Experimental Procedure
Introduction:

Students will use the following procedure to conduct the **Adsorption of Neodymium Experiment**. This is broken into several sections. First, students will experimentally determine the adsorbent capacity via the batch method. Next, they will saturate a packed bed column by running a Neodymium solution through it. The packed bed will then be stripped using a nitric acid solution. All samples taken will be diluted and run through ICP-MS to determine concentration. The final part of the lab module is the calculations, where students will determine their adsorbent capacity, create their breakthrough curve, and calculate their stripping efficiency.
Experimental Procedure

Equipment Needed:
- Premade adsorption column with adsorbent (column should have distilled water in it)
- Premade adsorbent batch (200 ml beaker with adsorbent submersed in distilled water)
- Plastic tubing (3 ft. long)
- Peristaltic pump
- 500 ml beaker
- (2) 100 ml beaker
- 50 ml beaker
- 10 ml graduated cylinder
- 5- vial and caps

Chemicals Needed:
- 1M nitric acid solution (Prepared by TA as instructed)
- Distilled water (Prepared by TA as instructed)
- Neodymium solution (Prepared by TA as instructed)

Adsorbent Capacity Determination Procedure:
1) Setup
   a. Remove Parafilm from 200 ml beaker with adsorbent. Pour out excess water so just enough remains to keep the adsorbent submerged. **Make sure all of the adsorbent stays in the beaker when pouring out liquids.**
   b. Remove Parafilm from the first 200 ml neodymium solution. Take out 20 ml using 20 ml bulb pipette and respective pump. Put three 5 ml samples into three separate vials for analysis of initial concentration. Be sure to label vials I-1, I-2, & I-3. Discard extra 5ml in to 500 ml waste beaker.
   NOTE: Be sure to record all samples volumes.
   NOTE: Use the small glass vials with green tops for sample collection.
   c. Pour the rest of the Nd solution in with adsorbent.
   d. Cover mixture with Parafilm and gently shake to homogenize.
   e. **Place mixture in shaker bath until Column Operation Procedure is complete.**
   f. Estimated time: 20 min.
2) Adsorption
   a. Remove beaker from shaker bath.
   b. Gently shake mixture to homogenize.
   c. Remove Parafilm from beaker.
   d. Fill three vials with 5 ml of solution from the beaker. Measure using 10 ml graduated cylinder. Label Q1, Q2, & Q3.
   e. Pour remainder of solution into waste beaker. **Make sure all of the adsorbent stays in the beaker. Leave a small amount of solution left to keep adsorbent submersed.**
   f. Estimated time: 10 min.

3) Wash batch
   a. Fill beaker to the 200 ml line with distilled water.
   b. Shake gently to homogenize and allow adsorbent to settle to bottom.
   c. Fill a vial with 5 ml of wash solution. Label Q4.
   d. Pour remaining contents into waste beaker, again leaving enough liquid to keep adsorbent submersed.
   e. Repeat wash twice.
   f. Label the second vial Q5.
   g. Estimated time: 10 min

4) Stripping the adsorbent
   a. Remove Parafilm from first 50 ml 1M Nitric Acid solution.
   b. Pour contents in with adsorbent
   c. Allow mixture to sit for about 5 minutes. Stir occasionally
   d. Fill three vials with contents of beaker. Label Q6, Q7, & Q8
   e. Pour remainder of solution into waste beaker. **Make sure all of the adsorbent stays in the beaker. Leave a small amount of solution left to keep adsorbent submersed.**
   f. Estimated time: 20 min.

5) Wash batch
   a. Fill beaker to the 200 ml line with distilled water.
   b. Shake gently to homogenize and allow adsorbent to settle to bottom.
   c. Fill a vial with 5 ml of wash solution. Label Q9.
   d. Pour remaining contents into waste beaker, again leaving enough liquid to keep adsorbent submersed.
   e. Repeat Twice. Label the second vial Q10.
   f. Estimated time: 10 min.
6) Cleanup
   a. Fill beaker to the 200 ml line with distilled water for storage.
   b. Cover the top of the beaker with Parafilm.
   c. Store beaker in appropriate location.
   d. Pour contents of 500 ml waste beaker into appropriate storage container.
   e. Wash all beakers, graduated cylinders, and hose.
   f. Estimated Total time: 80 min.

Adsortion Column Procedure:
1) Setup
   a. Remove Parafilm from top of column
   b. Pour 75 ml of 1M nitric acid solution in a 100 ml beaker
   c. Remove Parafilm from the second 200 ml neodymium solution. Take out 20 ml using 20 ml bulb pipette and respective pump. Put three 5 ml samples into three separate vials for analysis of initial concentration. Be sure to label vials I-4, I-5, & I-6. Discard extra 5ml in 500 ml waste beaker.
   d. Drain column from 10 cm to 0.5 cm above sand line. Collect excess in 500 ml waste beaker.
   e. NOTE: Keep adsorbent submerged in solution at all times
   f. Insert tubing into 200 ml neck flask of neodymium solution
   g. Run tubing through peristaltic pump (may already be done)
   h. Estimated time: 20 min.

2) Adsorption column operation: Sample collection for breakthrough.
   NOTE: When taking samples, label each vial. Use B1, B2...for breakthrough, W1 for wash, and S1,S2...for stripping
   a. Place the 10 mL graduated cylinder under the column.
   b. Start pump while tubing is not connected to column so the tubing can fill with Neodymium solution. Pump speed should be at 1.1 ml per minute. Once tubing is nearly filled, attach tubing to column cap as shown in Apparatus Diagram to begin flow of solution into adsorption column
   c. Once solution has accumulated in the column 10 cm above the sand, open the valve to start collection of effluent into the graduated cylinder.
   d. Collect the first 4 ml of solution that accumulates in the graduated cylinder and pour into a vial. Make sure to label vial starting at B1 and record sample volumes for use later. Next, Collect 5 samples of 10mL and fill a vial for each one. Pour excess in waste beaker.
Make sure to record the volume of each sample before pouring it into the vial. Finally, collect 25 samples of 5ml.

**NOTE:** On B10 and B20, collect 6 ml and split between three vials (each would be a 2 ml sample). Triplicates allow for error analysis.

**NOTE:** Follow the sample measurements table (page 35) to get a visual of how many samples should be taken.

**NOTE:** Use several 10 ml graduated cylinders and switch them out after taking a sample.

**NOTE:** The first sample will contain a small amount of water left over from when it was stored (about 0.2 ml)

e. Upon completion, close column valve and turn pump off.

f. Estimated time: 3 hours.

3) **Wash column**

a. Remove plastic tubing from neodymium solution and top of column. Wash in sink.

b. Pour 25 ml of distilled water into a 50 ml beaker.

c. Place graduated cylinder under column. Make sure graduated cylinder is clean.

d. Pipette 10 ml of distilled water into column 1ml at a time using a 1ml pipette.

e. Open column valve and allow collection into graduated cylinder.

f. Collect 10 ml from column in graduated cylinder. Once this is done, the water level should be 0.5 cm above the sand line so the adsorbent always stays wet. Use the waste beaker to drain off excess water.

   g. Close column valve.

   h. Fill a vial with collected wash water for analysis.

   i. Estimated time: 20 min.

4) **Stripping the adsorbent.**

a. Place plastic tubing in second 1M nitric acid solution.

b. Run tubing through peristaltic pump.

c. Start pump while tubing is not connected to column so the tubing can fill with nitric acid solution. Pump speed should be at **1.1ml per minute**. Once tubing is nearly filled, attach tubing to column top as shown in Apparatus Diagram to begin flow of solution into adsorption column.
d. After attaching tubing to column, place the 10 ml graduated cylinder under the column and open column valve to start the collection of samples.

e. Take samples in the following order and store in vials: 4 samples of 1.5 ml, followed by 2 samples of 3 mL, 2 samples of 4 ml, 2 samples of 9 ml, and 1 sample of 10 ml. For the two 9 ml samples, split each between three vials (3 of 3 ml) to make triplicates. Use same procedure for sampling.

*See sample measurement table for to get a visual of how many samples should be taken.

f. Once the nitric acid is pumped through and the solution reaches 0.5 cm above the top sand line, close column valve and turn off pump.

g. Estimated time: 50 min.

5) Wash column
a. Remove plastic tubing from nitric acid solution and column top and wash in sink.

b. Pour 25 ml of distilled water into a 50 ml beaker.

c. Place graduated cylinder under column.

d. Pipette 10 ml of distilled water into column 1ml at a time using a 1 ml pipette.

e. Open column valve and allow collection into graduated cylinder.

f. Collect 10 ml from column in graduated cylinder.

g. Fill a vial with collected wash water for analysis.

h. Close column valve.

i. Estimated time: 20 min

6) Cleanup
a. Fill column to 10 cm line with distilled water for storage.

b. Cover the top of the column with Parafilm. Column top should not be attached.

c. Store the column upright and keep in the stand.

d. **Remember to finish Adsorbent Capacity Determination Procedure**

e. Pour contents of 500 ml waste beaker into appropriate storage container

f. Wash all beakers, graduated cylinders, and hose.

g. Estimated total experimental time: 5 hours
Samples Measurement Table:
The following is a chart of the samples that should be taken and the dilutions of each that are required. Dilution instructions are specified in the next section.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Volume (ml)</th>
<th>Dilution Factor</th>
<th>Sample</th>
<th>Volume (ml)</th>
<th>Dilution Factor</th>
</tr>
</thead>
<tbody>
<tr>
<td>Q1 (Batch)</td>
<td>5</td>
<td>1000</td>
<td>B17</td>
<td>5</td>
<td>100</td>
</tr>
<tr>
<td>Q2</td>
<td>5</td>
<td>1000</td>
<td>B18</td>
<td>5</td>
<td>100</td>
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<tr>
<td>Q3</td>
<td>5</td>
<td>1000</td>
<td>B19</td>
<td>5</td>
<td>100</td>
</tr>
<tr>
<td>Q4</td>
<td>5</td>
<td>1000</td>
<td>B201</td>
<td>2</td>
<td>500</td>
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<tr>
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<tr>
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<td>500</td>
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<tr>
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<td>1000</td>
<td>B24</td>
<td>5</td>
<td>1000</td>
</tr>
<tr>
<td>I 1 (Initial)</td>
<td>5</td>
<td>1000</td>
<td>B25</td>
<td>5</td>
<td>1000</td>
</tr>
<tr>
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<td>B26</td>
<td>5</td>
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<tr>
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<td>5</td>
<td>1000</td>
<td>B27</td>
<td>5</td>
<td>1000</td>
</tr>
<tr>
<td>I 4</td>
<td>5</td>
<td>1000</td>
<td>B28</td>
<td>5</td>
<td>1000</td>
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<tr>
<td>I 5</td>
<td>5</td>
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<td>B29</td>
<td>5</td>
<td>1000</td>
</tr>
<tr>
<td>I 6</td>
<td>5</td>
<td>1000</td>
<td>B30</td>
<td>5</td>
<td>1000</td>
</tr>
<tr>
<td>B1 (Breakthrough)</td>
<td>4</td>
<td>100</td>
<td>B31</td>
<td>5</td>
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</tr>
<tr>
<td>B2</td>
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<td>100</td>
<td>W1 (Wash)</td>
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<td>1000</td>
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<td>S1 (Strip)</td>
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<tr>
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<td>S2</td>
<td>1.5</td>
<td>5000</td>
</tr>
<tr>
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<td>S3</td>
<td>1.5</td>
<td>5000</td>
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<tr>
<td>B6</td>
<td>10</td>
<td>100</td>
<td>S4</td>
<td>1.5</td>
<td>1000</td>
</tr>
<tr>
<td>B7</td>
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<td>S5</td>
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<td>5</td>
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<td>S6</td>
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<td>1000</td>
</tr>
<tr>
<td>B9</td>
<td>5</td>
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<td>S7</td>
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<td>1000</td>
</tr>
<tr>
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<td>S902</td>
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<td>S903</td>
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</tr>
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<td>100</td>
<td>S1001</td>
<td>3</td>
<td>100</td>
</tr>
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<td>100</td>
<td>S1002</td>
<td>3</td>
<td>100</td>
</tr>
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<td>S1003</td>
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<tr>
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<td>100</td>
<td>S11</td>
<td>10</td>
<td>100</td>
</tr>
<tr>
<td>B16</td>
<td>5</td>
<td>100</td>
<td>W2</td>
<td>10</td>
<td>100</td>
</tr>
</tbody>
</table>
Dilutions Procedure:

In order for samples to be measured by the ICP-MS, they must be diluted. Following the instructions below, dilute each of the samples as stated by the Sample Measurements Table. Review equipment diagram before starting.

*NOTE: This is a time consuming process. Results will not be accurate if the process is rushed. Use a different pipette for each dilution factor. Estimated time: 3 hours.

1) Dilution Factor of 100:
   a. Using the pipette, extract 0.1 ml of sample and add to the larger 10 ml vial
   b. Using a different pipette, add 0.5 ml of 1M Nitric Acid Solution to the 10 ml vial
   c. Using a large pipette, add 9.4 ml of DI water to the 10 ml vial

2) Dilution Factor of 500:
   a. In a 50 ml neck flask, pipette 1 ml of sample in and fill to the 50 ml line with DI water. Cover with Parafilm and shake. (This creates a dilution of 50, which will now be diluted by 10)
   b. From the neck flask, pipette 1 ml of sample into a 10 ml vial.
   c. Using a different pipette, add 0.5 ml of 1M Nitric Acid Solution to the 10 ml vial.
   d. Using a large pipette, add 8.5 ml of DI water to the 10 ml vial.

3) Dilution Factor of 1000:
   a. In a 50 ml neck flask, pipette 1 ml of sample in and fill to the 50 ml line with DI water. Cover with Parafilm and shake. (This creates a dilution of 50, which will now be diluted by 20)
   b. From the neck flask, pipette 0.5 ml of sample into a 10 ml vial.
   c. Using a different pipette, add 0.5 ml of 1M Nitric Acid Solution to the 10 ml vial.
   d. Using a large pipette, add 9 ml of DI water to the 10 ml vial.

4) Dilution Factor of 5000:
   a. In a 50 ml neck flask, pipette 1 ml of sample in and fill to the 50 ml line with DI water. Cover with Parafilm and shake. This creates a dilution of 50.
   b. Follow the Dilution Factor of 100 procedure using this solution. This will create a DF of 5000.
Standards

Dilute the 1000 ppm Nd standard solution to make a 1 ppm solution. Using the 1 ppm solution, make a 50, 100, 250, and 500 ppb solution. Place in vials for analysis on ICP-MS.
Adsorption Experiment Calculations

1. Adsorption batch capacity calculation.

Concentrations are obtained from ICP-MS results. The amount of neodymium sorbed is calculated by measuring the initial concentration and final concentration in the batch.

\[
sorbed \text{ amount} = C_i * V_{tot} - C_f * V_{tot} - C_{w1} * V_{w1} - C_{w2} * V_{w2}
\]

\[
q_{batch} = \frac{sorbed \text{ amount}}{\text{adsorbent weight}}
\]  

\[
stripped \text{ amount} = C_{stripping} * V_{stripping} + C_{w3} * V_{w3} - C_{w4} * V_{w4}
\]

\[
stripping \text{ efficiency} = \frac{stripped \text{ amount}}{sorbed \text{ amount}} * 100
\]

2. Adsorption column capacity calculation

The amount of adsorbed neodymium ions is calculated by performing a mass balance between the initial and effluent concentrations. Effluent concentrations of Neodymium are obtained from ICP-MS results.

\[
sorbed \text{ amount} = C_i * V_t - \Sigma(C_{eff \ in \ loading} * V_{eff \ in \ loading})
\]

\[
q_{batch} = \frac{sorbed \text{ amount}}{\text{adsorbent weight}} = \frac{C_i * V_t - \Sigma(C_{eff \ in \ loading} * V_{eff \ in \ loading})}{W}
\]

3. Breakthrough point curve and calculation based on the adsorption capacity.

Plot \( C_{eff}/C_i \) for each sample versus time to get the breakthrough curve. From equation 1 and 2, the breakthrough volume can be calculated.

\[
V_t = \frac{W * q_{batch}}{C_i}
\]

Assume: no leaching of neodymium during the column experiment

**Caution:** adsorption capacity is very sensitive to pH, which should be at 3.5 ± 0.1. This can greatly affect adsorbent capacity.
4. Stripping Efficiency

The amount of Neodymium stripped is calculated by measuring the concentration of the column effluent.

\[
\text{stripped amount} = \sum (C_{\text{eff in stripping}} \times V_{\text{eff in stripping}})
\]  \hspace{1cm} (8)

\[
\% \text{ strip} = \frac{\text{stripped amount}}{\text{sorbed amount}} \times 100 = \frac{\sum (C_{\text{eff in stripping}} \times V_{\text{eff in stripping}})}{C_i \times V_i - \sum (C_{\text{eff in loading}} \times V_{\text{eff in loading}})}
\]  \hspace{1cm} (9)
Nomenclature

$C_i = \text{average initial concentration of neodymium solution (mg/L)}$

$C_f = \text{final concentration of neodymium solution (mg/L)}$

$C_{\text{eff in loading}} = \text{effluent concentration during column loading (mg/L)}$

$C_{\text{eff in stripping}} = \text{effluent concentration during column stripping (mg/L)}$

$C_{\text{Stripping}} = \text{Concentration of neodymium in batch stripping solution (mg/L)}$

$C_{w1} = \text{Concentration of Nd in first wash for batch adsorption (mg/L)}$

$C_{w2} = \text{Concentration of Nd in second wash for batch adsorption (mg/L)}$

$C_{w3} = \text{Concentration of Nd in first wash solution for batch stripping (mg/L)}$

$C_{w4} = \text{Concentration of Nd in second wash solution for batch stripping (mg/L)}$

$q_{\text{batch}} = \text{adsorption capacity in batch experiment (mg of neodymium /g of adsorbent)}$

$V_{\text{eff in loading}} = \text{effluent volume during column loading (L)}$

$V_{\text{eff in stripping}} = \text{effluent volume during column stripping (L)}$

$V_i = \text{initial volume of neodymium solution (L)}$

$V_{\text{stripping}} = \text{total volume of batch stripping solution (L)}$

$V_{\text{tot}} = \text{total volume of neodymium solution in batch (L)}$

$V_{w1} = \text{volume of first wash for batch adsorption (L)}$

$V_{w2} = \text{volume of second wash for batch adsorption (L)}$

$V_{w3} = \text{volume of first wash for batch stripping (L)}$

$V_{w4} = \text{volume of second wash for batch adsorption (L)}$

$W = \text{weight of adsorbent (g)}$
Adsorption Experiment Proper
Introduction:

The following is the lab report written after a test run of the Adsorption of Neodymium Experiment was conducted using the original procedure. This is similar to what students will write after they conduct the experiment. The report contains a theory section, an explanation of the experimental setup, the experimental results, discussions, and recommendations for the procedure. These recommendations are now included in the Adsorption Experiment Preparation and the Adsorption Experimental Procedure.
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Abstract

The intention of this experiment was to extract Neodymium from a solution by running it through a packed bed column with a specialized adsorbent. This experiment was done until breakthrough was achieved. Using the calculated breakthrough curve, it was determined that 0.5g of adsorbent extracted 27.81 mg of Neodymium, resulting in an adsorbent capacity of 0.385 mmol/g. This is lower than the theoretical maximum value of 0.6 mmol/g, obtained from equilibrium isotherm data\(^4\). The capacity is lower than the theoretical value most likely because the ionic strength of the solution used was higher than anticipated.

A 1M nitric acid solution was then run through the column after it had been washed to strip the adsorbent. The total amount of Neodymium stripped from the nitric acid solution was calculated to be 30.24 mg, which results in a stripping efficiency of 108.7\%. This is higher than the expected value of 100\% and most likely due to the experimental uncertainty of the initial concentration.

Theory

Adsorption is a process used very frequently in the nuclear industry, specifically in reprocessing. A common method of reprocessing is called the UREX +1a process, which involves four separate steps. The final step is called the TALSPEAK (Trivalent Actinide - Lanthanide Separation by Phosphorus reagent Extraction from Aqueous Komplexes) process, which separates lanthanides over actinides. Current practice utilizes liquid-liquid extraction (LLE) for the TALSPEAK process. However, this is not the only sorption process which can be used. Adsorption is currently being researched as an alternative to LLE. The reason for this is because LLE produces a significant amount of waste. In the case of nuclear fuel reprocessing, this waste is radioactive. An adsorbent on the other hand can be regenerated and used multiple times so less radioactive waste is produced.

The adsorbent studied in this experiment is a 2-ethylhexyl phosphonate based adsorbent. It is known to be excellent for extracting metal ions like the lanthanides. When a Lanthanide-Actinide solution includes the compound DTPA (diethylenetriaminepentaacetic acid) and is under specific conditions, this adsorbent favors the extraction of Lanthanides over Actinides, thus making it a viable method for separation\(^4\). The following figure shows the chemical structure of the adsorbent being used and the reaction made to synthesize it. The maximum neodymium adsorption capacity of this adsorbent at pH 3 is 0.56 mmol/g, however can be as low as 0.29 if the ionic strength is high\(^2\). The maximum capacity at pH 3.5 was estimated to be 0.6 mmol/g and as low as 0.3 mmol/g.
The amount of neodymium sorbed can be calculated using the following equation:

$$\text{sorbed amount} = C_i \times V_i - \sum \left( C_{\text{eff in loading}} \times V_{\text{eff in loading}} \right)$$  \hspace{1cm} (1)

Where $C_i$ is the concentration and $V_i$ is the effluent volume. The adsorbent capacity “$q$” can be calculated using the following equation.

$$q_{\text{batch}} = \frac{\text{sorbed amount}}{\text{adsorbent weight}} = \frac{C_i \times V_i - \sum \left( C_{\text{eff in loading}} \times V_{\text{eff in loading}} \right)}{W} \hspace{1cm} (2)$$

Another important feature of the adsorption column is the breakthrough volume, which is the volume at which the solution breaks through the adsorbent, meaning all adsorbent sites are used so sorbed material is no longer being extracted. This is calculated using the following equation:

$$V_i = \frac{W \times q_{\text{batch}}}{C_i} \hspace{1cm} (3)$$

Where $W$ is the adsorbent weight and $q_{\text{batch}}$ is the capacity of the adsorbent in mg/g.
The breakthrough volume can be seen on the breakthrough curve. The breakthrough curve shows when the adsorbent reaches its capacity. This can be seen by the spike in relative concentration. The figure below shows a theoretical breakthrough curve. The ineffective bed volume is when the bed reaches its capacity and no longer extracts the desired ion.

![Theoretical Breakthrough Curve](image)

A major advantage to adsorption columns is that they can be regenerated and used again. This is done via a process called stripping. When an acid such as nitric acid is introduced onto the adsorbent, the metal ion is removed. The amount of neodymium stripped can be calculated using the following equation:

$$\text{stripped amount} = \sum (C_{\text{eff in stripping}} \times V_{\text{eff in stripping}})$$

The stripped amount can then be used in combination with the sorbed amount to calculate the stripping efficiency, as shown in the equation below:

$$\% \text{ Strip} = \frac{\text{stripped amount}}{\text{sorbed amount}} \times 100 = \frac{\sum (C_{\text{eff in stripping}} \times V_{\text{eff in stripping}})}{\sum (C_{\text{eff in loading}} \times V_{\text{eff in loading}})} \times 100$$

Once the adsorption capacity decreases below an acceptable level (~60-70% maximum) and/or the stripping efficiency also decreases below an acceptable level (~60-70%), the adsorbent is no longer able to be used and needs to be replaced.
Experimental

In this experiment, an adsorbent was placed in a column and used to extract Neodymium, one of the Lanthanides. The column used was 0.7 cm ID and 20 cm in length. A Cole-Parmer Masterflex L/S Peristaltic Pump was used to pump solutions into the column. The following is a diagram of the experiment.

**Figure 3: Experimental Setup**

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>S-1</td>
<td>Stream 1</td>
</tr>
<tr>
<td>P-1</td>
<td>Pump 1</td>
</tr>
<tr>
<td>S-2</td>
<td>Stream 2</td>
</tr>
</tbody>
</table>
The first part of the experiment measured the breakthrough. Here, a 2.5 mmol/L solution of neodymium at pH 3.5 was run through approximately 0.5g of adsorbent in a packed bed column at a rate of 1.1 ml/min. Samples were taken periodically at the bottom of the column to determine the effluent concentration. A wash run was then conducted to flush out the remaining neodymium solution.

The second part of the experiment was the stripping. Here, 50 ml of 1M nitric acid solution was run through the column. Samples were taken frequently so that the amount of neodymium stripped could be determined. The column was then washed again and filled with water for storage.

Each of the samples was then diluted by a specified amount between 100x and 5000x. Each sample was run through the ICP-MS to determine the concentration so calculations could be performed.

Results & Discussion

**Breakthrough**

Figure 4 shows the breakthrough curve, which is a plot of C/Co vs. Bed Volume. All concentrations were determined with the ICP-MS. A 2-period moving average trendline was used to fit these data. See Appendix I for Excel data.

![Breakthrough Curve](image)

The breakthrough curve resembles the theoretical model. The spike in relative concentration is very apparent. The point at Bed Volume 88 is high, most likely...
because of experimental uncertainty. The amount of neodymium sorbed was calculated from these data. This calculation was done by measuring the total amount of Nd in the initial solution and subtracting from that the total amount out in accordance with equation (1). This value was then used to calculate the adsorbent capacity using equation 2. The table below shows the calculated amount of neodymium sorbed and the calculated capacity.

Table 1: Calculated Adsorbent Capacity

<table>
<thead>
<tr>
<th>Initial Mass (mg)</th>
<th>61.10</th>
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<tbody>
<tr>
<td>Total Mass out (mg)</td>
<td>33.29</td>
</tr>
<tr>
<td>Sorbed Amount (mg)</td>
<td>27.81</td>
</tr>
<tr>
<td>q (mmol/g)</td>
<td>0.386</td>
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</table>

This capacity of 0.386 is lower than the theoretical maximum value of 0.6 mmol/g. This could be due to several reasons. The breakthrough curve’s shape is as expected, so the main contributor to this difference is probably the ionic strength of the solution. At pH 3, the adsorbent’s capacity can be between 0.56 mmol/g and 0.29 mmol/g. This suggests that the ionic strength of the solution used may have been on the high end.

**Stripping**

Figure 5 shows the Nd concentration as a function of bed volume while stripping. Concentrations were determined by diluting samples and running them through the ICP-MS.

![Figure 5: Concentration vs. Bed Volume During Stripping](image)
It can be seen from the graph that the largest amount of the neodymium is extracted from the column during the beginning of the stripping. The first sample taken shows a concentration much lower than that of the others. This is most likely due to excess water which was still in the column when the stripping started. The concentration of the effluent was then used to determine the total amount of Neodymium that was stripped. Table 2 shows the results of the stripping efficiency calculation.

Table 2: Stripping Efficiency

<table>
<thead>
<tr>
<th>Sorbed Amount (mg)</th>
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<td>Stripped Amount (mg)</td>
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<tr>
<td>Stripping Percentage</td>
<td>108.7</td>
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Figure 6 is a plot of the Stripping Percentage as a function of bed volume. The stripping percentage was calculated using equation 5.

Ideally, the stripping percentage would be at or just under 100%. There are several possible reasons why our results produced an efficiency of over 100%, but one reason looks to be the main contributor. The solution which was used was 2.5 mmol/L Nd, which corresponds to 360.6 µg/L. The initial sample taken was measured to be 345 µg/L. Most likely what happened is an air bubble got into the pipette when the sample was diluted or an incorrect measurement was made.
Table 3 shows the calculated adsorbent capacity and stripping efficiency based on the actual initial concentration, rather than the measured one.

### Table 3: Calculations Based on the Actual Concentration of Nd Solution

<p>| | |</p>
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<td>Stripped Amount (mg)</td>
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<tr>
<td>Stripping Percentage</td>
<td>98.88</td>
</tr>
<tr>
<td>$q_{\text{batch}}$ (mmol/g)</td>
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</table>

As shown from Table 3, the stripping percentage would be closer to the expected value of 100% had the actual concentration been used. Also, the adsorbent capacity would be 0.424 mmol/g, which is closer to the theoretical maximum value of 0.6 than 0.386. This supports the conclusion that the initial concentration measurement was not correct.

### Assumptions in Model Development

The first assumption made is that no leaching occurs when calculating the breakthrough volume. Leaching is when some of the neodymium ions desorb off the adsorbent and become free ions before it is stripped. This is a reasonable assumption because the results were relatively accurate and accounting for leaching would make the calculations more difficult.

Another assumption is that samples are perfectly mixed. This assumption is valid because all samples were shaken before being used.

### Origin of Errors

The experiment itself offered many sources of error. The dilutions part of the experiment was very time consuming and needed to be conducted in a precise and accurate manor. It would appear that the dilution of the initial solution was contained some experimental error. Perhaps an air bubble entered the pipette when extracting the sample with the pipette or an incorrect volume measurement was made. This could have happened to other samples as well.

Cleanliness is another source of error. All glassware and pipettes need to be carefully monitored to make sure they are clean and free of foreign material. This applies more so to the dilution part of the experiment, where multiple pipettes are being used simultaneously for water, sample, and acid. Some samples are 500 times the concentration of others, so pipette tips need to be switched often when extracting samples.
**Experimental Problems**

Overall, this experiment is long and tedious but produces very accurate and relevant results. Calculations done on experimental data resemble what is expected theoretically. The experimental breakthrough curve for example, bears close resemblance to the theoretical breakthrough curve. The main obstacle is to make sure the experiment is not rushed and to take the time needed to produce accurate results.

**Conclusion**

In this experiment, an adsorbent was used in a column to extract the lanthanide neodymium from solution. The adsorbent capacity was determined to be 0.386 mmol/g. This value is lower than theoretical maximum of 0.6 mmol/g and most likely due to two factors. First, the ionic strength of the solution was probably too high. Secondly, the initial solution concentration was measured to be lower than it actually was. This result impacts the calculations in that it appears that less neodymium was sorbed, thus decreasing the capacity. Overall, the breakthrough curve closely resembles the theoretical one, shown in Figure 2.

The stripping efficiency was determined to be 108.7%. Had the actual concentration of the initial solution been used rather than the measured concentration, the stripping efficiency would’ve been 98.9%, which is almost ideal.

The biggest improvement to the experiment would be to take triplicates of the initial concentration samples and average them. This should minimize experimental error since concentration played such a critical part in calculations.
Nomenclature

$C_{\text{eff in loading}}$ = effluent concentration during column loading (mg/L)

$C_{\text{eff in stripping}}$ = effluent concentration during column stripping (mg/L)

$C_i$ = initial concentration of neodymium solution (mg/L)

$q_{\text{batch}}$ = adsorption capacity in batch experiment (mg of neodymium/g of adsorbent)

$V_{\text{eff in loading}}$ = effluent volume during column loading (L)

$V_{\text{eff in stripping}}$ = effluent volume during column stripping (L)

$V_i$ = initial volume of neodymium solution (L)

$W$ = weight of adsorbent (g)
### Appendices

**I. Breakthrough Data**

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<tr>
<th>Batch ID</th>
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<th>DF</th>
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<th>Tot Solut Vol (L)</th>
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<th>Total Bed mg out</th>
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## II. Stripping Data

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<th>Stripped Volume</th>
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Written Summary of Capstone Project:

Nuclear fuel reprocessing is the separation of spent nuclear fuel into individual components. These components include nonradioactive materials, unused fuel and radioactive materials. The purpose of this process is to separate out the usable radioactive material so that volume of radioactive waste is consolidated. Current reprocessing techniques are able to decrease the waste to less than 5 percent of the original amount. Separated materials can be recycled or reused. This process is industrially utilized in countries such as France and Great Britain but not in the United States. The reason for this is because the original intention of nuclear fuel reprocessing dates back to the Manhattan Project, where spent nuclear fuel was used to obtain high concentrations of plutonium. Due to America’s disarmament policies, reprocessing is only being researched at laboratories such as Oak Ridge National Laboratory until a specific process is perfected that is in alignment with these policies.

The UREX+1a process is one of the leading methods to separate dissolved spent fuel and is the one studied in this project. It is broken into four separate solvent extractions. That is, the dissolved spent fuel is chemically separated into individual and thus more useful components. The first step is called UREX, which is short for uranium extraction. The second part is called CCD-PEG, which removes strontium and cesium from the dissolved spent fuel. The third step is called the TRUEX, which is short for TransUranic extraction and is used to remove the remaining fission products left other than the lanthanides and actinides. The final step is the TALSPEAK process, which is the separation of
lanthanides from actinides. The purpose of this project was to study the TALSPEAK process specifically. Lanthanides are elements 57-71 in the F-block and actinides are elements 89-103, also in the F-block of the Periodic Table.

TALSPEAK is short for Trivalent Actinide - Lanthanide Separation by Phosphorus reagent Extraction from Aqueous Komplexes. Basically, this means separating lanthanides which are non-radioactive from actinides which are radioactive. Lanthanides are also more abundant than actinides in nuclear waste, so by separating them out, the volume is reduced. Another benefit is that these separated elements have practical applications. According to USGS (U.S. Geological Study), lanthanides are used in the creation of fiber-optics, cathode-ray tubes, and liquid-crystal displays. Several actinides also have uses such as californium, which can be used to start nuclear reactors.

The first part of my Capstone Project was a study of the TALSPEAK process and an alternative method being researched here at Syracuse. Dr. Hyung Jun Park developed an adsorbent which could be used as an alternative to solvent extraction. An adsorbent is a material, often a powder or bead, which when a solution or gas passes through it, it extracts a specific ion. In this case, the metal lanthanides were adsorbed. The chief benefit of using an adsorbent is that it can be stripped. Thus, when an acid is run through the adsorbent, it strips off the bonded metal ion so the adsorbent can be reused. In essence this is similar to using a sponge to soak up water and once you are done, you can wring the sponge and use it again. In Dr. Park’s experiments, he ran a solution of lanthanides through the adsorbent in a column, known as a packed bed column. The resulting
solution had a much lower concentration of lanthanide ions, indicating a successful extraction. An acid was then run through the column and the extracted lanthanides were collected separately. Different parameters, such as the effect of pH and concentration on the adsorbent capacity is studied in my analysis paper of his work, titled *Separation of Lanthanides Over Actinides Using Phosphorus Based Adsorbent*.

Syracuse University has just recently introduced the new Nuclear Engineering Track, starting with the course: Introduction to Nuclear Power and Reactor Safety. This track is a combination of courses focusing on nuclear power stations and nuclear fuel reprocessing. Starting in the spring 2012 semester is the supplementary lab. The laboratory will consist of three experiments, all based around current nuclear fuel reprocessing techniques. One of these experiments will be a simulation of the TALSPEAK process using Dr. Park’s phosphorus based adsorbent. The basis of my Capstone Project was to design this experiment, conduct the experiment, prove that it extracts the desired metal lanthanide ion and show that the experiment is repeatable. For my experiment, only the lanthanide neodymium is extracted. Obviously, since the actinides in spent nuclear fuel are radioactive, they cannot be used in the laboratory. However, the adsorption of Neodymium ions from solution will effectively demonstrate the process to students.

To accomplish this goal, I was required to create two procedures. The first is titled *Adsorption Experiment Preparation*. This procedure gives specific instructions for what the TA needs to do prior to class. It includes making specific
solutions and reloading the column when the adsorbent needs to be replaced. The second procedure is the actual procedure that students will use in lab, titled **Adsorption Experimental Procedure**. This includes determining the adsorption capacity in a batch and in a packed bed. For this experiment, the packed bed is a small column filled with powdered resin in which fluid flows through and is collected at the bottom. Students will also strip the adsorbent to determine how much Neodymium can be recovered. All samples will be measured using an inductively coupled plasma mass spectrometer (ICP-MS). This instrument is a very high powered machine used often in research and industry and will show students how very small concentrations of materials in solution are measured.

The next part of my Capstone Project was to conduct the experiment I had designed. Not only did this show that acceptable results could be achieved but I was also able to fine-tune my procedure and take pictures so students would be able to see what the setup looks like. All laboratory equipment was purchased specifically for this experiment by Dr. Park using a grant secured by Dr. Tavlarides.

The write-up of my experiment is titled **Adsorption Experiment Proper**. Excellent results were achieved. An adequate adsorbent capacity was determined and the stripping efficiency was very high. From this, I learned that experimentally determining the capacity would be more beneficial than referencing a theoretical value, so now the experiment includes a batch section to support that. The capacity is determined by taking samples after allowing a batch of adsorbent and solution to come to equilibrium.
The Adsorption of Neodymium Experiment is set up and ready for use in the nuclear engineering lab. This is an experiment which closely simulates what happens in actual nuclear fuel reprocessing. According to most professionals in the field, reprocessing is the best option for dealing with spent nuclear fuel and it is very possible that reprocessing will soon become a commercial operation in the US as it has in France and Britain. This experiment, combined with the two others currently being designed, will give students a very good understanding of current practices. In addition, using adsorption for extraction of ions is a very common method not just in nuclear engineering, but also in many other engineering disciplines so the benefits of understanding this process are not limited just to the nuclear field.

With greenhouse gases now being given more consideration than ever, nuclear power has reemerged as an alternative to coal and fossil fuel plants. The Achilles heel to nuclear power has always been the highly toxic radioactive spent fuel. As the incident at Japan’s Fukushima Daiichi Nuclear Power Plant has shown, if nuclear power is to continue then a plan for dealing with the spent fuel must be created. Reprocessing nuclear fuel is currently the best solution and hopefully in the near future there will be progress towards commercializing it. This adsorption experiment will support a course geared toward gaining a better understanding of nuclear fuel reprocessing.
Sources Cited or Consulted:


