

**OKLAHOMA STATE UNIVERSITY**  
**COLLEGE OF ENGINEERING, ARCHITECTURE, AND TECHNOLOGY**  
**DEPARTMENT OF CIVIL AND ENVIRONMENTAL ENGINEERING**

**SENIOR DESIGN REPORT**

**PRODUCED WATER: RARE-EARTH ELEMENT RECOVERY AND CLEAN  
WATER PRODUCTION**

OKLAHOMA STATE UNIVERSITY

world wide water.

Produced Water: Rare-Earth Element Recovery and Clean Water Production

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## EXECUTIVE SUMMARY

Rare earth elements (REEs) are required in technologies critical for many sectors of society, including transportation, defense and communication. Traditional sources of REEs are mines, but high concentration REE-rich ores are scarce. Produced water from oil and gas operations provide an appealing alternative for the retrieval of REEs. A byproduct of oil and gas processes as well as other natural and industrial operations, the immense volumes of produced water generated every year are typically disposed of as waste. Produced water at an oil field site is often generated at a ratio of 10:1 or even more<sup>1</sup>, meaning that for every barrel of oil produced, there's often at least one barrel of produced water generated. This is a massive amount of waste generation, and produced water injection has led to increased seismic activity and other issues in several regions of the world<sup>2</sup>. A water recycling and reuse system that incorporates REE recovery offers a more economically feasible and environmentally responsible solution to replace existing practices of deep well injection.

Trace amounts of europium, neodymium and lanthanum are REEs found in produced water from Wyoming, USA oil and gas basins. Recovering a high enough concentration of these metals provides potential for an economically lucrative and environmentally more sustainable approach to produced water management.

This report describes the model design and full-scale application of a system capable of removing and separating the REEs from solution as well as removing total dissolved solids (TDS) to levels at or below National Secondary Drinking Water Acts recommendations of 500 mg/L.

System feasibility is tested using a synthetic produced water mixture containing concentrations of compounds similar to produced water solutions fracked from Wyoming and Texas wells. The solutions have extremely high salt concentrations, as well as a few organic and inorganic compounds.

The proposed solution is divided into two main processes: REE removal and TDS removal. REEs are concentrated in an ion exchange column before the remainder of the solution is processed through a thermal desalination unit.

The ion exchange column contains a resin with iminodiacetic (IDA) functional groups. The IDA chelating resin has a high affinity for large, trivalent heavy metals. The REEs thus attach to the functional groups, which from there can be backwashed with hydrochloric acid (HCl) to yield a concentrated solution of the REEs. Since the elements each have different affinities for the column, they will exit at different points in time. Thus, the elements can be separated by recovering the backwash from the column at the appropriate time.

After the solution is run through the ion exchange column it flows towards the thermal desalination unit. The unit uses mirrors and lamps to heat the solution, causing it to vaporize and move up the evaporation column. The TDS, left behind in a solid form, collects on the inside of the glass compartment. The solution then exits the evaporator and moves through a large Teflon condensing tube, which dispenses the clean water into a container. The membrane does have to be periodically backwashed.

The lab prototype is fully functioning, though lacking in efficiency. The thermal desalination system could be further optimized to maximize its filtration capacity. The resin easily pulls out a

large amount of the soluble REEs, though it also pulls out the much higher concentrations of Fe(III) as well as smaller percentages of several other compounds.

The components of the system are theoretically capable of working, and have yielded meaningful, though preliminary, data— further testing and experimentation should be carried out to construct a feasible system. For example, systematic and long-term testing would need to be conducted on the ion exchange column to determine the time and heights at which target REEs can be extracted under closely controlled conditions. This would optimize the resale and reuse value of the REEs, greatly increasing the economic feasibility of the system.

The thermal desalination unit has many different variables, allowing much room for optimization. If the unit's efficiency was optimized, the capacity and cleaning efficiency would increase, which would make the unit a more valuable asset at drilling sites.

Based on theory and data collected from the bench-scale model, an up-scaled unit could collect profitable amounts of REEs and clean large volumes of produced water to dischargeable levels. This would allow for reuse or discharge into the environment. This solution offers a lucrative and sustainable solution to the problem statement.

## DESIGN REPORT

### INTRODUCTION

This report presents design and application plans regarding an economical and efficient produced water reuse and rare earth elements (REEs) collection system.

The designed water-treatment process has two main primary objectives:

1. To obtain a REE recovery efficiency of over 70%
2. To remove total dissolved solids (TDS) to levels at or below drinking water standards of 500 mg/L.

Also included in these objectives was cost analysis for the construction and maintenance of a full-scale, operating system capable of cleaning 2000 gal/day of produced water. The cost estimate includes the disposal costs of wastes generated by the system as well as the revenue generated from the collected REEs.

As shown in Figure 1, the design outlined in this report includes an ion exchange column followed by a thermal desalination unit. The iminodiacetic (IDA) functional groups contained on the ion exchange resin have a high affinity for +3 valence heavy metals, thus capturing the target REEs on the resin. The remainder of the solution then gravity-feeds to temporary storage receptacles before being processed through to the thermal desalination unit.

The bench-scale thermal desalination unit consists of a lightbulb located at the bottom and a reflector plate at the top of the unit. In between the two plates is a chamber that contains a ceramic cylinder coated with nanosprings in which the solution is gravity fed. The nanosprings increase the surface area of the chamber, which wick the water to create surface area and allow for faster evaporation of the solution. Once evaporated, the water vapor travels out of the top of the reactor through a condenser and finally deposits into storage.

The following information was collected and analyzed in a collaborative environment and aims to address the procedures and assessment used during the design process to address the project objectives.

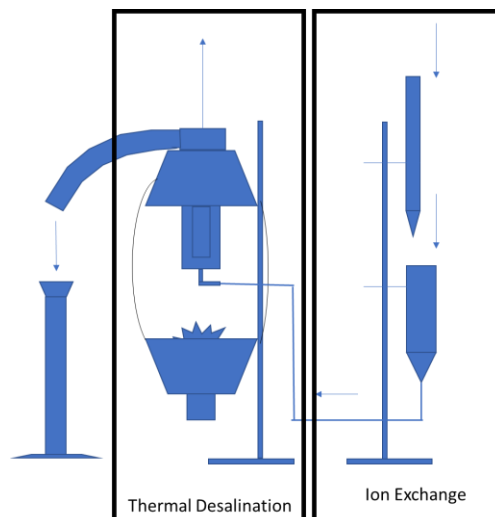


Figure 1: Bench-Scale System Overview

### ALTERNATIVES ANALYSIS

Given the task constraints and objectives, literature research was conducted for research done on, a) REE removal from aqueous, preferably saline, solutions, and b) desalination of high salt solutions.

Due to the high concentration of salts contained within the given stock solution, traditional polymer reverse osmosis membranes were determined to be economically infeasible. Membrane processes require sufficient pressure to overcome the osmotic gradient, which is extremely energy intensive in high salinity solutions. Polymer membranes will foul rapidly for high



concentration solutions. The design was narrowed down to three types of desalination units: solvent extraction, thermal desalination, and membrane distillation.

1. **Solvent extraction.** Solvent extraction is a separation technique that requires no membrane, which means no backwash system is necessary for a descaling process. It utilizes a temperature-dependent solvent that selects water over salts. It was an appealing option as it works in much the same way as reverse osmosis but is capable of handling hypersaline brines<sup>3</sup>. Unfortunately, studies for this removal method are in very early stages and technology remains outside the project budget.
2. **Membrane distillation.** Membrane distillation uses porous, hydrophobic plates. They are different temperature on each side which causes a partial pressure difference, transferring the vapor molecules across the membrane<sup>4</sup>. They have been proven to be an effective solution for high quality desalination of water, but even so, it only seems to be effective for low to medium brines<sup>5</sup>.
3. **Thermal desalination.** Thermal desalination evaporates the water, leaving behind any salts, minerals, and other contaminants originally contained in the solution.

Due to the high salinity and desire for simplicity, thermal desalination was selected for the system.

Most research on REE recovery from produced water is relatively recent. Before selecting the ion exchange resin solution, several ideas were researched that ultimately were discarded. These ideas either didn't have enough experimental data supporting their effectiveness in high saline solutions or didn't meet the efficiency goals set by the project parameters. The alternatives for REE recovery were: (1) Biopolymers, (2) Ion Exchange Resins, and (3) Filtration Systems.

1. **Biopolymers.** Literature on naturally occurring biopolymers presented a solution that was environmentally sustainable and has the potential to become extremely economically feasible<sup>6</sup>. Biopolymers are incredibly diverse in use and readily available. The polymer has to be correctly functionalized to attract targeted ions. Currently, there are no functionalized polymers on the market aimed at REE removal, thus not good solution for this project.
2. **Ion Exchange resins.** Using an appropriate ion exchange column would allow for the ions of the +3 valence REEs to be exchanged with the ions contained on the functional group of the resin, which is an insoluble material of high molecular weight.
3. **Filtration system.** Using a pretreatment filtration system enable the TSS concentration to be drastically reduced before the influent is further treated. It would remediate the high fatigue related to the high TSS on the later treatment processes.

A similar product is an ion exchange resin with IDA functional groups and is available commercially. This is the route chosen for the system.

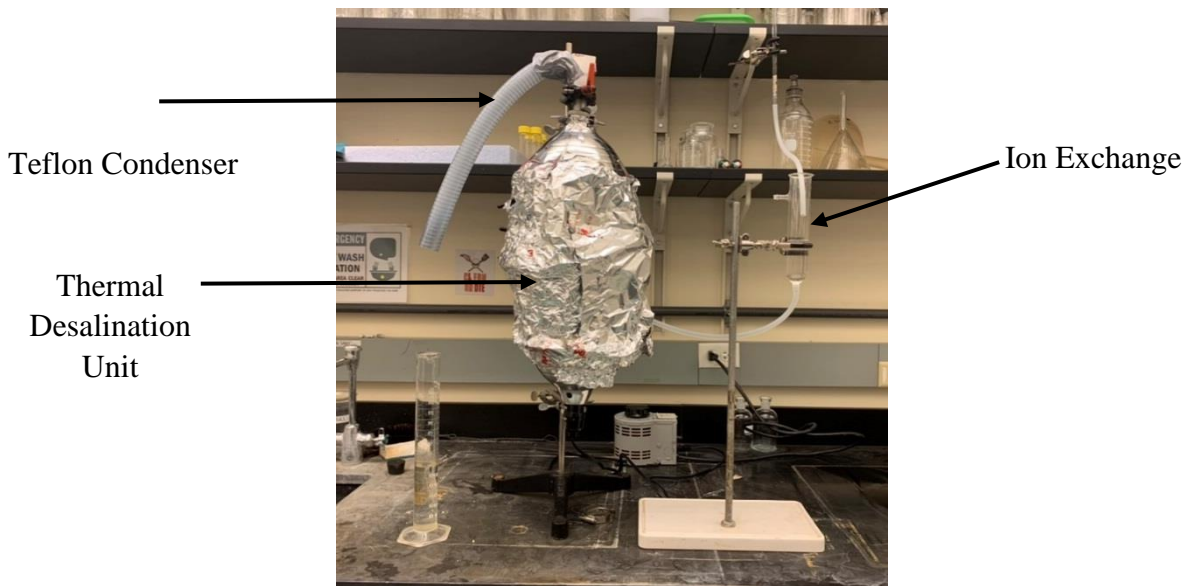
Another potential pretreatment that remained in the design for a long time was a primary filtration system targeting large amounts of the present TDS. This aimed to prevent fouling of the more expensive and more delicate nano-filters. A solution for this was black walnut media filtration systems which could potentially provide a relatively cheap, long lasting, and sustainable solution. Another appeal to these systems is that they are already used at oil and gas

operational sites, and there are already companies that produce the necessary equipment<sup>7,8</sup>. However, primary filtration was ultimately eliminated from the bench scale design due to time and cost constraints.

## **BENCH SCALE/PROTOTYPE**

A lab scale model of the selected approach was designed and assembled. Experiments testing the performance of the system yielded data displaying the efficiency and feasibility of the system. This data was evaluated, providing the information necessary to upscale the unit into a full-size design. During these tests, the team always made sure to follow proper lab protocol and safety recommendations when handling harmful chemicals.

The prototype set up can be seen in the Figure 2.



*Figure 2: Bench-scale Design Setup*

## **Design**

The final designs selected were chosen for their feasibility, ease of access, and their ability to prove a concept. The unit is composed of three primary systems: the REE recovery filtration system, the desalination and purification system, and product and waste management. Figure 2 shows the flow schema of the design process.

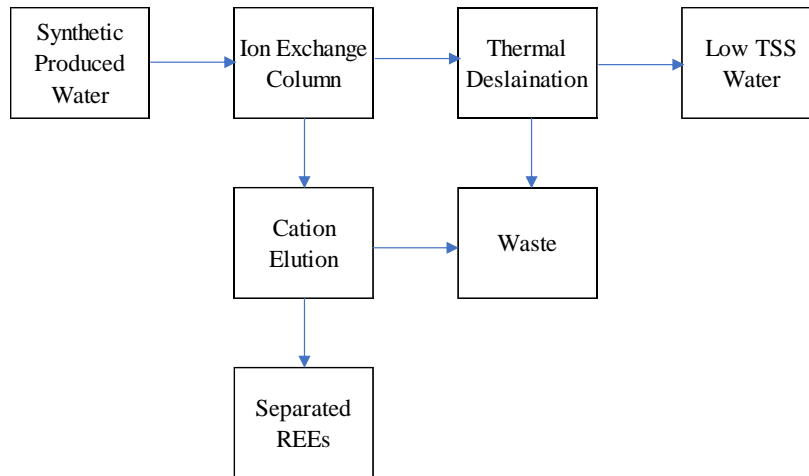


Figure 3: Bench-Scale Schematic

### Ion Exchange Column

An ion exchange column is used to separate the REEs from the produced water solution. No pretreatment was selected for the reactor based on the assumption from the specifications that the salts in the synthetic produced water were dissolved. The synthetic produced water flows into the column at a controlled rate, using a flow equalization tank. The incoming water will be stored in a tank and released to the ion exchange column at a controlled rate. Figure 3 shows an overview of the Ion Exchange system.

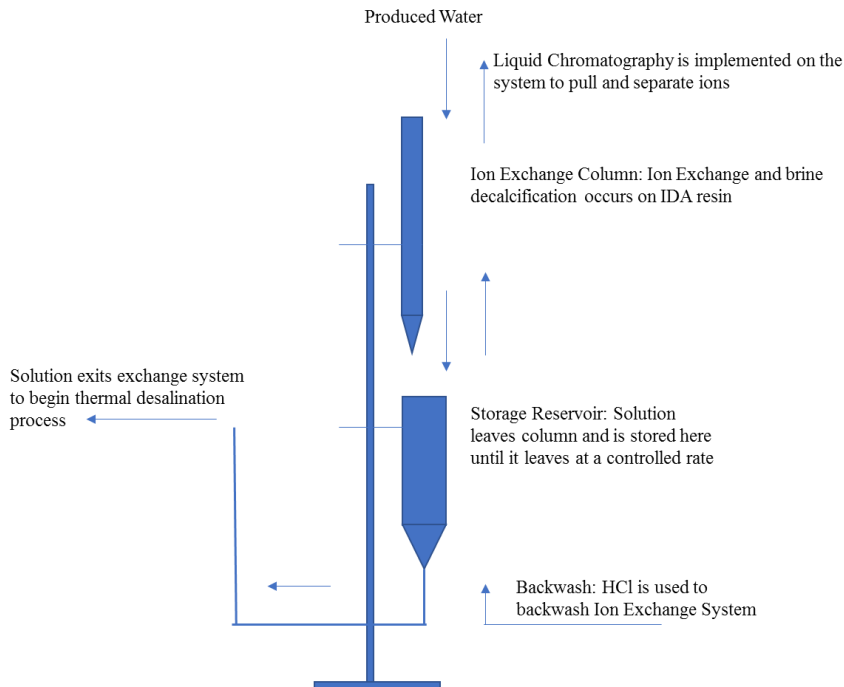


Figure 4: Detailed Ion Exchange System Diagram

The column contains an ion exchange resin with IDA functional groups<sup>9</sup>. These resins are commonly used for brine decalcification, commonly referred to as water softening. A common problem in high salt solutions is that the saturation point of the water is exceeded, and the chlorine and hydroxide ions contained within the resin begin to react and form solids. When using an ion exchange resin to target specific ions, the precipitated salt quickly clogs the column. In addition to having to backwash the column more frequently, it also decreases the optimized life expectancy of the resin, resulting in higher operation costs and waste production rates. The composition of the resin allows the salts to more readily filter through the column, and the other compounds have a higher chance of bonding to the functional groups.

The anion of iminodiacetic acid can act as a tridentate ligand, meaning it has three atoms that can function as donor atoms<sup>10</sup>. This quality is what gives IDA chelating agents a high affinity to +3 heavy metals – including the REEs targeted in this design – coordination complexes. The high capacity and osmotic stability of the resin used in this design offer a cost effective and efficient system.

Once the solution runs through the column it is collected and distributed at a controlled pace into the column that gravity-feeds the thermal desalination unit. The REEs and other minerals collected on the ion exchange column can be collected by backwashing the column with HCl. Due to the extremely high ligand capacity of the selected resin, it would only have to be backwashed periodically.

Hydrochloric acid was chosen for backwashing because of the REEs natural affinity for chlorine. Once the HCl is run through the column, the REEs will prefer to complex with the chlorine rather than the IDA on the resin. The affinities of complexations for each REE differ; those with a higher affinity for the backwash solution will be bound first, and consequently exit the column first. The order and time interval of the REE-chlorine complexations leaving the column can be measured to successfully recover the elements separately, according to the multicomponent chromatography theory.

### Thermal Desalination

The thermal desalination unit serves as the final treatment to remove the remaining total dissolved solids in the produced water. It uses the simple process of evaporation but utilizes emerging technology in material production. With thermal lamps the water is wicked up on the surface of a ceramic column coated with silica nanospring. The prototype was tested as described in the Performance Data section.

Per Dr. David McIlroy with Oklahoma State University's Department of Physics, the nanospring surface increases the area of evaporation from about 140cm<sup>2</sup>, to approximately 105m<sup>2</sup>, over 7,500 times more surface area than without the nanospring surface<sup>11</sup>. This surface area creates a wicking effect that allows the evaporation rate to increase, speeding up the treatment and using less energy. The nano springs also act as a nanofilter to further remove solids in the water. After the nanospring column, the vaporized water is condensed in a Teflon condensing tube and is low TSS water.

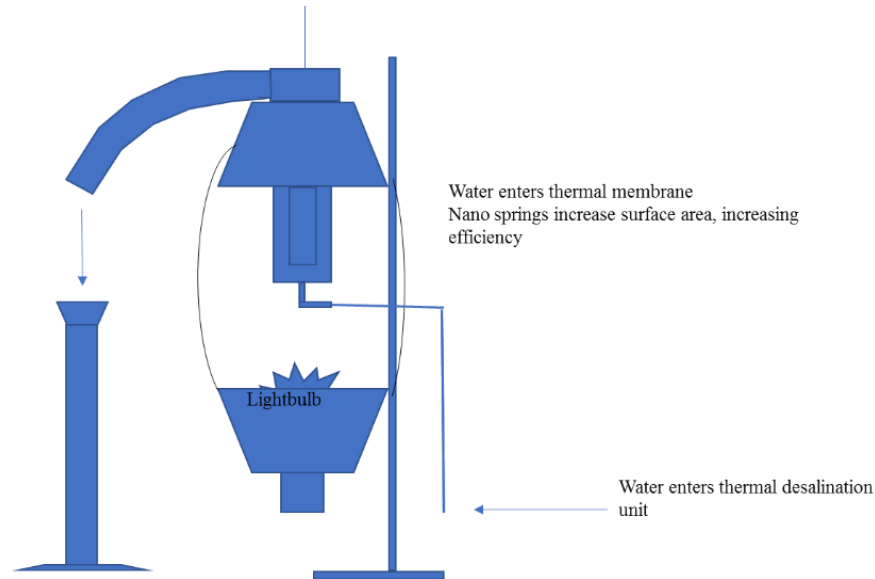


Figure 5: Detailed Thermal Desalination Diagram

The maintenance of the thermal desalination unit consists of maintenance of the heating source as well as the backwash for the nanospring column. The nano surface is durable to most conditions the system would have. The main source of damage to the column is physical removal of the surface. The springs can be removed with a simple scratch with a fingernail. The nanosprings occasionally lose their efficiency and, thus, the ceramic rod has to be recoated. This is a simple maintenance procedure, requiring new nanosprings simply coated over the old ones or the old ones be wiped off and new ones grown onto the surface<sup>12</sup>. A summarized diagram can be seen in Figure 4.

### Products and Wastes

The system will have three separate final products: REEs in solution, potable water, and waste byproducts. There will also be intermediate wastes from the ion exchange resin and the HCl backwash of both the column and the thermal membrane.

The final output of the thermal desalination will be distributed in a container as it exits the unit. This water should meet the 500 mg/L of TDS requirement.

The backwash from the ion exchange column will distribute to another column. This contains the retrieved REEs as well as some other solutions as byproducts of the ion exchange process. This byproduct will further be refined into the individual compounds. All collected compounds as well as the HCL backwash from the synthetic solution are able to be washed down the drain.

The waste reciprocal will contain all products backwashed from the desalination unit as well as the 0.5M HCl used for the backwash. The process flow diagram for the full-scale model is more complex and has more waste outputs. This can be seen in the 'Full-Scale Design Description' section on Page 14.

### Performance Data

The first ion exchange column tests set out to prove the resin selects and binds to +3 heavy metals. Tests were run at the following resin concentrations and compound combinations. Concentrations of the REEs and other components in water were determined by analysis with ICP/optical emission spectrometry<sup>13</sup>.

*Table 1: Milligrams of Resin per Liter Treated*

<b>Experimental Resin Concentrations (mg/L):</b>
565
1130
5650

*Table 2: Ion Combinations*

<b>Combinations of Ions Tested:</b>
La <sup>+3</sup>
Nd <sup>+3</sup>
Eu <sup>+3</sup>
La <sup>+3</sup> , Nd <sup>+3</sup> , Eu <sup>+3</sup>
Full Synthetic Produced Water Solution

All concentrations yielded similar results. The following graphs show the results of the ICP Data at the 5650 concentration. As shown, very significant removal of the REEs was achieved with this method.

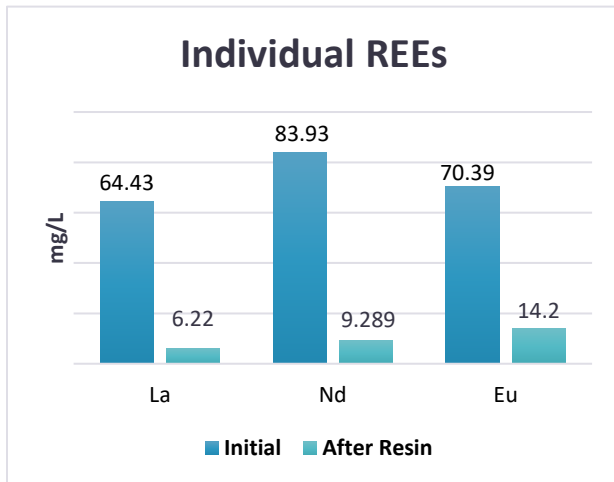


Figure 6: Resin Data - Individual REEs

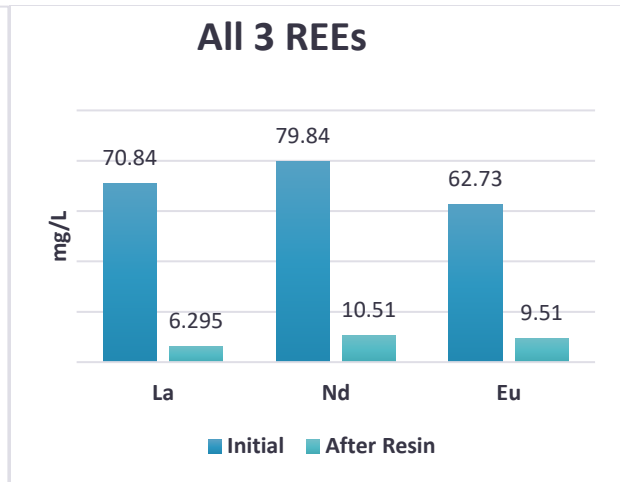


Figure 7: Resin Data – All Three REEs

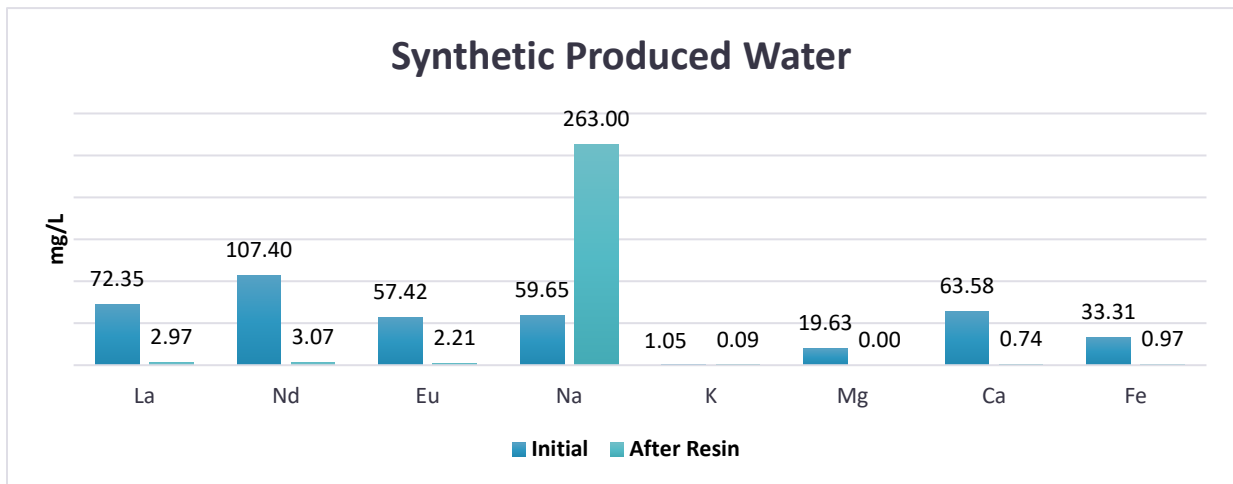


Figure 8: Resin Data – Synthetic Produced Water

Initial testing on the thermal desalination unit was performed using a stock solution of 10 g/L NaCl. Because of the strong correlation between the salinity of water and conductivity, conductivity was the parameter that was measured to assess the % removal of NaCl. Test result were as follows:

Table 3: Thermal Desalination Proof of Concept

<b>Initial Conductivity:</b>	16.91mS
<b>Final Conductivity:</b>	78.5µS
<b>% Removal:</b>	99.5%

Several secondary runs exhibited similar results. Without pretreatments, at 99.5% removal, the systems TSS output would be just above the 500 mg/L mark, at around 800 mg/L. With a pretreatment filtration system in place, the extra removal would bring the system outputs to below the TSS requirement.

### **Analysis/Technical Evaluation**

Results from testing the ion exchange resin, seen in Figure 5, validated the usefulness of the resin. It contained over 70% of the REEs in every given scenario. The increased soluble  $\text{Na}^+$  ions show functional groups are binding to the +3 heavy metal ions and the increased sodium in the solution implies that the  $\text{Na}^+$  ionic forms attached to the functional group are being released from the resin into the solution.

Initial test results from the thermal desalination unit, as displayed in Table 3, gave a high TDS removal level, proving the system was capable of filtering water to a high-quality level. The system also had no visible scaling and no decreased efficiency after tests using low TDS concentration level solution, which increased confidence in its ability to handle higher levels of solids.

## **FULL-SCALE DESIGN DESCRIPTION**

The scale-up consisted of the components presented in the bench-scale as well as additional technologies:



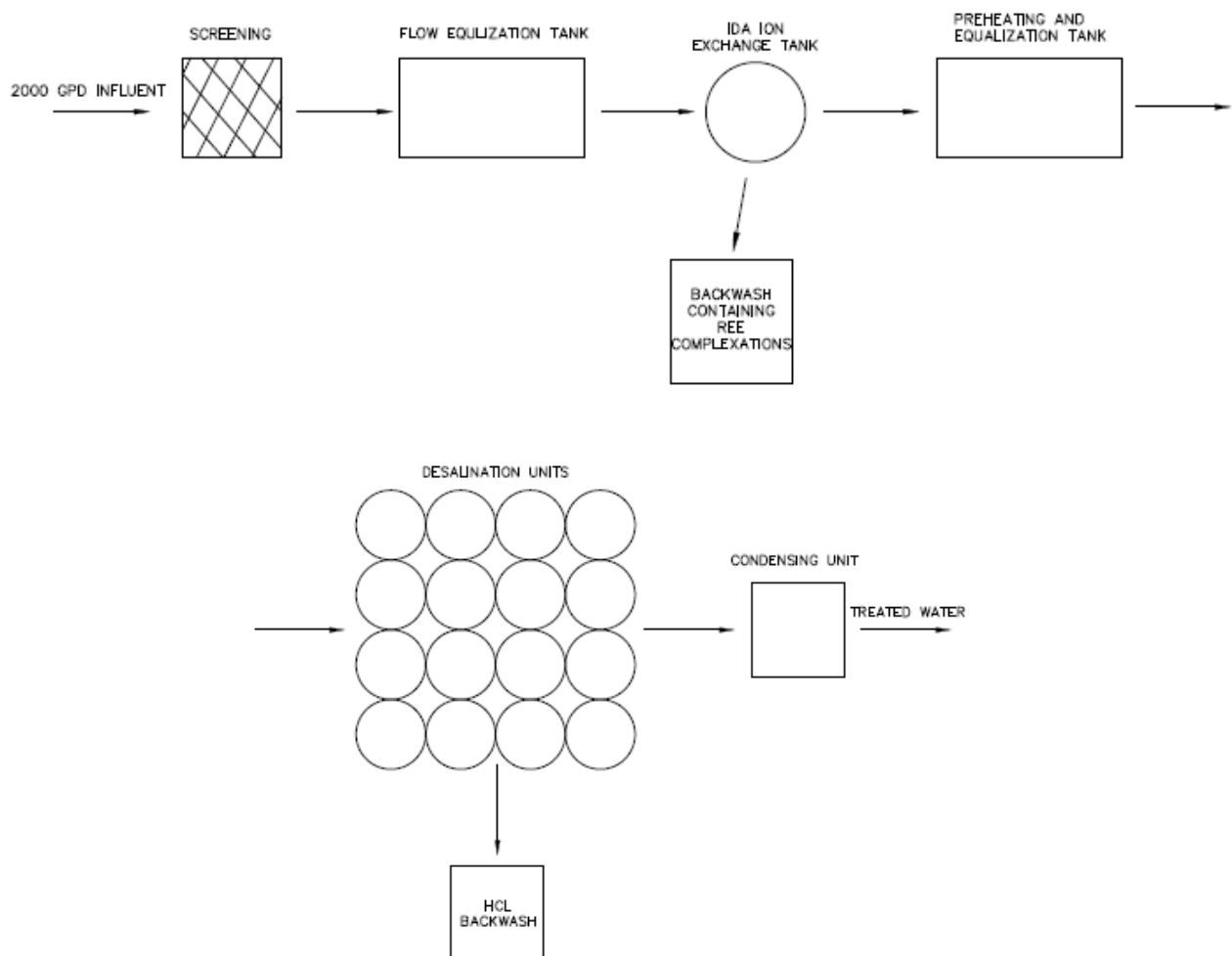


Figure 9: Scale-Up Layout Diagram

The full-scale system consists of pre-screening treatments and a standard flow equalization tank. The system has been scaled to fit on a trailer for easy transport on and off site, and to accommodate the fluctuations in ambient temperature. The ion exchange tank and desalination units are scaled up from the bench-scale model and sizing and pricing estimations can be seen below in the Business Plan.

The system also has accounted for energy efficiencies. The consideration of an alternative power source – such as solar thermal, geothermal, or flare gas – was an initial idea that became practical with the nanosprings. They allow the evaporation rate to increase, hence decreasing the energy demand. When solar power is not available, whatever onsite power is being used can be connected.

Once the water exits the condensing unit, the treated water may need additional treatment to meet Water Quality Standards promulgated by the state or EPA prior to discharge. Additionally, any Effluent Limitation Guidelines under 40 CFR 435 must be met. The developed system is limited to 2,000 gal/day; the amount of available profit is limited to this system parameter unless

further scale-up is implemented. The business plan outlines the application and implementation of the full-scale system as well as cost analysis and targeted demographics.

## BUSINESS PLAN

### Mission

Our mission is to provide an avenue for the produced water industry to affordably treat and dispose of produced water. Our system's technology allows for a cost effective and even lucrative opportunity for the industry. It also enhances national security and decreases domestic reliance on foreign powers. Waste disposal accounts for a large percentage of the handling of produced water. The solution presented in this report minimizes costs by removing the high salt content and creates a new revenue stream from the sale of REEs. Our vision is simple: make the best solution for both the current environmental and cost concerns in the industry. With the advancement of our technology in the Mobile Treatment Unit, this is more than achievable.

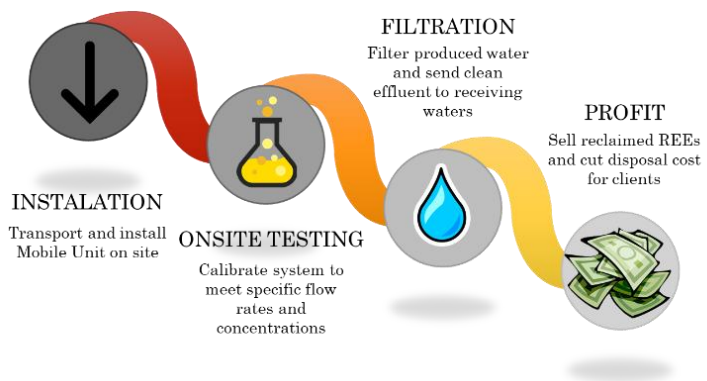


Figure 10: Business Flow Schematic

### Business Description

The treatment of produced water with the IDA Ion Exchange resin and Thermal Desalination Unit is a modified solution using familiar technology to the current industry. The ion exchange process is a common method used to remove specific ions. However, the IDA resin has not currently been utilized for REE removal. Our solution takes this specific resin as well as the current method of ion exchange and applies it in a new way. Similarly, evaporation is well known but rarely used due to energy inefficiencies. Yet the advancement in nanospring technology and potential for integration with low-grade heat from renewable energy sources increases the evaporation rate and further decreases the energy consumption. Due to the lack of data on REE concentration within produced waters, initial business contact would be to implement an ICP-OES to measure REE concentrations to test for the economic feasibility of system implementation. Our solution is the utilization of advancing technologies with the familiarity of current methods.

### Market Analysis

The FracTracker alliance has reported over 1.7 million oil and natural gas well sites in the U.S.<sup>14</sup> There is limited data on REE levels in produced water, however. Less than 1% of

the 150,000 well sites listed on the USGS National Produced Waters Geochemical Database reported that they had REE data (Figure 8).

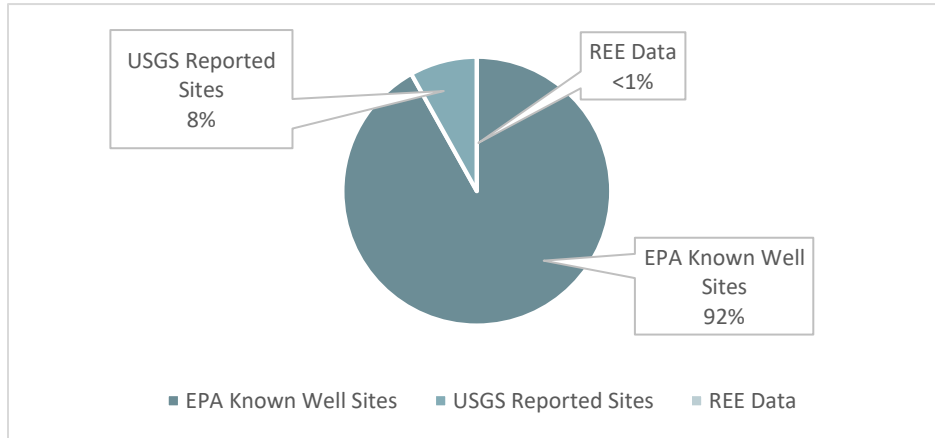


Figure 11: Well Sites with Reported REE Data

According to existing literature, there are trace amounts of REEs found in all produced water at the ng/L levels. Concentrations can be six orders of magnitude higher in select locations<sup>15</sup>. There is a need for further research on the exact levels of REE concentrations as well as how many sites contain these economic commodities. There is no known technology currently attempting to obtain REEs from the water, thus market saturation would be slow and the need for the REEs high for the foreseeable future.

About 1.1 million hydraulic fracturing wells exist in the United State<sup>14</sup>. Project parameter indicate our system would be tested on oil sites at Wyoming oil basins as well as the Permian Basin in Texas that use fractured or conventional wells. Active wells in Wyoming and Texas total 131,990 as of 2018<sup>15</sup>. The Permian Basin’s high land acquisition cost and higher regulations make it a less ideal candidate for implementation of our system. The total number of service optimal wells now stands at 30,724 wells, the number of active oil wells across Wyoming’s oil basins.

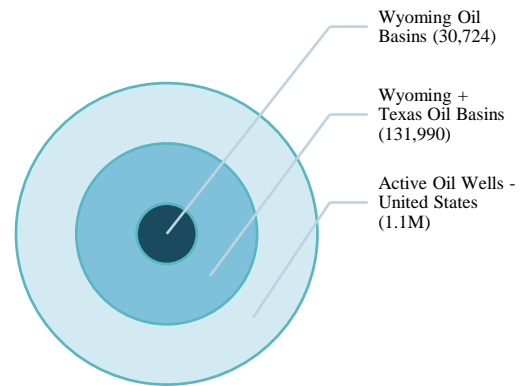


Figure 12: Targeted Business Demographic

**Market and Sales/Savings**

The economic feasibility of the IDA resin paired with the nanospring desalination unit is promising. The ion exchange system has technology able to regenerate the surface of the

resin. This cuts the cost of waste disposal and the cost of replacing the resin. The addition of nanosprings increases the surface area, which increases the evaporation rate and reduces the energy needed to evaporate the water. Additionally, solar thermal energy can be implemented on site to remediate any energy consumption costs. Figure 10 summarizes the anticipated cost savings.

*Table 4: Nanospring Cost Analysis*

Savings from Nanosprings			
Test Method	Tested Volume (mL)	Units Need for 2000GPD	Cost of Power for 2000GPD
Without NS	51.0	10,267	\$ 13,344.64
With NS	11.3	46,336	\$ 2,956.75
		Amount Saved by Nanosprings	\$ 10,387.88

Included below (Table 4) is the estimation for our mobile unit as well as the annual operation costs. Most of our anticipated costs is from training and yearly expenses from the operators. The solar panel system cost estimation was based on an estimate given by a solar energy company. Table 5 presents the anticipated operation costs.

Table 5: Scale-Up Cost Analysis

<b>Price of Mobile Treatment Unit</b>			
Qty	Item	\$/item	Total \$
1	Trailer	15,000	15,000
1	Solar Energy Requirements <sup>16</sup>	110,000	110,000
1	4" dia 5" clear pvc pipe	25	25
	40 Day Supply of Resin	374	15,345
2	Electric tankless water heater	461	923
2	Water Heater Booster	1,394	2,788
10	22 Gal. Vert. Tank	178	1,782
60	4"dia. Nanospring	150	9,000
10	50' 0.25" copper tube	39	390
3	2500 gallon holding tank	910	2,730
Sum			\$ 157,982
Contingency			25%
Adjustment			\$ 39,495
Projected Cost			<b>\$ 197,477</b>

Table 6: Operation Cost Analysis

<b>Annual Operating Cost</b>	
Task	cost/year
Transportation On-Site	1,500
Resin	4,626
Trained Personnel On-Site	312,000
System Maintenance and Repairs	86,000
Waste disposal	150,000
Total	<b>\$ 554,125</b>

Sale of the reclaimed REE compounds is another idea that has a lot of potential. Our system currently removes over 95% of them from the influent. The scale-up could potentially recover around 70% during elution from the column. The only parameters that our system cannot change are the concentrations entering our system from the source, and the market prices of REEs. This is discussed in the Risks and Uncertainties portion of this report on page 22. The revenue estimation of the current market price with assumed 1mg/L and a 2000GPD system is in located in Table 6.

*Table 7: Predicted REE Revenue*

Profit			
REE	USD/kg <sup>17</sup>	Annual Reclaimed	Annual Profit
La	1.96	27.594	\$ 54.08
Nd	50.4	27.594	\$ 1,391
Eu	0.0588	27.594	\$ 1.62
Total Annual Profit			<b>\$1,446.44</b>

### **Product Development**

Our bench scale model has generated data that has been very promising. The lab scale system has proven that our selection of resin has an affinity for REE ions in the produced water. The thermal desalination unit has proven to reduce energy needed to evaporate water and remove salinity. We are anticipating a design for 2000 gallons per day. Furthering our research on our bench scale would include investigations to maximize the REE removal using IDA, like adjusting temperature and pH. Included in this testing would be maximizing the salt removal and energy efficiency of the Thermal Desalination Unit. After optimizing these conditions, the next step would be upsizing to a full-scale system. Beta testing would be necessary before making the technology a reliable and usable throughout the industry.

### **Waste Generation Considerations**

As designed, the system will have a recurring waste stream. The amounts and cost estimates are tabulated on a monthly basis. There are also several one-time construction byproducts that must be managed and properly disposed of.

#### Construction Generated Waste

The system is designed to be mobile. This minimizes initial construction waste generation as no soil is disrupted and minimal material waste is generated.

### Recurring Waste Expenses

The actual system will have three sources of recurring waste: the spent resin, the 0.5M HCl backwash solution, and the total dissolved solids pulled from the solution. The table below shows each system byproduct and its information.

*Table 8: Waste Analysis*

<b>Waste</b>	<b>State of Matter</b>	<b>Hazardous?</b>	<b>Disposal Method</b>
TDS Waste	Solid	No	Landfill
IDA Resin	Solid	No	Reuse/Recycle
0.5 M HCl	Liquid	No	Injection Well

All wastes are non-toxic in the forms applied to this project. The TDS waste can be dried and potentially disposed of in a landfill. When using real produced water instead of the synthetic, hazardous compounds, including naturally occurring radioactive materials (NORMs), may be present. This would have to be considered, thus increasing disposal costs for the landfill disposal process. The HCl waste would probably be disposed of in a traditional sludge injection well<sup>18</sup>. The IDA resin can potentially be regenerated by washing with NaOH, increasing sustainability and economic feasibility. If deemed inefficient, the resin can either be disposed of or the resin company could be contacted for potential pickup. For simplification of cost estimation, resin is assumed to be solid waste. Due to the relatively low volumes and the low molarity of the HCl solution, it is non-hazardous.

Table 8 displays the expected recurring monthly expenses associated with the system. Costs are current and based on estimates in the Western United States, as the projects' guidelines work with a mixture synthesized based on Wyoming and Texas oil basins. From the 99.5% salt removal, estimated TDS waste is estimated to be around 2,440 pounds per day for the 2,000 GPD system. Using the ion exchange resins capacity, the HCl backwash capacity is estimated to be about 590L per day, converting to approximately 3.67 barrels of sludge waste.

Table 9: Waste Disposal Cost Analysis - with System

Expense	Cost/Unit	Amount of Waste	Estimated Expense
Injection Well - Sludge	3.50/bbl <sup>18</sup>	3.67 bbl	\$12.85
Solid Waste Disposal	\$38.27/ton <sup>19</sup>	1.22 tons	\$46.69
<b>Total Estimated Expense:</b>			\$59.54

In comparison, the EPA states that it costs approximately \$0.25 per barrel to dispose of produced water in an injection well. The following cost estimation uses equivalent volumes and units to allow for comparison of the new design systems waste generation and injection well waste generation.

Table 10: Waste Disposal Cost Analysis - Injection Well

Cost/ Unit	Equivalent Amount	Estimated Expense
\$0.5/bbl <sup>18</sup>	47.1 bbl	\$23.53
<b>Total Estimated Expense:</b>		\$23.53

Though the actual waste disposal process is more expensive, the cost of transporting 2000 gallons of water to an injection well site is significantly more than the cost of transporting 1 ton and 4 barrels of waste to potentially much closer locations.

The above expense calculations were taken into consideration throughout the Business Management Plan. The amount of generated waste as well as the type of waste also factors substantially into the sustainability, risk, and community relations plans.

The system has to be moved on and off site before and after operation. This cost varies from one project to the next but can be easily estimated prior to project initiation<sup>20</sup>.

Table 11: Transportation of System Cost

Duration of Transport (mi)	Cost per Mile (\$)
0-500	5.00
>500	1.75



## Health, Safety, and Environmental Regulations

The system should meet and exceed all necessary codes and standards. All general safety guidelines should be followed as well as a number of EPA regulations. Most of the oil and natural gas industries waste generation procedures and regulations fall under state jurisdiction. The following Federal and Wyoming State Regulations are applicable to the project.

Relevant EPA guidelines include<sup>14</sup>:

- Safe Drinking Water Act (SDWA)
  - National Secondary Drinking Water Regulations (NSDWR),
  - National Primary Drinking Water Regulations (NPDWR), and
  - Underground Injection Control (UIC)
- Clean Water Act (CWA),
  - National Pollutant Discharge Elimination System (NPDES), and,
  - Water Quality Standards

The NPDWR and NSDWR protect public health by limiting contaminants in drinking water sources. An assigned task for this project asks that the system’s water output meet the NSDWR standard of TDS levels at or below 500 mg/L.

All aspects of CWA should be followed. As the output stream of treated water would ideally be discharged back into the environment, the output should be regularly tested to make sure contaminant levels are, at minimum, fulfilling safety standards as set forth in NPDES.

Additionally, injection wells are monitored under some guidelines of the SDWA which requires Congress be informed of waste disposal practices to ensure the safety and quality of underground sources of drinking water are maintained<sup>21</sup>. Under the SDWA falls Underground Injection Control (UIC), which provides standards for injection well sites.

Additional permitting regarding stormwater management construction would be required.

In addition to federal guidelines, there are even industry niche standards set forth in the regions of interest – Wyoming and Texas. Most oil and gas field waste management is regulated at the state level. Regulatory authorities for produced water disposal in the target states are shown in the following tables<sup>22</sup>:

*Table 12: State Regulatory Boards*

State	Underground Injection Control	Land Application	Water Discharge via NPDES	Recycling
Texas	TRRC	TRRC	USEPA	TRRC
Wyoming	WOGCC	WOGCC	WDEQ	WDEQ

Table 13: Regulatory Board Acronyms

Acronym	Agency
TRRC	Railroad Commission
USEPA	United States Environmental Protection Agency
WOGCC	Wyoming Oil and Gas Conservation Commission
WDEQ	Wyoming Department of Environmental Quality Agency Specific Provisions

Similar to federal regulations, the state codes govern disposal of produced water to protect water quality, ecosystems, and local municipalities. If water is intended to be drinkable instead of injectable, many other regulations and concerns have to be considered.

Construction work required to build and maintain the system should follow all industry standards and guidelines to ensure worker safety.

Using the EnvisionV3 Pre-Assessment Checklist from the Institute for Sustainable Infrastructure. Envision uses a rating system as a way to implement sustainability into building projects, allowing them to meet sustainability goals and gain public recognition<sup>23</sup>. A detailed sustainability analysis is set forth below.

Table 14: Sustainability Analysis

	Credit Assessment Status	Evaluation Questions Assessed		Assessment Status						Assessed Maximum Points Available	Total Maximum Points
		Yes	No	Improved	Enhanced	Superior	Conserving	Restorative	Points		
Total Points:	All Credits Assessed	142	76	43	64	67	119	72	365	856	1000

Possible Award Level:	Gold
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Without use of the water purification and REE collection system detailed in this report, the conventional or fracturing well site would obtain only an Award level of bronze. This would be mainly due to the industry’s high safety, leadership, and allocation levels. The system adds significant points to the natural world and resource allocation sections, allowing the site to be upgraded to gold status.

The increase in points for the scaled up design are mainly due to significant increases in resource allocation and increased natural environment protection measures.

**Risk and Uncertainty Considerations**

There are many variables affecting both the system and the oil and natural gas industry that introduce increased risks and uncertainties into the project.

Associated risks include heat variation within the thermal desalination unit – it could potentially overheat and create a fire hazard.

Several uncertainties exist within the system. The composition of the influent is going to be inconsistent and certain compounds have the potential to clog the resin, desalination unit, or otherwise yield the system ineffective. Certain regulated compounds could potentially not filter out of solution, causing the effluent to have levels beyond what EPA and state regulation allow.

Economical risks exist due to fluctuating nature of the oil and natural gas industries. As oil and natural gas prices rapidly rise and fall, the value or necessity of reusing produced waters could decrease overnight, resulting in the system becoming economically unfeasible. The relatively stable value of the rare earth elements reduces this risk. The Environmental risks associated with drought are another economical concern for the oil and natural gas industries. Water becomes increasingly important and the cost increased, which would cause the demand for a water reuse system to increase.

Along with fluctuating oil and gas price, the market and availability for rare earth elements fluctuates. Regulatory, economic, environmental, and political situations limit the consistency of REE availability and demand, making REE prices unpredictable and unstable. This could cause the demand the system to fluctuate, with that the profit margins and sales of the unit.

The following chart show the prices and predictions for two of the projects rare earth elements. Europium data was unavailable<sup>17</sup>:

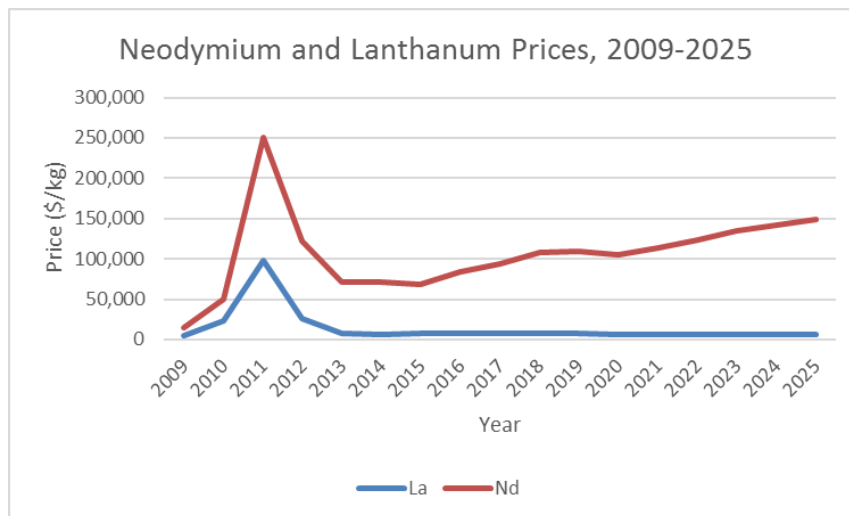


Figure 13: REE Price Prediction

A final risk lies in the limited prospects for clientele. The technology is niche and can only be used on produced containing certain REEs. There may not be a large enough client base to make the technology viable.

## Community Relations Plan

The project task requires that produced water be treated to levels that allow them to be released back into the environment. Untreated produced waters are hazardous to natural water streams and ecosystems. State and local municipalities may be understandably concerned about the prospect hazardous materials being discharged into their communities.

All state and local permits and regulations will be obtained and adhered to with documentation of such being made available to the public at all times. All site personnel

will be properly trained. An emergency operations plan is assessed and as many possible situations are accounted and planned as necessary.

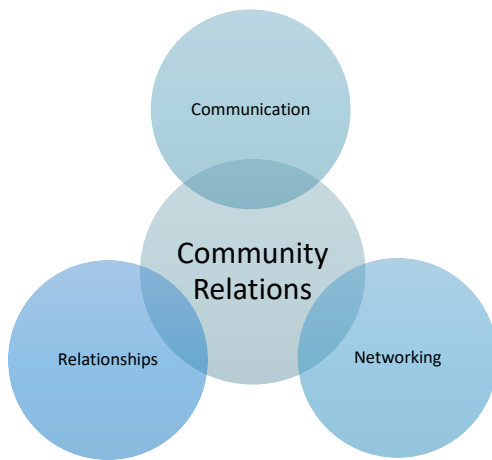


Figure 14: Community Relations Plan

To alleviate these concerns, trusted public leaders will be approached before the system is utilized. They will be given documentation for proof of design as well as testing data and results. Officials are encouraged to maintain an open and transparent line of communication where all qualms and concerns can be voiced. If possible, a thorough presentation will be presented to the public where they are encouraged to ask questions and make recommendations. All

final equipment use plans will be reviewed by town officials. Any and all voiced questions, comments, and concerns will be acknowledged and analyzed.

## Public Involvement Plan

Public representatives will be given a thorough outline of all project plans and schedules, system processes, and planned utilization, as well as access to any further requested information on implemented equipment and systems. They will also be granted limited site access and outside auditing will be implemented at the public's request.

Contact will first be made in writing. Representatives will be given the schedules, details, and preliminary site information, as well as their designated person of contact for the duration of the project. Any questions and concerns can then be sent back, and an in-person meeting can be scheduled if the public representatives wish. Any concerns will be addressed as promptly. Compromises and decisions will be reached based on relevant legislation, prior relevant court proceedings, and economic, environmental, cultural, and political feasibility. Public forums would ideally be conducted semiannually to reassess and present project updates and system implications.

Public representatives of the relevant town, counties, and states should elect a person of contact. All communication and proceedings between company and public should be made through the parties' respective contact before proceeding forward. Contacts should at a minimum provide a telephone number and email address that they can be reached at during work hours.

In addition to all non-proprietary system and operational information being publicly available online, informational pamphlets can be provided at request. The company can host a public meeting if the public wishes to have their questions and concerns publicly addressed.

The system's output will be tested regularly a pre-determined increment, considering state suggestions and community input. These reports will be directed to the community representative via the company's representative. If either party abuses or neglects their right to contact, contracts written previously to project initiation allow for court subpoenas.

Due to the necessity of a well-functioning public relations plan, an added salary must be factored in for a full-time communications and public affairs employee.

According to multiple job recruiting sites, monetary values tabulate out to:

*Table 15: Public Relations Pricing*

<b>Expense</b>	<b>Cost/Unit</b>	<b>No. of Units Used</b>	<b>Estimated Expense</b>
Public Relations Specialist <sup>24</sup>	\$60,000/year	2 weeks	\$2,307.69

A typical schedule for community contact and involvement is as follows:

Gantt Chart displays the communication and involvement plan for all governments potentially effected by use of system.

Plan Duration

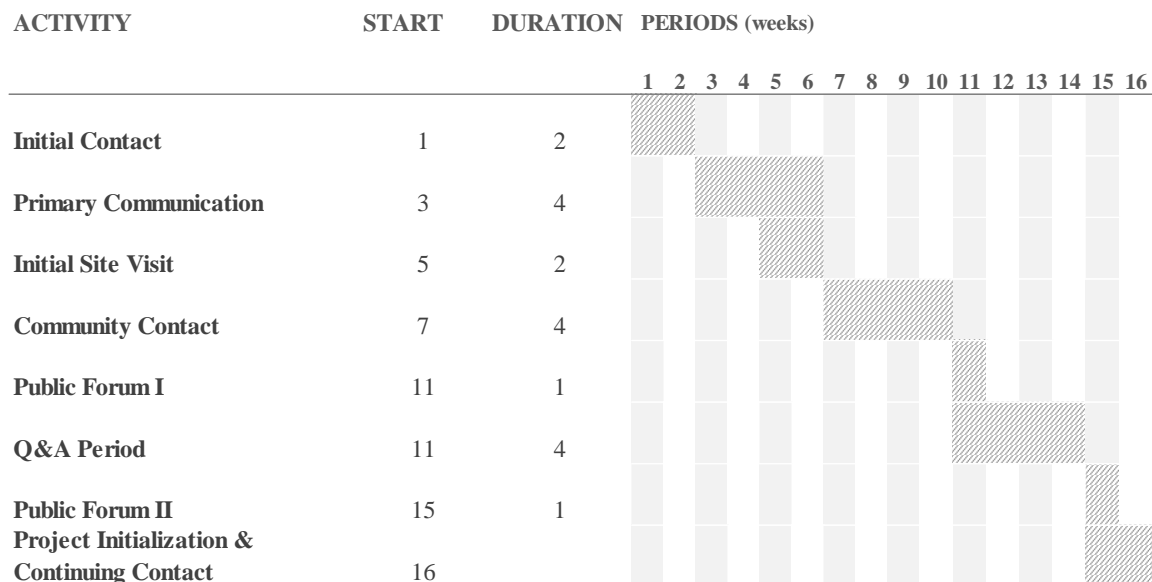


Figure 15: Community Contact Plan

## CONCLUSIONS

REEs are becoming increasingly more important, valuable, and rare. Current reliance of foreign powers and the necessity of REEs are an increasing concern for national security. As mining ore sources are depleted, it's important we find ways to conserve and reuse REEs. With high concentrations of REEs found in most oil basins, REE recovery and water cleaning systems offers potentially lucrative and beneficial technologies.

The system described in this report uses an ion-exchange column with a high capacity IDA resin, ion exchange columns, and a solar thermal desalination unit. It retrieves and separates the REEs and then purifies the water to TDS levels at or below required EPA standards. The industry employing the technology not only potentially decreases their waste disposal costs by cleaning the produced water to levels allowing it to be injected back into the environment, but also stands to make large profits on the sale of the retrieved REEs, potentially drastically increasing profit margins.

Further testing and experimentation need to be conducted on the ion exchange column to determine the relative affinities of ions and the time at which they need to pull out of the column during the backwashing process. Furthermore, with more testing, the maximum capacity at which the system is capable of operating could be increased. Optimizing efficiencies in these areas would allow the size and weight of the system to be decreased and would optimize the resale value of the recovered REEs. These proposed improvements, properly researched and optimized, have the potential to yield a system that is both profitable and sustainable.

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## AUDITS

### Legal Issues Audit

Auditor: Shellie R. Chard, Director of Water Quality Division, Oklahoma Department of Environmental (shellie.chard@deq.ok.gov)

Overarching comments:

- Might explain:
  - why synthetic produced water versus actual produced water
  - how you “know” the produced water quality from WY and TX
  - explain which producing basin in the TX and WY
  - explain WY is a coal bed methane formation which is very different from other types of formations and is more easily treatable
- Need to address (maybe just a sentence or two) the potential cost and regulatory requirements of disposing of by products
  - Wastewater – POTW through pretreatment requirements, direct discharge, industrial surface impoundment
  - Solids – landfill (hazardous wastewater landfill versus typical solid waste landfill)

Introduction comments:

- Minor grammatical corrections: “In between the two plates is a chamber that contains a ceramic cylinder coated with nanosprings in which ~~that~~ the solution is gravity fed ~~into~~.”

Alternative Analysis

- In this sentence “Another appeal to these systems is that they are already used in oil fields....” Explain what is meant in “oil fields.” Do you mean in oil and gas operations or specifically at an exploration and production site? This is an important distinction because location will trigger different regulations

Bench Scale/Prototype

- Backwashing and disposal will be a key cost and important to address from a regulatory perspective
  - can it go to a sanitary sewer system?
  - Will a direct discharge permit be needed (will take a minimum of 6 months to obtain)
  - What are the chemical constituents?

Full Scale

- Figure 6. You show “clean water” as an output. That term means different things to different people. If it is potable that should be the term. If it is low TDS water, perhaps that is a better identifier.
- The term “cleaned” water is used. I think “treated” water is more appropriate
- Edit the following sentence “Once the water exits the condensing unit, the cleaned water exits the system, where is it may or may not need to be chlorinated to reach allowable discharge levels.” You might consider “ Once the water exits the condensing unit, the treated water may need to received disinfection prior to discharging into a surface water body.” You may also want to reference more than just disinfection. Something like “...the treated water may need additional treatment to meet Water Quality Standards promulgated by the State or EPA prior to discharge. Additionally, any Effluent Limitation Guidelines under 40 CFR 435 must be met.”
- Need to address the limitation of 2,000 gallons. The volume of produced water is well over 1T gallons/year.
- You may want to talk about how and when market saturation may occur which will limit how aggressively this method is implemented

#### Waste Generation

- Throughout the paper you address salts which are the majority of the total dissolved solids. In this section you talk about total suspended solids as one of you waste streams. I think it is TDS
- Table 7 – you could have some landfill disposal depending on liquid content. You could also have some hazardous waste when using actual produced water versus synthetic due to concentrating contaminants including naturally occurring radionuclides (NORM)
- I have never heard of a sludge injection well (Class II UIC wells are the oil and gas related wells; Class I is the industrial)

#### Health, Safety and Environmental Regulations

- Safe Drinking Water Act (SDWA)
  - Underground Injection Control (UIC)
  - National Primary Drinking Water Regulations (NPDWR) – These are referred to as Primary Standards
  - National Secondary Drinking Water Regulations (NSDWR) – These are referred to Secondary Standards
- Clean Water Act
  - National Pollutant Discharge Elimination System (NPDES)
  - Water Quality Standards
- For drinking water it is TDS and turbidity not TSS

- Treated water would be discharged into the environment not injected into the environment. It could be injected into a formation. Testing would be done in accordance with permits
- Additional construction requirements include storm water management permits

#### Community Relations Plan

- Communities are typically more concerned with discharges rather than injection, except when in an area with induced seismicity
- Want to include general public, trusted leaders, local and state government
- May want to do community outreach during the public meetings or other public participation requirements of environmental permits or authorizations.

## **Economic Audit – World Wide Water, Oklahoma State University**

Date: March 19, 2020

Auditor: Jared Boehs, HyQ Technologies, LLC (jared@hyqwater.com)

First, I commend Oklahoma State University's 'World Wide Water' team for their efforts, thoroughness and due diligence in completing their report. For simplicity of presentation and ease of review, I am outlining the conclusion of my economic audit in bullet list form. Below are my comments, recommendations and questions to the team.

### General:

1. In the team report, TSS removal is discussed quite regularly, rather TDS removal as instructed by the Problem Statement. Ensure correct terminology is used throughout the report.
2. In general and regarding the financial analysis, does the team foresee the ion exchange column and thermal desalination unit performing with the same level of efficiency and removal success across any produced water stream encountered, regardless of salinity?
3. The team will need to prove their solution is an oilfield conscious option that can be easily adopted by E&P companies.
4. Can the system be scaled in size? If so, what is the estimated commercial volume range?
5. What is the site footprint of the system? Will the size be burdensome to the oil and gas operator?
6. Can the system operate in all ambient temperatures experienced in the oilfield?
7. Is the system electric driven or natural gas driven? Have those variable input costs been accounted for in the financial analysis? If so, describe those assumptions.
8. Have transportation logistics of the byproducts been accounted for in the business plan? Are the REEs easily sold, are markets easily accessible and in regions where oil and gas is produced and produced water is present? Are special means of transportation required?
9. If the team intends to operate in a 24/7 operation, solar power will not power the system through the night or cloudy/rainy days. In addition to solar, I recommend the team consider a flexible, hybrid energy system that could include multiple sources of energy, depending on resources available in a given region, including line power, wind, flare gas, diesel powered generators, and so forth.
10. Has downtime for maintenance and cleaning/backwash cycles been considered and accounted for in annual revenue projections? Additionally, has consideration been taken for storage of produced water during downtime? I.e. redundancy, storage tanks etc.
11. As illustrated in the reclamation table, is it safe to assume the team will recover the exact same volume of each REE (Europium, Neodymium and Lanthanum) annually? Worst case scenarios should also be considered.
12. Recommend providing graphs of the historical commodity prices for the REEs over time, illustrating their price consistency. Graphs depicting demand for the REEs would also be helpful in de-risking the use of the team's solution.
13. What is the useful life of the equipment? What do yearly repair costs look like?
14. Recommend describing the build, mobilization and demobilization time of the system.

## Financial:

1. What are the operating costs per feed gallon, or barrel, for operating the total process? Illustrate the individual price per gallon costs to operate the ion exchange column and thermal desalination unit. Next, for business planning, convert to cost per barrel and outline the price per barrel to be charged to customers in the market.
2. Recommend the team complete a chart illustrating both costs and revenues in a per barrel format (bbl = 42 gallons) for ease of analysis by potential industry customers. Potential customers will want to see if the offering provides cost savings to their current, traditional means of disposal. Customers will then decide to utilize the team's system or direct their produced water straight to disposal.
3. Recommend providing analysis for return on investment.
4. Ensure all input costs have been accounted for. The 25% contingency seems high and could raise questions about level of detail in estimate.
5. The trailer cost is nearly 40% of your mobile unit capital cost to build. Recommend locating a used, more economical option.
6. The water heater "booster" is 3x the cost of the tankless water heater, which appears to be the primary heat source. As discussed in bullet 9 above, is there a more efficient heat source available?
7. Are there cost savings in building a fixed "brick and mortar" structure in conjunction with a produced water gathering system, rather than a mobile unit?
8. Recommend clarifying input cost assumptions for maintenance and waste disposal. Is waste disposal by barrel or by gallon? Must the waste be trucked? Where is it disposed? Maintenance of the system (\$624,000) is nearly 2.4 times greater than the projected CAPEX cost. This seems high; therefore, further clarification of assumptions should be considered.
9. Does the 'cost of transportation' in the team's Operating Cost table account for the storage, transportation, logistics and so forth associated with capturing, storing and transporting the REEs to market?
10. The "Price of Mobile Treatment Unit" table shows projected cost. Is this actual manufactured cost or sell price? Are all manufacturing/assembly costs, overhead and profit included in the mobile unit cost?
11. The revenue table for REEs currently depicts 'Annual Revenue' from each REE, not profit. Profit is a net figure after all costs (fixed, variable, labor, cost of capital, etc.) have been accounted for. Recommend including an additional chart depicting revenue, expenses, EBITDA and profit.
12. What are the worst case economics in a soft market? Include minimum sale price that the process can operate at and still be profitable.
13. Regarding commercial disposal fees, per barrel, fees in the Permian Basin and Oklahoma at a commercial disposal well will likely range from \$0.50 to \$0.75 per barrel. In Wyoming, commercial disposal fees are likely closer to \$2.50 per barrel, excluding trucking.
14. All figures in your report should have a consistent look, regarding dollar signs, commas, etc. Rather than a mix of both.
15. Round all figures to whole dollars, no cents.

Thank you for the opportunity to review the report. Please follow-up with any questions, as I am happy to further discuss any of the above comments or the industry in general. Good work and all the best with this endeavor.

## Health Issues Audit

Auditor: Michael Taylor, Cowan Group Engineering, LLC (michael@cowangroup.co)

### REEs Report Review

- Health and safety
  - While removing TSS and REEs are there other elements of concern. May need to discuss the entire MCL list from EPA. Typically, oil field industry uses many chemicals in the produces water to acquire the intended results of fracking and the produced water typically has solvents and gasoline/oil byproducts. Could these items foul the system or does the pre-filtration typically take care of them?
  - Protection from explosion will be important in the full scale unit.
  - Are by-products such as TTHMs expected from the use of chlorine?
  - Is the intent to remove TDS or TSS?
  - Discuss general chlorine and HCL use safety. Maybe reference the mds sheets for them. Eye protection and gloves for liquid solution.
  - It might be useful to provide the table for the secondary mcl standards and discuss whether you anticipate any of them being an issue.
  - Is the produced water ever intended to be drinkable or just put back into a stream or injection well? If drinkable, all NSF requirements should be met for the materials used or at least discuss the intent of acquiring NSF certification for full scale. If just into a stream a discharge permit would most likely be required from the state as eluded to in the report.
  
- General thoughts:
  - Should discuss the amount of produced water that one oil well site produces. It is important to discuss the size of scale that full-scale unit would need to be and if it is feasible. Produced water at an oil field site is typically one to one or even more. With that said it is possible that for every barrel of oil produced one barrel of water is produced.
  - What kind of energy costs are anticipated at full scale? Will solar provide enough energy?
  
- Grammar and formatting recommendations:
  - Page 6 third paragraph should read – As shown in Figure 1.....
  - Page 6 ALTERNATIVE ANALYSIS heading needs moved to left side of page
  - Page 9 second paragraph of Thermal Desalination should read – 7,500 times more surface area than without the nanospring surface.
  - Page 10 references page 11 rather than 12 for “Full-Scale Design Description”
  - Page 10 move Table 1 to page 11
  - Page 14 market and sales/savings sentence two needs revised/reordered.
  - Page 16 move header for recurring waste expenses to next page.
  - Page 17 move table 9 to page 18 to prevent cut off.
  - Page 22 paragraph four sentence three uses the word wither which should be either.