

# Form B: IGEM-HERC Full Proposal Cover Sheet

Idaho State Board of Education

PROPOSAL NUMBER: TOTAL AMOUNT REQUESTED: \$140,000

Proposal Track (select one): Proof of Concept

TITLE OF PROPOSED PROJECT: Recovery of Critical Materials from E-Waste

SPECIFIC PROJECT FOCUS: The proposed research focuses on recovering rare earth elements (REEs) from electrical and electronic waste (e-waste). A novel anhydrous three-step treatment process is proposed to be developed for transforming the e-waste into a resource. Specific objectives for the project are: 1. Demonstration of the technical feasibility of process steps, 2. Optimization of the operating parameters, and, 3. Preliminary techno-economic and environmental life cycle analysis of the process.

We envision this project as a seed for future research and development activities, ultimately establishing Idaho as a leader in tackling national and global challenges in the critical materials resource recovery and environmental remediation arenas.


PROJECT START DATE: July 1, 2024 PROJECT END DATE: June 30, 2025

NAME OF INSTITUTION: University of Idaho DEPARTMENT: Chemical and Biological Engineering

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NAME: TITLE: SIGNATURE:

PROJECT DIRECTOR/PRINCIPAL INVESTIGATOR	Vivek Utgikar	Professor	
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CO-PRINCIPAL INVESTIGATOR	Krishnan Raja	Professor	
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NAME OF PARTNERING COMPANY: N/A COMPANY REPRESENTATIVE NAME:

SIGNATURE:

Authorized Organizational Representative

NAME: Heather Clark, Assistant Director, Sponsored Accounting

tc 3/12/24

SIGNATURE:



Digitally signed by Heather Clark  
DN: cn=Heather Clark, o=University of Idaho, ou=Office of Sponsored Programs, email=heatherclark@uidaho.edu, c=US  
Date: 2024.03.12 11:23:32 -07'00'

# Proposal Narrative

<b><u>Idaho Public Institution</u></b>	University of Idaho (UI)
<b><u>Project Title</u></b>	Recovery of Critical Materials from E-Waste
<b><u>Principal Investigator</u></b>	Vivek P. Utgikar, Professor, Chemical and Biological Engineering, University of Idaho, Moscow, ID-83844 ( <a href="mailto:vutgikar@uidaho.edu">vutgikar@uidaho.edu</a> )
<b><u>Key Project Team Member</u></b> (Co-Principal Investigator)	Krishnan S Raja, Professor, Nuclear Engineering and Industrial Management, University of Idaho, Idaho Falls, ID-83402 ( <a href="mailto:ksraja@uidaho.edu">ksraja@uidaho.edu</a> )

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## **Project Objectives**

The proposed research focuses on recovering rare earth elements (REEs) from electrical and electronic waste (e-waste). REEs are deemed critical materials by the U.S. due to their importance to the economy and the risk of disrupting their supply. REEs are indispensable to practically every product or device used by modern society, and an interruption in their supply will greatly diminish the living standards of communities. A novel anhydrous three-step treatment process that combines elements of different feasible technologies is proposed to be developed for transforming the e-waste stream into a resource.

The specific objectives for the project are to:

1. Demonstrate the technical feasibility of the steps of the proposed process through experimental investigations;
2. Optimize the operating parameters for the first step of the process; and,
3. Conduct preliminary techno-economic and environmental life cycle analysis of the process.

We envision this project as a seed for future research and development activities, ultimately establishing Idaho as a leader in tackling national and global challenges in the critical materials resource recovery and environmental remediation arenas.

**Total Amount Requested:** \$140,000

## **Resource Commitment**

The PI and the co-PI have the necessary equipment (autoclaves, furnaces, controlled environment chambers, electrochemical instruments) in their laboratories to perform the experiments. Both the Moscow and Idaho Falls campuses of the University of Idaho campuses have the needed analytical and computational facilities to support the proposed research. Details of these facilities

are provided in Appendix A. The chemical reagents and other supplies needed for the project will be procured from the vendors as needed.

## **Specific Project Plan and Timeline**

### **Introduction**

Rare earth elements (REEs) are integral constituents of components in energy systems – electric vehicles, wind turbines, photovoltaic thin films, and high-tech products – hard disk drives, television screens, magnets, optics, and many others<sup>1</sup>. Technological advances in these sectors have caused explosive growth in consumer and industrial products that utilize REEs worldwide. The supply of these materials continues to be dominated by a single country – China. The near monopoly enjoyed by China allows it to control the global supply and manipulate prices of REEs, as reflected in the significant spike in the rare earth oxide (REO) prices in 2011<sup>1</sup>. The World Trade Organization (WTO), mediating in the trade dispute brought forth by the consumer nations, ruled against China, and the relaxation of supply resulted in a precipitous price drop<sup>2</sup>. Such dramatic price

fluctuations make the economies of the majority of countries vulnerable to the policies and actions of the sole supplier. These materials are at a high risk of supply disruption that will have severe consequences on the economy and are designated as critical materials, as shown in Figure 1<sup>3</sup>.

At the same time, the rapidity of technological advances has also hastened the obsolescence of these products, creating a challenge for managing the electrical and electronic products discarded by consumers. The global generation of such e-waste (also referred to as waste electric and electronic equipment – WEEE), estimated to be more than 52 million metric tons in 2021, i.e., over 6 kg per person, is predicted to rise to as much as 75 million metric tons by 2030<sup>4,5</sup>. The complexity of the physical and chemical characteristics of e-waste makes its management exceptionally challenging. Along with the valuable materials, it also contains a vast number of toxic and hazardous substances that have the potential for severe environmental and human health impacts. Improper disposal practices, as well as incomplete and low-cost primitive recycling approaches, have facilitated the release of many harmful chemicals into the environment, creating an urgency for developing effective recovery and disposal techniques<sup>6</sup>.

The current inventory of unrecycled e-waste is nearly 350 million metric tons, and less than 20% of the e-waste is properly collected and/or recycled<sup>5</sup>. There is a growing realization that the voluminous e-waste can serve as an anthropogenic mine for these critical resources<sup>7</sup>. Recovery

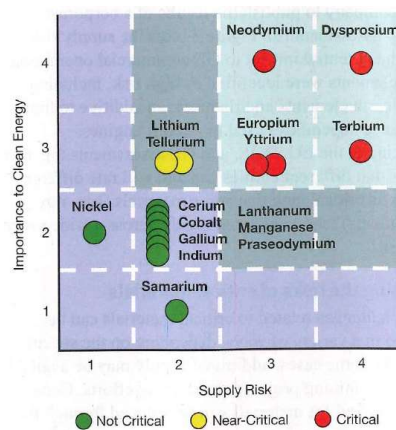


Figure 1. Categorization of materials based on significance and supply risk

of materials from e-waste can contribute to alleviating primary supply limitations while at the same time minimizing and mitigating environmental impacts. Proposed research thus contributes to both remediation of the environment and reducing the vulnerability of the economy to potential supply disruptions.

#### Limitations of the Current Technology

Conventionally, aqueous processes have been employed or investigated for recovering these materials. The aqueous or hydrometallurgical processes typically proceed via the dissolution of metals into concentrated acids followed by the removal of dissolved iron as iron (III) hydroxide using a strong alkali<sup>8</sup>. A solvent extraction step separates REEs, which are then precipitated as oxalates. Further separation of individual REE requires molten salt electrolysis, for which the oxalates need to be converted to oxides through calcination<sup>9</sup>. The large number of processing steps involving many different reagents results in the generation of large quantities of wastewater and other streams with varying toxicity characteristics, creating a significant environmental hazard. Managing these secondary waste streams responsibly to protect the environment substantially burdens the process, reducing its economic viability<sup>10</sup>. Pyrometallurgical processes include high-temperature (up to 1500 °C) molten salt or liquid metal extraction, gas phase extraction, or electrochemical processing<sup>10</sup>. While energy intensive, these processes involve far fewer processing steps that involve mostly non-aqueous reagents, avert problems of wastewater generation are averted<sup>9</sup>. Concentrations of target species in process streams are also higher, making the separations thermodynamically and kinetically more favorable than those in hydrometallurgical processes. However, the high temperatures, severe processing conditions, and difficult-to-handle reagents are drawbacks of these alternatives.

An innovative process will be investigated in this research to recover neodymium (Nd) from iron-neodymium-boron ( $\text{Fe}_{12}\text{Nd}_2\text{B}$ ) magnets that are ubiquitous in e-products and e-waste.

#### Overview and Novelty of the Proposed Process

The innovative three-step pyrometallurgical process will involve 1) solid-state ammonium chloride mediated chlorination forming metal chlorides, 2) reduction of ferrous chloride ( $\text{FeCl}_2$ ) to elemental iron using molten magnesium, and 3) electroreduction of Nd in an electrorefiner using a LiCl-KCl based chloride molten salt. Volatile  $\text{BCl}_3$  formed in the solid state chlorination will be trapped and converted to boric acid, adding further value to the process.

Among the advantages of the overall process are lower consumption and use of less expensive reagents, lower operating temperatures, better control, and effective separations resulting in high-purity products while averting the generation of challenging waste streams. The novelty of the process arises from leveraging favorable thermodynamics for selective reductions and separations that also exhibit faster kinetics than alternative processes<sup>9</sup>. Our preliminary experimental work has focused on the first step of the process, where we have varied operating

conditions and successfully converted the metals into their chlorides. Additional details of the experimentation are described later in the proposal.

#### Relevance to Idaho HERC Strategic Plan and Mission

Idaho's higher education research strategic plan envisions Idaho's institutions continuing to build a vibrant higher education research ecosystem to catalyze the creation of knowledge, technologies, products, and industries, leading to increased student success, advances and opportunities for economic growth, and enhanced quality of life in Idaho and beyond. Research is the foundation for innovation and application that provides wider benefits across the state, including economic growth and workforce quality improvement, leading to productivity enhancement and, ultimately, improved quality of life for the state and beyond.

HERC has identified five key areas where Idaho's institutions have emerging strengths. The proposed research is closely aligned primarily with energy, sustainability, and resilience. As mentioned earlier, REEs are integral to energy systems, particularly renewable energy systems, that will continue to gain ever-increasing prominence in the future. Reliance on uncertain and potentially hostile foreign entities for critical components can have devastating consequences, and the proposed research promises an alternative for mitigating such a situation. The research also serves the objective of resource conservation and recovery and improving the environment and health of human beings and ecosystems.

The proposed project will also train graduate and undergraduate students, providing them an opportunity for learning and professional development through participation in research and scholarly activities at the University of Idaho. It will also build the research capabilities at the institution, positioning it to be competitive in attracting research funding from federal and non-federal sources, including industry.

#### RESEARCH & DEVELOPMENT PLAN

##### Process Description

The innovative process consists of three steps: The first step of the process consists of converting the metals present in the magnet into chlorides for further separation and REE recovery. The chlorination will be effected by hydrogen chloride gas generated through the decomposition of ammonium chloride. This process, termed solid-state chlorination, is superior to other chlorination alternatives, such as direct chlorination using chlorine gas, as it is conducted at much lower temperatures (~300°C as compared to 800-1000°C), minimizes the hazards associated with the handling of high-pressure chlorine gas, and affords much better control of the reaction. Solid-state chlorination is also more cost-effective than chlorination using concentrated aqueous hydrochloric acid, which additionally creates a toxic wastewater stream. Also, boron (B) present in the magnet will be removed from the system as gaseous boron trichloride (BCl<sub>3</sub>), leaving behind a solid residue consisting of chlorides of Fe, Nd, and other trace REEs such as dysprosium

(Dy) present in the magnet.

The second step of the process involves reduction of iron from its chloride form back to the metallic form by contacting the residue with molten magnesium (Mg) at ~650-700°C. The thermodynamics of the reactions indicate that only iron chloride will undergo selective reduction, with REE chlorides remaining inert to Mg. At the end of this second step, iron will be separated from the melt of REE- and Mg- chlorides. As with the first step, this step is superior to other processing alternatives that require much higher temperatures and longer processing times.

The final step of this process involves the electro-reduction of REEs – reduction of rare earth (RE) chlorides into metallic form using electric current. As mentioned in step 2, RE chlorides cannot be reduced to RE metals simply by using heat (high temperature) and chemical agents (Mg). This reduction also requires the input of work, which is accomplished through the input of electrical energy. Chlorides formed in step 2 of this process will be dissolved in a molten chloride salt medium (lithium chloride-potassium chloride or lithium chloride-magnesium chloride mixture), and an electric current will be passed through the mixture. This electrowinning will be conducted at ~500 °C, and Nd will be recovered at the cathode. Chloride salt media are superior to fluoride salts for electrorefining and electrowinning of REEs (recovery of REEs in purified form using electrical energy) due to lower toxicity and processing temperatures, and less demanding requirements for materials of construction for process equipment.

### Research Objectives

The specific research objectives for the project are to:

1. Demonstrate the technical feasibility of each step of the proposed project through experimental investigations,
2. Optimize the operating parameters for the first step of the process and
3. Conduct preliminary techno-economic and environmental life cycle analysis of the process.

### Prior Work

Several experimental runs were conducted on solid-state chlorination of the permanent magnets. The magnets in the disc form (30 mm dia. X 1.5 mm thickness) were first demagnetized by heating them to temperatures ranging from 200 - 250 °C, and holding them at that temperature for 15-30 minutes. The demagnetized discs were then treated in a Planetary Ball Mill (Model PQ-N2, Accessory Corporation, New Jersey) for size reduction. The resulting coarsely ground powder was contacted with solid NH<sub>4</sub>Cl in an autoclave and heated to temperatures ranging from 325 – 350 °C. The percentage excess of the chlorinating agent, the particle size, and the reaction time were varied in the experiments. It was found that the chlorination step was successful in converting the elements into their chlorides. The conversion and yield increased with excess chlorinating agent (NH<sub>4</sub>Cl) and the reaction time, as shown below in the accompanying table. The conversion and yield also increased with a decrease in the particle size.

Thermodynamic analysis of the metal chloride reduction using Mg metal indicates that reductions of iron chlorides are highly favorable with the Gibbs energy changes of nearly -300 kJ/mol for FeCl<sub>2</sub> and -550 kJ/mol for FeCl<sub>3</sub>. Reductions of Nd (other REE chlorides), on the other hand are thermodynamically unfavorable with positive Gibbs energy changes.

Condition of Magnet Powder	% Excess of NH <sub>4</sub> Cl	Reaction Time, h	Conversion range, %
Coarsely ground	10	2	15-25
Coarsely ground	25	4	40-45
Coarsely ground	25	6	68-75
Finely ground (<150 μm)	25	2	>90

These transformations will be experimentally studied to prove the feasibility of the second step.

### Research Activities and Tasks

The following tasks are defined for achieving the project goal and objectives:

*Task 1 – Development of Process Flow Sheet:* We will develop a detailed flow sheet for the overall process, followed by material balances on each step to quantify the materials flows of all significant species, including those mentioned above. We will identify potential treatment schemes for managing the secondary streams to ensure that the proposed recovery process will not result in pollution of the environment. We will complete theoretical thermodynamic analyses of the processing steps and finalize the experimental design for each process step.

*Task 2 – Experimental Investigations of the Individual Process Steps:* We will conduct experimental investigations on all three steps of the proposed innovative process. For step 2 (reduction of chlorides of iron and other non REEs) and step 3 (electrorefining of REEs), the focus will primarily be on demonstrating the feasibility of the processing step. The bench-scale experimentation will be conducted with surrogate materials representing the products of the reactions of the previous step. The extent of conversion of the reactants will be used to assess the feasibility of the processing step. For the first step (solid-state chlorination), the focus will be on the optimization of reaction conditions. We will vary the following parameters in these experiments: particle size, reaction temperature, time, and the excess chlorination reagent. We will conduct detailed material and energy balances to quantify the conversion/yield and relate them to the operating conditions.

*Task 3 – Preliminary Technoeconomic Analysis (TEA) and Life Cycle Assessment (LCA):* The flow sheet developed in Task 1 will be refined based on the experimental results obtained in Task 2. This flowsheet will provide the basis for developing a scaled-up process model. the technoeconomic analysis and the LCA. This scaled-up process will serve as the basis for the technoeconomic analysis and the environmental LCA of the recovery process.

**REFERENCES:** 1. Lorenz T, Bertau M, Recycling of rare earth elements from FeNdB magnets via solid-state chlorination, *Journal of Cleaner Production*, 2019, 215: 131-43. 2. World Trade Organization, *China-*

*Measures Related to the Exportation of Rare Earths, Tungsten, and Molybdenum*, 2014. 3. Ku AY, Hung S, Manage raw material supply risks, *CEP*, 2014, 110(9): 28-35. 4. Jyothi RK, et al, Review of rare earth elements recovery from secondary resources for clean energy technologies: Grand opportunities to create wealth from waste, *Journal of Cleaner Production*, 2020, 267: 122048. 5. Ruiz A, Latest Global E-Waste Statistics And What They Tell Us, *TheRoundup.org*, 2023. [https://theroundup.org/global-e-waste-statistics/#:~:text=57.4%20Mt%20\(Million%20Metric%20Tonnes,produce%20the%20most%20e%2Dwaste](https://theroundup.org/global-e-waste-statistics/#:~:text=57.4%20Mt%20(Million%20Metric%20Tonnes,produce%20the%20most%20e%2Dwaste). Accessed October 22, 2023. 6. Ismail H, Hanafiah MM, An overview of LCA application in WEEE management: Current practices, progress and challenges, *Journal of Cleaner Production*, 2019, 232: 79-93. 7. Xavier et al, A comprehensive review of urban mining and the value recovery from e-waste materials, *Resources, Conservation & Recycling*, 2023, 190: #106840. 8. Isildar et al, Electronic waste as a secondary source of critical metals: Management and recovery technologies, *Resources, Conservation & Recycling*, 2018, 135: 296-312. 9. Hua et al, Selective extraction of rare earth elements from NdFeB scrap by molten chlorides, *ACS Sustainable Chemistry & Engineering*, 2014, 2: 2536-43. 10. Abbasalizadeh et al, Neodymium extraction using salt extraction process, *Mineral Processing and Extractive Metallurgy (Transactions of Institute of Mining and Metallurgy C)*, 2015, 124(4): 191-6.

### **Potential economic impact**

The global market value of neodymium is \$2.07 billion and is expected to expand at a compound annual growth rate (CAGR) of 15.0% from 2022 to 2030<sup>i</sup>. The market is anticipated to be driven by the increasing usage of permanent magnets in the automotive industry. The boric acid market value was \$ 1.1 billion, and the anticipated CAGR is 6.1% from 2023 – 2032<sup>ii</sup>. Boric acid is used in various applications ranging from pharmaceuticals to building materials, including in light water nuclear reactors as neutron absorbers. Recycling e-waste is an emerging market with a lot of potential for the exponential growth of the regional economy and the creation of high-paying jobs. Successful implementation of this project will help Idaho lead the efforts of the U.S.'s energy security and safety.

### **Criteria for measuring success**

Successful accomplishment of the project objectives will be based on the progress made in each of the tasks. The progress in each of tasks will be reviewed in the context of the major milestones defined under project management. Milestone 1 is related to the first task, and success in the first task/first objective will be based on closing the material balance and identifying the treatment alternatives for effluent streams of the proposed process. Milestones 2-4 relate to the experimental investigations of the three process steps, and successful demonstration of each step, as well as identification of the optimum operating conditions for step will serve as the criteria for measuring success. Completion of the TEA/LCA will serve as the measure of success for task 3 and

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<sup>i</sup><https://www.grandviewresearch.com/industry-analysis/neodymium-market-report>

<sup>ii</sup><https://www.marketresearchfuture.com/reports/boric-acid-market-7294>



attainment of milestone 5. The overall success of the project will be based on the success in reaching all the milestones. An additional metric for measuring the success of the project will be submission of at least two manuscripts to refereed journals for publication.

**The overall success of the project will be based on the integration of above metrics into the assessment of the advancement in the Technology Readiness Level (TRL) of the process from 2 (Concept Formulation) to 3 (Experimental Proof of Concept).**

**Anticipated Development Challenges/barriers**

There are two potential challenges to successful completion of tasks and accomplishment of the project objectives: 1) The experimental work in the project is building upon and extending known techniques and practices. As with any experimental work, there can be unforeseen delays in procurement of needed materials as well as component/equipment/instrument malfunction. We will design the experiments and develop flexible timeline that can accommodate such situations. 2) The tasks outlined above will be carried out by graduate students under the guidance and direction of the senior personnel. For any research project, availability or recruitment of qualified students can be asynchronous with the project timeline. A non-availability can lead to a delay in the project and compress the timeline for conducting the tasks. Cognizant of this difficulty, we will proactively engage with the current undergraduate student pool to recruit promising, motivated students at UI, as well as reach out to the other universities in the State of Idaho and the Pacific Northwest region to fill the junior personnel need.

**Budget**

The amount requested for the proof of concept is \$140,000, as shown in the following table.

LINE ITEM REQUEST	JUSTIFICATION	TOTAL REQUEST
Personnel (salary and fringe)	Support for PI, Co-PI, and 2 graduate students	\$87,800
Equipment	--	0
Travel	--	0
Participant Support	--	0
Other Direct Costs	Materials/supplies, publication, and tuition and health insurance for graduate students	\$52,200
		<b>\$140,000</b>

Details of the budget are provided in the accompanying Form D: IGEM-HERC Budget Form Spreadsheet.

**Budget justification**

The explanation of costs indicated in the budget above is as follows:

1. Personnel Costs (salary and fringe): PI Utgikar and co-PI Raja will spend 1 and 0.8 summer months working on the project. The project tasks will be executed by two graduate students. The salary and fringe bases are as shown in the attached Form D worksheet. An anticipated fringe rate is applied to this budget. These are requested to DHS CAS but not yet approved. Per the university guidance on new proposals, anticipated rates as ‘estimated fringe rates’ for projects that will begin 7/1/24 and later are applied. FY25 anticipated fringe rates are as follows: Faculty: 31.7%; Staff: 40.1%; Students: 2.0% ; Temporary:10.1%. Current FY24 fringe rates available at: <http://www.uidaho.edu/research/faculty/resources/fringe-rates> are: Faculty: 31%; Staff: 41.3%; Students: 2.5% ; Temporary: 8.3%.
2. Other direct costs include supplies cost of \$24,200 (itemized list in Form D), publication costs of \$2,600, and graduate student tuition (\$21,000) and health insurance (\$4,400) for 2 students, 2 semesters.

**Project management**

The project schedule is shown below in a Gantt Chart.

Objective/Tasks	Year 1			
	Quarter			
	1	2	3	4
<u>Task 1: Flow Sheet Development</u>	[Gantt bar spanning Q1]			
<u>Task 2: Experimentation</u>	[Gantt bar spanning Q1, Q2, Q3, Q4]			
2.1 Reaction Step 1	[Gantt bar spanning Q1, Q2, Q3, Q4]			
2.2 Reaction Step 2	[Gantt bar spanning Q1, Q2, Q3]			
2.3 Reaction Step 3	[Gantt bar spanning Q3, Q4]			
<u>Task 3: TEA/LCA Analysis</u>	[Gantt bar spanning Q4]			

The high level major milestones for the project are: 1) Milestone 1: Process Flow Sheet at the end of quarter 1; 2) Milestone 2: Parameter Optimization for process step 1 at the end of quarter 3; 3) Milestone 3: Feasibility analysis of process step 2 at the end of quarter 2; 4) Milestone 4: Feasibility analysis of process step 3 at the end of quarter 4, year 1; 5) Milestone 5: Preliminary TEA/LCA at the end of quarter 4, year 1.

PI Utgikar will serve as the overall project director and will lead activities related to the first process step, as well as flow sheet development and TEA/LCA. Co-PI Raja will lead the activities related to the second and third steps. The project activities will be conducted by two graduate students. The research group will hold biweekly meetings to review the progress and analyze the data. PI and co-PI have collaborated extensively on several research projects and developed a smooth modus operandi for successful accomplishment of project objectives.

### **Additional institutional and other sector support**

The institutional support for the project is manifest through the availability of the lab space as well as utilities and project administration support. An industrial partner is not involved in the project at the current time, due to the nature of project (proof of concept) and the low TRL of 2. However, potential industrial collaborations are identified as described below.

### **Future funding**

Several avenues including federal funding agencies and industrial partnerships are identified for securing funding for future research and technology development. Federal funding agencies/programs include: Critical Materials Innovation Hub (<https://www.ameslab.gov/cmi>), a U.S. DOE innovation hub that seeks to accelerate innovative scientific and technological solutions to develop resilient and secure supply chains for rare-earth metals and other critical materials; National Science Foundation, specifically the Division of Chemical, Bioengineering, Environmental and Transport Systems (CBET) through its programs that support fundamental science and engineering research for the development of novel materials, mechanisms, and/or tools to improve the efficiency, resource utilization, and/or intensification of chemical processes, and energy and environmental sustainability; the Office of Energy Efficiency and Renewable Energy (EERE) of the U.S. DOE which is vested in ensuring the supply of materials critical to the clean renewable energy; the Department of Defense (DoD) programs aimed at ensuring rare earth supply chain resiliency; and Advanced Research Projects Agency – Energy (ARPA-E) programs.

Several industrial companies in the U.S. are engaged in the recycling of critical materials. *Critical Materials Recycling, Inc.* (<https://criticalmaterialsrecycling.com/>) is one such company that focuses upon reclaiming REE and other critical materials from magnets and e-waste. *Idaho Strategic Resources, Inc.* (<https://idahostrategic.com/>) is another organization with significant interest in REEs. We will reach out to these and other industries during the later part of the project to develop collaborations, once we have the indication of the feasibility of the process and consequently, success in advancing the TRL from 2 to 3. We will involve UI's Office of Technology Transfer to ensure that the intellectual property issues are handled properly.

## Appendix A: Facilities and Equipment

Facilities and equipment available at the University of Idaho (UI) for conducting the research are as follows:

#### *Moscow Campus*

The facilities available at the Department of Chemical and Biological Engineering at UI include wet and dry chemistry laboratory including controlled environment chambers, fume hoods and separations equipment.

Analytical instrumentation and laboratory facilities available at UI include gas chromatographs; mass spectrometers (ICP, HPLC); optical, electron and atomic force microscopes; surface area analyzers; and microfabrication equipment. The details of the available equipment are as follows:

- Field-emission scanning electron microscopy (FE-SEM, AMRAY 1830), SEM (Carl Zeiss, Supra 35), energy dispersive spectroscopy (EDS), transmission electron microscopy (TEM), powder x-ray diffraction (XRD), single-crystal XRD.
- *Spectroscopic facility*: Raman spectrometer (i-Raman with 532 nm laser, Gamry Instruments) and in-situ UV-Vis spectroscope (Spectro 115E, 350-1050 nm, Gamry Instruments) for in-situ spectroscopic investigation during electrochemical testing.
- *Material synthesis*: Two controlled atmosphere furnaces with ramping capabilities with a maximum temperature of 1300 °C (Nabertherm, Model: RD/30/200/13) and 1700 °C (SentraTech, Model: STT-1700C), Controlled atmosphere glove box with recirculation and regeneration capacity (mBraun, Model: LABStar), Vacuum Oven: (Max: 250 °C, 1 cu.ft., 0 – 0.1 MPa acuum reading, Jeio Tech Co., Model: OV-11-120)
- *Material testing*: A thermo-mechanical analyzer (TA Instruments, model: TMA Q400), a microbalance (Radwag USA), high resolution DCPD crack length measurement system, non-destructive testing capabilities including ultrasonic flaw detector (GE Inspection Technologies, Model: USM Go), Eddy current tester, magnetic flux detector, Netzsch Simultaneous Thermal Analyzer STA-409. Several potentiostats (Gamry Instruments, Models: Reference 600 and Interface 1000, Princeton Applied Research (AMETEK), Model: VersaSTAT MC (4-channels), and CH Instruments: Model 400C Electrochemical quartz micro balance).

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- IBEST Optical Imaging Core: fluorescence (Leica MZ16F stereo), epifluorescence and transmission (Nikon Eclipse), confocal and multiphoton (Olympus Fluoview) microscopy facilities from 405-1040 nm excitation and related hardware/software w/ full-time staff.
- Mass spectrometry facilities with access to i) Waters Q-ToF Premier quadrupole time-of-flight MS equipped with electrospray ionization, nanoESI, MALDI, and Triazaic nanotile ion sources, routinely interfaced to Waters nanoAcquity UPLC or Acquity HPLC; ii) Waters Xevo TQ tandem quadrupole MS equipped with electrospray ionization, nanoESI, and Triazaic nanotile ion sources and interfaced to nanoAcquity UPLC; iii) GC-MS composed of a single quadrupole Hewlett-Packard 5973 Mass Selective Detector interfaced to model 6890 GC. Inductively coupled plasma-MS is available through reciprocal agreement with Washington State University (~7 miles away).
- A 1700 sq. ft. chemistry lab has two HP 5890A Gas Chromatographs, an HP 8453 Photodiode Array Spectrometer, a BAS CV50w Voltammograph.
- A controlled atmosphere glove box capable of maintaining < 0.2 ppm oxygen and moisture (mBraun, Model: LabStar), a microbalance (Radwag USA, Model: XA110/2X), and Millipore water purification system capable of supplying high purity water with 18.2 Megaohm-cm resistivity, several non-destructive testing capabilities including ultrasonic flaw detector (GE Inspection Technologies, Model: USM Go), Eddy current tester, magnetic flux detector.

The Department of Chemical and Biological Engineering also employs one full-time instrumentation specialist (BS ME), and one full-time, PhD-level research engineer to assist with the research. Both are expert at machining the required process equipment for experimental investigations.

*Idaho Falls Center of University of Idaho (UI)-Center for Advanced Energy Studies (CAES)*

CAES Research Facility in Idaho Falls is also accessible for conducting the research. Laboratories located in CAES include: analytical chemistry laboratory, advanced materials laboratory (AML), fluids laboratory, and radiochemistry laboratory. Microstructure and Characterization Suite (MaCS) in AML is a state-of-the-art microstructural characterization

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facility housed inside the having the following instruments: Scanning Transmission Electron Microscope (STEM) - ThermoFisher Spectra 300 - monochromated, double-corrected, 30 - 300kV; Scanning Transmission Electron Microscope - FEI Tecnai TF30-FEG STwin TEM with EDS, EELS (GIF), EFTEM & TopSpin; Dual Beam Focused Ion Beam Microscope (FIB) - FEI QUANTA 3D FEG with EDS & EBSD; Scanning Electron Microscope (SEM) - JEOL JSM 6610LV with EDS, EBSD, & CL; Local Electrode Atom Probe (LEAP) - Cameca LEAP 4000X HR; Nano Indenter Atomic Force Microscope - Hysitron TI950 TriboIndenter; X-Ray Diffractometer (XRD) - Rigaku SmartLab. In addition, instruments available at the Idaho Falls Center include Frontier FTIR Spectrometer, Mettler-Toledo Thermal Analysis System TGA 2, and Micromeritics Nanoplus-I Particle Size Analyzer.

### **Summary**

The facilities available include those needed for conducting chemical and electrochemical experimentation described in the work plan. Analytical and characterization facilities are available and accessible at both Moscow and Idaho Falls campuses of the university.

## Appendix B: Biographical Sketches



**IDENTIFYING INFORMATION:**


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 NAME: Utgikar, Vivek Prabhakar
 

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 ORCID iD: <https://orcid.org/0000-0001-9039-9091>


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 POSITION TITLE: Professor
 

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 PRIMARY ORGANIZATION AND LOCATION: University of Idaho, Moscow, Idaho, United States
 

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**Professional Preparation:**

ORGANIZATION AND LOCATION	DEGREE (if applicable)	RECEIPT DATE	FIELD OF STUDY
University of Dayton, Dayton, Ohio, United States	Postdoctoral Fellow	08/1995	Environmental Technology
University of Cincinnati, Cincinnati, Ohio, United States	Postdoctoral Fellow	07/1994	Chemical Engineering
University of Cincinnati, Cincinnati, Ohio, United States	PHD	06/1993	Chemical Engineering
University of Mumbai, Mumbai, Not Applicable, N/A, India	MENG	07/1985	Chemical Engineering
University of Mumbai, Mumbai, Not Applicable, N/A, India	BENG	06/1983	Chemical Engineering

**Appointments and Positions**

2001 - present Professor, University of Idaho, Moscow, Idaho, United States

2015 – 2020 Associate Dean of Research, University of Idaho

2014 – 2015 Interim Director, Nuclear Engineering Program, University of Idaho

2007 – 2014 Associate Professor, University of Idaho

2001 – 2007 Assistant Professor, University of Idaho

1998 – 2001 Research Associate, National Research Council, Cincinnati, Ohio, United States

1995 – 1998 Assistant Professor, University of Dayton, Dayton, Ohio, United States

1985 – 1988 Development Officer, Indian Organic Chemicals Limited, Khopoli, Not Applicable, N/A, India

## **Products**

### *Products Most Closely Related to the Proposed Project*

1. Goettsche H, Raja K, Sabharwall P, Utgikar V. Treatment of Off-Gas Emissions: Kinetics of Silver Mordenite Catalyzed Methyl Iodide Decomposition. *Chemical Engineering Journal Advances*. 2022 May; 10:100290-. Available from: <https://linkinghub.elsevier.com/retrieve/pii/S2666821122000515> DOI: 10.1016/j.cej.2022.100290
2. Balumuru C, Stanford J, Raja K, Sabharwall P, Utgikar V. Investigations into Plasma-Mediated Decomposition of Organoiodide Species as a Pretreatment for Mitigation of Radioiodine Emissions. *Industrial & Engineering Chemistry Research*. 2022 January 03; 61(1):269-278. Available from: <https://pubs.acs.org/doi/10.1021/acs.iecr.1c04049> DOI: 10.1021/acs.iecr.1c04049
3. Vaidya T, Stanford J, Rooyen N, Raja K, Utgikar V, Sabharwall P. Capture of Volatile Organic Iodine Species Using Mordenites. *Journal of Nuclear Fuel Cycle and Waste Technology (JNFCWT)*. 2023 June 30; 21(2):205-224. Available from: <https://www.jnfcwt.or.kr/journal/article.php?code=86875> DOI: 10.7733/jnfcwt.2023.016
4. Alhindawy I, Tolan D, Elshehy E, El-Said W, Abdelwahab S, Mira H, Taketsugu T, Utgikar V, El-Nahas A, Youssef A. A novel pH-Dependent sensor for recognition of strontium ions in water: A hierarchically structured mesoporous architectonics. *Talanta*. 2023 February; 253:124064. Available from: <https://linkinghub.elsevier.com/retrieve/pii/S0039914022008608> DOI: 10.1016/j.talanta.2022.124064
5. Stanford J, van Rooyen N, Vaidya T, Raja K, Sabharwall P, Utgikar V. Photodecomposition of methyl iodide as pretreatment for adsorption of radioiodine species in used nuclear fuel recycling operations. *Chemical Engineering Journal*. 2020 November; 400:125730-. Available from: <https://linkinghub.elsevier.com/retrieve/pii/S1385894720318581> DOI: 10.1016/j.cej.2020.125730

### *Other Significant Products, Whether or Not Related to the Proposed Project*

1. Shrotri A, Niphadkar P, Bokade V, Utgikar V, Nandanwar S. A short review on recent advances in porous adsorbents for separation of oxygen from atmospheric air. *Asia-Pacific Journal of Chemical Engineering*. 2023 March 04; 18(2):- . Available from: <https://onlinelibrary.wiley.com/doi/10.1002/apj.2896> DOI: 10.1002/apj.2896
2. Gupta A, Armatis P, Sabharwall P, Fronk B, Utgikar V. Energy and exergy analysis of Ca(OH)<sub>2</sub>/CaO dehydration-hydration chemical heat pump system: Effect of reaction temperature. *Journal of Energy Storage*. 2021 July; 39:102633-. Available from: <https://linkinghub.elsevier.com/retrieve/pii/S2352152X21003753> DOI: 10.1016/j.est.2021.102633
3. Gupta A, Armatis P, Sabharwall P, Fronk B, Utgikar V. Thermodynamics of Ca(OH)<sub>2</sub>/CaO reversible reaction: Refinement of reaction equilibrium and implications for operation of

chemical heat pump. *Chemical Engineering Science*. 2021 February; 230:116227-. Available from: <https://linkinghub.elsevier.com/retrieve/pii/S0009250920307594> DOI: 10.1016/j.ces.2020.116227

4. Shrestha N, Raja K, Utgikar V. Mg-RE Alloy Anode Materials for Mg-Air Battery Application. *Journal of The Electrochemical Society*. 2019 September 17; 166(14):A3139-A3153. Available from: <https://iopscience.iop.org/article/10.1149/2.0131914jes> DOI: 10.1149/2.0131914jes
5. Karlsson T, Fredrickson G, Yoo T, Vaden D, Patterson M, Utgikar V. Thermal analysis of projected molten salt compositions during FFTF and EBR-II used nuclear fuel processing. *Journal of Nuclear Materials*. 2019 July; 520:87-95. Available from: <https://linkinghub.elsevier.com/retrieve/pii/S0022311518317367> DOI: 10.1016/j.jnucmat.2019.04.016

### **Synergistic Activities**

1. **Infrastructure**: Lead PI for FY 17 DOE- Nuclear Energy University Program (DOE-NEUP) Scientific Infrastructure Grant for University of Idaho.
2. **Faculty Development**: Lead PI U.S. Nuclear Regulatory Commission (NRC) faculty development grant, 2015-2018.
3. **DOE-NEUP Research Grants**: (a) Lead PI: FY 12 Project #12-3363 “Advanced Reactors-Intermediate Heat Exchanger (IHX) Coupling: Theoretical Modeling and Experimental Validation”; FY 13 Project #13-4761 “Off-Gas Treatment: Evaluation of Nano-structured Sorbents for Selective Removal of Contaminants,”; FY18 Project #18-14998 “Novel Processes for Capture of Radioactive Iodine Species from Vessel Off-Gas Streams”; FY18 Project #18-14963 “Development of Hybrid Energy Systems: Temperature Amplification through Chemical Heat Pumps for Industrial Applications.” (b) Co-PI of FY 17 project #17-12757 “Elucidation of Electrochemical Behavior of Technetium, Tellurium and Iodine in Molten Salt Solutions.”
4. **Author**: “Fundamental Concepts and Computations in Chemical Engineering,” 2017, and “Chemical Processes in Renewable Energy Systems,” 2021, Publisher: Pearson, Boston.
5. **Member of the Technical Review Group (TRG)** advising DOE’s Idaho Cleanup Project treating sodium-bearing radioactive waste in the **Integrated Waste Treatment Unit (IWTU)**.

### **Certification:**

When the individual signs the certification on behalf of themselves, they are certifying that the information is current, accurate, and complete. This includes, but is not limited to, information related to domestic and foreign appointments and positions. Misrepresentations and/or omissions may be subject to prosecution and liability pursuant to, but not limited to, 18 U.S.C. §§ 287, 1001, 1031 and 31 U.S.C. §§ 3729-3733 and 3802.

Certified by Utgikar, Vivek Prabhakar in SciENcv on 2023-11-10 13:07:18

**IDENTIFYING INFORMATION:**


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NAME: Raja, Krishnan Selva

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ORCID iD: <https://orcid.org/0000-0003-4746-2272>

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POSITION TITLE: Professor of Nuclear Engineering

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**PRIMARY ORGANIZATION AND LOCATION:** University of Idaho, Idaho Falls, Idaho, United States

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**Professional Preparation:**

ORGANIZATION AND LOCATION	DEGREE (if applicable)	RECEIPT DATE	FIELD OF STUDY
Indian Institute of Technology, Chennai, Not Applicable, N/A, India	PHD	07/1993	Welding and Stress Corrosion Cracking Metallurgical Engineering
Indian Institute of Technology, Chennai, Not Applicable, N/A, India	MENG	02/1988	Industrial Metallurgy
College of Engineering, Guindy (Anna University), Chennai, Not Applicable, N/A, India	BENG	05/1986	Mechanical Engineering

**Appointments and Positions**

2023 - present Professor of Nuclear Engineering, University of Idaho, Idaho Falls, Idaho, United States

2021 - 2022 Professor of Materials Engineering, University of Idaho, Moscow, Idaho, United States

2016 - 2021 Associate Professor, Materials Engineering, University of Idaho, Moscow, Idaho, United States

- 2011 - 2016 Assistant Professor of Materials Engineering, University of Idaho, Moscow, Idaho, United States
- 2001 - 2011 Research Faculty - Chemical and Materials Engineering, University of Nevada, Reno, Nevada, United States
- 1997 - 2000 Research Associate, Tohoku University, Sendai, Not Applicable, N/A, Japan
- 1993 - 1997 Research Executive Engineer, Larsen & Toubro Ltd., Mumbai, Not Applicable, N/A, India

## **Products**

### *Products Most Closely Related to the Proposed Project*

1. Day B, Zillinger J, Utgikar V, Raja K. The Electrochemical Behavior of Tellurium Tetrachloride in LiCl-KCl Eutectic Molten Salt at 450 °C. Journal of The Electrochemical Society. 2021 May 18; 168(5):056514-. Available from: <https://iopscience.iop.org/article/10.1149/19457111/abfeff> DOI: 10.1149/1945-7111/abfeff
2. Shrestha N, Day B, Utgikar V, Raja K, Fredrickson G, Frank S. Electrochemistry of Iodide in LiCl-KCl Molten Salts and Chemla Effect: An Overview. ECS Transactions. 2018 May 04; 85(4):15-23. Available from: <https://iopscience.iop.org/article/10.1149/08504.0015ecst> DOI: 10.1149/08504.0015ecst
3. Misra M, Raja K, Ruppert J. Electrochemical Corrosion Behavior of Refractory Metals in LiClLi<sub>2</sub>O Molten Salt. ECS Transactions. 2010 October 01; 33(7):181-192. Available from: <https://iopscience.iop.org/article/10.1149/1.3484775> DOI: 10.1149/1.3484775
4. Misra M, Raja K, Jaques A, Baral S. Effect of Addition of Multi-Component Lanthanides to LiCl-KCl Eutectic on Thermal and Electrochemical Properties. ECS Transactions. 2010 October 01; 33(7):351-360. Available from: <https://iopscience.iop.org/article/10.1149/1.3484793> DOI: 10.1149/1.3484793
5. Brown R, Struhs E, Mirkouei A, Raja K, Reed D. Sustainability Assessment of Mixed Rare Earth Metals Production from Surface Soil in Idaho, USA. [Preprint]. 2023. DOI: 10.2139/ssrn.4605019

### *Other Significant Products, Whether or Not Related to the Proposed Project*

1. Vaidya T, Stanford J, Rooyen N, Raja K, Utgikar V, Sabharwall P. Capture of Volatile Organic Iodine Species Using Mordenites. Journal of Nuclear Fuel Cycle and Waste Technology(JNFCWT). 2023 June 30; 21(2):205-224. Available from: <https://www.jnfcwt.or.kr/journal/article.php?code=86875> DOI: 10.7733/jnfcwt.2023.016

2. Dhabarde N, Ferrer A, Tembo P, Raja K, Subramanian V. Chalcogenide-Based Complex Transition Metal Electrocatalyst for Hydrogen Peroxide Production. *Journal of The Electrochemical Society*. 2023 January 16; 170(1):016506-. Available from: <https://iopscience.iop.org/article/10.1149/1945-7111/acafa5> DOI: 10.1149/1945-7111/acafa5
3. Goettsche H, Raja K, Sabharwall P, Utgikar V. Treatment of Off-Gas Emissions: Kinetics of Silver Mordenite Catalyzed Methyl Iodide Decomposition. *Chemical Engineering Journal Advances*. 2022 May; 10:100290-. Available from: <https://linkinghub.elsevier.com/retrieve/pii/S2666821122000515> DOI: 10.1016/j.ceja.2022.100290
4. Dhabarde N, Carrillo-Ceja O, Tian S, Xiong G, Raja K, Subramanian V. Bismuth Vanadate Encapsulated with Reduced Graphene Oxide: A Nanocomposite for Optimized Photocatalytic Hydrogen Peroxide Generation. *The Journal of Physical Chemistry C*. 2021 October 26; 125(43):23669-23679. Available from: <https://pubs.acs.org/doi/10.1021/acs.jpcc.1c05315> DOI: 10.1021/acs.jpcc.1c05315
5. Shrestha N, Raja K, Utgikar V. Mg-RE Alloy Anode Materials for Mg-Air Battery Application. *Journal of The Electrochemical Society*. 2019 September 17; 166(14):A3139-A3153. Available from: <https://iopscience.iop.org/article/10.1149/2.0131914jes> DOI: 10.1149/2.0131914jes

### **Synergistic Activities**

1. Licensed Professional Engineer (Metallurgical and Materials Engineering in the state of Idaho (P16254);
2. NACE Certified Materials Selection and Design Specialist (Certification # 25861) 3. Editorial Board Member of Metallurgical and Materials Transactions A, Springer

### **Certification:**

When the individual signs the certification on behalf of themselves, they are certifying that the information is current, accurate, and complete. This includes, but is not limited to, information related to domestic and foreign appointments and positions. Misrepresentations and/or omissions may be subject to prosecution and liability pursuant to, but not limited to, 18 U.S.C. §§ 287, 1001, 1031 and 31 U.S.C. §§ 3729-3733 and 3802.

Certified by Raja, Krishnan Selva in SciENCv on 2024-03-08 14:29:59