## Madrid Engineering Group, Inc.

# 2005 Annual Report

LAZY "S" RANCH PHOS-PHILTER™ PROJECT Phosphorus Reduction Program



Prepared for:

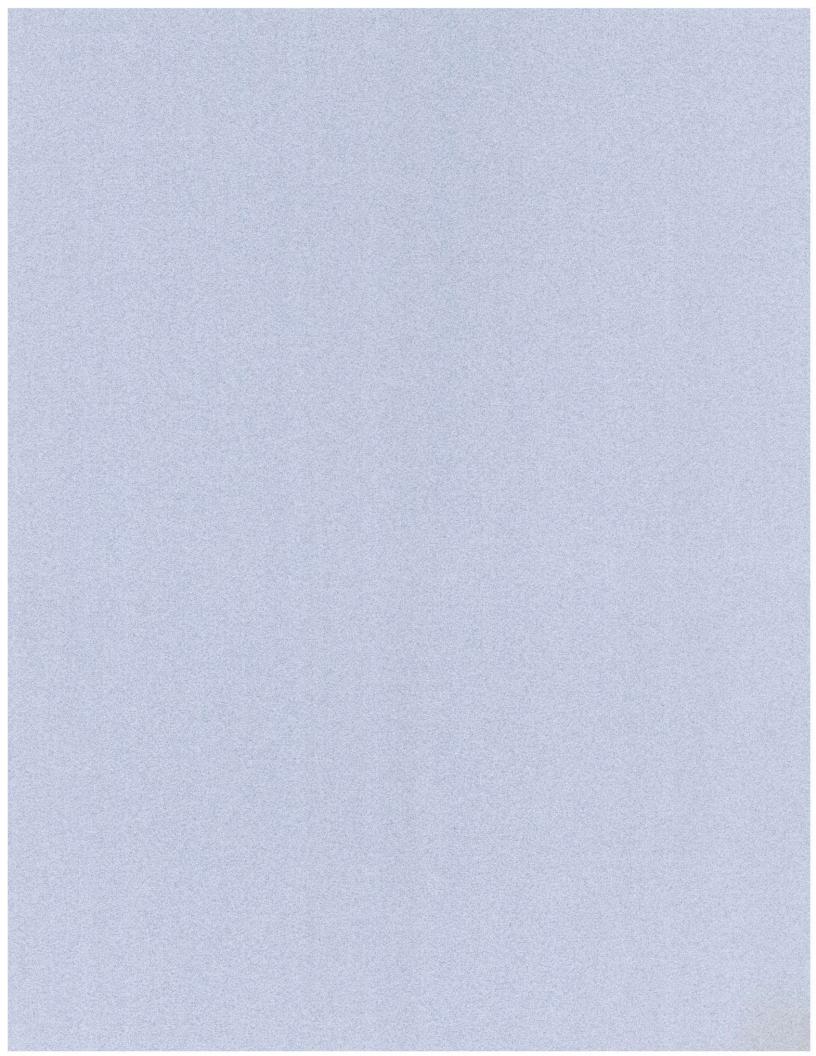
Eugene Stokes and South Florida Water Management District

Prepared by:

MADRID ENGINEERING GROUP, INC. P.O. Box 2506 Bartow, FL 33831 863-533-9007

> Project No. 3083.2 February 2006





#### **CERTIFICATIONS**

#### **Engineering Certification**

I hereby certify that I am a registered professional engineer in the State of Florida practicing with Madrid Engineering Group, Inc. under license number EB 0006509 issued by the Florida Department of Business and Professional Regulation and the Board of Professional Engineers. I certify that I, or others under my direct supervision, have prepared the geotechnical engineering evaluations, findings, opinions and conclusions represented in this report.

Lazy "S" Ranch Phos-Philter™ Project Phosphorus Reduction Program 2005 Annual Report MEG Project No. 3083.2

SIGNATURE:

NAME:

Larry D. Madrid, P.E.

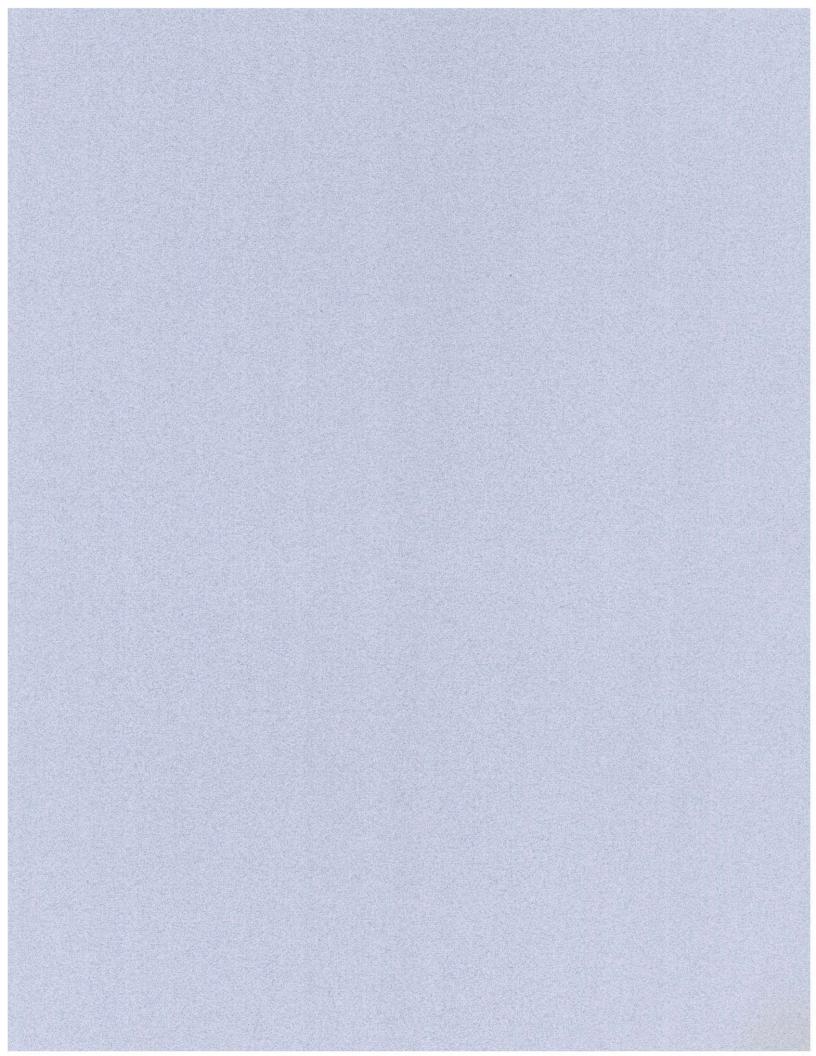
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## Lazy "S" Ranch Phosphorus Source Control Grant Program Project 2005 Annual Report

Madrid Engineering Group, Inc. now has approximately one year of monitoring completed on the Lazy "S" Ranch PhosPhilter™ project located on the Eugene Stokes Lazy "S" Ranch in Venus, Highlands County, Florida. This project is being completed as part of the ongoing **Phosphorus Source Control Grant** program of the South Florida Water Management District. The phosphorus reduction system was patented, engineered, designed, and constructed by Madrid Engineering Group, Inc. in Bartow, Florida, and was put into operation in February 2005.

#### **General System Description**

The system uses a patented non-toxic environmentally friendly iron humate material named PhosPhilter™, which pulls phosphorus from the phosphorus-enriched water by chemically bonding to it as the water flows through the media. At the Lazy "S" Ranch a canal goes around the western perimeter of the property, joins another canal from the center of the property, and collects the water at a central point (sump). On an as-need basis, a 400 GPM diesel pump transfers the phosphorus rich water from the canal to a 2.5-acre settling pond area (Photo 1).



Photo 1 - North View of Settling Pond, from NE Corner

From there it flows into one of two 50 feet x 50 feet humate treatment ponds containing the PhosPhilter<sup>TM</sup> material (Photo 2). The phosphorus enriched water gravity-flows through the PhosPhilter<sup>TM</sup> where it chemically bonds to the humate molecules. At the bottom of the humate treatment ponds is a sand bed with an underdrain system that collects the water and discharges to the stream that goes off the property, ultimately discharging into Fish Eating Creek. At the downstream end of each underdrain, there is a flexible pipe (Photo 3) that can be raised or lowered to control the rate of water being discharged. The system was designed to operate at a rate of 200 GPM for each treatment pond, for a total of 400 GPM.



Photo 2 - Humate Bed with water infiltrating



Photo 3 - Flexible Hose on Outlet from Humate Beds

#### Efficacy of the PhosPhilter™ System

In the original design calculations, it was assumed that one pound of iron humate would be able to remove 0.3 pounds of phosphorus from the water it came in contact with. In addition, we added a safety factor of 3.0 to the design. Therefore, we constructed the system at this site assuming that one pound of iron humate would remove 0.1 pound of phosphorus, on a mole-per-mole basis.

Recently, Madrid Engineering Group hired an independent company specializing in water chemistry to complete additional laboratory bench scale testing to determine the effectiveness of iron humate in removing phosphorus. Their report is attached. Also attached is the resume' of Mr. Don Luke, the principal investigator of the project. Mr. Luke determined that the iron humate is considerably more efficacious in phosphorus removal than previously believed. Although he was not completely able to achieve "breakthrough", he determined that iron humate can uptake at least 1.48 times its weight, at a rate of 95% removal by total weight.

The humate is therefore about 15 times more effective than previously thought. For this project, in the original plan we designed the system to last about 4 to 5 years before needing filter replacement, based on the pumping records of Mr. Stokes and phosphate concentrations previously obtained by the District. It is now reasonable to assume that the PhosPhilter<sup>TM</sup> can last 20 to 25 years without needing replacement, based on the new information from Clean Water Technologies. With the extended duration of

the humate it now becomes significantly more economically feasible for the property owner.

The beginning months of the 2005-year were in the dry season and therefore there were minimum opportunities to run the system. During March, the rainy season began and we started getting some water quality tests results that indicated how efficient and effective the PhosPhilter™ system would work. From June through the end of October we were able to run the system nearly continually and we have a number of lab test results (attached) showing the amount of phosphorus reduction. We are pleased to report an 81% overall phosphorus reduction average through the first year, with over 90% removal at the end of the year.

There are several items of note, based on our observations.

- Flow Rate The system must be regulated to maintain a maximum flow rate of 200 gallons per minute per pond in order to have the proper retention time required for the chemical reaction to take place to bond the phosphorus to the PhosPhilter™ medium. On March 15-16, 2005, the flow was increased to determine if the rapid flow would affect the phosphorus removal amount. As can be seen on the attached test results, the uptake percentage dipped to 58% as a result of reduced retention time.
- Maintenance by July, a thin layer of organic silt sediments had built up on top of the humate bed that ultimately sealed off and clogged the flow of water, thereby bringing the system to a halt. On July 18, 2005 MEG sent a crew to the site and raked the surface to break up the layer. This disked the organics into the sand, and the flow picked up to the normal rate (approximately 200 gpm) for several weeks. However, after a few weeks the beds clogged again and the flow rate significantly decreased. We discovered it will be necessary to remove the thin layer of organic silt completely to keep the system running at a normal pace. Photo 4 shows the build-up of silt on the top of the humate beds.



Photo 4 - Organic Silt Buildup on Humate Bed

- Test Results there were a few additional water quality test samples obtained by Mr. Stokes after July 12<sup>th</sup>, however the samples were never tested due to miscommunication with the testing laboratory. Soon after the last date shown, July 12 the water table was low enough at the ranch that pumping was no longer required from a ranching standpoint, so the PhosPhilter™ system did not need to be turned on except for the hurricanes that passed through. The system has been off all winter as there has been no need to run the system.
- Intake Modification the pump intake hose was lying on the bottom of the canal "sump" area and we believe that allowed the water to pick up phosphorus and silts that were settling to the bottom of the canal. In November, we therefore raised the intake hose to approximately two feet beneath the water surface, and are holding it in place with a polyurethane (Styrofoam) float (See Photo 5) that will rise and fall with the water surface. This should reduce the amount of silts in the intake water, and hopefully prolong the time between silt removal maintenance in the humate beds.



Photo 5 - Intake Hose from Canal tied to Floats

• Settling Pond Intake Modification - On the discharge side of the pump, where it discharges into the settling pond, there was no provision to prevent water from discharging directly onto the bottom of the settling pond, thereby causing significant erosion of the pond bottom, and associated release of silts and fines into the water column. This may have been a source of some of the silts, organics, and fines that was clogging off the humate ponds. To alleviate the erosion, we installed a large plastic (6' diameter) tub at the discharge location, and ran the discharge hose upward, so the incoming water will now spray up and not be directed at the bottom silts. (See Photo 6). The plastic tub is protecting the bottom from erosion, as is the upward flow direction.



Photo 6 - Water at Pond Intake Discharging Upwards

- Flow Path Proposed Modification After seeing how the water flows in the settling pond, we determined that the water could "short-circuit" to the humate beds without flowing through the majority of the pond. We therefore recommend changing the course of the water flow in the large settling pond. This will require Mr. Stokes to use a backhoe or bulldozer during the winter months to build a small, 50-foot long berm at the northwest corner of the west humate bed that will prevent short-circuiting (see Figure 4). This will create a considerably longer flow path and provide more time for settling to occur, which should improve system performance.
- Flow Meters The flow, to this point, has been estimated based on the pump rate and hours of pumping. We have checked the results in the field with time to fill a 5-gallon bucket, which is a crude but effective test. We have determined that flow meters can be used to provide a better estimate of the quantity of flow coming through the filter(s). One flow meter has been installed and another is ready to install before the system is run in 2006. Undoubtedly, some of the water is being lost to groundwater infiltration and/or evaporation. Therefore the quantity of water, and associated phosphorus quantities, is considered an estimate.

As soon as the spring rains begin and pumping starts for 2006, Mr. Stokes is prepared to scrape the top layer of organics off the humate bed and start the system back up. He has been given a copy of an operation and maintenance manual for the system and has agreed to run the system in general accord with the document.

Finally, we are working on a revised estimate of the cost per pound of phosphorus removal using this system, and will be submitted to the District under separate cover.

PHOSPHORUS TEST RESULTS SPREADSHEET

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			900							Pounds	
	Phosphorus	Phosphorus	Phosphorus	Gallone	Hour Motor			Phosphorus	Pounds	Phosphorus	
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1/19/05		0.100	91.2	200	0.00	7 0	144,000	0.783	0.95		
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4/19/05	1.230	0.420	65.9	250	254.7	7 (C	000,000	1.050	1.58		
6/8/05		0.266	78.4	225	273.8	0 7	200,000	0.810	0.61		
6/9/05		0.390	71.5	250	297.8	- VC		0.904	98.89		
6/10/05	0.940	0.060	93.6	200	0. FCS	, V	000,000	0.900	2.94		
6/17/05	1.100	0.085	92.3	200	480 B	1 0		0.880	2.12		
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6/21/05	1 100	0.070	0.20	200	0.100	47	288,000	1.022	2.46	82.69	
7/12/05	1420	0 300	2 0.4 0	7 700	0.000.0	47 010	288,000	1.021	2.45	85.14	
7/28/05	1 000	0000	0.0	100	0.50.0	7.767	2,269,800	1.120	21.22	106.36	
8/6/05		0000		75	0.24.0	1 0	96,000	1.000	08.0	107.16	
8/10/05		000.0		0 0	0.178	1	76,500	0.600	0.38	107.54	
8/12/0E	000.	0.000		00	886.0	12	45,000	1.000	0.38	107.92	
20/0/0	0.000	0.000		40	0.006	14	33,600	0.800	0.22	108.14	
9/9/05	1.000	0.000		30	918.0	00	32,400	1.000	0.27	108.41	
8/23/05	0.600	0.000		20	932.0	14	16,800	0.600	0.08	108.50	
		Average	81.3					Total P Removed	(sql) pe	108.5	
Comments											
	Removed outlet riser-not enough retention time	riser-not enoug	ah refention fin	at							
1	Hour meter not installed on pump until March	installed on pur	np until March	2005							

PHOSPHORUS TEST RESULT GRAPH SHEET

■ Phosphorus Before mg/L ■ Phosphorus After mg/L Date Sampled 0.000 Phosphorus (mg/L) 7.000 6.000 5.000 2.000 1.000

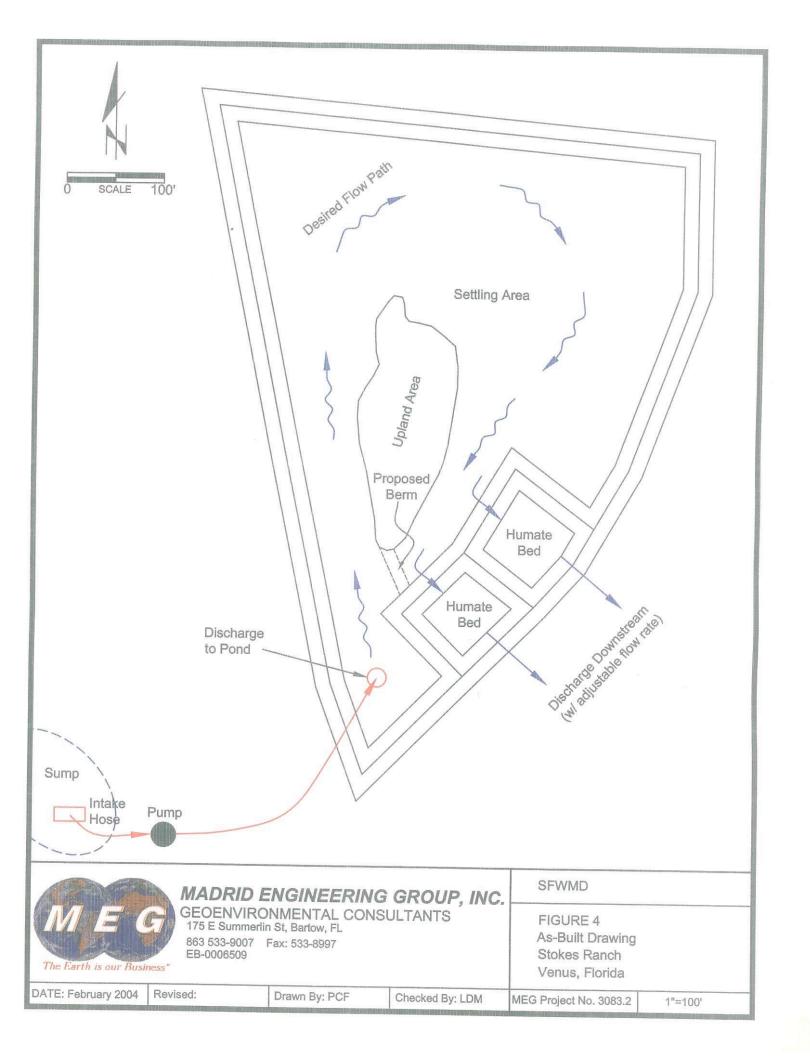
Stokes Test Results
Madrid Engineering Group, Inc.

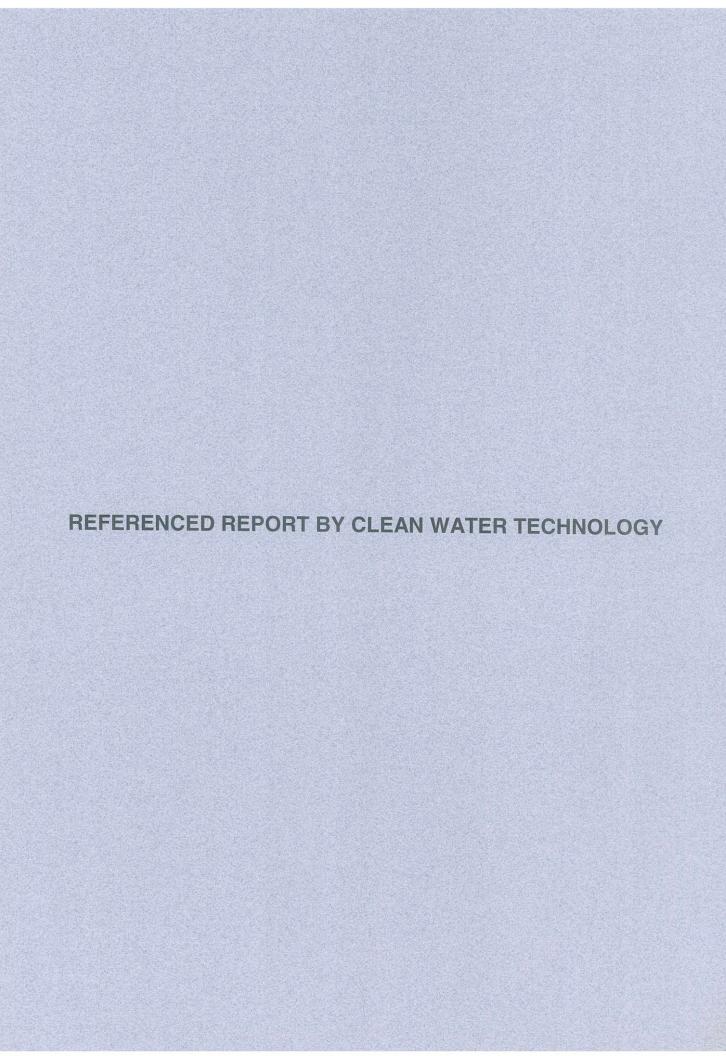


Rainfall amounts for 2005 taken from sfwmd.gov website located at the Archbold station starting January 31

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( M=MISSING I=INACTIVE T=TRACE E=ESTIMATED)





# Clean Water Technologies

Innovative Industrial Process Solutions



# TECHNICAL MEMORANDUM

REMOVAL OF PHOSPHORUS WITH IRON HUMATE (FEHU)

DECEMBER 2005

# Clean Water Technologies

Innovative Industrial Process Solutions



# TECHNICAL MEMORANDUM

REMOVAL OF PHOSPHORUS WITH IRON HUMATE (FEHU)

DECEMBER 2005

# REMOVAL OF PHOSPHORUS FROM WATER USING IRON HUMATE MEDIA

#### Introduction

Humic Acids are present in all natural water systems and result generally from the decay of vegetation. These materials are highly refractory and give the water a yellowish to tan coloration. The generic name "Humic Acids" actually refers to a broad class of materials of the above origin, and is generally separated into three major categories. Humin is the fraction of this material soluble neither in an acid or a base, Humic Acid is the fraction soluble at pH 2 or less, and Fulvic Acid is the fraction soluble at all pH values. The majority of the materials soluble in surface waters are Fulvic Acid and Humic Acid. The molecular weight of these materials ranges from approximately 500 to 2000, and the substances consist essentially of polymerized molecules containing large amounts of carboxy, hydroxyl, phenolic and ketonic groups to which their subsequent activity as detergent type molecules is attributed. Iron Humate is produced in typical municipal water treatment processes where an iron slat such as ferric Sulfate, or Ferrous Sulfate is utilized as a coagulant to remove colloidal material and to remove the color from the water prior to additional biological treatment. The sludge produced by this process is removed and dried. A number of innovative uses for this "Iron Humate" as the sludge has named, have been developed to exploit the properties of the Iron Humate (FeHu) as a material that will bind with phosphorus and trace metals. PhosFilter is such an application and is currently being utilized to remove phosphorus load from waters flowing from agricultural properties into the Everglades ecosystem. As a part of this work it was desired to develop a mean by which the life span of a given mass of FeHu could be estimated. Without getting into detail about the specific media content, it is necessary to mix the FeHu with a more porous media such as processed quartz sand to insure a reliable flow of water through the bed of FeHu containing media.

### **Experimental Description**

FeHu is utilized in a bed of material several feet thick in full scale applications, and water flows essentially vertically through the media bed and is collected in an under drain system. To simulate this application scenario, it was determined that a column configuration would be appropriate. Two, three inch diameter columns were constructed to operate in series. The raw water was fed with a metering pump to allow consistent and measurable flow. The first column was filled with washed quartz sand that had been screened to remove all plus 16 mesh material. The purpose of this column was simply to remove any particulate material that might clog the FeHu column. The second column was filled with a media comprised of 10% raw Iron Humate obtained from KemIron. The FeHu originated at the City of Tampa municipal water treatment plants. A pump was

placed on the discharge side of the Humate column and operated at a rate equal to the raw water feed pump. A large volume of phosphorus containing water was obtained from a local phosphate company. The water pH was adjusted to a pH of 6, and an analysis of the phosphorus, nitrogen and trace metals was obtained from an EPA certified laboratory utilizing appropriate EPA listed analytical methods. The pumps were started and operated for approximately 20 hours. Hourly samples of the column permeate were collected and submitted for appropriate analysis by the same laboratory as above.

### Experimental Results:

Sample Identification Feed Sample	Total P	Tot N 1	P Removal
Column Permeate-post treatment-initial	3.73	230	96.31%
Column Permeate-post treatment - 1330	4.34		
Column Permeate-post treatment - 1430	4.37	225	
Column Permeate-post treatment - 1530	4.52	222	
Column Permeate-post treatment - 1630	4.6	226	
Column Permeate-post treatment - 0845	4.3	223	95.74%
Column Permeate-post treatment - 0945	4.5	221	95.54%
Column Permeate-post treatment - 1145	4.5	225	95.54%
Column Permeate-post treatment - 1245	4.5	223	95.54%
Column Permeate-post treatment - 1345	4.5	225	95.54%
Column Permeate-post treatment - 1445	4.5	219	95.54%
Column Permeate-post treatment - 1545	4.5	217	95.54%
Column Permeate-post treatment - 1645	4.5	238	95.54%
Column Permeate-post treatment - 0845	2.72	233	97.31%
Column Permeate-post treatment - 0945	4.08	227	95.96%
Column Permeate-post treatment - 1045	4.23	225	95.81%
Column Permeate-post treatment - 1145	4.19	228	95.85%
Column Permeate-post treatment - 1245	3.97	230	96.07%
Column Permeate-post treatment - 1345	4.08	225	95.96%
Column Permeate-post treatment - 1445	4.06	230	95.98%
Column Permeate-post treatment - 1545	3.95	228	96.09%
Column Permeate-post treatment - 1645	3.98	228	96.06%
Column Permeate-post treatment - Final	3.61	176	96.43%

At this point, the test was stopped due to mechanical failure of the humate column internal supports. Solid media began to appear in the column permeate indicating that the lower bed support filter media had failed. Upon inspection, this was found to be the case.

## Phosphorus Removal Efficiency and Humate Loading

The actual mechanism of phosphorus removal by FeHu is not precisely understood. It is likely that a combination of chelation, and adsorption mechanisms are at work in this process as opposed to a specific redox reaction. Regardless, the above attempt to estimate

the maximum loading was preempted by equipment failure prior to actual breakthrough of the column. Ultimate loading will exceed the following estimates.

Based on the testing above, 242.88 L of raw water containing 24.53 grams of phosphorus was processed over a FeHu media bed containing 15.89g of FeHu suspended in a bed of sand. Based on the analytical results above, an average removal efficiency of 95.86% was calculated for the duration of the 23 hour test.

#### Conclusions

A design basis for phosphorus removal utilizing FeHu material would be as follows:

The above testing demonstrates a minimum loading of 1.48 Lb of Phosphorus per pound of FeHu at removal efficiencies in excess of 95%.

#### Education

University of South Florida -1973 -1978 Chemical Engineering, Partial Completion of Masters in Chemical Engineering. Thesis topic: "Non-Newtonian Flow of Two Phase Liquid Systems in a Solvent Extraction (Uranium) Environment. Tampa, Florida

#### <u>Professional experience</u>

8/2001 - Present

#### CleanWater Technologies.

Tampa, Florida

CleanWater Technologies provides expert Water Treatment consulting services for a variety of water treatment processes in use generally throughout the Phosphate Industry. Particular expertise exists in design and operational optimization of thickening processes for waste phosphatic clays, phosphoric acid clarification, phospho-gypsum process water treatment utilizing lime or other neutralizing materials. Patent pending processes have been developed for utilization in the removal of phosphorus, nitrogen, and trace metal contaminants from industrial process water systems. Consulting assignments have included the following:

#### Senior Consultant CDM Constructors – Piney Point Site

Responsibilities included characterization of the double lime treatment system in use at the Piney Point Site. Operational evaluation of the clarifier operation. Polymer control systems and sludge disposal processes were a part of the scope. This assignment is ongoing.

# Senior Consultant Madrid Engineering Group

Responsibilities included development of laboratory testing protocol for the development of process alternatives for the remediation of Lake Hancock in Polk County. Specific responsibility was for the development of the on shore dewatering and solids disposal process for the lake bottom sediments. This assignment is ongoing, and has resulted in the award of additional work on the project to further evaluate the processes developed in the original scope of work.

# Senior Consultant URS Engineering Corp

Responsibilities included development of laboratory testing protocol for the development of process alternatives for the remediation of North Lake in the city of Hollywood. Specific responsibility is for the development of the on shore dewatering and solids disposal process for the lake bottom sediments. This assignment is ongoing.

#### <u>Director, Business Development, Manager of Projects</u> <u>Florida Engineering and Design</u>

Responsibilities include an oversight role in most all projects technical, or other, and a direct role in Project Management for Most large projects undertaken by Florida Engineering and Design. Projects recently completed by Florida Engineering and Design, include the Neochim Fertilizer feasibility studies for the USTDA, The Progress Energy Cooling Pond Dam Expansion, Thermal Definition Study, and Spray Cooling System Design, Scale up of the Piney Point Industrial Waste Water Treatment process for offshore disposal, Feasibility Study for CF Industries for the implementation of fine fraction phosphate recovery. Retained as the process consultant by IMC Phosphates(now Mosaic) for their three operating fine phosphate recovery and waste disposal thickening processes.

Administrative responsibilities in addition to the above technical responsibilities include proposal preparation, and business development activities and contribution to most formal reports and technical opinions.

8/1993 - 8/2001

#### Ciba Specialty Chemicals

Florida

#### <u>Director, Business Development – NAFTA</u> <u>Global Key Account Manager, Phosphates</u>

Responsibilities included developing and implementing value based business development strategies within NAFTA region. Responsible for direct operation of the Florida Phosphate projects, associated customers and contractors. Identified project champions and facilitated development through various project assessment methods (Stagegate, VBM). Created implemented a NAFTA wide strategy for the rollout of the TC business and technology model. Managed process for identifying and evaluating new technology which could be implemented into one or more value based strategies. Additional responsibilities included developing sales strategies for 8 to 10 technical sales experts, as well as various sales support staff and administrative support personnel including development of sales strategies for

selling to public, municipal and private marketplaces.

A high level of technical success was achieved and resulted in seven (7) US and numerous associated foreign Patents for products and technology ranging from a novel clumping cat litter, to new Waste Disposal Technology that is currently being implemented in the phosphate industry. Two phosphate recovery processes described in US Patents 5688404, and 6077441 processes were developed, with both being implemented at one or more locations in the United States and Canada. The purpose of these recovery processes was to enhance the recovery of fine phosphatic materials lost to the waste (slimes) fraction. Investment by phosphate producers utilizing these processes exceeds \$50MM USD.

Responsibility during the development of these processes was to conduct much of the hands on developmental work, design and construct pilot scale operations, oversee field trials and manage the development of process improvements. Full scale processes were constructed at 3 locations, a pilot scale process is under construction in Western Canada, and this research was the topic of a technical presentation at the 2001 Paste Thickening Conference in Johannesburg SA, as well as numerous papers and public presentations throughout the phosphate industry.

During my tenure I was responsible for technical evaluation of potential projects, acquisitions and allocation of related technical resources on a global basis for projects in Jordan (Phosphate), Tunisia(Phosphate), Alberta Canada (Oil Sands), Northwest Territories, Canada (BHP Diamond Mining), Ghana (Aluminum), Belgium (Phosphoric Acid), Chile (Copper), and South Africa (Diamonds),

7/1984 - 8/1993

### Bloomingdale Enterprises

Brandon, FL

#### President

President and Chief Executive of a \$5MM Construction Industry related business. A large part of my function was as the primary Business Development person. Our business was both public and private sector based to achieve a diversified pertfolio of work to avoid a dependence on any particular market segment. Customer base ranged from individual property owners, housing and apartment developers, and governmental entities. We completed projects for State and Federal agencies like the Veterans Administration, Florida Department of Transportation, and many local government entities.

6/1981 - 6/1984

Allied Colloids

Suffolk, Virginia

#### **Technical Representative**

Technical Sales Representative serving the Water Treatment industry, specifically the Phosphate Industry in Central Florida, North Florida, and North Carolina. Responsible for developing own marketing plan and was very self directed and independent.

7/1980 - 6/1981

#### Jacobs Engineering Group

Lakeland Florida

#### **Process Engineer**

Process Engineering Consultant to various industries. Jacobs Engineering in Lakeland served primarily the phosphate industry, but my previous experience in the Uranium recovery field allowed me to branch out and develop work for firms outside the phosphate industry. My initial employment was with Zellars-Williams, Inc, an industry leading process development and consulting engineering firm focusing on phosphate beneficiation technology. ZWI was acquired by Jacobs Engineering. During my tenure there, I was a responsible team member for numerous international feasibility studies, notably in Peru (Bayovar), Chile, Brazil, and numerous US phosphate deposits.

4/1976 - 7/1980

#### **United Nuclear Corporation**

Mulberry, Florida

#### Development Group Leader

Started employment as a co-op engineering student in the Florida Uranium Recovery Facility. Work primarily focused on scale up of process improvements, on process development, and on development of pilot scale simulations of full scale unit operations. Within 18 months, I was promoted to the position of Development Group Leader, partly in recognition of significant patented process improvement technologies. At the time, I was the youngest person to have held that position within the United Nuclear Organization.

#### Professional memberships

Society of Mining and Metallurgical Engineers - 1992

#### **Publications**

Recent Advances in Waste Disposal Technology, June 2001 SME Application of Flocculation to Paste Tailings Disposal, April 2001. Paste Seminar, Johannesburg South Africa.

#### **Patents**

#### **US Patents**

Patent App - 2005 - In-Situ process for treatment of acidic waste industrial process waters

Patent App- 2005 – Process for removal and recovery of phosphorus, nitrogen and trace metals from industrial process waters

Number 6, 213,416 - Treatment of Phosphate Containing Rock - April 2001

Number 6,077,441 - Mineral Recovery Process - June 2000

Number 6,039,189 - Mineral Solids Separation Process - March 2000

Number 5,688,404 - Phosphate Recovery Process - November 1997

Number 5,609,123 – Animal Litter Compositions and Processes for Making Them – March 1997

Number 4,325,918 - Deprotonation of an Alkylphenyl Acid Phosphate Extractant - April 1982

Number 4,289,609 – Process for Removing Solid Organic Materials and Other Impurities from Wet Process Phosphoric Acid – September 1981.