Fiscal Year 2013/2014 Annual Report

Florida Industrial and Phosphate Research Institute

The FIPR Institute's Education Building

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Phosphate Research and Activities Board Members

Michael Daigle, The Mosaic Company

Herschel Morris, CF Industries, Inc.

Ann Paul, Audubon Florida

Vishwas Sathe, Florida Department of Environmental Protection, Tampa Office

*__Ava Parker__, Florida Polytechnic University

*Appointed by the FPU's Board of Trustees to serve until the President is hired

Florida Industrial and Phosphate Research Institute

Directorial Staff

Brian K. Birky, Ph.D., Interim Executive Director

Research Director, Public and Environmental Health

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

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

Vacant, Research Director, Chemical Processing

Gary Albarelli, Director of Information Programs

Karen J. Stewart, MLS, Library Director

Interim Executive Director's Message – Dr. Brian Birky

The phosphate industry plays an important role in ensuring a plentiful, low-cost food supply in the United States and helps ensure that there is sufficient food to feed a burgeoning world population. The industry also plays an important role in the economies of central and northern Florida. However, the industry's mining and chemical processing operations and the large scale of the phosphate industrial impacts raise public concerns about the environment and public health. Some of these concerns are perceived but lack scientific grounding, while some have a basis in fact.

The people in the phosphate regions worry that the industry may adversely impact the public water supply, spill acidic water or clay into the environment, and may not leave land in a useable form after mining.

The Florida Institute of Phosphate Research (FIPR) was established by the Florida Legislature in 1978 to study these issues, to help the public understand the extent and scope of any problems, and to find solutions. In 2010, the Institute's mission was expanded in legislation to include industries other than the phosphate industry and to encourage commercialization of its research products and intellectual property. On April 20, 2012, legislation was signed that immediately created Florida Polytechnic University as a new institution in the State University System of Florida. The Institute was re-established within this new university. Following the polytechnic model, the university will emphasize the application of science, technology, engineering, and mathematics.

The Institute's role is to conduct scientific investigations that will give lawmakers, regulators, members of the industry, environmentalists, and the general public the information they need to make decisions relating to issues of industrial influence or origin. Through science, we at the Institute endeavor to present the facts to the people of Florida. We strive to develop the research that will provide better solutions for phosphate industrial issues. The Institute's research principally addresses solutions for process water, clay settling areas, phosphogypsum management, reclamation science, energy and water conservation, and resource recovery.

In the Institute's 36 years of operation, it has made great strides in addressing the most vexing problems both within the phosphate industry and in the environment and communities it occupies.

Process Water - Process water is used in the chemical plant that converts mined phosphate rock into the phosphoric acid that is used to make fertilizer. There are billions of gallons of this acidic water generated in phosphate processing. Process water is

stored on and adjacent to phosphogypsum stacks that rise 200 feet into the air and can cover 400 or more acres of land. Our research is looking at ways to reduce the quantity and improve the quality of the water. We also study ways to clean the water so it can be safely released into the environment. The Institute has funded research on the effectiveness of secondary liners in phosphogypsum stacks in reducing the amount of water to be treated at stack closure. The Institute also funded the development of a pretreatment method coupled with reverse osmosis that was used to treat water during an emergency situation at Piney Point when acidic water was close to overflow levels. We have also examined the effectiveness of neutralization techniques in the event of an acidic water spill.

Dicalcium phosphate product recovered from process water treatment.

Clay Settling Areas - Phosphate rock in Florida is found in association with clay. The clay must be removed before the rock can be processed and converted into products. The clay is separated as slurry and is stored in ponds where the solid clay particles settle out over many years. The Institute's research is helping to understand how the clay settling areas impact the surface and groundwater flow in the watershed around them and how they can be reclaimed and put to environmentally sound and economically viable use. Past work investigated stabilization of phosphatic clay using lime columns for construction stability. Waste clay has been studied for use as a green building material in tiles, lightweight aggregates, and other construction materials.

Research into techniques to rapidly dewater the clay is the key component to clay pond reclamation. Recent work focused on use of a deep cone paste thickener for clay consolidation that could dewater clay in minutes rather than decades.

Deep cone pilot plant with thickened clay paste.

Phosphogypsum Management – The phosphate chemical processing plants produce about 5 tons of calcium sulfate (gypsum) for every ton of phosphoric acid. This gypsum is known as “phosphogypsum” due to its origin and the small amount of phosphate it contains. Phosphogypsum contains most of the radium that was contained in the beneficiated rock used to produce it. The Institute has conducted many studies on safe and beneficial uses of phosphogypsum and has gathered international studies as well as evidence of its use as an item of commerce.

Many uses of phosphogypsum have been directly studied at this Institute including use as a road base, marine barrier and oyster cultch, construction material, in the manufacture of glass and glass/ceramic products, as a daily cover in landfills to enhance microbial digestion of municipal wastes, in agriculture, and as a soil amendment.

Spreading asphalt over a phosphogypsum road base.

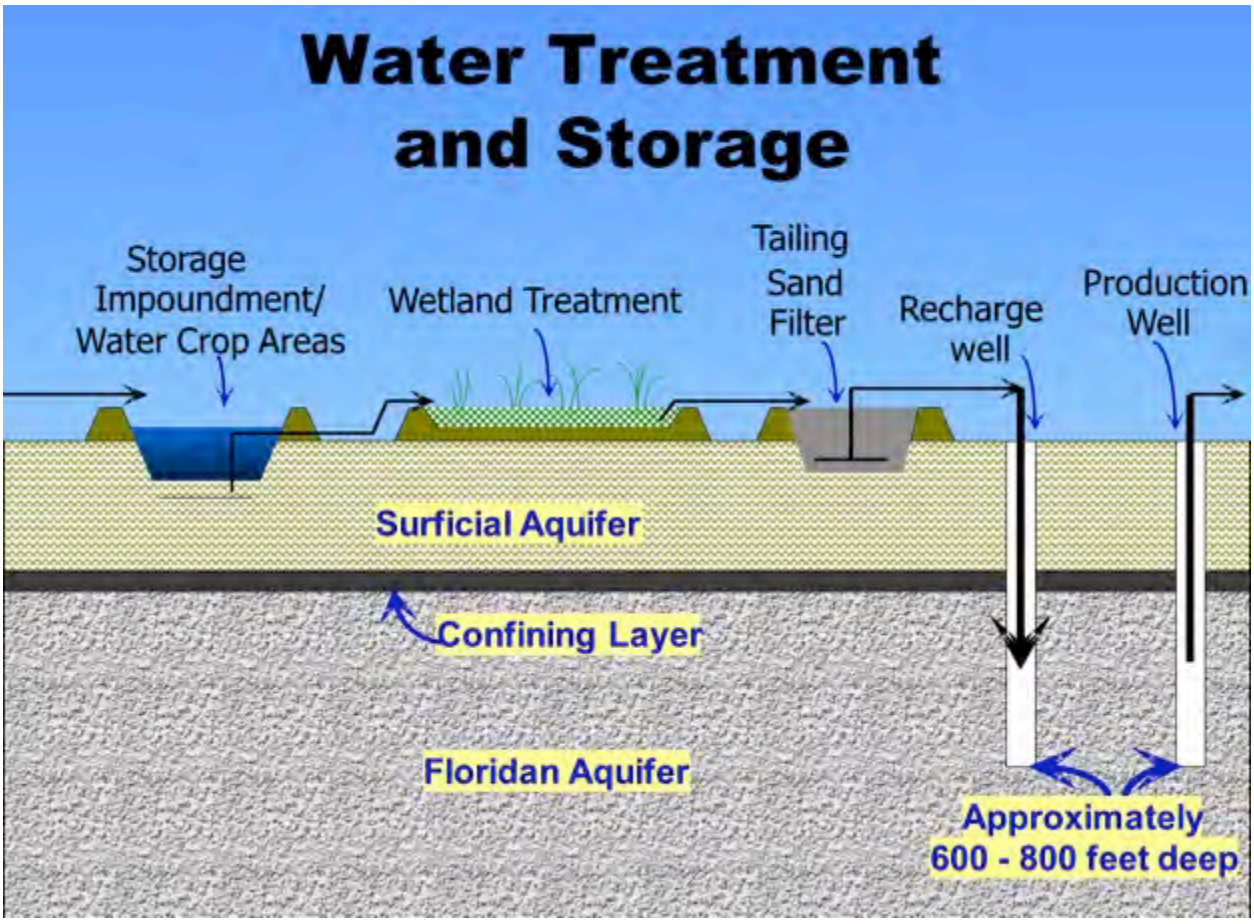
Reclamation Science – Reclamation may be defined as the process of returning mined land to a useful condition. The process includes reshaping the land and establishing vegetation. For natural systems, native plants must be established and exotic plants or weeds must be controlled. The public has expressed concerns about the value and potential/possible uses (both economic and ecological) of mined lands, the quantity and quality of surface and ground waters, and the restoration of natural ecological and hydrologic systems. The mining industry is concerned about obtaining mining permits (which require a reclamation plan) and achieving the reclamation and environmental requirements in a cost-effective manner. More recently, the possible ecological, hydrologic and economic impacts of lakes and reservoirs on reclaimed lands have come under increased scrutiny. The Institute continues to improve reclamation technologies, and to measure the success of reclamation efforts.

Tree farming on clay settling areas.

Energy and Water Conservation – Energy use is frequently discussed in research proposals to the Institute; mainly because energy cost may determine if a new technology can be economically applied. In addition, sulfuric acid production generates heat, which is used to generate power at the chemical processing site. The Institute has been investigating ways to recover much more energy from this process.

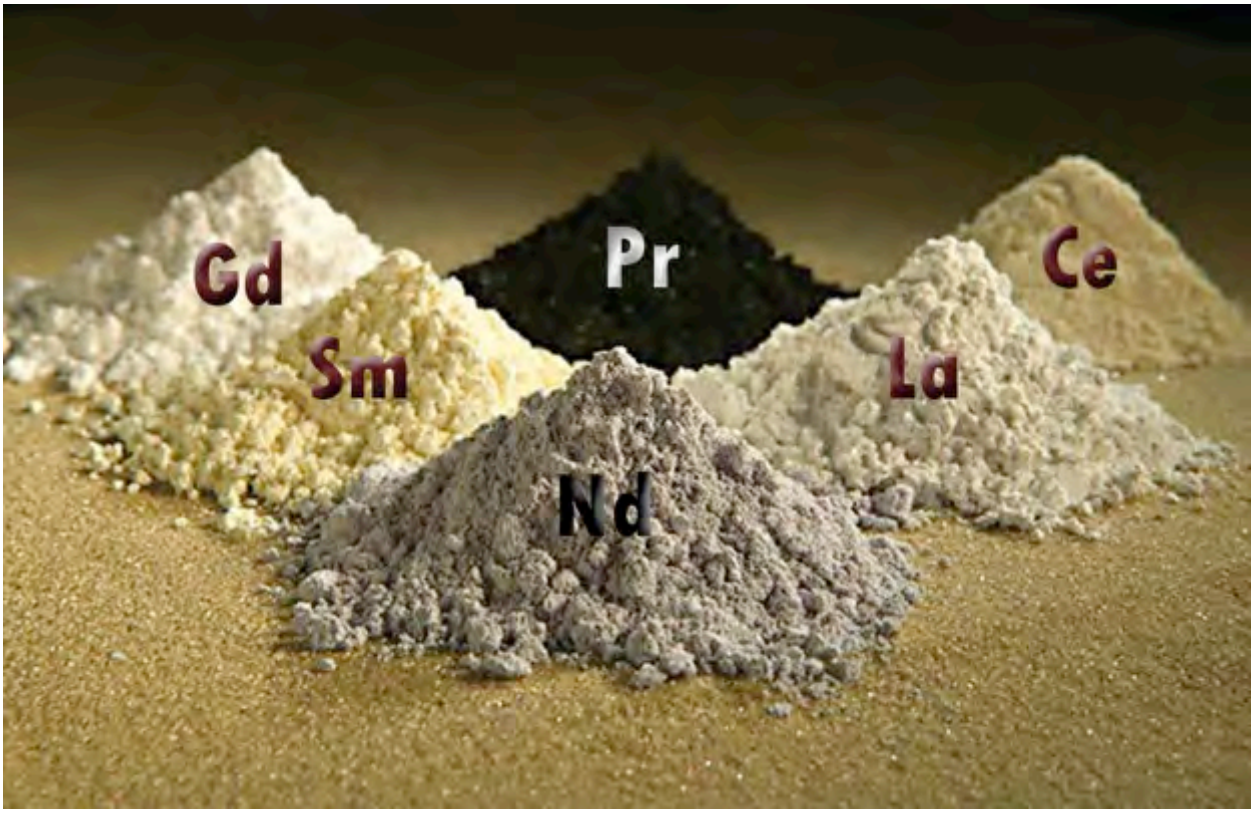
Water consumption and conservation is often of wider interest. The phosphate industry pumps and uses large volumes of fresh water as a necessary part of its operation, both on the mining and beneficiation sites and the chemical processing sites. Groundwater withdrawal led to serious effects in the past and is still a matter of contention. The industry has made tremendous strides in water recycling, but improvements can be made to reduce the amount of water needed. The Institute has studied ways to reduce or replace groundwater consumption for the industry.

Concern for water quality goes hand-in-hand with water conservation. The Institute has funded extensive research of the use of mined lands in water treatment. This research has been successfully applied on a large scale by the industry.



Water treatment using wetlands and sand tailings.

Resource Recovery – Mining has environmental and economic consequences. In order to optimize both, we should recover as much of the useful material as practical. In addition to phosphate rock, which is essential to our food security, uranium and rare earth elements are also present in the mined matrix and may be economically extracted to enhance our energy and national security. In this report, you will see that we are heavily involved in rare earth assessment and extraction research using our own funding and as part of an elite national team funded by the US Department of Energy.



Photograph by Peggy Greb, USDA, Agricultural Research Center

Six of the seventeen Rare Earth Elements (REE).

The Institute has conducted research to find new technologies to: separate phosphate rock from clay and sand, rapidly settle clays and recover water, determine what rock should be left in the ground and what should be mined, remove dolomite from rock, control nuisance and exotic plants, describe the hydrology of clay settling areas, use phosphogypsum in safe and beneficial ways, recover uranium and rare earth elements, assess sulfuric acid mist exposure in the workplace, describe natural radioactivity in the pre- and post-mining environment, and evaluate hazardous air pollutants in communities near processing plants.

The Institute's mission includes sharing the information it generates and collects. Toward this end the Institute routinely interacts with all stakeholders, hosts technical conferences, workshops and meetings, operates a library that is open to the public, and conducts a Kindergarten-Grade 12 education program. As our information program expands we are always looking for new ways to share our wealth of information. This report is part of that effort and is available through our web page (www.fipr.state.fl.us).

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, and was 6.2% in fiscal year (FY) 2013-2014. The tax rate for FY 2013-2014 was $1.61 per ton of rock severed.

Mine permitting has been a contentious issue between the industry and other entities, and the severance tax is based on mining production. The factors affecting the total tonnage of rock severed from Florida's lands also create uncertainty for planning and management of the research and operational budgets. However, the Institute strives to provide scientifically sound information to all of its stakeholders with the resources it is given.

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, 2013	$8,208,903
June 30, 2014	$8,399,762

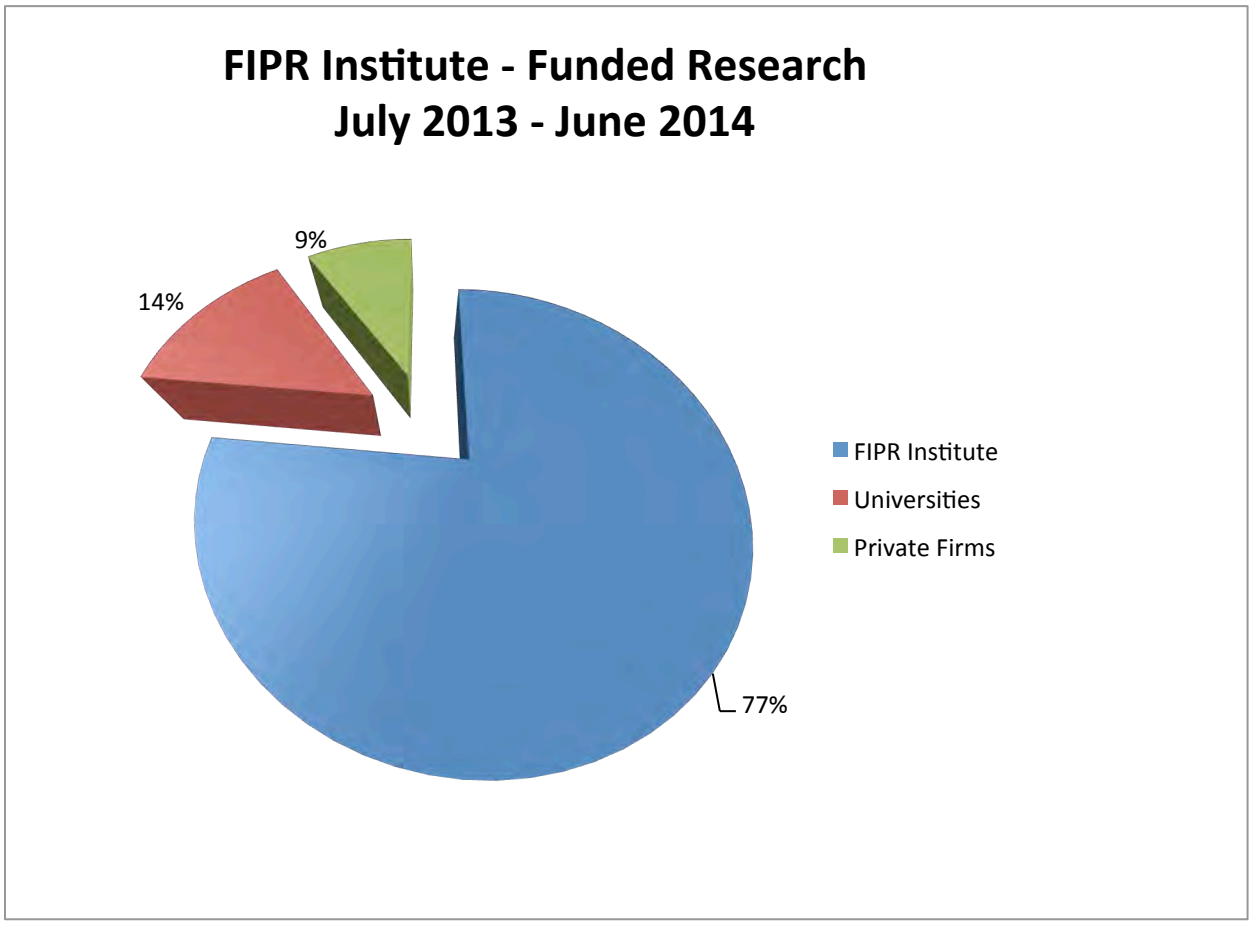
Operations

Income	
Gross Severance Tax[1]	$2,206,884
LESS Fees to Dept. of Revenue	($218,685)
Net Income	$1,988,199
PLUS Interest	$56,359
PLUS Residual Transfer from USF	$79,747
Total Income	$2,124,305

Expenses	
Research	$295,312
Internal Operations[2]	$1,638,134
Total Expenses	$1,933,446
Change in Trust Fund	$190,859

[1]Per Section 211.3103, F.S. the severance tax rate was $1.61 per ton of rock severed from the earth. The FIPR Institute received 6.2% of the proceeds from the severance tax.

[2]"Internal Operations" includes staff salaries and benefits, and expenses for the Education Program, Library, and Laboratories. The Institute's Research Directors and technicians (52% of the staff) spend most of their time directing or conducting research. Of the remaining staff, 24% deliver information and education services to the public and schools, and another 24% provide office and IT 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.



FIPR Institute research projects approved July 1, 2013 to June 30, 2014 by percentage of total funds awarded to universities versus private firms.

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, 2013	$84,786
June 30, 2014	$650,290
Change in Auxiliary Account	$565,503

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, Business Networking International (BNI), and the Bartow Chamber of Commerce use the FIPR Institute Conference facilities for their regular weekly and monthly meetings.

Three of the Institute's employees, Shannon Medley, Marie Wilmot, and Aaron Medley, are graduates of Bartow Leadership Program and are active in the Bartow Chamber of Commerce.

- FIPR representatives provide information about the Institute and its connection with Florida Polytechnic University.
- Two staff members, Shannon Medley and Marie Wilmot, are Chamber Ambassadors who help recruit and retain chamber members and promote functions.
- Shannon Medley is on the Bartow Chamber Board of Directors serving as Vice President of Public Affairs, and is Chair of the Bartow Leadership Alumni. Shannon was recently selected as Class Chair of the Bartow Chamber's upcoming two-year Leadership Program. She also serves as Day Chair for Leadership Polk's Bartow/ Ft. Meade/ Mulberry Day.

Awards and Achievements

There were no specific awards bestowed upon the Institute's staff during the fiscal year. However, they continue to serve on prestigious panels and committees, and their advice and counsel is in constant demand internationally.

Dr. Brian Birky is Convener of the NORM Task Force (Expert Panel) of the International Fertilizer Industry Association (IFA) Technical Committee. He and Dr. Patrick Zhang are also 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. Patrick is an Organizing Committee member for the 1st International Conference: Rare Earth Minerals/Metals – Sustainable Technologies for the Future, and served as Session Chair for the 7th International Conference on Rare Earths Development and Application.

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.

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 expansion of research topics beyond phosphate into related areas will become incorporated into the Information Program work areas to support and reflect research activities. The Institute uses social media to share information about its activities and promotes the websites www.fipr.state.fl.us and www.floridapolytechnic.org.

FIPR Institute has cooperated with Florida Polytechnic to incorporate the Institute's website as an integral part of the University website. The updated FIPR Institute site will have design features and elements consistent with Florida Polytechnic's site. This highlights the Institute's firm commitment to the growth and development of the University's academic and research programs. Development of the site continues and will deploy early in the next fiscal year.

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.

In a further effort to incorporate the Institute into the operations of Florida Polytechnic, FIPR Institute Library staff has worked with the University Library Staff and the Florida Virtual Library Center in Gainesville to transfer the FIPR Library catalog holdings and circulation to form the basis of a physical collection for Florida Polytechnic. In addition, the Institute's Library Director, Karen Stewart, has provided guidance to the University on the development of its virtual collection.

The K-12 Education Program continues to expand its focus to not only train teachers, but also deliver resources and content directly to classrooms in the state. In the past year, the Institute's educational resources have been brought to hundreds of classrooms, reaching thousands of Florida students directly. In addition, the variety of curriculum offered continues to grow as lesson plans, activities, and teaching units are added as a result of contributions by the Institute's staff and community teachers.

The FIPR Institute's K-12 Education Program activities for the fiscal year are summarized below.

- The FIPR Institute mentored award winning science fair students in the community.
- The FIPR Institute Education Program is constantly visiting public and private schools throughout Florida and emphasizes STEM education.
- The Institute continues to expand its offerings of STEM curriculum.
- Gary Albarelli, the Institute's Information Program Director, is on the Board of Florida Ag in the Classroom.
- Bartow Elementary Academy lists the FIPR Institute as a Corporate Partner for the school's STEM Lab and Garden.
- The Institute is involved in a variety of community education efforts. For example, Boktoberfest at Bok Tower Gardens, Central Florida Eco Tours, etc.
- The Institute hosted a STEM, STEAM, and STREAM spring break workshop that incorporated the fine arts and language arts into STEM curriculum.
- Staff served as Boy Scout merit badge counselors for STEM activities.
- The Institute's Education Coordinator, Ms. Indira Sukhraj, participates in a variety of community organizations:
    - Mulberry Phosphate Museum's Advisory Board
    - Polk Regional Science Fair's SRC (Scientific Review Committee)
    - Involved with LE/AD (Lakes Education Action Drive) to educate the public about phosphate in the environment
    - Aquatics expert for Tampa Bay Regional Envirothon
    - Reviews grant proposals for National Science Teachers Association (NSTA)
    - Served as judge for the International Science and Engineering Fair (ISEF)
    - Gave presentation on how to incorporate STEM into curriculum at Florida Association of Science Teachers (FAST) conference.

Technical Exchange

Journal Articles

- *Control and Management of Cogongrass and Other Exotic Grasses on Disturbed Lands in Florida.* FIPR Publication
- *A New Collector - Activator Scheme for Low Grade Phosphate Flotation*. Under review by International Journal of Mineral Processing
- *Upgrading Phosphate Flotation Tailings for REE Extraction.* Under review by Minerals and Metallurgical Processing Journal
- *REE Extraction from Wet Process Products: Phosphoric Acid, Phosphoric Acid Sludge and Phosphogypsum*. Under review by Hydrometallurgy
- *REE Extraction from Phosphate Rock: In-line REE Extraction from Wet Process*. Under review by Chemical Engineering and Processing: Process Intensification
- *Development of a Personal Sampler for Simultaneous Sampling of Gases and Aerosols.* Under review by Annals of Occupational Hygiene
- *Impacts of Hazardous Air Pollutants Emitted from Phosphate Fertilizer Production Plants on their Ambient Concentration Levels in the Tampa Bay Area*. Under review by Air Quality, Atmosphere & Health
- *A New Collector - Activator Scheme for Low Grade Phosphate Flotation*. Under review by International Journal of Minerals Processing
- *Upgrading Phosphate Flotation Tailings for REE Extraction*. Under review by Minerals and Metallurgical Processing Journal
- *REE Extraction from Wet Process Products: Phosphoric Acid, Phosphoric Acid Sludge, and Phosphogypsum*. Under review by Hydrometallurgy
- *REE Extraction from Phosphate Rock: In-Line REE-Extraction from Wet Process*. Under review by Chemical Engineering: process intensification Journal

Meetings and Presentations

FIPR co-organized a workshop with CMI at FIPR on uranium and rare earths from phosphate, which took place January 16-17, 2014. Over 40 people participated in the workshop, representing 4 national labs, 3 phosphate companies, 5 universities, and several engineering firms and consultants. This workshop identified challenges and

opportunities, and brainstormed strategies for recovering REE and uranium from four phosphate processing products and byproducts: amine flotation tailings, waste clay, phosphogypsum and phosphoric acid.

FIPR Institute staff members have been actively involved in technical exchange at various forums during the last several years, as highlighted below:

- Presentation to teachers: *Rad 101* (Radioactivity and Radiation) July 25, 2013
- Panel Session of Summer Workshop, July 26, 2013
- *Comprehensive Recovery of Valuable Elements from Phosphate*. Presented at the 7th International Conference on Rare Earth Development and Application, Ganzhou, China, August 10-13, 2013
- IFA NORM Working Group, Istanbul. Convenor and presenter. Presentation: *Overview of Industrial NORM in the USA with Emphasis on the Phosphate Industry,* September 26-28, 2013

- *Prescribing the Right 'Sleeping Pills' for the Phosphogypsum Stack Managers.* Presented at the 2013 Annual Regional Phosphate Conference, Lakeland, FL, October 9-10, 2013

- Doctoral Committee, Ms. Lin Shou dissertation defense, UF. *Diffusional Release of Pollutants into Ambient Air and Method for Enhancing Diffusional Collection in Air Sampling,* October 29, 2013

- UF Geotechnical and Waste Management Program, *Phosphogypsum Stack Management and Utilization,* October 2013

- FAMU School of the Environment, *Environmental Policy and Sustainability in the Phosphate Industry*, November 2013

- IAEA Fellowship, Amine Fourati, Uranium and REE Recovery from Phosphoric Acid, January 13-17, 2014

- CMI Workshop, Hosted Team Member Workshop, January 16-17, 2014

- *Reducing MgO Content in Florida Phosphate Concentrate*. Presented at the 2014 SME Annual Meeting, Salt Lake City, Utah, February 23-26-2014

- Bradley Public Meeting, New Wales Gyp Stack Expansion, February 25, 2014

- Mulberry Public Meeting, New Wales Gyp Stack Expansion, February 27, 2014

- GIW International Pumping Class, March 14, 2014

- Mosaic Manatee ICAP, *Phosphogypsum Stack Management and Utilization,* March 27, 2014

- Bartow Leadership Phosphate Day, Presentations: *FIPR Overview* and *Natural Radioactivity and Public Health in the Florida Phosphate Mining Region,* March 20, 2014

- Polk Leadership, Presentation: *Phosphate 101*, April 24, 2014

- *Reducing MgO Content in Florida Phosphate Concentrate*. Presented at the 2014 SME Annual Meeting

- IFA Technical Symposium with NORM Working Group Meeting, Amsterdam, March 31-April 3, 2014

- Florida Exotic Pest Plant Council Conference, *Selective Control of Invasive Exotic Grasses,* April 29, 2014

- *Phosphogypsum Valorization and Water Consequences*. Florida Association for Water Quality Control, Naples FL, June 11, 2014
- *Uranium and REE Recovery from Florida Phosphates – Looking Back and Going Forward*, Presented at the International Symposium on Uranium Raw Material for the Nuclear Fuel Cycle: Exploration, Mining, Production, Supply and Demand, Economics and Environmental Issues, June 23-27, 2014, Vienna
- IAEA Consultancy Symposium on Uranium Production from Phosphate Rocks. IAEA Headquarters, Vienna, June 30- July 3, 2014

Recent FIPR Institute Publications

- Update of *Radiation in the Phosphate Industry*, set of 3 DVDs divided into 15 Lessons
- 02-184-250. New Mobile Pre-Processing Equipment for Florida Phosphate Mining, Dr. Hagen Muller, HAVER Engineering GmbH. June 2014

Research Areas

Projects were funded from the Phosphate Research Trust Fund and managed by the FIPR Institute. Some projects were conducted in-house while many were conducted by various universities and private companies. FIPR Institute Research Directors serve as Contract Managers for all projects. Projects that were completed, ongoing, or initially funded during the fiscal year are described in the following text and organized according to the Institute's research areas.

Research projects, however, often pertain to more than one of the Institute's research areas because they have components that fit under more than one heading. For example, research on phosphogypsum, a by-product of phosphate fertilizer production, may include chemical processing of the phosphate, production and treatment of the process water, use of the by-product, public health, and reclamation of a closed phosphogypsum stack.

The Institute's Strategic Plan, available on our web site, covers the period 2011 to 2016 and was presented to the Institute's Advisory Board and the public in January 2011. After revision, its adoption was recommended by the Board at the January 2011 meeting and it was adopted on February 17, 2011. It discusses goals and approaches to achieve them in each of the Institute's research and programmatic areas. Although substantial progress has been made towards achieving these goals, more work is still needed. In addition, the plan contains new goals and approaches applicable to non-phosphate work.

Progress made in this fiscal year in Mining and Beneficiation, Chemical Processing, Public & Environmental Health, and Reclamation is described in the pages that follow. 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 provided in categories of completed projects, ongoing projects, and projects funded this fiscal year. In each category, the projects are described by title, funded organization, and a brief description of the objectives and accomplishments.

Mining and Beneficiation

Completed Projects

Innovative RTS Technology for Efficient Separation of Dolomite from Phosphate
University of Kentucky

The goal of this project is to develop and evaluate an innovative high efficiency triboelectrostatic separation technology, referred to as a rotary triboelectrostatic separator (RTS), for beneficiation of dolomitic phosphate samples without use of any chemicals or water. The RTS technology is based on differential surface charge acquired between phosphate and gangue minerals such as dolomite and quartz upon their mutual contact or friction with a charging medium or charger. This allows separation in an external electric field resulting from different motion trajectories for different particles.

Although charge density measurements showed large difference between dolomite and phosphate, the actual separation tests failed to achieve the proposed objectives.

Ongoing Projects

Remote Real-time Industrialized Analyzer of Phosphate Rock
R Squared S, Inc.

A decade-long research collaboration between Dr. Michael Gaft of Laser Distance Spectrometry (LDS) of Israel and the FIPR Institute has resulted in the development and commercialization of the world's first on-line analyzer for wet minerals using the laser-induced breakdown spectroscopy (LIBS) technology already deployed in locations around the world. The FIPR Institute and the Israeli research team have also set the goal long ago of developing a LIBS-based analyzer for remote analysis of ores and overburden before these materials are dug out or while in transit to the beneficiation plant. The application of ore evaluation in the exposed mine face and in the dragline bucket can increase reserves by including and screening phosphate deposits with high Mg content from southern Florida in economically viable production. Because of selective excavation and dumping, most of the dolomite will be separated near the open pit, thus reducing ore transportation costs significantly. Additional advantages may be the lowering of use energy and flotation reagents.

In this summer, the first industrial scale remote LIBS module was shipped to an operating phosphate mine of Mosaic. Field evaluation of the analyzer demonstrated its capabilities for multi-element analysis from distance as long as 25 meters. The analytical data from the remote LIBS correlated well with lab results.

Industrial prototype remote LIBS analyzer.

Isolation and Characterization of RE Mineral Particles in Florida Phosphate Rock by DE Rapid Scan Radiography and HRXMT

University of Utah

This research was designed to provide guidance for developing mineral separation and metal extraction technologies for recovering REE from Florida phosphate. Two advanced mineral characterization techniques are used in this research: high resolution X-ray micro tomography (HRXMT) and dual energy (DE) rapid scan radiography. Three sample streams, shaking table concentrate, acid plant feed, and phosphogypsum, were separated into three size classes: >106 μm, 75-106 μm, and 53-75 μm. DE radiographs

were taken at two energy levels and the ratio calculated. The images were thresholded to show only potential rare earth particles and then those particles were removed to prepare HRXMT samples. The samples were digitally reconstructed and the concentration of rare earth particles found using digital processing software. The overall concentrations for the three size classes were found to be 2157 ppm in the shaking table concentrate, 104 ppm in the acid plant feed, and 284 ppm in the phosphogypsum, respectively. Based on the degree of liberation, the best particle size to find fully liberated REE-containing monazite particles is 75-106 µm, although other sizes can reasonably be considered for acid plant feed and phosphogypsum. The ongoing project is determining the occurrence of REE in tailings & waste clay, identifying and quantifying REE minerals in phosphate rock, characterizing REE forms in phosphogypsum, and investigating liberation of REE minerals in ground and unground phosphate rock.

Projects Funded This Fiscal Year

Recovery of REEs and Uranium from Phosphate Ore Processing
FIPR

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). FIPR is a member of CMI undertaking the project on recovery of uranium and REE from phosphate mining and processing products as well as byproducts. All the CMI members are shown in the figure above.

In less than a year, FIPR has made significant progresses in concentrating REE from flotation tailings and waste clay.

Continuous separation of waste clay using a hydrocyclone in pilot testing.

Improving the Dolomite Flotation Technology for Florida Phosphate Pebbles: Removing the Last Hurdle to Commercialization

FIPR-Bluestar Lehigh Engineering Corporation

In collaboration with Jacobs Engineering, Mosaic and its predecessor IMC, and China Bluestar Lehigh Engineering Corporation (Bluestar), FIPR has developed a feasible technology for processing the high-dolomite phosphate pebbles in Florida. The process involves grinding the pebbles to liberate dolomite, dolomite flotation with phosphoric acid as a phosphate depressant, and silica flotation. In 2000, pilot testing was conducted on two pebble samples with Pebble #1 analyzing 3.5% MgO and Pebble #2 containing 2.8%, achieving more than 67% MgO removal at over 75% P2O5 recovery. Feasibility analysis by Jacobs Engineering demonstrated both the technical and economic feasibility of the dolomite flotation process. For a 300 tph battery limits beneficiation plant employing the FIPR beneficiation process, the construction cost was estimated to be 32 million dollars with a total operating cost of $15.62 per ton of concentrate. These numbers were considered to be very competitive.

Encouraged by these results, Mosaic conducted further testing and had decided to build a plant for processing their high dolomite reserves at the Four Corners mine. However, the project was suspended indefinitely due to the concern about treatment cost for P-containing waste water.

This new project is designed to further improve the dolomite flotation technology thus providing the industry with more confidence in commercializing the process. The proposed research includes the following five major components: 1) water balance analysis for the dolomite flotation plant to determine whether the flotation water could be recycled 100%; 2) modification of the dolomite flotation process by using less or zero phosphoric acid; 3) evaluation of strong phosphate depressants; 4) feasibility and environmental analysis; and 5) technology transfer workshop.

The project has achieved new breakthroughs in the following three aspects:

1. Dolomite flotation without using phosphoric acid
2. A new, more selective dolomite collector that performs well in sulfuric acid only
3. A new phosphate depressant, which could improve P2O5 recovery by over 3%.

Extraction and Recovery of Rare Earth Elements from Phosphate Using PX-107 and Chelok® Polymers

Periodic Products, LLC

Periodic Products has developed a series of unique, insoluble, non-toxic, biodegradable polymer compounds for removing metals from water. These products have the following features: 1) high adsorption capacities, 2) easy separation of metals from the polymer, 3) amenable to solution of different pH values, and 4) capable of removing +1, +2, +3 and +4 ions. In addition, Periodic Products has developed some proprietary aqueous based leaching agents for extracting metals from solid materials.

In 2013, Periodic Products initiated an in-house research program on REE extraction from phosphogypsum, and developed a two-step technology. In the first step, dry PG was ground and extracted with their proprietary extraction solution, PX-107, achieving 72-100% dissolution. In the second step, the leaching solution was treated with Chelok® Polymers to remove REE from the solution, resulting in removal of 94-100% of the dissolved REE from PG. Preliminary economic analysis showed positive return on investment in the technology. Preliminary experiments using waste clay and flotation tails also achieved encouraging results.

The FIPR Board awarded Periodic Products a grant to demonstrate the efficacy of their technologies on various intermediate products and by-products of phosphate mining and phosphoric acid production, including phosphate rock, phosphoric acid, waste clay, flotation tailings, and phosphogypsum.

Chemical Processing

Progress in chemical processing research was hampered by both the vacancy of the Research Director's position. Nevertheless, progress has been made in fulfillment of the Institute's mission. We have made a concerted effort to upgrade the Institute's laboratories to support research in chemical processing as well as ore characterization and beneficiation. Laboratory facilities include an Analytical Chemistry Lab (with a wet bench chemistry room and a separate analytical instrument room), and a Metallurgical Lab (with an adjacent secure outdoor compound). Instrumentation and procedures have been updated and enhanced to improve the range and quality of analyses we perform.

A partial list of analytical capabilities and instruments includes:

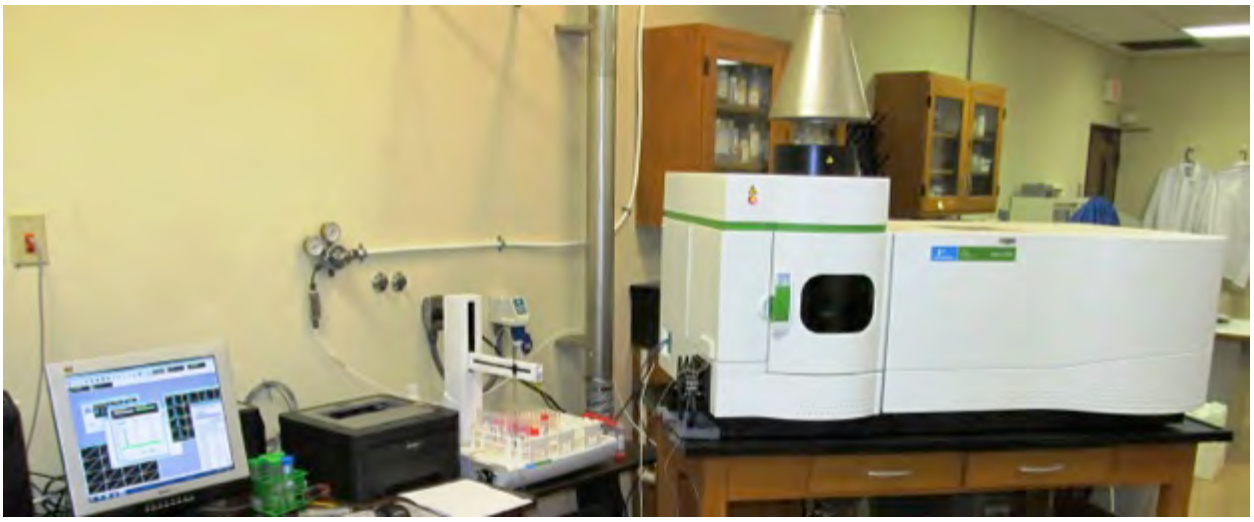
- Sample preparation: homogenization, cone and quartering, Bico rock crusher (Jaw crusher), grinding (Bico-Braun ball or rod mill and Bico-Braun Pulverizer/grinder), Blue-M drying ovens, Tyler Ro-Tap, sieve screening (wet & dry), riffle sample splitter
- IEC Centrifuge
- pH/ISE meter
- X-ray diffraction
- Petrographic microscope analysis
- P2O5 and CaO analysis (Lachat QuickChem 8500 Series 2 Flow Injection Analyzer)
- Inductively Coupled Argon Plasma (ICP) analyses (Perkin Elmer Optima 8300 Spectrometer)

Special sample preparation and/or procedure methods include:

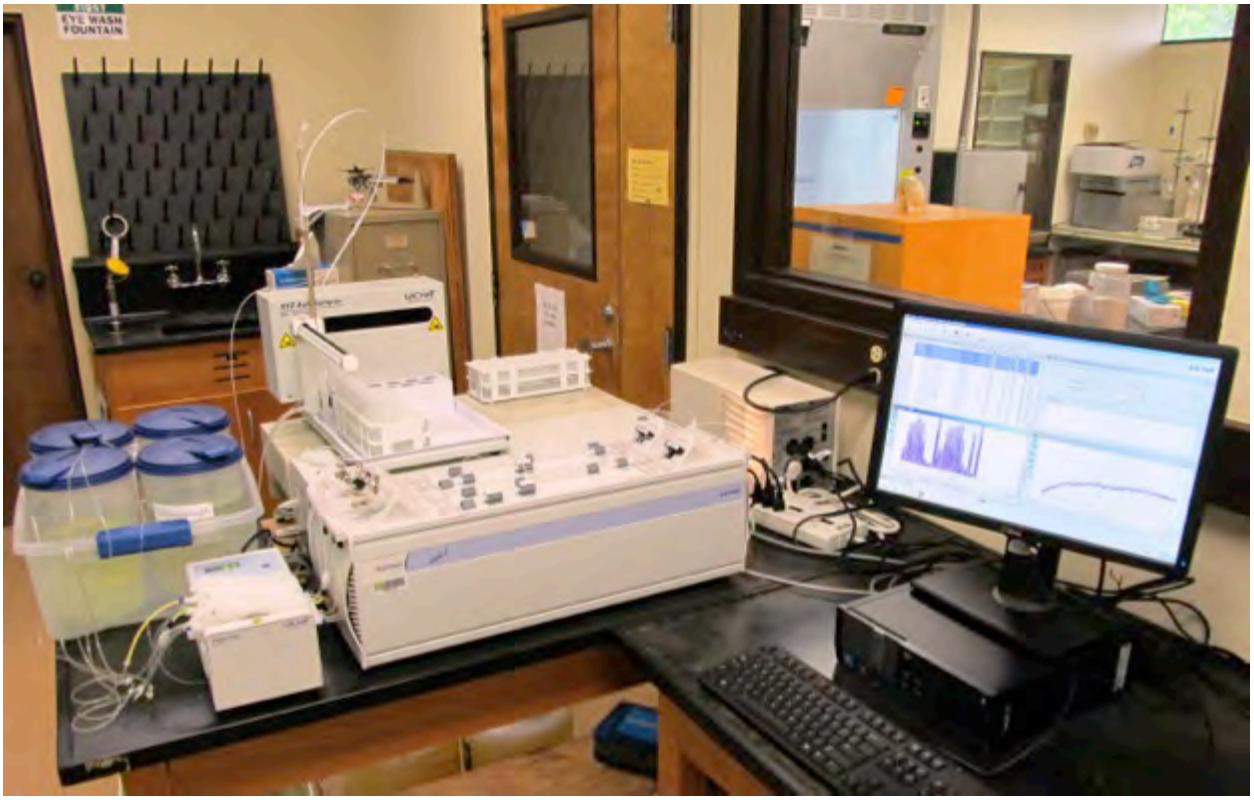
- Heavy mineral separation
- Magnetic separation
- Electrostatic separation
- Sonic sieving
- Cyclosizing
- Insol determinations
- ICP select elements (including uranium and rare earth elements)

Chemical analyses are performed with the most recent methods described in the Association of Florida Fertilizer and Phosphate Chemists (AFPC) manual. Only minor modifications are made to accommodate instrumentation available at FIPR Institute, but quality assurance is verified using controls and standards. Reagents are ACS reagent

grade and volumetric glassware is class A, as required. Analytical results of phosphate rock samples are monitored by analyzing AFPC-certified Check 22 standards along with the project samples. Spikes are incorporated to monitor recovery of cations by the ICP.



Perkin Elmer Optima 8300 Spectrometer (ICP-OES).



Lachat QuickChem 8500 Series 2 Flow Injection Analyzer.

Completed Projects

Characterization of an Undersea Phosphate Deposit

FIPR: funded by Odyssey Marine Exploration, Inc.

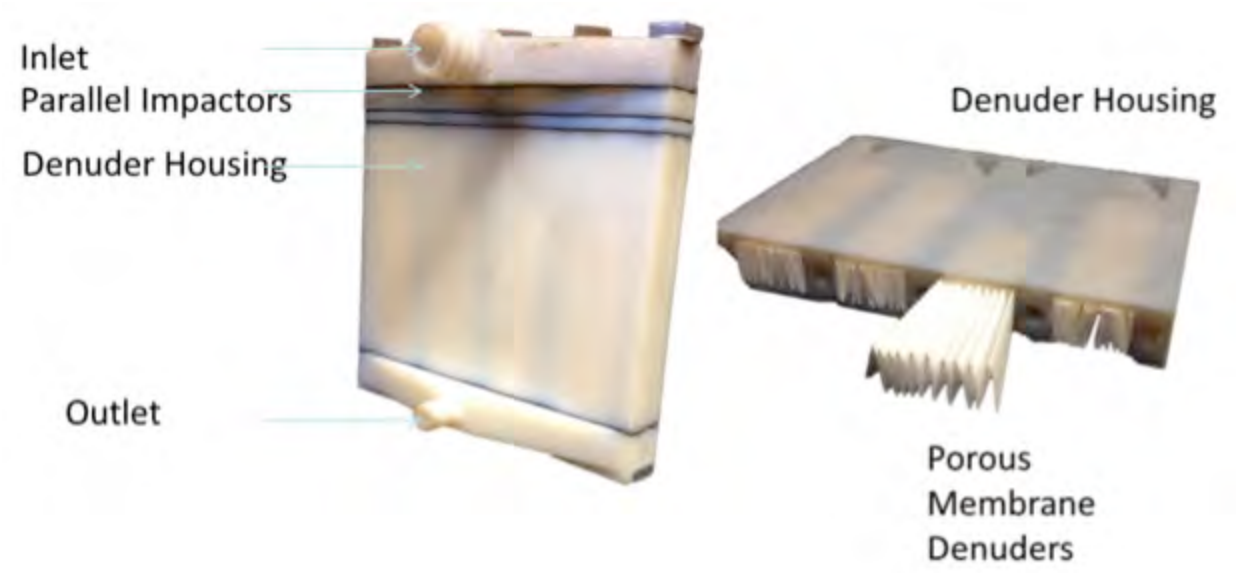
The Institute worked with Odyssey Marine Exploration, Inc. throughout the fiscal year to characterize marine sediment core samples from the Oceanica deposit controlled by Odyssey's subsidiary, Oceanica Resources, S. de. R.L. Over 760 total core samples representing a portion of the mineral deposit were tested at the Institute. The measured and indicated phosphorite resources total 327.2 million tonnes of ore at 18.5% P_2O_5. Assaying and metallurgical testing of the core samples at approximately one meter intervals indicates the potential to produce a phosphate rock concentrate at 28% to 30% P_2O_5 with a favorable CaO/P_2O_5 ratio of 1.5 to 1.55 and a Minor Element Ratio (MER) of 0.07 to 0.08. The chemical analysis suggests that the concentrate would be suitable for the production of phosphoric acid using the wet process methods.

The Dorado Discovery was used to gather marine sediment core samples.

Ongoing Projects

Commercial Development and Validation of a Disposable Personal Sampler for Inorganic Acid Mist Measurement

University of Florida

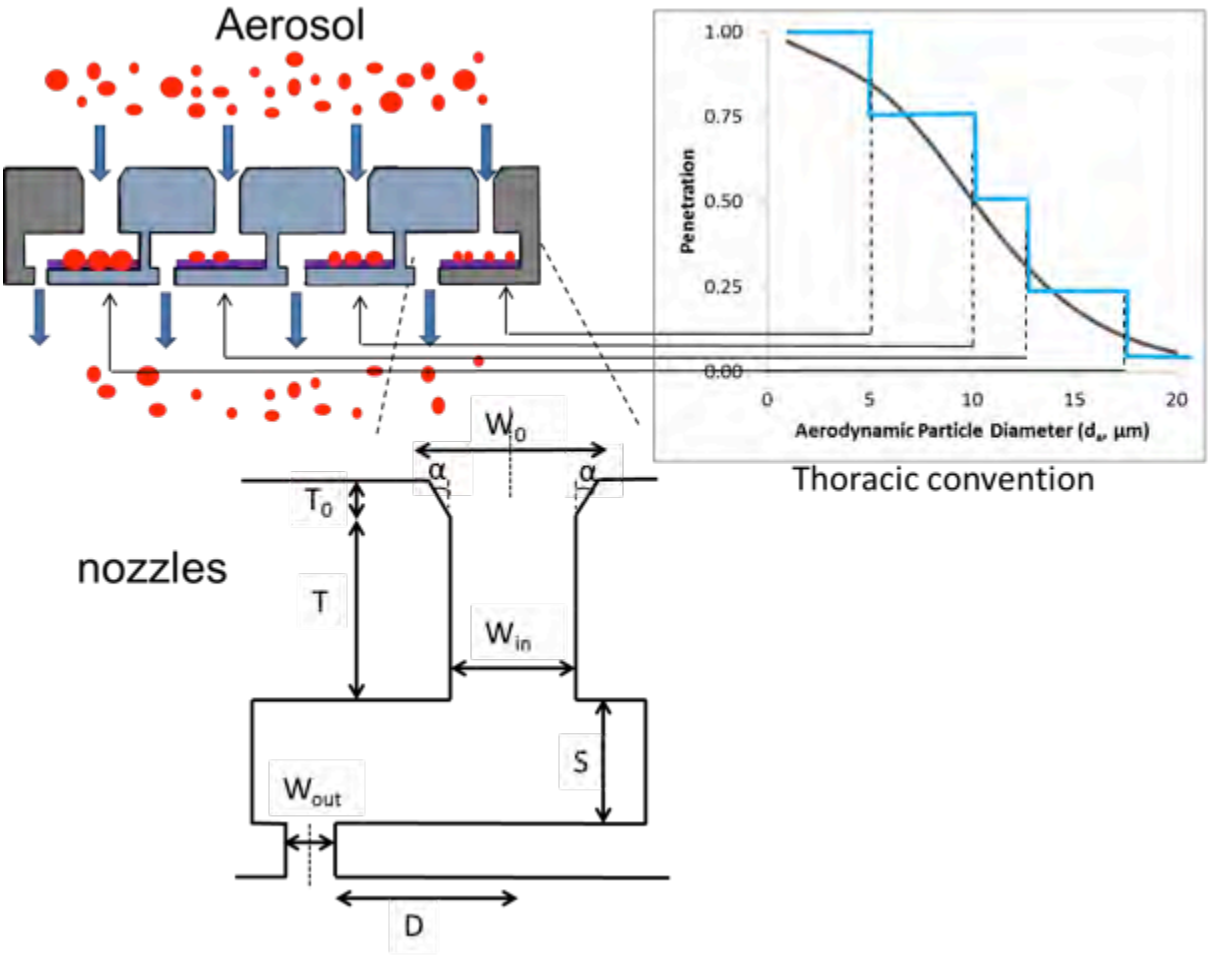


Experimental personal air sampler.

This is a continuation of previous research requested by the phosphate industry in response to new lower exposure limits that were proposed based on decades old measurements that are not representative of the modern facilities. A series of studies were conducted. The first study indicated that sulfuric acid mist exposures to Florida phosphate industry workers are below the new, more restrictive standards. However, the standard method used to measure sulfuric acid concentration overestimates those concentrations in sulfuric/phosphoric acid manufacturing plants and could result in reports of excessive exposure when they have not actually happened. Subsequent studies identified the physical sources of the errors and described methods to exclude them and correct the method. Finally, a porous membrane denuder (PMD) that excludes interfering aerosols from the sampling stream to give a more realistic measurement of the acid concentration in air was designed, constructed and patented.

The current study is designed to transform the FIPR personal sampler prototype into a ready-to-manufacture commercial sampler. Modifications that are critically important to

the commercialization of the sampler include a completely disposable and ergonomic design, and adaptation to the new thoracic fraction standards set by European Union. The commercial prototype will be validated by scientists at OSHA's Salt Lake City Testing Center. In addition, the project will explore the sampler's feasibility for ammonia adsorption to expand its capability from acidic to basic gases. The current design, PMD IV, has the capacity to absorb 10 ppm SO_2 for 8 hours and co-sample HNO_3, HCl, and SO_2 for 4 hours. SO_2 is almost entirely collected by denuders. No SO_2 interference occurs in other parts of the personal sampler. Such interference would result in overestimation of the actual exposure to sulfuric acid mist in the workplace.

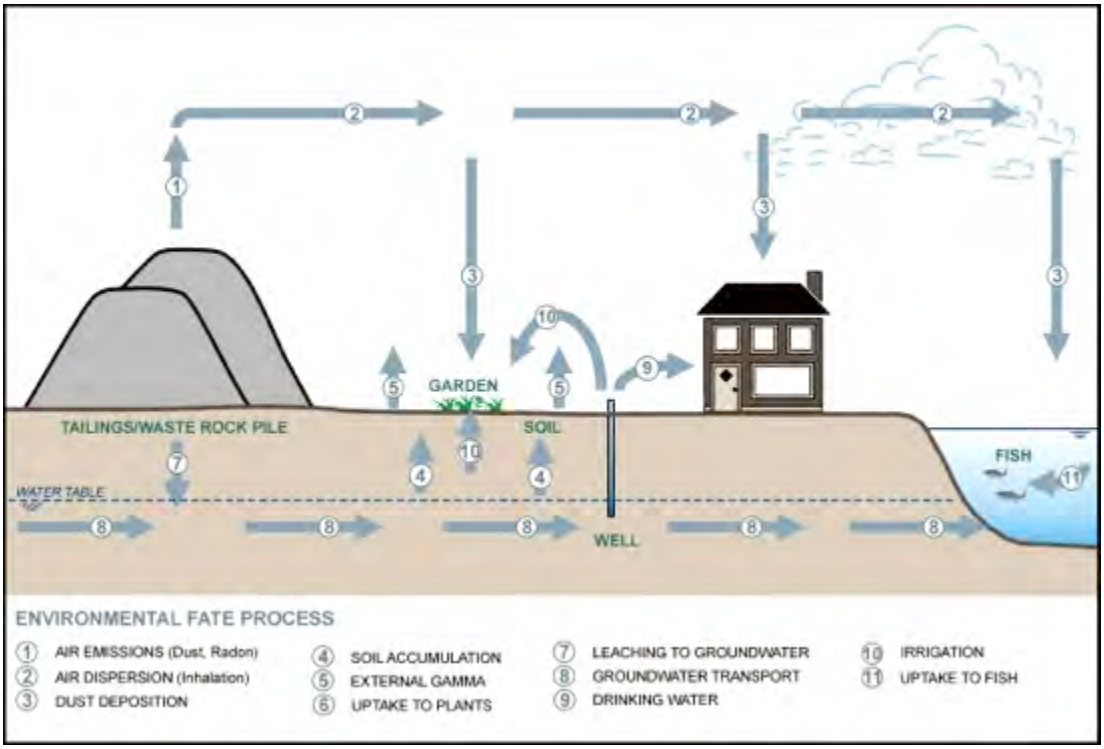


Conceptual Design of the FIPR thoracic sampler's parallel impactor.

Impact of Phosphate Fertilizer, Phosphoric Acid and Animal Feed Production Processes on Levels and Distribution of Toxic Metals in Air, Water and Soils
SENES

The University of Florida conducted and completed the main study to characterize the concentration levels and distribution of hazardous air pollution (HAP) toxic metals from production processes of phosphate fertilizer, phosphoric acid and animal feed. Such information is of seminal importance in assessing the potential exposure levels and environmental impact resulting from these pollutants and it forms the solid foundation for the USEPA to set new emission standards for the phosphate industry. Data for potentially toxic metals in discharged water and air emissions, including mercury, arsenic, cadmium, chromium, manganese, and nickel, were compiled and analyzed.

SENES was contracted to conduct a follow-up radiological risk assessment resulting from releases of the main naturally-occurring radionuclides (^{238}U, ^{235}U, ^{226}Ra, ^{232}Th, ^{228}Ra, ^{210}Pb, ^{210}Po, ^{214}Bi, ^{214}Pb, ^{40}K) to the environment using EPA-approved models (e.g., RESRAD, AERMOD) to the extent possible. In addition to releases associated with the production of phosphoric acid, ammoniated phosphates, and animal feeds, releases from co-product phosphogypsum storage in stacks and potential releases to the public and environment in commercial applications of phosphogypsum will also be assessed.



Typical Pathway Components Evaluated by SENES.

Reclamation

Ongoing Projects

Hydrology of Clay Settling Areas
University of South Florida

There are more than 120,000 acres of clay settling areas (CSAs) in Florida. What effects are these areas having on the surface and ground water regimes in the basins where phosphate mining occurs? How do CSAs behave hydrologically, and how can this behavior be modeled and predicted? How should CSAs be designed and reclaimed to optimize their hydrologic functionality? It is important to understand and be able to predict internal and external hydrologic relationships. The internal CSA hydrology is related to supporting functional wetlands on a CSA, and the "external" hydrology is related to impacts on surrounding surface and ground water systems.

Previous research has shown the importance of water storage in depressions and surface desiccation cracks. In addition, the depressional storage increases with time following reclamation due to further clay consolidation and surface subsidence. Progress has been made in improving the ability to predict clay consolidation and to account for it in modeling CSA hydrology. However, uncertainties in making accurate estimates of evapotranspiration and ground water seepage will remain. This project is evaluating and modeling the complete water balance of a CSA and includes methodology for more accurate determination of evapotranspiration and ground water seepage. One interesting finding has been that the vertical and horizontal permeability of the top meter of clay is much greater than previously thought due to cracks and old root channels.

Natural Channel Design of Low-Order Streams at Florida Phosphate Mines
AMEC, University of Florida

There is a great deal of public interest in protecting or restoring essential hydrological and ecological functions of streams. To obtain permits to mine, companies must provide reasonable assurance that streams, including headwaters, can be restored and that downstream areas will not be degraded. A fundamental component of restoration of stream function is that the channel shape and dimensions must be in dynamic equilibrium with the watershed. This project aims to provide much needed information to enable designers to rapidly and accurately fit equilibrium channels to reclaimed basins.

Stream Restoration.

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 is testing the performance of a leaf-gall-forming psyllid insect 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.

Projects Funded This Fiscal Year

Establishment and Management of Vegetation Cover on Phosphogypsum Stacks

The initial research was conducted 1989-2004. The current project includes 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.

Vegetation on Phosphogypsum Stack.

Vegetation Management on Reclaimed Lands

We have been working closely with staff of the Department of Environmental Protection, Bureau of Mining and Minerals Regulation, to further develop methods and strategies for controlling invasive exotic vegetation and for replacing it with native plant communities or other more desirable and useful vegetation.

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