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EVALUATION OF LIME TREATMENT SLUDGE ALTERNATIVE DISPOSAL METHODOLOGIES INCLUDING UTILIZATION IN CLOSURE OF PHOSPHOGYPSUM STACKS

FINAL REPORT

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FINAL REPORT

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PERSPECTIVE

Steven G. Richardson, FIPR Institute Reclamation Research Director

When phosphate rock is treated with sulfuric acid to produce phosphoric acid, the resulting phosphogypsum and process water byproducts are sent to a stack and pond system for settling of the phosphogypsum and clarification of the process water. The process water is acidic, high in dissolved solids, and contains some fluorine plus some plant nutrients (e.g., P, N, K, S, Ca and Mg). While a phosphogypsum stack system is operational, the process water is recycled through the chemical plant. When a chemical plant ceases operation and a stack is closed, the acidic process water entrained in the system drains, and the leachate must be treated prior to discharge. Conventional treatment uses lime applied in two stages (see Executive Summary or Introduction for more details on stage I and stage II lime treatment) to raise the pH of the water and precipitate elements such as fluorine and phosphorus. Ammonium ions are also converted to ammonia and removed as a gas. The precipitates, or lime sludges, are retained in settling ponds.

Closure of a representative phosphogypsum stack and pond system could require the treatment of over 3 billion gallons of acidic process water, thus generating 2400 acre-feet of lime sludge and 3 billion gallons of effluent. Thus, in addition to the phosphogypsum stack itself, the lime sludge settling basins must also be capped in the closure procedure. Any alternative methods that could reduce the amount of lime sludge in settling basins or reduce the amount of treated process water discharged to surface waters could be environmentally and perhaps economically beneficial. The research included evaluation of:

- Lime requirements for treatment, plus the properties and volumes of the sludges and the effluents produced, in relation to the characteristics of the process waters;
- Engineering of sludge settling ponds and their closure;
- Alternative sludge disposal methods, such as sludge use as an agricultural amendment for establishing vegetation on stack side slopes;
- Possible consumptive use of process water treated to various degrees for irrigation of grass on stack side slopes or in nearby pastures.

Other FIPR research related to phosphogypsum stack closure includes:

- Establishing Vegetation Cover on Phosphogypsum in Florida. FIPR Publication No. 01-086-116.
- Phosphogypsum Stack Closure: Evaluation of Phosphogypsum as an Alternate Final Cover. FIPR Publication No. 03-125-195.
- Hydrologic Evaluation of Final Cover System Alternatives for Closure of Phosphogypsum Stacks. FIPR Publication No. 03-126-212.

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The contributions of Dr. J. Bryan Unruh, Extension Turfgrass Specialist at the University of Florida's West Florida Research and Education Center, and Dr. Laurie E. Trenholm, Urban Turfgrass Specialist at the University of Florida, in executing the greenhouse irrigation studies are gratefully acknowledged.

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ABSTRACT

A characterization of the engineering properties of lime sludges derived from doublelime treatment of phosphogypsum stack system process waters is presented along with an evaluation of lime treatment sludge production quantities and CaO utilization (i.e., lime requirements) as a function of chemical characteristics of the process waters.

Utilization of lime-treatment sludges as an agronomic amendment in gypsum stack side slope final covers in lieu of dolomitic limestone was investigated. Greenhouse plant growth and irrigation studies were performed to determine whether various turfgrass species (bermudagrass, bahiagrass and seashore paspalum) can be successfully grown on leached and unleached phosphogypsum amended with either dolomitic limestone and/or different-type process water lime treatment sludges, and to evaluate performance of grass species grown in these media and in natural sandy soils when irrigated with fluids derived from lime treatment of process water.

Evaluations of conventional lime sludge disposal and alternative disposal methods for lime sludge and supernatant to minimize on-land disposal within sedimentation ponds and discharge of Stage II effluent to surface water are presented.

Lime-treatment sludge could potentially be mixed and added to process water in a cooling pond system during closure. Some lime sludge constituents (including free lime present in the sludge) will dissolve and/or react with the acidic process water such that the quantities of lime needed for ultimate treatment of the process water are reduced, and the pond system could be simultaneously filled with sludge in preparation for closure construction and capping.

Co-disposal of lime-treatment sludge with phosphogypsum slurry atop an active phosphogypsum stack may be feasible when adding: (i) up to 2.5% of Stage I sludge to the gypsum (dry weight basis); and (ii) up to 1 percent Stage II sludge to the gypsum (dry weight basis), with the slurried mix discharged at initial solids contents on the order of 20-30%. Co-disposal needs to be controlled so as not to adversely impact stack stability, and the handling, dewatering and compaction characteristics of the gypsum.

If the lime sludge contains some free lime (which it often does), application rates of 1% and up to about 6% of Stage II sludge (dry weight basis) appear to be suitable for amending leached and unleached gypsum, respectively, prior to grassing a side slope as part of phosphogypsum stack closure.

Bermudagrass and bahiagrass grown in sandy soil can be irrigated with effluent from either pH 7 single-stage treatment or conventional Stage II double-lime treatment (diluted or undiluted). Bermudagrass can successfully grow in both leached or unleached phosphogypsum media, properly amended with dolomitic limestone or with single-stage (pH 7.5) or Stage II lime-treatment sludges, and irrigated with effluents from diluted single-stage and Stage II lime treatment of process water. Seashore paspalum turfgrass can grow reasonably well in sandy soil when irrigated even with the more acidic Stage I (pH 5) effluent. Barring restrictions imposed by surface water runoff and groundwater quality requirements, recycling of effluents generated by lime-treatment of the process water to irrigate the grass cover on a closed phosphogypsum stack or grass pasture nearby appears to be technically viable. Spray irrigation would provide a substantial benefit in that reliance on valuable fresh water resources for dilution of the treated effluent for the sole purpose of achieving surface water discharge standards could be substantially reduced or eliminated.

If the land areas available for spray irrigation of turfgrass are limited and/or the quality of surface water runoff from the irrigated areas is of concern, water consumption by spray evaporation could be a viable option for disposing of lime-treatment effluents.

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LIST OF SYMBOLS AND ABBREVIATIONS

<u>Symbol</u>	Description
$A_{\rm f}$	Pore pressure parameter at failure
ANOVA	Analysis of Variance
AWC_{40}	Apparent water content at 40°C
AWC ₁₁₀	Apparent water content at 110°C (normalized based on mass of dry solids at 40°C)
AWC ₂₀₀	Apparent water content at 200°C (normalized based on mass of dry solids at 40°C)
ΔAWC_{110-40}	$AWC_{110} - AWC_{40}$
ΔAWC_{200-40}	$AWC_{200} - AWC_{40}$
В	Pore pressure coefficient
C _c	Compression index
C_v	Coefficient of consolidation
C_{α}	Coefficient of secondary compression
CR	Compression ratio
CIUC	Isotropically consolidated-undrained triaxial compression
D	Diameter
DAP	Days After Planting
DASF	Days After Starting Fluid Irrigation
e	Void ratio
$e_{\rm f}$	Final void ratio
ei	Initial void ratio
ET	Evapotranspiration
$\epsilon_{\rm v}$	Vertical strain
G	Conductivity
Gs	Specific gravity
H_{i}	Initial height
H_{f}	Final height
HDPE	High density polyethylene
IF	Irrigation Fluid
k	Hydraulic conductivity
k _v	Saturated vertical hydraulic conductivity
k ₂₀	Saturated hydraulic conductivity at 20°C

LIST OF SYMBOLS AND ABBREVIATIONS (CONT.)

<u>Symbol</u>	Description
L	Length
LI	Liquidity index
LL	Liquid limit
LSD	Least Square Difference
MCL	Maximum Contaminant Level
MC	Moisture content
MC_{40}	Moisture content determined using a drying temperature of 40°C
MC_{110}	Moisture content determined using a drying temperature of 110°C
M _{CaO}	Mass of -14 Hi-Calcium quick lime added per volume raw pond water treated
M_{sludge}	Dry mass of settled sludge per volume raw pond water treated
n	Total porosity
n _e	Effective porosity
NS	Not significant
\bar{p}	Average effective principal stress
Р	Probability
Р	Rainfall
PL	Plastic limit
PI	Plasticity index
q	Half principal stress difference
S	Degree of saturation
S	Solids content
S_{f}	Final solids content
S_i	Initial solids content
$\mathbf{S}_{\mathbf{t}}$	Sensitivity
Si	Initial degree of saturation
Su	Undrained shear strength
TDS	Total dissolved solids
u _b	Back-pressure
$\Delta V/V_o$	Volume change/Initial volume (volumetric strain)
V _{CaO}	Volume of 10% lime slurry added per volume raw pond water

LIST OF SYMBOLS AND ABBREVIATIONS (CONT.)

Symbol	Description
Vi	Initial settling velocity
Veffluent	Volume of clarified effluent water per volume raw pond water treated
V _{sludge}	Volume of settled sludge per volume raw pond water treated
V _v	Quantity of synthetic rain water permeated through sample divided by the initial volume of voids in the sample (calculated using a specific gravity of 3.0)
V_v^*	Interval of void volumes of leachate flow through sludge sample
V _{vi}	Initial void volume
$V_{\rm vf}$	Final void volume
$\overline{\varphi}$	Effective friction angle
W _c	Water content
W _{c,110}	Water content at 110°C
W _{c,40}	Water content at 40°C
W _f	Final water content
Wi	Initial water content
W _{ps}	Preshear water content
WDS	Weight of dry solids
γd,ps	Preshear dry density
$\gamma_{\rm W}$	Unit weight of water
$\bar{\sigma}_c$	Average isotropic effective confining stress
$\overline{\sigma_v}$	Vertical effective stress
$\overline{\sigma}_{vc}$	Effective vertical consolidation stress
$\overline{\sigma}_{vo}$	In situ vertical effective stress
$\bar{\sigma}_{vm}$	Pre-consolidation vertical effective stress
$\bar{\sigma}_1$	Major principal effective stress
$\bar{\sigma}_3$	Minor principal effective stress
-200	Percent material by dry weight passing the U.S. No. 200 sieve size (<0.075 mm), Fines Content

EXECUTIVE SUMMARY

STATEMENT OF PROBLEM AND GOALS

For every ton of P_2O_5 produced, about five tons of by-product phosphogypsum are generated. More than 850 million tons of phosphogypsum have already been stockpiled in some 30 stacks in Florida and, at current production levels, about 30 million tons of phosphogypsum are added each year. Active phosphogypsum stacks are almost entirely saturated with acidic process water (pH typically between 1.5 and 2.0). The entrained pore water accounts for about 60 billion gallons, and the pore water inventory is projected to increase by approximately 2 billion gallons per year until stack closures begin to reduce the volume of drainable pore water. Approximately half of the acidic pore water entrained within gypsum stacks, or 30 billion gallons, is in "temporary storage," as it will eventually drain once the stacks are closed, and the seepage water will then have to be treated prior to discharge (unless it can be transferred or recycled to the phosphogypsum stack system of an active phosphoric acid plant). Lime treatment is the most common method employed by the industry. For a typical two-stage lime treatment unit cost of \$20 per thousand gallons, the treatment cost for the industry could eventually reach 800 million dollars.

Sludge Generation from Conventional Double Lime Treatment of Process Water

Conventional lime treatment typically employs a two-stage neutralization process. Stage I treatment consists of mixing ground limestone (CaCO₃), lime (CaO) or slaked lime (Ca(OH)₂) with process water to elevate the pH to about 5 which results in precipitation of fluoride and other dissolved solids (i.e., metals, radionuclides and some of the phosphate) that settle and form the Stage I sludge. During Stage II treatment, clarified Stage I supernatant is mixed with lime once again to further raise the pH to precipitate the phosphorus. Underflow from the Stage II treatment reactor is then routed into a clarifier or settling pond for sedimentation of the Stage II sludge. Where the process water contains a low ammonia nitrogen concentration, a Stage II target pH on the order of 8 or 9 is typically used. When ammonia nitrogen concentrations are elevated, a higher Stage II target pH on the order of 11 is used to convert the ammonium ion to ammonia and aid in its removal via air stripping. Effluent from "Stage II" (i.e., dissolved solids removal without air-stripping) or "Stage II+" (i.e., Stage II plus aeration at pH~11 for ammonia removal) is subsequently acidified to a pH of about 7 prior to discharge to surface waters. Significant quantities of lime sludge are, therefore, generated during the double lime treatment process, and these soft by-product waste materials are typically disposed of in large settling areas that will ultimately have to be closed.

Surface Water Discharge Issues

The conductivity of double-lime treated process water often exceeds the 1,275 µmhos/cm surface water standard by a factor of up to 5. Moreover, lime treatment is not

always effective in removal of nitrogen for compliance with un-ionized ammonia criteria, unless second-stage treatment to high pH levels and extensive air-stripping is undertaken. Long-term reliance on continued use of substantial fresh water resources for dilution of lime-treated process water for the sole purpose of discharging treated water in compliance with the Class III Standard for conductivity, as mandated under Rule 62-302, Florida Administrative Code (FAC), is a non-beneficial use of a scarce and valuable fresh water resource and is not in the best interest of the industry or the State; moreover, discharges containing elevated nutrient loadings may lead to greater eutrophication in receiving water bodies, so it would be important to explore other means for beneficial use or consumption of the treated effluents as the nutrients can then become a resource (if used properly).

Research Objectives

Considering the large potential volumes of acidic process water eventually requiring treatment as part of gypsum stack closures in Florida, the associated treatment costs and the lime sludge disposal requirements, alternative utilization or disposal methodologies that would reduce the volume of lime sludge requiring on-land disposal within sedimentation ponds and/or that would minimize the volume of treated effluents discharged to surface waters of the State were evaluated.

The research was specifically focused to address the following topics relevant to treatment of acidic process water during closure of phosphogypsum stacks and/or during operation of phosphogypsum stack systems:

- Characterization of the engineering properties of the lime sludges relevant to disposal, CaO utilization (i.e., liming requirements) and the volumes of Stage I and Stage II lime sludge and supernatants generated during treatment of acidic process water as a function of the initial chemical composition of the process water;
- Evaluation of conventional lime sludge disposal methodologies relevant to settling pond sizes, and capping of settling ponds during reclamation;
- Evaluation of alternative disposal methods for the lime sludge such as dissolution in acidic process water during closure of a cooling pond system, co-disposal with gypsum slurry in the phosphogypsum stack during its active life, or use as an agronomic amendment in gypsum stack side slope final covers (in lieu of commercially available dolomitic limestone) to promote grass growth; and
- Irrigating turf grass with Stage II effluent to reduce discharges to surface waters and consumptive use of fresh water resources that would otherwise be required for dilution of treated waters prior to discharge.

Evaluation of sludge production consisted of characterizing the quantities of Stage I and Stage II lime sludge produced per 1000 gallons of treated process water for 3 different acidic process waters representing the range of total dissolved solids concentrations typically observed in Florida phosphoric acid plant process waters by performing laboratory sludge

production tests. Laboratory tests were also performed for evaluating the sludge engineering properties. Stage I and Stage II lime sludge properties relevant to conventional disposal in sedimentation ponds were characterized. Evaluation of the sludge agronomic properties consisted of mixing Stage I lime sludge, Stage II lime sludge and Stage II supernatant from each of the 3 selected representative sites with both leached (i.e., pH>4) and unleached (i.e., pH<3) phosphogypsum from the corresponding sites to determine if an amended phosphogypsum target pH of 4.8 to 5.2 can be achieved and verify if these lime-treatment materials could be used as an agronomic amendment in phosphogypsum stack side slope final covers in lieu of dolomitic limestone. Greenhouse plant growth and irrigation studies were performed to determine whether various turfgrass species (bermudagrass, bahiagrass and seashore paspalum) can be successfully grown on leached and unleached phosphogypsum amended with dolomitic limestone and/or different-type process water treatment sludges, and to evaluate performance of grass species grown in these media and in natural sandy soils when irrigated with fluids derived from lime treatment of process water. A total of 49 growth media/irrigation fluid combinations were studied under greenhouse controlled conditions at the University of Florida in Gainesville and at the West Coast Florida Research Education Center in Milton under the direction of Dr. Laurie E. Trenholm and Dr. J. Bryan Unruh.

Conventional lime sludge disposal was evaluated in addition to the following alternative disposal methods:

- Dissolution of lime sludge in acidic process water;
- Recycling lime-treatment sludge within phosphogypsum stack systems by codisposal of gypsum and sludge within the stack;
- Amendment of phosphogypsum with lime sludge to promote grass growth on the phosphogypsum stack slopes during closure;
- Irrigating turf grass with Stage II effluent to reduce discharges to surface water; and
- Using lime-treatment effluents in spray evaporation systems to reduce the need for fresh water resources that would otherwise be required for dilution of the treated effluents prior to discharge.

CaO UTILIZATION AND SLUDGE PRODUCTION

Lime treatment sludge production and CaO utilization (i.e., lime requirements) were determined from laboratory testing as a function of chemical characteristics of the process waters used. In general, the TDS of the process water yielded much better correlations with CaO utilization and sludge production quantities than process water pH.

The laboratory experiments yielded useful correlations between CaO utilization (i.e., lime requirements) and initial process water total dissolved solids (TDS) concentrations. The experiments showed that the rates of CaO utilization can vary significantly from about 20 to 200 lbs/1000 gallons of process water during Stage I treatment (to pH~5), and from 40 to 250 lbs/1000 gallons of process water for both Stage I and Stage II treatments combined (to

pH~11), considering process water TDS concentrations ranging from about 18,000 mg/l to 45,000 mg/l, respectively. Note that the corresponding CaO utilizations during Stage II of 20 to 50 lbs/1000 gallons are much lower than the lime requirements during Stage I. (The rate of CaO utilization ranged from 30 to 200 lbs/1000 gallons of process water during single-stage treatment to pH 7.) Actual field CaO utilization requirements are expected to be higher than derived from the laboratory experiments because mixing in the field is not expected to be as effective as the almost perfect mixing in a laboratory environment, and, hence, some unreacted lime (about 5-10%) will likely be present in the field treatment sludges. Note also that CaO utilization is the main controlling factor dictating the cost of treatment of the process water.

Test results indicated a typical range of Stage I sludge production (at pH~5) of about 50 to 400 lbs per 1000 gallons for process water having typical TDS in the range of 18,000 to about 45,000 mg/l, respectively. The Stage II sludge production (at pH~11) ranged from 100 to 250 lbs per 1000 gallons of process water , with sludge generation from Stage I and Stage II treatments combined ranging from about 150 to 650 lbs per 1000 gallons of process water (having TDS in the range of 18,000 to about 45,000 mg/l). The typical range of sludge generation from single-stage treatment to pH 7 varied from 150 to 500 lbs per 1000 gallons of process water quantities of sludge are, therefore, generated and these quantities are highly dependent on the TDS of the process water.

SLUDGE ENGINEERING PROPERTIES

After performing initial screening tests on 5 lime treatment plants in Florida at phosphoric acid plants, 3 sites and corresponding Stage I and Stage II sludges, representative of the range of index properties observed for lime sludge, were selected for measurement of engineering properties relevant to evaluating conventional disposal in and capping of sedimentation ponds. Field and laboratory prepared Stage I and Stage II samples were tested as part of the sludge engineering properties characterization.

The measured pH of the Stage I, Stage II and single-stage sludges was in the typical range for these type materials, ranging from 4.2 to 12.3. Laboratory testing confirmed that Stage II sludge settles to lower solids contents (less than 10-15%) than the Stage I sludge (20-30%). Stage I undisturbed sludge sample dry densities yielded solids contents of 26 to 33%. Stage II undisturbed sludge samples exhibited solids contents of 19-32%, higher than derived from laboratory tests probably due to consolidation and aggressive desiccation. The measured specific gravity of the lime sludges ranged from 2.75 to 3.05. As expected, both Stage I and Stage II sludge was typically finer than the Stage I sludge, with often more than 50% clay-sized particles. The Stage II sludge typically exhibited a higher plasticity than the Stage I sludge, and lower initial settling velocities.

Laboratory testing also indicated that the lime sludge behavior is comparable to that of an elastic MH-type silt (i.e., high-plasticity silt), and the rate of consolidation is 10 to 100 times faster than typical for more plastic CH-type (highly plastic) waste phosphatic clays. The hydraulic conductivity of the sludges typically ranged from 10^{-4} to 10^{-6} cm/sec. The hydraulic conductivity of the Stage I lime sludge was about one to two orders of magnitude higher than that of the Stage II lime sludge at equivalent void ratios. At solids contents in the range of 20-30%, the shear strengths measured in miniature vane shear tests indicate that lime sludge can be described as a very soft, very low strength material (i.e., with undrained shear strengths potentially less than 30 psf and no greater than about 200 psf). Although the undrained shear strength of lime sludge at initial settled solids content (i.e. just after deposition in a sludge pond) is quite low, at higher solids content the drained shear strength can become relatively high (characterized by an angle of internal friction in excess of 45°).

In situ samples of sludge were checked to determine the amount of unreacted lime present in sludge produced by field mixing equipment. The results yielded a free lime content ranging from less than 1% to more than 20% as CaO. The free lime content of Stage II sludge was higher than that of Stage I sludge, and was typically in the range of 5-10%. Hence, the sludge typically retains some buffering capacity.

SLUDGE AGRONOMIC PROPERTIES

The suitability of utilizing Stage II sludge (and/or single-stage pH7 sludge) generated from lime treatment of process water as an agronomic amendment in gypsum stack side slope final covers (in lieu of commercially available dolomitic limestone) was investigated and deemed feasible for achieving the target amended gypsum pH of 4.8 to 5.2 needed to promote grass growth. Application rates of 1% and up to about 6% of Stage II sludge (dry weight basis) appear to be suitable for amending leached and unleached gypsum, respectively, if the sludge contains some free lime. These percentages correspond to the addition of about 8 to 45 tons per acre of Stage II sludge (dry weight basis) to amend the upper 6 inches of gypsum stack slopes. For a Stage II solids content of 20%, it would be necessary to spread and then gradually mix a layer of Stage II sludge about 0.3 to 1.7 inches thick into the upper 6 inches of the surface of gypsum stack slopes. Based on results of the greenhouse studies, the sludge-amended leached and unleached phosphogypsum were good growth media for Bermuda turf grasses. Conductivity of the pore water of the sludge amended phosphogypsum was about twice to four-fold the Class III surface water Standard of 1275 µmhos/cm, consistent with the pore water conductivity of gypsum amended with dolomitic limestone.

Leaching tests were performed on Stage II sludge samples to check if any metals contained in the sludge would leach into the environment. Synthesized rain water (pH~5) was permeated through reconstituted lime sludge samples and there was no exceedance of any of the Maximum Contaminant Level (MCL) standards for metals after two void volumes of flow. Leaching of Stage I sludge did not result in exceedances either, except for the

secondary MCL standard for aluminum upon leaching of a single sludge sample from one of the investigated sites.

CONVENTIONAL SLUDGE DISPOSAL METHODS

Conventional double lime-treatment sludge disposal typically involves discharging underflow from the Stage I and II treatment processes into Stage I and II settling ponds for clarification of the treated process water and sedimentation and settling of the sludges for permanent storage and disposal.

A "typical" phosphogypsum stack closure requires the treatment of 3.6 billion gallons of acidic process water (combined drainable pore water and ponded water) characterized by an elevated TDS concentration, say on the order of 40,000 mg/l, and 0.8 billion gallons of rainfall infiltration during the 50-year post-closure care period at a TDS concentration on the order of 10,000 mg/l. Based on the sludge production test results and considering typical *in situ* solids contents of 25% for the Stage I sludge and 12.5% for the Stage II sludge, the estimated corresponding storage capacity requirements for Stage I and Stage II sludge settling ponds would be on the order of 1,330 and 2,625 acre-feet, respectively. Settling area footprints on the order of 50 and 90 acres, respectively, would be needed considering 30- to 35-foot high retaining dikes with some allowance for freeboard and for sludge settling/decanting.

Construction and operation costs associated with building and operating the sludge settling areas, and reclamation costs during abandonment, need to be factored in when evaluating alternative disposal methods. The settling, consolidation, hydraulic conductivity and strength characteristics of the Stage I lime sludge will result in a somewhat less demanding closure effort of a Stage I sludge pond than a Stage II sludge pond. Nevertheless, it is important that a relatively low water level be maintained when feasible during the life of the pond to promote periodic desiccation of the sludge to facilitate closure construction. Sludge surfaces are typically regraded during closure using very low ground pressure equipment as needed to provide sheetflow of runoff. Considering the relatively low undrained strengths of the lime sludge, dewatering of the upper portion of the sludge in the settling pond will be required to promote further surface desiccation and consolidation of the sludge deposit (e.g., through the use of dewatering ditches or a drain system) and minimize mudwaving. Gradual placement of soil cover and mixing of the sludge surface with soil may be necessary to allow access for construction equipment in developing a network of dewatering ditches. Bermuda turf grass is usually seeded in sludge surface areas although natural vegetation often grows voluntarily. Gradually capping a sludge pond in this manner is tedious and time consuming. Expedited closure of a sludge pond may be undertaken using high tensile strength woven geotextile fabric to control and limit mudwaving of the sludge during placement of the soil cover. Associated costs with such a closure scheme can be significant.

ALTERNATIVE SLUDGE DISPOSAL METHODS

Solubility of Lime Sludge in Process Water

Results of solubility tests indicated that some of the Stage I and II lime sludge constituents dissolve and/or react with acidic process water by increasing the pH and reducing the conductivity of the process water, depending on the lime sludge loading rate and the quantity of free lime available in the sludge.

At low loading rates of 5% or less, the pH and conductivity of the process water remains relatively unchanged. At relatively high loading rates, the pH can be increased significantly, particularly using Stage II lime sludge (which is likely to contain more free lime than Stage I sludge). Note that in order to get full benefit of the neutralization, the sludge needs to be mixed vigorously with the process water (e.g., by discharging the slurried-sludge outflow from the dredge into a flowing process water ditch discharging into the cooling pond).

At operating plants, where raising the pH of the process water may not be desirable from an operational standpoint, relatively low loading rates of 5% may be feasible as long as the facility can cope with adverse impacts on process water balance and available surge storage capacity. At facilities where an entire phosphogypsum stack system is being closed, the data suggest that both Stage I and Stage II lime sludge can be beneficially used to assist in neutralizing acidic process water prior to treatment, thus reducing the quantity of lime ultimately needed for treatment. Settled solids resulting from the neutralization process will need to be contained along with settling of the reacted sludge. It is expected that after reacting with process water, the volume of sludge will not change substantially, particularly at low loading rates. In such a scheme, the settled sludge can be advantageously used in filling a below grade cooling pond system in preparation for closure.

Disposal of Sludge with Gypsum Slurry

Settling tests were used to give an indication of how lime sludge-gypsum mixtures may tend to segregate in a rim ditch after partially reacting with acidic process water in the slurry tank and slurry pipeline.

In general, lime-treatment sludge co-disposal with gypsum will result in a slight reduction of the storage life of a gypsum stack. Moreover, the process water treatment cost may be affected by co-disposal of lime-treatment sludges with gypsum during plant operation depending on the settled solids content of the mix and its dewatering characteristics. Higher settled solids content after co-disposal will result in larger volumes of decant water that may need to be treated if a facility has a positive water balance. Depending on the dewatering characteristics of the settled mix, the treatment cost could potentially be reduced if the finer lime-treatment sludge reduces to some extent the volume of drainable pore water. On the other hand, any additional decant water could be re-circulated and re-used in the plant processes for a facility having a negative water balance (i.e., a facility where net water consumption exceeds the net water inputs), with the added benefit of recovering some of the phosphate from the sludge (i.e., improved P_2O_5 recovery).

Co-disposal of lime-treatment sludge with phosphogypsum slurry atop a gypsum stack may be feasible when adding: (i) up to 2.5% of Stage I sludge to the gypsum (dry weight basis); and (ii) up to 1 percent Stage II sludge to the gypsum (dry weight basis), with the slurried mix discharged at initial solids contents on the order of 20-30%. Additional testing will be needed to determine sludge co-disposal rates that can be tolerated by any given facility from a stack stability standpoint, and preclude adverse impacts on handling, dewatering and compaction characteristics of the gypsum.

Amending Phosphogypsum with Lime Sludge to Promote Grass Growth

Application rates of 1% and up to about 6% of Stage II sludge (dry weight basis) appear to be suitable for amending leached and unleached gypsum, respectively, if the sludge contains some free lime.

Considering a typical phosphogypsum stack with 200 acres of slope area, and a gross average application rate of 1 inch of Stage II sludge at a solids content of 20% (dry density of 15 pcf), amendment of the gypsum slope area to promote grass growth would consume about 15 acre-feet of Stage II sludge (5,445 dry tons) or only about 1% of the volume of Stage II lime sludge produced during closure of a typical phosphogypsum stack system.

CONSUMPTION OF LIME TREATMENT EFFLUENTS

For the "typical" phosphogypsum stack example used to illustrate sludge storage requirements, more than 3.5 billion gallons of Stage II effluent would have to be significantly diluted with fresh water resources prior to discharge to surface waters of the State, unless other suitable disposal methods are adopted to consume or beneficially use the lime treatment effluents.

Amending Phosphogypsum with Stage II Effluents

The agronomic screening test results indicated that Stage II effluents do not have adequate buffering capacity to neutralize the acidity in unleached phosphogypsum, although Stage II effluents could be used to amend and sweeten leached phosphogypsum to achieve the target pH of 4.8 to 5.2 needed to promote grass growth. Nevertheless, results of the greenhouse study indicate that the media consisting of phosphogypsum amended with Stage II effluents will not be very suitable for healthy turfgrass growth. Lime-treatment effluents for phosphogypsum amendment (as opposed to lime sludges) will, therefore, have very limited use in stack slope closure applications associated with amending the phosphogypsum to promote grass growth. Nevertheless, Stage II effluents may be beneficially used for irrigating turfgrasses.

Spray Irrigation of Turfgrasses Using Treated Effluents

Based on results of the greenhouse study, both bermudagrass and bahiagrass grown in sandy soil can be irrigated with effluent from either (pH 7) single-stage treatment or conventional Stage II double-lime treatment effluent (diluted or undiluted). The current study also indicates that bermudagrass can successfully grow in both leached or unleached phosphogypsum media, properly amended with dolomitic limestone or with single-stage (pH 7.5) or Stage II lime-treatment sludges, and irrigated with effluents from diluted single-stage and Stage II lime treatment of process water. The study also indicated that seashore paspalum turfgrass can grow reasonably well in sandy soil when irrigated even with the more acidic Stage I (pH 5) effluent.

In general, the quality and density of turfgrass were relatively better during early stages of irrigation with lime-treatment effluents containing remnant nutrients such as nitrogen. The quality and density declined substantially, however, when irrigation continued with undiluted effluents, likely due to accumulation of salt in turfgrass growth media. Steady declines in the turfgrass quality were also observed over the study period when irrigated with diluted lower-conductivity, lower-nutrient effluents, likely due to a lack of nutrients over time, as fertilization was not undertaken during the irrigation treatment period. The decline in quality did not appear to be any worse than that exhibited by grass irrigated with freshwater (without any fertilization of the pots of grass). Additional fertilization fluid lacks nutrients.

Based on the findings of this study and barring restrictions imposed by surface water runoff and groundwater quality requirements, recycling of effluents generated by limetreatment of the process water to irrigate the grass cover on a closed phosphogypsum stack or grass pasture nearby appears to be technically viable. Therefore, spray irrigation, if properly managed, can be a feasible alternative to surface water discharge for "consumption" of treated process water. Of particular interest is the finding that grasses irrigated with effluents from conventional two-stage lime treatment up to a neutral pH on the order of 7.5 can sustain reasonable long-term vigor and health provided the application is controlled at rates that afford long-term dilution via rainfall leaching at dilution ratios greater than 3:1. Since the grasses appear to react reasonably well to irrigation with undiluted Stage II effluent over a short duration, a longer-term target "dilution" ratio can be achieved with the higher TDS effluents by adjusting spray irrigation rates and schedules in response to seasonal and cyclical rainfall patterns as needed to preclude elevated electric conductivity of the growth media. Plant tissue samples were obtained at terminal harvest for testing fluoride and arsenic contents of plants irrigated with treated effluents. The fluoride contents of plants irrigated with Stage II and Stage II+ effluents ranged from 30 to 80 µg/g, consistent with the tissue content of plants irrigated with tap water, and arsenic was not detected in any of the tested samples.

In terms of turfgrass health, the success of a spray irrigation system will depend heavily on the ability to manage long-term effects on soil conductivity. For instance, the application rate may need to be limited in order to maintain healthy grass which in turn provides higher evapotranspiration potential that in the long term will enable higher consumption of treated water. Other factors affecting irrigation rates from a turfgrass health standpoint relate to the characteristics of the treated process water and turfgrass growth media.

Another major consideration that may limit spray irrigation rates is related to surface and groundwater compliance issues in and around the perimeter of the land application area. In Central Florida, rainfall averages about 54 inches per year. For average climatic conditions (temperature, relative humidity, etc.) and typical hydrogeologic conditions, natural evapotranspiration is on the order of 37 inches per year and potential evapotranspiration from turfgrass is on the order of 62 inches per year. The difference between these values, after accounting for infiltration, base flow and runoff is about 26 inches per year on average. This gross irrigation requirement or safe average irrigation demand corresponds to about 0.5 inches of spray irrigation per week or about 1.3 gpm per acre. Large tracts of land will, therefore, be required to consume the treated water via spray irrigation. For example, consumption of a typical rate of 1,000 gpm of effluent will require a minimum 800 acres of irrigable land area.

Because the concentrations of sodium (~800 mg/l) and sulfate (2,500 to 5,000 mg/l) in the lime-treated water exceed the corresponding Class G-II groundwater primary and secondary drinking water standard MCLs (160 mg/l for sodium and 250 mg/l for sulfate, respectively), significant dilution with groundwater (and/or rainwater) is needed to reduce sodium concentrations (by a factor of up to 5) and sulfate concentrations (by a factor up to 20) in order to ensure compliance with groundwater standards at the edge of the regulated zone of discharge. Moreover, without dilution/dispersion, any seepage outcrops into adjacent relief ditches or wetlands may exhibit elevated specific conductance in excess of the 1275 umhos/cm Class III surface water standard. On the other hand, assuming nitrogen consumption of 300 lb/acre/year, and considering that the spray irrigation rate is limited to about 0.2 to 0.5 inches per week (i.e., 0.5 to 1.3 gpm/acre) or less, approximately 50 mg/l to 125 mg/l of the nitrogen concentration is expected to be readily consumed by the grass (i.e., without reliance on dispersion, dilution or other attenuation mechanisms). Similarly, assuming phosphorus uptake of 50 lb/acre/year, the corresponding consumption of phosphorus by the grass is expected to be on the order of 10 mg/l to 25 mg/l, and that may be the limiting factor. From the nutrient uptake standpoint, therefore, the post-aeration and acidulation Stage II+ (pH 7.5) effluent would be better suited for spray irrigation, because the fluid will be lower in phosphorus. For high nutrient effluents, such as Stage II (pH 7.5) or single-stage (pH 7.5) effluents, larger dilutions with freshwater (e.g., rainfall) and/or larger areas will be required in order to achieve nutrient reduction via grass uptake to reasonable levels.
Management tools that can be implemented to minimize impacts to groundwater and surface water and to preclude salt accumulation in the soil include: (i) dilution of the treated effluent with another "fresh water source" (e.g., R.O. permeate); (ii) rotational irrigation or use of alternating active and dormant plots for land application; (iii) reducing the spray irrigation rate to a fraction of the "safe average irrigation demand" (e.g., to less than 0.2 to 0.3 inches per week, or 0.5 to 0.8 gpm/per acre) to provide for adequate dilution by rainfall; (iv) maintaining a safe buffer zone between the land application area and the compliance point at the edge of the zone of discharge; and/or (v) modifying the application rate as needed based on results of water quality monitoring data in a downgradient seepage collection relief ditch/drain or downgradient monitor well.

Because much of the remnant nitrogen, including un-ionized ammonia (and some of the phosphorus) are effectively "removed" by root uptake, Stage II+ treatment with air stripping at high pH, may not be needed if spray irrigation can be implemented as an alternative to surface water discharge. Stage II (pH~7.5) effluent may then be directly used for spray irrigation unless a higher level of treatment is needed (say to pH~9) to further reduce remnant arsenic concentrations, inorganic constituents and conductivity from a groundwater or surface water compliance standpoint. Note also that as a result of appeals by various groups (including the Florida Department of Environmental Protection, FDEP) to rescind the numeric nutrient criteria recently proposed by the United States Environmental Protection Agency (USEPA) and the fact that the FDEP is in the process of finalizing its own equivalent criteria, it is unclear at this time to what extent the process water will have to be treated and polished prior to discharge. Nevertheless, if it can be implemented, spray irrigation would provide a substantial benefit in that reliance on valuable fresh water resources for dilution for the sole purpose of achieving Class III surface water standards prior to discharge could be substantially reduced or eliminated. Hence, the consumptive use of fresh groundwater for dilution would be significantly reduced, and the precious water resource saved for other beneficial uses. Moreover, if spray irrigation can be implemented with the reduced Stage II (pH~7.5) level of treatment, significant treatment cost savings would be realized.

The extent to which potential benefits may be realized would depend on the available land area for spray irrigation and the treatment rate employed for execution of a facility's water management plan. In some cases, such as during the post closure care period of an idled facility (particularly the latter years once pore water drainage rates from the closed gypsum stack system have declined substantially and the water quality has improved), spray irrigation could be very effective from cost and resource conservation perspectives. Another major benefit is the reduction in nutrient loadings to sensitive waters of the State.

Spray Evaporation of Treated Effluents

If the land areas available for spray irrigation of turfgrass are limited and/or the quality of surface water runoff from the irrigated areas is a concern, water consumption by spray evaporation in self-contained area(s) could be a viable option for disposing of lime-

treatment effluents. This practice has recently been one of the key factors in the successful closure of two phosphogypsum stack systems in Central Florida.

The spray evaporation system(s) can be placed on self-contained land or on ponded areas, whereby the influent water minus the evaporation plus the rain water will be collected on-site and re-sprayed. The containment dikes surrounding the system will need to be designed considering the water balance of the system to prevent overflows. If an impact to groundwater is of concern, lined areas may be used.

The success of a spray evaporation system depends on consistent maintenance. The small diameter spray nozzles used in the system are prone to plugging, which has to be manually cleaned, a labor-intensive task. Safety of maintenance personnel for spray system operated on ponds is another important issue with this system. Fluoride emissions could also be a limiting factor in low pH water. Nevertheless, a properly designed and maintained spray evaporation system can be very cost-effective in evaporating substantial quantity of treatment effluents, thus reducing or eliminating the need for fresh water resources that would be needed for dilution of lime-treatment effluents prior to discharge.

INTRODUCTION AND RESEARCH OBJECTIVES

The lime sludge disposal problem facing concentrated phosphate fertilizer plant operators in Florida is briefly described below along with the objectives and goals of the research. Past and on-going research by Ardaman & Associates, Inc. on measurement of lime sludge engineering properties and evaluation of lime sludge disposal methods relevant to establishing the research methodology and evaluation plan are also presented.

STATEMENT OF PROBLEM AND GOALS

For every ton of P₂O₅ produced from phosphate rock, about five tons of by-product phosphogypsum are generated. More than 850 million tons of phosphogypsum have been stockpiled in some 30 stacks covering over 5,000 acres of land in Florida in Polk, Hillsborough, Manatee and Hamilton counties. Even though the phosphate industry and the Florida Industrial and Phosphate Research Institute, formerly Florida Institute of Phosphate Research (FIPR), are engaged in research to find markets for utilization of phosphogypsum (e.g., road construction and agricultural amendments), it is unlikely that any significant amount of the 30 million tons per year of phosphogypsum typically generated in Florida will be utilized by alternative markets in the near future. The current inventory of phosphogypsum stacks, therefore, will remain, and the construction of new and/or contiguous expansions to existing phosphogypsum stacks will continue in the foreseeable future to sustain production of concentrated phosphate fertilizer and associated products.

Active phosphogypsum stacks are almost entirely saturated with acidic process water. For a typical overall average phosphogypsum stack *in situ* dry density of 80 to 85 pounds per cubic foot, the entrained pore water accounts for approximately 40% of the total volume of the stack. Hence, the estimated 850 million tons of phosphogypsum generated to date could contain up to 60 billion gallons of process water. For the current phosphogypsum generation rate in Florida of 30 million tons per year, the inventory of entrained process water in Florida phosphogypsum stacks will continue to increase by approximately 2 billion gallons per year (i.e., about 3% per year) until stack closures begin to reduce the inventory.

Seepage of pore water from a closed phosphogypsum stack occurs by gravity drainage, resulting in a gradual lowering of the phreatic water surface within the stack, and the reduction in the phosphogypsum moisture content from saturated to field capacity conditions. The field capacity moisture content of phosphogypsum is typically on the order of half the saturated moisture content. Accordingly, approximately half of the acidic pore water entrained within gypsum stacks, or 30 billion gallons, is in "temporary storage" since it will eventually drain once the stacks are closed. Further, a substantial volume of pore water will also drain from the stacks as a consequence of post-closure drained creep, and this could account for on the order of an additional 10 billion gallons of process water drainage. For a typical two-stage lime treatment cost of \$20 per thousand gallons of acidic process water, the treatment cost for the industry could eventually reach 800 million dollars.

The rate of pore water drainage from a closed phosphogypsum stack depends on many factors, including foundation conditions, type of internal drains, if any, stack size and height, and the phosphogypsum permeability and drained creep characteristics. Post-closure drainage studies undertaken by Ardaman & Associates, Inc. for phosphogypsum stacks in Florida indicate typical drainage rates of approximately 500 to 2,000 gallons per minute (gpm) during the first year of closure, and overall gross average drainage rates during the first 5 years of closure on the order of 200 to 500 gpm.

Depending on the plant water balance and process water surge storage capacity, some facilities may be able to consume a significant amount of process water while others may have to regularly treat and discharge excess process water. Closure of a gypsum stack at a facility already treating water will require increased treatment to handle the drainage of pore water. Similarly, other facilities that do not regularly treat may need to initiate treatment upon closure of a stack or upon plant shutdown to maintain Florida Department of Environmental Protection (FDEP)-mandated surge storage capacities within the phosphogypsum stack systems. Furthermore, long-term water management (i.e., during and after final closure of a phosphogypsum stack system and chemical plant) will eventually require treatment at all facilities.

Conventional Process Water Treatment Methods

Lime treatment is the most common method in use for treatment of excess acidic process water from phosphogypsum stack systems. Reverse osmosis systems have also been used (or tested on a pilot or demonstration scale) at several facilities. Attempts at development of a few more unconventional treatment methods are currently being made, and consumption of excess process water using fixed or floating spray evaporation systems has also been achieved at several facilities. Nonetheless, lime treatment remains the most commonly accepted method.

Conventional lime treatment typically employs a two-stage neutralization process, primarily because the solubility of some of the dissolved solids that precipitate as the pH is initially raised tend to increase at elevated pH levels. Hence, in order to effectively remove certain dissolved solids (e.g., fluoride), it is necessary to remove their precipitated solids from the treatment process prior to elevating the pH to the final target level required for removal of dissolved solids having low solubility at higher pH levels (e.g., phosphorus). For facilities having process water containing elevated ammonia nitrogen concentrations, the final target pH typically needs to be raised to more than 11 as needed to enable air-stripping of the unionized ammonia prior to final acidification back to a neutral pH suitable for discharge. A schematic of a typical double lime treatment system is presented in Figure 1.



Figure 1. Schematic of Typical Double-Lime Treatment System.

Stage I of a two-stage lime treatment process consists of mixing ground limestone (CaCO₃), lime (CaO) or slaked lime (Ca(OH)₂) with acidic (pH typically between 1.5 and 2.0) process water to elevate the pH to about 5 which results in precipitation of dissolved solids (i.e., most of the fluoride and some of the phosphate) through the formation of calcium insoluble salts such as calcium fluoride (CaF₂), chukhrovite (Ca₃Al₂(R.E.)SO₄F₁₃·10H₂O) where R.E. = rare earth mix with yttrium), monocalcium phosphate (Ca(H₂PO₄)₂·H₂O) and dicalcium phosphate (Ca(HPO₄)·2H₂O). Underflow from Stage I treatment is typically discharged into a settling pond for clarification and sedimentation of Stage I lime sludge, which consists mainly of calcium salts and some unreacted limestone and/or lime. Most of the metals and radionuclides, where present in the process water, also precipitate during Stage I treatment.

During Stage II treatment, clarified Stage I supernatant is mixed with lime once again to raise the pH to a target value dictated primarily by the level of treatment required and type of constituents in the process water (which depend mainly on the type of products produced at the plant). Underflow from the Stage II treatment reactor is typically routed into a clarifier or settling pond for sedimentation of the Stage II sludge which typically consists primarily of dicalcium phosphate, calcium hydroxyphosphate (which forms a gelatinous floc that has very poor settling and thickening properties), calcium fluoride and a fluoro-apatite complex. Where the process water contains a low ammonia nitrogen concentration, a Stage II target pH on the order of 9 is used in order to precipitate phosphate, remnant fluoride and remnant arsenic. When ammonia nitrogen concentrations are elevated, a higher Stage II target pH on the order of 11 is used to aid in removal of ammonia via air stripping. Effluent from "Stage II" (i.e., dissolved solids removal without air-stripping) or "Stage II+" (i.e., Stage II plus aeration at pH~11 for ammonia removal) is ultimately acidified to a pH of about 7 prior to discharge.

In some instances, such as when dilute process water at an idled facility is only partially treated prior to discharge during emergencies, single-stage lime neutralization may be employed. In some cases, pH of the remnant process water inventory in an idle stack system can approach the target first-stage pH of 4 to 5 due to long-term dilution, particularly if partial neutralization occurs over time due to disposal of lime treatment sludge (having an unreacted lime component) in process water impoundments and where acidic pore water drainage rates from idled phosphogypsum stacks have declined to low levels, along with dilution by rainfall infiltration. In these cases, the process water can be similar to the first-stage effluent of a conventional two-stage treatment system and hence single–stage treatment is akin to second-stage treatment. In other cases, the treated water quality requirements for a particular consumptive use (e.g., reuse at the plant or spray irrigation) may enable single-stage treatment to a neutral pH.

Stage I treatment generates lime sludge at a rate of less than about 100 to more than 300 dry pounds per 1,000 gallons of treated process water. Stage II treatment sludge generation is on the order of 100 to more than 200 dry pounds per 1,000 gallons of treated process water. The underflow from Stage I treatment typically has a solids content of 10-15%, and settles to a solids content of 20-30%. Stage II underflow typically contains 5-10% solids, and settles to a solids content of 10-15%. Corresponding sludge volumes equal 5 to more than 20 cubic feet of Stage I sludge per 1,000 gallons of treated process water, and 10 to 30 cubic feet of Stage II sludge per 1,000 gallons of treated process water.

A "typical" phosphogypsum stack system closure requiring the treatment of 3.6 billion gallons of acidic process water (Figure 2) will require the disposal of 2,400 acre-feet of Stage I and Stage II lime sludge and the discharge of approximately 2.8 billion gallons of Stage II effluent to surface water. The entire potential drainable process water inventory in Florida of 40 billion gallons will require the disposal of 25,000 acre-feet of lime sludge within sedimentation ponds, and the release of 30 billion gallons of Stage II effluent into surface waters. The treatment of acidic process water, therefore, as part of gypsum stack closures could generate considerable volumes of lime sludge requiring permanent on-land disposal and significant quantities of Stage II effluent discharge to surface water. The specific conductance of the effluent is typically significantly greater than the Class III surface water standard (i.e., 1275 µmhos/cm), and the industry frequently has to rely on significant dilution by fresh water and/or seek a variance from the Florida Department of Environmental Protection (FDEP) or a mixing zone for such discharges.



Figure 2. Typical Process Water Inventory and Lime Sludge Volume Generated from Treatment During Final Closure of a Phosphogypsum Stack System.

Surface Water Discharge Issues

Criteria for surface water quality applicable to discharge of treated water to Class III surface water is mandated under Rule 62-302, Florida Administrative Code (FAC). Although conventional double-lime treatment (with air stripping) effectively removes metals, radionuclides, fluoride, phosphorous and other nutrients, inorganic constituents including calcium, sodium and sulfate typically remain at elevated levels, and hence the conductivity normally exceeds the 1,275 µmhos/cm Class III Standard (Rule 62-302.350, FAC) by a factor of about 5. Hence, discharge of double-lime treated process water to Class III surface waters requires dilution to reduce conductivity. Considering that well water typically used at Florida facilities as a source for dilution water is mineralized to some extent, and hence displays a somewhat elevated conductivity itself, discharge of diluted effluent of double-lime treatment involves substantial use of fresh water resources.

In addition to conductivity issues, double-lime treated (i.e., Stage II) process water at facilities that produce (or historically produced) ammoniated products typically exceed the Class III surface water standard for un-ionized ammonia (i.e., ≤ 0.02 mg/l as NH₃). Lime treatment alone is not effective in removal of nitrogen, unless second-stage treatment to high pH levels and air-stripping is undertaken (i.e., Stage II+ treatment). Even then, neutralized

Stage II+ effluent may contain remnant ammonia nitrogen concentrations that, although low, can still be sufficient to have undesirable effects on the receiving water environment at high discharge rates and, hence, high nitrogen loadings (which is in violation of the Class III Standard for nutrients which states that "in no case shall nutrient concentrations of a body of water be altered so as to cause an imbalance in natural populations of aquatic flora or fauna"). Hence, even when extensive air-stripping is undertaken, treatment rate and discharge restrictions may still be necessary particularly in light of recently proposed numerical nutrient criteria being promulgated to further minimize nutrient loadings.

Considering that long-term reliance on continued use of substantial fresh water resources for dilution of lime-treated process water for compliance with the Class III surface water standard is not in the best interest of the industry or the State, alternative means for onsite consumption or utilization of treated process water need to be developed. In concept, spray irrigation can be used as an alternative to discharge of treated effluents to surface waters by taking advantage of natural or enhanced evapotranspiration losses. By uniformly applying treated or partially treated process water at controlled rates over a sufficiently large land area having a healthy vegetation cover, seasonal deficits between rainfall and evapotranspiration can be exploited as a means of consumption. At least two facilities in Florida have utilized spray irrigation systems in conjunction with their water management plans. Long-term effectiveness of spray irrigation depends on the ability to maintain healthy vegetation within the application area since evapotranspiration rates need to be sustained as high as possible. Further, although infiltration of land-applied effluents can be controlled by carefully adjusting spray application rates in response to seasonal climate and growth variations as well as in response to shorter-term weather systems, or by alternating the use of several spray fields through active and dormant cycles, collection of seepage near the boundary of the application area may be required if leaching of constituents contained in the effluent is projected to present either surface water or groundwater compliance issues over time. In particular, lime treated process waters generally exceed the Class G-II groundwater standard for sodium (160 mg/l - primary Maximum Contaminant Level (MCL) standard) and sulfate (250 mg/l - secondary MCL standard) by factors of more than 5 and 15, respectively, and the Class III surface water standard for conductivity (1275 µmhos/cm) by a factor of 5 or more.

Lime Sludge Characterization

Presently, there is no significant data base of lime sludge engineering properties available for use in disposal planning and design, or correlations between process water composition and actual mass of lime sludge produced for estimating disposal requirements. One of the objectives of the research, therefore, was to provide a data base of lime sludge engineering properties that can be used for disposal planning and design.

Conventional Sludge Disposal Methods

The conventional disposal methods currently used will need to be evaluated to address: consolidation behavior during deposition within settling ponds as related to pond sizing; dewatering, desiccation and capping as related to reclamation of settling ponds; and drainage, dewatering, desiccation and capping as related to potential disposal of lime sludge in lined ponds atop closed gypsum stacks.

Alternative Disposal of Sludge and Treated Water

A "typical" phosphogypsum stack system could require the disposal of 2,400 acrefeet of lime sludge and discharge of approximately 3 billion gallons of Stage II supernatant. Another objective of this research was to evaluate alternative disposal methods for lime sludge and supernatant to reduce on-land disposal in sedimentation ponds and discharges of Stage II effluent to surface water. The following alternative disposal methods were evaluated:

- **Co-disposal** of lime sludge underflow or lime sludge dredged from sedimentation ponds with phosphogypsum slurry atop gypsum stacks in a manner that does not adversely impact wet-stacking operations, the life of the stack, or stack stability. The effects of co-disposal on plant water balance and treatment costs also need to be considered.
- **Dissolution** of or reactivity of lime sludge within acidic process water in a manner that does not adversely impact the process water system of an operating plant, or in the acidic process water of a phosphogypsum stack system undergoing closure in a manner that utilizes the neutralization capacity of the sludge to reduce Stage I lime requirements. The effects of dissolution of lime sludge on water balance and treatment costs will need to be addressed.
- **Recycling** of Stage II supernatant to irrigate the grass cover on a closed phosphogypsum stack or a grass pasture nearby to reduce the volume of Stage II effluent discharged to surface water.
- Utilization of lime sludge as an agronomic amendment in gypsum stack side slope final covers, in lieu of dolomitic limestone, to raise the pH of *in situ* phosphogypsum and provide nutrients for grass growth.

PAST AND PRESENT RELATED RESEARCH

Vegetating Amended Phosphogypsum

The ability to vegetate a 6-inch thick amended phosphogypsum layer on the side slope of a stack was well demonstrated by Richardson and others (1995) based upon research conducted over a 4-year period from 1990 to 1994 at the IMC-Agrico (now Mosaic) New

Wales, U.S. Agri-Chemicals Bartow and Estech Silver City phosphogypsum stacks. The results of that study indicated that the addition of dolomitic limestone at a rate of about 1 ton per acre into weathered/leached phosphogypsum with an initial pH ranging from about 4.5 to 5.0 noticeably enhanced plant (i.e., grass) growth. The enhanced plant growth achieved by addition of dolomitic limestone was attributed by Richardson and others (1995) primarily to the increased supply of magnesium, although it was also in part a result of increasing the pH by half a unit (see Figure 3).

At locations where the phosphogypsum pH is less than 4, such as may occur in relatively unweathered/unleached phosphogypsum or in phosphogypsum below the seepage line on a stack slope, higher application rates of dolomitic limestone will be needed to increase the pH to above 4.

Richardson and others (1995) and Fuleihan and others (2005) have shown that weathered/leached phosphogypsum typically displays a pH above 4, which is satisfactory for establishing a vegetation cover. Accordingly, the pH of initially highly acidic surfaces in a phosphogypsum stack (e.g., below the line of seepage) will increase if the phosphogypsum is allowed to weather and leach, i.e., once a stack begins to dewater and/or after side slope drains are installed. Although it is uncertain how long the weathering/leaching process will need to occur in the field, the data demonstrate the beneficial effects of allowing a stack to drain as much as possible and the acidity of phosphogypsum at the surface to decrease as much as possible by weathering and rain water infiltration prior to installing a side slope final cover.

The field studies conducted by Richardson and others (1995) indicated that common Bermuda grass was the best adapted grass, of those tested during the investigation (i.e., common Bermuda grass, weeping love grass and Pensacola Bahia grass), for establishing a grass cover on phosphogypsum. Fuleihan and others (2005) also showed that common Bermuda grass performs well in dolomitic limestone amended phosphogypsum side slope final covers.



Source: Adapted from Richardson and others (1995).

Figure 3. Effect of 1 Ton/Acre of Dolomitic Limestone Amendment on Raising pH of Leached and Unleached Phosphogypsum.

Properties of Lime Sludge

Several laboratory test programs have been conducted by Ardaman & Associates, Inc. over the past 15 years to characterize the engineering properties of sludge generated from lime treatment of phosphogypsum stack system acidic process waters. The following geotechnical properties of lime sludge were determined from the studies.

Index Properties

Stage I and Stage II lime sludge typically display specific gravities of 2.90 to 3.00. Typical particle-size distributions of Stage I and Stage II lime sludge, from sieve and hydrometer test methods, are presented in Figures 4 and 5 and compared with the typical range of particle-size distribution for phosphogypsum. As shown, lime sludge displays a particle-size distribution finer than phosphogypsum. Stage I sludge contains equivalent siltand clay- size fractions of 40-50%, with minor amounts of sand-size particles. Stage II sludge is finer than Stage I sludge, and is characterized by 70% clay-size particles and minor amounts of silt- and sand-size particles. Because of the finer particle-size and gelatinous nature, Stage II lime sludge generally settles to a lower solids content, consolidates slower, and is less permeable, more compressible, and exhibits lower strength than Stage I lime sludge.



Figure 4. Particle Size Distribution of Stage I Lime Sludge.

U.S. STANDARD SIEVE SIZE



Figure 5. Particle Size Distribution of Stage II Lime Sludge.

The Atterberg limits (i.e., liquid limit and plasticity index) of lime sludge are presented in Figure 6. As shown, the lime sludge displays Atterberg limits characteristic of elastic silts, and classifies as an MH-type silt in accordance with the Unified Soil Classification system.



Figure 6. Plasticity of Lime Sludges.

Consolidation Properties

The consolidation properties of lime sludge are important for estimating behavior during deposition in sedimentation ponds, sizing sedimentation ponds, and estimating postclosure settlements following installation of a soil cap. The results of one-dimensional laboratory slurry consolidation tests on a Stage I and Stage II sludge are presented on Figure 7. As shown, the Stage II sludge displays a significantly lower solids content at a given effective consolidation stress and is more compressible than the Stage I sludge.

The coefficient of consolidation, which governs the rate of primary consolidation, and coefficient of secondary compression, which governs the rate of drained creep following primary consolidation, measured on a Stage I and Stage II sludge are presented in Figure 8. As shown, the lime sludge displays a coefficient of consolidation of 0.002 to 0.02 cm² per second, which is relatively rapid. Stage I sludge consolidates at a rate approximately twice that of the finer-grained Stage II sludge. Because of the elastic silt nature of the lime sludge, the rate of consolidation is still 10 to 100 times faster than typical for plastic, CH-type, waste phosphatic clays. The rates of secondary compression for lime sludge are relatively low, varying from 0.1-0.3%, and are about 3 to 10 times less than values exhibited by plastic CH-type, waste phosphatic clays.



Figure 7. Compressibility of Stage I and Stage II Lime Sludges.



Figure 8. Consolidation Properties of Stage I and Stage II Lime Sludges.

Hydraulic Conductivity

The hydraulic conductivity of lime sludge, which governs the rate at which a fluid can flow through the sludge, is presented in Figure 9. As shown, based on limited data, the hydraulic conductivity varies from 10^{-4} to 10^{-6} cm/sec and decreases with increasing solids content. The hydraulic conductivity of Stage I sludge is 100 times higher than that of the finer-grained Stage II sludge at the same solids content, and 3 to 10 times higher when consolidated under the same effective stress.



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Figure 9. Hydraulic Conductivity of Stage I and Stage II Lime Sludges.

Shear Strength

The shear strength of lime sludge is of interest for evaluating the requirements for placement of a soil cap atop a sedimentation pond during closure. The undrained shear strengths of saturated lime sludge measured with a laboratory vane at solids contents ranging from 10-50% are shown on Figure 10. At low solids contents of 15-30%, typical of values that occur at the end of settling, the undrained shear strength is less than 30 psf, characteristic of a very soft, very low strength material. At solids contents approaching 40 percent, typical of values following self-weight consolidation in sedimentation ponds, the undrained shear strength approaches 200 to 300 psf, characteristic of a soft, weak material. Based on these limited data, the soft consistency and low strength of lime sludge will require surface dewatering, desiccation and potentially staged construction to install a soil cap during closure. Although the undrained strength of lime sludge at low solids contents is relatively low, the drained strength will be higher because of the relatively high angle of internal friction of about 40° exhibited by lime sludge (Figure 11).



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Figure 10. Undrained Shear Strength of Stage II Lime Sludge.

Solubility of Lime Sludge in Process Water

A series of experiments were previously performed by Ardaman & Associates, Inc. to investigate the effects of adding lime sludge to acidic process water (pH of 2.0 and conductivity of 21,000 μ mhos/cm) to document the amount of lime sludge that can be dissolved or reacted in process water, and the resulting changes in pond water chemistry as a function of lime sludge loading rate.



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Figure 11. Effective Stress Paths from Consolidated Undrained Triaxial Compression Tests on Undisturbed Lime Sludge Samples.

The effect of lime sludge addition on the pH and conductivity of acidic process water is illustrated in Figure 12. As shown, at low loading rates of 5% or less, the pH and conductivity of the process water remains relatively unchanged. At relatively high loading rates, the pH can be increased significantly, particularly using Stage II lime sludge. At operating plants, where raising the pH of the process water may not be desirable from an operation standpoint, relatively low loading rates of 5% are feasible. At facilities where an entire phosphogypsum stack system is being closed, the data suggest that both Stage I and Stage II lime sludge can be used to effectively neutralize acidic process water prior to treatment and reduce the quantity of lime needed for treatment.



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Figure 12. Effect of Lime Sludge Addition on pH and Conductivity of Process Water.

Sedimentation Characteristics of Phosphogypsum-Lime Sludge Mixtures

Lime sludge particles are finer than phosphogypsum particles (Figures 4 and 5). Hence, segregation of lime sludge from phosphogypsum during settling within rim-ditches or compartments atop a stack could potentially adversely impact wet-stacking construction operations and slope stability. Further, lime sludge will react with acidic process water, and, as a result, some lime sludge solids will dissolve, and some of the solids will change in chemical and physical composition. Depending on contact time, the settling behavior of lime sludge-gypsum mixtures in rim ditches may differ from the settling behavior in a stack compartment. Settling tests can be used to give an indication of how lime sludge-gypsum mixtures may tend to segregate in a rim ditch after partially reacting with acidic process water in the slurry tank and slurry pipeline. Hydrometer and solubility tests, however, may give a better indication of how lime sludge-gypsum mixtures behave in a settling compartment after fully reacting with acidic process water. The properties of reacted lime sludge, rather than unreacted lime sludge, therefore, will be important for characterizing the settling behavior and potential effects of the settled material on stack operation and stability.

A series of limited experiments were previously performed by Ardaman & Associates, Inc. to determine the effect of lime sludge addition on the settling behavior of phosphogypsum slurry, and evaluate the feasibility of the co-disposal of lime sludge with phosphogypsum slurry atop a gypsum stack. The results of laboratory settling tests on mixtures of phosphogypsum slurry with 2-12% lime sludge added by dry weight (Figures 13 and 14) indicate that at low concentrations (i.e., $2\%\pm$) Stage I sludge can be added to phosphogypsum slurry without adversely affecting settling behavior. Stage II sludge had more of an effect on settling behavior, and the experiments indicate that about 1% can be added to phosphogypsum without significantly affecting settling behavior. Hence, these limited experiments suggest that it will be feasible to co-dispose of at least some lime sludge with phosphogypsum atop active gypsum stacks in a controlled manner, without adversely affecting stack operations and stability.



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Figure 13. Effect of Stage I Lime Sludge Addition on the Settling Behavior of Phosphogypsum Slurry.



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Figure 14. Effect of Stage II Lime Sludge Addition on the Settling Behavior of Phosphogypsum Slurry.

RESEARCH PURPOSE AND OBJECTIVES

Considering the potential volume of acidic process water eventually requiring treatment as part of gypsum stack closures in Florida, and the associated treatment costs and lime sludge disposal requirements, any alternative utilization or disposal methodologies reducing the volume of lime sludge requiring on-land disposal within sedimentation ponds or reducing the volume of Stage II effluent discharged to surface water will benefit the industry and the environment. The objectives of this research, therefore, were to investigate the following topics, illustrated in Figure 15, related to treatment of acidic process water during closure of phosphogypsum stacks or phosphogypsum stack systems.

- Characterization of the engineering properties of lime sludge relevant to disposal, and the volumes of Stage I and Stage II lime sludge and supernatant generated during treatment of acidic process water as a function of the initial chemical composition of the process water
- Evaluating conventional lime sludge disposal methodologies relevant to characterizing settling pond sizes, capping settling ponds during reclamation, and potential disposal of lime sludge in lined ponds atop closed gypsum stacks.
- Evaluating alternative disposal methods for lime sludge and supernatant to

reduce on-land disposal within sedimentation ponds and minimize discharge of Stage II effluent to surface water. The alternative disposal methods include: (i) recycling lime sludge within phosphogypsum stack systems, such as by codisposal with phosphogypsum slurry within settling compartments atop gypsum stacks; (ii) redissolution of Stage II sludge within acidic process water; and (iii) irrigating grass with Stage II effluent to reduce discharges to surface water.

• Utilization of lime sludge or supernatant as an agronomic amendment in gypsum stack side slope final covers, in lieu of dolomitic limestone, to raise the pH of *in situ* phosphogypsum and provide nutrients for grass growth.

RESEARCH OVERVIEW

The research was divided into the following four tasks to accomplish the specified objectives:

- **Task A—Evaluation of Sludge Production**: Characterized the quantities of Stage I and Stage II lime sludge produced per gallon of treated process water for different initial process water pH and total dissolved solids.
- **Task B—Evaluation of Sludge Engineering Properties**: Characterized Stage I and Stage II lime sludge properties relevant to conventional disposal in sedimentation ponds.
- Task C—Evaluation of Sludge Agronomic Properties: Investigated the suitability of utilizing Stage II sludge, Stage II supernatant, or single-stage pH 7 sludge as agronomic amendments in gypsum stack side slope final covers in lieu of dolomitic limestone using green house plant growth studies.
- **Task D—Evaluation of Alternative Disposal Methods**: Evaluated alternative disposal methods for lime sludge and supernatant to minimize on-land disposal within sedimentation ponds and discharge of Stage II effluent to surface water. The evaluated alternative disposal methods included: (i) recycling lime sludge within phosphogypsum stack systems, by co-disposal of lime sludge with gypsum within the stack; (ii) dissolution of lime sludge in acidic process water; and (iii) irrigating grass with Stage II effluent to reduce discharges to surface water.

Task A—Evaluation of Sludge Production

The quantity of Stage I and Stage II lime sludge produced as a function of initial process water quality was evaluated in laboratory experiments for 3 different acidic process waters representing the range of total dissolved solids concentrations typically observed in Florida phosphoric acid plant process waters. Adequate lime was added to achieve a target pH value of 5 for Stage I treatment, and 11 for Stage II treatment of the decanted Stage I supernatant. The Stage II supernatant was re-acidified to a pH of 7 as typical prior to discharge to surface water.

The following parameters were measured on each of the experiments:

- Mass and solids content of settled sludge
- Mass of lime added to achieve target pH
- Chemistry of the decanted supernatant
- Particle-size distribution of settled sludge
- Specific gravity of settled sludge
- Atterberg limits of settled sludge







B. ALTERNATE DISPOSAL IN PHOSPHOGYPSUM STACK SYSTEM



C. RECYCLING FOR USE IN CLOSURE OF PHOSPHOGYPSUM STACK SYSTEM

Figure 15. Conventional and Alternative Disposal of Process Water Treatment Sludge and Supernatant.

These experiments allowed a characterization of the quantity and type (i.e., particlesize and plasticity) of lime sludge, and quantity and quality of supernatant produced from 2stage lime treatment of process waters at various initial pH and total dissolved solids concentrations. The experimental data were then used to refine estimates of volumes of lime sludge and supernatant generated during conventional treatment and disposal, and the reductions in disposal volumes and Stage II supernatant discharges achievable utilizing alternative recycling and disposal methods.

Task B—Evaluation of Sludge Engineering Properties

Stage I and Stage II lime sludge were sampled from 5 lime treatment plants for characterization of engineering properties relevant to conventional disposal in sedimentation ponds. Lime sludge was sampled from the following plants: IMC- Agrico (now Mosaic) Nichols and Faustina plants (Plants N and A, respectively), CF Industries Bartow Plant (Plant C), PCS Phosphate Dorr-Oliver Plant (Plant D), and Farmland (now Mosaic) Green Bay Plant (Plant B). Each sample of sludge was initially screened with a series of index tests to characterize the solids content, pH, particle-size, specific gravity, Atterberg limits, settling characteristics, solubility in pH 2 process water, lime content, and supernatant pH, conductivity, SO₄, F, total P and NH₄ concentrations.

From the screening tests, 3 sites and corresponding Stage I and Stage II sludge, representative of the range of index properties observed for lime sludge, were selected for measurement of engineering properties relevant to evaluating conventional disposal in and capping of sedimentation ponds. The additional testing included field sampling of *in situ* sedimented Stage I and Stage II lime sludge at each site, and the laboratory measurement and characterization of the consolidation and strength properties of sedimented Stage I and Stage II sludge for comparison with field measured values. The field and laboratory experimental data were used to characterize the engineering properties of lime sludge, and provide a data base from which to select engineering properties of lime sludge for sizing and capping sedimentation ponds.

Task C—Evaluation of Sludge Agronomic Properties

Stage I lime sludge, Stage II lime sludge and Stage II supernatant from each of the 3 selected representative sites were evaluated for use as an agronomic amendment in phosphogypsum stack side slope final covers in lieu of dolomitic limestone. Each of the sludge samples, and Stage II supernatants was mixed with both leached (i.e., pH>4) and unleached (i.e., pH<3) phosphogypsum from the corresponding sites at 3 different application rates to determine if an amended phosphogypsum target pH of 4.8 to 5.2 can be achieved. The pH, conductivity and moisture content of each of the mixtures was measured to identify an application rate to achieve the target pH without introducing too high a salt concentration to the gypsum that would limit plant growth.

Based upon these experiments, selected mixtures of phosphogypsum amended with the selected application rates of Stage I sludge, Stage II sludge, and Stage II supernatant with both leached and unleached phosphogypsum were further analyzed for pore fluid SO₄, total P, F and NH₃ concentrations, total and effective porosity, and hydraulic conductivity.

Selected mixtures were also used in greenhouse plant growth studies to demonstrate the ability of the mixtures to support the growth of Bermuda and Bahia grass.

Task D—Evaluation of Alternative Disposal Methods

Based upon the findings of the experiments from Tasks A, B and C, an evaluation was performed to identify the reductions in lime sludge disposal volumes within conventional sedimentation ponds and discharges of Stage II supernatant to surface water potentially achievable using the alternative recycling and disposal methods of: (i) codisposal of lime sludge with phosphogypsum atop the gypsum stack; (ii) dissolution of lime sludge within acidic process water; and (iii) irrigating grass with Stage II effluent.

The co-disposal and dissolution alternatives resulted in the reaction of lime sludge with acidic process water. Hence, the properties and characteristics of lime sludge after reacting with acidic process water, in addition to the properties and characteristics of conventionally disposed of lime sludge, are important for comparing and evaluating alternative disposal methods. The effects of co-disposal and dissolution disposal methods on plant water balance and treatment costs were also considered. The feasibility of irrigating grass with Stage II effluent was evaluated as part of the Task C greenhouse plant growth study, and the corresponding potential reduction in discharges of Stage II effluent to surface water was evaluated.

EVALUATION OF SLUDGE PRODUCTION

SAMPLE COLLECTION AND PLAN OF STUDY

Acidic process waters representing the range of total dissolved solids concentrations typically observed in Florida phosphoric acid plant process waters were sampled to analyze and characterize the water and to use in sludge production experiments. Process water samples were obtained from the following plants:

- Plant A sampled from the cooling pond on July 10, 2001.
- Plant B sampled from the cooling pond on April 20, 2001 and January 22, 2002.
- Plant C essentially idle facility since 1985; sampled from the cooling pond on April 19, 2001 and January 22, 2002.
- Plant D sampled from the cooling pond on April 27, 2001.
- Plant N essentially idle facility since 1998; sampled from the cooling pond on April 17, 2001 and January 23, 2002.
- Mulberry Phosphates Inc. (MPI) facility facing bankruptcy proceedings; sampled from the cooling pond on March 14, 2001, February 15, 2002 and May 22, 2002¹.
- Piney Point Phosphates (PPP) facility facing bankruptcy proceedings; sampled from the cooling pond on March 14, 2001, February 15, 2002 and May 22, 2002¹.

The pH and conductivity of the process water at each plant was field checked at several locations in the cooling ponds and settling compartments atop the gypsum stacks. Samples were collected based on lower pH and higher conductivity measurements of the process water obtained from each plant.

Samples of supernatant from Stage I and Stage II Treatment Settling Ponds were obtained from Plants B, C, D and N. Underflow effluents were obtained and analyzed from Plant A.

The laboratory testing program on the collected process water samples consisted of:

- Characterization of acidic process water samples;
- Stage I and Stage II lime treatment experiments; and
- Single-stage lime treatment experiments.

The Stage I, Stage II and single-stage lime treatment experiments were evaluated with regard to:

¹ Some of these data were previously documented in a letter to FIPR titled "Feasibility Study to Screen Emergency Treatment Alternatives for Mulberry and Piney Point Phosphates Process Waters," Fuleihan NF and Ingra TS, October 2001. Identified on figures as FIPR/FDEP (2001).

- Sludge production
- CaO utilization
- Settling test results
- Characterization of treated effluent
- Engineering properties of produced sludges (presented in subsequent section titled "Evaluation of Sludge Engineering Properties")

CHARACTERIZATION OF ACIDIC PROCESS WATER SAMPLES

The test results on the chemical composition of the process water samples are presented in Table 1. As shown in Table 1, pH of the process water samples varied from 1.8 to 3.1, and the conductivity ranged from 8,670 to 28,000 μ mhos/cm as measured in the Ardaman laboratory.

Samples of the process waters were sent to either ENCO, TestAmerica, Inc. or Pembroke Laboratories, Inc. for additional chemical analyses. These results indicate that the acidic solutions exhibited a low pH in the range of 1.6 to 2.8 and contained high amounts of dissolved solids (TDS ~ 9,600 to 41,500 mg/l). Constituents with the greatest concentrations include sulfate (2,684 to 6,225 mg/l), sodium (760 to 4,000 mg/l), phosphorus (1,400 to 7,150 mg/l), ammonia nitrogen (154 to 1,240 mg/l), fluoride (150 to 5,560 mg/l) and calcium (185 to 1,400 mg/l).

The process waters sampled from Plants C, N, MPI and PPP are higher in pH and lower in total dissolved solids than waters sampled from Plants A, B and D because the former plants had been shut down for some time prior to sampling. The average pH and total dissolved solids for the idle plants equaled 2.6 and 18,000 mg/l, respectively, while the average pH and total dissolved solids for the active plants equaled 1.9 and 37,000 mg/l, respectively.

STAGE I AND STAGE II LIME TREATMENT EXPERIMENTS

The quantity of Stage I and Stage II lime sludge produced as a function of initial process water quality was evaluated. Process waters from Plants B, C, D, N, MPI and PPP were used. The process water was initially treated with lime to a target pH of 5.0 to produce Stage I sludge. The supernatant from the Stage I sludge was then treated with additional lime to a target pH of 11.0 to produce the Stage II sludge. The quantity of lime slurry needed to attain the target pH, the mass and solids content of sludge generated, and the quantity and quality of the treated effluent were determined for each experiment.

The quantity of Stage I sludge produced at the initial pH of each acidic process water (i.e., pH ranging from 1.6 to 3.1), was determined experimentally in the laboratory. For process water from Plants B, C, D, and N, about 4.8 gallons of process water were placed in a 5-gallon bucket to where lime slurry was added and agitated to produce Stage I sludge. For

Facility	Plant A	P	ant B	Pla	nt C	Plant D	Plant N		MPI Site PPP Site			
Sample I.D.	PW-A1	PW-B1	PW-B2	PW-C1	PW-C2	PW-D1	PW-N1	PW-N2	PW-M1	PW-P1	PW-P2	PW-P3
Sampling Date	7/10/01	4/20/01	1/22/02	4/19/01	1/22/02	4/27/01	4/17/01	1/23/02	3/14/01	3/14/01	2/15/02	5/22/02
Lab Parameters (Ardaman)												
Lab pH	1.9	1.9	1.8 [A]	2.7 [A]	2.7 [A]	1.8	2.1 [A]	2.1 [A]	2.4	3.1	2.9	2.9
Conductivity (µmhos/cm)	21,100	28,000	26,100 [A]	11,400	8,670 [A]	23,250[A]	17,700 [A]	11,170[A]	22,100	13,800	9,500	10,750
TESTING LABORATORY	TA	TA	PL	TA	PL	TA	TA	PL	TA	TA	ENCO	ENCO
Major Constituents (mg/l)												
Calcium, Ca	1,190	1,400	185	1,110	260	1,150	570	-	540	600	580	551
Magnesium, Mg	462	236	401	196	387	213	177	-	223	242	230 [A]	229
Sodium, Na	2,475	2,080	4,000	1,270	760	1,240	1,350	-	1,720	1,560	1,300	1,290
Potassium, K	485	153	652	196	530	253	149	-	210	226	210	196
Iron, Fe	181	142	184	15	13	120	32	-	59	6	5	6
Manganese, Mn	30	14	17	10	11	16	7	-	15	3	2 [A]	3
Chloride, Cl	56	448 [M]	763	33	64	108	79	-	140	144	110	110
Fluoride, F	3,040	5,560	5,140	362	250	4,170	1,490	1,000	3,800 [A]	304 [A]	169 [A]	150
Sulfate, SO ₄	5,200	5,900	5,595	2,780 [P]	2,684	4,300	5,280	-	6,150 [A]	6,225 [A]	4,200	5,200
Total Phosphorus, P	6,050 [P]	7,150	6,830	3,750	3,340	5,600	3,400	2,860	6,400 [A]	1,650 [A]	1,530 [P]	1,400
Total Kjeldahl Nitrogen, N	450	760 [M]	889	280	349	650	700	619	1,340	780	641 [P]	650
Ammonia Nitrogen, as N	154	830 [M]	723	220	-	645	650	-	1,240	730	635 [P]	600
Ammonium Ion, NH ₄	-	0.33	-	0.09	-	0.26	0.26	-	0.50	-	-	-
	22 700	20.000	22.445	5 000 (E)		20.000	16000					
Acidity as $CaCO_3$	33,700	38,000	32,445	7,000(E)	7,787	28,000	16,800	-	-	-	-	-
Alkalinity as CaCO ₃	1.0 [U]	1.0[U]	0.1 [U]	1.0 [U]	0.1 [U]	1.0[U]	1.0 [U]	-	-	-	2[U]	0.65 [U]
Solids, Total Dissolved	34,400	41,500	38,015	19,300	15,086	32,900	20,000 [R]	17,376	36,900[P]	16,800	9,600 [A]	11,500
Solids, Total Suspended	-	-	-	-	-	-	-	-	22	144	58	120
Other Parameters								. –				
Lab pH	1.7	1.6	1.9	2.6	2.6	1.7	2.0	1.7	2.1	2.8 [A]	2.9	2.4
Conductivity (µmhos/cm)	-	14,800	29,800	19,600	11,630	23,400[P]	58,600	20,100	23,780[P]	-	10,000	10,983
NOTES : $TA = Test America, In$	c.; PL = Pe	mbroke La	aboratories, In	ic.; ENCO =	= ENCO La	boratories,	lnc.					

Table 1. Chemical Composition of Process Water Samples.

[A] Reported value is the average of two or more determinations; [P] Reported value determined by Pembroke Laboratories, Inc.

[R] Result confirmed by rerun analysis; [M] Matrix interference prevented accurate determination;
[U] Material was analyzed for but not detected. (The reported value is the minimum detection limit); (E) Estimated value.

Plants MPI and PPP process water, the initial quantity was about 0.9 gallons. Chemical Lime -14 Hi Calcium Quicklime (90% average available CaO) was hydrated to create a 10% slurry by mass (slaked lime). The hydrated lime slurry was gradually added to the process water in the 5-gallon bucket and continuously stirred until a target pH value of 5.0 was achieved for Stage I treatment. The elapsed time and volume of hydrated lime slurry added was recorded. The slurry of treated process water and sludge generated was poured into a Plexiglass settling column to an initial height of about 100 cm for Plants B, C, D and N; 45 cm for MPI; and 58 and 100 cm for PPP. The height of the settling interface was monitored with time. After 24 hours, the settling tests were taken down and the final water content, volume of effluent, and solids content of the settled sludge were determined. A sample of the treated process water was removed for chemical analysis.

The collected effluent from the Stage I treatment experiments was adjusted to a target pH value of 11.0 by adding additional lime slurry to simulate Stage II treatment. Thereafter, the Stage II supernatant was acidified to a pH of 7, as typically done before discharge to surface water. Aeration typically undertaken in the field was not performed in the laboratory experiments, thus, treated ammonia and N parameters are not intended to be representative of fully treated Stage II+ aerated water. The elapsed time and volume of hydrated lime slurry added were recorded. The resultant slurries were poured into settling columns to an initial height of about 90 to 100 cm for Plants B, C, D and N; and 58 and 100 cm for the PPP Plant and settling tests were performed. After 24 hours, the settling tests were taken down and the final water content, volume of effluent, and solids content of the settled sludge were determined. A sample of the treated process water was removed for chemical analysis.

SINGLE-STAGE LIME TREATMENT EXPERIMENTS

Process waters from Plants B, C and N were single-stage treated with lime to a target pH value of 7.0. Process waters from MPI and PPP Plants were treated in a single-stage with lime to target pH values ranging from 5.0 to 8.0. The quantity of lime slurry needed to attain the target pH, the mass and solids content of lime sludge generated, and the quantity and quality of the treated effluent were determined for each experiment.

For process water from Plants B, C and N, about 4.8 gallons of process water were placed in a 5-gallon bucket to where lime slurry was added and agitated to produce single-stage sludge. Chemical Lime -14 Hi·Calcium Quicklime (90% average available CaO) was hydrated to create a 10% slurry by mass (slaked lime). The hydrated lime slurry was gradually added to the process water in the 5-gallon bucket and continuously stirred until a target pH value of 7.0 was achieved for single-stage treatment. The elapsed time and volume of hydrated lime slurry added was recorded. The slurry of treated process water and sludge generated was poured into a Plexiglas settling column to an initial height of ranging from 60 to 130 cm. The height of the settling interface was monitored with time. After 24 hours, the settling tests were taken down and the final water content, volume of effluent, and solids content of the settled sludge were determined. A sample of the treated process water was removed for chemical analysis.

For the process water from Plants MPI and PPP, the single-stage treatment experiments were conducted by treating 0.9 gallon (3500 cm³) batches of process water with a 10% slurry of Chemical Lime -14 Hi · Calcium Quicklime (90% average available CaO) to target pH values ranging from 5.0 to 8.0. The produced sludge samples that were neutralized to a pH value of 7.0 to 7.9 are included in the single-stage results. The 10% lime slurry (slaked lime) and process water were mixed in 4.2-inch diameter by 18-inch high plexiglas containers for 30 minutes using an impeller type mixer revolving at 300 rpm. At the end of the mixing period, the treated water was allowed to clarify for 24 hours prior to recording the volume of settled sludge and volume of clarified treated water, and removing a sample of clarified treated process water for chemical analysis. The final water content and solids content of the settled sludge were then determined.

Treated effluent from the MPI Plant with a pH between 5.9 and 7.2 developed a viscous gel within the clarified water attributed to post-precipitation syndrome from continuing reactions after mixing was stopped. The gel was not present after 24 hours, but was present 72 hours after mixing was stopped. The gel was removed from the effluent water by filtering through a 0.45 micron filter prior to taking effluent samples for chemical analyses.

SLUDGE PRODUCTION RESULTS

The results of the sludge productions from the Stage I, Stage II and single-stage lime treatment experiments are summarized in Tables 2 through 5 and in Figures 16 through 22. Included with data from this study are results previously reported in a "Feasibility Study to Screen Emergency Treatment Alternatives for Mulberry and Piney Point Phosphates Process Waters", by Fuleihan and Ingra, (2001) [identified as FIPR/FDEP (2001)] and in an experiment performed by Jacobs Engineering, Inc. for Ardaman & Associates, Inc. in May 1990 [identified as Jacobs (1990)].

The measured masses of sludge generated in the laboratory are generally consistent with backfigured industry average values. The laboratory experiments, however, provided useful correlations between sludge production and initial process water pH and total dissolved solids that allow estimating the expected mass of Stage I and Stage II sludge or single-stage sludge produced once the pH and total dissolved solids concentration of the process water are known with the total dissolved solids concentration being the more critical indicator.

The Stage I sludge production quantity as a function of process water pH and total dissolved solids concentration is summarized in Figures 16 and 17, respectively. Results indicate that the typical range of Stage I sludge production is about 80 to 400 lbs per 1000 gallons of process or pond water (Figures 16 and 17, and Table 2). The PPP Plant process water had a lower initial total dissolved solids concentration than process waters from active plants and was treated to a slightly lower pH of 4.5 accounting for the significantly lower quantity of sludge production. Although the pH of the Stage I effluents are similar, the

conductivity, total dissolved solids and chemistry of the effluent vary accounting for the wide range in sludge quantity produced during Stage II treatment.



Figure 16. Sludge Mass Generation in Laboratory Stage I Treatment Versus Process Water pH.



Figure 17. Sludge Mass Generation in Laboratory Stage I Treatment Versus Process Water Total Dissolved Solids.

Results indicate that the Stage II sludge production is 80 to 200 lbs per 1000 gallons of process water (Figure 18 and Table 3) and increases fairly linearly on a semi logarithmic scale with increasing total dissolved solids. The Stage II sludge production does not correlate well with initial process water pH or Stage I effluent pH.

The Stage II sludge settles to lower solids contents than the Stage I sludge (i.e., the average settled solids content of Stage II sludge equaled about 6% whereas the Stage I sludge settled solids content equaled about 20%). Therefore, Stage II sludge will occupy a much larger volume than the Stage I sludge (when comparing equal masses of sludge).

The sludge generation from Stage I and Stage II treatments combined ranged from 100 to 600 lbs per 1000 gallons of process water (Figures 19 and 20) depending on the process water pH and total dissolved solids concentration.

The typical range of sludge generation from single-stage treatment to pH 7 varies from 100 to 500 lbs per1000 gallons of process water (Figures 21 and 22 and Table 4) which is generally in agreement with the trend observed in the Stage I treatment experiments. As expected, the single-stage treatment experiment generated more sludge than the Stage I treatment since the target pH was 7 rather than 5.

Pond Water				Tonnat	CaO Addition			Stage	1 Efflue	nt (After 24-hr Se	Settled Sludge						
Sample No.	pН	Conductivity (µmhos/cm)	TDS (mg/l)	Vol. (gal)	pH	Time (min)	V _{CaO} (gal/gal)	M _{CaO} (lb/gal)	Sample No.	pН	Conductivity (µmhos/cm)	V _{effluent} (gal/gal)	V _{sludge} (gal/gal)	w _{c,110} (%)	w _{c, 40} (%)	S _{f, 40} (%)	M _{sludge} (lb/gal)
PW-B1	1.9	28,000	41,500	4.73	5.0	275	0.193	0.144	B1-L1	5.00	(20,000)	0.90	0.12	260	226	30.7	0.373
PW-D1	1.8	23,250	32,900	4.89	5.0	198	0.120	0.090	D1-L1	4.87	8,460	0.87	0.14	455	(427)	19.0	0.259
PW-M1	2.4	22,100	36,900	0.92	5.0	30	0.121	0.101	M1-L1	4.74	13,800	0.86	0.16	-	570	14.9	0.217
PW-N1	2.1	17,700	20,000	4.88 4.95	5.0 5.0	50 100	0.047 0.044	0.035 0.033	N1-L1a N1-L1b	4.90 4.65	10,700 12,600	0.95 0.96	0.08 0.07	885 725	800 (686)	11.1 12.7	0.083 0.082
PW-C1	2.7	11,400	19,300	4.85 4.81	5.0 5.0	155 95	0.025 0.028	0.018 0.021	C1-L1a C1-L1b	4.86 4.85	10,800 10,200	0.97 0.97	0.04 0.04	365 400	330 362	23.3 21.6	0.086 0.074
PW-P1	3.1	13,800	16,800	0.92	6.0	30	0.022	0.020	P1-L1	5.6	11,200	0.93	0.09	-	1270	7.3	0.060
PW-P2	2.9	9,500	9,600	4.92 4.94 4.98 4.95	4.5 4.5 4.5 4.5	69 83 84 88	0.006 0.007 0.006 0.006	0.005 0.006 0.005 0.005	P2-L1a P2-L1b P2-L1c P2-L1d	4.41 4.43 4.16 4.17	8,800 8,770 8,730 8,660	0.99 0.99 0.99 0.99	0.01 0.01 0.01 0.01	1445 1160 1115 1385	(1368) (1100) (1054) (1312)	6.8 8.3 8.7 7.1	0.006 0.006 0.007 0.006

Table 2. Summary of Laboratory Stage I Treatment Test Results.

Where: TDS = Total dissolved solids.

 V_{CaO} = Volume of 10% lime slurry added per gallon raw pond water.

M_{CaO} = Mass of -14 Hi · Calcium quick lime added per gallon raw pond water treated (based on 90% available CaO in -14 Hi · Calcium quick lime).

V_{effluent} = Volume of clarified effluent water (excluding lime slurry water) per gallon raw pond water treated.

 $V_{sludge} = Volume of settled sludge per gallon raw pond water treated.$

 $w_{c,110}$ = Settled sludge water content (dried at 110EC); $w_{c,40}$ = Settled sludge water content (dried at 40EC).

 $S_{f, 40}$ = Final solids content by mass based on water content (dried at 40EC).

 M_{sludge} = Dry mass of settled sludge (dried at 40EC) per gallon raw pond water treated .

 $(\mathbf{x}) =$ calculated value.



Figure 18. Sludge Generation in Laboratory Stage II Treatment Versus Stage I Effluent Total Dissolved Solids.

The volume of sludge generated in the single-stage treatment experiments of MPI and PPP process waters to various pH endpoints is presented in Table 5. As shown, treatment of MPI process water to pH of 4.7 to 8.0 resulted in the production of 150 to 380 gallons of sludge per 1000 gallons of process water. A lower to similar sludge volume of 90 to 380 gallons per 1000 gallons of process water was produced with the higher pH PPP process water. An essentially equivalent volume of sludge was produced with the higher pH PPP process water (when treated to a pH greater than 7) in spite of the fact that the mass of sludge produced was much lower. This occurred because the *in situ* dry density and solids content of the settled PPP sludge (about 9 lb/ft³ and 13%, respectively) were much lower than those of the MPI sludge (about 3 lb/ft³ and 5%, respectively).

As expected, for Stage I, Stage II and single-stage treatment, the quantity of sludge correlates to the total dissolved solids more closely than to the pH of the process water. The final settled solids contents of the sludges are very dependent on the treatment scenario and the particle size and characteristics of the sludge. The particle size distributions of the sludges are presented in the subsequent section titled "Evaluation of Sludge Engineering Properties."

Stage I Effluent					T	CaO Addition			Stage	II Efflue	ent (After 24-hr S	Stage II Settled Sludge					
Sample	лU	Conductivity	TDS	Volume	n	Time	V_{CaO}	M _{CaO}	Sample	ոՍ	Conductivity	V _{effluent}	V _{sludge}	W _{c,110}	W _{c,40}	$S_{f, 40}$	M _{sludge}
No.	рн	(µmhos/cm)	(mg/l)	(gal) *	pn	(min)	(gal/gal)	(lb/gal)	No.	pm	(µmhos/cm)	(gal/gal)	(gal/gal)	(%)	(%)	(%)	(lb/gal)
B1-L1	5.00	(20,000)	(25,000)	4.73 / 4.69	11.0	88	0.073	0.055	B1-L2	10.56	(10,000)	0.55	0.36	1684	1510	6.2	0.193
D1-L1	4.87	8,460	15,100	4.89 / 4.28	11.0	56	0.052	0.039	D1-L2	11.54	4,780	0.68	0.19	1350	(1300)	7.1	0.122
N1-L1a	4.90	10,700	(15,500)	4.89 / 4.15	11.0	40	0.065	0.049	N1-L2a	10.77	-	0.67	0.26	1480	1361	6.8	0.150
N1-L1b	4.65	12,600	19,700	4.95 / 4.35	11.0	75	0.054	0.040	N1-L2b	10.74	7,250	0.71	0.23	1224	(1157)	8.0	0.160
C1-L1a	4.86	10,800	11,100	4.85 / 4.19	11.0	145	0.035	0.026	C1-L2a	11.20	[10,200]нн	0.81	0.16	1529	1305	7.1	0.100
C1-L1b	4.85	10,200	12,800	4.82 / 4.28	11.0	45	0.039	0.029	C1-L2b	10.72	[7,920] НН	0.75	0.22	1931	1670	5.7	0.108
P2-L1b	4.43	8,770		4.94 / 4.87	7.0	135	0.023	0.017	P2-L2a	7.34	8,170	0.97	0.03	341	(320)	23.8	0.066
P2-L1c	4.16	8,730	8,950	4.98 / 4.77	9.0	234	0.041	0.031	P2-L2b	9.04	7,590	0.76	0.24	2410	(2310)	4.1	0.084
P2-L1d	4.17	8,660		4.95 / 4.82	11.0	210	0.062	0.047	P2-L2c	11.02	6,310	0.77	0.23	2017	(1915)	5.0	0.093
		6,960		- / 4.96 **	7.0	76	0.024	0.018		7.04	5,440	0.96	0.05	435	(340)	22.7	0.087
С-F1н 4.80	6,950	0 000	- / 4.92 **	7.0	74	0.024	0.018	Ш 2	7.06	5,480	0.96	0.05	388	(318)	23.9	0.088	
	4.80	6,930 8,8	0,000	- / 4.90 **	7.0	192	0.029	0.021	11-3	7.03	5,300	0.98	0.03	226	(220)	31.2	0.083
	6,960		- / 4.94 **	7.0	194	0.028	0.021		7.06	5,300	0.98	0.03	232	(215)	31.8	0.086	

Table 3. Summary of Laboratory Stage II Treatment Test Results.

Where: V_{CaO} = Volume of 10% lime slurry added per gallon raw pond water.

 M_{CaO} = Mass of -14 Hi Calcium quick lime added per gallon raw pond water treated (based on 90% available CaO in -14 Hi Calcium quick lime).

V_{effluent} = Volume of clarified effluent water (excluding lime slurry water) per gallon raw pond water treated.

 V_{sludge} = Volume of settled sludge per gallon raw pond water treated.

 $w_{c,110}$ = Settled sludge water content (dried at 110EC); $w_{c,40}$ = Settled sludge water content (dried at 40EC).

 $S_{f,40}$ = Final solids content by mass based on water content (dried at 40EC).

M_{shdge} = Dry mass of settled sludge (dried at 40EC) per gallon raw pond water treated.

*Initial raw pond water volume / Initial Stage I effluent volume treated in second stage.

**Volume of Stage I field sample used in second stage laboratory treatment test.

H Field sample from Stage 1treatment Pond (Plant C).

HH Conductivity measured after acidulation to pH7.

(x) Calculated value.



Figure 19. Sludge Generation for Laboratory Stage I and Stage II Treatment Combined Versus Process Water pH.



Figure 20. Sludge Generation for Laboratory Stage I and Stage II Treatment Combined Versus Process Water Total Dissolved Solids.


Figure 21. Sludge Generation in Laboratory Single-Stage Treatment Versus Process Water pH.



Figure 22. Sludge Generation in Laboratory Single-Stage Treatment Versus Process Water Total Dissolved Solids.

		Pond Water	r		Transf		CaO Addit	ion	Effl	uent (Af	ter 24-hr Settli	ng)		Sett	led Sludge		
Sample	ъЦ	Conductivity	TDS	Volume	1 arget	Time	V _{CaO}	M _{CaO}	Sample	лIJ	Conductivity	V _{effluent}	V _{sludge}	W _{c,110}	W _{c,40}	$S_{f,40}$	M _{sludge}
No.	pm	(µmhos/cm)	(mg/l)	(gal)	pm	(min)	(gal/gal)	(lb/gal)	No.	pm	(µmhos/cm)	(gal/gal)	(gal/gal)	(%)	(%)	(%)	(lb/gal)
DW/D1	1.0	28,000	41 500	4.91	7.0	157	0.230	0.173	B1-LSSa	7.01	8,820	0.83	0.19	332	(300)	25.0	0.466
PW-DI	1.9	28,000	41,300	3.87	7.0	89	0.239	0.179	B1-LSSb	7.15	9,070	0.79	0.22	416	(375)	21.0	0.452
				4.94	7.0	136	0.281	0.211		7.09	9,190	0.76	0.25	508	(477)	17.3	0.410
DW DO	1.0	26 100	28.000	4.57	7.0	131	0.280	0.210	IE 4	7.13	9,250	0.78	0.25	480	(447)	18.3	0.413
PW-B2	1.8	20,100	38,000	4.91	7.0	202	0.250	0.188	11-4	7.12	9,880	0.74	0.28	539	(505)	16.5	0.422
				4.91	7.0	117	0.258	0.194		7.12	9,860	0.73	0.29	578	(540)	15.6	0.420
DW/M1	2.4	22,100	20,800	0.02	7.0	30	0.181	0.152	M1-LSSa	7.15	12,500	0.72	0.28	-	625	13.8	0.3510.
PW-MI	2.4	22,100	39,800	0.92	7.5	30	0.197	0.165	M1-LSSb	7.41	12,300	0.71	0.30	-	670	13.0	358
				4.93	7.0	140	0.088	0.066	N2-LSSa	7.06	7,320	0.74	0.27	1354	(1260)	7.4	0.170
PW-N2	2.1	11,170	17,800	4.91	7.0	207	0.104	0.078	N2-LSSb	6.99	7,510	0.74	0.27	1289	(1215)	7.6	0.175
				4.90	7.0	212	0.104	0.078	N2-LSSc	6.99	7,490	0.74	0.26	1296	(1215)	7.6	0.174
PW-C2	2.7	8,670	15,100	4.89	7.0	192	0.054	0.040	C2-LSS	7.11	5,410	0.95	0.06	321	(300)	25.0	0.139
PW-P1	3.1	13,800	16,800	0.92	7.0	30	0.040	0.033	P1-LSS	7.00	10,480	0.76	0.32	2560	2560	3.8	0.107
PW-P2	2.9	9,500	9,600	4.94	7.0	113	0.034	0.026	P2-LSS	7.22	8,390	0.91	0.10	1178	(1115)	8.2	0.070
W	here: V	$V_{CaO} = Volume of$	of 10% lime	e slurry adde	d per gallo	on raw p	ond water.										
	M_{CaO} = Mass of -14 Hi Calcium quick lime added per gallon raw pond water treated (based on 90% available CaO in -14 Hi Calcium quick lime).																
		$V_{effluent} = V_{0}$	olume of cla	arified efflue	ent water (excludin	g lime slur	rv water) n	er gallon rav	v pond y	water treated.			-			

Table 4. Summary of Single-Stage Laboratory Treatment Test Results.

) per ga

 $V_{\text{studge}} = \text{Volume of settled sludge per gallon raw pond water treated.}$

 $w_{c,110}$ = Settled sludge water content (dried at 110EC); $w_{c,40}$ = Settled sludge water content (dried at 40EC).

 $S_{f, 40}$ = Final solids content by mass based on water content (dried at 40EC).

 $M_{sludge} = Dry$ mass of settled sludge (dried at 40EC) per gallon raw pond water treated.

(x) denotes calculated value.

	pН		Ca	O Utilization		N/	Settled SI	ludge at Ver	y Low Co	nsolidation		
Target	End of Mixing (30 minutes)	At 24 hours	V _{CaO} (gal/gal)	M _{CaO} (lb/gal)	M _{CaO} * (lb/gal)	v _{effluent} (gal/gal)	V _{sludge} (gal/gal)	M _{sludge} (lb/gal)	S _f (%)	$\gamma_{\rm d}$ (lb/ft ³)		
			MPI Proce	ess Water Trea	tment							
5.0	4.8	4.7	0.12	0.11	0.10	0.98	0.15	0.22	14.9	10.3		
5.5	5.9	5.9	0.14	0.13	0.12	0.97	0.22	В	В	В		
6.0	6.2	6.2	0.15	0.14	0.13	0.95	0.22	В	В	В		
6.5	6.7	6.7	0.17	0.16	0.14	0.94	0.24	0.36	15.8	11.0		
7.0	7.2	7.2	0.18	0.17	0.15	0.89	0.28	0.35	13.8	9.5		
7.5	7.4	7.4	0.20	0.19	0.18	0.90	0.30	0.36	13.0	8.9		
8.0	8.0	8.0	0.21	0.20	0.18	0.84	0.38	0.39	11.4	7.6		
	PPP Process Water Treatment											
6.0	5.8	5.6	0.022	0.020	0.018	0.93	0.09	0.06	7.3	4.8		
6.5	6.5	6.4	0.037	0.032	0.029	0.95	0.12	0.10	5.7	3.7		
7.0	7.2	7.0	0.040	0.037	0.033	0.80	0.32	0.11	3.8	2.4		
7.5	7.9	7.9	0.042	0.039	0.035	0.65	0.39	0.12	3.6	2.3		
8.0	8.3	8.1	0.044	0.041	0.037	0.67	0.38	0.12	3.8	2.4		
Where: $V_{CaO} = Volume of 10\%$ lime slurry added per gallon raw pond water; $M_{CaO} = Mass of -14 Hi$ ·Calcium quick lime added per gallon raw pond water treated (based on 90% available CaO in -14 Hi·Calcium quick lime) $M_{CaO}^* = Mass of CaO added per gallon raw pond water treated (based on 90% available CaO in -14 Hi·Calcium quick lime) V_{effluent} = Volume of clarified treated water;V_{sludge} = Volume of settled sludge;M_{sludge} = Mass of settled sludge;S_f = Final sludge solids content; and\gamma_d = Sludge dry density.$												

Table 5. Summary of Lab Single-Stage Treatment Experiments for MPI and PPP Process Water.

CaO UTILIZATION RESULTS

The laboratory experiments also yielded useful correlations between CaO utilization (i.e., lime requirements) and initial process water pH and total dissolved solids concentrations. These correlations allow estimating CaO utilization rates from the initial process water pH and total dissolved solids concentration. The experiments show that the rates of CaO utilization can vary significantly from 20 to 200 lbs/1000 gallons of process water during Stage I treatment (Figures 23 and 24, and Table 2), and from 40 to 250 lbs/1000 gallons of process water for both Stage I and Stage II treatments combined (Figures 25 and 26), depending on the process water pH and total dissolved solids (TDS) concentration. As expected, the CaO utilization correlations with TDS are more reliable than the correlations with pH. The rate of CaO utilization ranges from 20 to 200 lbs/1000 gallons of process water during single-stage treatment to pH 7 (Figures 27 and 28, and Table 4).

Note that the field CaO utilization requirements in actual applications are expected to be higher than derived from the laboratory experiments because mixing in the field is not expected to be as effective as the almost perfect mixing in a laboratory environment, and, hence, some unreacted lime will likely be present in the field treated sludges. The unreacted lime measured in six field sludge samples varied from 0.7-22% (as noted in subsequent section titled "Evaluation of Sludge Engineering Properties," Table 28).

The mass of CaO utilization in the single-stage treatment experiments for MPI and PPP process water is presented on Figure 29 and Table 5. For these experiments, a range of target pH values from 4.8 to 8.0 was selected. As shown, the MPI process water required from 100 to 180 lbs of CaO per 1000 gallons of process water to achieve pH values of 4.7 to 8.0. Much lower quantities of about 20 to 40 lbs of CaO per 1000 gallons of process water water required to treat the PPP process water. This variation is attributed to the lower total dissolved solids and higher pH of the PPP process water.



Figure 23. CaO Utilization for Laboratory Stage I Treatment Versus Process Water pH.



Figure 24. CaO Utilization for Laboratory Stage I Treatment Versus Process Water Total Dissolved Solids.



Figure 25. CaO Utilization in Laboratory Stage I and Stage II Treatment Combined Versus Process Water pH.



Figure 26. CaO Utilization in Laboratory Stage I and Stage II Treatment Combined Versus Process Water Total Dissolved Solids.



Figure 27. CaO Utilization for Laboratory Single-Stage Treatment Versus Process Water pH.



Figure 28. CaO Utilization for Laboratory Single-Stage Treatment Versus Process Water Total Dissolved Solids.



Figure 29. CaO Utilization for Laboratory Single-Stage Treatment Versus Treated Effluent pH for MPI and PPP Process Water.

SETTLING TEST RESULTS

Settling tests were performed on the Stage I, Stage II and single-stage sludge samples at initial solids contents ranging from 0.8-3.5%. The Stage I sludge samples settled to solids contents ranging from 10-28%, except for PPP Stage I sludge samples which settled from an initial solids content of 0.1% to a solids content of about 6-8%. Although the initial pond water pH, and total dissolved solids of Plant B and MPI were similar, the Stage I sludge samples from Plant B settled to almost twice the solids content of MPI sludge samples. The final settled dry density of Stage I sludge varied from about 7 to 21 lb/ft³. The initial settling velocities ranged from 1.7 to 5.8 cm/min. (Table 6)

The Stage II sludge samples that were treated to a pH of 11 settled from initial solids contents of 0.7-1.9% to final solids contents ranging from about 4-8% and the settled dry density varied from 2.5 to 5.0 lb/ft^3 . In comparison with the Stage I sludge, the particle sizes

of Stage II sludge are finer as evidenced by slower initial settling velocities that ranged from 0.2 to 1.3 cm/min. The Plant C, Stage II sludges that were acidified to a pH of 7 displayed final settled solids contents ranging from 18-30% (Table 7).

The single-stage sludge samples exhibited more variability in settled solids content, settled dry density, and initial settling velocities than either the Stage I or Stage II sludges. The settled solids contents varied from 4-24%, the dry density varied from 3 to 17 lb/ft³ and the initial settling velocities ranged from 0.3 to 10.0 cm/min (Table 8).

The settling curves are presented in Appendix B. For the single-stage sludge, the settling is more than 95% complete within 100 minutes for most samples, while for the Stage II sludge, it took around 1000 minutes to complete 95% of the settling. The majority of the particles in the Stage II sludge are finer than the particles in the single-stage sludge and, therefore, Stage II sludge was expected to require longer settling times.

CHARACTERIZATION OF TREATED EFFLUENTS

During Stage I, Stage II and single-stage laboratory treatments, the process waters displayed substantial reductions in total dissolved solids as shown in Tables 9 through 13. The reductions in concentrations of phosphorus, fluoride, calcium, iron and manganese were significant. Minor to moderate reductions occurred in the concentrations of sodium, sulfate and ammonia nitrogen.

Supernatant samples were also obtained from Stage I and Stage II settling ponds. The chemical composition of the Stage I and Stage II field water samples are presented in Tables 14 and 15. The chemical compositions of the supernatant in Stage I, Stage II and single-stage laboratory treatment experiments are comparable to the chemical compositions of the surface water obtained from the Stage I and Stage II settling ponds.

The laboratory treatment experiments allowed a characterization of the quantity and type (i.e., particle-size and plasticity) of lime sludge, and quantity and quality of supernatant produced from Stage II lime treatment of process waters at various initial pH and total dissolved solids concentrations. Additionally, where sufficient sludge mass was generated, the particle-size distribution, specific gravity and Atterberg limits of the sludge were determined. Results are presented in the subsequent section titled "Evaluation of Sludge Engineering Properties."

	Pe	ond Water			St	age 1 Effluent		Column	Initial	Sample		Final Set	tled Slud	ge	V _{si}
Sample No.	pН	Conductivity (µmhos/cm)	TDS (mg/l)	Sample No.	pН	Conductivity (µmhos/cm)	TDS (mg/l)	Dia. (cm)	H _i (cm)	S _{i,110} (%)	H _f (cm)	S _{f,110} (%)	w _f (%)	$\gamma_{d,f}$ (lb/ft ³)	(cm/ min)
B1	1.9	28,000	38,000	B1-1	5.00	-	(25,700)	15.25	118.2	2.8	11.7	27.8	260	21.3	1.7
D1	2.0	23,250	32,900	D1-1	4.87	8,460	15,100	15.25	114.1	2.3	14.4	18.2	455	12.9	4.6
M1	2.1	22,100	39,800	M1-1	4.74	13,800	23,000	10.70	44.6	2.0	6.1	14.8	570	10.3	-
N1	2.1	17,700	20,000	N1-1a N1-1b	4.90 4.65	12,400 12,600	19,700	15.25 15.25	106.2 107.0	0.8 0.8	8.5 7.0	10.2 12.6	885 725	6.8 8.6	2.7 3.4
C1	2.7	11,350	19,300	C1-1a C1-1b	4.86 4.85	[10,800]	11,100	15.25 15.25	103.4 103.7	0.8 0.8	3.9 4.4	21.5 20.0	365 400	15.4 11.6	5.8 -
P2	2.9	9,510	16,800	P2-1a P2-1b P2-1c P2-1d	4.41 4.43 4.16 4.17	8,800 8,770 8,730 8,660	8,950	15.25 20.32 20.32 15.25	102.9 58.2 58.5 103.3	0.1 0.1 0.1 0.1	1.2 0.6 0.6 1.2	6.5 8.0 8.2 6.7	1445 1160 1115 1385	3.4 4.4 4.9 3.8	-
Where:	iere: TDS = Total dissolved solids; H_i = Initial height of slurry; H_f = Final height of settled sludge. $S_{i,110}$ = Initial slurry solids content (using a drying temperature of 110°C); $S_{f,110}$ = Final sludge solids content (at end of the 24-hour settling period) (using a drying temperature of 110°C). w_f = Final water content (using a drying temperature of 110°C). $\gamma_{d,f}$ = Final dry density; v_{si} =Initial settling velocity. (x) denotes calculated walve and [x] denotes estimated value.														

Table 6. Summary of Settling Test Results for Sludge from Laboratory Stage I Treatment Experiments.

	Stag	ge 1 Effluent			Stag	e 2 Effluent		Column	Initial S	Sample	Stag	ge II Final	Settled S	Sludge	
Sample No.	pН	Conductivity (µmhos/cm)	TDS (mg/l)	Sample No.	pН	Conductivity (µmhos/cm)	TDS (mg/l)	Dia. (cm)	H _i (cm)	S _{i,110} (%)	H _f (cm)	S _{f, 110} (%)	w _f (%)	$\frac{\gamma_{df}}{(lb/ft^3)}$	v _{si} (cm/min)
B1-1	5.00	-	(25,700)	B1-2	10.56	-	8,700	15.25	105.1	1.9	35.3	5.6	1675	3.6	0.21
D1-1	4.87	8,460	15,100	D1-2	11.54	4,780	5,170	15.25	94.3	1.4	19.7	6.9	1350	4.5	0.87
N1-1a N1-1b	4.90 4.65	12,400 12,600	19,700	N1-2a N1-2b	10.77 10.74	7,250	-	15.25 15.25	94.3 96.0	1.8 1.8	26.7 23.2	6.3 7.6	1480 1225	3.9 5.0	0.19 0.26
C1-1a C1-1b	4.86 4.85	- [10,800]	- 11,100	C1-2a C1-2b	11.20 10.72	[10,200] [7,920]	5,170 2,910	15.25 15.25	90.7 93.1	1.1 1.2	16.3 22.1	6.1 4.9	1530 1930	4.0 3.2	1.27 0.68
P2-1b P2-1c P2-d	4.43 4.16 4.17	8,770 8,730 8,660	8,950	P2-2a P2-2b P2-2c	7.34 9.04 11.02	8,170 7,590 6,310	5,800 5,200 4,700	20.32 20.32 15.25	58.4 58.4 106.7	0.7 1.0 1.0	1.7 13.9 23.5	22.7 4.0 4.7	340 2410 2015	16.0 2.5 3.0	- - -
C-1F	4.80	6,950	8,800	IF-3	7.04 7.06 7.03 7.06	5,440 5,480 5,300 5,300	4,100	20.32 15.25 15.25 20.32	59.7 105.2 105.2 59.6	0.9 0.9 0.9 0.9	2.9 4.7 3.0 1.7	18.7 20.5 30.6 30.1	435 390 225 230	12.4 13.5 20.7 20.7	- - -
Where:	Where: TDS = Total dissolved solids; H_i = Initial height of slurry; H_f = Final height of settled sludge. $S_{i,110}$ = Initial slurry solids content (using a drying temperature of 110°C); $S_{f,110}$ = Final sludge solids content (at end of the 24-hour settling period) (using a drying temperature of 110°C). w_f = Final water content (using a drying temperature of 110°C). $\gamma_{d,f}$ = Final dry density; v_{si} =Initial settling velocity. (x) denotes calculated value and [x] denotes estimated value. w_{10}														

 Table 7. Summary of Settling Test Results for Sludge from Laboratory Stage II Treatment Experiments.

	Po	nd Water			Single	-Stage Effluent		Column	Initial	Sample		Final Sett	tled Slud	ge	
Sample No.	рН	Conductivity (µmhos/cm)	TDS (mg/l)	Sample No.	pН	Conductivity (µmhos/cm)	TDS (mg/l)	Dia. (cm)	H _i (cm)	S _{i,110} (%)	H _f (cm)	S _{f,110} (%)	w _f (%)	γ_{df} (lb/ft ³)	(cm/min)
B1	1.9	28,000	38,000	B1-S1a B1-S1b	7.01 7.15	8,820 9,070	8,130	15.25 15.25	126.4 100.2	3.5 3.5	19.4 18.0	23.1 19.4	330 415	16.9 13.9	10.05 4.51
B2	1.8	17,470	38,000	IF-4	7.12 7.12	9,880 9,860	-	20.32 15.25	72.3 129.0	3.4 3.4	15.9 29.7	15.6 14.7	540 580	10.8 10.2	-
M1	2.1	22,100	39,800	M1-S1 M1-S2	7.15 7.41	12,500 12,300	13,200	10.40 10.40	48.2 49.4	3.3 3.3	11.4 12.4	13.8 13.0	625 670	9.4 8.7	-
N2	2.1	10,900	17,800	N2-S1a N2-S1b N2-S1c	7.06 6.99 6.99	7,320 7,510 7,490	7,280	20.32 20.32 15.25	63.0 63.8 112.6	1.7 1.7 1.7	15.4 15.3 26.7	6.9 7.2 7.2	1355 1290 1295	4.5 4.9 5.0	0.29
C2	2.9	7,990	15,100	C2-S1	7.11	5,410	5,160	15.25	107.5	1.3	6.1	23.7	320	16.4	6.19
P1	3.0	12,570	16,800	P1-S1 P1-S2	7.00 7.88	10,480 10,010	9,300	10.40 10.50	45.7 42.0	1.2 1.4	13.3 15.7	4.8 3.5	2005 2720	2.5 2.5	-
Where:	Where: $TDS = Total dissolved solids; H_i = Initial height of slurry; H_f = Final height of settled sludge.$														

Table 8. Summary of Settling Test Results for Sludge from Laboratory Single-Stage Treatment Experiments.

 $S_{i,110}$ = Initial slurry solids content(using a drying temperature of 110°C);

 $S_{f,110}$ = Final sludge solids content (at end of the 24-hour settling period) (using a drying temperature of 110°C).

 w_f = Final water content (using a drying temperature of 110°C).

 $\gamma_{d,f}$ = Final dry density; v_{si} = Initial settling velocity.

Facility	Plant B	Pla	nt C	Plant D	Plant N	MPI Site	PPF	P Site				
Pond Water Sample I.D.	PW-B1	PW	′-C1	PW-D1	PW-N1	PW-M1	PW-P1	PW-P2				
Stage I Effluent Sample I.D.	B1-L1	C1-L1a	C1-L1b	D1-L1	N1-L1b	M1-L1	P1-L1	P2-L1a				
Sample Preparation Date	6/28/01	6/06/01	7/11/01	7/16/01	8/14/01	3/30/01	4/06/01	2/19/02				
Lab Parameters (Ardaman)												
Lab pH (at 24 hours)	5.0	4.9	4.8	4.9	4.9	4.7	5.6	4.4				
Conductivity (µmhos/cm)	-	-	-	8,460	10,700	13,800	11,200	8,800				
TESTING LABORATORY	TA	TA	ТА	TA	TA	TA	TA	ENCO				
Major Constituents (mg/l)												
Calcium, Ca	306	470	576	472	1,320	476	380	-				
Magnesium, Mg	253	224	224	290	225	201	195	234 [A]				
Sodium, Na	2,140	1,350	1,250	1,080	1,560	2,060	1,408	1,300				
Potassium, K	273	194	203	213	164	267	220	210				
Iron, Fe	0.05	0.08	0.06	0.07	0.03	0.05	0.03 [U]	0.04 [T]				
Manganese, Mn	3.5	6.3	6.7	6.9	4.5	5.0	1.2	2.3 [A]				
Chloride, Cl	38	52	36	47	20	23	160	120				
Fluoride, F	80	38	104	67	45 [A]	19	52	21 [A]				
Sulfate, SO ₄	4,200	2,800	2,800	2,800	4,600	4,500	6,000	4,800				
Total Phosphorus, P	3,180	2,110	2,620	1,890	2,850	4,400	950	1,480 [P]				
Nitrogen, TKN as N	745	270	260	590	725	-	800	625 [P]				
Nitrogen, Ammonia as N	640	255	188	510	645 [A]	1,220	720	620 [P]				
Ammonium Ion, NH ₄	0.41	0.10	0.12	0.33	0.40	0.49	0.29	-				
Other Parameters												
Lab pH	5.0	5.0	5.0	4.9	4.9 [A]	4.6	5.5	4.4				
Conductivity (umhos/cm)	-	10.800	10.200	-	11.550 [A]	-	-	8.100				
Solids. Total Dissolved	25.000 (E)	11.100	12.800	15,100	19.700	23.000	12.800	8,950				
Acidity as CaCO ₃	4,750	3,970	4,570	5,140	4,910	7,870	2,950	-				
Alkalinity as CaCO3 85 78 80 47 126 153 125 2 [U]												
NOTES : TA = Test America, Inc.; ENCO = ENCO Laboratories, Inc.												
Reported value is the ave	Reported value is the average of two or more determinations											
[P] Reported value deter	rmined by Pembro	ke Laboratories,	Inc.; [T] Reporte	d value determine	ned by Thornton	Laboratories, Ir	IC.					

 Table 9. Chemical Composition of Effluents from Laboratory Stage I Treatment Experiments.

[U] Material was analyzed for but not detected (the reported value is the minimum detection limit) (E) Estimated value

Facility Plant B Plant C Plant D Plant N PPP Site Stage I Effluent Sample I.D. B1-L1 C1-L1a C1-L1b D1-L1 N1-L1b P2-L1a P2-L1a P-F1										
Stage I Effluent Sample I.D.	B1-L1	C1-	Lla	C1-L1b	D1-L1	N1-L1b	P2-L1a	P2-L1a	P-F1	
Stage II Effluent Sample I.D.	B1-L2	C1-	L2a	C1-L2b	D1-L2	N1-L2b	P2-L2	P2-L2'	IF-3	
Sample Preparation Date	7/30/01	6/08	3/01	7/11/01	7/18/01	8/16/01	2/18/02	2/18/02	6/5/02	
Lab Parameters (Ardaman)										
Lab pH	7.2 н	7.1	Н	7.0 н	7.0 н	7.2 н	6.6 н	7.3	6.8	
Conductivity (µmhos/cm)	-	-	-	-	6,790	9,800	7,210	8.170	9,600	
TESTING LABORATORY	TA	TA	TA *	TA	TA	TA-Lab	ENCO	ENCO	P-L	
Major Constituents (mg/l)										
Calcium, Ca	49	113	12.6	4.1	98	-	430	140	-	
Magnesium, Mg	0.9	2.1	2.5	2.0	0.5 [U]	-	7.5	84	-	
Sodium, Na	1,890	1,101	1,340	1,280	950	-	1,300	1,300	957	
Potassium, K	235	170	167	200	170	-	200	200	-	
Iron, Fe	0.03	0.33	0.34	0.03	0.1 [U]	-	0.15	0.32 [A]	-	
Manganese, Mn	0.01 [U]	0.02	0.23	0.01 [U]	0.05 [U]	-	0.01 [U]	0.24 [A]	-	
Chloride, Cl	28 (e)	48	48	38	76	-	48	110	180	
Fluoride, F	2.0	6.5	6.5	18.6	3.3	2.0	1.1 [A]	17 [A]	5.7	
Sulfate, SO ₄	5,500 (?)	3,000	3,000	3,200	3,600	5,410 (?)	4,300	4,400	6,156	
Total Phosphorus, P	2.3	114	114	95	0.8	8.0	1.3	39	54	
Nitrogen, TKN as N	<520>	<180>	<190>	<195>	<354>	-	<<270>>>	480	647	
Nitrogen, Ammonia as N	<430>	<160>	<140>	<184>	<330>	<470>	<<270>>>	480	525	
Ammonium Ion, NH ₄	<5.48>	<0.59>	<0.52>	<1.86>	<2.11>	-	-	-	-	
Other Parameters										
Lab pH	7.3	7.1	7.1	7.2	7.0	7.1	6.7	7.4	6.7	
Conductivity (umbos/cm)	16.100	7.920	9.720 (?)	10.200	7.470	16.300	6.300	6.900	7.800	
Solids. Total Dissolved	8,700	2,910	4.510	5,170	5,170	-	4,700	5,800	4,100	
Acidity as CaCO ₃	131	158	188	14	7	-	-	-	-	
Alkalinity as $CaCO_3$	53	164	168	114	48	-	18	150	-	
NOTES : TA = Test America, Inc.;	PL = Pembroke	e Laboratories,	Inc.; ENCO =	ENCO Laboratorio	es, Inc.	•	•	•		
[A] Reported value is	the average of	two or more de	eterminations:	* Duplicate sampl	e: (?) Indicates	questionable re	ported value			

Table 10. Chemical Composition of Effluents from Laboratory Stage II Treatment Experiments.

Material was analyzed for but not detected. (The reported value is the minimum detection limit); [U]

Measured after addition of 1N H₂SO₄ as needed to adjust from pH 11 to pH 7 Η

No attempt made at air-stripping ammonia at Stage II pH 11. (Laboratory results not representative of field aerated Stage II+ treated effluents); <>

Results not representative of field aerated Stage II+ effluent due to poor laboratory simulation of air-stripping at pH 11 << >>

	r		1									
Facility Plant B Plant C Plant N MPI Site PPP Site Pond Water Sample I.D. PW-B2 PW-C2 PW-N2 PW-M1 PW-P1 PW-P2 PW-P3												
Pond Water Sample I.D.	PW-B2	PW-C2	PW-N2	PW-M1	PW-P1	PW-P2	PW-P3					
Single-Stage Effluent Sample I.D.	B2-LSS*	C2-LSS	N2-LSSa	M1-LSS	P1-LSS	P2-LSS	P3-LSS / IF-4					
Sample Preparation Date	1/22/02	2/06/02	2/07/02	3/30/01	4/06/01	2/18/02	5/23/02					
Lab Parameters (Ardaman)												
Lab pH (at 24 hours)	7.0	7.1	7.1	7.2	7.0	7.2	7.2					
Conductivity (µmhos/cm)	8,820	5,410	7,320	12,500	10,480	8390	9,290					
TESTING LABORATORY	PL	PL	PL	TA	TA	ENCO	PL					
Major Constituents (mg/l)												
Calcium, Ca	5.2	0.5	2.0	21	142	93	-					
Magnesium, Mg	74	130	75	28	89	154 [A]	-					
Sodium, Na	5,97	849	791	2,080	1,448	1,300	861					
Potassium, K	285	179	151	263	213	200	-					
Iron, Fe	0.1 [U]	0.1 [U]	0.1 [U]	0.03 [U]	0.03 [U]	0.03	-					
Manganese, Mn	0.01 [U]	0.5	0.09	0.1	0.04	0.04	-					
Chloride, Cl	400 [A]	55	50	44	140	120	150					
Fluoride, F	67	29	57	113	27	30 [A]	19.5					
Sulfate, SO ₄	3,560	2,050	3,612	4,200	5,850	3,900	5,016					
Total Phosphorus, P	1,020	159	202	1,350	230	280 [P]	139					
Nitrogen, TKN as N	619	244	613	-	800	600 [P]	632					
Nitrogen, Ammonia as N	506	186	470	1,060	690	550 [P]	500					
Ammonium Ion, NH ₄	-	-	-	3.92	2.55	-	-					
Other Parameters												
Lab pH	6.8	6.7	6.3	7.2	7.0	7.2	-					
Conductivity (µmhos/cm)	11,600	6,530	9,930	17,900	18,200	7,900	-					
Solids, Total Dissolved	8,130	5,160	7,280	13,200	9,300	5,700	7,775					
Acidity as CaCO ₃	500	700	380	1,540	600	-	-					
Alkalinity as CaCO ₃ 614 478 320 1,220 329 290 -												
NOTES : TA = Test America, Inc.; PL = Pembroke Laboratories, Inc.; ENCO = ENCO Laboratories, Inc.												
[A] Reported value is the a	average of two or	more determin	ations.									
[P] Reported value determined by Pembroke Laboratories, Inc.												
[U] Material was analyzed for but not detected. (The reported value is the minimum detection limit);												
 Composite sample from 	m duplicate trials	(B2-LSSa and	B2-LSSb).									

 Table 11. Chemical Composition of Effluents from Laboratory Single-Stage Treatment Experiments.

	Untreated		Treated W	/ater pH at	24 Hours	
Parameter	Process Water	5.6	6.4	7.0	7.9	8.1
Lab pH	2.8	5.5	6.4	7.0	7.6	8.1
Conductivity (µmhos/cm)	13,800	11,200	10,750	10,480	10,010	10,320
Turbidity (NTU), 24 hours	В	2.5	1.1	1.9	2.0	3.8
Lab Turbidity (NTU)	6.5	1.7	0.7	0.8	1.4	0.6
Color (Pt/Co units)	50	40	40	45	40	45
Calcium, Ca (mg/l)	602	380	200	142	54	65
Magnesium, Mg (mg/l)	242	195	129	89	33	50
Sodium, Na (mg/l)	1556	1408	1464	1448	В	1410
Potassium, K (mg/l)	226	220	217	213	145	208
Iron, Fe (mg/l)	5.8	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03
Manganese, Mn (mg/l)	3.4	1.2	0.2	0.04	< 0.03	< 0.03
Chloride, Cl (mg/l)	144	160	150	140	145	145
Fluoride, F (mg/l)	320	52	37	27	40	24
Sulfate, SO ₄ (mg/l)	6200	6000	5960	5850	5700	5800
Total Phosphorus, P (mg/l)	1740	950	410	230	В	60
Ammonia, Nitrogen, N (mg/l)	780	720	700	690	670	400
Acidity, CaCO ₃ (mg/l)	В	2950	1350	600	181	108
Alkalinity, CaCO ₃ (mg/l)	В	125	290	329	В	В
Solids, Total Dissolved (mg/l)	16,800	12,800	10,400	9,300	8,600	8,700
Solids, Total Suspended (mg/l)	144	58	78	46	9	32

Table 12. Chemical Composition of Effluents from Laboratory Single-Stage Treatment of
PPP Process Water to Various pH Levels (FIPR-FDEP Study).

Table 13. Chemical Composition of Effluents from Laboratory Single-Stage Treatment of
MPI Process Water to Various pH Levels (FIPR-FDEP Study).

Parameter Process Treated Water pH at 24 Hour								
Parameter	Process Water	4.7	5.9	6.2	6.7	7.2	7.4	8.0
Lab pH	2.1	4.6	5.8	6.1	6.6	7.2	7.5	7.9
Conductivity (µmhos/cm)	22,100	13,800	13,000	12,700	12,400	12,500	12,300	12,000
Turbidity (NTU), 24 hours	В	1.3	8.9	21	17	13	5.0	1.4
Turbidity (NTU), 72 hours	В	В	32.0/1.8*	32.0/1.3*	39.1/1.2*	31.1/2.4*	В	В
Lab Turbidity (NTU)	0.9	1.5	2.0	1.5	1.2	2.5	7.5	1.5
Color (Pt/Co units)	300	130	45	120	150	120	150	150
Calcium, Ca (mg/l)	538	476	57	39	24	21	22	17
Magnesium, Mg (mg/l)	223	201	102	68	47	28	19	9
Sodium, Na (mg/l)	2260	2060	2100	2180	2160	2080	1928	1936
Potassium, K (mg/l)	210	267	281	268	258	263	270	271
Iron, Fe (mg/l)	59	0.05	< 0.03	< 0.03	< 0.03	< 0.03	0.33	0.33
Manganese, Mn (mg/l)	15	5.0	0.9	0.6	0.3	0.1	0.1	< 0.03
Chloride, Cl (mg/l)	140	23	46	51	48	44	39	39
Fluoride, F (mg/l)	4120	19	55	87	107	113	112	120
Sulfate, SO ₄ (mg/l)	6200	4500	4750	4500	4200	4200	4000	4000
Total Phosphorus, P (mg/l)	6600	4400	2240	2900	1860	1350	1320	1210
Ammonia Nitrogen, N (mg/l)	1240	1220	1010	1120	1110	1060	1000	1040
Acidity, CaCO ₃ (mg/l)	В	7870	5060	2560	2700	1540	600	320
Alkalinity, CaCO ₃ (mg/l)	В	153	524	529	984	1220	1270	1300
Solids, Total Dissolved (mg/l)	39,800	23,000	16,900	15,600	13,600	13,200	10,900	10,800
Solids, Total Suspended (mg/l)	22	6	226	86	54	222	В	58
*Turbidity at 72 hours with gel in	suspension/T	urbidity a	72 hours aft	er filtering th	rough 0.45 m	icron membra	ane filter.	

Facility	Plant B*	Plan	t C	Plant D	Plant N*	PPP Site	Plant A						
Sampling Location	Stage 1 Pond	Stage 1 Pond	Stage 1 Pond	North Pond	Pond I	Clarifier	Underflow						
Sample I.D.	B-F1	C-F1a	C-F1b	D-F1	N-F1	P-F1	A-1U						
Sampling Date	4/19/01	4/19/01	2/21/02	4/27/01	4/17/01	11/12/01	7/10/01						
Lab Parameters (Ardaman)													
Lab pH	4.2	4.5 [A]	4.8 [A]	2.9	4.7 [A]	5 \	4.2						
Conductivity (µmhos/cm)	6,150	10,000 [A]	6,950 [A]	9,810	14,350 [A]	(e)	9,820						
TESTING LABORATORY	TA	TA	ENCO	TA	TA	FDEP	ТА						
Major Constituents (mg/l)	ĺ												
Calcium, Ca	285	1,160	1,000	384	727	-	1,074						
Magnesium, Mg	89	201	240	151	224	270	630						
Sodium, Na	756	1,210	1,300	1,470	1,960	1,100	2,156						
Potassium, K	67	187	220	48	176	190	466						
Iron, Fe	0.35	0.15	0.56	2.9	1.0 (E)	2.1	0.23						
Manganese, Mn	anganese, Mn 1.6 7.5 5.6 3.2 3.1 2.4 6.1												
	$\begin{array}{c c c c c c c c c c c c c c c c c c c $												
Chloride, Cl	aloride, Cl 124 46 46 68 [M] 36 100 46												
Fluoride, F	Ioride, C1124464668 [M]3610046Ioride, F453149160483913												
Sulfate, SO ₄	2,500	2,920	2,300	3,750	5,670	4,600	3,700						
Total Phosphorus, P	470	3,250	1,800	1,320	2,220	1,200	-						
Nitrogen, TKN as N	270	280	220	220	760	610	354						
Nitrogen, Ammonia as N	285	240	210	230	690	600	138						
Ammonium Ion, NH ₄	0.11	0.10	-	0.09	0.28	-	0.09						
Other Parameters													
Lab pH	4.1	4.5	4.9	3.0	4.7	-	4.0						
Conductivity (µmhos/cm)	6,930	14,100	6,600	18,000 (?)	-	-	17,900 (?)						
Solids, Total Dissolved	5,930	17,500	8,800	10,900	19,200	10,000	17,900						
Acidity as CaCO ₃	1,330	7,970	3,800	3,760	5,960	-	7,440						
Alkalinity as CaCO ₃ 1.0 [U] 1.0 [U] 53 1.0 [U] 34 - 1.0 [U]													
NOTES : [TA = Test America, Inc.; PL = Pembroke Laboratories, Inc.; ENCO = ENCO Laboratories, Inc.; FDEP = Florida Department of Environmental Protection.													
NOTES : [A] Reported value is the a	average of two or more	determinations.											
[M] Matrix interference p	revented accurate deterr	mination.											
[U]Material was analyzed for but not detected. (The reported value is the minimum detection limit);													
(E) Estimated value; (?) Ir	(E) Estimated value; (?) Indicates questionable reported value.												
 * Inactive treatment faci 	lity; Surface water sam	ple not necessarily rep	presentative of Stag	ge I treated effluent.									

Table 14. Chemical Composition of Surface Water Samples from Stage I Settling Ponds.

Parameter	Parameter Plant B* Plant C Plant D Plant N* PPP Site Plant A Sampling Location Stage 2 Pond Pond 4A* Pond 4B* Outfall H South Pond Pond II Aeration Pond Underflow													
Sampling Location	Stage 2 Pond	Pond 4A*	Pond 4B*	Outfall H	South Pond	Pond II	Aeration Pond	Underflow						
Sample I.D.	B-F2	C-F2a	C-F2b	C-F2c	D-F2	N-F2	IF-2d0	A-2U						
Sampling Date	04/19/01	04/19/01	04/19/01	2/21/02	04/27/01	04/17/01	5/09/02	07/10/01						
Lab Parameters (Ardaman)														
Lab pH	5.8	10.4 [A]	9.1 [A]	6.9 [A]	5.3	11.0 [A]	9.7	12.3						
Conductivity (µmhos/cm)	3,900	6,260 [A]	6,030 [A]	4,515 [A]	5,980	7,660 [A]	6,240	7,820						
TESTING LABORATORY	TA	TA	TA	ENCO	TA	TA	ENCO/ECT	TA						
Major Constituents (mg/l)														
Calcium, Ca	369	6.6	4.3	11	160	5.9	550	22						
Magnesium, Mg	90	1.0	3.1	4.1	177	1.9	9.1	0.1						
Sodium, Na	438	1,080	1,010	1,200	879	1,740	1,300	1,764						
Potassium, K	54	168	157	180	34	149	200	400						
Iron, Fe	0.13	0.03	0.03 [U]	0.16	1.0	0.04	0.15	0.05						
Manganese, Mn 1.4 0.03 [U] 0.03 [U] 0.01 [U] 0.10 1.0 0.04 0.13 0.03 0.03 [U] 0.03 [U] 0.03 [U] 0.01 [U] 0.1 0.03 [U] 0.01 [U] 0.04 [U]														
Manganese, Min 1.4 $0.03 [U]$ $0.01 [U]$ 0.1 $0.03 [U]$ $0.01 [U]$ $0.004 [U]$														
Chloride, Cl 143 49 53 48 73 28.7 120 45														
Fluoride, F	Chloride, Cl 143 49 53 48 73 28.7 120 45 Fluoride, F 4.6 [U] 1.0 0.5 9.6 2.6 0.8 1.3 11.6													
Sulfate, SO ₄	1,880	2,290	2,850	1,900	2,750	2,920	4,300	2,500						
Total Phosphorus, P	170	180	10.7 [P]	4.3	150	160	1.3	10						
Nitrogen, TKN as N	40	110	13 [P]	11	41	70	5.6	266						
Nitrogen, Ammonia as N	40	40	11	10	40	3.0	3.3	199						
Ammonium Ion, NH ₄	0.02	-	3.08	-	0.05	2.4	-	-						
Other Parameters														
pH	4.6	10.4	9.0	6.8	6.6	10.7	1.0	12.0						
Conductivity (µmhos/cm)	6,600	8,660	5,910	4,300	8,000	12,800	6,100	9,430						
Solids, Total Dissolved	3,520	4,320	4,320	2,200	4,320	5,570	4,700	4,480						
Acidity as CaCO ₃	346	1.0 [U]	1.0 [U]	24	20	1.0 [U]	-	1.0 [U]						
Alkalinity as CaCO ₃ 1.0 [U] 365 110 [P] 75 148 281 18 115														
NOTES : TA = Test America, Inc.; ENCO = ENCO Laboratories, Inc.; ECT= Environmental Consulting & Technologies, Inc.														
[A] Reported value is the average of two or more determinations; [P] Reported value determined by Pembroke Laboratories, Inc.														
[U] Material was analyzed for but not detected (the reported value is the minimum detection limit);														
* Inactive treatment	nent facility; Surfac	ce water sample	not necessarily r	representative of	Stage II treated	effluent.								

Table 15. Chemical Composition of Surface Water Samples from Stage II Settling Ponds.

H Sampled post-aeration and acidulation to neutral pH.

EVALUATION OF SLUDGE ENGINEERING PROPERTIES

OVERVIEW OF LABORATORY TESTING

Stage I and Stage II lime sludges were sampled from existing lime treatment/sludge disposal facilities at five phosphogypsum stack systems (Plants A, B, C, D and N) for characterization of engineering properties relevant to conventional disposal in sedimentation ponds. Bulk samples and undisturbed tube samples from existing Stage I and Stage II settling ponds were obtained to evaluate the sludge engineering properties.

Initially, bulk samples of sludge were obtained and characterized using a series of index tests, including solids content, pH, conductivity, particle-size (ASTM D422), specific gravity (ASTM D854) and Atterberg limits (ASTM D4318). The settling behavior was characterized and the free lime content (amount of unreacted lime remaining in the sludge) was measured. Based on results of the index tests, three sites (Plants B, C and N) and the corresponding Stage I and Stage II sludges were selected to obtain undisturbed samples.

Laboratory hydraulic conductivity, consolidation and shear strength tests were performed on the undisturbed Stage I and Stage II lime sludge samples and on samples of sludges produced during the laboratory process water treatment experiments. The laboratory measurement and characterization of the consolidation and strength properties were used for evaluating engineering properties relevant to conventional disposal in and capping of sedimentation ponds.

FIELD SAMPLING AND LABORATORY PRODUCTION OF LIME SLUDGE

Bulk samples of Stage I, Stage II, and single-stage lime sludge were obtained from sedimentation ponds of four inactive treatment plants (Plants B, N, C, and D) and from one active plant (Plant A), as indicated in Table 16.

In addition to the collection of bulk samples for testing, undisturbed samples of sludge were obtained. The sampling took place from an airboat using a hand-operated fixed-piston sampler at two locations in each settling pond. The change in solids content and water content with depth in the ponds was measured. Strength, hydraulic conductivity and consolidation tests were performed on these undisturbed samples of sludge.

In addition, Stage I and Stage II lime sludges were experimentally produced in the laboratory as described in the previous section titled "Evaluation of Sludge Production." Where a sufficient mass of sludge was generated, the particle-size distribution, specific gravity and Atterberg limits of the sludge were determined.

Site	Stage	Sampling Location
Dient B	Ι	Settling Pond I
Flain B	II	Settling Pond II
Dlant N	Ι	Sludge Pond I
Flait	II	Sludge Pond II
	Ι	Stage I Pond
Plant C	II	Sludge Pond 4A [Old Sludge]
Flain C	II	Sludge Pond 4B [New Sludge]
	Single-Stage	Sludge Pond 5A
Plant D	II	South Sludge Pond
Dlont A	Ι	Clarifier Underflow
Fiant A	II	Clarifier Underflow

 Table 16.
 Sludge Sampling Locations.

pH and Conductivity

The pH and conductivity of each lime sludge sample were measured using a 2:1 dilution ratio of de-ionized water to air-dried solids. Tables 17 and 18 present the pH and conductivity data for field sludge samples and sludge generated in laboratory treatment experiments, respectively.

The pH of the sludge ranged from 4.2 to 12.3 and the surface water pH ranged from 4.2 to 11 for the field sampling. From the laboratory treatment studies, the pH from the generated sludge samples ranged from 5.9 to 9.1 and treated effluent water pH ranged from 4.8 to 11. The field and laboratory samples encompassed a wide range of pH, and typically the pH of the sludge was similar to the pH of the corresponding surface water or treated effluent, even though the sludge tends to be closer to neutral than the surface water or treated effluent.

	Comple	Depth	Data	(Sludge*	Sur	face Water	
Facility	No.	Range (ft)	Sampled	pН	Conductivity (µmhos/cm)	pН	Conductivity (µmhos/cm)	
Stage I Sludg	ge							
PLANT B	1A	0 -3	5/07/01	8.8	3,440	4.2	6,150	
DI ANT N	1A	0 -3	5/03/01	5.9	5,590	17	14 250	
FLANT N	1B	0 -3	5/03/01	5.7	6,290	4.7	14,350	
PLANT A	1 ^H	n/a	7/20/01	4.2	9,820	n/a	n/a	
Stage II Sludge								
PLANT C	2A	3 -6	4/24/01	8.3	9,780	10.4	6 260	
Pond 4A	2B	2 -5	4/24/01	8.0	8,030	10.4	0,200	
	2C-1	0 -3	4/25/01	7.3	4,810			
PLANT C	2C-2	6 -9	4/25/01	7.8	2,730	0.1	6 020	
Pond 4B	2D-1	0 -4	4/25/01	7.8	3,990	9.1	0,030	
	2D-2	8 -12	4/25/01	7.8	2,340			
DLANTD	2A	3 -7	5/04/01	8.7	3,400	5.0	2 000	
PLANI B	2B	3 -5	5/04/01	7.8	3,140	5.8	3,900	
DI ANT N	2A	1 -3	5/03/01	11.5	4,320	11.0	7.00	
PLANT N	2B	2 -4	5/03/01	11.5	4,860	11.0	7,000	
PLANT D	2A	1 -3	4/27/01	8.0	11,870	5.3	5,980	
PLANT A	2 ^H	n/a	7/2001	12.3	7,820	n/a	n/a	
Single-Stage	Sludge							
PLANT C	SSA	-	4/25/01	5.5	1,952			
Pond 5A	SSB	-	4/25/01	4.7	2,810	-	-	
*Pore water of	dilution usi	ng <u>a 2:1</u> ra	tio of deioniz	zed water	to air-dried solids	8.		
^H Sample from	n mechanic	al clarifier	underflow.					

 Table 17. pH and Conductivity Data for Field Sludge Samples.

	Treated Eff	luent Water	Sludge*		
Facility	pН	pH Conductivity (µmhos/cm)		Conductivity (umhos/cm)	
Stage I	I		I	(particos, erri)	
PLANT C	4.9	10,500	6.7	5,140	
PLANT N	4.8	12,600	5.9	15,050	
Stage II					
PLANT C	11.0	-	9.1	12,560	
PLANT N	10.7	7,250	7.8	14,090	
*Pore water dilut	ion using a 2:1 ration	o of de-ionized wat	ter to air-dried so	lids.	

 Table 18. pH and Conductivity Data for Sludge Generated in Laboratory Treatment

 Experiments.

Solids Content and Water Content

The solids content and water content were measured on each lime sludge sample (Stage I, Stage II and single-stage) in general accordance with ASTM D2216 using drying temperatures of 40°C, 110°C and 200°C. Results of the tests showed an apparent change in water content by drying from 40°C to 200°C (ΔAWC_{200-40}) of 6.8-12.7% for field sludge samples indicating the presence of hydrated compounds such as chukhrovite (Ca₃Al₂(R.E.)SO₄F₁₃·10H₂O where R.E. = rare earth mix with yttrium), monocalcium phosphate (Ca(H₂PO₄)₂·H₂O), dicalcium phosphate (Ca(HPO₄)·2H₂O) and hydroxy-phosphate.

The laboratory treatment experiment samples exhibited a similar apparent change in water content by drying from 40°C to $110^{\circ}C$ (ΔAWC_{110-40}) of 7.4-13.7%. Plots of the water content of the field samples versus depth of Stage I and Stage II lime sludge samples are presented in Figure 30. Tables 19 and 20 present the water content results for field sludge samples and sludge produced from laboratory treatment experiments, respectively.



Figure 30. Water Content of Stage I and Stage II Lime Sludge Samples with Depth.

		Depth				Wa	ter Content		
Facility	No.	Range (ft)	Date Sampled	AWC ₄₀ (%)	WC ₁₁₀ (%)	AWC ₁₁₀ (%)	AWC ₂₀₀ (%)	ΔAWC ₁₁₀₋₄₀ (%)	ΔAWC ₂₀₀₋₄₀ (%)
				Stag	ge I Sludge				
PLANT C	1A* 1B*	0 -6 0 -7	5/07/01 5/07/01	228.1 222.9	241.5 238.5	232.2 227.5	235.3	4.1 4.6	7.2
PLANT B	1A S1	0 -3 Surface	5/07/01 4/19/01	233.1 111.8	251.2 120.0	238.2 115.5	241.2	5.1 3.7	8.1
PLANT N	1A 1B	0 -3 0 -3	5/03/01 5/03/01	201.0	186 222.0	- 207.4	212.5	- 6.4	- 11.5
PLANT A	1 ^H	n/a	7/2001	980	1,048	981	-	1.0	-
Stage II Sludge									
PLANT C Pond 4A	2A 2B	3 -6 2 -5	4/24/01 4/24/01	826.8 693.6	859.0 723.3	830.2 697.2	-	3.4 3.6	-
PLANT C Pond 4B	2C-1 2C-2 2D-1 2D-2	0 -3 6 -9 0 -4 8 -12	4/25/01 4/25/01 4/25/01 4/25/01	422.4 331.4 375.0 351.0	441.4 345.4 391.4 369.2	425.9 334.5 378.4 355.0	358.4	3.5 3.1 3.4 4.0	7.4
PLANT B	2A 2B	3 -7 3 -5	5/04/01 5/04/01	1,375.2 566.5	1,447.4 612.3	1,380.0 573.0	- 579.2	4.8 6.5	- 12.7
PLANT N	2A 2B	1 -3 2 -4	5/03/01 5/03/01	255.4	269.2 281	259.2	262.2	3.8	6.8 -
PLANT D	2A	1 -3	4/27/01	1,480.8	1,576.7	1,486.6	-	5.8	-
PLANT A	2 ^H	n/a	7/2001	1,212.6	1,270.2	1,216.8	-	4.2	-
				Single	-Stage Slud	ge			
PLANT C Pond 5A	SSA SSB		4/25/01 4/25/01	153.6 177.8	162.2 186.2	156.9 180.7	-	3.3 2.9	-
Where AWC	$_{40} = Appare$	ent water co	ontent at 40°	C;					

Table 19. Water Content and Drying Characteristics of Field Sludge Samples.

 $WC_{110} = Water content at 110^{\circ}C;$

 AWC_{110} = Apparent water content at $110^{\circ}C$ (normalized based on mass of dry solids at $40^{\circ}C$);

 $AWC_{200} = Apparent$ water content at $200^{\circ}C$ (normalized based on mass of dry solids at $40^{\circ}C$);

 $\begin{array}{l} \Delta AWC_{100} = AWC_{110} - AWC_{40}; \\ \Delta AWC_{200-40} = AWC_{200} - AWC_{40}; \\ \end{array}$ *Pore water dilution using a 2:1 ratio of de-ionized water to air-dried solids.

^HSample from mechanical clarifier underflow.

	pU of		Water Content						
Facility	Effluent	AWC ₄₀	WC ₁₁₀	AWC ₁₁₀	ΔAWC_{110-40}				
	Emuent	(%)	(%)	(%)	(%)				
		Stage I							
DI ANT C	4.86	416.2	458.8	423.8	7.6				
FLANTC	4.85	396.5	437.1	404.1	7.6				
DI ANT N	4.90	868.8	963.1	877.7	8.9				
PLANT N	4.85	919.4	1,017.8	929.1	9.7				
PLANT B	5.00	237.2	270.1	246.1	8.9				
		Stage II							
DI ANT C	11.20	1,404.6	1,643.5	1,418.3	13.7				
FLANIC	10.72	1,701.5	1,966.6	1,714.3	12.8				
DI ANT N	10.77	1,420.9	1,544.8	1,428.4	7.5				
PLANT IN	10.70	1,857.7	2,014.0	1,865.1	7.4				
PLANT B	10.56	1,519.3	1,683.8	1,528.5	9.2				
Where $AWC_{40} =$	Apparent water	r content at 40°C	•						
$WC_{110} = Water co$	ontent at 110°C	1.							
$AWC_{110} = Appare$	ent water conte	nt at 110°C (norr	nalized based	l on mass of o	dry solids at				
40°C);									
$\Delta AWC_{110-40} = AV$	$WC_{110} - AWC_{40}$	0;							

Table 20. Water Content and Drying Characteristics of Sludge Generated in
Laboratory Treatment Experiments.

Specific Gravity

The specific gravity was measured on each lime sludge sample (Stage I, Stage II and single-stage) in general accordance with ASTM D854 using lime saturated water in the pycnometer and a drying temperature of 40°C. The measured values ranged from 2.75 to 3.05 considering both field and laboratory samples as presented in Tables 21 and 22.

Facility	Boring No.	Sample No.	Sample Depth (ft)	Specific Gravity, G					
		Stage I Sludge	- ·F ()						
PLANT C	TH-1A	US-3	6-8	3.00					
PLANT B	TH-1A	US-2	1-3	2.75					
PLANT N	TH-1B	US 2	0-2	2.86					
Stage II Sludge									
PLANT C	TH-2A	Bucket	3-6	3.03					
(Pond 4A)	TH-2B	Bucket	2-5	3.08					
PLANT C	TH-2C	US-1 Bucket 1 US-3 Bucket 2	1-3 0-3 7-9 6-9	2.93 3.01 2.97 2.95					
(Pond 4B)	TH-2D	US-2 Bucket 1 US-3 Bucket 2	3-5 0-4 6-8 8-12	3.05 2.94 2.80 2.76					
DI ANT D	TH-2A	US-2	5-7	2.86					
FLANI D	TH-2B	US-2	5-7	2.79					
PLANT N	TH-2B	US-2	2-4	2.82					
	S	Single-Stage Sludge							
PLANT C (Pond 5A)	TH-A TH-B	Bucket Bucket	-	2.98 3.04					

Table 21. Specific Gravity of Field Sludge Samples.

Table 22. Specific Gravity of Sludge Generated in Laboratory Treatment Experiments.

Facility	pH of Treated Effluent	Specific Gravity, G _s		
Stage I Sludge				
PLANT B	5.0	3.02		
PLANT C	4.9	2.75		
PLANT D	4.9	3.02		
Stage II Sludge				
PLANT B	10.6	2.89		
PLANT C	11.0	3.05		
PLANT D	11.5	2.93		

Total Unit Weight and Dry Density

The undisturbed tube lime sludge samples (Stage I and Stage II) were extruded, and the total unit weight and dry density were determined. Tables 23 and 24 present the results of density determinations and index tests on Stage I and Stage II undisturbed sludge samples, respectively.

Stage I undisturbed sludge sample dry densities ranged from 19.6 to 26.7 lb/ft³ with solids contents of 26-33%. The Stage II undisturbed sludge samples exhibited lower values with dry densities ranging from 14.0 to 24.9 lb/ft³ and solids contents of 19-32%.

Particle-Size Distribution

Particle size analyses were performed on the lime sludge samples in accordance with ASTM D422 using sieve and hydrometer methods. The hydrometer specimens were tested using surface water from a corresponding sampling location for the field samples or using lime saturated water for the laboratory generated sludge as well as for the reacted sludge.

Results of the sieve and hydrometer tests on field and laboratory treatment experiment sludge samples are summarized in Table 25. Figures 31 and 32 present the ranges of particle-size distribution for the Stage I and Stage II lime sludges, respectively, along with the typical range for phosphogypsum. Particle size distribution curves are presented in Appendix A. The percentage of particles passing the 75 mm sieve-size (i.e., fines content) for Stage I sludge ranged from 67 to 100. Results of fines content determinations from field and laboratory generated samples of Stage II sludge yielded values ranging from 38-100% passing the 75 mm sieve-size. The Stage II undisturbed samples exhibited a large variability in particle-size with few samples containing sand-sized particles or cemented fragments.

Facility	Boring No.	Sample No.	Sample Depth (ft)	WC ₁₁₀ (%)	S ₁₁₀ (%)	γ_d (lb/ft ³)	γ_t (lb/ft ³)	Gs	e	S (%)	-200 (%)
		US-1	1-3	200.5	33	26.7	80.2	(3.0)	6.02	100	75, 96, 97
PLANT C	TH-1A	US-2	3-5	199.2	33	26.4	79.1	(3.0)	6.09	98	90, 99
		US-3	6-8	205.0	33	26.1	79.6	3.00	6.68	100	86, 96, 98
PLANT B	TH-1A	US-1	1-3	233.4	30	22.9	76.5	(2.8)	6.63	99	84, 100
		US-2	1-3	280.0	26	19.6	74.6	2.75	7.76	99	85, 98, 99
		US-1	0-2	233.5	30	22.8	75.9	(2.8)	6.67	98	-
	IH-IA	US-2	0-2	216.3	31	23.2	74.6	(2.8)	6.54	95	67, 80
PLANTIN	TIL 1D	US 1	0-2	240.3	29	22.1	75.1	(2.8)	6.91	97	90, 99
	IH-IB	US 2	0-2	237.8	31	24.3	78.5	2.78	6.14	101	87, 98, 99
Where: WC	$L_{110} = $ Water con	tent (determ	nined using a	a drying tem	perature	of 110 EC	C); $S_{110} =$	Solids c	ontent (1	for a dr	ying
tem	perature of 110	EC); $\gamma_d = I$	Dry density;	$\gamma_t = \text{Total } \mathbf{u}_t$	nit weigh	it; $G_s = Sp$	ecific gra	vity (esti	imated v	alues in	1
pare	ntheses); $e = Ve$	oid ratio; S =	= Degree of	saturation;	-200 = p	ercent ma	terial by d	lry weigh	nt passin	g the U	.S. No. 200
sieve	e size (<0.075 n	nm)	-		-		-		-	-	

 Table 23. Density Determinations and Index Tests on Undisturbed Stage I Sludge Samples.

Engility	Boring	Sample	Sample	WC ₁₁₀	S ₁₁₀	γ_{d}	γ_t	G		S	-200
Facility	No.	No.	Depth (ft)	(%)	(%)	(lb/ft^3)	(lb/ft^3)	Gs	е	(%)	(%)
		US-1	1-3	357.6	22	16.0	73.2	2.73	9.65	101	24, 37, 93
	TH-2C	US-2	4-6	304.9	25	18.3	74.3	(2.9)	8.89	99	45, 72, 89
DI ANT C		US-3	7-9	310.5	24	17.0	69.9	2.88	9.58	93	70, 98
(Pond 4P)		US-1	1-3	334.2	23	16.8	72.7	(3.0)	10.19	98	57
(Folia 4D)	TU 2D	US-2	3-5	239.2	30	22.9	77.7	3.05	7.31	100	42, 52
	IH-2D	US-3	6-8	283.6	26	18.6	71.3	2.73	8.16	95	68, 99
		US-4	9-11	284.5	26	19.3	74.4	(2.8)	8.06	99	95
PLANT B		US-1	2-4	383.8	21	15.0	72.5	(2.8)	10.65	101	-
	TH-2A	US-2	5-7	271.1	27	20.4	75.6	2.78	7.51	100	83, 88, 89
		US-3	8-10	406.7	20	14.0	70.7	(2.8)	11.49	99	-
	TU 2D	US-1	2-4	349.5	22	15.7	70.4	(2.8)	10.13	97	79,90
PLANT C (Pond 4B) PLANT C (Pond 4B) PLANT B TH-2 PLANT B TH-2 TH-2 TH-2 TH-2 TH-2 TH-2 TH-2 TH-2	1п-2D	US-2	5-7	397.4	19	14.2	73.3	(2.8)	11.31	102	83, 97
	тц эл	US-1	0-2	216.6	32	24.9	78.8	(2.8)	6.02	101	50
	1 H- 2A	US-2	2-4	270.5	27	20.0	74.1	(2.8)	7.74	98	57, 89, 94
PLANT N		US-1	0-2	261.5	28	21.0	76.0	(2.8)	7.32	100	-
	TH-2B	US-2	2-4	255.4	22	21.2	75.5	2.76	7.13	99	52, 76, 97
Where: WO	$C_{110} = $ Water con	ntent (detern	nined using a	drying tem	perature	of 110 EC	$(C); S_{110} =$	Solids	content (f	or a dry	/ing
tem	perature of 110) EC); $\gamma_d = I$	Dry density; γ	$v_t = Total un$	it weigh	$ft; G_s = Sp$	ecific gra	vity (est	timated v	alues in	-
pare	entheses); $e = V$	oid ratio; S	= Degree of s	aturation; -	-200 = p	ercent ma	terial by d	lry weig	ht passing	g the U.	S. No. 200
siev	e size (<0.075)	mm).	-		1		2	. 0		_	

 Table 24. Density Determinations and Index Tests on Undisturbed Stage II Sludge Samples.

Facility	Boring No.	Sample No.	Sample Depth (ft)	-200 (%)	Percent less than 5µ (%)
		Sta	ge I Sludge	-	-
		US-1	1.25	88	25
	TH 1A	US-2	4.5	99	30
PLANT C	1 Π- 1 Α	US-3	6.75	91	50
		1A	0-6	86	40
	Lab Sample C	C-1 (pH=5)	-	90	5
	TII 1 A	US-2	2.0	98	30
PLANT B	IH-IA	1A	0-3	70	32
	Lab Sample E	B-1 (pH=5)	-	91	18
DI ANT N	TU 1D	US 2	2.0	98	41
FLANT N	IП-ID	1B	0-3	85	44
		Sta	ge II Sludge		
DLANTC	TH-2A	2A	3 - 6	74	33
(Pond 4A)	TH-2B	2B	2 - 5	75	33
(1 0110 477)	Lab Sample C	C-1(pH=11)	-	78	30
		US-1	1.75	93	46
		US-2	4.25	89	51
DLANTC		2C-1	0-3	59	26
PLANT C (Pond 4B)		2C-2	6 – 9	85	24
(I Olid 4D)		US-4	10.5	95	33
	TH-2D	2D-1	0 - 4	52	23
		2D-2	8 - 12	68	36
	TH-2A	US-2	6.0	83	57
DI ANT D		US-2	5.5	97	38
PLANI D	1п-2D	2B	3 – 5	82	67
	Lab Sample (pH=11)	-	39	15
DLANTD	Lab Sample I	D-1(pH=5)	-	86	15
PLANT D	Lab Sample I	D-1(pH=11)	-	82	29
	TH-2B	US-2	4.0	98	30
PLANT N	TH-2A	2A	1 – 3	70	32
	Lab Sample N	N-1(pH=5)	-	91	18
		Single	e-Stage Sludge	-	
PLANT C	TH-5A	SS-A	0-1	51	24
(Pond 5A)	TH-5B	SS-B	0-1	70	22
Where: -200 =	= percent materi	al by dry weigh	ht passing the U.S.	No. 200 sieve siz	ze (<0.075 mm).

 Table 25. Particle Size Analyses on Field and Laboratory Sludge Samples.

U.S. STANDARD SIEVE SIZE



Figure 31. Particle-size Distribution of Stage I Lime Sludge.



Figure 32. Particle-Size Distribution of Stage II Lime Sludge.

Atterberg Limits

The liquid limit and plastic limit were measured on the lime sludge samples in general accordance with ASTM D4318. Tables 26 and 27 present the results of Atterberg limits determinations for Stage I and Stage II sludge samples, respectively. The Atterberg limits are plotted on the plasticity chart in Figure 33.

Results of the Atterberg limit tests yielded liquid limit values ranging from 54-131% and plasticity index values ranging from 14-64% for Stage I field samples. The Stage I laboratory treatment samples were determined to be non-plastic. The laboratory samples contained more silt and less clay size particles than the field samples as shown in Appendix A, Figures A-1 (Plant C) and A-2 (Plant B). These variations in particle size were reflected on the sludge plasticity.

Stage II field samples exhibited liquid limit values ranging from 86-336% with plasticity index values ranging from 17-221%. The Stage II laboratory treatment samples yielded liquid limit values ranging from 137-188% and plasticity index values ranging from 31-88% except for the laboratory sample generated from Plant B which was deemed to be non-plastic. This sample was coarser than the other samples with only 34% of the particles passing the U.S. Standard No. 200 sieve and, therefore would classify as silty sand, SM. The non-plastic samples classify as low plasticity silt, ML. All of the other samples classify as elastic silt, MH in accordance with the Unified Soil Classification System (ASTM D2487). Depending on the particle-size, some additional qualifiers such as sandy or with sand are appropriate for each specific sample.

Free Lime Content

The amount of unreacted lime in selected Stage I and Stage II sludge samples was determined by titration method. Free Lime Content is equal to the combined percent by dry weight of quicklime (CaO), hydrated lime (Ca(OH)₂), and limestone (CaCO₃) as determined by calibrated titration methods, reported as CaO or CaCO₃ equivalent. In addition, the carbonate content was measured on the sludge samples using a gasometric method, ASTM D4373 "Rapid Determination of Carbonate Content of Soils" that calculates carbonate content as a calcite equivalent.

Results of the carbonate content and free lime content tests are presented in Tables 28 and 29 for field samples and for sludge generated in laboratory treatment experiments, respectively. The field samples yielded a free lime content ranging from 0.7-22.7% as CaO and from 1.3-40.6% as CaCO₃. In general, the free lime content ranged from less than 1% to more than 20% as CaO. The free lime content of Stage II sludge was higher than that of Stage I sludge, and was typically in the range of 5-10%. Hence, the sludge typically retains some buffering capacity. The single-stage sludge generated in the laboratory treatment experiments yielded free lime content values ranging from 0.2-0.7% as CaO and from 0.3-1.3% as CaCO₃. It should be noted that the laboratory controlled mixing produced almost

total reaction of the lime whereas the field samples exhibited, on occasions, significant amounts of free lime likely indicating that field treatment is less effective than laboratory mixing.

Facility	Boring No.	Sample No.	Sample Depth	W _{C110} (%)	LL	PL	PI	LI	-200 (%)
Stage I Slud	ge Pond S	Samples	. <u> </u>						
		US-1	1-3	200.5	92	46	46	3.4	75
PLANT C	TH-1A	US-2	3-5	199.2	98	56	42	3.4	99
		US-3	6-8	205.0	88	49	39	4.0	86
		US-1	1-3	233.4	92	59	33	5.3	100
DI ANT D	TH-1A	US-2	1-3	280.0	130	73	57	3.6	98
FLANT D		Bucket	-	207.4	83	53	30	5.1	-
	Surfac	e Sample	(Coarse)	120.0	54	40	14	5.7	-
	TH-1A	US-2	0-2	221.4	126	63	63	2.5	80
DI ANT N		Bucket	-	186.0	106	65	41	3.0	-
FLANT N	TU 1D	US 1	0-2	240.3	125	70	55	3.1	99
	1П-1D	US 2	0-2	222.9	131	67	64	2.4	98
Stage I Labo	oratory Tre	eatment Sl	ludge Samp	les					
	PLAN	IT B		270.1		Non-I	Plastic		91
	PLAN	NT C		458.8		Non-I	Plastic		90
	PLAN	IT D		468.9		Non-I	Plastic		86
Where: W _{C1}	$_{10} = Water$	r content d	etermined	using a dr	ying ten	nperatu	re of 1	10EC;	
LL =	= Liquid li	mit; PL =	Plastic lim	it; PI = Pl	asticity	index;	LI = Li	quidity	index;
-20	0 = percer	nt materia	l by dry we	ight passi	ng the U	J.S. No	. 200 si	eve siz	e
(<0.	075 mm).								

 Table 26. Atterberg Limits Determinations on Stage I Sludge Samples.

Facility	Boring No.	Sample No.	Sample Depth (ft)	W _{C110} (%)	LL	PL	PI	LI	-200 (%)
Stage II Slu	dge Pond	Samples							
PLANT C	TH-2A	Bucket		859.0	94	76	18	43.5	74
(Pond 4A)	TH-2B	Bucket		723.3	96	79	17	37.9	75
		US-1	1-3	357.6	336	115	221	1.1	93
		US-2	4-6	304.9	297	108	189	1.0	89
	TH-2C	US-3	7-9	310.5	146	57	89	2.8	-
		Bucket		441.4	98	78	20	18.2	59
PLANT C		Bucket		345.5	110	77	33	8.1	85
(Pond 4B)		US-2	3-5	239.2	141	68	73	2.3	52
		US-3	6-8	283.6	133	63	70	3.2	68
	TH-2D	US-4	9-11	284.5	86	53	33	7.0	-
		Bucket		391.4	101	75	26	12.2	52
		Bucket		358.1	129	74	55	5.2	68
		US-2	5-7	271.1	110	66	44	4.7	88
	TH-2A	US-3	8-10	406.7	173	81	92	3.5	-
DI ANT D		Bucket		1447	196	108	98	13.7	-
PLANT B		US-1	2-4	349.5	134	66	68	4.2	90
	TH-2B	US-2	5-7	415.0	121	69	52	6.7	83
PLANT C (Pond 4B) PLANT B PLANT D PLANT N Stage II Lat		Bucket		603.7	234	108	126	3.9	-
PLANT D	TH-2	Bucket		1577	173	88	85	17.5	-
PLANT N	TH-2B	US-2	2-4	255.4	149	73	76	2.4	97
Stage II Lab	oratory T	reatment S	Sludge Sar	nples	-				
	PLAN	ТВ		1684		Non-F	Plastic		34
	PLAN	ТС		1805	137	106	31	54.8	78
	PLAN	T D		1372	188	107	81	15.6	82
	PLAN	ΤN		1608	187	99	88	17.1	72
Where: W _{C1}	$_{10} = Wate$	er content	determine	ed using	a dryin	g tempe	rature c	of 110°	$^{\mathbb{C}}$; LL =
Liqu	uid limit; I	PL = Plast	ic limit; Pl	I = Plastic	ity inde	ex; LI – I	Liquidit	y index	; -200 =
perc	ent mater	ial by dry	weight pas	ssing the	U.S. No	5. 200 si	eve size	e (<0.07	5 mm).

 Table 27. Atterberg Limits Determinations on Stage II Sludge Samples.



Figure 33. Plasticity Chart of Stage I, Stage II and Single-Stage Lime Sludge Samples.
	Samula	Depth	Carbonate	Free Lime C	Content (%)
Facility	Sample	Range	Content		
	INO.	(ft)	% as CaCO ₃	as CaO	as $CaCO_3$
Stage I Sludge					
PLANT B	1A	0-3	9.1	8.6	15.3
PLANT N	1A	0 – 3	1.0	0.7	1.3
PLANT A	1 [∺]	n/a	0.7	-	-
Stage II Sludge					
PLANT C	2A	3 -6	4.6	-	-
Pond 4A	2B	2 -5	4.4	-	-
	2C-1	0 -3	3.2	-	-
PLANT C	2C-2	6 -9	7.5	12.2	21.8
Pond 4B	2D-1	0 -4	5.0	8.8	15.7
	2D-2	8 -12	13.0	-	-
DI ANT D	2A	3 -7	12	-	-
PLANI D	2B	3 -5	7.7	5.9	10.5
DLANT N	2A	1 -3	21	-	-
PLANT N	2B	2 -4	20	22.7	40.6
PLANT D	2A	1 -3	5.0	-	-
PLANT A	2 ^H	n/a	7.5	-	-
Single-Stage Sludg	ge				
PLANT C	SSA	-	1.0	-	-
Pond 5A	SSB	-	0.6	-	-
^H Sample from med	chanical clarif	ier underflow			

Table 28. Free Lime and Carbonate Content of Field Sludge Samples.

Table 29. Free Lime and Carbonate Content of Sludge Generated in Laboratory Treatment Experiments.

		Carbonate	Free Lime	Content (%)
Facility	рН	Content % as CaCO ₃	as CaO	as CaCO ₃
Stage I Sludge				
PLANT D	4.9	0.8	-	-
PLANT N	4.8	1.8	-	-
Stage II Sludge				
PLANT C	11.0	2.0	-	-
PLANT D	11.5	1.8	-	-
PLANT N	10.7	1.1	-	-
Single-Stage Sludge				
DI ANT D	7.0	0.3	0.2	0.3
PLANI D	7.2	0.5	-	-
PLANT N	7.1	2.0	0.7	1.3

Solubility in Process Water

Field sludge samples from Plants B and C were tested to determine the effect of adding Stage I and Stage II sludge to process water. As shown in Table 28, some of the field sludge samples contained significant quantities of free lime. When the sludge was mixed with process water, some of the sludge constituents dissolved or reacted with process water causing the pH to increase and some of the dissolved solids in the process water to precipitate.

The tests were performed by mixing approximately 2 liters of process water with 20g, 50g or 100g of sludge. After mixing, the slurry was allowed to settle. The settled sludge (i.e., reacted sludge) was collected for testing. Sieve and hydrometer analyses were performed on selected reacted sludges with results presented in Appendix A, Figure A-11 and A-12. The particle-size distribution for reacted Stage I sludge is very similar to that of unreacted sludge. The particle size of the reacted Stage II sludge is somewhat coarser than that of the corresponding unreacted sludge.

The supernatant from the reacted sludges was sampled for testing pH and conductivity (results shown in Table 30). The supernatant pH increased, and in most cases, conductivity decreased from the prior-to-reaction values of the corresponding process water. These changes indicate beneficial treatment of the process water with the addition of both the Stage I and Stage II sludges. These results are consistent with prior findings presented in Figure 12.

	Pı	rocess Water	Dm	Average	S	upernatant
Facility	рН	Conductivity (µmhos/cm)	Mass of Added Sludge (g)	Ratio Lime Sludge/ Pond Water (by Volume)	рН	Conductivity (µmhos/cm)
Stage I Sludge						
PLANT B	1.7	26,000	20	0.030	2.0	20,900
PLANT B	1.7	26,000	50	0.079	2.4	19,930
PLANT C	2.9	8,670	20	0.024	3.5	8,740
Stage II Sludge						
PLANT B	1.7	26,000	100	0.256	2.9	14,520
PLANT C	2.9	8,670	20	0.033	3.3	7,920

 Table 30. pH and Conductivity of Supernatant from Reacted Sludges Generated in Laboratory Treatment Experiments.

SETTLING AND CONSOLIDATION PROPERTIES

Sedimentation Characteristics of Lime Sludge

Laboratory settling tests were performed in graduated plexiglass settling columns on sludge (Stage I, Stage II and single-stage) from the field (bucket and undisturbed samples). The sludge was adjusted to an initial solids content of about 2%, and the sludge slurry was poured into a settling column to a desired initial sample height. The sludge slurry was placed in a 10.0-10.8 cm diameter column to a height of 27.0 cm. The slurry was mixed with a hand-held stirrer to provide a homogeneous sample, and remove any segregation of particles which occurred during placement of the slurry into the column. The columns were securely covered with clear plastic wrap to prevent evaporation of the supernatant fluid during the test period. The settling tests were performed in a fluorescent-lighted laboratory and were not exposed to direct sunlight.

Performance of the settling tests consisted of visually monitoring the height of the sludge slurry-supernatant interface versus time. Depending on the behavior of the sludge, initial readings were obtained of height versus time in the range of one reading every 1 to 10 minutes. Subsequent readings were obtained at increasing time intervals. The tests were continued for a period of at least 24 hours, or until the settled height remained constant.

After completion of the test, the solids content of the settled sludge was measured for comparison to the calculated value based on final height and initial solids content. The supernatant pH and specific conductance at the end of the test were measured to determine if a significant change in supernatant chemistry occurred over the test period. Results of the settling tests performed on the field sludge samples are presented in Table 31.

The initial settling velocities of the field sludge samples (i.e., the initial slope of the settled height versus time curve) ranged from 0.3-0.6, 0.02-0.3 and 0.8-1.2 cm/min for Stage I, Stage II and single-stage sludge samples, respectively (except for one anomalous sample of Stage II sludge that exhibited a higher settling velocity of 2.0 cm/min). The initial settling velocities of the laboratory generated sludge samples (see previous section titled "Evaluation of Sludge Production") ranged from 1.7-5.8, 0.2-1.3, and 0.3-10.0 cm/min for Stage I, Stage II and single-stage sludge samples, respectively, which were generally higher than the corresponding sludge-type initial settling velocities observed in the field samples. In general, the Stage II sludge yielded the lowest initial settling velocities.

The settling tests for Plant B-Stage I sludge settled to final water contents (260% and 370%) which are slightly higher than the measured water contents of the field samples (200 to 205%). The settled final water content for all of the other Stage I and Stage II settling tests from Plants B, C and N were significantly higher, ranging from 795 to 1325%, than the field sample measured water contents that ranged from 210 to 410%. The difference between field sludge measured water contents and the water contents measured in the laboratory settling tests on remolded samples likely reflect the fact that the laboratory samples are subjected to lower effective stresses and much shorter time for consolidation and creep than the field samples.

		Sludge Sample				Slurry Water		Col.	Init	ial		Final Se	ettled Slue	lge	
Facility	Course	"IJ×	Conductivity*	WDS	Source	Conductivity	TDS	Dia.	H _i	Si	$H_{\rm f}$	Sf	Wf	γ_{df}	V_{si}
	Source	рп∘	(µmhos/cm)	(gm)	(Pond)	(µmhos/cm)	(mg/l)	(cm)	(cm)	(%)	(cm)	(%)	(%)	(lb/ft^3)	(CIII/IIIII)
Stage I Pond	l Samples														
DI ANT D	TH-1A	8.8	3,440	48.37	1	6 150	5 020	10.64	27.0	2.0	2.6	21.3	370	15.5	0.64
I LANI D	Surface	11.8	9,170	46.52	1	0,150	5,950	10.70	27.0	1.9	1.5	27.8	260	21.5	0.50
DI ANT N	TH-1A	5.9	5,590	45.91	1	14 250	10.200	10.41	27.0	2.0	6.4	8.1	1,135	5.3	0.35
FLANT N	TH-1B	5.7	6,290	47.98	1	14,550	19,200	10.69	27.0	2.0	7.0	7.3	1,270	4.8	0.40
Stage II Pon	d Samples			-											
	TH-2A	8.3	9,780	51.09	4A	6,260	4,320	10.70	27.0	2.1	8.0	6.8	1,360	4.4	0.11
PLANT C	TH-2B	8.0	8,030	50.47	4A	6,260	4,320	10.68	27.0	2.1	6.1	8.7	1,050	5.8	0.19
	TH-2C	7.3	4,810	50.25	4B	6,030	4,320	10.67	27.0	2.1	4.6	11.2	795	7.6	0.33
	TH-2D	7.8	2,340	49.06	4B	6,030	4,320	10.63	27.0	2.0	6.7	7.8	1,180	5.2	0.25
DI ANT D	TH-2A	8.7	3,400	49.70	2	2 000	2 520	10.66	27.0	2.1	6.1	8.8	1,035	5.8	0.20
FLANI D	TH-2B	7.8	3,140	49.82	2	3,900	5,520	10.68	27.0	2.1	7.7	7.0	1,325	4.6	0.11
PLANT N	TH-2A	11.5	4,320	44.81	2	7.660	5 570	10.40	27.0	1.9	5.7	8.7	1,045	5.8	0.08
	TH-2B	11.5	4,860	46.77	Z	7,000	3,370	10.65	27.0	1.9	6.2	8.0	1,145	5.3	0.08
PLANT D	Stage II	8.0	11,870	43.12	2	5,980	4,320	10.08	27.0	2.0	9.8	5.3	1,785	3.4	0.02
PLANT A	R.U.	10.1	10,420	46.19	2	7,820	4,480	10.63	27.0	1.9	2.3	21.5	365	15.6	2.00
Single-Stage	e Pond Sam	ples													
PLANT C	TH-A	5.5	1,950	49.15	Stage I	10,000	17,500	10.70	27.0	2.0	2.5	19.2	420	13.6	0.80
(Pond 5A)	TH-B	4.7	2,810	46.56	Stage I	10,000	17,500	10.38	27.0	2.0	1.8	25.3	295	19.1	1.15
Where: WD	S = Weight	t of dry s	solids; $TDS = Tot$	tal dissolv	ved solids;	$H_i = Initial heighted H_i = Initial height$	ht; $S_i = Ini$	tial slurry	solids c	ontent;	$H_f = Fir$	nal heigł	nt; $S_f = Fi$	nal slurry	solids
cont	ent (at end	of the 24	4-hour settling pe	riod); e _f =	= Final voi	d ratio (compute	ed assumin	g a specif	fic gravit	y of 3.0	for sluc	lge solic	ls); $w_f = I$	Final water	content
(usin	(using a drying temperature of 100 ^o C); $\gamma_{d,f}$ = Final dry density; v_{si} = Initial settling velocity.														

Table 31.	Summary of	of Settling	Test	Results for	Field	Sludge	(Bucket)	Samples.
							(

* Pore water dilution using a 2:1 ratio of de-ionized water to air-dried samples.

Conventional Consolidation Tests

One-dimensional incremental consolidation tests were performed on each undisturbed lime sludge (Stage I and Stage II) sample in general accordance with ASTM D2435. The results of the consolidation tests are presented in Table 32. The consolidation test data, in terms of void ratio versus effective vertical consolidation stress and the coefficient of consolidation versus effective vertical consolidation stress, are presented in Figures C-1 through C-8 for Stage I samples and Figures C-9 through C-19 for Stage II samples in Appendix C. The coefficient of consolidation (C_v) , which governs the rate of primary consolidation, and coefficient of secondary compression based on strain (C_{α}), which governs the rate of drained creep following primary consolidation, measured on Stage I and Stage II sludge samples are presented in Figure 34. The C_{α} values of the undisturbed sludge samples are fairly consistent with values from previous research (Figure 8) at vertical effective consolidation stresses from 0.1 to 1.0 kg/cm² and are equal to or greater than previously measured for vertical effective consolidation stresses from 1.0 to 5.0 kg/cm². The C_v values are slightly higher than previously determined values for a vertical effective consolidation stress, $\bar{\sigma}_{vc}$, less than 1 kg/cm² and are slightly lower compared to previously determined values for $\overline{\sigma}_{yc}$ greater than 1 kg/cm². The lime sludge behavior is comparable to an elastic silt and, therefore, the rate of consolidation is 10 to 100 times faster than typical for plastic, CHtype, waste phosphatic clays.

The compression index, C_c , is plotted versus the *in situ* water content (NM), initial void ratio (e_o) and sludge liquid limit (LL), in Figure 35. One set of test results for Plant B-Stage II does concur with the general trend. As shown, a relationship with a good correlation coefficient is determined between C_c and e_o and NM. The C_c for both Stages I and II sludge follows the same trend with increasing NM and e_o . Figure 36 presents the relationship between the coefficient of secondary compression (C_α) and virgin compression ratio (CR) in terms of strain from laboratory consolidation tests on undisturbed samples. As shown, the C_α to CR ratio ranges from 0.025 to 0.010 with an average of 0.016.

Slurry Consolidation Tests

One-dimensional incremental consolidation tests were performed on three lime sludge samples (Plant C-Stage II, Plant N-Stage I and Plant N-Stage II) at an initial solids content corresponding to the underflow solids content. Sufficient slurry was sedimented in 10.2-cm diameter by 60-cm tall settling columns within a one-dimensional consolidometer to produce a specimen height at the end of settling of approximately 10 to 15 cm. After gravity settling was substantially complete, the specimens were loaded one-dimensionally under stresses of 0.001 to 1.0 kg/cm² using a load increment ratio of 1 (i.e., 14 load increments). The tests were performed using specially designed and fabricated equipment with a counterbalance pulley system (to counteract a portion of the normal load associated with the top loading piston) that is similar to a conventional one-dimensional consolidation device except that the equipment used allows for evaluation of the consolidation behavior at stresses as low as 0.001 kg/cm². Stress increments greater than 0.5 kg/cm² can also be applied by a

pneumatic loading frame after locking the piston. The slurry consolidation equipment has the added feature of allowing for the ability to perform constant head hydraulic conductivity tests after consolidation at each effective stress increment.

The change in specimen height with time under each load was monitored and evaluated to characterize the one-dimensional compressibility, consolidation and drained creep properties of the sludge. The hydraulic conductivity of the lime sludge at the end of selected load increments was measured by the constant-head method using a small hydraulic gradient. Results of these tests are presented in Figures C-20 through C-22 in Appendix C and in Tables 33 and 34.



Figure 34. Consolidation Properties of Stage I and Stage II Lime Sludges.



Figure 35. Compression Index Versus Sludge Index Properties.



Figure 36. C_{α} and CR Relationship for Undisturbed Samples.

Boring Sample Sample Index Properties Initial Conditions Final Conditions Consolidation Paran							aramete	ers									
Site	No.	No.	Depth	-200		PI	Wi	γ_i	ei	S_i	W _f	$\gamma_{\rm f}$	e _f	$\overline{\sigma}_{vo}$	$\overline{\sigma}_{vm}$	C _c	c_v
	G1 1		(11)	(%)	(%)	(%)	(%)	(pcf)		(%)	(%)	(pcf)		(kg/cm^2)	(kg/cm ²)		(cm^{-}/s)
Stage I Lim	e Sludge																3
PLANT B	TH-1A	US 1	2.0	100	92	33	193	27.4	5.84	99	122	40.1	3.67	0.016	0.310	1.80	6×10^{-3}
		US 2	2.0	85	130	57	265	21.0	7.93	100	151	33.7	4.55	0.014	0.352	3.15	1x10 ⁻²
		US 1	2.0	97	92	46	220	24.7	6.58	100	144	35.1	4.34	0.017	0.342	2.21	$2x10^{-2}$
PLANT C	TH-1A	US 2	4.0	90	98	42	215	25.1	6.46	100	134	37.4	4.00	0.034	1.172	2.18	8×10^{-3}
		US 3	7.5	98	88	39	233	23.2	7.07	99	165	31.5	4.95	0.063	1.172	2.26	1×10^{-2}
	TH-1A	US 2	1.5	80	126	63	208	26.0	6.21	100	128	38.6	3.85	0.012	0.928	2.85	8x10 ⁻³
PLANT N	TH-1B	US 1	1.0	99	125	55	226	23.9	6.83	99	139	36.1	4.19	0.007	1.001	2.53	$8x10^{-3}$
	TH-1B	US 2	1.5	99	131	64	298	18.9	8.92	100	180	29.3	5.39	0.012	1.050	4.63	3x10 ⁻³
Stage II Lin	ne Sludge													-			
PLANT B	TH-2A	US 2	6.5	89	10	44	182	26.4	6.08	90	124	39.5	3.74	0.037	0.952	2.70	$4x10^{-2}$
	TH-2B	US 1	3.0	90	134	68	468	12.2	14.35	98	302	18.5	9.12	0.011	0.350	3.82	$7x10^{-3}$
	TH-2B	US 2	5.5	83	121	52	261	21.2	7.85	100	180	29.3	5.39	0.023	0.410	2.65	$7x10^{-3}$
	TH-2C	US 1	2.5	37	336	221	292	18.6	9.06	97	189	27.8	5.71	0.012	1.465	4.32	$4x10^{-3}$
	TH-2C	US 2	5.5	45	297	189	364	15.1	11.41	96	235	23.3	7.02	0.029	1.709	6.84	$4x10^{-3}$
	TH-2C	US 3	8.25	98	146	89	377	15.0	11.49	98	225	24.1	6.77	0.043	0.415	5.20	$7x10^{-3}$
PLANT C	TH-2D	US 2	5.0	42	141	73	141	35.4	4.29	98	87	51.8	2.61	0.023	1.074	1.18	5×10^{-3}
	TH-2D	US 3	7.5	99	133	70	298	18.7	9.03	99	200	26.8	5.98	0.039	0.488	4.98	$3x10^{-3}$
	TH-2D	US 4	10.5	95	86	33	241	22.4	7.35	98	155	33.1	4.66	0.054	0.635	3.03	$4x10^{-3}$
	TH-2A	US 2	3.75	94	-	-	299	18.8	8.97	100	190	28.0	5.69	0.027	0.366	2.92	5x10 ⁻³
PLANT N	PLANT N TH-2B US 2 3.25 52 149 76 325 17.4 9.76 100 222 24.5 6.65 0.023 0.280 4.82 $4x10^{-4}$																
Where: -20	00 = percent	t material by	y dry weigh	t passing	g the U.	S. No. 2	00 sieve	e size (<	0.075 mr	n); LL =	Liquid	Limit; l	PI = Pla	sticity Index;	; w _i = Initial	moistu	re
co	ontent; $\gamma_i = \mathbf{I}$	nitial dry de	ensity; $e_i =$	Initial vo	oid ratio	; $\mathbf{S}_{i} = \mathbf{In}$	itial deg	gree of s	aturation	; $\mathbf{w}_{\mathrm{f}} = \mathrm{Fi}$	inal moi	sture co	ntent; γ_{f}	= Final dry	density; e _f =	Final v	void
ra	tio; $\overline{\sigma}_{vo} = \mathrm{Es}$	stimated in s	s <i>itu</i> vertical	effectiv	e stress	; $\overline{\sigma}_{vm} =$	Estimat	ed pre-c	onsolida	tion ver	tical effe	ective st	ress; C _c	= Compressi	on Index; c	v = Coe	fficient
of	of consolidation at effective vertical consolidation stress = $2\sigma_{vm}$.																

 Table 32. Summary of Laboratory Consolidation Test Results for Undisturbed Sludge Samples.

Index Properties **Initial Conditions Final Conditions Consolidation Parameters** Sample Boring Site Depth -200 LL S_i $\frac{c_v}{(cm^2/s)}$ PI Wi W_{f} k γ_i $\gamma_{\rm f}$ $\overline{\sigma}_{v}$ (kg/cm²) No. ei ef (pcf) (pcf) (ft) (%) (%) (%) (%) (%) (%) (cm/s) PLANT N TH-1B 0-2 99 125 55 506 15.19 100 188 28.3 5.62 0.001 1.5E-03 11.6 -2.4E-03 0.002 0.004 1.4E-03 6.3E-05 0.008 7.6E-04 _ 0.016 1.0E-03 1.6E-05 0.032 1.7E-03 -0.064 1.8E-03 -4.0E-03 0.128 -0.250 6.2E-03 -1.1E-02 0.500 -1.000 9.6E-03 Where: -200 = percent material by dry weight passing the U.S. No. 200 sieve size (<0.075 mm); LL = Liquid Limit; PI = Plasticity Index; w_i = Initial water content; γ_i = Initial dry density; e_i = Initial void ratio; S_i = Initial degree of saturation; w_f = Final water content; γ_f = Final dry density; e_f = Final void ratio; $\overline{\sigma}_v$ = Vertical effective stress; c_v = Coefficient of consolidation; and k=Hydraulic conductivity

 Table 33. Summary of Slurry Consolidation Test Results for Stage I Sludge Sample.

	D	Sample	Inde	x Prope	rties		Initial C	onditions		Fina	l Condit	tions	ns Consolidation Parameters		
Site	Boring	Depth	-200	LL	PI	Wi	γ _i		Si	Wf	$\gamma_{\rm f}$		- (1 (2)	C _v	k
	INO.	(ft)	(%)	(%)	(%)	(%)	(pcf)	ei	(%)	(%)	(pcf)	e_{f}	σ_v (kg/cm)	(cm^2/s)	(cm/s)
PLANT	TH-2D	0-4	90	141	73	737	8.0	22.55	98	205	25.8	6.27	0.001	4.5E-03	-
С													0.002	2.8E-03	7.4E-05
													0.004	2.1E-03	-
													0.008	1.7E-03	2.9E-05
													0.016	1.4E-03	-
													0.032	1.4E-03	-
													0.064	1.9E-03	-
													0.128	1.5E-03	-
													0.250	-	-
													0.500	1.3E-03	-
													1.000	1.8E-03	-
PLANT	TH-2A	0-4	96	-	-	737	8.0	22.55	98	205	25.8	6.27	0.001	4.5E-03	-
Ν													0.002	2.8E-03	-
													0.004	2.1E-03	2.1E-04
													0.008	1.7E-03	-
													0.016	1.4E-03	3.7E-05
													0.032	1.4E-03	-
													0.064	1.9E-03	-
													0.128	1.5E-03	-
													0.250	-	-
	0.500 1.3E-03 -														
													1.000	1.8E-03	-
Where: -	200 = perce	ent material	by dry	weight p	bassing t	he U.S.	No. 200) sieve siz	ze (<0.0	75 mm)	; $LL = L$	Liquid Li	mit; PI = Plastici	ty Index; $w_i = Initian$	al water
content; y	$t_i = $ Initial di	ry density; e	e _i = Initia	al void 1	atio; S _i	= Initial	degree	of saturat	tion; w _f	= Final	water co	ontent; γ	= Final dry densi	ity; e _f = Final void	ratio; $\overline{\sigma}_{v} =$
Vertical e	Vertical effective stress; $c_v = Coefficient of consolidation; and k=Hydraulic conductivity$														

 Table 34. Summary of Slurry Consolidation Test Results for Stage II Sludge Samples.

HYDRAULIC CONDUCTIVITY AND LEACHING CHARACTERISTICS

Hydraulic Conductivity Tests

A total of six laboratory constant-head, flexible-wall hydraulic conductivity tests were performed on cylindrical test specimens trimmed from undisturbed Stage I and Stage II lime sludge samples in general accordance with ASTM D5084. The samples were back-pressure saturated and consolidated to isotropic effective confining stresses approximately equal to the *in situ* vertical effective stress estimated based on the sample depth. The consolidated samples were then permeated with lime sludge-saturated water until the outflow-to-inflow ratio had stabilized under hydraulic gradients ranging from about 9 to 16.

Results of the laboratory hydraulic conductivity tests are summarized in Table 35. The results show vertical saturated hydraulic conductivity values ranging from 2.6×10^{-7} cm/sec to 5.7×10^{-6} cm/sec for the Stage II sludge. The vertical hydraulic conductivity for one sample of Stage I sludge equaled 3.8×10^{-6} cm/sec. Although this is within the range of measured values for Stage II sludge, the sample was at a lower void ratio and much higher dry density than the Stage II sludge samples. Results from the hydraulic conductivity tests are presented in Figure 37 along with the results from the hydraulic conductivity tests on the slurry consolidation tests. In general, the hydraulic conductivity of the Stage II lime sludge is about one to two orders of magnitude higher than that of the Stage II lime sludge at equivalent void ratios.



Figure 37. Hydraulic Conductivity of Stage I and Stage II Sludge Samples.

Leaching Tests

Leaching tests were performed to document that metals contained in lime sludge will not readily leach into the environment by rainwater runoff and/or infiltration. Three leaching tests on Plant B-Stage I, Plant B-Stage II, and Plant N-Stage II were conducted by permeating synthesized rainwater (pH-5) through reconstituted lime sludge samples within stainless steel cylindrical molds. The outflow was collected and chemistry determined at approximately each void volume (total initial volume of voids within a specimen) for over five void volumes of flow. The pH, conductivity, As, Cd, Cr, Pb, Ag, Mn, Fe, Ni, Al, Na, P and F were measured on the collected outflow samples. Tables 36 and 37 present summaries of physical sample data and leachate collection, and Table 38 presents results of chemical analyses on selected leachate samples. As shown, there is no exceedance of any of the standards for metals after two void volumes of flow except in the case of the secondary standard for aluminum upon leaching the Plant B-Stage I sludge sample.

Table 35. Summary of Constant Head Flexible-Wall Hydraulic Conductivity Test Results for Undisturbed Sludge Samples.

-200
(/0)
94
79
70
57
89
76
-6 -6 -7 -6

Where: L= Sample Length; D= Sample Diameter; w_c = Water content; γ_d = Dry density; e = Void ratio.

S = Calculated degree of saturation using an assumed specific gravity of 3.0.

 $\Delta V/V_o$ = Volume change from initial to final condition (negative values denote consolidation).

 k_{20} = Saturated hydraulic conductivity at 20°C; and -200 = percent material by dry weight passing the U.S. No. 200 sieve size (<0.075 mm).

All samples were back-pressure saturated at a confining pressure of 100 lb/in², and consolidated under an average isotropic effective confining stress, $\overline{\sigma}_{c} = 3.0 \text{ lb/in}^{2}$.

 Table 36. Summary of Constant-Head Rigid-Wall Hydraulic Conductivity Results from Laboratory Remolded Sludge Leaching Tests.

	Composite	Sample			Init	ial Co	nditions			Average		Fina	l Condi	tions		Average
Site	Sample	Depth	L	D	WDS	Wi	$\gamma_{d,i}$	Vvi		Hydraulic	W _f	$\gamma_{d,f}$		Vv _f	$\Delta V/V_o$	Flow Rate
	No.	(ft)	(cm)	(cm)	(gm)	(%)	(lb/ft^3)	(cm^3)	ei	Gradient	(%)	(lb/ft^3)	$e_{\rm f}$	(cm^3)	(%)	(Vv/day)
Stage I Sluc	lge															
PLANT B	1A	0 - 3	12.7	10.13	423.3	259	21.8	1068	7.77	2.7	194	26.8	5.80	834	-18.7	0.26
Stage II Slu	ıdge															
PLANT B	2B	3 - 5	16.0	10.08	247.2	514	11.7	1233	15.42	1.0	461	12.9	13.83	1114	-9.1	0.21
PLANT N	2A	1 - 3	16.5	10.08	414.5	294	19.7	1178	8.82	2.1	280	19.9	8.40	1162	-1.2	0.21
Where: L=	Initial samp	le length;	, D = Sa	ample c	liameter	r; WD	$\overline{S} = Weig$	ght of dry	y sludge	solids;						
w _i =	= Initial wate	er content	(deter	mined	using a	drying	g tempera	ature of 1	10EC);	$\gamma_{d,i} = Initial$	dry der	nsity.				
Vv	$_{i} =$ Initial voi	id volume	e (calcı	lated u	using a s	specifi	c gravity	of 3.0);	$e_i = Init$	ial void ratio).					
w _f =	= Final water	content ((at 110	EC); γ _d	i,f = Fina	al dry	density.									
Vv_{f}	= Final void	l volume	(calcul	ated us	ing a sp	ecific	gravity of	of 3.0); e	f = Fina	l void ratio.						
ΔV	$/V_{o} = Volum$	e change	from i	nitial to	o final c	onditi	on (nega	tive valu	es deno	te consolidat	tion).					

	Composite	Elapsed	Time	Average	Increr	nental	Cumu	lative	Leachate			
Site	Sample	Time	Increment	Hydraulic	Fl	ow	Flo	ow	Conductivity			
	No.	(days)	(days)	Gradient	ml	$V_{\rm V}$	ml	Vv	(µmhos/cm)			
Stage I S	ludge	-						-	-			
		4.97	4.97	1.6	1008	1.18	1008	1.18	2,300			
		10.21	5.24	2.2	1215	1.44	2223	2.62	1,705			
PLANT	1.4	14.35	4.14	2.6	913	1.08	3136	3.70	978			
В	IA	19.50	5.15	2.9	1105	1.31	4241	5.01	753			
23.75 4.25 3.0 895 1.06 5136 6.07 688												
28.52 4.77 3.0 1011 1.20 6147 7.27 692												
Stage II	Sludge											
		5.88	5.88	1.0	1271	1.11	1271	1.11	-			
		10.84	4.96	1.0	1267	1.14	2479	2.25	1,115			
PLANT	28	14.97	4.13	1.0	1083	0.97	3562	3.22	939			
В	20	20.11	5.14	0.8	1373	1.23	4935	4.45	-			
		24.24	4.13	1.1	1034	0.93	5969	5.38	629			
		31.32	7.08	1.1	1190	1.07	7159	6.45	551			
		6.89	6.89	1.4	1022	0.87	1022	0.87	8,170			
		11.87	4.98	2.0	1068	0.92	2089	1.79	7,770			
PLANT	24	16.11	4.24	2.2	902	0.78	2991	2.57	7,200			
Ν	21	20.24	4.13	2.2	1034	0.89	4026	3.46	5,910			
24.37 4.13 2.2 1179 1.01 5205 4.47 5,110												
		28.42	4.05	2.2	1335	1.15	6540	5.61	5,160			
Where:	Where: $Vv = Quantity$ of synthetic rain water permeated through sample divided by the initial											

 Table 37. Summary of Leaching Test Results for Remolded Sludge Samples.

volume of voids in the sample (calculated using a specific gravity of 3.0).

D. (Class G-II	Synthetic	Stag Plan	ge I It B	Stag Plai	ge II nt B	Stag Plai	ge II nt N	
Parameter	Standards	Rainwater	2.6-3.7	6.1-	2.3-3.2	5.4-6.5	1.8-2.6	4.5-5.6	
		(Blank)	V_V^*	7.3V _v *	V_V *	V _v *	V _v *	V _v *	
INITIAL									
PARAMETERS									
(BY ARDAMAN)		1							
pH	6.0 - 8.5	4.6	8.6	8.0	7.8	8.2	12.1	12.0	
Conductivity	1,275	16	978	692	939	551	7,200	5,160	
(µmhos/cm)									
LABORATORY ANAL	YSES (By Tes	tAmerica, In	c.)					_	
Major Constituents									
(mg/l)									
Sodium, Na	160(P)	0.87 [I]	85.7	17.7	60.7	20.7	274	80	
Fluoride, F	4.0(P)/2.0(S)	0.1 [U]	10.4	9.0	2.0	1.7	5 [U]	5 [U]	
Total Phosphorus, P	-	0.05 [U]	0.05 [U]	0.05 [U]	0.21	0.08	0.05 [U]	0.05 [U]	
Metals (µg/ml)	200(0)	50	4 1 0 2	1.064	50	50	50	50	
Aluminum, Al	200(S)	50 [U]	4,183	1,064	50 [U]	50 [U]	50 [U]	50 [U]	
Arsenic, As	10(P)	5 [U]	5 [U]	5 [U]	5.8	5 [U]	5 [U]	5 [U]	
Cadmium, Cd	5(P)		0.1[0]	0.1[U]	0.1[U]	0.1[U]	0.1[U]	0.1[U]	
Chromium, Cr	100(P)	5 [U]	5 [U]	5 [U]	5 [U]	5 [U]	5 [U]	5 [U]	
Iron, Fe	300(S)	3.9 [I]	11.1[1]	12.8[1]	8.3 [I]	5.1 [I]	36.2	30.1	
Lead, Pb	15(P)	3 [U]	3 [U]	3 [U]	3 [U]	3 [U]	9.8	4.3	
Manganese, Mn	50(S)	5 [U]	5 [U]	5 [U]	5 [U]	5 [U]	5 [U]	5 [U]	
Nickel, Ni	100(P)	3.3 [I]	5.2 [1]	0.7 [I]	7 [U]	7 [U]	117.5	36.6	
Silver, Ag	100(S)	3 [U]	3 [U]	3 [U]	3 [U]	3 [U]	3 [U]	3 [U]	
Other Parameters									
nH	60-85	5 19	8 47	7 96	8 1 2	8 39	123	123	
Conductivity	1 275	13	1 020	715	929	573	6 970	5 200	
(umhos/cm)	1,275	15	1,020	/15	141	515	0,270	3,200	
NOTES \cdot [I] = Labor:	atory detection	limit		<u> </u>					
[I] = Report	ed value is betw	veen the labo	ratory met	hod detec	tion limit	and the l	aboratory	,	
practic	val quantitation	limit	iutory mee	nou actee	tion min.	uno me i	ubbruibry		
$V_{\rm w}^*$ = Interv	val of void volu	mes of leach	ate flow th	rough slu	dge samp	le			
$V_V = $ Interval of volumes of leachate flow through studge sample (P) = Primary standard; (S) = Secondary standard									

Table 38.	Chemical An	alyses on 1	Laboratory	Leachate	Samples.
		•			1

(P) = Primary standard; (S) = Secondary standard

STRENGTH PROPERTIES

The shear strength of lime sludge is of interest for evaluating the requirements for placement of a soil cap atop a sedimentation pond during closure. The strengths of the undisturbed sludge samples were determined using laboratory miniature vane shear tests and triaxial compression tests. The strengths of remolded sludge samples were determined using triaxial compression tests.

Laboratory Miniature Vane Shear Tests

Laboratory miniature vane shear strength tests were performed on undisturbed Stage I and Stage II sludge samples in general accordance with ASTM D4648 to characterize the undrained shear strength of lime sludge as a function of solids content. Results of laboratory vane shear strength tests on undisturbed sludge samples are presented in Table 39. The results show peak undrained shear strengths ranging from 19 to 225 psf for Stage I undisturbed sludge samples and peak undrained shear strengths from 9 to 280 psf for Stage II undisturbed sludge samples. Two Stage II samples from Plant N and one Stage II sample from Plant B displayed undrained shear strengths less than 20 psf, indicative of a very soft, very low strength material.

Triaxial Compression Tests on Undisturbed Samples

Isotropically consolidated-undrained triaxial compression tests are used to characterize the drained angle of internal friction and normalized undrained shear strength of lime sludge which are of interest for capping sedimentation ponds.

Isotropically consolidated-undrained triaxial compression tests with pore pressure measurements **CIUC** were performed in general accordance with ASTM D4767 on specimens trimmed from selected undisturbed tube samples taken from Plants B, C and N for both Stage I and Stage II sludges. The specimens were back-pressure saturated and then isotropically consolidated in increments to effective consolidation stresses of 0.25 or 0.5 kg/cm². The test specimens were subsequently sheared in compression at a constant rate of axial strain of about 1%/hour without allowing drainage. Summaries of the **CIUC** test data and results for the undisturbed samples are presented in Tables 40 and 41, respectively. The measured stress-strain behavior of these samples is presented in Figures 38 and 39 for Stage I and Stage II samples, respectively. Figures D1 through D7 in Appendix D summarize results of the samples from each plant.

Effective stress paths from the triaxial compression tests performed on undisturbed samples are presented on Figure 40 for both Stage I and Stage II sludge. The effective stress paths for each plant are presented in Figure D8 through D10 in Appendix D. The failure envelope, or K_f-line, passes through the origin with a corresponding angle of internal friction ranging from 44.4-55.6° for Stage I sludge and from 44.4-56.9° for Stage II sludge.

Facility	Boring	Sample	Sample Depth	WC_{110}	S_{110}	LL (%)	PI	LI	Lal Str	b Vane Sho rength (lb/f	ear t ²)		
	INO.	INO.	(ft)	(70)	(70)	(70)	(70)		Peak	Remolded	St		
Stage I Sluc	lge Sample	es											
		US-1	1-3	200.5	33	92	46	3.4	31	12	2.6		
PLANT C	TH-1A	US-2	3-5	199.2	33	98	42	3.4	35	8	4.3		
		US-3	6-8	205.0	33	88	39	4.0	31	9	3.4		
DIANTR	ТН 1Λ	US-1	1-3	233.4	30	92	33	5.3	19	5	3.8		
I LANI D	111-1A	US-2	1-3	280.0	26	130	57	3.6	64	12	5.3		
	ТЦ 1 А	US-1	0-2	233.5	30	-	-	-	95	24	4.0		
DI ANT N	IП-1А	US-2	0-2	221.4	31	126	63	2.5	115	40	2.9		
FLANT IN	TII 1D	US 1	0-2	240.3	29	125	55	3.1	90	19	4.7		
	IП-ID	US 2	0-2	222.9	31	131	64	2.4	225	29	7.8		
Stage II Sludge Samples													
		US-1	1-3	357.6	22	336	221	1.1	173	13	13.3		
	TH-2C	US-2	4-6	304.9	25	297	189	1.0	280	28	10.0		
PLANT C		US-3	7-9	310.5	24	146	89	2.8	140	33	4.2		
(Pond 4B)		US-2	3-5	239.2	30	141	73	2.3	21	8	2.6		
	TH-2D	US-3	6-8	283.6	26	133	70	3.2	160	30	5.3		
		US-4	9-11	284.5	26	86	33	7.0	34	26	1.3		
	TH-2A	US-2	5-7	271.1	27	110	44	4.7	55	14	3.9		
PLANT B		US-1	2-4	349.5	22	134	68	4.2	98	12	8.2		
	1 п-2 р	US-2	5-7	415.0	19	121	52	6.7	16	2	8.0		
	TH-2A	US-2	2-4	270.5	27	-	-	-	12	8	1.5		
PLANT N		US-1	0-2	261.5	28	-	-	-	126	42	3.0		
	TH-2B	US-2	2-4	255.4	22	149	76	2.4	9	3	3.0		
Where: WC cont	$\begin{array}{c c c c c c c c c c c c c c c c c c c $												

 Table 39. Miniature Vane Shear Strength Tests on Undisturbed Sludge Samples.



Figure 38. Stress-Strain Behavior of Undisturbed Stage I Sludge Samples in Triaxial Compression.



Figure 39. Stress-Strain Behavior of Undisturbed Stage II Sludge Samples in Triaxial Compression.



Figure 40. Effective Stress Paths for Undisturbed Stage I and Stage II Sludge Samples.

Triaxial Compression Tests on Laboratory Sedimented Samples

Two consolidated-undrained **CIUC** triaxial compression tests were performed on laboratory prepared specimens of Plant N-Stage II sludge. The specimens were prepared by placing slurry into a 4-inch cylindrical device. The sample settled and then a small normal load was applied and gradually increased while allowing drainage until a void ratio of 5.4 was reached. The sample was removed from the cylinder and specimens were trimmed for triaxial compression testing. The two specimens were back-pressure saturated and isotropically consolidated in increments to effective consolidation stresses of 0.25 or 0.5 kg/cm^2 , respectively, and then sheared undrained at a constant axial strain rate of about 1%/hour. The measured stress-strain behaviors and effective stress paths for these specimens are presented on Figures D7 and D11 in Appendix D, respectively. Tables 40 and 41 include the \overline{CIUC} test data and results for the sedimented specimens. Results of the \overline{CIUC} triaxial compression tests performed on the laboratory sedimented specimens of Plant N-Stage II sludge yielded an effective friction angle on the order of 44-50° (see Figure D-11), in agreement with results obtained for undisturbed Stage II sludge test specimens. (At stresses in excess of 1 kg/cm², the failure envelope may potentially be slightly lower than shown in Figure D-11 due to a curvature of the envelope at increasing stresses.)

Shear Strength Implications

The shear strength of lime sludge is of interest for evaluating the requirements for soil cap placement atop lime sludge ponds during closure. At solids contents in the range of 20-30%, the shear strengths measured in miniature vane shear tests indicate that the sludge can be described as a very soft, very low strength material (i.e., with undrained shear strengths potentially less than 30 psf and no greater than about 200 psf). If means for increasing the solids content are provided (e.g. through dewatering, desiccation or staged loading) the sludge can potentially exhibit relatively high drained strengths as measured in undrained triaxial compression tests.

Although the undrained shear strength of lime sludge at initial settled solids content (i.e. just after deposition in a sludge pond) is quite low, at higher solids content the drained shear strength can be relatively high. During closure construction, sludge ponds will require dewatering and staged construction to allow controlled consolidation and shear strength increase prior to installation of a soil cap.

	Dening	C 1.	Sample	200	Init	ial Condit	ions	Pre-Sh	ear Condit	tions	Consolida	ation Data	
Site	Boring	Sample	Depth	-200	Wi	γ _{d,i}	$\mathbf{S}_{\mathbf{i}}$	W _{ps}	$\gamma_{d,ps}$	В	$\overline{\sigma}_{c}$	$\Delta V/V_o$	
	INO.	INU.	(ft)	(70)	(%)	(lb/ft^3)	(%)	(%)	(lb/ft^3)	(%)	(kg/cm^2)	(%)	
Stage I Lim	e Sludge - I	Undisturbe	ed Samples	5									
DI ANT D	TH-1A	US 1	1.5	84	138	36.0	98	124	39.6	99	0.50	-9.0	
PLANI D	TH-1A	US 2	1.5	99	266	20.8	100	220	24.7	99	0.25	-15.7	
DI ANT C	TH-1A	US 1	2.5	96	171	30.5	100	135	37.2	98	0.25	-18.0	
PLANIC	TH-1A	US 3	7.25	96	238	23.1	100	166	31.4	99	0.50	-26.5	
	TH-1A	US 2	1.0	67	210	25.7	100	197	27.1	99	0.25	-5.3	
PLANT N	TH-1B	US 1	1.25	90	236	22.8	98	202	26.5	99	0.25	-13.8	
	TH-1B	US 2	1.25	87	197	27.1	100	183	28.8	99	0.25	-6.1	
Stage II Lime Sludge - Undisturbed Samples													
PLANT B	TH-2A	US 2	6.0	88	251	22.0	100	227	23.9	100	0.25	-8.1	
	TH-2B	US 2	6.0	97	330	17.3	100	232	23.5	100	0.50	-26.4	
DI ANT C	TH-2C	US 1	1.25	24	452	12.8	100	263	21.0	100	0.50	-39.1	
FLANIC	TH-2C	US 2	5.75	72	324	17.5	100	275	20.2	99	0.50	-13.5	
	TH-2A	US 1	1.25	50	191	27.3	98	157	32.8	100	0.25	-16.7	
PLANT N	TH-2A	US 2	3.0	57	308	18.3	100	224	24.3	99	0.50	-24.5	
	TH-2B	US 2	3.5	97	271	20.5	100	202	26.5	98	0.50	-22.6	
Stage II Lin	ne Sludge -	Sedimente	ed Samples	s									
DI ANT N	Composit	e Sample	0 to 3	68	182	28.7	100	172	30.2	100	0.25	-5.0	
	From 7	TH-2A	0103	70	187	28.3	100	159	32.2	100	0.50	-12.1	
Where: -200	0 = percent	material b	y dry weig	ght passi	ng the U.	S. No. 20	0 sieve si	ze (<0.07	5 mm); w _i	= Initi	al water co	ntent; $\gamma_i =$	
Init	ial dry dens	sity; $S_i = In$	itial degre	e of satu	uration; v	$w_{\rm ps} = {\rm Pres}$	hear wate	er content	$\gamma_{d,ps} = Pr$	reshear	dry density	; B =	
Por	e pressure c	coefficient	; $\overline{\sigma}_{c} = \text{Isot}$	tropic ef	ffective co	onsolidati	on stress;	$\Delta V/V_{o}$	= Volumet	tric stra	ain (negativ	e values	
indi	icate compr	ression).		-							-		

 Table 40. Summary of Consolidated-Undrained Triaxial Compression Test Specimen Data.

	Boring	Sample	Sample		Stren	igth at $(\overline{\sigma_1}/\overline{\sigma_1})$	$\overline{\sigma_3}$) _{max}			Stre	ngth at $(\overline{\sigma_1} - \overline{\sigma_1})$	$\overline{\sigma_3}$) _{max}		
Site	No.	No.	Depth (ft)	ε _v (%)	م (deg)	$\overline{\mathbf{p}}$ (kg/cm ²)	q (kg/cm ²)	$A_{\rm f}$	ε _v (%)	(deg)	$\overline{\mathbf{p}}$ (kg/cm ²)	q (kg/cm ²)	$A_{\rm f}$	
Stage I Lime S	ludge – Und	isturbed Sam	ples											
PLANT B	TH-1A TH-1A	US 1 US 2	1.5 1.5	10.1 15.2	54.5 49.2	0.290 0.202	0.236 0.153	1.08 0.69	6.8 14.7	53.6 50.9	0.308 0.196	0.248 0.152	0.89 0.68	
PLANT C	TH-1A TH-1A	US 1 US 3	2.5 7.25	15.8 18.0	49.6 54.1	0.285 0.306	0.217 0.248	0.42 0.89	14.6 15.5	49.7 52.8	0.287 0.314	0.219 0.250	0.42 0.87	
PLANT N	TH-1A TH-1B TH-1B	US 2 US 1 US 2	1.0 1.25 1.25	15.6 15.4 15.3	50.3 58.2 58.6	0.290 0.300 0.287	0.223 0.255 0.245	0.41 0.40 0.42	10.9 15.4 5.4	49.8 58.2 56.1	0.296 0.300 0.311	0.226 0.255 0.258	0.40 0.40 0.38	
Stage II Lime Sludge – Undisturbed Samples														
PLANT B	TH-2A TH-2B	US 2 US 2	6.0 6.0	14.9 15.1	59.0 53.3	0.252 0.303	0.216 0.243	0.50 0.90	13.0 5.4	57.3 47.9	0.258 0.364	0.217 0.270	0.48 0.75	
PLANT C	TH-2C TH-2C	US 1 US 2	1.25 5.75	15.0 12.2	43.6 50.2	0.312 0.414	0.215 0.318	0.94 0.64	2.6 4.6	34.0 47.7	0.418 0.473	0.234 0.350	0.67 0.54	
PLANT N	TH-2A TH-2A TH-2B	US 1 US 2 US 2	1.25 3.0 3.5	14.8 11.9 12.1	53.9 51.7 47.7	0.234 0.340 0.381	0.189 0.267 0.282	0.54 0.80 0.71	14.8 3.1 2.7	53.9 45.1 41.2	0.234 0.425 0.457	0.189 0.301 0.301	0.54 0.62 0.57	
Stage II Lime S	Sludge – Rer	nolded Samp	oles											
PLANT N	ANT N Composite Sample 0 to 3 from TH-2A				50.5 46.2	0.332 0.658	0.256 0.475	0.34 0.33	7.8 15.6	49.7 44.6	0.358 0.703	0.273 0.494	0.30 0.30	
Where: $\overline{\sigma_1} = M$ stress difference	ajor principa e; A _f = Pore	al effective s pressure par	tress; $\overline{\sigma_3} = N$ rameter at fai	Minor princ lure; $\overline{\phi} = E$	ipal effect	ive stress; ε_v iction angle	= Vertical s assuming no	train; $\mathbf{\overline{p}} = A$ cohesion	Average ef	fective pri	ncipal stress	q = Half prices	incipal	

 Table 41. Summary of Consolidated-Undrained Triaxial Compression Test Results.

EVALUATION OF SLUDGE AGRONOMIC PROPERTIES

OVERVIEW OF TESTING PROGRAM

The suitability of utilizing sludge generated from two-stage (Stage I and II) and single-stage lime treatment of process water, as well as second stage treated effluent water (Stage II+), as an agronomic amendment in gypsum stack side slope final covers in lieu of dolomitic limestone was investigated.

The sludge samples and Stage II supernatants were mixed with both leached (i.e., pH>4) and unleached (i.e., pH<3) phosphogypsum from the corresponding sites at selected application rates to determine if an amended phosphogypsum target pH of 4.8 to 5.2 could be achieved. The pH, conductivity and moisture content of each of the mixtures were measured to determine a suitable application rate to achieve the target pH without introducing too high salt concentration to the gypsum that would limit plant growth.

The phosphogypsum amended with the selected application rates of dolomitic limestone, Stage II sludge and effluent, single-stage sludge and effluent, and Stage II supernatant were analyzed for pore fluid sulfate, total phosphorus, fluoride and ammonia concentrations. Total and effective porosity, hydraulic conductivity and sedimentation characteristics tests were also performed on selected amended gypsum samples.

LEACHED AND UNLEACHED PHOSPHOGYPSUM PROPERTIES

Construction of amended gypsum side slope covers on typical gypsum stacks involves amendment of both leached and unleached phosphogypsum. Unleached gypsum is usually encountered on the lower slopes where process water seepage saturates the gypsum. Unleached gypsum contains pore water having chemical composition similar to process water, and is characterized as having a pH less than 3. Gypsum near the surface of upper slopes where the phreatic water surface is well below the surface is commonly leached by rain water infiltration to some extent. Leached gypsum is conventionally characterized as having a pH greater than 4 and conductivity ranging from 2,000-2,200 µmhos/cm.

A series of laboratory screening tests were conducted on amended phosphogypsum specimens. Samples of phosphogypsum from three plants (i.e., Plants B, C and N) were used for amendment with selected lime-treatment materials.

Material Sampling and Characterization

Phosphogypsum samples were obtained from existing gypsum stacks at four Florida facilities in April 2001. Bulk samples were obtained from locations that, based on preliminary field extract pH measurements, were representative of leached gypsum having a

pH equal to slightly greater than 4, and representative of the lower pH unleached gypsum from several trial sampling areas.

Samples from three of the four sites (i.e., Plants B, C and N) were selected for use in the agronomic screening tests. Table 42 presents pH and conductivity measurements on the phosphogypsum samples used in the agronomic screening testing along with their moisture content characteristics.

Source	Por	e Water*		Mo	isture Content								
Site	ъU	Conductivity	AWC ₄₀	AWC ₂₀₀	ΔAWC_{200-40}	CaSO ₄ ·2H ₂ O							
Bite	рп	(µmhos/cm)	(%)	(%)	(%)	(%)							
Unleached	Gypsum												
Plant C	2.8	4,990 [A]	22.4	42.1	19.7	94							
Plant B	2.4 [A]	6,950 [A]	28.2	46.8	18.6	89							
Plant N	2.9	3,430	23.6	43.5	20.0	96							
Leached Gy	psum												
Plant C	4.8 5.1	2,270 [A]	13.1	32.0	18.9	90							
Plant B	4.2 [A]	2,235 [A]	6.5	25.9	19.4	93							
Plant N	4.9	2,240	7.6	23.6	16.0	77							

Table 42.	Characteristics	of Gypsum	Samples [†]	Used in Agrou	nomic Screening	P Tests.
	Character istics	or Gypsum	Sumples	Coca m rigi oi		

* Pore water extracted using de-ionized water at a dilution ratio of 2:1 relative to air-dried weight of solids.

[A] = Reported value is the average of two or more determinations.

 $AWC_{40} = Apparent Moisture Content at 40°C.$

AWC₂₀₀= Apparent Moisture Content at 200°C.

 $\Delta AWC_{200\text{-}40} = AWC_{200} - AWC_{40}.$

 $CaSO_4 \cdot 2H_2O =$ Percent dihydrate gypsum backfigured from apparent moisture contents at 40°C and 200°C.

The unleached gypsum samples exhibited a backfigured $CaSO_4 \cdot 2H_2O$ content ranging from 89-96% and the pH ranged from 2.4 to 2.9. The leached gypsum samples yielded $CaSO_4 \cdot 2H_2O$ concentrations ranging from 77-93% and a pH ranging from 4.2 to 4.9. Note that the pH of the leached gypsum samples from Plants C and N were already within the range of amended phosphogypsum target pH of 4.8 to 5.2.

Settling column tests were performed on unleached gypsum samples using the procedures and equipment described in the settling column tests performed on lime sludge (i.e., as described in the sludge engineering properties section). Summary of the settling characteristics of the gypsum samples used in the agronomic screening tests are presented in Table 43.

	Source		Process Water		Settling Column	Sample	Height	Dry Gypsum	Si	$\mathbf{S}_{\mathbf{f}}$		WC ₄₀	γdf	v _{si}
	Site	pН	Conductivity (µmhos/cm)	TDS (mg/l)	Diameter (cm)	Initial (cm)	Final (cm)	Solids (g)	(%)	(%)	e _f	(%)	(lb/ft ³)	(cm/min)
11	Unleached Gy	psum												
	PLANT C	2.7	11,400	19,300	10.42	27.0	7.5	663.0	24.8	65.1	1.25	53.7	64.6	0.53
-	PLANT B	1.9	28,000	41,500	10.42	27.0	8.6	658.5	24.6	59.4	1.59	68.4	56.1	0.90
	PLANT N	2.1	17,700	19,982	10.40	27.3	9.3	658.5	24.8	56.8	1.80	76.0	52.0	0.45
	Where: $S_i = Int$ $\gamma_{df} = Final dry$	itial solids density; v _s	content; $S_f = Fir$ _i = Initial settling	nal solids c g velocity.	content; $e_f = 1$	Final void	ratio; WC	40= Final wa	ter conte	nt (using	g a dryi	ng tempe	rature of 4	40°C);

Table 43. Sedimentation Characteristics of Gypsum Samples Used in Agronomic Screening Tests.

LIME-TREATMENT SLUDGES

Lime-treatment sludges and effluents were also obtained from Plants B, C and N for use in the agronomic screening tests. Characteristics of the sludges and treated effluents used in the agronomic screening tests are presented in Table 44.

					-	
Source		MC	Free L	ime (FL)	Por	e Water ¹
Source	Туре	(0())	% as	% as	μIJ	Conductivity
Site		(%)	CaO	CaCO ₃	рп	(µmhos/cm)
	Stage II+ Sludge	206	0.0	15.7	7 0	2 000
	(Pond 2D Field Sample)	390	8.8	(5.0)	7.8	5,990
Dlass C	Stage II+ Treated Effluent				10.4 †	$c a c o \dagger$
Plant C	(Pond 4A Surface Sample)	n/a	-	-	10.4	6,260
	Single-Stage Sludge	1.67		(1,0)	= =	2 000
	(Pond 5A Field Sample)	10/	-	(1.0)	5.5	5,990
	Stage II+ Sludge	222	5.0	10.5	7 0	2 1 4 0
	(Field Sample from TH-2B)	332	5.9	(7.7)	7.8	5,140
Dlant D	Stage II+ Treated Effluent	n /a			10.4 †	6 260 †
Flain D	(Stage II Pond Surface Sample) ²	II/a	-	-	10.4	0,200
	Single-Stage (pH 7.5) Sludge	400	0.2	0.5	7 0 ^{††}	o o o ††
	(From Laboratory Treatment)	400	0.5	(0.5)	7.0	8,820
	Stage I Sludge	222		(1.1)	57	6 200
Dlant N	(Field Sample from TH-1B)		-	(1.1)	5.7	0,290
Plant N	Stage II+ Sludge	265	22.7	40.6	11.5	1 960
	(Field Sample from TH-2B)	205	22.1	(20.0)	11.5	4,800

Table 44. Characteristics of Sludge and Treated Effluent Used in Agronomic Screening Tests.

 MC_{110} = Moisture content determined using a drying temperature of 110°C;

FL = Combined percent by dry weight of quicklime (CaO), hydrated lime (Ca(OH)₂), and limestone (CaCO₃) as determined by calibrated titration methods (Values in parentheses are calcite equivalent as CaCO₃ percent by dry weight determined by the CO₂ gas pressure method).

- ¹ Pore water extracted using de-ionized water at a dilution ratio of 2:1 relative to air-dried weight of solids (except as noted otherwise).
- ² Stage II effluent from Plant C (Pond 4A) used with leached gypsum from Plant B since no representative Stage II effluent was available from Plant B.
- ^b pH and conductivity of field surface water sample from Stage II sludge settling pond.
- ^{††} pH and conductivity measured on treated clarified effluent water (assumed essentially the same as pore water of settled sludge).

AGRONOMIC SCREENING TESTS

Lime sludge and treated effluent materials from the three selected facilities were mixed with selected amounts of leached and unleached phosphogypsum from the corresponding facility to evaluate suitable application rates to achieve a target amended gypsum pH in the range of 4.8 to 5.2. The conductivity and pH of each mixture was measured on an extract sample prepared using a 2:1 ratio of de-ionized water to air dried mixture by weight at 1 hour, 1 day, 2 days, 4 days and 8 days after initial mixing. The moisture content of each mixture was also measured in accordance with ASTM D 2216 using a drying temperature of 40°C. A total of 41 mixtures were prepared and tested as part of the agronomic screening tests.

Results from the agronomic screening tests are presented in Tables 45 and 46 for the leached and unleached phosphogypsum, respectively. Figures 41 and 42 depict the relationship between the mix pore water pH and Stage II sludge addition for the leached and unleached phosphogypsum, respectively. Test results indicate that the amended phosphogypsum target pH of 4.8 to 5.2 can be reached on leached phosphogypsum with an initial pH of 4 with the addition of less than 1% of Stage II sludge by dry weight (Figure 41). As shown on Figure 42, unleached phosphogypsum will require from about 1-10% of Stage II sludge by dry weight to achieve the amended phosphogypsum target pH depending on the amount of unreacted lime present in the sludge. For an *in situ* gypsum dry density of 75 pcf, these percentages correspond to the addition of about 8 to 80 tons per acre (dry weight basis) of Stage II sludge to amend the upper 6 inches of a gypsum stack slope. For a Stage II sludge solids content of 20%, and corresponding dry density of 15 pcf, it would be necessary to spread and then mix a layer of Stage II sludge about 0.3 to 3 inches thick into the upper 6 inches of the surface of gypsum stack slopes.

Results of the agronomic screening tests indicate that the amended phosphogypsum target pH of 4.8 to 5.2 may potentially be achieved on leached phosphogypsum with an initial pH of about 4 with the addition of about 5% of single-stage sludge by dry weight, provided the pH of the sludge is on the order of 7 or more and as long as the sludge contains some free lime. For an *in situ* gypsum dry density of 75 pcf, this percentage correspond to the addition of about 40 tons per acre (dry weight basis) of single-stage sludge to amend the upper 6 inches of a gypsum stack slope. For a single-stage sludge solids content of 33%, and corresponding dry density of 25 pcf, it would be necessary to spread and then mix a layer of single-stage sludge about 0.9 inches thick into the upper 6 inches of the surface of gypsum stack slopes. On the other hand, it appears that amendment of unleached phosphogypsum to the target pH of 4.8 to 5.2 with single-stage sludge may not be practical considering that additions in excess of 25% (dry weight basis) will likely be needed particularly if the free lime content of the sludge is low (as is characteristic of laboratory prepared samples).

	Gyps	sum Cl	haracteristics		Ar	nendme	ent Materia	al		Ad	ldition R	ate			Mix Por	e Wate	er ¹
Source	MC ₄₀	Р	ore Water ¹	T	MC110	Free	e Lime	Ро	ore Water ¹	% by	Wet Tons	Approx.	Mix MC ₄₀	1	Day After Mixing	1	Week After Mixing
Site	(%)	pН	Conductivity (µmhos/cm)	Туре	(%)	% as CaO	% as CaCO ₃	pН	Conductivity (µmhos/cm)	Dry Weight	Per Acre	(in)	(%)	pН	Conductivity (µmhos/cm)	pН	Conductivity (µmhos/cm)
										1	40.5	0.31	15.6	5.6	2,300	5.9	2,460
	12.4	4.0	2 270	Stage II	207	0.0	15.7	70	2 000	1	40.5	0.31	16.4	5.6	2,300	6.0	2,310
7)	12.4	4.8	2,270	Sludge	396	8.8	(5.0)	7.8	3,990	- 3 10	122	0.93	23.2 46.3	6.3 6.8	2,580	6./	2,680
1T C										16	403 648	4.97	64.5	0.8 7.1	3,840	0.) 7.1	4,000
PLAN	12.4	4.8	2,270	Stage II Effluent (Pond 4A)	n/a	-	-	10.4 †	6,260 [†]	1 10	8.2 82	0.07 0.72	13.2 22.3	5.0 5.4	2,290 2,470	5.1 5.4	2,330 2,450
	13.0	5.1	2,170	Single-Stage Sludge	167	- (1.0)		5.5	3,990	5	109	0.72	19.6	4.9	2,400	4.5	2,550
										1	35.2	0.26	8.5	5.3	2,290	5.6	2,370
	6.1	4.1	2,250	Stage II	332	5.9	10.5	7.8	3,140	5	176	1.32	(21.6)*	7.1	3,070	7.2	3,280
~				Sludge			(7.7)			10 20	352 705	2.65	33.6 57.7	7.5	2,950	7.3 7.6	3,210
PLANT I	6.0	4.4	2,220	Stage II Effluent (Pond 4A) ³	n/a	-	-	10.4 †	6,260 [†]	15 25	122 204	1.08 1.80	(21.1)* 30.8	4.9 5.2	2,370 2,640	4.7 5.0	2,440 2,580
	6.0	4.4	2,220	Single-Stage Sludge (Lab)	400	0.3	0.5 (0.5)	7.0 **	8,820 ††	5 15	207 622	1.56 4.68	(24.8)* 57.1	5.5 5.7	3,400 5,570	5.3 5.5	3,620 5,790
PLANT N	7.2	4.9	2,240	Stage II Sludge	265	22.7	40.6 (20.0)	11.5	4,860	0.5 1 5 10 20	14.9 29.8 149 298 595	0.11 0.22 1.08 2.17 4.34	13.1 9.4 19.1 30.3 47.2	6.5 6.9 8.6 9.7 11.3	2,220 2,210 2,510 2,520 2,710	6.5 6.9 8.1 9.0 11.3	2,330 2,320 2,770 3,000 3,420

Table 45. Summary of Agronomic Test Results for Leached Phosphogypsum.

NOTES: MC_{40} = Moisture content determined using a drying temperature of 40°C; MC_{110} = Moisture content determined using a drying temperature of 110°C; Free Lime =

Combined percent by dry weight of quicklime (CaO), hydrated lime (Ca(OH)₂), and limestone (CaCO₃) as determined by calibrated titration methods, reported as CaO

equivalent or CaCO₃ equivalent (Values in parentheses are calcite equivalent as CaCO₃ percent by dry weight determined by CO₂ gas pressure method).

Pore water extracted using de-ionized water at a dilution ratio of 2:1 relative to air-dried weight of solids (except as noted otherwise).

² Equivalent thickness to amend upper 6 inches of phosphogypsum having an assumed *in situ* dry density of 75 lb/ft³.

³ Stage II effluent from Plant C (Pond 4A) used with leached gypsum from Plant B since no representative Stage II effluent was available from Plant B.

pH and Conductivity of field surface water sample from Stage II sludge settling pond.

thpH and Conductivity measured on treated clarified effluent water (assumed essentially the same as pore water of settled sludge)

* Mix moisture content estimated based on measured component moisture contents and mix proportion

	Gyps	um Cl	naracteristics		Ar	nendme	ent Materia	al		Ad	ldition R	ate			Mix Pore	e Wate	er ¹
Source	MG	Po	ore Water ¹	Matarial	MG	Free	e Lime	Po	ore Water ¹	% By	Wet	Approx.	Mix MC40	1	Day After Mixing	1	Week After Mixing
Site	MC ₄₀ (%)	pН	Conductivity (µmhos/cm)	Туре	MC ₁₁₀ (%)	% as CaO	% as CaCO ₃	pН	Conductivity (µmhos/cm)	Dry Weight	Per Acre	Thick. ² (in)	(%)	pН	Conductivity (µmhos/cm)	pН	Conductivity (µmhos/cm)
	21.6	2.8	5,290	Stage II	396	8.8	15.7	7.8	3,990	1 3 10	40.5 122 405	0.31 0.93 3.10 2.10	27.9 33.2 59.5	3.7 4.1 4.6	3,120 4,790 5,180 2,750	3.6 4.0 4.6	3,940 4,940 5,250
NT C				Sludge			(3.0)			10 16 30	405 648 1215	3.10 4.97 9.31	56.5 74.6 114	4.8 5.5 6.2	6,050 6,780	4.7 5.8 6.5	4,640 6,280 7,020
PLA	21.6	2.8	5,290	Stage II Effluent	n/a	-	-	10.4 †	6,260 [†]	1 10	8.2 82	0.07 0.72	(22.6)* 33.6	2.7 2.6	5,040 5,470	2.6 2.7	5,210 5,450
23.	23.4	2.8	4,960	Single-Stage Sludge	167	-	(1.0)	5.5	3,990	20 35	435 762	2.89 5.06	50.0 (60.6)*	4.1 4.3	4,930 5,200	3.9 4.0	5,030 5,500
NT B	29.9	2.5	6,820	Stage II Sludge	332	5.9	10.5 (7.7)	7.8	3,140	1 5 10 20	35.2 176 352 705	0.26 1.32 2.65 5.29	35.5 44.8 57.5 80.6	3.4 4.8 5.7 6.4	6,010 5,970 6,280 6,580	3.3 4.6 5.5 6.8	6,300 6,340 6,720 6,940
УТd	33.6	2.4	7,080	Single-Stage Sludge (Lab)	400	0.2	0.3 (0.5)	7.0 ^{††}	8,820 ††	25	1021	7.81	108	5.1	8,180	4.6	8,180
PLANT N	23.2	2.9	3,430	Stage II Sludge	265	22.7	40.6 (20.0)	11.5	4,860	0.5 1 5 10 20	14.9 29.8 149 298 595	0.11 0.22 1.08 2.17 4.33	24.0 26.7 34.7 45.1 62.3	5.3 6.7 8.6 10.0 11.2	2,690 2,510 2,690 2,580 2,670	5.2 6.8 8.3 9.5 10.9	2,890 2,720 2,910 2,930 3,190

Table 46. Summary of Agronomic Screening Test Results for Unleached Phosphogypsum.

NOTES: MC_{40} = Moisture content determined using a drying temperature of 40° C; MC_{110} = Moisture content determined using a drying temperature of 110° C; Free Lime = Combined percent by dry weight of quicklime (CaO), hydrated lime (Ca(OH)₂), and limestone (CaCO₃) as determined by calibrated titration methods, reported as CaO equivalent or CaCO₃ equivalent (Values in parentheses are calcite equivalent as CaCO₃ percent by dry weight determined by CO₂ gas pressure method)

¹Pore water extracted using de-ionized water at a dilution ratio of 2:1 relative to air-dried weight of solids (except as noted otherwise)

² Equivalent thickness to amend upper 6 inches of phosphogypsum having an assumed *in situ* dry density of 75 lb/ft^3

pH and Conductivity of field surface water sample from Stage II sludge settling pond

[†]pH and Conductivity measured on treated clarified effluent water (assumed essentially the same as pore water of settled sludge)

*Mix moisture content estimated based on measured component moisture contents and mix proportion.

The agronomic screening test results also indicate that Stage II effluent may not be practically used to amend unleached phosphogypsum to the target pH of 4.8 to 5.2. The Stage II effluents did not display enough buffering capacity to neutralize the acidity in unleached phosphogypsum. It appears, however, that Stage II effluent may be used to sweeten leached phosphogypsum. Test results indicate that a pH 4 leached phosphogypsum would likely require about 40,000 to 50,000 gallons per acre of Stage II supernatant to amend the upper 6 inches of a gypsum stack slope.



SLUDGE ADDITION (DRY TONS/ACRE)

SLUDGE ADDITION (% By Dry Weight)

			GYPSUM				LIME SLU	DGE	
SYMBOL	SOURCE	рН	Conductivity (µmhos/cm)	MC ₄₀ (%)	TYPE	pН	Conductivity (µmhos/cm)	MC (%)	Free Lime (% as CaO)
•	PLANT B	4.1	2,250	6	Stage II	7.8	3,140	332	5.9
	PLANT C	4.8	2,270	12	Stage II	7.8	3,990	396	8.8
	PLANT N	4.9	2,240	7	Stage II	11.5	4,860	265	22.7

Figure 41. pH of Leached Gypsum Versus Stage II Sludge Addition.



STAGE II SLUDGE ADDITION (DRY TONS/ACRE)

			GYPSUM				LIME SLUE	DGE	
SYMBOL	SOURCE	рΗ	Conductivity (µmhos/cm)	MC 40 (%)	TYPE	рН	Conductivity (µmhos/cm)	MC (%)	Free Lime (% as CaO)
•	PLANT B	2.5	6,820	30	Stage II	7.8	3,140	332	5.9
A	PLANT C	2.8	5,290	22	Stage II	7.8	3,990	396	8.8
	PLANT N	2.9	3,430	23	Stage II	11.5	4,860	265	22.7

Figure 42. pH of Unleached Gypsum Versus Stage II Sludge Addition.

AMENDED PHOSPHOGYPSUM GROWTH MEDIA

Based on results of the agronomic screening tests, viable mixtures were selected for preparation of amended phosphogypsum growth media. Combinations of leached and unleached phosphogypsum and dolomitic limestone, single-stage and Stage II sludges, and

single-stage and Stage II effluents were prepared for use in greenhouse plant growth studies to evaluate the ability of the mixtures to support the growth of turfgrass species.

Samples of process water, Stage II sludge and lime-treatment effluents were obtained from Plants B, C and N for use in preparation of the amended phosphogypsum growth media mixtures. Laboratory lime neutralization was utilized to produce simulated single-stage treatment sludge and effluent.

Characteristics of the amended phosphogypsum growth media mixtures are presented in Table 47. As indicated in Table 47 the prepared growth media were analyzed for pH, conductivity, sulfate (SO₄), total phosphorus (total P), fluoride (F), ammonia (NH₃), and total Kjeldahl Nitrogen (TKN) concentrations.

Dolomitic limestone used in preparation of amended phosphogypsum growing media AG-1 (using leached phosphogypsum) and AG-2 (using unleached phosphogypsum) consisted of 'Soil Doctor' brand dolomitic limestone. This product is a ground dolomitic limestone with 60% of the material (dry weight basis) consisting of coarse to fine sand-size particles and 40% of the material finer than the U.S. Standard No. 200 sieve. The dolomitic limestone addition rates needed to increase pH of the leached and unleached gypsum were selected based on results of screening tests previously performed by Ardaman in conjunction with FIPR Publication 03-126-212. Leached and unleached phosphogypsum from Plant B were selected for dolomitic limestone amendment considering that these materials exhibited the lowest pore water pH of the plant source sites sampled. The selected dolomitic limestone addition rates were 2.5% and 0.25% by dry weight for unleached and leached phosphogypsum, respectively. For an *in situ* gypsum dry density of 75 pcf, these percentages correspond to the addition of about 20 and 2 tons per acre (dry weight basis) of dolomitic limestone to amend the upper 6 inches of an unleached and leached gypsum stack slope, respectively.

As indicated in Table 47, the pH of the pore fluid of the amended gypsum growth media were in general compliance with the target pH (4.8 to 5.2). Conductivity of the pore water of the amended leached gypsum was about twice the Class III surface water standard of 1275 μ mhos/cm, while the conductivity of the pore water of the amended unleached gypsum was about two- to four-fold the Class III surface water standard, consistent with pore water conductivity of gypsum amended with dolomitic limestone.

	Gyps	sum Ch	aracteristics		A	mendm	ent Material					Mix I	Pore Water	.1		
Source		Po	ore Water ¹	Growth		Po	ore Water ¹	Addition	Mix							
Site	MC ₄₀ (%)	рН	Conductivity (µmhos/cm)	Media I.D.	Material Type	рН	Conductivity (µmhos/cm)	Rate by Dry Weight (%)	MC ₄₀ (%)	pН	Conductivity (µmhos/cm)	F (mg/l)	Total P (mg/l)	SO ₄ (mg/l)	NH3 (mg/l)	TKN (mg/l)
Leached Gy	ypsum															
				AG-1	Dolomitic Limestone	-	-	0.25	11.7	4.74	2,790	7.3	110	1,582	17.2	17.5
DIANTR	6.0	4.1	2 250	AG-3A	Stage II Sludge	7.8	3,140	1.0	12.6	5.83	2,480	7.7	1.5	1,869	0.41	0.84
	4.1	2,230	AG-4A	Single-Stage Sludge [Lab]	7.1	8,900	2.5	11.9	4.83	2,860	9.4	73.6	1,869	29.0	29.3	
			AG-7	Single-Stage Effluent [Lab]	7.1	8,900	20.0	26.5	4.78	3,190	8.1	49.8	2,665	44.1	44.5	
				AG-3B	Stage II Sludge	11.5	4,860	0.5	9.5	6.51	2,380	7.3	2.3	1,901	0.02	1.06
PLANT N	7.2	4.9	2,240	AG-4B	Single-Stage Sludge [Lab]	7.1	7,320	2.0	14.9	6.40	2,630	10.1	20.4	1,710	6.48	6.83
				AG-6	Stage II Effluent	10.4	6,730	1.0	8.5	5.31	2,370	10.0	13.3	1,678	0.43	1.21
Unleached	Gypsum															
PI ANT B	20.0	2.5	6 820	AG-2	Dolomitic Limestone	-	-	2.5	21.8	4.86	4,780	70.3	666	2,919	82.7	83.8
I LANI D	29.9	2.5	0,820	AG-5A	Stage II Sludge	7.8	3,140	6.0	52.3	4.95	4,240	67.5	316	3,238	56.5	57.3
PLANT N	23.2	2.9	3,430	AG-5B	Stage II Sludge	11.5	4,860	1.0	25.6	6.10	2,790	8.9	2.4	2,155	11.6	11.6
PLANT C	21.6	2.8	5,290	AG-5C	Stage II Sludge	7.8	3,990	10.0	47.4	5.58	3,930	39.7	4.9	3,110	45.5	45.6
NOTES:	$MC_{40} =$ ¹ Pore w	Moistu vater ext	re content deter tracted using de	mined usi -ionized v	ng a drying temp vater at a dilution	erature 1 ratio (of 40°C.	o air-dried	weight of	solids (except as noted	otherwis	se).			

Table 47. Characteristics of Amended Phosphogypsum Greenhouse Growing Media.
Characterization of the amended phosphogypsum growth media also included total and effective porosity testing in general accordance with ASTM D 2325. Results of these tests are presented in Table 48. As shown, the measured effective porosity of phosphogypsum amended with Stage II sludge is lower than values measured on the corresponding phosphogypsum without amendment, indicating that the addition of Stage II sludge will likely reduce to some extent the theoretical volume of water that could drain from the gypsum pores under a suction of 1/3 atmosphere.

C		Stage II Sludge	Initial Co	onditions	F Con	inal ditions	Porosity				
Media I.D. Source Facility		Addition Rate by Dry Weight (%)	Wi (%)	γ_{di} (lb/ft ³)	w _i (%)	$\gamma_{\rm df}$ (lb/ft ³)	Total, n	Effective, n _e			
AG-5B	Plant N	0 1.0	22.5 23.9	75.4 77.7	8.4 20.2	78.5 77.4	0.46 0.43	0.35 0.16			
AG-5A	Plant B	0 6.0	11.5 31.9	75.4 76.5	4.5 13.3	76.7 78.1	0.47 0.46	0.42 0.30			
AG-5C	Plant C	0 10.0	16.2 23.6	74.8 75.8	7.8 19.2	83.7 77.4	0.42 0.47	0.32 0.23			
Where: $\gamma_{df} = Fina$	Where: w_i = Initial moisture content; γ_i = Initial dry density; w_f = Final moisture content; γ_{df} = Final dry density										

Table 48. Summary of Effective Porosity Test Results for PhosphogypsumLime Sludge Mixtures.

Hydraulic conductivity tests were also performed on remolded unleached gypsum and amended gypsum specimens in general accordance with ASTM D 5084. Results of these hydraulic conductivity tests are presented in Table 49. Amended phosphogypsum specimens were prepared by adding 1%, 6% and 10% sludge (dry weight basis) to unleached phosphogypsum from Plants N, B and C, respectively. As expected, the hydraulic conductivity of the phosphogypsum specimens decreases with sludge addition.

	Percent		Moldi	ng Cono	ditions		, ج		Average		Final Conditions				kao	200
Site	Sludge Added	L (cm)	D (cm)	W_c	$\gamma_{\rm d}$ (1b/ft ³)	e	σ_c (lb/in ²)	(lb/in^2)	Hydraulic Gradient	W_c	$\gamma_{\rm d}$ (1b/ft ³)	e	$\Delta V/V_o$	S	(cm/sec)	-200 (%)
Unleached	Gypsum	(CIII)	(CIII)	(70)	(10/11)				Gradient	(70)	(10/11)		(70)	(70)		L
PLANT N	0	7.61	3.56	24.1	79.6	0.83	3.0	97.0	13.4	28.3	87.6	0.66	-9.1	100	7.2x10 ⁻⁵	83
PLANT B	0	7.10	3.58	12.0	80.5	0.81	3.0	97.0	2.3	32.0	83.3	0.74	-3.4	100	4.0×10^{-4}	50
PLANT C	0	7.65	3.56	16.6	79.6	0.83	3.0	97.0	13.6	26.0	90.7	0.60	-12.2	100	6.0x10 ⁻⁵	87
Gypsum-Li	me Sludge	Mixture	s													
PLANT N	1	6.90	3.72	25.1	87.1	0.67	3.0	97.0	12.4	23.8	93.6	0.55	-6.9	100	1.2×10^{-5}	84
PLANT B	6	7.52	3.58	31.0	80.3	0.81	3.0	97.0	11.0	31.2	84.2	0.73	-4.7	100	2.6×10^{-5}	46
PLANT C	10	7.45	3.58	23.1	93.1	0.56	3.0	97.0	11.7	21.8	96.3	0.51	-3.3	100	2.6x10 ⁻⁷	79
Where: L= Specimen length; D= Specimen diameter; w_c = Moisture content; γ_d = Dry density; e = Void ratio; σ'_c = Average isotropic effective confining																
stress; $u_b =$	stress; $u_b = Back$ -pressure; $\Delta V/V_o = Volume$ change from initial to final condition (negative values denote consolidation); S = Calculated degree of															
saturation;k	$a_{20} = Satura$	ated hydr	aulic co	nductivi	ty at 20°C	; -200 =	= Fines con	tent.								

Table 49. Summary of Constant Head Flexible-Wall Hydraulic Conductivity Test Results for Phosphogypsum-Lime Sludge Mixtures.

GREENHOUSE PLANT GROWTH STUDIES USING IRRIGATION FLUIDS DERIVED FROM TREATMENT OF PROCESS WATER

The primary objective of the greenhouse plant growth and irrigation studies was to determine whether various turfgrass species can be successfully grown on leached and unleached phosphogypsum amended with dolomitic limestone and different-type process water treatment sludges when irrigated with fluids derived from process water treatment. In particular, the study focused on evaluating the extent to which the inorganic constituents and nutrients retained in treated process water can impact sustained health of grass vegetation.

OVERVIEW OF GREENHOUSE STUDY PLAN

Leached and unleached gypsum were obtained from three different phosphogypsum plants (i.e., Plants B, C and N) for use in preparing samples for the greenhouse plant growth and irrigation studies. Amended phosphogypsum growth media were prepared as described in the evaluation of sludge agronomic properties section. Turfgrass species used in the greenhouse studies were bermudagrass, bahiagrass and seashore paspalum. Irrigation fluids consisted of fresh water, diluted and undiluted Stage I, single-stage, Stage II and Stage II+ effluents from process water treatment. A total of 49 growth media/irrigation fluid combinations were studied under greenhouse controlled conditions.

The plant growth tests were undertaken in 2002 at the greenhouse facilities of: (i) the University of Florida (UF) in Gainesville (Gainesville study) under the direction of Dr. Laurie E. Trenholm, Urban Turfgrass Specialist; and (ii) the UF West Florida Research and Education Center in Milton, Florida (Milton study) under the direction of Dr. J. Bryan Unruh, Extension Turfgrass Specialist.

The Gainesville study focused on determining whether bermudagrass can be successfully grown and maintained on leached and unleached gypsum amended with dolomitic limestone when irrigated with diluted and undiluted effluent waters derived from process water treatment. The Gainesville study also evaluated the performance of bermudagrass, bahiagrass and Paspalum grass on Arredondo sand used as growth media when irrigated with diluted and undiluted effluent waters derived from process water treatment for comparison purposes with the amended gypsum growth media plots.

The Milton study focused on establishing whether bermudagrass can be successfully grown and maintained on gypsum amended with different type of process water treatment sludges used as growth media when irrigated with fresh water and undiluted Stage II effluents.

IRRIGATION TEST FLUID PREPARATION

A total of four irrigation fluid types derived from the process water treatment of Plants B and C obtained in early 2002 were prepared for use in the greenhouse plant growth study. In addition, control irrigation fluids consisting of fresh water from a deep supply well (i.e., for Milton study) and tap water (i.e., for Gainesville study) were used.

Irrigation Fluid Types

<u>Irrigation Fluids IF-1g and IF-1m</u>: are fresh water control fluids (i.e., tap water and well water from the greenhouse facilities in Gainesville and Milton, respectively). These tap and well waters were used during the greenhouse germination and establishment periods for the corresponding test pots.

<u>Irrigation Fluid IF-2</u>: is effluent from the operating double-lime treatment system at Plant C post-aeration and acidulation to a target pH of about 7.5. The resulting neutralized "Stage II+" effluent was remixed and homogenized prior to final preparation of diluted and undiluted irrigation test fluids.

<u>Irrigation Fluid IF-3</u>: is an effluent from laboratory double-lime treatment to a neutral pH of about 7.5, which induces precipitation of most available phosphorous but does not achieve a pH level adequate for air-stripping ammonia. This "Stage II" fluid was prepared by adding sufficient laboratory-hydrated lime to Stage I effluent obtained from Plant C. Upon completion of 5-gallon laboratory batch neutralizations, a composite of the effluents was remixed and homogenized prior to final preparation of diluted and undiluted irrigation test fluids.

<u>Irrigation Fluid IF-4</u>: is an effluent from laboratory single-stage lime treatment of process water to a neutral pH of about 7.5. This "single-stage" fluid was prepared by gradually adding laboratory-hydrated lime to process water samples obtained from Plant B. Upon completion of 5-gallon laboratory batch neutralizations, a composite of the effluents was remixed and homogenized prior to final packaging for shipment to the greenhouse.

Irrigation Fluid IF-5: corresponds to "Stage I" effluent obtained from Plant C.

Laboratory Dilution

Irrigation fluids were prepared for use in the greenhouse studies in both diluted and undiluted forms. Ardaman Orlando laboratory tap water was used for diluting the test fluids. The diluted fluids were included in the greenhouse studies to simulate field "dilution" that would occur as a result of rainfall to an extent depending of the spray irrigation rate. Dilution ratios of 3:1 and 10:1 were used for double-lime treated effluent fluids (IF-2 and IF-3) with the intent of producing irrigation fluids that: (i) marginally comply with the primary

MCL standard of 160 mg/l for sodium; and (ii) marginally comply with the Class III surface water standard for conductivity.

Undiluted neutralized Stage II+ treated water was used as a test irrigation fluid (IF-2d0), and two diluted Stage II+ irrigation fluids were prepared using tap water at 3:1 and 10:1 dilution ratios (IF-2d1 and IF-2d2, respectively). Similarly, undiluted neutralized Stage II treated water was used as a test irrigation fluid (IF-3d0), and two diluted Stage II irrigation fluids were prepared using tap water at 3:1 and 10:1 dilution ratios (IF-3d1 and IF-3d2, respectively).

Irrigation Fluid Characterization

Upon completion of laboratory preparation of undiluted and diluted irrigation fluids, a representative sample of each test fluid was sent to Pembroke and ENCO laboratories for chemical analyses. The chemical composition of the undiluted irrigation fluid sources is presented in Table 50. Results of chemical analyses performed on representative samples of the undiluted and diluted effluent source fluids used in this study are summarized in Table 51. Notable trends in the analytical results for the lime-effluent fluids include the following:

- Measured pH values for all the irrigation fluids used in the greenhouse study ranged from 6.9 to 8.1, generally within the range for compliance with Class III surface water standards (6.5 to 8.0), with the exception of Stage I undiluted irrigation fluid (IF-5) which exhibited a lower pH value of 4.8.
- The conductivity of the four undiluted lime treated effluent fluids (IF-2d0, IF-3d0, IF-4 and IF-5) remained elevated at levels ranging from about 4,500 to 9,600 µmhos/cm (about 4 to 7 times the Class III Standard of 1,275 µmhos/cm). The sodium and sulfate concentrations for these undiluted lime-treated fluids remained elevated.
- Fluoride was present in the undiluted Stage II (pH7.5; IF-3d0), undiluted singlestage (pH 7.5; IF-4) and undiluted Stage I (pH 5; IF-5) effluents at levels ranging from about 24 to 54 mg/l, whereas the fluoride concentration in the undiluted Stage II+ effluent (which had its pH raised to near 11 in two stages) declined to about 10 mg/l.
- Total phosphorus concentrations in the undiluted single-stage treated effluent (pH 7.5; IF-4) and undiluted Stage I (pH 5; IF-5) effluents remained elevated at levels ranging from 1,100 to 1,800 mg/l. It was estimated that the phosphorus concentration in the undiluted Stage II+ effluent (which had its pH raised to near 11 for air-stripping prior to final neutralization) was about 4.3 mg/l.
- Ammonia nitrogen concentrations in the undiluted Stage II (pH7.5; IF-3d0), undiluted single-stage treated effluent (IF-4) and undiluted Stage I (IF-5) effluents remained elevated at levels ranging from 170 mg/l to 590 mg/l, whereas the undiluted Stage II+ (air-stripped) effluent (IF-2d0) contained about 10 mg/l ammonia nitrogen.

• Measured constituent concentrations and conductivity of the diluted irrigation fluids are generally consistent with expected values considering the reported parameter values for the source and dilution fluid. It should be noted that the conductivity and sodium concentration of the 10:1 diluted Stage II+ and Stage II (pH 7.5) fluids comply with the applicable Class III surface water and groundwater standard MCLs, respectively.

As expected, the fresh water irrigation fluids (IF-1g and IF-1m) and diluting fluid (Ardaman tap water), exhibited very low constituent concentrations, and hence low conductivities, when compared even to highly diluted lime treated effluents.

	Fresh	Water	C II.	C/ II	Single	C/ I
			Stage II+	Stage II	Stage	Stage I
			(pH 7.5)	(pH 7.5)	(pH 7.5)	(pH 5)
Parameter	Gainesville	Milton	Effluent	Effluent	Effluent	Effluent
	(Tap)	(Well)	Plant C	Plant C	Plant B	Plant C
			(Field)	(Field/Lab)	(Lab)	(Field)
Irrigation Fluid I.D.	IF-1g	IF-1m	IF-2	IF-3	IF-4	IF-5
INITIAL PARAMETERS						
nH		74	69	71	71	48
Conductivity (umhos/cm)		107	4.510	5.450	9.640	6.950
LABORATORY ANALYSES			.,	-,	,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,	
Sample/Prep. Date	8/17/02	8/26/02	2/22/02	3/01/02	3/01/02	2/22/02
Testing Lab	Pembroke	Pembroke	ENCO	ENCO	ENCO	ENCO
Maior Constituents (mg/l)						
Calcium. Ca	-	-	11	24	29	1.000
Magnesium, Mg	-	-	4.1	110	52	240
Potassium, K	-	-	180	220	350	220
Sodium, Na	9.4	1.9	1.200	1.200	2.300	1.300
Chloride, Cl	40	6.6	48	46	420	46
Fluoride, F	0.9	0.1 [U]	9.6	24	54	49
Sulfate, SO_4	81	1 [Ū]	1,900	2,200	3,800	2,300
Total Phosphorus, P	0.2	0.1 [U]	4.3	1,500	1,100	1,800
Nitrogen, as N				,	,	,
Total	0.05 [U]	1.48	13	230	750	220
Ammonia	0.05 [U]	0.05 [U]	10	170	590	210
Total Kjeldahl (TKN)	0.05 [U]	1.48	11	230	740	220
$NO_3 \& NO_3$	-	-	0.7	0.4	17	0.2
Alkalinity as CaCO ₃	-	-	75	510	920	53
Acidity as CaCO ₃	-	-	2 [U]	450	470	3,800
Solids, Total Dissolved	244	111	2,200	4,100	5,700	8,800
Solids, Total Suspended	-	-	38	56	340	80
Metals (µg/l)						
Aluminum, Al	-	-	440	150	800	240
Antimony, Sb	-	-	5 [U]	-	-	5 [U]
Arsenic, As	-	-	31	390	2,100 [R]	400
Barium, Ba	-	-	100 [U]	100 [U]	100 [U]	100 [U]
Beryllium, Be	-	-	1 [U]	1 [U]	1 [U]	3.4
Cadmium, Cd	-	-	1.0	4.1	4.0	30
Chromium, Cr	-	-	10 [U]	32	39	91
Copper, Cu	-	-	1 [U]	5.4	13	1.7
Iron, Fe	-	-	160	360	290	560
Lead, Pb	-	-	5 [U]	5 [U]	5 [U]	5 [U]
Manganese, Mn	-	-	10 [U]	1,400	95	5,600
Mercury, Hg	-	-	0.5 [U]	-	-	0.5 [U]
Nickel, Ni	-	-	30	410	290	780
Selenium, Se	-	-	10[U]	11	27	18
Silver, Ag	-	-	0.5 [U]	-	-	0.5 [U]
Thallium, TI	-	-	6.0	7.2	31	11
Zinc, Zn	-	-	100 [U]	100 [U]	100 [U]	180
Other Parameters						1.0
Lab pH	8.94	7.41	6.8	6.8	7.0	4.9
Lab Conductivity (μ mhos/cm)	346	104	4,300	4,600	8,600	6,600
Color (Pt/Co Units)	-	-	40	-	-	60
Turbidity (NTU)	-	-	10	4.0	6.0	8.0
NOTE: [U] Material was analyzed	1 for but not de	tected. (The re	eported value	is the minimum	detection lim	nt)
[K] Result confirmed by r	erun analysis					

 Table 50. Chemical Composition of Undiluted Irrigation Fluid Source Materials.

		Fresh Water								Single-	
Parameter	Dilutin g Fluid (A&A Tap)	Gainesville (Tap)	Milton (Well)	Stage II (Fie and	+ (pH 7.5 Plant C ld Sample Aeration d Acidulat) Effluent Post- ion)	Stage I (Stag Lab-T	Stage II (pH 7.5) Effluent Plant C (Stage I Field Sample Lab-Treated to pH 7.5)		Stage (pH 7.5) Effluent Plant B (Lab)	Stage I (pH 5) Effluent Plant C (Field)
IRRIGATION FLUID I.D.	n/a	IF-1g	IF-1m	IF-2d0	IF-2d1	IF-2d	IF-3d0	IF-3d1	IF-3d2	IF-4	IF-5
DILUTION Dilution Water Dilution Ratio	n/a	n/a	n/a	None	A&A Tap 3:1	A&A Tap 10:1	None	A&A Tap 3:1	A&A Tap 10:1	None	None
INITIAL PARAMETERS pH Conductivity (µmhos/cm)	7.9 333		7.4 107	6.9 4,510	8.0 1,545	8.1 782	7.1 5,450	7.1 1,929	7.5 956	7.1 9,640	4.8 6,950
LABORATORY ANALYSES Sample / Preparation Date Testing Lab Lab pH Lab Conductivity (µmhos/cm)	7/10/02 P-Lab 8.02 300	8/17/02 P-Lab 8.94 346	8/26/0 2 P-Lab 7.41 104	2/22/0 2 ENCO 6.8 4,300	3/07/02 P-Lab 8.28 1,412	3/07/02 P-Lab 8.32 716	3/01/0 2 ENCO 6.8 (6,500)	3/07/02 P-Lab 7.09 1,802	3/07/02 P-Lab 7.40 886	3/01/02 ENCO 7.0 8,600	2/22/02 ENCO 4.9 6,600
Major Constituents (mg/l) Sodium, Na Chloride, Cl Fluoride, F Sulfate, SO ₄ Total Phosphorus, P Nitrogen, as N Ammonia Total Kjeldahl (TKN) Total Nitrogen Total Dissolved Solids	12.7 17 0.9 5.1 0.1 [U] 0.12 2.25 2.31 201	9.4 40 0.9 81 0.2 0.05 [U] 0.05 [U] 0.05 [U] 244	1.9 6.6 0.1 [U] 1 [U] 0.1 [U] 0.05 [U] 1.48 1.48 111	(800) 48 9.6 (2,600) (10) 10 11 13 (4,000)	207 26 3.1 644 2.4 0.12 2.7 6.02 1,127	82.2 23 1.9 246 0.9 0.09 2.25 3.44 579	(800) (70) 24 2,200 (600) 170 230 230 (5,500)	202 29 7.8 592 145 41.5 43.6 50.4 1,532	90 22 (3.0) 234 56.5 17 17.8 20.3 971	2,300 420 54 3,800 1,100 590 740 750 5,700	1,300 46 49 2,300 1,800 210 220 220 8,800
NOTES: () Values in parentheses are estimated values.											

Table 51. Chemical Composition of Diluted and Undiluted Irrigation Fluids.

[U] - Constituent was analyzed for but not detected (the reported value is the minimum detection limit). A & A = Ardaman and Associates Inc.; P-Lab = Pembroke Laboratories Inc.; ENCO = Environmental Conservation Laboratories, Inc.

GAINESVILLE GREENHOUSE STUDY

Experimental Study Plan

A total of 30 growth media/irrigation fluid combinations, each of which replicated three times, were used in the Gainesville greenhouse study. The combinations of grass type, growth media and irrigation fluids used in the Gainesville study are summarized in Table 52. As shown, leached and unleached gypsum from Plant B amended with dolomitic limestone were selected as media to grow bermudagrass. In addition, one plot of unleached gypsum amended with dolomitic limestone was seeded with seashore paspalum. Bermuda, bahia and seashore paspalum grass were also seeded on growth control plots made using Arredondo sand (fine sand with silt, SP-SM). The selected undiluted and diluted irrigation fluids are indicated in Table 52.

Seeding and Grass Establishment

Seeding of the growth media began on March 12, 2002. Bermudagrass (*Cynodon dactylon* L.) cultivar 'Sahara' and bahiagrass (*Paspalum notatum* Flugge) cultivar 'Pensacola' were seeded into 6-inch diameter pots at a rate of 3 lb/1000 ft² (i.e., 0.27 g/pot, which corresponds to a land application rate of 130 pounds per acre) and 8 lb/1000 ft² (i.e., 0.82 g/pot, which corresponds to a land application rate of about 350 pounds per acre), respectively. On March 14, 2002, seashore paspalum (*Paspalum vaginatum* Swartz) cultivar 'Sea Isle 1' was sprigged into 6-inch diameter pots at a volumetric rate of 60 mls of sprig material per pot (i.e., about 17 cubic yards per acre). The sprigs were lightly top dressed to prevent desiccation.

During an establishment period of 30 days, the pots were kept well-irrigated with tap water. Thirty days after planting (30 DAP) all pots received fertilization including nitrogen at an application rate of about 22 pounds per acre.

Irrigation

Irrigation treatment started 38 days after planting (38 DAP) when grasses were uniformly established in the pots. Each pot was irrigated at a rate of 100 to 150 ml per day, 5 to 6 days per week as needed to balance with the evapotranspiration rate in the greenhouse. Higher irrigation rates were needed to balance greenhouse evapotranspiration for grasses grown in Arredondo Sand due to its lower water holding capacity relative to amended gypsum.

Clipping and Terminal Harvest

Grass in each pot was trimmed to a height of two inches at 30, 38, 45 and 74 days after planting. The clippings from each harvest were dried and weighed. Irrigation was stopped at 96 days after planting. Terminal harvest was conducted at 97 days after planting where shoots and roots were collected, dried, and weighed.

Table 52.	Summary of Grass Type, Growth Media and Irrigation Fluid Combinations
	Used in Gainesville Greenhouse Study.

			Gro	wth Media		Irrigation Fluid			
Pot	Grass		Amendm	nent Material	Gungum				
Nos.	Туре	I.D.	Material Type	Addition Rate by Dry Weight (%)	Source Site	I.D.	Dilution	Description	
Leached Phospho	ogypsum								
101/201/301						IF-1g	0	Tap Water	
103/203/303			itic			IF-2d0	0	Stage II+	
105/205/305	Bermuda	AG-1	lom nest	0.25	Plant B	IF-2d1	3:1	Stage II+	
107/207/307			Dol			IF-3d0	0	Stage II (pH 7.5)	
111/211/311						IF-4	0	Single-Stage (pH 7.5)	
Unleached Phosp	hogypsum								
102/202/302						IF-1g	0	Tap Water	
104/204/304			e			IF-2d0	0	Stage II+	
106/206/306			ston		Plant B	IF-2d1	3:1	Stage II+	
108/208/308	Bermuda		me			IF-3d0	0		
109/209/309		AG-2	c Li	2.5		IF-3d1	3:1	Stage II (pH 7.5)	
110/210/310			Jolomitic			IF-3d2	10:1		
112/212/312						IF-4	0	Single-Stage (pH 7.5)	
127/227/327	Seashore Paspalum		D			IF-4	0	Single-Stage (pH 7.5)	
Control Pots in S	and								
113/213/313						IF-1g	0	Tap Water	
114/214/314						IF-2d0	0	C. II.	
115/215/315						IF-2d1	3:1	Stage II+	
116/216/316	Bermuda					IF-3d0	0		
117/217/317						IF-3d1	3:1	Stage II (pH 7.5)	
118/218/318						IF-3d2	10:1		
119/219/319						IF-4	0	Single-Stage (pH 7.5)	
120/220/320						IF-1g	0	Tap Water	
121/221/321			Arre	dondo Sand		IF-2d0	0	Store II.	
122/222/322			(51-5141)		IF-2d1	3:1	Stage II+	
123/223/323	Bahia					IF-3d0	0		
124/224/324						IF-3d1	3:1	Stage II (pH 7.5)	
125/225/325]					IF-3d2	10:1		
126/226/326						IF-4	0	Single-Stage (pH 7.5)	
128/228/328	C 1					IF-1g	0	Tap Water	
129/229/329	Paspalum						0	Single-Stage (pH 7.5)	
130/230/330	i uspaium					IF-5	0	Stage I (pH 5)	

Grass Establishment Assessment

Results of percent cover assessments during establishment, prior to beginning irrigation treatments of bermudagrass, are summarized in Table 53. Growth of bermudagrass in Arredondo sand exhibited somewhat greater percent cover than that in leached or unleached gypsum throughout the establishment period. According to Gainesville greenhouse observations establishment of bermudagrass was not significantly affected by tap water irrigation.

Growth	No. of	Percent Cover (6 to 20 Days After Planting) *									
Media	Pots	6 Days	8 Days	10 Days	13 Days	16 Days	20 Days				
Leached Gypsum	15	19.2 a	29.5 ab	34.9 a	50.0 a	63.3 a	74.7 a				
Unleached Gypsum	21	13.3 a	17.8 b	24.0 a	40.7 a	55.9 a	67.4 a				
Arredondo Sand	21	20.5 a**	31.4 a	36.0 a	52.6 a	69.7 a	75.9 a				
ANOVA - P		0.10	0.05	0.10	NS	NS	NS				
*Mean value of all corresponding pots.											
** Mean values follo	** Mean values followed by the same letter do not differ significantly at the 0.05 probability level.										
NS: Not significant											

 Table 53. Results of Percent Cover Assessments During Establishment of Bermudagrass.

Photographs depicting an overview of the turfgrass at thirty days after planting (30 DAP) are presented in Figure 43. Based on Gainesville greenhouse observations, germination of bahiagrass in Arredondo sand lagged behind bermudagrass (as shown on certain pots in Figure 43).

Visual Quality Ratings

Visual quality, color and density ratings, as applicable, were assigned to each pot every 7 to 10 days throughout the irrigation study, and the results were averaged over the evaluation period. These rankings are based on a scale from 1 to 9, where 1 represents dead grass and 9 represents the highest quality grass. Conventionally, a rating of 5 is considered a minimally acceptable score for turfgrass.

In some cases, visual quality declined to poor during the study due to lack of vigor and color of shoot tissue, while the density remained relatively high. Results of the greenhouse study are presented by grass species in light of the differences in growth media/irrigation treatments.

Figure 44 corresponds to an overview photograph of the turfgrasses at 17 days after starting irrigation (i.e., 54 days after planting). Selected comparative photographs from weeks 7 (i.e., 17 days after starting fluid irrigation) and 11 (i.e., 46 days after starting fluid





Figure 43. Gainesville Greenhouse Study Overview - Grass Establishment Period (30 Days after Planting and Irrigation with Tap Water).



Figure 44. Gainesville Greenhouse Study Overview during Irrigation Period (17 Days after Starting Irrigation; 54 Days after Planting).

irrigation) are presented in Figures 45 and 46. Photographs that illustrate the turfgrass condition at the end of the irrigation period are presented in Figures 47 through 54.

Visual Ratings for Bermudagrass

Visual ratings encompassing quality, color and density, assessed 26 days after starting fluid irrigation (i.e., 63 days after planting) on growth of bermudagrass are presented in Table 54. As shown, bermudagrass grown in amended leached gypsum and irrigated with tap water generally exhibited poor quality. Pots grown in sand and irrigated with undiluted single-stage (pH 7.5) effluent exhibited the highest turfgrass quality 26 days after starting fluid irrigation. The ratings indicate that irrespective of growth media, bermudagrass exhibited, on average, higher quality when irrigated with Stage II (pH 7.5) fluids than when irrigated with Stage II + fluids 26 days after starting fluid irrigation. This observation was verified after further irrigation with Stage II and single-stage fluids during weeks 7 and 11, as illustrated by the high quality grass in Figure 45, and likely reflects the higher nutrient (i.e., nitrogen) concentration in these fluids.

Week 7 after Planting



Diluted Stage II (pH 7.5) Effluent Undiluted Stage II (pH 7.5) Effluent

Unleached Gypsum With Dolomitic Limestone _____

Arredondo Sand Week 11 after Planting



Single-Stage	Undiluted
(pH 7.5)	Stage II (pH 7.5)
Effluent	Effluent
Arredondo	Arredondo
Sand	Sand

Figure 45. Bermudagrass in Growth Media Irrigated with Various Effluents.

Irrigated with Fresh Water



Week 7 after Planting

Week 11 after Planting

8



Irrigated Stage I (pH 5) Effluent



Week 7 after Planting

Week 11 after Planting









Figure 47. Turfgrass Irrigated with Tap Water (IF-1g) (at End of Irrigation Period).





Bermuda	Bermuda	Bermuda	Bahia
on	on	on	on
Leached	Unleached	Arredondo	Arredondo
Gypsum	Gypsum	Sand	Sand
amended	amended		
with	with		
Dolomitic	Dolomitic		
Limestone	Limestone		

Figure 48. Turfgrass Irrigated with Undiluted Stage II + (IF-2d0) Fluid (at End of Irrigation Period).







Bermuda on Leached Gypsum amended with Dolomitic Limestone	Bermuda on Unleached Gypsum amended with Dolomitic Limestone	Bermuda on Arredondo Sand	Bahia on Arredondo Sand
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Figure 49. Turfgrass Irrigated with 3:1 Diluted Stage II + (IF-2d1) Fluid (at End of Irrigation Period).



Figure 50. Turfgrass Irrigated with Undiluted Stage II (pH 7.5) (IF-3d0) Fluid (at End of Irrigation Period).









Figure 51. Turfgrass Irrigated with 3:1 Diluted Stage II (pH 7.5) (IF-3d1) Fluid (at End of Irrigation Period).



Figure 52. Turfgrass Irrigated with 10:1 Diluted Stage II (pH 7.5) (IF-3d2) Fluid (at End of Irrigation Period).



Figure 53. Turfgrass Irrigated with Single-Stage (pH 7.5) (IF-4) Fluid (at End of Irrigation Period).



Figure 54. Seashore Paspalum on Arredondo Sand Irrigated with Stage I (pH 5) (IF-5) Fluid (at End of Irrigation Period).

Average quality and density ratings for bermudagrass growth over and at the end of the 60-day irrigation study period are presented in Table 55. Pots irrigated with undiluted and 3:1 diluted Stage II (pH 7.5) effluents (IF-3d0 and IF-3d1) and undiluted single-stage effluent (IF-4) generally exhibited the highest turf quality and density over the 60-day irrigation period. Pots irrigated with tap water (IF-1g) exhibited relatively poor visual quality over the 60-day irrigation period. On average, the visual quality ratings for bermudagrass grown in amended leached gypsum were lower than those obtained in other media.

As indicated in Table 55, by the end of 60-day irrigation period, quality of most pots had declined below acceptable levels (i.e., the quality rating is below 5). According to Gainesville greenhouse observations, much of the shoot tissue had turned either gray-green or light green-yellow in color. Density ratings remained relatively high indicating that the grasses were not yet losing biomass, but were suffering from severe deficiencies or toxicities. It should be noted that at the end of the 60-day irrigation period, pots irrigated with higher conductivity fluids, i.e., the undiluted lime-treated effluents (IF-3d0 and IF-4) exhibited poor quality, after having exhibited relatively good quality 26 days after the start of irrigation. Turf quality declines were also observed in pots irrigated with lower-nutrient irrigation fluids, including tap water (IF-1g) and very dilute Stage II (pH 7.5) fluid (IF-3d2), likely indicating that bermudagrass irrigated with these fluids suffered from lack of nutrients.

Bermudagrass visual quality ratings averaged over and at the end of the 60-day irrigation period were also summarized by growth media type as presented in Table 56. As noted above bermudagrass irrigated with Stage II (pH 7.5) fluids (IF-3d0, IF-3d1 and IF-3d2) and single-stage (pH 7.5) (IF-4) exhibited better visual quality ratings than grass irrigated with lower nutrient Stage II + fluids (IF-2d0 and IF-2d1) over the 60-day irrigation period, irrespective of growth media.

	Irrigation Fluid		Quality	Color	Density	
I.D.	Description	Dilution	N (mg/l)			
Amended 0	Gypsum - Leached					
IF-1g	Tap Water	0	0.05†	5.1 b*	5.2 b	5.3 c
IF-2d0	Stage II+	0	13	5.9 b	5.7 b	6.5 b
IF-2d1	Stage II+	3:1	6	5.6 b	5.4 b	6.2 b
IF-3d0	Stage II (pH 7.5)	0	230	7.4 a	7.7 a	7.8 a
IF-4	Single-Stage (pH 7.5)	0	750	7.5 a	7.6 a	7.8 a
Amended (Gypsum - Unleached	-			-	
IF-1g	Tap Water	0	0.05†	6.5 ab	6.7 abc	6.8 ab
IF-2d0	Stage II+	0	13	5.9 b	6.0 c	5.7 b
IF-2d1	Stage II+	3:1	6	6.2 ab	6.3 bc	6.6 ab
IF-3d0	Stage II (pH 7.5)	0	230	7.3 a	7.8 a	7.4 a
IF-3d1	Stage II (pH 7.5)	3:1	50.4	6.9 ab	7.1 abc	7.4 a
IF-3d2	Stage II (pH 7.5)	10:1	20.3	7.4 a	7.4 ab	7.9 a
IF-4	Single-Stage (pH 7.5)	0	750	6.6 ab	7.2 abc	7.3 ab
Arredondo	Sand		•			
IF-1g	Tap Water	0	0.05†	5.4 c	5.4 c	6.2 c
IF-2d0	Stage II+	0	13	5.6 c	5.1 c	6.1 c
IF-2d1	Stage II+	3:1	6	5.1 c	4.9 c	5.8 c
IF-3d0	Stage II (pH 7.5)	0	230	7.6 ab	7.8 a	8.1 ab
IF-3d1	Stage II (pH 7.5)	3:1	50.4	7.1 b	6.5 b	7.6 b
IF-3d2	Stage II (pH 7.5)	10:1	20.3	5.6 c	5.5 c	6.2 c
IF-4	Single-Stage (pH 7.5)	0	750	7.9 a	8.3 a	8.5 a

Table 54. Visual Quality, Color and Density Ratings for Bermudagrass 26 days AfterStarting Fluid Irrigation.

Ratings are based on a scale from 1-9, where 1= dead and 9= excellent (a rating of 5 is conventionally considered minimally acceptable.

* Mean values followed by the same letter do not differ significantly at the 0.05 probability level.

† Material was analyzed for but not detected. (The reported value is the minimum detection limit).

	Irrigation Fluid			Averaged C Irrigation S	Over 60-Day Study Period	At the End of the 60-Day Irrigation Study Period	
I.D.	Description	Dilution	N (mg/l)	Quality	Density	Quality	Density
Irrigation	Fluid (Averages over all 3 grow	th media)					
IF-1g	Tap Water	0	0.05†	5.6 d*	6.2 c	4.2 ab*	6.5 ab
IF-2d0	Stage II+	0	13.0	5.9 cd	6.6 bc	4.5 ab	6.0 ab
IF-2d1	Stage II+	3:1	6.0	5.7 cd	6.5 bc	4.8 a	5.7 b
IF-3d0	Stage II (pH 7.5)	0	230.0	6.4 a	7.3 a	3.5 b	7.3 a
IF-3d1	Stage II (pH 7.5)	3:1	50.4	6.5 a	7.2 a	3.9 ab	4.4 c
IF-3d2	Stage II (pH 7.5)	10:1	20.3	6.0 bc	6.9 ab	4.2 ab	6.0 ab
IF-4	Single-Stage (pH 7.5)	0	750.0	6.3 ab	7.2 a	3.6 ab	5.7 bc
Growth N	Media (Averages over all irrigation	on fluids)					
	Arredondo Sand	-	-	6.1 a	7.1 a	4.5 a	6.2 a
	Amended Unleached Gypsum	-	-	6.2 a	6.9 a	4.1 a	5.8 ab
	Amended Leached Gypsum	-	-	5.8 b	6.3 b	3.8 a	5.3 b
ANOVA							
Irrigation	Fluid			P = 0.0001	P = 0.0006	P=0.07	NS
Growth N	Aedia			<i>P</i> = 0.03	P = 0.001	NS	P= 0.003
Irrigation Fluid & Growth Media $P = 0.01$ $P = 0.15$ $P = 0.02$ $P = 0.02$							P= 0.004
Ratings a minimall * Mean v	re based on a scale from 1-9, why y acceptable). alues followed by the same letter	ere 1=dead	and 9=exo	cellent (a scor	e of 5 is conv .05 probabilit	entionally cor y level.	isidered

Table 55. Visual Quality and Density Ratings for Bermudagrass.

[†] Material was analyzed for but not detected. (The reported value is the minimum detection limit).

NS: Not significant.

Even though bermudagrass visual quality was slightly higher in growth media irrigated with Stage II (pH 7.5) effluent during the initial period of irrigation, the ratings at the end of the study suggest that grass irrigated with Stage II+ fluid may be able to perform better over time, probably reflecting the effect of the lower conductivity of this fluid. Over time, the irrigation with higher conductivity fluids (i.e., undiluted lime-treated effluents such as IF-2d0, IF-3d0 and IF-4) appear to cause phytotoxicity on the amended gypsum growth media.

As shown in Table 56, and as described above, at the end of 60-day irrigation period, bermudagrass quality ratings indicate that quality of most pots had declined below acceptable levels. In particular, bermudagrass had suffered significantly from irrigation with the higher conductivity undiluted Stage II (pH 7.5) fluid (IF-3d0) in the amended leached and unleached gypsum growth media, with a less pronounced effect on the grass grown in Arredondo sand. Bermudagrass grown on both amended leached gypsum and Arredondo sand exhibited somewhat higher quality ratings at the end of the 60-day irrigation period when irrigated with Stage II+ fluid than grass grown in amended unleached gypsum irrigated with the same fluid. Pots irrigated with tap water (IF-g) exhibited similar quality rankings across the growth media at the end of the irrigation period. The observed quality ratings below acceptable levels when irrigating with lower conductivity fluids (e.g., IF-3d2) and tap water (IF-1g) are most likely due to the lack of supplemental nutrition and it is typical of grass responses when left unfertilized in a pot where nutrients become limiting.

Irrigation Fluid				Averaged 9	Over 60-Day Study Period	Irrigation	At the End of the 60-Day Irrigation Study Period				
I.D.	Description	Dilution	N (mg/l)	Arredondo Sand	Amended Unleached Gypsum	Amended Leached Gypsum	Arredondo Sand	Amended Unleached Gypsum	Amended Leached Gypsum		
IF-1g	Tap Water	0	0.05†	5.7 c*	5.7 a	5.3 b	4.2 a-c*	4.0 a	4.3 a		
IF-2d0	Stage II+	0	13.0	5.9 c	5.8 a	6.0 ab	5.4 a	3.3 a	4.8 a		
IF-2d1	Stage II+	3:1	6.0	5.7 c	6.1 a	5.4 ab	4.8 ab	5.0 a	4.5 a		
IF-3d0	Stage II (pH 7.5)	0	230.0	7.0 a	6.3 a	6.0 ab	5.3 a	3.3 a	2.0 c		
IF-3d1	Stage II (pH 7.5)	3:1	50.4	6.3 b	6.5 a	-	3.6 ac	4.2 a	-		
IF-3d2	Stage II (pH 7.5)	10:1	20.3	5.5 c	6.5 a	-	3.8 a-c	4.5 a	-		
IF-4	Single-Stage (pH 7.5)	0	750.0	6.7 ab	6.2 a	6.1 a	3.0 c	4.2 a	3.5 b		
ANOVA				P = 0.0001	NS	P = 0.06	P = 0.0001	NS	P = 0.06		
Ratings a	Ratings are based on a scale from 1-9, where 1-dead and 9- excellent (a rating of 5 is conventionally considered minimally										

Table 56. Visual Quality Ratings for Bermudagrass as a Function of Growth Media.

Ratings are based on a scale from 1-9, where 1= dead and 9= excellent (a rating of 5 is conventionally considered minimally acceptable).

* Mean values followed by the same letter do not differ significantly at the 0.05 probability level.

† Material was analyzed for but not detected (The reported value is the minimum detection limit).

NS: Not significant

Visual Ratings for Bahiagrass and Seashore Paspalum

Visual quality, color and density ratings of bahiagrass and seashore paspalum grown in Arredondo sand and irrigated with various treatment fluids are presented in Table 57. As shown in Table 57, bahiagrass grown in Arredondo sand and irrigated with tap water exhibited poor turfgrass quality 26 days after starting irrigation, similarly to bermudagrass grown in this-type media when irrigated with the same fluid (see Table 54).

The seashore paspalum grass grown in sand exhibited better quality ratings when irrigated with undiluted and diluted Stage II (pH 7.5) effluents. This observation concurs with observations made for bermudagrass irrigated with these higher nutrient fluids. Moreover, seashore paspalum grass grown in sand and irrigated with Stage I (pH 5) effluent performed better than when irrigated with fresh water (see Figure 46).

Visual quality and density ratings of bahiagrass and seashore paspalum averaged over the 60-day irrigation period are summarized in Table 58. Bahiagrass grown in Arredondo sand irrigated with undiluted and diluted Stage II (pH 7.5) fluids (IF-3d0, IF-3d1, and IF-3d2) exhibited higher visual ratings over and at the end of the 60-day irrigation study period than bahiagrass grown in the same growth media when irrigated with tap water (IF-1g) or Stage II+ fluids (IF-2d0 and IF-2d1), reflecting the effect of the higher nutrient fluid.

Seashore paspalum exhibited slightly higher visual ratings than bermudagrass and bahiagrass when grown Arredondo sand and irrigated with single-stage (pH 7.5) effluent (IF-

4) over the 60 day irrigation period. Seashore paspalum, bermudagrass and bahiagrass grown in sand and irrigated with single-stage (pH 7.5) effluent (IF-4) deteriorated over time and exhibited poor ratings by the end of the irrigation period. In particular, bahiagrass grown in Arredondo sand irrigated with single-stage (pH 7.5) effluent (IF-4) had died completely by the end of the irrigation period. (It should be noted that bermudagrass is normally more salt-tolerant than bahiagrass.)

By the end of the 60-day irrigation period, two replicate pots of seashore paspalum grown in unleached gypsum irrigated with single-stage (pH 7.5) fluid (IF-4) also died completely, while a rapid decline in quality was being observed in the third replicate.

	Irrigation Fluid					
I.D.	Description Dilution N (mg/l)		Quality	Color	Density	
Bahiagras	s (in Arredondo Sand)					
IF-1g	Tap Water	0	0.05†	5.4 b	5.2 b	6.1 ab
IF-2d0	Stage II+	0	13.0	5.5 b	5.8 ab	5.7 b
IF-2d1	Stage II+	3:1	6.0	6.2 ab	6.3 ab	6.3 ab
IF-3d0	Stage II (pH 7.5)	0	230.0	6.9 a	6.9 a	6.9 a
IF-3d1	Stage II (pH 7.5)	3:1	50.4	6.1 ab	6.2 ab	6.8 ab
IF-3d2	Stage II (pH 7.5)	10:1	20.3	5.3 b	6.5 ab	6.3 ab
IF-4	Single-Stage (pH 7.5)	0	0.05†	5.8 b	6.2 ab	6.1 ab
Seashore	Paspalum (in Arredondo Sand)		-			
IF-1g	Tap Water	0	0.05†	-	6.9 b	8.3 b
IF-3d0	Single-Stage (pH 7.5)	0	230.0	-	8.5 a	8.8 ab
IF-5	Stage I (pH 5)	0	220.0	-	8.2 a	8.9 a

Table 57. Visual Quality, Color and Density Ratings for Bahiagrass and SeashorePaspalum 63 Days After Planting (26 Days of Irrigation).

Ratings are based on a scale from 1-9, where 1= dead and 9= excellent (a rating of 5 is conventionally considered minimally acceptable.

*Mean values followed by the same letter do not differ significantly at the 0.05 probability level.

† Material was analyzed for but not detected. (The reported value is the minimum detection limit).

Irrigation Fluid				Averaged Over 60-Day Irrigation Study Period				At the End of the 60-Day Irrigation Study Period			
inigation Fluid			Bahiagrass		Seashore Paspalum		Bahiagrass		Seashore Paspalum		
I.D.	Description	Dilution	N (mg/l)	Quality	Density	Quality	Density	Quality	Density	Quality	Density
IF-1g	Tap Water	0	0.05†	5.5 c*	5.9 bc	6.4 a	7.4 b	4.2 b*	4.8 b	4.4 a	6.0 a
IF-2d0	Stage II+	0	13.0	5.4 c	5.7 c	-	-	4.2 b	5.0 b	-	-
IF-2d1	Stage II+	3:1	6.0	5.6 bc	5.8 c	-	-	4.9 b	5.3 b	-	-
IF-3d0	Stage II (pH 7.5)	0	230.0	6.0 ab	6.6 ab	-	-	4.4 b	6.9 a	-	-
IF-3d1	Stage II (pH 7.5)	3:1	50.4	6.2 a	6.8 a	-	-	4.0 b	5.3 b	-	-
IF-3d2	Stage II (pH 7.5)	10:1	20.3	6.0 ab	6.5 b	-	-	6.1 a	6.5 a	-	-
IF-4	Single-Stage (pH 7.5)	0	750.0	3.8 d	4.2 d	6.9 a (Sand) 7.1 a (UL)	8.1 a (Sand) 8.0 a (UL)	0.0 c	0.0 c	3.8 a (Sand) 1.7 a (UL)	4.8 a (Sand) 2.2 a (UL)
IF-5	Stage I (pH 5)	0	220.0	-	-	7.3 a	8.1 a	-	-	4.9 a	6.8 a
ANOVA				P = 0.0001	P = 0.0001	P = 0.10	P = 0.04	P = 0.0001	P = 0.0001	NS	NS
Ratings a	Ratings are based on a scale from 1-9, where $1 = \text{dead}$ and $9 = \text{excellent}$ (rating of 5 is conventionally considered minimally acceptable.										

Table 58. Visual Quality and Density Ratings for Bahiagrass and Seashore Paspalum.

*Mean values followed by the same letter do not differ significantly at the 0.05 probability level. † Material was analyzed for but not detected. (The reported value is the minimum detection limit).

UL: Amended unleached gypsum. NS: Not significant.

Shoot and Root Growth

Bermudagrass Shoot and Root Growth

Results of Bermuda grass shoot and root biomass measurements are summarized in Table 59. As shown, the irrigation fluid type had a significant effect on shoot and root biomass measurements. bermudagrass irrigated with undiluted and 3:1 diluted Stage II (pH 7.5) fluids (IF-3d0 and IF-3d1) and single-stage (pH 7.5) effluent (IF-4) exhibited the highest shoot biomass. According to the Gainesville greenhouse observations, the subject pots exhibited the best visual quality and density throughout the study. However, the quality of these plots declined by the end of the study, while the density was still high in many of these plots.

Irrigation with lime-treated undiluted effluents (IF-2d0, IF-3d0 and IF-4) exhibited the lowest root biomass at the end of the irrigation period, yet the Stage II+ undiluted effluent performed slightly better than tap water. Results indicate that the lower conductivity diluted irrigation fluids (IF-2d1, IF-3d1 and IF-3d2) did not adversely affect root or shoot biomass compared to grass irrigated with tap water (IF-1g).

Bermudagrass grown in amended leached gypsum exhibited the lowest total biomass accumulation when ranked by growth media.

	Irrigation Fluid	Biomass (g/pot)							
I.D.	Description	Dilution	Dilution N (mg/l)		Root	Total			
Irrigation Fluid (Averaged Over All 3 Growth Media)									
IF-1g	Tap Water	0	0.05†	5.0 d*	5.9 bc	10.9 c			
IF-2d0	Stage II+	0	13.0	5.5 d	6.0 bc	11.6 c			
IF-2d1	Stage II+	3:1	50.4	4.9 d	6.4 b	11.3 c			
IF-3d0	Stage II (pH 7.5)	0	230.0	15.9 a	4.9 bc	20.7 a			
IF-3d1	Stage II (pH 7.5)	3:1	50.4	11.3 b	11.3 a	22.6 a			
IF-3d2	Stage II (pH 7.5)	10:1	20.3	8.0 c	6.0 bc	14.0 bc			
IF-4	Single-Stage (pH 7.5)	0	750.0	11.3 b	4.0 c	15.3 b			
Growth M	Iedia (Averaged Over All Irrigat	tion Fluids)							
-	Arredondo Sand	-	-	8.3 b	6.8 a	15.1 a			
-	Amended Unleached Gypsum	-	-	10.5 a	5.7 a	16.2 a			
-	Amended Leached Gypsum	-	-	7.0 b	5.7 a	12.7 b			
				P = 0.0001	P = 0.0001	P = 0.0001			
P = 0.0002 NS $P = 0.01$									
*Mean values followed by the same letter do not differ significantly at the 0.05 probability level. †Material was analyzed for but not detected (The reported value is the minimum detection limit). NS: Not significant.									

Table 59. Summary of Bermudagrass Shoot and Root Biomass Measurements (at
the End of the 60-Day Irrigation Study Period).

Bahiagrass and Seashore Paspalum Shoot and Root Growth

Results of terminal shoot and root biomass measurements of bahiagrass are summarized in Table 60. As observed for different media seeded with bermudagrass, and irrespective of growth media, bahiagrass irrigated with undiluted and 3:1 diluted Stage II (pH 7.5) fluids (IF-3d0 and IF-3d1) yielded the greatest amount of shoot biomass. Pots irrigated with diluted lime-treated fluids (IF-2d1, IF-3d1 and IF-3d2) did not adversely affect root biomass compared to grass irrigated with tap water (IF-1g). On the other hand, pots irrigated with higher conductivity fluids (i.e., undiluted lime-treated effluents IF-2d0, IF-3d0 and IF-4) exhibited lower root biomass at the end of the irrigation study than grass irrigated with tap water (IF-1g).

Results of terminal shoot and root biomass measurements of seashore paspalum are summarized in Table 61. Seashore paspalum irrigated with undiluted Stage I and singlestage fluids (IF-5 and IF-4) yielded the greatest amount of shoot biomass. However, these higher conductivity fluids yielded smaller amount of root biomass when compared to grass irrigated with tap water (IF-1g). Pots irrigated with tap water (IF-1g) showed significant amount of root biomass and a limited shoot biomass.

	Irrigation Fluid	Biomass (g/pot)						
I.D.	Description	Dilution	N (mg/l)	Shoot	Root	Total		
IF-1g	Tap Water	0	0.05†	3.0 bc*	7.5 a	10.4 b		
IF-2d0	Stage II+	0	13.0	2.8 c	4.1 b	6.9 c		
IF-2d1	Stage II+	3:1	6.0	2.7 c	7.5 a	10.1 b		
IF-3d0	Stage II (pH 7.5)	0	230.0	8.7 a	4.0 b	12.7 b		
IF-3d1	Stage II (pH 7.5)	3:1	50.4	8.6 a	8.3 a	16.9 a		
IF-3d2	Stage II (pH 7.5)	10:1	20.3	4.6 b	8.4 a	13.0 b		
IF-4	Single-Stage (pH 7.5)	750.0	2.9 bc	2.5 b	5.4 c			
ANOVA		P=0.0001	P=0.0001	P=0.0001				
*Mean values followed by the same letter do not differ significantly at the 0.05 probability level.								
†Materia	l was analyzed for but not detec	ted (the rep	orted valu	e is the mini	mum detect	ion limit).		

Table 60. Summary of Bahiagrass Shoot and Root Biomass Measurements (at the End of the 60-Day Irrigation Study Period).

Table 61. Summary of Seashore Paspalum Shoot and Root Biomass Measurements(at the End of the 60-Day Irrigation Study Period).

	Irrigation Fluid	Biomass (g/pot)							
I.D.	Description	Dilution	N (mg/l)	Shoot	Root	Total			
IF-1g	Tap Water (Soil)	0	0.05†	3.1 c*	10.3 b	13.4 b			
IF-4	Single-Stage (pH 7.5) (Soil)	0	750.0	13.3 b	4.7 b	18.0 a			
IF-4	Single-Stage (pH 7.5) (UL)	0	750.0	12.4 b	1.9 b	14.3 b			
IF-5	Stage I (pH 5.0) (Soil)	0	220.0	18.3 a	3.5 b	21.8 a			
ANOV	ANOVA P=0.001 P=0.02								
* Mean values followed by the same letter do not differ significantly at the 0.05 probability level.									
† Material was analyzed for but not detected (The reported value is the minimum detection limit).									
UL: Ai	mended unleached gypsum.								

Fluoride and Arsenic Content of Plant Tissues

Plant tissue samples from selected pots with Arredondo sand as growth media were obtained at terminal harvest for testing fluoride and arsenic contents. Selected samples were sent to Pembroke Laboratories, Inc. to perform the analyses.

Moist plant samples were dried at about 65° C until they became crispy. Samples were then passed through a stainless steel grinder with a mesh opening equivalent to US sieve No. 20 (850 µm) and mixed thoroughly. Samples were dried again at about 65° C for 2 hours and cooled in a desiccator prior to weighing for analysis.

Fluoride was extracted by shaking 0.50 to 1.0 g of dried plant material in 20 mL of 0.05M H_2SO_4 for 15 minutes. Then, 20 mL of 0.01M NaOH were added followed by an additional 15 minutes of shaking. Potential interference from Al, Si and Fe were reduced by adding 5 ml of 3M NaOAc and 10 mL of 0.5M sodium citrate buffers before analysis.

Fluoride content was then analyzed by the Ion Selective Electrode method on extract samples. Table 62 presents results of the measured fluoride content along with the arsenic content on the selected plant tissues. Measured fluoride contents ranged from about 30 to 200 μ g/g. Considering that excessive fluoride in feed plants can cause fluorosis in grazing animals, longer-term evaluation of the turf grass irrigated with lime-treatment effluents will be needed in combination with the effect of seasonal and cyclical rainfalls to address the possibility of using this grass for feeding grazing animals. Notwithstanding the potential concern about fluorosis, the fluoride contents of plants irrigated with Stage II and Stage II+ effluents ranged from 30 to 80 μ g/g, consistent with the tissue content of plants irrigated with tap water.

Arsenic was not detected in any of the tested samples.

Pot	Growth	Grass		Irrigation	Tissue Content (µg/g)				
No.	Media	Туре	I.D.	Description	Dilution	F (mg/l)	Fluoride	Arsenic	
113/313		Bermuda	IF-1g	Tap Water	0	0.9	60	0.1 [U]	
114/214/314		Bermuda	IE 240	Stago II.	0	0.6	82 [A]	0.1 [U]	
121/221		Bahia	16-200	Stage II+	0	9.0	50	0.1 [U]	
122/222	ndo Sanc	Bahia	IF-2d1	Stage II+	3	3.1	31	0.1 [U]	
116/216/316	юра	Bermuda	IE 240	Stage II	0	24	52 [A]	0.1 [U]	
123/223	Arre	Bahia	16-300	(pH 7.5)	0		59	0.1 [U]	
119/219/319	~	Bermuda					93 [A]	0.1 [U]	
126/226/326		Bahia	IF-4	Single-Stage (pH 7.5)	0	54	199 [A]	0.1 [U]	
[U] Material was analyzed for but not detected (the reported value is the minimum detection limit). [A] Average value from two analyses (on samples from replicate pots).									

 Table 62. Summary of Fluoride Analyses on Plant Tissue Samples.

MILTON GREENHOUSE STUDY

Experimental Study Plan

A total of 19 growth media/irrigation fluid combinations replicated three times were used in the Milton greenhouse study. Leached and unleached gypsum amended with dolomitic limestone, single-stage (pH 7.5) sludge, single-stage (pH 7.5) effluent, Stage II sludge, and Stage II effluent were used as growth media. The combinations of grass type, growth media and irrigation fluids used in the Milton study are summarized in Table 63.

Seeding and Grass Establishment

Bermudagrass (*Cynodon dactylon* L.) cultivar 'Sahara' was seeded into 6-inch pots at a rate of 3 lb/1000 ft² (i.e., 0.27 g/pot, which corresponds to a land application rate of 130 pounds per acre). In addition, a control plot using Orangeburg sandy loam (silty sand) was also used as growth media.

Pots were placed under a potable water mist irrigation system and watered as needed during establishment. Initial conductivity measurements were taken 15 days after planting. After three weeks pots were transferred to a greenhouse with a higher temperature.

All pots received about 22 pounds of nitrogen per acre prior to the initiation of the irrigation treatment regime.

Irrigation

Irrigation treatments were initiated 50 days after planting. Pots were irrigated with either undiluted Stage II+ effluent (IF-2d0) or well water (IF-1m). Pots were irrigated at a rate of 100 ml (using a pipette) six times a week as needed to maintain evapotranspiration (1.3 inches per week irrigation rate). The irrigation treatment volume was increased to 150 ml six times a week (2.0 inches per week) 72 days after planting to balance evapotranspiration. Irrigation continued until 116 days after planting, at which time the treated effluent irrigation fluid had been depleted.

Clipping and Terminal Harvest

Pots were trimmed to a uniform height of 3 inches 50 days after planting (just prior to the start of irrigation). Shoot biomass was determined every two weeks thereafter. Clippings from each harvest were dried and weighed.

A terminal harvest was conducted at 129 days after planting where all shoots were clipped to the soil level. After shoot removal, final conductivity measurements were recorded, and roots were separated from the growth media. Length, and fresh and dry weights of roots were determined.

	Growth Med		Irrigation Fluid			
I.D.	Amendment Material Material Type	Addition Rate by Dry Weight (%)	Gypsum Source Site	Grass Type	I.D.	Description
Leached	Phosphogypsum					
AG-1	Dolomitic Limestone	0.25	В		IF-1m IF-2d0	Well Water Stage II+ Undiluted
AG-3A	Stage II Sludge	1.0	В		IF-1m IF-2d0	Well Water Stage II+ Undiluted
AG-3B	Stage II Sludge	0.5	Ν	ıda	IF-1m	Well Water
AG-4A	Single-Stage (pH 7.5) Sludge	2.5	В	Bermı	IF-1m IF-2d0	Well Water Stage II+ Undiluted
AG-4B	Single-Stage (pH 7.5) Sludge	2.0	N		IF-1m	Well Water
AG-6	Stage II Effluent	1.0	N		IF-1m	Well Water
AG-7	Single-Stage (pH 7.5) Effluent	20	В		IF-1m IF-2d0	Well Water Stage II+ Undiluted
Unleache	d Phosphogypsum		-	-		
AG-2	Dolomitic Limestone	2.5	В		IF-1m IF-2d0	Well Water Stage II+ Undiluted
AG-5A	Stage II Sludge	6.0	В	nuda	IF-1m IF-2d0	Well Water Stage II+ Undiluted
AG-5B	Stage II Sludge	1.0	N	ern	IF-1m	Well Water
AG-5C	Stage II Sludge	10.0	С	В	IF-1m	Well Water
	Silty Sand		IF-1m IF-2d0	Well Water Stage II+ Undiluted		

Table 63. Summary of Growth Media and Irrigation Fluid Combinations Used in
Milton Greenhouse Study.

Grass Establishment Assessment

Results of germination assessment during grass establishment are summarized in Table 64. As indicated, a significant germination of bermudagrass was observed in pots with silty sand used as growth media by 8 days after planting. Only limited germination of bermudagrass grown in leached gypsum amended with Stage II sludge (AG-3A) was observed by 8 days after planting. In general, the grass germinated progressively over time in the different-type growth media.

Bermudagrass germinated in leached gypsum amended with Stage II (pH 7.5) sludge (AG-3A) exhibited the best establishment at 17 days after planting, which was somewhat better than the grass establishment observed in the silty sand control pot. Bermudagrass in leached gypsum amended with Stage II and single-stage (pH 7.5) effluents (AG-6 and AG-7, respectively) exhibited significantly lower amount of germination compared to pots having leached gypsum amended with sludge or dolomitic limestone as growth media.

Bermudagrass germination was relatively poor by 17 days after planting on unleached gypsum amended with dolomitic limestone (AG-2) and unleached gypsum amended with 1% addition of Stage II sludge as growth media (AG-5B).

Overall, the poorest bermudagrass germination was observed in unleached gypsum amended with 10% addition of Stage II sludge as growth media (AG-5C). According to the greenhouse observations, the original growth media of AG-5C was water logged, probably indicating that the sludge percent addition was too high, adding excessive moisture to the growth media. The condition of this growth media improved after the material was allowed to dry, pulverized, and pots were reestablished.

Photographs of the turfgrass condition 50 days after planting (just prior to the start of the irrigation period) are presented in Figures 55 through 63. Selected comparative photographs are presented in Figures 64 through 66.



Figure 55. Bermudagrass on Leached Gypsum Amended with Stage II Sludge (AG-3A) During Establishment (50 Days after Planting and Irrigation with Well Water).



Figure 56. Bermudagrass on Unleached Gypsum Amended with Stage II Sludge (AG-5A) During Establishment (50 Days after Planting and Irrigation with Well Water).


Figure 57. Bermudagrass on Leached Gypsum Amended with Single-Stage (pH 7.5) Effluent (AG-7) During Establishment (50 Days after Planting and Irrigation with Well Water).



Figure 58. Bermudagrass on Leached Gypsum Amended with Single-Stage (pH 7.5) Sludge (AG-4A) During Establishment (50 Days after Planting and Irrigation with Well Water).



Figure 59. Bermudagrass on Unleached Gypsum Amended with Stage II Sludge (AG-5C left, and AG-5B right) During Establishment (50 Days After Planting and Irrigation with Well Water).



Figure 60. Bermudagrass on Leached Gypsum Amended with Stage II Sludge (AG-3B Top Left); Stage II Effluent (AG-6 top right); and Single-Stage (pH 7.5) Sludge (AG-4B) During Establishment (50 Days after Planting and Irrigation with Well Water).



Figure 61. Bermudagrass on Leached Gypsum Amended with Dolomitic Limestone (AG-1) During Establishment (50 Days after Planting and Irrigation with Well Water).



Figure 62. Bermudagrass on Unleached Gypsum Amended with Dolomitic Limestone (AG-2) During Establishment (50 Days after Planting and Irrigation with Well Water).



Figure 63. Bermudagrass on Silty Sand During Establishment (50 Days After Planting and Irrigation with Well Water).



Dolomitic Limestone

Stage II Sludge

Single-Stage (pH 7.5)

Single-Stage (pH 7.5)

Figure 64. Bermudagrass in Amended Leached Gypsum Irrigated with Fresh Water.



Dolomitic Limestone

Stage II Sludge

Figure 65. Bermudagrass in Amended Unleached Gypsum Irrigated with Fresh Water.



Figure 66. Bermudagrass in Unleached Gypsum Amended with Stage II Sludge Irrigated with Fresh Water.

	Growth Media			Seedlings per Pot					
	Amendment Material								
I.D.	I.D. Material Type		Gypsum Source Site	4/02/02 (8 DAP)	4/05/02 (11 DAP)	4/08/02 (14 DAP)	4/11/02 (17 DAP)		
Leached F	Phosphogypsum								
AG-1	Dolomitic Limestone	0.25	В	0.7 c* 1.0 c	20.7 d-g 23.3 c-f	70.7 cde 84.0 bcd	94.3 bc 97.7 bc		
AG-3A	Stage II Sludge	1.0	В	1.0 c 5.3 bc	39.7 bc 68.0 a	96.3 abc 123.3 a	117.7 ab 135.7 a		
AG-3B	Stage II Sludge	0.5	N	0 c	20.3 d-g	40.7 fgh	53.0 e-h		
AG-4A	Single-Stage (pH 7.5) Sludge	2.5	В	0.3 c 0.3 c	26.3 cde 34.3 bcd	61.3 d-g 69.0 c-f	85.0 cde 97.7 bc		
AG-4B	Single-Stage (pH 7.5) Sludge	2.0	Ν	1.0 c	26.3 cde	41.7 e-h	57.7 d-g		
AG-6	Stage II Effluent	1.0	N	0 c	15.3 e-h	37.7 ghi	44.7 f-l		
AG-7	Single-Stage (pH 7.5) Effluent	20.0	В	0 c 0 c	6.7 h 2.0 fgh	21.7 hij 22.0 hij	29.7 g-j 32.0 g-j		
Unleached	l Phosphogypsum								
AG-2	Dolomitic Limestone	2.5	В	0 c 0 c	0.0 h 0.0 h	23.3 hij 8.7 ij	49.7 fgh 17.0 if		
AG-5A	Stage II Sludge	6.0	В	0 c 0 c	6.0 gh 2.7 h	64.7 d-g 21.3 hij	88.7 bcd 37.7 g-j		
AG-5B	Stage II Sludge	1.0	N	0.3 c	3.3 h	17.7 hij	21.7 hij		
AG-5C [†] Stage II Sludge			С	0 c	0.7 h	4.3 j	7.3 ј		
	Silty Sand		27.0 a 12.7 c	36.7 bcd 44.0 b	65.0 d-g 100.7 ab	71.7 c-f 104.0 abc			
	LSD (P=0.05)	7.7	16.7	29.4	32.4				
*Mean va	lued followed by the same letter do	not differ sig	gnificantly at	the 0.05 pro	bability level.				

 Table 64. Germination of Bermudagrass in Amended Phosphogypsum.

*Mean valued followed by the same letter do not differ significantly at the 0.05 probability level. LSD = Least Square Difference which characterizes the minimum difference between statistically significant differences in measurement or rating at the 95% confidence level.

AG-5C was initially water logged and was allowed to dry and pulverized to reestablish the growth media.

Electrical Conductivity

Electrical conductivity measurements were made on the growth media in each pot at the end of the irrigation period. Table 65 summarizes the effect of irrigation fluid on final growth media conductivity. As expected, the higher conductivity irrigation fluid; i.e., undiluted Stage II+ (pH 7.5) effluent, IF-2d0, significantly altered the soil pore water conductivity. As indicated in Table 65, irrigation with high-conductivity undiluted Stage II+ (pH 7.5) fluid (IF-2d0) resulted in higher growth media conductivities when compared to pots irrigated with well water (IF-1m).

As indicated in Table 65, growing media (pore water) conductivities in the range of 800 to 7,200 μ mhos/cm were measured for pots irrigated with the high-conductivity

undiluted Stage II (pH 7.5) fluid (IF-2d0). In general, irrigation with well water had little effect on low conductivity growth media. Irrigation with well water (IF-1m) on pots with unleached gypsum amended with 6% and 10% addition of Stage II sludge (AG-5A and AG-5C, respectively) yielded to a reduction in pore water conductivity from 2,600 to 1,200 μ mhos/cm and from 5,600 to 2,000 μ mhos/cm, respectively.

Growth Media					Gro Irrigation Fluid Co (μ					
	Amendment Materi									
I.D.	Material Type	Addition Rate by Dry Weight (%)	dition te by DryGypsum SourceOrySiteeight (%)		Description	Conductivity (µmhos/cm)	Initial (4/09/02)	Final (7/22/02)		
Leached P	hosphogypsum									
AG-1	Dolomitic Limestone	0.25	В	IF-1m IF-2d0	Well water Undiluted Stage II+	100 4,500	330 d* 370 d	400 d 800 cd		
AG-3A	Stage II Sludge	1.0	В	IF-1m IF-2d0	Well water Undiluted Stage II+	100 4,500	330 d 370 d	500 cd 1,600 bc		
AG-3B	Stage II Sludge	0.5	0.5 N		Well water	100	330 d	300 d		
AG-4A	Single-Stage (pH 7.5) Sludge (Lab)	2.5	В	IF-1m Well water IF-2d0 Undiluted Stage II+		100 4,500	430 d 330 d	400 d 900 bcd		
AG-4B	Single-Stage (pH 7.5) Sludge (Lab)	2.0	N	IF-1m	Well water	100	330 d	400 d		
AG-6	Stage II Effluent	1.0	N	IF-1m	Well water	100	300 d	300 d		
AG-7	Single-Stage (pH 7.5) Effluent (Lab)	20.0	В	IF-1m IF-2d0	Well water Undiluted Stage II+	100 4,500	470 d 330 d	400 d 1,300 bcd		
Unleached	l Phosphogypsum									
AG-2	Dolomitic Limestone	2.5	В	IF-1m IF-2d0	Well water Undiluted Stage II+	100 4,500	430 d 470 d	500 cd 1,600 bc		
AG-5A	Stage II Sludge	6.0	В	IF-1m IF-2d0	Well water Undiluted Stage II+	100 4,500	2,600 c 3,900 b	1,200 bcd 7,200 a		
AG-5B	Stage II Sludge	1.0	N	IF-1m	Well water	100	370 d	400 d		
AG-5C [†]	Stage II Sludge	10.0	С	IF-1m	Well water	100	5,600 a	2,000 b		
Silty Sand					Well water Undiluted Stage II+	100 4,500	100 d 100 d	200 d 1,200 bcd		
LSD (P=0.05) 1.1 1.2							1.2			
*Mean val [†] AG-5C w	lues followed by the same vas initially water-logged a	*Mean values followed by the same letter do not differ significantly at the 0.05 probability level.								

 Table 65. Effect of Irrigation Fluid on Final Growing Media Conductivity.

Visual Quality Ratings

Table 66 presents visual ratings of bermudagrass with respect to the effect of growth media and irrigation fluid combination during the study period. Prior to initiation of irrigation treatments, significant differences in quality of bermudagrass were attributed to properties of the growing medium such as conductivity, nitrogen content, etc. bermudagrass

grown in most of the growing media initially exhibited acceptable quality, with the exception of leached gypsum amended with single-stage (pH 7.5) sludge, Stage II effluent and single-stage (pH 7.5) effluent (AG-4B, AG-6 and AG-7, respectively), which exhibited only marginally acceptable visual qualities just prior to the start of the irrigation treatment period (i.e., 50 days after planting).

As indicated in Table 66, bermudagrass quality ultimately declined over the duration of the study irrespective of the irrigation fluid. In general, pots irrigated with well water (IF-1m) initially displayed good quality that gradually declined to poor quality towards the end of study period. At the end of the study period, bermudagrass grown in leached and unleached gypsum amended with dolomitic limestone (AG-1 and AG-2) and irrigated with undiluted Stage II+ (pH 7.5) fluid (IF-2d0) exhibited the best quality among the growth media/irrigation fluid combinations, which was only at the marginally acceptable level for turf. In general, pots irrigated with undiluted Stage II + effluent (IF-2d0) exhibited higher quality than pots irrigated with well water (IF-1m) at the end of the irrigation period, likely reflecting the effect of the higher nutrient (i.e., nitrogen) concentration in the undiluted lime-treated effluent. Similar observations were noted in the Gainesville greenhouse study.

Visual quality observed on grass grown on leached gypsum amended with Stage II and single-stage (pH 7.5) sludges (i.e., AG-3A and AG-4A, respectively) when irrigated with undiluted Stage II + effluent (IF-2d0) was only slightly lower than the quality observed in the leached and unleached gypsum growth medium amended with dolomitic limestone (i.e., AG-1 and AG-2, respectively) when irrigated with the same fluid, indicating that these two-type lime-treatment sludges can be used as viable alternatives for amending phosphogypsum in closure applications when applied at the approximate rates used in this study (i.e., 1.0% and 2.5% dry weight addition for the Stage II and single-stage sludges, respectively).

Bermudagrass grown on unleached gypsum amended with Stage II sludge (AG-5A, AG-5B and AG-5C) exhibited very poor visual quality ratings at the end of the study period, irrespective of irrigation fluid. By the end of the study very poor ratings were also observed on bermudagrass grown on sand, suggesting that there was probably a generalized lack of supplemental nutrition in the pots at that time as only one fertilization was undertaken prior to initiation of the irrigation treatment regime.

Shoot Growth

Results of terminal shoot biomass measured during final destructive sampling, and cumulative shoot biomass measured during periodic clippings are summarized in Table 67. Throughout the study, and particularly at the terminal shoot harvest, bermudagrass yielded the greatest amount of shoot biomass (both fresh and dry weights) when grown in leached gypsum amended with single-stage (pH 7.5) sludge (AG-4A) and unleached gypsum amended with Stage II sludge (AG-5A) when irrigated with undiluted Stage II+ fluid (IF-2d0). Shoot biomass was lower in the unleached gypsums amended with Stage II sludge when irrigated with well water (AG-5A and AG-5C) than similar growth media (AG-5A)

irrigated with undiluted Stage II+ fluid (IF-2d0). In general, pots irrigated with the undiluted Stage II+ effluent yielded higher biomass than those irrigated with well water.

Leached gypsum amended with Stage II effluent (AG-6) irrigated with well water (IF-1m) exhibited the lowest shoot biomass. Shoot biomass was also relatively low on leached gypsum amended with single-stage (pH 7.5) effluent (AG-7) when irrigated with well water (IF-1m). It should be noted that these growth media pots had exhibited the lowest establishment percentage at 17 days after planting (Table 64).

Growth Media Irrigation Flu			rigation Fluid	.id Visual Quality Rating							
	Amendment Mater	ial									Final Growing
I.D.	Material Type	Addition Rate by Dry Weight (%)	Gypsum Source Site	I.D.	I.D. Description (:		06/15/02 (82 DAP) 32 DASF	06/24/02 (91 DAP) 41 DASF	07/10/02 (107 DAP) 57 DASF	07/22/02 (119 DAP) 69 DASF	Media Conductivity (µmhos/cm)
Leached	Phosphogypsum						·				•
AG-1	Dolomitic Limestone	0.25	В	IF-1m IF-2d0	Well water Undiluted Stage II+	7.3 ab* 7.0 abc	4.3 5.0	4.0 4.7	3.3 4.3	2.3 c 5.0 a	400 d 800 cd
AG-3A	Stage II Sludge	1.0	В	IF-1m IF-2d0	F-1m Well water F-2d0 Undiluted Stage II+		5.0 4.3	4.3 4.0	4.3 3.7	3.0 bc 4.7 a	500 cd 1,600 bc
AG-3B	Stage II Sludge	0.5	Ν	IF-1m	IF-1m Well water		5.0	5.0	4.3	3.0 bc	300 d
AG-4A	Single-Stage (pH 7.5) Sludge (lab)	2.5	В	IF-1m IF-2d0	F-1m Well water F-2d0 Undiluted Stage II+		4.3 4.7	4.3 4.3	4.0 3.7	3.3 bc 4.0 ab	400 d 900 bcd
AG-4B	Single-Stage (pH 7.5) Sludge (lab)	2.0	N	IF-1m	Well water	6.3 cde	4.0	3.7	3.3	3.0 bc	400 d
AG-6	Stage II Effluent	1.0	Ν	IF-1m	Well water	6.0 de	4.3	3.3	2.7	2.7 c	300 d
AG-7	Single-Stage (pH 7.5) Effluent (lab)	20.0	В	IF-1m IF-2d0	Well water Undiluted Stage II+	5.7 e 6.7 bcd	4.0 5.0	3.7 4.7	3.0 5.3	2.7 c 4.7 a	400 d 1,300 bcd
Unleache	d Phosphogypsum										
AG-2	Dolomitic Limestone	2.5	В	IF-1m IF-2d0	Well water Undiluted Stage II+	7.0 abc 6.3 cde	4.3 4.7	4.0 5.0	3.7 5.0	2.7 c 5.0 a	500 cd 1,600 bc
AG-5A	Stage II Sludge	6.0	В	IF-1m IF-2d0	Well water Undiluted Stage II+	7.3 ab 7.3 ab	4.3 5.7	4.0 5.3	3.0 5.3	2.3 c 3.0 bc	1,200 bcd 7,200 a
AG-5B	Stage II Sludge	1.0	N	IF-1m	Well water	7.0 abc	4.7	4.7	4.3	3.3 bc	400 d
$AG-5C^{\dagger}$	Stage II Sludge	10.0	С	IF-1m	Well water	7.7 a	4.5	2.7	3.6	3.0 bc	2,000 b
Silty Sand IF-1m Well water IF-2d0 Undiluted Stage II+			7.7 a 7.7 a	4.7 5.0	4.7 5.0	4.3 4.7	2.7 c 4.7 a	200 d 1,200 bcd			
LSD (P=0.05)					0.8	NS	NS	NS	1.2		

Table 66. Effect of Irrigation Fluid Type and Growing Media on Bermudagrass Quality.

Ratings are based on a scale from 1-9, where 1= dead and 9= excellent (a rating of 5 is conventionally considered minimally acceptable). * Mean values followed by the same letter do not differ significantly at the 0.05 probability level.

LSD - Least Square Differences which characterizes the minimum difference between statistically significant differences in measurement or rating at the 95% confidence level. [†]AG-5C was initially water logged and was allowed to dry and pulverized to reestablish the growth media.

NS: Not significant.

Growth Media				Irrigation Fluid		Terminal Shoot Weight (g/pot)		Total Shoot Weight (g/pot)		Cumulative Shoot Weight (g/pot)		Incremental Shoot Weight (g/pot)	
	Amendment Mate	erial											
I.D.	Material Type	Addition Rate by Dry Weight (%)	Gypsum Source Site	I.D.	Description	Fresh	Dry	Fresh	Dry	Fresh	Dry	Fresh	Dry
Leached P	hosphogypsum					-	-			-			
AG-1	Dolomitic Limestone	0.25	В	IF-1m IF-2d0	Well water Undiluted Stage II+	11.24 c-f* 16.03 a-d	6.52 bcd 7.84 abc	20.99 22.34	9.70 10.42	15.77 19.90	8.22 9.68	4.53 3.87	1.70 1.84
AG-3A	Stage II Sludge	1.0	В	IF-1m IF-2d0	Well water Undiluted Stage II+	12.52 b-f 11.21 c-f	7.40 a-d 6.12 bcd	25.13 20.02	11.35 8.73	17.46 14.98	9.50 7.90	4.94 3.77	2.10 1.78
AG-3B	Stage II Sludge	0.5	Ν	IF-1m	Well Water	11.12 c-f	5.92 bcd	24.48	10.65	22.36	10.00	11.24	4.08
AG-4A	Single-Stage (pH 7.5) Sludge (Lab)	2.5	В	IF-1m IF-2d0	Well water Undiluted Stage II+	16.14 abc 19.88 ab	9.23 ab 11.17 a	26.63 35.16	12.95 16.45	21.13 29.39	11.52 15.03	4.99 9.51	2.29 3.86
AG-4B	Single-Stage (pH 7.5) Sludge (Lab)	2.0	Ν	IF-1m	Well water	7.60 efg	4.32 cde	12.30	6.25	10.37	5.62	2.77	1.30
AG-6	Stage II Effluent	1.0	Ν	IF-1m	Well water	2.92 g	1.49 e	12.27	4.75	8.10	3.56	5.18	2.07
AG-7	Single-Stage (pH 7.5) Effluent (Lab)	20.0	В	IF-1m IF-2d0	Well water Undiluted Stage II+	7.95 efg 15.19 a-e	4.37 cde 8.17 abc	22.67 26.02	9.93 12.51	20.16 21.30	9.20 11.34	12.21 6.11	4.83 3.17
Unleached	Phosphogypsum												
AG-2	Dolomitic Limestone	2.5	В	IF-1m IF-2d0	Well water Undiluted Stage II+	10.98 c-f 12.54 b-f	6.09 bcd 6.58 bcd	16.58 27.83	8.22 11.00	13.83 20.21	7.64 9.27	2.85 7.67	1.55 2.69
AG-5A	Stage II Sludge	6.0	В	IF-1m IF-2d0	Well water Undiluted Stage II+	13.00 f 20.85 a	6.61 bcd 11.04 a	33.38 34.60	13.42 16.01	24.37 30.52	11.17 15.01	11.37 9.67	4.56 3.97
AG-5B	Stage II Sludge	1.0	Ν	IF-1m	Well water	8.29 d-g	4.41 cde	24.36	9.48	16.75	7.47	8.46	3.06
AG-5C [†]	Stage II Sludge	10.0	С	IF-1m	Well water	16.49 abc	9.38 ab	36.28	16.02	28.02	13.94	11.53	4.56
Silty Sand IF- IF-		IF-1m IF-2d0	Well water Undiluted Stage II+	6.31 fg 12.27 b-f	3.58 de 6.54 bcd	16.90 25.65	7.21 11.34	10.82 21.78	5.67 10.29	4.51 9.51	2.09 3.75		
		LSD (P=0.0)5)			7.82	4.35	-	-	-	-	-	-

Table 67. Effect of Irrigation Fluid Type and Growing Media on Bermudagrass Shoot Biomass.

Ratings are based on a scale from 1-9, where 1= dead and 9= excellent (a rating of 5 is conventionally considered minimally acceptable).

*Mean values followed by the same letter do not differ significantly at the 0.05 probability level.

LSD – Least Square Differences which characterizes the minimum difference between statistically significant differences in measurement or rating at the 95% confidence level. [†]AG-5C was initially water logged and was allowed to dry and pulverized to reestablish the growth media.

Root Growth

Table 68 presents the results of bermudagrass root length and root biomass measurements during the study period. Bermudagrass grown in silty sand and leached gypsum amended with single-stage (pH 7.5) sludge (AG-4A) exhibited greater root lengths than the remainder growth media, irrespective of the irrigation fluid. It should be noted that leached and unleached gypsum amended with Stage II sludge growth media (AG-3A, AG-3B, AG-5A, AG-5B and AG-5C) yielded similar root length among these media, irrespective of irrigation fluid.

The data suggest that gypsum amendment with lime-treated sludges is likely to promote more root growth than gypsum amendment with dolomitic limestone.

As indicated in Table 68, root length is slightly greater in leached gypsum amended with dolomitic limestone (AG-1) than in unleached gypsum amended with dolomitic limestone (AG-2) when irrigated with well water and undiluted Stage II+ effluent fluids.

Root lengths were relatively lower on leached gypsum amended with Stage II and single-stage (pH 7.5) effluents (AG-6 and AG-7, respectively), and irrigated with well water (IF-1m), when compared to other growth media.

The combination of growing media and irrigation fluids had significant effect on fresh and dry root biomass. Bermudagrass grown in leached gypsum amended with single-stage (pH 7.5) sludge (AG-4A) exhibited greater fresh and dry root masses than those measured in other growth media. It appears that for this particular growth media the irrigation fluid had only a minor effect on the measured root biomass during the irrigation period of study.

Bermudagrass grown in leached gypsum amended with Stage II effluent (AG-6) irrigated with well water (IF-1m) yielded the lowest root weight.

Although the root length measured in the silty sand was relatively high, the measured root weight in this growth medium was very low.

	Growth Media			Irrigation Fluid			Root Weig		Final
	Amendment Material					Root			Growing
I.D.	Material Type	Addition Rate by Dry Weight (%)	Gypsum Source Site	I.D.	Description	Length (cm)	Fresh	Dry	Media Conductivity (µmhos/cm)
Leached P	hosphogypsum								
AG-1	Dolomitic Limestone	0.25	В	IF-1m IF-2d0	Well water Undiluted Stage II+	9.7 10.7	3.37 b-f 4.57 a-f	1.87 c-f 2.47 a-d	400 d 800 cd
AG-3A	Stage II Sludge 1.0		В	IF-1m IF-2d0	Well water Undiluted Stage II+	11.8 12.0	2.56 b-f 1.25 def	1.34 c-g 0.69 efg	500 cd 1,600 bc
AG-3B	Stage II Sludge 0.5		Ν	IF-1m	Well water	10.2	5.12 а-е	2.07 b-e	300 d
AG-4A	Single-Stage (pH 7.5) Sludge (Lab) 2.5		В	IF-1m IF-2d0	Well water Undiluted Stage II+	12.3 12.0	7.71 a 8.20 a	2.73 abc 3.50 ab	400 d 900 bcd
AG-4B	Single-Stage (pH 7.5) Sludge (Lab)	ab) 2.0 N		IF-1m	Well water	7.7	1.36 def	1.06 d-g	400 d
AG-6	Stage II Effluent	1.0	Ν	IF-1m	Well water	8.8	0.48 f	0.25 g	300 d
AG-7	Single-Stage (pH 7.5) Effluent (Lab)	20.0	В	IF-1m IF-2d0	Well water Undiluted Stage II+	8.7 11.3	3.46 b-f 6.49 ab	1.30 c-g 1.89 c-f	400 d 1,300 bcd
Unleached	Phosphogypsum	1	1	r	1	1	1	1	
AG-2	Dolomitic Limestone	2.5	В	IF-1m IF-2d0	Well water Undiluted Stage II+	8.2 7.8	5.63 abc 2.28 c-f	1.93 c-f 1.76 c-g	500 cd 1,600 bc
AG-5A	Stage II Sludge	6.0	В	IF-1m IF-2d0	Well water Undiluted Stage II+	10.0 10.2	2.23 c-f 5.28 a-e	1.74 c-g 2.77 abc	1,200 bcd 7,200 a
AG-5B	Stage II Sludge	1.0	Ν	IF-1m	Well water	9.5	1.97 c-f	1.45 c-g	400 d
AG-5C [†]	Stage II Sludge	10.0	С	IF-1m	Well water	10.3	5.42 a-d	3.81 a	2,000 b
Silty Sand				IF-1m IF-2d0	Well water Undiluted Stage II+	12.7 12.3	1.20 ef 1.70 c-f	0.50 fg 1.19 d-g	200 d 1,200 bcd
	LSD (P	=0.10 and 0.03	5)			-	4.17*	1.53**	-

Table 68. Summary of Terminal Root Length and Root Biomass Measurements.

*Mean values followed by the same letter do not differ significantly at the 0.10 probability level.

**Mean values followed by the same letter do not differ significantly at the 0.05 probability level

LSD – Least Square Differences which characterizes the minimum difference between statistically significant differences in measurement or rating at the 95% confidence level.

[†]AG-5C was initially water logged and was allowed to dry and pulverized to reestablish the growth media.

PRACTICAL FINDINGS OF GREENHOUSE STUDIES

Results of the greenhouse studies indicate that bermudagrass and bahiagrass grown in sandy soil can be irrigated with pH 7.5 effluents from diluted single-stage and double-stage lime treatment of phosphogypsum industry process water. A similar conclusion had been reported by Fuleihan and Werner (2007) when irrigating bermudagrass grown in sandy soils with diluted double-lime treated process water from the Piney Point site. That FIPR report titled "Plant Growth Study Using Irrigation Fluids Derived from Treatment of Process Water from the Piney Point Phosphogypsum Stack System," Florida Institute of Phosphate Research, FIPR Project Number: 00-03-143S, is available from the FIPR Institute upon request. In general, bermudagrass exhibited higher quality than bahiagrass when irrigated with lime-treatment effluents.

In addition, the current studies also indicate that bermudagrass can successfully grow in leached and unleached phosphogypsum media amended with dolomitic limestone and irrigated with pH 7.5 effluents from diluted single-stage and double-stage lime treatment of process water. In particular, bermudagrass exhibited reasonable quality when irrigated with Stage II+ (pH 7.5) effluent diluted with fresh water at a ratio of about 3:1.

Seashore paspalum can grow in sandy soil when irrigated with Stage I (pH 5) effluent from lime-treatment of process water. However, it appears that this-type turfgrass will exhibit very limited quality when grown in unleached gypsum amended with dolomitic limestone and irrigated with Stage I (pH 5) effluent.

Amendment of leached and unleached gypsum with Stage II and single-stage (pH 7.5) sludges resulting from lime-treatment of process water appear to be suitable options for growing bermudagrass. Application rates of up to about 1-6% of Stage II sludge by dry weight appear to be suitable for amending leached and unleached gypsum, respectively. In the subject study, an application rate of about 2.5% of single-stage (pH 7.5) sludge was effective for amending leached gypsum. On the other hand, results of the studies indicate that phosphogypsum amendment with lime-treatment effluents will not be as suitable for turfgrass growth. Moreover, it is not practical to amend unleached phosphogypsum with relatively large percentages of Stage II sludge (i.e., in excess of about 6%) as the media will display excessive moisture for turf grass growth to occur.

Overall, quality and biomass of the turfgrasses were relatively higher during the early period of irrigation with lime-treatment fluids containing remnant nutrients such as nitrogen. The quality and biomass, however, declined substantially with time when irrigation continued with the higher-conductivity undiluted fluids, such as undiluted Stage II (pH 7.5) and single-stage (pH 7.5) effluents. In actual spray irrigation applications, rates could be adjusted to achieve a target dilution ratio in response to rainfall seasonal patterns. In addition, alternating irrigation with fresh water and lime-treatment effluents could be implemented to preclude elevated electric conductivity of the growth media.

Relatively high fluoride concentrations were measured on turfgrass treated with higher conductivity fluids (e.g., single-stage effluent). Rainfall leaching will need to be evaluated in longer-term studies to moderate salt accumulation and reduce fluoride concentrations, particularly if the resulting biomass is intended to be used in feeding grazing animals.

Results of the studies also indicate that grasses irrigated with low-nutrient, lowconductivity effluents (i.e., 3:1 and 10: ratio diluted double lime treatment effluents) did not adversely affect root or shoot biomass compared to grass irrigated with fresh water. The lower grass quality observed at the end of the studies was primarily attributed to lack of nutrients. Results of the Gainesville and Milton studies suggest that additional fertilization would be needed to sustain healthy turf grass for longer periods of time.

EVALUATION OF ALTERNATIVE DISPOSAL METHODS

Alternative disposal methods for process water lime-treatment sludge and treated effluent were evaluated with the objective of reducing on-land disposal within sedimentation ponds and treated effluent discharges to surface waters. In addition to conventional lime sludge disposal, the following alternative disposal methods were evaluated: (i) dissolution of lime sludge in acidic process water; (ii) recycling lime-treatment sludge within phosphogypsum stack systems by co-disposal of gypsum and sludge within the stack; (iii) amendment of phosphogypsum with lime sludge to promote grass growth on the phosphogypsum stack slopes during closure; (iv) irrigating turf grass with Stage II effluent to reduce discharges to surface water; and (v) using lime-treatment effluents in spray evaporation systems to reduce the need for fresh water resources that would otherwise be required for dilution of the treated effluents prior to discharge.

CONVENTIONAL SLUDGE DISPOSAL METHODS

Conventional double lime-treatment sludge disposal typically involves discharging underflow from the Stage I and II treatment processes into Stage I and II settling ponds for clarification of the treated process water and sedimentation and settling of the sludges for permanent storage and disposal.

In general, the coarser Stage I sludge will settle at higher rates than the finer Stage II sludge. As deposition continues into each corresponding settling pond, the sludges undergo self-weight consolidation, with the usually coarser Stage I sludge consolidating at a faster rate than the Stage II sludge. Typically, the Stage I sludge settling ponds are likely to contain material with higher solids contents, higher hydraulic conductivity, and higher undrained strength than the Stage II sludge. It is generally expected that for similar treatment rates, the storage capacity of an equal-size settling pond filled with Stage II lime sludge will be depleted sooner than when filled with Stage I sludge. The settling, consolidation, hydraulic conductivity and strength characteristics of the Stage I lime sludge will also result in a somewhat less demanding closure effort of a Stage I sludge pond than a Stage II sludge pond.

As illustrated in Figure 2, a "typical" phosphogypsum stack closure would require the treatment of 3.6 billion gallons of acidic process water (combined drainable pore water and ponded water) characterized by an elevated TDS concentration, say on the order of 40,000 mg/l. In addition, rainfall infiltration through the closed slopes of the stack during the 50-year long term care post-closure period could contribute as much as 0.8 billion gallons of additional lower TDS leachate, characterized say by a TDS concentration on the order of 10,000 mg/l. From the experimental results in Figure 17, Stage I lime treatment is expected to generate 300 lb of Stage I sludge with treatment of each 1000 gal of elevated TDS process water (i.e., 300 lb/1000gal), and only 10 lb/1000 gal of Stage I sludge with the lower TDS rainfall leachate. In addition, experimental results in Figure 20 suggest that as much as 550 lb/1000 gal and 100 lb/ 1000 gal of combined Stage I + Stage II sludges would be generated

through double lime treatment of the 40,000 mg/l and 10,000 mg/l TDS waters, respectively. Hence, with these TDS process waters, Stage II sludges would be generated at rates of 250 lb /1000 gal and 90 lb /1000 gal, respectively. Considering typical *in situ* solids contents of 25% for the Stage I sludge and 12.5% for the Stage II sludge, corresponding storage capacity requirements would be on the order of 1,330 acre-feet for the Stage I settling pond and 2,625 acre-feet for the Stage II settling pond. Settling area footprints on the order of 50 and 90 acres, respectively, would be needed considering 30 to 35-foot high retaining dikes with some allowance for freeboard and for sludge settling/decanting.

Construction and operation costs associated with building and operating the sludge settling areas, and reclamation costs prior to abandonment need to be factored in when evaluating alternative disposal methods. Feasible alternatives for reclamation of a sludge settling pond include: (i) possibly closing a Stage II sludge pond as a wetland or a lake after making allowance for leaching of the sludge; and (ii) conventionally closing the pond as an upland area. For the case of upland closure, it is desirable that a relatively low water level be maintained when feasible during the life of the pond to promote periodic desiccation of the sludge. Sludge surfaces are typically regraded during closure using very low ground pressure equipment as needed to provide sheetflow of runoff. Considering the relatively low undrained strengths of the lime sludge, dewatering of the upper portion of the sludge in the settling pond will be required to promote further surface desiccation and consolidation of the sludge deposit (e.g., through the use of dewatering ditches or a drain system). Gradual placement of soil cover and mixing of the sludge surface with soil may be necessary to allow access for construction equipment in developing a network of dewatering ditches. Turf grass is usually seeded in sludge surface areas where natural vegetation has not grown. Gradually capping a sludge pond in this manner is tedious and time consuming. Expedited closure of a sludge pond may be undertaken using high tensile strength woven geotextile fabric to control and limit mudwaving of the sludge during placement of the soil cover. Associated costs with such a closure scheme can be significant.

Special operational measures will need to be implemented for cases in which disposal of lime sludge is undertaken in lined ponds atop closed gypsum stacks. In particular, extreme care will need to be exercised when operating construction equipment in the vicinity of the installed liner. Moreover, dewatering of the lime sludge may present additional challenges when deposited hydraulically in the "bathtub" formed by the lined compartment.

For the "typical" phosphogypsum stack example used above to illustrate sludge storage requirements, more than 3.5 billion gallons of Stage II effluent would have to be significantly diluted with fresh water resources prior to discharge to surface waters of the State (as explained in a prior subsection titled "Surface Water Discharge Issues" within the main section titled "Introduction and Research Objectives").

ALTERNATIVE SLUDGE DISPOSAL METHODS

Solubility of Lime Sludge in Process Water

Laboratory testing was undertaken to determine the effects of adding lime sludge to acidic process water of an operating plant, or to ponded water within a phosphogypsum stack system undergoing closure, as an alternative to optimize use of the neutralization capacity of the sludge, thus reducing subsequent Stage I lime-treatment requirements. Another benefit of this scheme during closure is that it provides for disposal of the sludge (e.g., dredged from a nearby settling area) within the cooling pond system in conjunction with filling of the cooling pond in preparation for closure.

Results of solubility tests indicated that some of the Stage I and II lime sludge constituents dissolve and/or react with acidic process water by increasing the pH and reducing the conductivity of the process water, depending on the lime sludge loading rate and the quantity of free lime available in the sludge.

At low loading rates of 5% or less (see Figure 12 and Table 29), the pH and conductivity of the process water remains relatively unchanged. At relatively high loading rates, the pH can be increased significantly, particularly using Stage II lime sludge (which is likely to contain more free lime than Stage I sludge). Note that in order to get full benefit of the neutralization, the sludge needs to be mixed vigorously with the process water (e.g., by discharging the slurried-sludge outflow from the dredge into a flowing process water ditch discharging into the cooling pond).

At operating plants, where raising the pH of the process water may not be desirable from an operation standpoint, relatively low loading rates of 5% may be feasible as long as the facility can weather adverse impacts on process water balance and available surge storage capacity. At facilities where an entire phosphogypsum stack system is being closed, the data suggest that both Stage I and Stage II lime sludge can be beneficially used to assist in neutralizing acidic process water prior to treatment, thus reducing the quantity of lime needed for treatment. Settled solids resulting from the neutralization process will need to be contained along with settling of the reacted sludge. It is expected that after reacting with process water, the volume of sludge will not change substantially, particularly at low loading rates. In such a scheme, the settled sludge can be advantageously used in filling a below grade cooling pond system in preparation for closure.

Particle-size analyses indicate that there is no significant variation in the particle size distribution of the Stage I sludge after reacting with process water. On the other hand, reaction of Stage II sludge with process water resulted in somewhat coarser Stage II sludge particle sizes. Coarser than typical sludge particles may be advantageous for co-disposal with gypsum slurry to reduce the potential segregation during settling within rim-ditches.

Disposal with Gypsum Slurry

Lime sludge particles are finer than phosphogypsum particles. Hence, segregation of lime sludge from phosphogypsum during settling within rim-ditches or compartments atop a stack could potentially adversely impact wet-stacking construction operations and slope stability. Further, lime sludge will react with acidic process water, and, as a result, some lime sludge solids will dissolve, and some solids will change in chemical and physical composition. Depending on contact time, the settling behavior of lime sludge-gypsum mixtures in rim ditches may differ from the settling behavior in a stack compartment. Settling tests were used to give an indication of how lime sludge-gypsum mixtures may tend to segregate in a rim ditch after partially reacting with acidic process water in the slurry tank and slurry pipeline. These tests were evaluated in light of possible impacts on wet-stacking operations, life of the stack, plant water balance and treatment costs.

A series of settling tests were performed on: (i) phosphogypsum samples from selected plants; and (ii) mixes of either Stage I or Stage II lime-treatment sludges and phosphogypsum from the corresponding plant site (in as much as possible) at selected percent addition and target initial slurry solids contents (i.e., percent dry weight of solids to total weight of slurry). The initial solids contents were selected to be in general agreement with typical values used in the industry for gypsum slurry disposal. The settling tests were performed in graduated plexiglass settling columns. After preparing a sample to the desired initial solids content, the slurry mixture was poured into the column to a desired initial sample height. The sludge slurry was thoroughly mixed after placement within the column with a hand-held stirrer to provide a homogeneous sample, and remove any segregation of particles which occurred during placement of the slurry into the column. The columns were securely covered with clear plastic wrap to prevent evaporation of the supernatant fluid during the test period. The settling tests were performed in a fluorescent-lighted laboratory and were not exposed to direct sunlight.

Performance of the settling tests consisted of visually monitoring the height of the mixed slurry-supernatant interface versus time. Depending on the behavior of the mixture, initial readings were obtained of height versus time in the range of one reading every 1 to 10 minutes. Subsequent readings were obtained at increasing time intervals. The tests were continued for a period of at least 24 hours, or until the settled height remained constant.

Summary of settling test results for phosphogypsum and phosphogypsum/limetreatment sludge mixtures is presented in Table 69. Settling curves are presented in Appendix E.

The initial settling velocity (i.e., the initial slope of the settled height versus time curve) for the gypsum samples ranged from 0.50 to 2.60 cm/min for initial solids contents ranging from 22.2-28.0%. In general, the initial settling velocity of the slurry reduced with the addition of lime-treatment sludges, with the exception of Stage I sludge from Plant N mixed with gypsum from Plant W (Mosaic New Wales Plant) at initial solids contents of about 28%. Addition of Stage II sludge at the upper bound limit of 8% (dry weight basis) resulted in larger initial settling velocity reduction compared to Stage I sludge at similar percent addition.

Phosphogypsum and Process Water Source	Process Water		Sludge	Sludge	Settling Column	Initial Sample	S _i	Percent Sludge (Dry	V _{si}	Final Settled Solids	S _f	$\gamma_{\rm df}$
	pН	Conductivity (µmhos/cm)	Source	туре	(cm)	Height	(%)	Weight Basis) (%)	(cm/min)	Height (cm)	(%)	(10/IT ⁻)
			NI/A	NI/A	10.1	27.0	22.2	0.0	2.60	7.0	55.9	53.6
			IN/A	IN/A	10.6	27.0	27.0	0.0	1.29	8.9	56.3	53.7
					10.4	22.1	22.2	0.9	0.41	5.6	61.8	58.7
	1.7	26,100	PLANT B	Stage I	10.4	22.5	22.3	2.6	0.31	5.8	61.5	57.9
PLANT B					10.1	24.0	22.7	8.2	0.43	7.4	56.4	49.0
					10.1	22.1	27.0	0.5	0.22	5.9	57.4	77.5
				Stage II	10.4	22.5	26.8	0.9	0.75	7.0	62.7	60.6
					10.7	23.2	26.4	2.7	0.54	6.7	64.4	64.5
					10.7	26.0	25.3	8.3	0.04	10.7	49.2	43.2
			N/A	N/A	10.7	27.0	23.3	0.0	0.75	9.2	44.4	39.1
			IN/A	IN/A	10.7	27.0	28.0	0.0	0.50	11.2	46.4	41.3
PLANT W	1.2	48,000			10.6	21.5	28.0	0.9	0.50	8.9	47.4	44.2
			PLANT N	Stage I	10.7	22.0	28.1	2.5	0.48	9.1	55.7	51.3
					10.4	23.0	28.4	8.0	0.40	9.7	55.1	51.1
Where: S _i = Initia N/A: Not Applica	Where: $S_i = \text{Initial solids content}$; $V_{si} = \text{Initial settling velocity}$; $S_f = \text{Final settled solids content}$; $\gamma_{df} = \text{Final dry density}$ N/A: Not Applicable, i.e., no sludge added to gypsum.											

Table 69. Summary of Settling Test Results for Phosphogypsum-Lime Sludge Mixtures.

A reduction in the initial settling velocity will impact typical rim ditch operations of gypsum stacks, as relatively fewer solids will likely be accumulated in the rim ditch for subsequent excavation. The finer solids will probably have a tendency to remain in suspension, at least during initial settling. Substantial settling of this-type mixtures will require more time than slurry exhibiting higher settling velocities, which would likely affect the time period necessary for operation equipment to start excavating settled material from the rim ditch (or the settling compartment) during stacking operations and the time needed for the material to dewater prior to use in raising the dikes in conjunction with the upstream method of construction.

In general, at low concentrations (i.e., additions of less than about 3%, dry weight basis) the final settled solids content increased with the addition of lime-treatment sludge. At higher concentrations of about 8%, the Stage II sludge addition resulted in a decrease of settled solids content indicating that such percent addition will adversely impact the settling characteristics of the slurry and the engineering properties of the mixture.

Mixtures of gypsum and lime-treatment sludge exhibiting higher settled solids content than settled gypsum with no sludge addition will result in greater volumes of decant water that will need to be considered in a facility's water balance (in addition to the water introduced with the sludge). An operating plant site with a positive water balance (i.e., a plant where the net water inputs exceed the net water consumption) will be adversely impacted by the additional water volume that would need to be consumed (e.g., by treatment, or spray irrigation) or stored prior to consumption (e.g., in settling compartments or surge ponds). On the other hand, the additional decant water could be re-circulated and re-used in the plant processes for a facility having a negative water balance (i.e., a facility where net water consumption exceeds the net water inputs) with the added benefit of recovering some of the phosphate from the sludge (i.e., improved P_2O_5 recovery).

Based on the settling data shown in the figures included in Appendix E (and also in Figures 13 and 14), very little segregation of Stage I sludge appears to occur for sludge addition rates lower than about 2.5% (particularly when using gypsum and process water from Plant W). Significant segregation appears to occur when adding either Stage I or Stage II sludge in excess of about 2.5%. In particular, the data suggest that Stage II sludge addition up to 1% may not promote segregation of the slurry.

In general, lime-treatment sludge co-disposal with gypsum will result in a slight reduction of the storage life of a gypsum stack. Moreover, the process water treatment cost may be affected by co-disposal of lime-treatment sludges with gypsum during plant operation depending on the settled solids content of the mix and its dewatering characteristics. Higher settled solids content after co-disposal will result in larger volumes of decant water that may need to be treated if a facility with a positive water balance has no additional compartments or ponds for excess water storage and means for future consumption of the excess water. Depending on the dewatering characteristics of the settled mix, the treatment cost could potentially be reduced if the finer lime-treatment sludge reduces to some extent the volume of drainable pore water. In summary, co-disposal of lime-treatment sludge with phosphogypsum slurry atop a gypsum stack may be feasible when adding (dry weight basis): (i) up to 2.5% of Stage I sludge; and (ii) up to 1% Stage II sludge to gypsum slurry mixed to initial solids contents on the order of 20-30%. However, additional testing will be needed to determine sludge codisposal rates that can be tolerated by any given facility from a stability standpoint, and preclude adverse impacts on handling, dewatering and compaction characteristics of the gypsum.

Amending Phosphogypsum with Lime Sludge to Promote Grass Growth

Amendment of leached and unleached phosphogypsum with Stage II Sludge (and/or with single-stage pH 7 sludge) appears to be a suitable option for promoting grass growth on gypsum stack slopes in lieu of using commercially available dolomitic limestone. Application rates of about 1% and up to 6% of Stage II sludge (dry weight basis) appear to be suitable for amending leached and unleached gypsum, respectively, if the sludge contains some free lime. For an *in situ* gypsum dry density of 75 pcf, these percentages correspond to the addition of about 8 to 45 tons per acre of Stage II sludge (dry weight basis) to amend the upper 6 inches of gypsum stack slopes. For a Stage II solids content of 20%, and corresponding dry density of 15 pcf, it would be necessary to spread and then gradually mix a layer of Stage II sludge about 0.3 to 1.7 inches thick into the upper 6 inches of the surface of gypsum stack slopes.

Considering a typical phosphogypsum stack with 200 acres of slope area, and a gross average application rate of 1 inch of Stage II sludge at a solids content of 20% (dry density of 15 pcf), amendment of the gypsum slope area to promote grass growth would consume about 15 acre-feet of Stage II sludge (5,445 dry tons) or only 1% of the volume of Stage II lime sludge produced during closure of a typical phosphogypsum stack system.

ALTERNATIVE LIME-TREATMENT EFFLUENT CONSUMPTION

The following sections discuss potential alternatives to surface water discharge of lime-treatment effluents to reduce or eliminate long-term reliance on continued use of substantial freshwater resources for dilution of treated effluent prior to discharge.

Amending Phosphogypsum with Stage II Effluents

The agronomic screening test results have indicated that Stage II effluents do not have adequate buffering capacity to neutralize the acidity in unleached phosphogypsum, although Stage II effluents could be used to amend and sweeten leached phosphogypsum to achieve the target pH of 4.8 to 5.2 needed to promote grass growth. Nevertheless, results of the greenhouse study indicate that the media consisting of phosphogypsum amended with Stage II effluents will not be very suitable for healthy turfgrass growth. Lime-treatment effluents for phosphogypsum amendment will, therefore, have very limited use in stack slope closure applications.

Spray Irrigation of Turfgrasses

Based on results of the greenhouse study, both bermudagrass and bahiagrass grown in sandy soil can be irrigated with effluent from either pH 7 single-stage treatment or conventional Stage II double-lime treatment effluent (diluted or undiluted). This finding is consistent with the conclusion reported by Fuleihan and Werner (2007), which investigated irrigation of bermudagrass grown in sandy soils with the diluted double-lime treatment effluent obtained from the Piney Point phosphogypsum stack system. The current study has also indicated that bermudagrass can successfully grow in both leached or unleached phosphogypsum media, properly amended with dolomitic limestone or with single-stage (pH 7.5) or Stage II lime-treatment sludges, and irrigated with effluents from diluted single-stage and Stage II lime treatment of process water. The study also indicated that seashore paspalum turfgrass can grow reasonably well in sandy soil when irrigated even with the more acidic Stage I (pH 5) effluent.

In general, the quality and density of turfgrass were relatively better during early stages of irrigation with lime-treatment effluents containing remnant nutrients such as nitrogen. The quality and density declined substantially, however, when irrigation continued with undiluted effluents, likely due to accumulation of salt in turfgrass growth media. Steady declines in the turfgrass quality were also observed over the study period when irrigated with diluted lower-conductivity, lower-nutrient effluents, likely due to a lack of nutrients over time. The decline in quality, however, did not appear to be any worse than that exhibited by grass irrigated with freshwater.

Based on the findings of this study and barring restrictions imposed by surface water runoff and groundwater quality requirements, recycling of effluents generated by lime-treatment of the process water to irrigate the grass cover on a closed phosphogypsum stack or grass pasture nearby appears to be technically viable. Therefore, spray irrigation, if properly managed, can be a feasible alternative to surface water discharge for "consumption" of treated process water. Of particular interest is the finding that grasses irrigated with effluents from conventional two-stage lime treatment up to a neutral pH on the order of 7.5 can sustain reasonable long-term vigor and health provided the application is controlled at rates that afford long-term dilution via rainfall leaching at dilution ratios greater than 3:1. Since the grasses appear to react reasonably well to irrigation with undiluted Stage II effluent over a short duration, a longer-term target "dilution" ratio can be achieved with the higher TDS effluents by adjusting spray irrigation rates and schedules in response to seasonal and cyclical rainfall patterns as needed to preclude elevated electric conductivity of the growth media.

In terms of turfgrass health, the success of a spray irrigation system will depend heavily on the ability to manage long-term effects on soil conductivity. For instance, the application rate may need to be limited in order to maintain healthy grass which in turn provides higher evapotranspiration potential that in the long term will enable higher consumption of treated water. Other factors affecting irrigation rates from a turfgrass health standpoint relate to the characteristics of the treated process water and turfgrass growth media. For instance, the allowable application rate for treated water from an operating facility would likely need to be somewhat lower than from an idled facility where the process water has diluted over time. Similarly, the application rate may need to be lower for turfgrasses grown on unleached gypsum media than for grasses grown on amended leached gypsum media.

Another major consideration that may limit spray irrigation rates is related to surface and groundwater compliance issues in and around the perimeter of the land application area. In Central Florida, rainfall averages about 54 inches per year. For average climatic conditions (temperature, relative humidity, etc.) and typical hydrogeologic conditions, natural evapotranspiration is on the order of 37 inches per year and potential evapotranspiration from turfgrass is on the order of 62 inches per year. The difference between these values, after accounting for infiltration, base flow and runoff is about 26 inches per year on average (see Figure 67). This gross irrigation requirement or safe average irrigation demand corresponds to about 0.5 inches of spray irrigation per week or about 1.3 gpm per acre. Large tracts of land will, therefore, be required to consume the treated water via spray irrigation. For example, consumption of a typical rate of 1,000 gpm of effluent will require a minimum 800 acres of irrigable land area.

Because the concentrations of sodium (~800 mg/l) and sulfate (2,500 to 5,000 mg/l) in the lime-treated water exceed the corresponding Class G-II groundwater primary and secondary drinking water standard MCLs (160 mg/l for sodium and 250 mg/l for sulfate, respectively), significant dilution with groundwater (and/or rainwater) is needed to reduce sodium concentrations (by a factor of 5) and sulfate concentrations (by a factor up to 20) in order to ensure compliance with groundwater standards at the edge of the regulated zone of discharge. Moreover, without dilution/dispersion, any seepage outcrops into adjacent relief ditches of wetlands may exhibit elevated specific conductance in excess of the 1275 µmhos/cm Class III surface water standard. On the other hand, assuming nitrogen consumption of 300 lb/acre/year, and considering that the spray irrigation rate is limited to about 0.5 to 0.2 inches per week (i.e., 1.3 to 0.5 gpm/acre) or less, approximately 50 mg/l to 125 mg/l of the nitrogen concentration is expected to be readily consumed by the grass (i.e., without reliance on dispersion, dilution or other attenuation mechanisms). Similarly, assuming phosphorus uptake of 50 lb/acre/year, the corresponding consumption of phosphorus by the grass is expected to be on the order of 10 mg/l to 25 mg/l. From the nutrient uptake standpoint, therefore, the post-aeration and acidulation Stage II+ (pH 7.5) effluent would be better suited for spray irrigation, because the fluid will be lower in phosphorus. For high nutrient effluents, such as Stage II (pH 7.5) or single-stage (pH 7.5) effluents, larger dilutions with freshwater (e.g., rainfall) and/or larger areas will be required in order to achieve nutrient reduction via grass uptake to reasonable levels.

Management tools that can be implemented to minimize impacts to groundwater and surface water and to preclude salt accumulation in the soil include: (i) dilution of the treated

effluent with another "fresh water source" (e.g., R.O. permeate); (ii) rotational irrigation or use of alternating active and dormant plots for land application; (iii) reducing the spray irrigation rate to a fraction of the "safe average irrigation demand" (e.g., to less than 0.2 to 0.3 inches per week, or 0.5 to 0.8 gpm/per acre) to provide for adequate dilution by rainfall; (iv) maintaining a safe buffer zone between the land application area and the compliance point at the edge of the zone of discharge; and/or (v) modifying the application rate as needed based on results of water quality monitoring data in a downgradient seepage collection relief ditch/drain (see Figure 67) or downgradient monitor well.

Because much of the remnant nitrogen, including unionized ammonia (and some of the phosphorus) are effectively "removed" by root uptake, Stage II+ treatment with air stripping at high pH, may not be needed if spray irrigation can be implemented as an alternative to surface water discharge. Stage II (pH7.5) effluent may then be directly used for spray irrigation unless a higher level of treatment is needed (say to pH9) to further reduce remnant arsenic concentrations, inorganic constituents and conductivity from a groundwater or surface water compliance standpoint. Note also that as a result of appeals by various groups (including the Florida Department of Environmental Protection, FDEP) to rescind the numeric nutrient criteria proposed by the United States Environmental Protection Agency (USEPA) and the fact that the FDEP is in the process of finalizing its own equivalent criteria, it is unclear at this time to what extent the process water will have to be treated and polished prior to discharge. Nevertheless, if it can be implemented, spray irrigation would provide a substantial benefit in that reliance on valuable fresh water resources for dilution for the sole purpose of achieving Class III surface water standards prior to discharge could be substantially reduced or eliminated. Hence, the consumptive use of fresh groundwater used for dilution would be significantly reduced, and the precious water resource saved for other beneficial uses. Moreover, if spray irrigation can be implemented with the reduced Stage II (pH7.5) level of treatment, significant treatment cost savings would be realized.

The extent to which potential benefits may be realized would depend on the available land area for spray irrigation and the treatment rate employed for execution of a facility's water management plan. In some cases, such as during the post closure care period of an idled facility (particularly the latter years once pore water drainage rates from the closed gypsum stack system have declined substantially and the water quality has improved), spray irrigation could be very effective from cost and resource conservation perspectives. Another major benefit is the reduction in nutrient loadings to sensitive waters of the State.

Spray Evaporation

If the land areas available for spray irrigation of turfgrass are limited and/or the quality of surface water runoff from the irrigated areas is a concern, water consumption by spray evaporation in self-contained area(s) could be a viable option for disposing of lime-treatment effluents. This practice has recently been one of the key factors in the successful closure of two phosphogypsum stack systems in Central Florida.

A typical spray evaporation system consists of a series of header pipes with risers with fine spray nozzles, arranged in a grid system. The risers are generally spaced 10 to 15 feet on-center along each header pipe. The header pipes are typically spaced 100 feet apart. Water is pumped to the spray nozzles at 50 to 70 psig pressure (at the nozzles). As a result, a stream of water is discharged into the air at high velocity. Friction between the air and water causes the formation of water droplets of varying sizes. With proper design and depending on the size of the droplets and their distribution patterns and pressure at the nozzle, evaporation of water could amount to as much as 10% of the inflow rate for typical Central Florida climatic conditions. The smaller the droplet, the more surface area there is for evaporation occurs at night and early morning. Another important factor that affects evaporation rates is wind transporting the water vapor away from the spray evaporation area. The wind helps increase evaporation by transporting drier air from adjacent areas to the spray evaporation field.

The spray evaporation system(s) can be placed on self-contained land or on ponded areas, whereby the influent water minus the evaporation plus the rain water will be collected on-site and re-sprayed. The containment dikes surrounding the system will need to be designed considering the water balance of the system to prevent overflows. If an impact to groundwater is a concern, the area(s) could be lined.

The success of a spray evaporation system depends on consistent maintenance. The small diameter spray nozzles used in the system are prone to plugging, which has to be manually cleaned, a labor-intensive task. Safety of maintenance personnel for spray system operated on ponds is another important issue with this system. Fluoride emissions could also be a limiting factor in low pH water. Nevertheless, a properly designed and maintained spray evaporation system can be very cost-effective in evaporating substantial quantity of treatment effluents, thus reducing or eliminating the need for fresh water resources for dilution of lime-treatment effluents prior to discharge.



a) Without Spray Irrigation



b) With Spray Irrigation

Figure 67. Simplified Water Balance of a Grassed Upland Area with and without Spray Irrigation.

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Appendix A

LIME SLUDGE INDEX TEST RESULTS



U.S. STANDARD SIEVE SIZE

SYMBOL	SAMPLE DESCRIPTION	-200 (%)	-5µ (%)
	FIELD: TUBE SAMPLE TH-1A US-1 (1.25')	88	25
	FIELD: TUBE SAMPLE TH-1A US-2 (4.5')	99	30
	FIELD: TUBE SAMPLE TH-1A US-3 (6.75')	91	50
	FIELD: COMPOSITE SAMPLE 1A (TH-1A; 0'-6')	86	40
0	LAB: TREATMENT EXPERIMENT (SAMPLE C-1; pH 5)	90	5

Figure A-1. Particle Size Analyses of Stage I Sludge Samples (Plant C).

U.S. STANDARD SIEVE SIZE



SYMBOL	SAMPLE DESCRIPTION	-200 (%)	-5µ (%)
	FIELD: TUBE SAMPLE TH-1A US-2 (2.0')	98	30
	FIELD: COMPOSITE SAMPLE 1A (TH-1A; 0'-3')	70	32
©	LAB: TREATMENT EXPERIMENT (SAMPLE B-1; pH 5)	91	18

Figure A-2. Particle Size Analyses of Stage I Sludge Samples (Plant B).



SAMPLE DESCRIPTION	-200 (%)	-5µ (%)
FIELD; TUBE SAMPLE TH-1B US-2 (2.0')	98	41
FIELD: COMPOSITE SAMPLE 1B (TH-1B; 0'-3')	85	44
	SAMPLE DESCRIPTION FIELD: TUBE SAMPLE TH-1B US-2 (2.0') FIELD: COMPOSITE SAMPLE 1B (TH-1B; 0'-3')	SAMPLE DESCRIPTION-200 (%)FIELD: TUBE SAMPLE TH-1B US-2 (2.0)98FIELD: COMPOSITE SAMPLE 1B (TH-1B; 0'-3')85

Figure A-3. Particle Size Analyses of Stage I Sludge Samples (Plant N).



Figure A-4. Particle Size Analyses of Stage II Sludge Samples (Plant C – Pond 4A).




SYMBOL	SAMPLE DESCRIPTION	-200 (%)	-5µ (%)
	FIELD: TUBE SAMPLE TH-2C US-1 (1.75')	93	46
	FIELD: TUBE SAMPLE TH-2C US-2 (4.25')	89	51
	FIELD: TUBE SAMPLE TH-2D US-4 (10.5')	95	33

Figure A-5. Particle Size Analyses of Stage II Sludge Samples (Plant C – Pond 4B).



Figure A-6. Particle Size Analyses of Stage II Sludge Samples (Plant C – Pond 4B).



SYMBOL	SAMPLE DESCRIPTION	-200 (%)	-5 µ (%)
	FIELD: TUBE SAMPLE TH-2A US-2 (6.0')	83	57
	FIELD: TUBE SAMPLE TH-2B US-2 (5.5')	97	38
	FIELD: COMPOSITE SAMPLE 2B (TH-2B; 3'-5')	82	67
0	LAB: TREATMENT EXPERIMENT (STAGE 2 - pH 11)	39	15

Figure A-7. Particle Size Analyses of Stage II Sludge Samples (Plant B).



SYMBOL	SAMPLE DESCRIPTION	-200 (%)	-5µ (%)
	LAB: TREATMENT EXPERIMENT (SAMPLE D-1; pH 5)	86	15
	LAB: TREATMENT EXPERIMENT (SAMPLE D-1; pH 11)	82	29

Figure A-8. Particle Size Analyses of Laboratory Stage II Sludge Samples (Plant D).



SYMBOL	SAMPLE DESCRIPTION	-200 (%)	-5µ (%)
-	FIELD: TUBE SAMPLE TH-28 US-2 (4.0')	98	30
	FIELD: COMPOSITE SAMPLE 2A (TH-2A; 1'-3')	70	32
<u></u> 0	LAB: TREATMENT EXPERIMENT (SAMPLE N-1; pH 5)	91	18

Figure A-9. Particle Size Analyses of Stage II Sludge Samples (Plant N).



Figure A-10. Particle Size Analyses of Single-Stage Sludge Samples (Plant C).



SYMBOL	SAMPLE DESCRIPTION	-200 (%)	-5µ (%)
	STAGE 1 COMPOSITE SAMPLE 1A - AS RECEIVED	86	40
0	STAGE 1 COMPOSITE SAMPLE 1A - REACTED	90	40
	STAGE 2 COMPOSITE SAMPLE 2D-2 - AS RECEIVED	68	36
&	STAGE 2 COMPOSITE SAMPLE 2D-2 - REACTED	70	29

Figure A-11. Particle Size Analyses of Unreacted and Reacted Sludge (Plant C).



Figure A-12. Particle Size Analyses of Unreacted and Reacted Sludge (Plant B).

Appendix B

LABORATORY SETTLING COLUMN TEST RESULTS ON STAGE I, STAGE II AND SINGLE-STAGE SLUDGE



Figure B-1. Stage II Sludge Height of Settled Solids with Time (Plant B).



Figure B-2. Stage II Sludge Height of Settled Solids at Initial 60 Minutes (Plant B).



Figure B-3. Stage II Sludge Percent Settling Completed with Time (Plant B).



Figure B-4. Single-Stage Sludge Height of Settled Solids with Time (Plant B).



Figure B-5. Single-Stage Sludge Height of Settled Solids at Initial 60 Minutes (Plant B).



Figure B-6. Single-Stage Sludge Percent Settling Completed with Time (Plant B).



Figure B-7. Stage II Sludge Height of Settled Solids with Time (Plant C).



Figure B-8. Stage II Sludge Height of Settled Solids at Initial 60 Minutes (Plant C).



Figure B-9. Stage II Sludge Percent Settling Completed with Time (Plant C).



Figure B-10. Single-Stage Sludge Height of Settled Solids with Time (Plant C).



Figure B-11. Single-Stage Sludge Height of Settled Solids at Initial 60 Minutes (Plant C).



Figure B-12. Single-Stage Sludge Percent Settling Completed with Time (Plant C).



Figure B-13. Stage II Sludge Height of Settled Solids with Time (Plant D).



Figure B-14. Stage II Sludge Height of Settled Solids at Initial 60 Minutes (Plant D).



Figure B-15. Stage II Sludge Percent Settling Completed with Time (Plant D).



Figure B-16. Single-Stage Sludge Height of Settled Solids with Time (Plant D).



Figure B-17. Single-Stage Sludge Height of Settled Solids at Initial 60 Minutes (Plant D).



Figure B-18. Single-Stage Sludge Percent Settling Completed with Time (Plant D).



Figure B-19. Stage II Sludge Height of Settled Solids with Time (Plant N).



Figure B-20. Stage II Sludge Height of Settled Solids at Initial 60 Minutes (Plant N).



Figure B-21. Stage II Sludge Percent Settling Completed with Time (Plant N).



Figure B-22. Single-Stage Sludge Height of Settled Solids with Time (Plant N).



Figure B-23. Single-Stage Sludge Height of Settled Solids at Initial 60 Minutes (Plant N).



Figure B-24. Single-Stage Sludge Percent Settling Completed with Time (Plant N).

Appendix C

LABORATORY CONSOLIDATION TEST RESULTS



Figure C-1. Consolidation Test Results for Stage I Undisturbed Sample (Plant B).



Figure C-2. Consolidation Test Results for Stage I Undisturbed Sample (Plant B).


Figure C-3. Consolidation Test Results for Stage I Undisturbed Sample (Plant C).



Figure C-4. Consolidation Test Results for Stage I Undisturbed Sample (Plant C).



Figure C-5. Consolidation Test Results for Stage I Undisturbed Sample (Plant C).



Figure C-6. Consolidation Test Results for Stage I Undisturbed Sample (Plant N).



Figure C-7. Consolidation Test Results for Stage I Undisturbed Sample (Plant N).



Figure C-8. Consolidation Test Results for Stage I Undisturbed Sample (Plant N).



Figure C-9. Consolidation Test Results for Stage II Undisturbed Sample (Plant B).



Figure C-10. Consolidation Test Results for Stage II Undisturbed Sample (Plant B).



Figure C-11. Consolidation Test Results for Stage II Undisturbed Sample (Plant B).



Figure C-12. Consolidation Test Results for Stage II Undisturbed Sample (Plant C).



Figure C-13. Consolidation Test Results for Stage II Undisturbed Sample (Plant C).



Figure C-14. Consolidation Test Results for Stage II Undisturbed Sample (Plant C).



Figure C-15. Consolidation Test Results for Stage II Undisturbed Sample (Plant C).



Figure C-16. Consolidation Test Results for Stage II Undisturbed Sample (Plant C).



Figure C-17. Consolidation Test Results for Stage II Undisturbed Sample (Plant C).



Figure C-18. Consolidation Test Results for Stage II Undisturbed Sample (Plant N).



Figure C-19. Consolidation Test Results for Stage II Undisturbed Sample (Plant N).



 SOURCE:
 Plant C - Stage II Pond 4B

 BORING NO:
 TH-2D (UPPER 1/3rd)

 DEPTH (tt):
 0 to 4 (Composite)

 DESCRIPTION:
 Gray sludge

PERCENT PASSING NO. 200: 90 SPECIFIC GRAVITY (Assumed): 3.00

SPECIMEN CONDITIONS	INITIAL	FINAL	
MOISTURE CONTENT (%): DRY DENSITY (Ib/ft ³): VOID RATIO:	647.9 9.2 19.44	232.1 23.5 6.96	
SATURATION (%):	100	100	

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Figure C-20. Slurry Consolidation Test Results (Plant C).



SOURCE:Plant N - Stage I PondBORING NO:TH-1BDEPTH (ft):0 to 2 (Composite)DESCRIPTION:Gray sludge

SPECIMEN CONDITIONS	INITIAL	FINAL
MOISTURE CONTENT (%):	492	169
DRY DENSITY (lb/ft ³):	11.9	30.8
VOID RATIO:	14.77	5.08
SATURATION (%):	100	100

PERCENT PASSING NO. 200: 85 SPECIFIC GRAVITY (Assumed): 3.00

Figure C-21. Slurry Consolidation Test Results (Plant N, Stage I).



	SA	١MF	٢LΕ	DA.	TΑ
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SOURCE:Plant N - Stage II PondBORING NO:TH-2ADEPTH (ft):0 to 4 (Composite)DESCRIPTION:Gray sludge

 SPECIMEN CONDITIONS
 INITIAL
 FINAL

 MOISTURE CONTENT (%):
 737
 205

 DRY DENSITY (Ib/ft³):
 8.0
 25.8

 VOID RATIO:
 22.55
 6.27

 SATURATION (%):
 98
 98

PERCENT PASSING NO. 200: 96 SPECIFIC GRAVITY (Assumed): 3.00

Figure C-22. Slurry Consolidation Test Results (Plant N, Stage II).

Appendix D

LABORATORY STRENGTH TEST RESULTS



Figure D-1. Stress-Strain Behavior of Stage I Undisturbed Samples (Plant B).



Figure D-2. Stress-Strain Behavior of Stage I Undisturbed Samples (Plant C).



Figure D-3. Stress-Strain Behavior of Stage I Undisturbed Samples (Plant N).



Figure D-4. Stress-Strain Behavior of Stage II Undisturbed Samples (Plant B).



Figure D-5. Stress-Strain Behavior of Stage II Undisturbed Samples (Plant C).



Figure D-6. Stress-Strain Behavior of Stage II Undisturbed Samples (Plant N).



Figure D-7. Stress-Strain Behavior of Stage II Undisturbed and Remolded Samples (Plant N).



Figure D-8. Effective Stress Paths of Stage I and Stage II Undisturbed Samples (Plant B).



Figure D-9. Effective Stress Paths of Stage I and Stage II Undisturbed Samples (Plant C).



Figure D-10. Effective Stress Paths of Stage I and Stage II Undisturbed Samples (Plant N).



Figure D-11. Effective Stress Paths of Stage II Undisturbed and Remolded Samples (Plant N).

Appendix E

LABORATORY SETTLING COLUMN TEST RESULTS ON GYPSUM AND LIME-TREATMENT SLUDGE MIXTURES



Figure E-1. Gypsum and Stage I Sludge Mixtures Height of Settled Solids with Time (Plant B Gypsum, Process Water and Sludge).



Figure E-2. Gypsum and Stage I Sludge Mixtures Height of Settled Solids at Initial 60 Minutes (Plant B Gypsum, Process Water and Sludge).



Figure E-3. Gypsum and Stage I Sludge Mixtures Height of Settled Solids with Time (Plant W Gypsum, Process Water and Sludge).


Figure E-4. Gypsum and Stage I Sludge Mixtures Height of Settled Solids at Initial 60 Minutes (Plant W Gypsum, Process Water and Sludge).



Figure E-5. Gypsum and Stage II Sludge Mixtures Height of Settled Solids with Time (Plant B Gypsum, Process Water and Sludge).



Figure E-6. Gypsum and Stage II Sludge Mixtures Height of Settled Solids at Initial 60 Minutes (Plant B Gypsum, Process Water and Sludge).