Fiscal Year 2017/2018 Annual Report

Florida Industrial and Phosphate Research Institute

Mined Land Reclaimed with Native Vegetation for Gopher Tortoise Habitat

Approved by the Phosphate Research and Activities Board

September 28, 2018

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Phosphate Research and Activities Board Members

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Robert Fredere, Jr., The Mosaic Company, Vice-Chairman

Vishwas Sathe, Florida Department of Environmental Protection

Environmental Community Member, Awaiting Appointment

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Florida Industrial and Phosphate Research Institute

Directorial Staff

Brian K. Birky, Ph.D., Executive Director, Research Director, Public and Environmental Health

Steven G. Richardson, Ph.D., Research Director, Reclamation

Patrick Zhang, Ph.D., Research Director, Mining and Beneficiation

Vacant, Research Director, Chemical Processing

Gary Albarelli, MLS, Director of Information Programs

Executive Director's Message – Dr. Brian Birky

This report marks the 40th year of the Florida Industrial and Phosphate Research (FIPR) Institute's service to the citizens of Florida. The FIPR Institute was re-established within Florida Polytechnic University in 2012. Since that time, the Institute has integrated its administrative functions, library services, and K-12 STEM Education Program within the University. The Institute is funding cooperative research projects involving University faculty and students, which are described herein.

In this report, we describe the Institute's financial condition, community and business engagement, awards and achievements, information program, technical exchange, and research.

The Institute's financial condition is fairly stable, but is characterized by fluctuations up to hundreds of thousands of dollars per year. The Institute experienced some unusual expenses this year, but remained within its budget. However, these expenses decreased the Trust Fund and Auxiliary Account balances for the year as shown in the Financial Report section of this report. The unusual expenses included extensive facility repairs following Hurricane Irma and an unexpected audit that was charged to the Institute.

The Institute's staff is active in the community through organizations such as Florida Ag in the Classroom, Bartow Chamber of Commerce and its Leadership Program, Leadership Polk, Bartow Community Healthcare Foundation, Girls Incorporated, Polk Vision's Building a Healthier Polk, Girl Scouts of West Central Florida, and others.

Staff members serve on Boards and Expert Working Groups, as Honorary Chairs of conferences and universities, and as Science Fair Judges locally and internationally.

The Institute's library is open to the public, and contains both print and electronic resources. The in-house collection is largely phosphate-related, but there are broader scientific publications and periodicals, especially in energy and chemical engineering disciplines. In addition, the library is connected to other libraries of the state university system through Florida Polytechnic University.

The Education Program is fully integrated within the University's Community Outreach Program. STEM lessons developed at FIPR are used in the program to engage students and prepare them for further education and careers in high-tech fields.

The Institute, most notably through its Directors, continued to exchange technical information with other scientists and the public via workshops, conferences, publications, and presentations.

While the broad areas of research have remained the same, specific topics of interest change as needed. Mining, beneficiation, chemical processing, land reclamation, and public and environmental health remain focal areas for research. Current events may spawn new research, or evaluation of existing technology. However, our main drivers continue to be sustainability, comprehensive extraction, and critical materials.

Spiral Mineral Separation at the FIPR Institute.

Financial Report

FIPR Institute's research and operation are funded through the Phosphate Research Trust Fund. This trust fund receives its income from a portion of the severance tax paid to the state for each ton of beneficiated phosphate rock concentrate and pebble (not dry) as measured coming off the belt at the washer of each beneficiation plant.

Since the Institute's inception in 1978, the severance tax rates, distribution, and associated fees have varied greatly, as has the rate of mining. This has, in turn, resulted in widely variable rates of income for the Phosphate Research Trust Fund. The distribution to the Institute's trust fund has varied from 5% to 12.5% of the collected tax during our history. The distribution was 5.6% for this fiscal year, and the tax rate was $1.80 per ton of rock severed.

The FIPR Institute's Executive Director provides a summary of expenditures and the trust fund balance at public meetings of the Institute's Phosphate Research and Activities Board. A more detailed summary is included herein.

Trust Fund Balance	
July 1, 2017	$7,563,064
June 30, 2018	$7,146,732
Operations	
Income	
Gross Severance Tax[1]	$1,852,850
LESS Fees to Dept. of Revenue	($113,407)
Net Income	$1,654,873
PLUS Interest	$123,642
Total Income	$1,863,085
Expenses	
Research	($360,322)
Internal Operations[2]	($1,919,095)
Total Expenses	($2,279,417)
Change in Trust Fund	($416,332)

[1]Per Section 211.3103, F.S.

[2]"Internal Operations" includes staff salaries and benefits, and expenses for the Education Program, Library, and Laboratories. The Institute's Research Directors and technicians spend most of their time directing or conducting research. The remaining staff deliver information and education services to the public and schools, or provide office support within the Institute. Since "Internal Operations" also includes research done by the FIPR Institute staff, the portion of the annual income expended on research is substantial. Of the amount that is approved by our Board for specific research projects ("Research" in the summary table), the largest shares typically go to universities and private firms. Their research is conducted under the direction of the Institute.

Auxiliary Funds (Not from the Severance Tax)

According to Florida Statutes 1004.346 enacted in 2012, the FIPR Institute may also secure funding from grants and other available sources, enter into contracts, and provide consulting services. Revenue from these sources is deposited into an auxiliary account.

Auxiliary Account Balance	
July 1, 2017	$677,501
June 30, 2018	$661,972
Income	
Consulting	$31,740
PLUS Interest	$11,313
Total Income	$43,054
Total Expenses	($58,583)
Change in Auxiliary Account	($15,529)

Awards and Grants (Not from the Severance Tax)

Critical Materials Institute (CMI) Account	
Revenue	$142,891
Expenses	($142,891)
Change in CMI Account	$0
Florida Wildflower Foundation (FWF)	
Revenue[1]	$718
Expenses	($718)
Change in FWF Account	$0

[1]The remaining funds were approved as carry over from the previous fiscal year for additional research.

The overall financial status for FY 2017-2018, which combines the Trust and the Auxiliary Funds, was $8,240,565 as of July 1, 2017. The final amount, as of June 30, 2018, is $7,808,704; showing a net decrease of $431,861.

Community and Business Engagement

The FIPR Institute interacts with local community and business organizations in a variety of ways, such as providing our facilities for public use, participating in networking and fundraising activities, and supporting regional conferences and symposia. The Early Learning Coalition, the Polk County School Board, and the Bartow Chamber of Commerce use the FIPR Institute Conference facilities regularly for their meetings. Finally, the Institute's employees are active in the community and serve on various committees and boards.

- Brian Birky
  - Assists Polk County Public Schools with radon assessment and communications
  - Interprets radiological information and associated risk for community groups
  - Presents phosphate industry information to Leadership Polk, Bartow Leadership and other community groups upon request
- Gary Albarelli
  - Serves on the Board of Directors of Florida Ag in the Classroom
- Marie Wilmot
  - Graduate of both Bartow Chamber of Commerce Leadership and Leadership Polk
  - Serves on Girls Incorporated Board of Directors
  - Serves on Polk Vision's *Building a Healthier Polk* Alignment Team
  - Instructor at the Lake Wales YMCA and volunteers as an instructor at other community outreach facilities and events
    - Lakeland Coleman Bush Community Center
    - Bartow Carver Recreational Center
    - Polk County School Board Employee Wellness Programs
    - Bartow's Juneteenth event
    - Winter Haven's Fitness by the Fountain
  - Chairman for Bartow's fitness program - Fortress Fitness, which was recognized as a Healthy Weight Community Champion 2017 by the Florida Department of Health, State Surgeon General, Community Champion Recognition Program
  - Volunteers with Bartow Chamber of Commerce and Bartow Area Chamber Foundation Quality of Life Programs
- Kate Beamon
  - Volunteers each summer at Wekiva Youth Camp in Apopka, Florida with the 7th Grade Primitive Tent Camping Program
  - Volunteers as a Leader for a Girl Scouts of West Central Florida (GSWCF) with a focus on STEM and outdoor activities

    - Serves as volunteer GSWCF Service Unit Manager (South Lakeland/Mulberry)
    - Recruiter, Event Coordinator, Public Relations Contact and Assistant Product Sales Coordinator for the Mulberry Highlands Service Unit of GSWCF
    - Consults GSWCF staff on STEM activities and events
    - Elected as an Area Delegate for voting at GSWCF governance meetings
    - Selected and funded as one of five National Delegates to represent all GSWCF volunteers for governance voting at the National Council Session at the Girl Scouts USA national convention, G.I.R.L. 2017, in Columbus, Ohio in October 2017. This national convention of Girl Scouts only occurs once every three years.
- Indira Sukhraj
    - Serves on the Internal Review Board (IRB) for the Polk Regional Science Fair
    - Serves on Polk Vision's Talent Pipeline Team
    - Serves as a member Florida Polytechnic's THRIVE Team
    - Serves as a STEM Consultant with Texas Instruments'
    - Runs a local animal charity and volunteers at local animal welfare events
- Patrick Zhang
    - Dissertation Committee member for a Ph.D. student of Florida A&M University
    - Participated in Step Out for Education with Marie Wilmot to raise funds for Polk Country Education Foundation

Awards and Achievements

The Institute's staff members continue to serve on prestigious panels and committees, and their counsel is in constant demand internationally.

Dr. Brian Birky is Co-Convener of the PG NORM Working Group (Expert Panel) of the International Fertilizer Association (IFA). He is a member of the United Nations Economic Commission for Europe (UNECE) Expert Group on Resource Classification and he and Dr. Patrick Zhang are Technical Consultants (United States) to the International Atomic Energy Agency (IAEA).

Dr. Patrick Zhang is a member of the Editorial Board for Mineral Processing and Extractive Metallurgy Review journal. He is also Honorary Chair for the Center for Comprehensive Utilization and Sustainable Development of Phosphate Resources, China University of Geosciences.

Dr. Steve Richardson is an Advisory Committee Member, Polk County Bone Valley Special Area Study. He also serves on the Lake Wales Ridge Environmental Advisory Committee.

Ms. Indira Sukhraj is an International Science and Engineering Fair (ISEF) Judge, and reviews grant proposals for National Science Teachers Association (NSTA). She is also a judge for the Florida Junior Academy of Sciences and the Army Education Outreach Program's National eCybermission Challenge. She is a member of the Polk Regional Science Fair's SRC (Scientific Review Committee), is an aquatics expert for Tampa Bay Regional Envirothon, and is involved with LE/AD (Lakes Education Action Drive) to educate the public about phosphate in the environment. She also works with Texas Instruments on STEM Education, beta testing new equipment and integrating science and engineering. Indira earned her Master's Degree in Instructional Design from Saint Leo University this year.

Information Program

The Information Program primarily consists of the Institute's Library, widely considered the world's most extensive collection of phosphate-related reference materials, the K-12 Education Program, which brings the science of Florida's phosphate mining and processing into the classroom, and communications, which provides information on phosphate-related issues to the public. The Institute uses the internet to share information about its activities and promotes the websites www.fipr.state.fl.us and www.floridapoly.edu.

The FIPR Institute Library provides books, periodicals, maps, and many other reference materials for use by the public. Everyone is welcome to use the Library, both in person and through online services, and residents with a valid Florida driver's license may check out many of the books in our collection and other specific items. The Library also participates in an interlibrary loan program to enhance accessibility to information for all members and their patrons. As a part of the State University System (SUS), the Library can also access many research articles of interest to research scientists and students.

The Library continues to serve a diverse population of patrons, primarily phosphate industry professionals, but also students and members of the public, answering questions and providing literature searches about phosphate mining, technology, and history; phosphogypsum technology and potential utilization of this by-product; mine reclamation; and issues concerning the environment and public health as related to the phosphate industry.

The Library completed its survey of its collection to determine the scope of its planned digitization of the public domain works in the collection. Budgetary quotes have been obtained from vendors and the library will be applying for grant(s) opportunities for this work in the coming year.

As part of the assessment for the digitization project, the library conducted a weeding process to eliminate outdated materials and material not relevant to the mission of the Institute or University. As a result, approximately ten percent of the book collection was removed.

In keeping with the statutory charge of the Institute to be an information resource for the citizens of Florida on phosphate-related issues, the library continues to provide reference, circulation and interlibrary loan services to its stakeholders.

The FIPR Institute's K-12 STEM Education Program has substantially merged with Florida Polytechnic University's Education Outreach Team. Activities for the fiscal year are summarized below.

- Create STEM intensive lessons that integrate FIPR and Florida Polytechnic-specific content
- After school STEM Program partnership with the City of Winter Haven Cultural Center, City of Winter Haven Parks and Recreations after school STEM Programs, Polk County Parks and Recreation after school STEM Programs
- In-classroom STEM lessons - Polk, Hillsborough, Pasco, Sumter, Highlands, Duval, Clay, Lake, Indian River and Hardee Counties
- Water, Wings and Wild Things at Circle B Bar Reserve
- Collaboration with industry partner, Mosaic, in designing and presenting an activity at Agrifest
- Host on campus STEM activities at the University's iconic Innovation, Science and Technology Building (IST)
    - Independence Academy
    - RISK Group
    - Heartland Group
    - Polk County Gifted Students
- Earth Day - Sustainability
    - Florida Polytechnic Earth Day sponsored by SGA
    - Earth Day at Bok Tower Gardens
- Science and Engineering Fairs
    - Project Mentor
    - Judge at assorted Local School Fairs in Polk, Lake, and Indian River Counties
    - Judge and Senior categories captain at Polk Regional Science and Engineering Fair
    - Judge Heartland Regional Fair (Hardee, Okeechobee, Hendry, Glades, Highlands, Lee Counties)
    - Judge and category chair at Florida State Science and Engineering Fair
    - Judge at INTEL ISEF[1]

[1] 1The INTEL ISEF is the largest event of its kind. There are other international science fairs, but none as large as the INTEL-sponsored one. Approximately 60 countries have students and judges participating each year. The event is an excellent way to stay informed about STEM education around the world, and can be a venue for recruiting top international students to Florida Polytechnic University.

- Participated in the Great American Teach-In at several schools in Polk and Hillsborough Counties
- Florida Polytechnic Collaboration
    - Working with Admissions to inform them of upcoming activities; they are incorporating our team into some of the campus tours
    - Collaborating with the Presidential Ambassadors by providing them with educational outreach opportunities and professional development
    - Recruiting Florida Polytechnic student volunteers
- Florida Association of Science Teachers (FAST) Conference
- National Science Teacher Association (NSTA) Conference
- Texas Instruments T3 Leadership Summit

International Science and Engineering Fair (ISEF).

Technical Exchange

FIPR Institute staff shared technical information and expertise at conferences and workshops both locally and abroad. We frequently serve as organizers, session chairs, and presenters. In addition, we publish both in-house and external papers as a result of our research and information programs.

This fiscal year, there were some notable events involving environmental and public health concerns that prompted the press and community groups to seek scientific opinions and comments from FIPR, which were seen in newspaper articles and television segments. Examples of the Institute's technology transfer activities are summarized below.

Publications in Peer-reviewed Journals

Patrick Zhang

Simultaneous recovery of rare earths and uranium from wet process phosphoric acid using solvent extraction with D2EHPA, Hydrometallurgy, Vol. 175, pp. 146-153, 2018.

Rare-earth Leaching from Florida Phosphate Rock in Wet-process Phosphoric Acid Production. Minerals & Metallurgical Processing, Vol. 34, No. 3, pp.146-153, 2017.

The Ultimate Mineral Processing Challenge: Recovery of Rare Earths, Phosphorus and Uranium from Florida Phosphatic Clay. Minerals & Metallurgical Processing, Vol. 34, No. 4, pp. 183-188, 2017.

Rare Earths Recovery and Gypsum Upgrade from Florida Phosphogypsum. Minerals & Metallurgical Processing, Vol. 34, No. 4, pp. 201-206, 2017.

Recovery of Rare Earth Elements in Hydrometallurgical Processes of Phosphate Rock - A Critical Review. Chemical Engineering Journal, Vol. 335, pp. 774–800, 2017.

Brian Birky

Feasibility Test of Cellulosic Filter for Collection of Sulfuric Acid Mists. Separation and Purification Technology, Vol. 195, pp. 398-403, 2018.

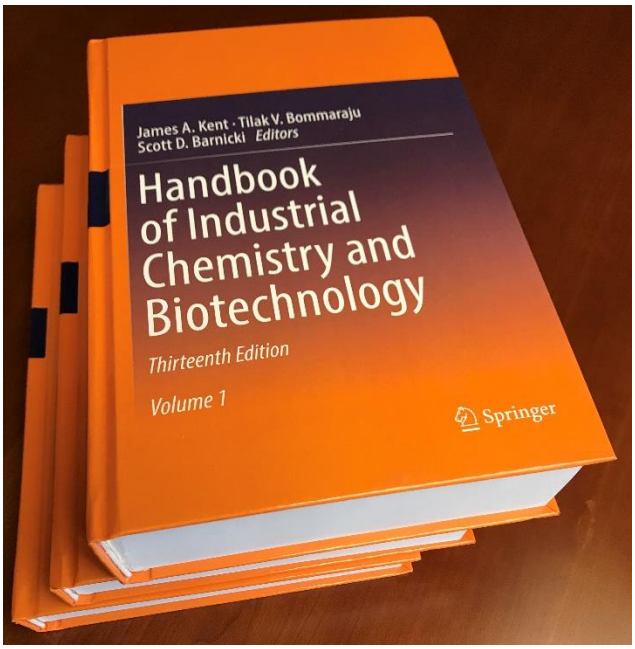
Development of a thoracic personal sampler system for co-sampling of sulfuric acid mist and sulfur dioxide gas. Journal of Occupational and Environmental Hygiene, 14(7), 562-571, 2017.

Book Chapter

Brian Birky

Handbook of Industrial Chemistry and Biotechnology: 13th Edition. Three-volume set. Volume 2, Section 3, pp. 1211-1239, 2017. Edited by James A. Kent, Tilak V. Bommaraju and Scott D. Barnicki
ISBN: 978-3-319-52285-2 (Print); 978-3-319-52287-6 (eBook)
DOI: 10.1007/978-3-319-52287-6
Volume 2, Section 3, Pages: 1211-1239, 2017
Website : https://link.springer.com/chapter/10.1007/978-3-319-52287-6_20

Updated Chapter "Phosphorus and Phosphates" by adding new process descriptions, illustrations, and emerging technologies.



This three-volume, 42-chapter set is a state-of-the-art professional sourcebook for the science and engineering and practices of the chemical process industry. All the chapters have been updated and written by individuals with demonstrated expertise in their respective fields. The chapters are organized into seven sections, each section being devoted to a specific area of industrial chemistry and biotechnology: Section 1: Raw Materials for the Chemical Process Industries, Section 2: Industrial Organic Chemistry, Section 3: Industrial Inorganic Chemistry, Section 4: Polymer Chemistry, Section 5: Biochemistry, Section 6: Emerging Fields of Industrial Chemistry, and Section 7: Industrial Processing and Engineering.

Dr. Birky updated the Volume 2, Section 3, Chapter “Phosphorus and Phosphates” by adding new process descriptions, illustrations, and emerging technologies. From the abstract:

> “Phosphate rock extraction from its ore, and its subsequent conversion into fertilizer materials and industrial chemicals, is a relatively mature art. While production of phosphoric acid from sedimentary phosphate rock via the “wet process” dominates the industry, the geology and mineralogy of source rock varies, and many processing choices are available. Changing global economics, social and environmental pressures affect modern market conditions. This chapter discusses phosphate mineralogy, deposits, mining, beneficiation, production, value, chemical processing technologies, products, by-products, and environmental factors.”

Presentations

Patrick Zhang

Rare Earths Occurrence in Florida Phosphate Ore and Their Fate during Mining and Chemical Processing. Beneficiation of Phosphates VIII, Cape Town, South Africa, April 29 – May 4, 2018.

Recovery of Rare Earths and P from Pre-concentrated Phosphate Flotation Tails by Sulfuric Acid Leaching. Beneficiation of Phosphates VIII, Cape Town, South Africa, April 29 – May 4, 2018.

Breakthroughs in Commercializing Two Mineral Separation Devices for Dramatic Reduction in Energy and Water Consumption. SME 2018 Annual Meeting, Minneapolis, Feb. 25 – 28, 2018.

Progressing Towards an Ultimate Solution to the Florida Waste Clay Pond Problem. SME 2018 Annual Meeting, Minneapolis, Feb. 25 – 28, 2018.

Simultaneous Recovery of Heavy Rare Earth Elements and Uranium from Wet Process Phosphoric Acid. Lakeland Regional Phosphate Conference, October 2017.

Brian Birky

“FIPR Institute Overview” with phosphate industry issues and radiation in perspective demonstration. Presentation to Bartow Leadership, March 15, 2018.

Gary Albarelli

Revisiting the Merseberg Process: Economic Opportunity and Environmental Benefit? Beneficiation of Phosphates VIII, Cape Town, South Africa, April 29 – May 4, 2018.

Phosphogypsum and Sustainability. Mosaic CAP Riverview, March 22, 2018.

Phosphogypsum and Sustainability. Mosaic CAP New Wales, Feb. 8, 2018.

Sustainability and the Florida Phosphate Industry. Lecture for Renewable Energy and Sustainability course at Florida Poly, November 16, 2017.

Indira Sukhraj

Cracking the Code - Integrating Computer Science Standards, Presented at the Florida Association of Science Teachers Annual Conference, Orlando, FL October 2017.

Poster Presentation

Brian Birky

Limitations and Modifications of Impregnated Filter Method for Sampling Sulfur Dioxide Gas. 36th Annual Conference of the American Association for Aerosol Research, 2017.

Leadership Role

Brian Birky

Provided technical education and expertise for residents of Oakbridge and Grasslands during radiation litigation

Provided radon expertise, education and mediation during administration and faculty meetings on behalf of the Polk County School Board

Florida Radiation Advisory Committee – Environmental Expert

University of Florida – Doctoral Committee

University of Florida – UF Change Chem Labs Advisory Board

Convened NORM and Phosphogypsum Working Group of the International Fertilizer Association at SHE Symposium in Madrid

Technical consultant for IAEA (Vienna)

Member of the UNECE Expert Group on Resource Classification (Geneva)

Patrick Zhang

Chair
Beneficiation of Phosphates VIII, Cape Town, South Africa, April 29 – May 4, 2018

Technical Committee Chair
1st International Conference on Sustainable Development of Phosphate Resources and involved in organization of the 2nd International Conference on Sustainable Development of Phosphate Resources, to be conducted in July 2019, Guiyang, China.

Ph.D. Dissertation Committee Member
Radionuclide and Heavy Metal Bioremediation of Florida Phosphogypsum
Ivory Council, Florida A & M University

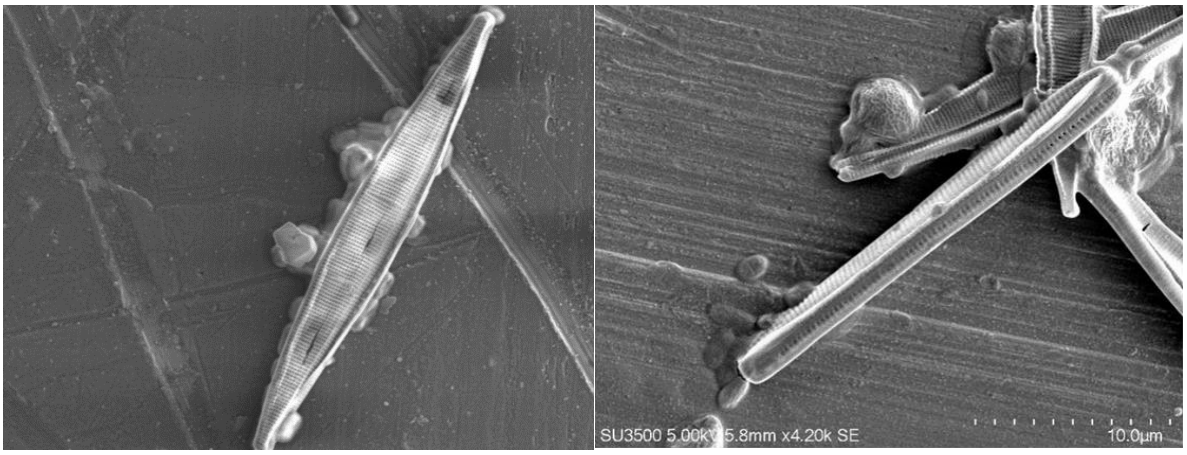
Beneficiation of Phosphates VIII, Cape Town, South Africa

Florida Polytechnic University Faculty-Student Research Program

This fiscal year the Institute initiated a FIPR-funded program whereby Poly faculty could apply for funding to address an applied research problem in their field of expertise that will engage students as research assistants. The goal of the program is to provide students with the opportunity to develop research skills to creatively address real-world problems. During the past year, FIPR has funded two such projects. Gary Albarelli, FIPR Director of Information Programs, oversees projects funded under this program.

Mass Production of Endemic Diatoms in Polk County with Concomitant Biofuel Extraction and Cost Analysis. Budget: $49,486; Duration: 1 year. Faculty Investigator: Dr. Melba Horton. Four student researchers have been engaged in the research.

The objectives of this study are to identify endemic diatom species in Polk County freshwater, and explore cost-effective means of mass-production of diatoms for lipid extraction and use of frustules.



SEM Images of Diatoms Collected from Polk County Lakes used to Seed Photobioreactors.

Lake samples from the water column of a wide variety of area lakes were provided by Polk County Parks & Recreation. Initial microscopy showed presence of diatoms in all samples, but two samples were chosen as most prolific for diatoms. Photobioreactors were designed, built and deployed both in a laboratory and exposed to the outdoor environment. Diatom growth in the reactors, treated with a commercially available growth medium, was mixed with green algae, and a distinct die-off of diatoms occurred within each treatment and trial. The peak population was measured to occur at two weeks, followed by the die-off. Lipid extraction amounts increased with growth time. However, the source of these lipids could not be distinguished from diatoms or competing green or

blue-green algae. Because of the die-off, reseeding of diatoms on a regular basis is necessary. Additional samples were collected to identify diatoms from bottom substrates of three additional lakes, and the samples were tested for growth in the photobioreactors. Future work will include assessment of additional water quality parameters that may account for die-offs and testing growth using a diatom-specific growth medium that can be formulated at a reduced cost.

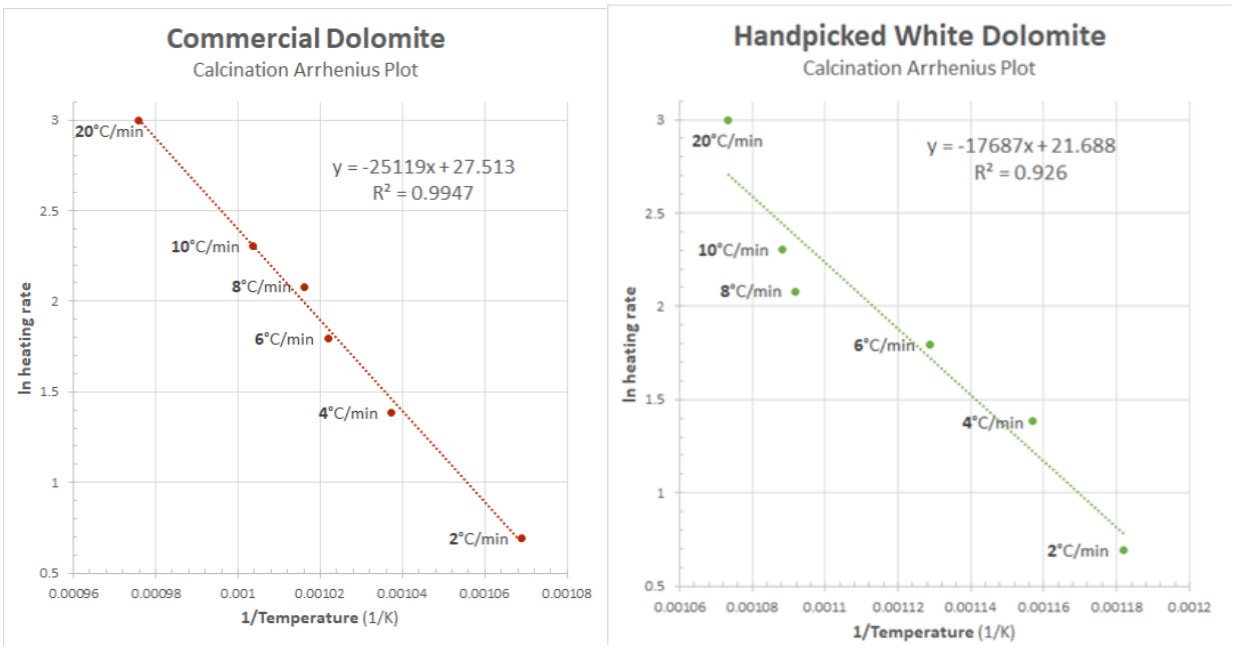
Research Team Collecting Substrate Samples from a Polk County Lake.

Feasibility Studies of Dolomites from Phosphatic Pebble for Thermochemical Energy Storage and CO_2 Sequestration. Budget: $54,857; Duration: 1 year. Faculty Investigator: Dr. Sesha Srinivasan. Three student researchers have been engaged in the research.

The objectives of this study are: to utilize dolomitic phosphate pebble to facilitate the calcination-carbonation cycle for thermochemical energy storage, and to investigate the potential for this system as a Carbon Capture and Sequestration (CCS) system. The hypothesis is that impurities present in the dolomitic pebble would avoid a sintering

phenomenon that has occurred in prior work using pure dolomite, which inhibits repeated carbonation-calcination cycling.

High-dolomite pebble samples were furnished by the FIPR Institute. Analyses of the calcination and carbonation reactions were performed on these samples with and without physical sorting by color to determine those with highest dolomite concentrations, thus having the greatest cycling potential. Samples were ball-milled under various conditions to increase particle surface area to allow for increased sites for carbonation. Further chemical and mineralogical analyses will be performed on these samples to help explain the differences due to elemental or crystalline structure differences. Further work will include demonstration of cycling both at ambient and increasing pressures to determine effects on the extent of carbonation.



Comparison of Arrhenius Plots from TGA Results used to Determine Activation Energy of Calcination Reactions.

FIPR Institute Research

FIPR Institute research projects are either conducted in-house or by various universities and private companies using Institute funds. FIPR Institute Research Directors serve as Contract Managers for all projects. Projects that were completed or ongoing during the fiscal year are described in the following text.

The Institute's projects that are funded by the Phosphate Research Trust Fund are directed at solving real-world problems identified with the mining and processing of phosphate rock in Florida in which the public has a substantial interest. Summaries of the Institute's research are described by title, funded organization, and a brief description of the objectives and accomplishments.

Recovery of Rare Earths and Uranium from Phosphate

FIPR Institute and the Critical Materials Institute

In early 2013, the US Department of Energy (DOE) awarded $120 million to the Critical Materials Institute (CMI) to establish a new Energy Innovation Hub. CMI focuses on developing and commercializing advanced technologies to secure the national supply for critical materials, particularly rare earth elements (REE). The FIPR Institute is undertaking the project on recovery of U and REE from phosphate mining and processing products as well as byproducts. All CMI members are shown in the figure below.

CMI member universities, national laboratories, and industry partners.

The FIPR Institute has hosted four annual workshops that reviewed progress of all member teams researching the recovery of REE and uranium from phosphate rock. There were 10 phosphate process streams characterized so that now the REE content, distribution and available forms are known. Beneficiation technologies to concentrate the REE have been developed for each of the six phosphate process streams. The FIPR Institute collaborates with other CMI member institutions and affiliates. ORNL research involving University of Tennessee students, and ThorOre in collaboration with University of Central Florida students, were hosted at the FIPR Institute laboratory facilities.

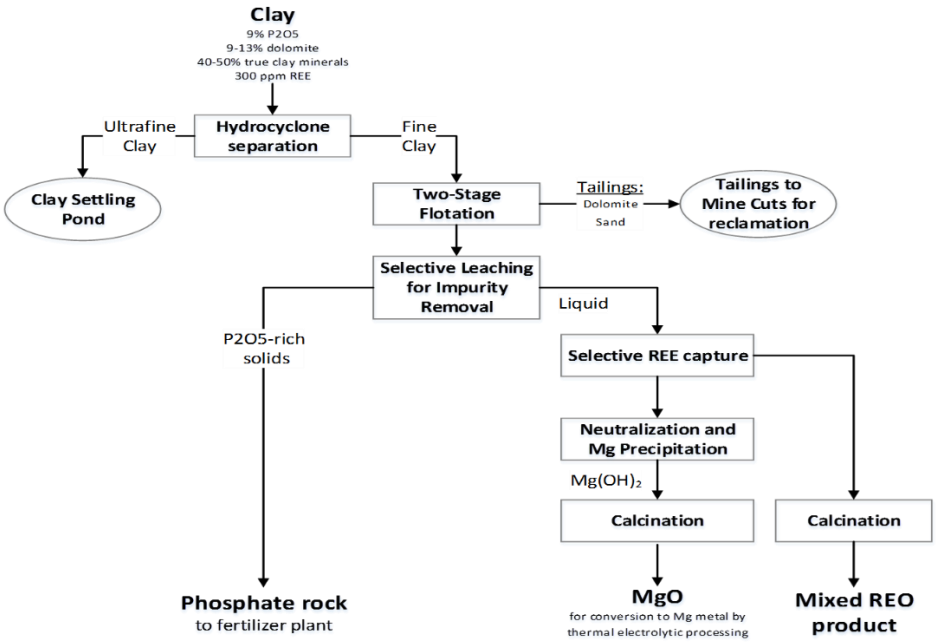
The major FIPR achievements under the CMI project are summarized as follows:

- gained a better understanding of rare earths occurrence in phosphate rock, phosphate flotation tailings, phosphogypsum (PG), acid sludge, and phosphoric acid, thus being able to develop suitable beneficiation and extraction schemes for each stream;
- conducted two in-plant pilot testing campaigns to concentrate REE minerals from waste clay and amine flotation tailings, with shaking table testing achieving roughly 50% REE concentration in about 6% of the total mass of flotation tailings;
- developed a multi-stage leaching scheme for recovering REE from PG using dilute sulfuric acid without infringing on the regulatory conditions of PG;
- discovered a significant REE source material: sludge from phosphoric acid concentration/clarification;
- an extraction process was developed for simultaneous recovery of heavy rare earths and uranium from phosphoric acid;
- a processing scheme was designed for producing high-value phosphoric acid and heavy rare earths-containing solids from phosphoric acid sludge;
- a laboratory, continuous testing system consisting of gravity separation and flotation was designed for long-term investigation on recovery of rare earths, phosphorus and uranium from phosphatic clay;
- five peer reviewed journal papers were published; and
- funding was approved for CMI Phase 2 (July 1, 2018 to June 30, 2023).

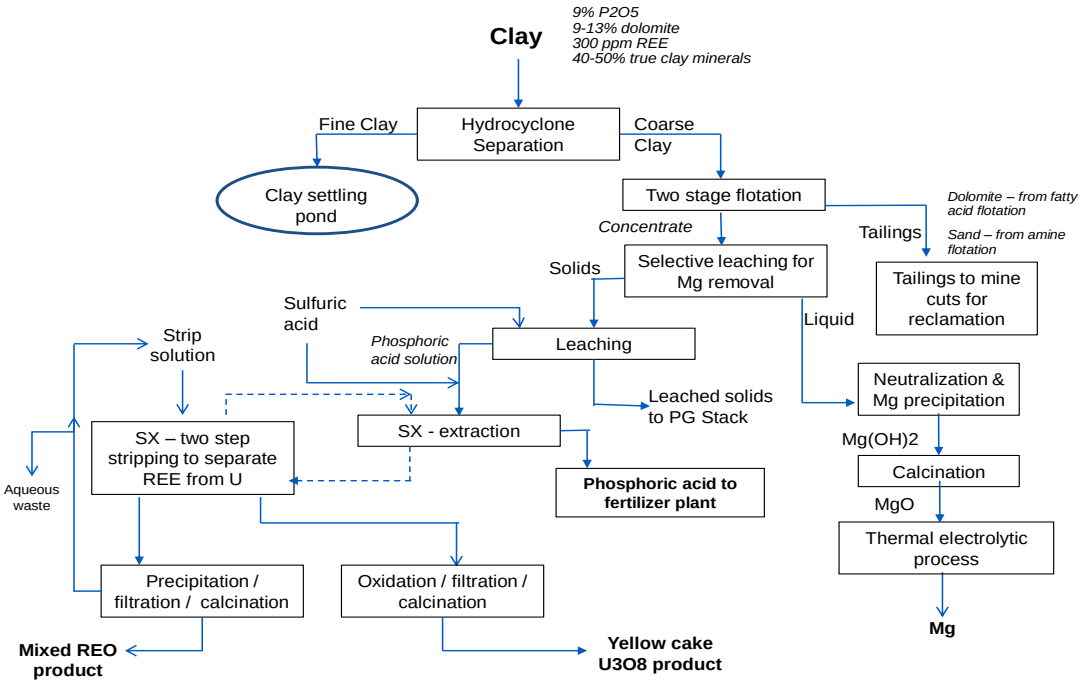
Funding was also approved for CMI team members (Colorado School of Mines, Oak Ridge National Laboratory and Idaho National Laboratory) for related testing of samples and technologies. The FIPR Institute gained the support of many industrial partners for Phase 2 work, including Mosaic, Nutrien, CAP Holdings Co., Clear Springs Land Company, Mineral Development LLC, Periodic Products, ThorOre, Jacobs Engineering, and K-Technologies.

Phase 2 research will focus on the recovery of phosphorus, rare earth elements, magnesium and uranium from waste phosphatic clay. Building on the Approach 1 – a

single-stage leaching process tested in Phase 1, a multi-stage Approach 2 has been conceptualized for Phase 2 testing.



Approach 1 of the FIPR CMI Project.



Approach 2 of the FIPR CMI Project.

Recent activities in preparation for Phase 2 include:

- collection of fresh samples of waste phosphatic clay;
- characterization of these samples; and
- acquisition of equipment needed for CMI Phase 2 research including a gravity and flotation separation testing system, and a hydrocyclone testing system.

Hydrocyclone Testing System.

Removal of Dolomite from Florida Phosphate Pebble Using Packed Column Jig

FIPR Institute

The project is designed to conduct continuous, pilot-scale testing of gravity separation using a packed column jig (PCJ) for separating dolomite from phosphate, thus allowing the use of large amounts of high-dolomite phosphate pebbles currently stockpiled or left in the ground. Dolomite, a natural contaminant in Florida phosphate rock, is a huge problem for fertilizer processing. The lower zone Hawthorne Formation being currently mined commonly has high MgO contamination. There are existing techniques for dolomite removal from phosphate rock, including: flotation, chemical treatment, and high-temperature treatment. All of these methods have some associated environmental issues.

PCJ is a column filled with packing plates, which are corrugated diagonally and set in an alternating configuration. The packing plates create a myriad of small cells in the column. A stream of mixed particles is fed through an inlet located near the top of the column. The

feed point depends on the feed characteristics and concentrate grade target. A steady state water flow enters the bottom of the column and a pulsating flow is also superimposed to create a jigging action that maintains all particles in suspension limiting stagnation problems. The unique features of PCJ can be summarized as follows: 1) low energy use, 2) long, nearly unlimited separation zone, 3) small footprint, 4) minimal water use, 5) no chemicals used, 6) high throughput, and 7) effective for both coarse and fine particles.

Laboratory-Scale Packed Column Jig at the FIPR Institute.

The objectives of the proposed research are to determine the optimum grinding size; obtain results comparable to the best flotation process for Florida dolomitic phosphate; demonstrate the process on a pilot scale; optimize all the operating parameters; and,

estimate capital and operating costs. A laboratory-scale packed column jig is ready for use, and a 3-ton/day pilot plant under construction. Testing will start soon.

Novel Technologies to Convert Dolomite Phosphate Rock into High Efficiency Slow Release Fertilizers

University of Florida

High dolomite phosphate rock is a worldwide problem in phosphate processing, and it is becoming more and more critical to the Florida phosphate industry. The wet process for phosphoric acid manufacturing is still the predominant technology for phosphate fertilizer production. This process generally requires a phosphate rock feed containing less than 1% MgO.

As the phosphate mining in central Florida moves farther south into the lower zone, the grade of the ore (matrix) gradually decreases, with dolomite being the major undesirable component. The flotation feed from the lower zone can generally be processed using the Crago double flotation process, but the pebble fraction averages 6% MgO and is currently stockpiled. The industry is estimated to generate close to one million tons of such high-dolomite pebbles per year. Other high dolomite phosphatic materials include waste clay and the oversize rejects from the washer. Finding beneficial uses for these dolomitic phosphate materials not only improves phosphate resource utilization efficiency dramatically, but also greatly reduces the environmental impacts of phosphate mining.

Recently, progress has been made in converting low grade phosphate rock into high efficiency, slow release P fertilizers by subjecting fine phosphate rock (PR) powder to reactions with some types of organic compounds called phosphate-activating agents. As a result, the surface of PR particles is covered with organic molecules, which markedly increase surface exposure to soil matrix and enhance physical, chemical, and biological reactions on the surface of PR. This two-year project includes 6 major tasks: 1) screening of activation agents to find the most effective; 2) optimization of the activation parameters; 3) physical and chemical characterization of the activated phosphate fertilizer; 4) greenhouse experiments to compare the new fertilizer with traditional phosphate fertilizers; 5) column leaching study to assess the environmental impact of the slow release fertilizer; and 6) economic analysis.

In the recently submitted project annual report, the research team presented the following encouraging findings:

- activation with selected organic molecules significantly increased water soluble P (WSP) in the dolomite phosphate rocks (DPRs), from ~5-15 to ~100-6000 mg/kg,

varying largely among the different activating agents or their combination, and between the DPR samples;
    - organic molecules were coded as HA, LSS, and CA;
    - based on WSP, the activating efficiency decreased in the order of HA > LSS > HA+CA > LSS+CA > LS >CA,
    - both HA and LSS are effective in activating DPR, enhancing P release from DPR by 300-700 times, but HA is overall superior to LSS;
- based on sequential extractions, supply of P from HA- or LSS-activated DPRs is of slow release characteristics with 30-60% released in the first extraction, as compared to >85% from soluble phosphate;
- quantitative XRD analysis indicated that the activation process significantly increased the ratio of apatite/dolomite, which could be attributed to partial dissolution of dolomite by the organic agents, and thus promoted release of P from DPRs;
- the results from the optimization study based on one rock sample indicate that the optimized activation conditions are:
    - particle size < 100 mesh, 8% dosage of activating agent, reaction for 20 min, and at 20% moisture;
    - under optimized conditions, activation with humic acid can raise water soluble P from 9.4 mg/kg to 8790 mg/kg, as estimated by one-time extraction; and
- activation processes enhanced release of Ca and Mg from DPR, but had minimal influence on water solubility of trace metals including Fe, Mn, Cu, Zn, Cr, Cd, Pb and Ni.

The project is nearly complete. The process produced effective slow release fertilizers, and the researchers have applied for patent protection for the technology. The research team conducted greenhouse experiments showing that the activated DPR fertilizers are similar or slightly better than water-soluble fertilizers, and they conducted column leaching studies indicating that the activated DPR fertilizers have much lower leaching loss of nutrients (P, Ca, and Mg) than water-soluble P fertilizers. The leaching potential of toxic metals (Pb, Cd, Cr, and Ni) is also much lower.

The economic analysis shows that the production cost for this fertilizer product is roughly $100/ton. The resulting product can be sold for about $230/ton. The available phosphorus of this fertilizer product is about 84% of TSP, making it a high quality fertilizer. Transportation costs are a big factor into the economic analysis, but there is a demonstrated market for the product.

Leaching Study for Select Process and Non-Process Waters Relative to Future Disposal through a Deep Injection Well

Ardaman & Associates, Inc.

The current practice of pond water treatment and subsequent surface discharge is not only expensive, but also consumes a tremendous amount of fresh water. In the current pond water treatment and surface discharge practice, at least 5000 gallons of fresh water are used as dilution water for every 1000 gallons of process water treated to meet the requirement for conductivity. A typical PG stack system closure could require the treatment of over 3 billion gallons of process water, consuming 15 billion gallons of fresh water.

The Ardaman & Associates project is designed to study the feasibility of deep well injection of treated/partially treated water as an alternative to surface water discharge for phosphogypsum stack closure. The objectives of this study are: determine the effect of single liming and double liming on water quality; prepare and characterize twenty rock cores for leaching tests; determine permeability and water quality changes over time through leaching tests; compare leaching test results with predicted data by geochemical modeling; and, analyze testing results to determine the feasibility of full-scale operation.

The scope of work involves the following major components:

- characterizing 8 water sources for 21 water quality parameters;
- conducting various treatment tests on the waters;
- modeling the behavior of treated or partially treated water in various carbonate cores;
- performing leaching tests to determine carbonate core integrity with time in various waters; and
- analyzing all test data to provide guidelines for implementing the deep well injection method.

Eight source waters were collected and treated in different ways to yield fourteen water samples for testing. Each sample was characterized for the following parameters: conductivity, pH, turbidity, TDS, calcium, magnesium, sodium, potassium, aluminum, iron, manganese, chloride, fluoride, total phosphorous, total nitrogen, silica, acidity, bicarbonate alkalinity, carbonate alkalinity, and total alkalinity. Permeability testing was performed on 11 geological core samples to determine suitability for leaching tests. A geochemical model was prepared for each water sample used in the leaching tests to determine the potential for the precipitation and dissolution of various minerals within the core sample. The final component of the study was a 180-day leaching test. This test is critical to determine the feasibility of deep well injection. The Institute expects to receive a final report soon.

Treatment of Chemical Processing Pond Water and Precipitation of Phosphorus Using Nclear

Nclear, Inc.

Currently, the primary strategy for treating process wastewater is double liming, which requires raising the wastewater to pH 11 in two steps, stripping the water of undissociated NH_3, and reducing the pH back to ~ 6.5 by adding acid.

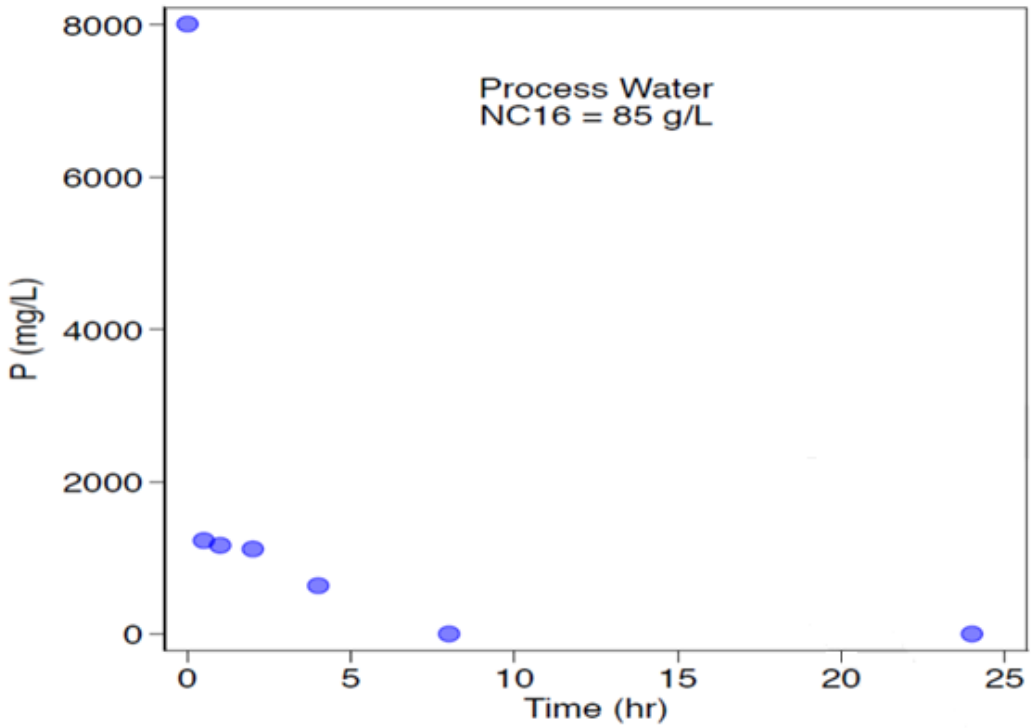
Nclear is a synthetic calcium (Ca) silicate mineral that induces and enhances phosphorus removal from water and wastewater. It requires lower pH for P removal than that required for double liming. Large-scale tests showed that Nclear was more effective than alum or ferric salts for P removal.

The primary objectives of the project are to:

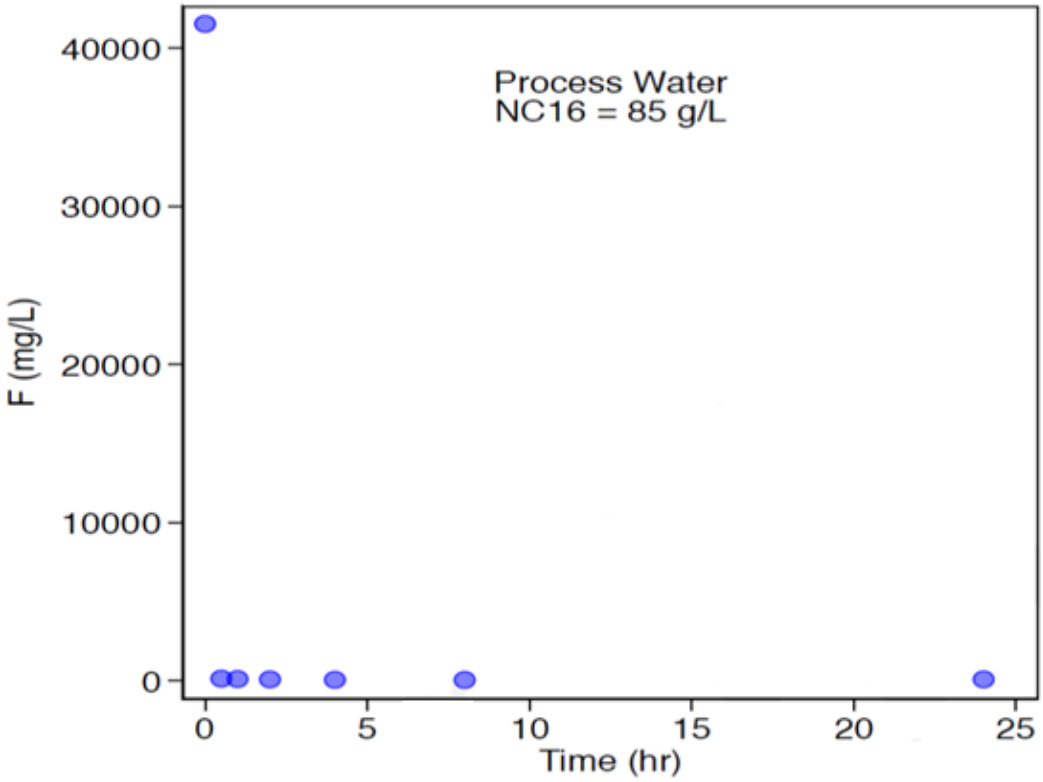
- understand the chemistry of pond and flotation waters;
- determine and compare removal efficacies of Nclear and double liming for removing phosphorus from chemical processing pond water;
- determine and compare removal efficacies of Nclear and alum/ferric salts for removing phosphorus from a synthetic flotation water;
- test the effectiveness of Nclear for ammonia (NH_3) removal; and,
- develop thermodynamic tools for comparing costs based on target treatment levels.

The project demonstrated that Nclear was effective for phosphorus removal from flotation water in a short period. For higher phosphorus levels to be tested, the samples were spiked with phosphorus to make a higher concentration in the synthetic flotation water. Nclear also was effective in phosphorus and fluorine removal from process water. Researchers are currently working on the economic analysis and testing the removal of ammonia from process water.

The dose response experiments indicate that the chemical cost of treating flotation water for phosphorus removal is cost-competitive with precipitation using alum. The chemical cost for treating 1,000 gallons of flotation water with NC16 was estimated to be closer to $1.90, while the alum cost was $3.80. Thermodynamic modeling indicates that the NC16 treatment cost may be further reduced through the recovery of P from the precipitating solids obtained from treatment with NC16 (P content 10.8%). As seen in the following figures, treatment of process water with NC16 is effective. However, the cost for treating process water was not very encouraging.



P Removal from Process Water with Nclear.



F Removal from Process Water with Nclear.

Beneficiation of Dolomitic Phosphate Pebble by Triboelectrostatic Belt Separation
Jacobs Engineering Group

This research project is designed to evaluate a non-chemical, electrostatic technology for processing high-dolomite phosphate pebbles. The objectives for this study include:

- acquire and characterize a representative high MgO sample;
- determine the dolomite liberation particle size;
- perform pilot plant testing using a bulk sample to optimize separation efficiency;
- characterize the products; and
- analyze the economic feasibility.

The Colorado School of Mines completed the dolomite liberation study, and Jacobs Engineering completed the metallurgical testing. Two tons of bulk sample were crushed and ground by FLSmidth, and ST Equipment & Technology completed the separation tests. The project is finished, and the final report is expected soon.

Screening of a New Candidate Biological Control Agent of Brazilian Peppertree
University of Florida

Brazilian peppertree (*Schinus terebinthifolius*) is a non-native, highly invasive shrub or small tree that infests thousands of acres of mined and unmined lands in Florida. The plant is not invasive in its native habitat in South America, indicating that its aggressive spread in Florida and elsewhere may be due to escape from its natural enemies. This suggests that importing Brazilian peppertree's natural enemies, such as certain insects, may help control the plant. The research has been testing the performance of two species of a leaf-gall-forming psyllid insect (*Calophya latiforceps and C. terebinthifolii*) on injuring Brazilian peppertree, testing the host specificity of the insect (attacking the target plant species, but not harming other plant species), and developing methods for rearing the insects. This is information needed before the insect can be considered for release in Florida.

Two insects have been and are being screened as possible biological control agents. Insects have been imported to quarantine labs both in Florida and in Brazil, and researchers have been working to rear them and assess their control performance and host specificity for Brazilian peppertrees.

Progress of the research project includes:

- a petition has been submitted to the USDA Animal and Plant Health Inspection Service (APHIS) requesting field release of *Calophya latiforceps* as a biological control agent on Brazilian peppertree;

- the USDA Technical Advisory Group recommended approval for release;
- the final report for this project was submitted to the FIPR Institute and is in the final review process;
- populations of *Calophya* species are being maintained at Fort Pierce for continued research and possible release.

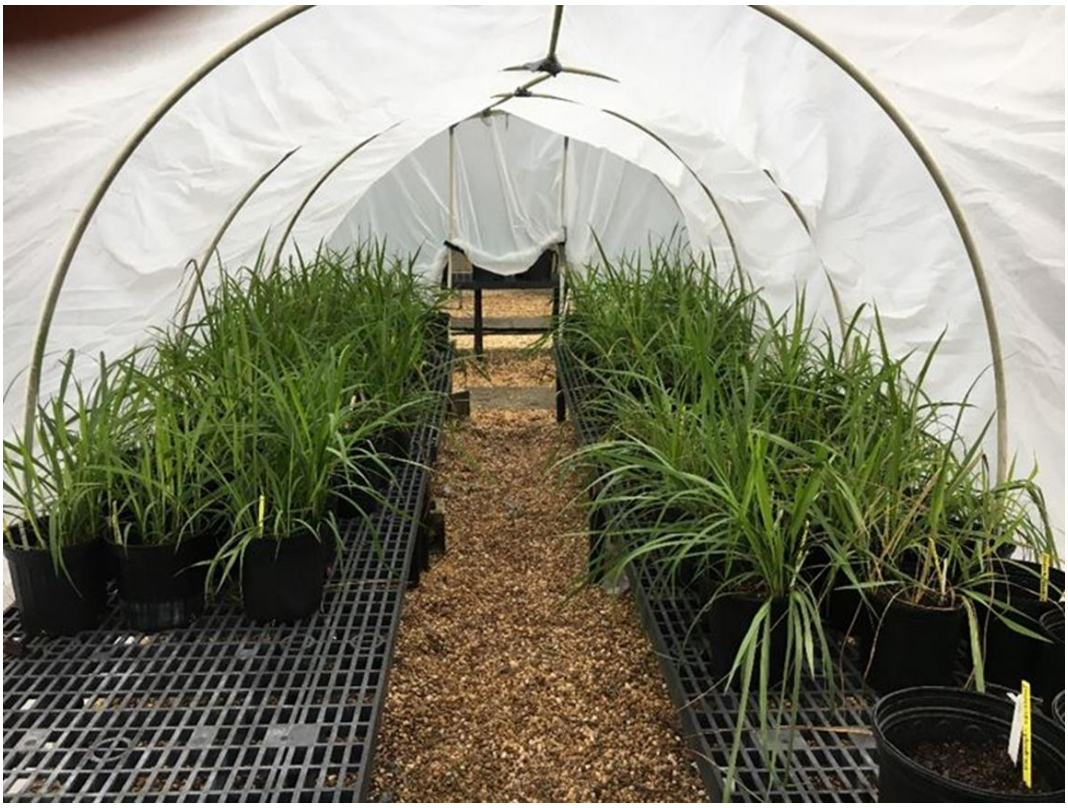
Brazilian Peppertree with Leaf Damage from Calophya latiforceps.

Potential Biological Control of Cogongrass with the Indonesian Gall Midge, Orseolia javanica

University of Florida

Cogongrass (*Imperata cylindrica*) is a non-native and highly invasive rhizomatous grass that has infested thousands of acres of mined and non-mined lands in Florida. A gall midge insect (*Orseolia javanica*) has been discovered in Indonesia that attacks Indonesian strains of cogongrass. Parasitic insects may be host-specific, even requiring a certain genotype plant. The UF research group has found preliminarily that Florida

cogongrass (that has two genotypes – Central and North Florida) is genetically similar to Indonesian cogongrass. This indicates that the gall midge could be potentially used to target Florida cogongrass. A preliminary project was funded to determine if the Indonesian gall midge would be able to complete its life cycle on Florida cogongrass and inflict damage to the grass. Cogongrass rhizomes were collected from northern and central Florida; permits were obtained; and the rhizomes were shipped to Indonesia for testing. The cogongrass is growing well, but there have been some issues in rearing the insect in greenhouses. High heat and parasites on the wild-collected gall midges are problematic. Although not yet funded, methods for a possible full study would include importation of insects to the quarantine lab; successful rearing of the insects; assessing the insect performance against cogongrass; and insurance of host specificity.



Florida Cogongrass Cultivation for Testing.

Establishment and Management of Vegetation Cover on Phosphogypsum Stacks

FIPR Institute

The initial research was conducted 1989-2004. Ongoing efforts include training of new industry personnel and consultants/contractors on the principles and methodology for establishing and managing vegetation cover on the side slopes of phosphogypsum stacks, plus evaluating and testing the effectiveness of additional techniques.

Grass Established on the New Wales South Phosphogypsum Stack via Hydro-seeding.

The FIPR Institute's reclamation department has conducted extensive research on this topic. During the previous fiscal year, Mosaic asked for FIPR's assistance in providing training on earlier findings and in conducting further tests on potentially more cost-effective methods for pH adjustment and grass establishment on closed phosphogypsum stack side slopes. The FIPR Institute has conducted a training workshop for industry consultants. Field tests are being performed on experimental methods of establishment and management of grass cover on closed stacks, to include the use of bermudagrass sod or hydroseeding and mulch applications.

Preliminary findings and/or recommendations include:

- do it right the first time (it is expensive to rework grass establishment areas);
- allow time for rainfall on the stack and subsurface drains to reduce the salinity and acidity before planting (this reduces the amount of lime that must be applied for pH balancing);
- if sod is to be used directly on phosphogypsum, it must be at least 95% bermudagrass (a grass species tolerant to acid and salt);

- mowing of grassed areas (primarily for weed control) should be reduced in frequency and mowing height should be increased (grass cover can be damaged by tractor tires sliding, even slightly, downslope and by mowing itself, particularly when the mower hits a high spot in the gypsum);
- tillage is required to loosen the phosphogypsum surface prior to planting.

Work this year included the use of better quality sod of tolerant grass, allowing natural leaching to raise pH and reduce lime application, and testing potentially more effective hydro-seeding and hydro-mulching methods.

On-going work was placed on hold while Mosaic worked on the sinkhole remediation, but they are now ready to continue.

Native Wildflower and Grass Establishment
FIPR Institute and Florida Wildflower Foundation

This completed research project was a cooperative effort of the FIPR Institute, the Florida Department of Environmental Protection (FDEP), and the Florida Fish and Wildlife Conservation Commission (FWC) in addition to the grant funds from the Florida Wildflower Foundation. The project focused on the establishment of native wildflowers and grasses on disturbed lands (including mined and non-mined lands) and the control of invasive, competitive weeds.

Most of the weed seeds occur in the uppermost layer of soil. Soil inversion, using a moldboard plow, is being tested as a way to bury the weed seeds and thus prevent or inhibit weed seed germination, compared to shallow tillage by disking. The moldboard plow treatment showed some success, but the soil inversion achieved was sometimes incomplete, especially when plowing through sod. Research staff members and cooperators are currently in search of a deeper plow that would create more complete soil inversion.

Use of Moldboard Plow for Soil Inversion with Some Incomplete Inversion (inset).

The application of pre-emergent herbicides immediately after planting of small container-grown plants was found to be effective for preventing broadleaf and grass weed seed germination, reducing weed competition, and promoting good plant establishment and growth. At two of the study sites on former mined lands, either not treated with pre-emergent herbicides or after the pre-emergent effect had worn-off, the weeds were predominantly broadleaved plants. Where grasses had been planted, application of a selective post-emergent (applied to the leaves) broadleaf herbicide, which did not injure the grasses, resulted in reduced weed competition and good grass establishment and growth. Unfortunately, we do not yet have a post-emergent herbicide that could control broadleaf weeds without also severely injuring the planted broadleaved flowers, although it is possible to apply some grass killers without injury to the broadleaved flowers. Selective control of various weeds with minimal or no injury to desired plants is the subject of ongoing research. The work includes control of very difficult perennial weeds as well as weeds from seeds.

The final research report has been submitted to the Florida Wildflower Foundation. FIPR will hold a workshop on weed management and the establishment of native flowers and grasses during fall 2018. The Florida Wildflower Foundation has encouraged submission

of a proposal for continued research. Dr. Steven Richardson of FIPR serves on the Research Advisory Committee of the Florida Wildflower Foundation.

Pre- + Post-emergent Herbicides (left) vs. No Herbicides (right).

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Please contact us for more information on the research or programs of the FIPR Institute.