

AGRONOMIC AND ENVIRONMENTAL CHARACTERIZATION OF PHOSPHORUS IN  
BIOSOLIDS PRODUCED AND/OR MARKETED IN FLORIDA

By

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To Mom, Dad, Amanda, Chris, and Richelle.

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## TABLE OF CONTENTS

	<u>page</u>
ACKNOWLEDGEMENTS.....	4
LIST OF TABLES.....	7
LIST OF FIGURES.....	8
ABSTRACT.....	12
CHAPTER	
1 INTRODUCTION.....	14
Hypotheses and Research Objectives.....	17
Study Approach.....	18
2 MATERIALS AND METHODS.....	19
Materials.....	19
Year 1 Glasshouse and Static Incubation Studies.....	19
Laboratory Characterization.....	20
Dynamic Laboratory Incubation.....	23
Glasshouse Study.....	26
Statistical Analysis.....	29
3 RESULTS AND DISCUSSION.....	31
Laboratory Characterization.....	31
Dynamic Laboratory Incubation.....	35
Glasshouse Study.....	42
Yields.....	43
Phosphorus Uptake and Relative Phytoavailability.....	45
Phosphorus Leaching.....	49
4 CONCLUSIONS.....	70
Dynamic Laboratory Incubation.....	70
Glasshouse Study.....	72
Land Applying BPR or BPR-like Residuals.....	74
GLASSHOUSE ANOVA AND CUMULATIVE DATA TABLES.....	76

LIST OF REFERENCES.....82

BIOGRAPHICAL SKETCH.....85

## LIST OF TABLES

<u>Table</u>		<u>page</u>
2-1	Selected properties of Immokalee fine sand.....	30
3-1	Materials received for laboratory characterization.....	51
3-2	Selected general chemical properties of materials.....	52
3-3	Selected phosphorus properties of materials.....	53
3-4	Comparison of TP concentrations determined by 3 digestion methods.....	54
3-5	Dynamic laboratory incubation: cumulative SRP released and SRP released (% of applied P).....	55
3-6	Cumulative Bahiagrass tissue P concentrations after 4 harvests.....	65
3-7	Relative phosphorus phytoavailability of biosolids to TSP. ....	66
3-8	Various RPP estimate model fit statistics.....	67
3-9	Point estimates of RPP for Milorganite and Lakeland NS biosolids.....	69
A-1	Cumulative Bahiagrass P uptake after 4 harvests ANOVA.....	76
A-2	Cumulative Bahiagrass yield after 4 harvests ANOVA.....	77
A-3	Cumulative P leached in glasshouse experiment ANOVA .....	78
A-4	Cumulative Bahiagrass yields after 4 harvests.....	79
A-5	Cumulative P uptake for Bahiagrass after 4 harvests.....	80
A-6	Cumulative P leached after 4 leachings.....	81

## LIST OF FIGURES

<u>Figure</u>	<u>page</u>
3-1a P rate: 56 kg P ha <sup>-1</sup> . Cumulative SRP released (% of applied P) as a function of P source.....	56
3-1b P rate: 224 kg P ha <sup>-1</sup> . Cumulative SRP released (% of applied P) as a function of P source.....	56
3-2a P rate: 56 kg P ha <sup>-1</sup> . Dynamic laboratory incubation: cumulative P released (% of applied P) as a function of biosolids-PWEP.....	57
3-2b P rate: 224 kg P ha <sup>-1</sup> . Dynamic laboratory incubation: cumulative P released (% of applied P) as a function of biosolids-PWEP.....	57
3-3a P rate: 56 kg P ha <sup>-1</sup> . Dynamic laboratory incubation: cumulative P released as a function of biosolids-PSI.....	58
3-3b P rate: 224 kg P ha <sup>-1</sup> . Dynamic laboratory incubation: cumulative P released as a function of biosolids-PSI.....	58
3-4a P rate: 56 kg P ha <sup>-1</sup> . Dynamic laboratory incubation: cumulative P released (% of applied P) per released event.....	59
3-4b P-Rate: 224 kg P ha <sup>-1</sup> . Dynamic laboratory incubation: cumulative P released (% of applied P) per released event. ....	60
3-5a P rate: 56 kg P ha <sup>-1</sup> . Cumulative Bahiagrass yields after 4 harvests.....	61
3-5b P-rate: 112 kg P ha <sup>-1</sup> . Cumulative Bahiagrass yields after 4 harvests.....	61
3-5c P rate: 224 kg P ha <sup>-1</sup> . Cumulative Bahiagrass yields after 4 harvests.....	62
3-6 Cumulative Bahiagrass yield-weighted tissue P concentrations after 4 harvests.....	62
3-7 P rate: 224 kg P ha <sup>-1</sup> . Bahiagrass N tissue concentrations in each harvest.....	63
3-8 P rate: 224 kg P ha <sup>-1</sup> . Bahiagrass yield-weighted N concentrations after 4 harvests.....	63
3-9a P rate: 56 kg P ha <sup>-1</sup> . Cumulative Bahiagrass P uptake after 4 harvests.....	64
3-9b P-rate: 112 kg P ha <sup>-1</sup> . Cumulative Bahiagrass P uptake after 4 harvests.....	64
3-9c P-rate: 224 kg P ha <sup>-1</sup> . Cumulative Bahiagrass P uptake after 4 harvests.....	64
3-1 0 Cumulative Bahiagrass P uptake as a function of P applied.....	66

3-11 Plateau model: P uptake from Milorganite treatments as a function of P applied.....68

3-12 Relative phosphorus phytoavailability (RPP) as a function of biosolids-PWEP.....69

## LIST OF ABBREVIATIONS

Al	aluminum
AN	ammonium nitrate
ANOVA	analysis of variance
BAP	biologically available phosphorus
BPR	biological phosphorus removal
C	carbon
Ca	calcium
DDI	distilled, de-ionized water
EC	electrical conductivity
FDEP	Florida Department of Environmental Protection
Fe	iron
ICP	inductively coupled plasma spectrophotometry
K	potassium
LOI	loss on ignition
Mg	magnesium
N	nitrogen
P	phosphorus
PSC	phosphorus source coefficient
PSI	phosphorus saturation index
PV	pore volume
PWEP	percent water-extractable phosphorus
RPA	relative phosphorus adsorption

RPP	relative phosphorus phytoavailability
SRP	soluble reactive phosphorus
S	sulfur
TC	total carbon
TN	total nitrogen
TP	total phosphorus
TRT	treatment
TSP	triple super phosphate
UF	University of Florida
USEPA	United States Environmental Protection Agency
WEP	water-extractable phosphorus
WTR	water treatment residual
WWTP	wastewater treatment plant

Abstract of Thesis Presented to the Graduate School  
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Land application of biosolids can result in an accumulation of soil phosphorus (P). While excess soil P is typically not harmful to crops, P can migrate offsite and can lead to surface and groundwater impairment. Increased concern over accelerated eutrophication of water bodies has led to heightened scrutiny and regulation of biosolids land application. Regulation could include limiting application rates to match crop P requirements (P-based rates). If P-based regulations are imposed, it will be critical to understand biosolids P phytoavailability and the potential for surface and groundwater impairment from biosolids P. We conducted laboratory and glasshouse studies to provide understanding. A laboratory incubation was conducted using small soil columns to assess P release potential: 11 biosolids and triple super phosphate (TSP) were individually mixed with 400 g of a typical low-P sorbing Florida soil (Immokalee fine sand) at two rates: 56 and 224 kg P ha<sup>-1</sup>.

Columns were leached bi-weekly for 14 weeks to attain 60 mL of drainage in each leaching. Soluble reactive P (SRP) was determined and summed over the 8 leachings. Cumulative P release (as a percentage of P applied) was greatest from biological P removal (BPR) and BPR-like biosolids. Phosphorus release from Milorganite, a thermally dried biosolids high in Fe and Al oxides (44 g kg<sup>-1</sup>), released ~39% of applied P at the 56 kg P ha<sup>-1</sup> rate,

indicating that P release was limited by association of P with Fe and Al and by heat drying. In contrast to the moderate P release from Milorganite, P release from the Lakeland NS biosolids, a BPR-like product with low percent solids (3%), and low Fe and Al content ( $\sim 12 \text{ g kg}^{-1}$ ), was  $\sim 90\%$  of P applied at  $56 \text{ kg P ha}^{-1}$ .

A glasshouse study utilizing large soil columns was conducted: 7 biosolids and TSP were individually mixed with 4 kg samples of Immokalee soil and Bahiagrass (*Paspalum notatum* Flugge) was grown. Bahiagrass was harvested monthly for 4 months and tissue total P (TP) was measured. Four of the biosolids chosen for the glasshouse study had high PWEF values ( $\geq 15\%$ ), and their P was expected to be highly plant available. Indeed, the OCUD S, Lakeland NS, GRU, and Boca Raton biosolids fit into the “high” category of relative P phytoavailability (RPP;  $>75\%$  of TSP). Two biosolids with low P-solubility (PWEF  $\leq 1.1\%$ ) fit into the moderate RPP category (25-75% of TSP): Milorganite, and GreenEdge. One biosolid (Disney) with a moderate PWEF (8.4%) also fit into the moderate RPP category. Results of the glasshouse study indicate that no change in biosolids application rate is needed (or justified) for BPR and BPR-like biosolids with RPP values in the high RPP category. However, application rates for Disney and GreenEdge could be approximately doubled to meet crop P needs. Phosphorus in Milorganite is only  $\sim 1/3$  as phytoavailable as P in TSP, indicating that Milorganite may be applied at P rates as great as  $224 \text{ kg P ha}^{-1}$  (based strictly on phytoavailability). Given the wide range of RPP values and leaching risks of various biosolids, land application of biosolids should not be regulated en masse. PWEF, and PSI are easily determined and excellent gauges of both biosolids agronomic value and P leaching hazard. PWEF is a better indication of how a biosolid might affect the environment than biosolids-TP or soil test P.

## CHAPTER 1 INTRODUCTION

Throughout history, human and animal wastes have been recycled to agricultural land to supply nutrients for crops. However, the development of centralized wastewater treatment systems, and an ever-increasing human population has created biosolids disposal challenges. The U.S. produced an estimated  $6.5 \times 10^6$  Mg of biosolids in 2000 and approximately 50-60% of biosolids were disposed of via land-based recycling (USEPA, 1999). Biosolids production is expected to increase to  $7.5 \times 10^6$  Mg yr<sup>-1</sup> by 2010. The state of Florida produces about  $2.7 \times 10^5$  dry Mg of biosolids per year; 66% of the biosolids are land applied, 17% are land filled, and 17% are marketed and distributed to the public (FDEP, 2005). In addition to biosolids produced within the state of Florida,  $9.1 \times 10^4$  Mg of class AA pelletized biosolids are imported each year. Given the large quantities of biosolids produced, land-based recycling of biosolids becomes a critical disposal route for municipalities. Biosolids land application can also be an environmentally sound and beneficial practice.

Biosolids are a source of essential plant elements such as N, P, sulfur, and micronutrients. Thus, land-based recycling of biosolids to agricultural land can supply farmers with an economic alternative to chemical fertilizers. Biosolids are also high in organic matter, which can be important in Florida, where soils are typically sandy and low in organic matter. While the land application of biosolids is beneficial, long-term biosolids application can result in accumulation of soil P (O'Connor et al., 2005). When biosolids are applied at an N-based rate to meet crop N requirements, P is typically oversupplied due to differences in biosolids N:P than crop needs. The excessive P accumulation it is not harmful to crops (Person et al., 1994), but leads to accumulation of P in amended soils and potential environmental problems. Migration of P off-site to ground and surface waters is a cause for concern as P is generally the limiting nutrient in

fresh water ecosystems for accelerated eutrophication (Carpenter et al., 1998). P can migrate off-site either in dissolved form, as biosolids particles, or attached to soil particles (Elliott et al., 2005). Most soils in the U.S. have sufficient P-sorbing capacity (due to Fe and Al oxides) to prevent P leaching. Elliott et al. (2002) showed that even sandy Florida soils with moderate P-sorbing capacity can prevent P leaching. In soils with adequate P-sorbing capacity, P loss occurs mostly through erosion and runoff, and erosion control measures can help control P loss in these soils. Atlantic Coastal Plain soils (including most Florida soils), however, are naturally low in Fe and Al oxides. In such soils, P leaching and loss of P dissolved in subsurface flow become the dominant P loss mechanism (He et al., 1999).

Excess P in water bodies can lead to accelerated eutrophication. Increased concern over water quality has led to amplified scrutiny of biosolids land application programs and recommendations that biosolids application be limited to meet crop P needs (P-based). Limiting biosolids land application to P-based rates would significantly reduce biosolids application rates. When application rates are decreased, more land area is required for disposal, increasing disposal costs. Lower biosolids application rates also mean additional N fertilizer is required, increasing costs to farmers, and making land-based recycling of biosolids for agronomic benefits less attractive.

Recent research suggests that limiting biosolids application to P-based rates is probably unnecessary for conventionally produced biosolids (Brandt et al., 2004). Biosolids-P is not necessarily as labile as P in mineral fertilizers or manures, and lability can be greatly influenced by the wastewater treatment process (Maguire et al., 2001). Treatment processes such as thermal drying can significantly reduce P lability of biosolids compared to conventionally produced cake (Smith et al., 2002). A possible exception may be biological P removal (BPR) biosolids, in

which the wastewater treatment process is engineered to promote luxury uptake of P by bacteria. While the BPR process reduces P concentrations in wastewater effluent, the resulting biosolids have a higher concentration of P than conventionally produced residuals, and labile P in BPR biosolids may be higher than for conventional or heat-dried biosolids (Elliott et al., 2002; Brandt et al., 2004; O'Connor et al., 2004).

Total P (TP) concentration of the biosolids is generally a poor indicator of P lability and phytoavailability. Only a small fraction of P from most conventionally produced biosolids is soluble, making most biosolids less likely to negatively impact the environment compared to soluble P sources (mineral fertilizer, manures). The water-extractable P (WEP) content of biosolids has been highly correlated to P lability (Brandt et al., 2004). For typical biosolids, percent water-extractable P ( $PWEP = WEP/TP \times 100$ ) is < 5% (Brandt et al., 2004), and phytoavailability (plant-available) P is only 40-50% of TSP (USEPA, 1995). Biosolids produced using iron (Fe) and aluminum (Al) salts may have even lower P lability and PWEP values (<0.5%) (Corey, 1992; Brandt et al., 2004). BPR biosolids may have much greater soluble P concentrations than conventionally produced biosolids, and greater PWEP (~ 14%) (Brandt et al., 2004).

The phosphorus saturation index (PSI) has also been correlated to environmental P leaching risk (Elliott et al., 2002). PSI is calculated as the molar ratio of oxalate-extractable P to Fe and Al ( $[P_{ox}]/[Al_{ox} + Fe_{ox}]$ ). When biosolids have PSI values less than 1.1, little P leaching is expected to occur, even in sandy, low P-sorbing soils (Elliott et al., 2002). Both PWEP and PSI can serve as *a priori* estimates of biosolids-P lability. Both measures can better estimate how P in biosolids will affect the environment when land applied than biosolids-TP or soil test P measures (Maguire et al., 2001; Elliott et al., 2002; Brandt et al., 2004; O'Connor et al., 2004).

Given the nutrient concerns that accompany biosolids land application, each state's Natural Resources Conservation Service (NRCS) is required by the Unified Strategy for Animal Feeding Operations (USDA/USEPA, 1999) to develop P management strategies. Under code 590, states have three options to manage P: agronomic soil test P recommendations, environmental soil P thresholds, or a P site index to evaluate vulnerability to potential P loss (Elliott and O'Connor, 2007). Most states have chosen a P-index approach as both P source and transport factors are taken into account. The P-index acknowledges that for negative environmental impacts to occur, both a soluble P source and a transport mechanism are required (Sharpley et al., 2003). The state of Florida is in the midst of developing a P-index to manage P nutrient concerns.

### **Hypotheses and Research Objectives**

A good understanding of biosolids P phytoavailability relative to fertilizer-P is critical to making adjustments to biosolids application rates should P-based application restrictions be imposed. All biosolids do not have the same potential to negatively affect the environment, hence, it is important to know which biosolids-P chemical characteristics can be used to judge *a priori* the environmental impact a biosolids will have once land applied.

- **Hypothesis 1:** Relative P phytoavailability is greater for BPR biosolids than conventionally produced biosolids.
- **Hypothesis 2:** P leaching is significantly greater from (sandy, low P-sorbing) soil amended with BPR residuals than the same soil amended with conventionally treated biosolids.
- **Hypothesis 3:** P lability of all biosolids is less than fertilizer-P (TSP).
- **Objective 1:** Characterize the agronomic value of various biosolids important in Florida so adjustments can be made to biosolids application rates if biosolids P-based regulations are imposed.

- **Objective 2:** Develop sufficient data to demonstrate that most biosolids pose less of an environmental hazard (with the possible exception of BPR biosolids) than readily soluble P sources.
- **Objective 3:** Expand the database of knowledge to include a wider variety of biosolids production schemes, specifically materials produced or marketed in Florida. Research was focused on materials not previously studied in detail, specifically BPR materials.

### **Study Approach**

A full laboratory characterization was conducted on 21 biosolids produced or marketed in Florida to identify materials warranting further study in glasshouse and laboratory experiments. Seven residuals were chosen for a glasshouse study to judge agronomic P availability and environmental lability. Eleven biosolids were chosen for a dynamic laboratory incubation, the purpose of which was to quantify water soluble P, examine the kinetics of P release, and determine the leaching hazard of various biosolids materials.

## CHAPTER 2 MATERIALS AND METHODS

### **Materials**

Wastewater treatment plants (WWTPs) throughout Florida were invited to participate in the research effort by completing a survey (developed by Robert Morrell of PBS&J consulting) of plant operations and materials characteristics. Completed surveys were submitted by 24 WWTPs, representing 22 residuals products, and results were forwarded to UF personnel. Based on survey results, 20 Florida products were requested from WWTPs for analysis. A few samples of biosolids produced in other states, but marketed in Florida, were also requested. Milorganite, a commercially available biosolids product from Wisconsin, was chosen to be included in this study. In total, 21 materials were subjected to laboratory characterization.

### **Year 1 Glasshouse and Static Incubation Studies**

In 2005, glasshouse and static laboratory incubation studies were conducted with 9 biosolids: Milorganite, GreenEdge, Jacksonville Cake, Tampa, Disney Compost, Pinellas Cake, Boca Raton, and Orange County Utilities Division (OCUD) east dry and cake materials. The residuals were individually amended to a typical Florida Spodosol (Immokalee fine sand) at 2 P-application rates: 56 and 224 kg P ha<sup>-1</sup> to represent P-based and N-based biosolids application rates, respectively. Most of the soil was used in the glasshouse study, but some was used in a static laboratory incubation. The glasshouse study was intended to provide measures of P leaching risk and relative P phytoavailability to pasture grass of various biosolids. Amended soils used in the static laboratory incubation were sampled at various times for WEP and TP determination, and the data were intended to yield information about the kinetics of biosolids-P release. Both the glasshouse study and the static laboratory incubation experiments were terminated in December 2005 because of apparent un-equal P loadings in treatments. We have

no explanation for the unequal P loads, but without equal P loadings, conclusions on relative P phytoavailability and P leaching were not possible. Similar studies were begun anew in 2006.

### **Laboratory Characterization**

Biosolids percent solids was determined by drying (105° C) “as is” materials to constant weight. Biosolids pH and electrical conductivity (EC) were determined using a 1:10 ratio of biosolids (dry weight equivalent) to distilled, de-ionized water (DDI), and equilibrated by static incubation at room temperature for 2 hours. After the 2 hr equilibration, the biosolids-DDI water mixture was stirred and allowed to settle, and pH and EC were determined on the solution. Total carbon/ total nitrogen (TC/TN) was determined on dried, ball-milled biosolids by combustion at 1010° C using a Carlo Erba (Milan, Italy) NA-1500 CNS analyzer.

Loss on ignition (LOI), a measure of biosolids organic matter content, was determined by measuring 0.2 g of oven-dried and ball-milled biosolids into pre-weighed 50 mL beakers. The materials were ashed at 250°C for 30 minutes, followed by ashing at 550°C for an additional 4 hours. The samples were allowed to cool, and the beakers were weighed again. Mass lost during the ash process is a measure of organic matter (Sparks, 1996).

To analyze for total phosphorus (TP), samples of biosolids were first oven-dried and ground in a ball mill to a fine powder. The biosolids (0.2 g) samples were then ashed (250°C for 30 minutes and 550°C for 4 hours) in a muffle furnace to destroy organic matter. The ashed samples were digested with ~2 mL of DDI water and 20 mL of 6 M HCl. The beakers were placed on a hotplate at 110° C until the HCl evaporated, and the residual was dry. The hotplate temperature was then increased to the hottest setting (~300°C) for 1 hour. The beakers were allowed to cool, and ~2 mL of DDI and 2.25 mL of 6 M HCl was added to the residual material and the beakers were placed on a hot plate (highest setting) until small bubbles started to form (solution began to boil). The solution was removed from the hotplate and allowed to cool. The

solution was transferred quantitatively to a funnel containing a Whatman #42 filter, and allowed to drain into 50 mL volumetric flasks. The filter paper was rinsed 3 times with DDI and allowed to drain completely between each rinse (Andersen, 1976). The solutions were brought to volume and analyzed for P using the molybdenum blue method (Murphey and Riley, 1962). Digests from the Andersen (1976) method were also analyzed for total Fe, Al, and Ca via inductively coupled plasma spectroscopy (ICP).

We also determined biosolids-TP using EPA method 3050A (USEPA, 1995) to verify TP values determined by the Andersen (1976) method were accurate. Method EPA3050A required 2 g (dry weight equivalent) of biosolids. Samples were pre-digested with 10 mL of 1:1 HNO<sub>3</sub>, covered with reflux caps, and heated at 110°C for 10-15 minutes. After the pre-digestion, an additional 5 mL of HNO<sub>3</sub> was added and the samples were refluxed for 30 minutes. This second step was repeated until brown fumes were no longer visible. The reflux caps were removed and the digestion solution was allowed to evaporate (2-3 h) to a volume of 5 mL. The flasks were removed from the hotplate and allowed to cool. Then, 30 mL of 30% hydrogen peroxide and 2 mL of DDI was added and new reflux caps were placed on top of the flasks. The samples were returned to the hotplate set at 110°C until effervescence subsided. Additional 1 mL aliquots of 30% hydrogen peroxide were added (not exceeding 10 mL) until effervescence ceased. After the peroxide reaction, 5 mL of concentrated HNO<sub>3</sub> was added and the samples were refluxed for 30 minutes. The samples were then cooled, and filtered through a Whatman #41 filter into 50 mL volumetric flasks. The filters were rinsed 3 times with DDI and allowed to drain completely between each rinse. Filtered digests were brought to volume and analyzed for P via ICP.

Biosolids-TP was also determined by an outside laboratory via perchloric acid digestion (Association of Official Analytical Chemists, 1990; The Fertilizer Institute, 1982) to further

confirm TP values. One gram of dried and powdered biosolids were boiled with 30 mL of concentrated HNO<sub>3</sub> for ~20 minutes. The samples were cooled and 20 mL of perchloric acid was added until the solutions were colorless and started to fume. Once samples were colorless, and white fumes were noted, the digestion was continued for an additional 20 minutes. Following the second 20-minute digestion, 30 mL of DDI was added and the samples were boiled for 5 minutes. Samples were then cooled and filtered through an Ahlstrom Grade 54, 12.5 cm filter paper into a 100 mL volumetric flask. Samples were brought to volume and analyzed for P via ICP.

Water-extractable P (WEP) was determined using a 1:200 biosolids (dry weight equivalent) to DDI water (Sharpley and Moyer, 2000). The residuals/water mixture was shaken on an orbital shaker at 200 strokes per minute for 1 hour. A sub-sample of the mixture was centrifuged for 10 minutes at 4000 rpm, and the supernatant was vacuum-filtered through a 0.45 µm- filter. The solution was analyzed for P using the molybdenum blue method (Murphey and Riley, 1962). Percent WEP (PWEP) was calculated by dividing WEP by TP and multiplying by 100 ( $PWEP = WEP/TP*100$ ). Brandt et al. (2004) concluded that PWEP was a good measure of the environmentally relevant portion of total P in biosolids and manures.

Oxalate-extractable P, Fe, and Al was determined by shaking 0.5 (dry weight equivalent) of biosolids with 30 mL of 0.175 M ammonium oxalate and 0.1 M oxalic acid for 4 hours on an orbital shaker at 200 strokes per minute (Loeppert and Inskeep, 1997). The samples were shaken in a sealed box to avoid exposure to light. After shaking, the samples were centrifuged for 10 minutes at 4000 rpm, and vacuum filtered through a Whatman #42 filter. The supernatant was analyzed for P, Fe, and Al via ICP. Moles of oxalate-extractable P, Fe, and Al were used to

calculate PSI, an indication of labile P in residuals [PSI = moles P/ (moles Fe + mole Al)] (Elliott et al., 2002).

Fe-oxide extractable P (Fe-strip P) in biosolids was determined using Fe-oxide impregnated strips (Chardon, 1996). Strips were prepared by soaking Whatman #42 filter paper in 0.65 M  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  + 0.6 M HCl solution overnight. The strips were removed and allowed to air dry. The strips were immersed for 30 seconds in 2.7 M  $\text{NH}_4\text{OH}$ , then rinsed twice in DDI water, left to sit in clean DDI water for 1 hour, and then air dried before use. Fe-strip P was determined by shaking the prepared strips with 1 g (dry weight equivalent) of biosolids and 60 mL of 0.01 M  $\text{CaCl}_2$  in a 120 mL bottle for 16 hours on an orbital shaker at 125 oscillations/minute. The original method was changed from 40 mL  $\text{CaCl}_2$  to 60 mL  $\text{CaCl}_2$  to ensure the biosolids were completely covered with solution. During the  $\text{CaCl}_2$  wash, P is removed from the biosolids and is retained on the Fe-strip. After shaking, the strips were removed from the glass bottles and rinsed thoroughly with DDI to remove biosolids particles. The rinsed strips were placed in 125 mL Erlenmeyer flasks and shaken with 40 mL of 0.1 M  $\text{H}_2\text{SO}_4$  on an orbital shaker for 1 hour at 125 oscillations/minute. The 0.1 M  $\text{H}_2\text{SO}_4$  extracts P from the Fe-strip, and the solutions were then analyzed for P using the molybdenum blue method (Murphey and Riley, 1962). Fe-strip P is measure of biologically available P in soil (Sharpley, 1993a and b).

### **Dynamic Laboratory Incubation**

Eleven biosolids and TSP were chosen for a dynamic laboratory incubation. Biosolids were mixed at 2 rates, equivalent to 56 and 224 kg P ha<sup>-1</sup>, with 400 g samples of Immokalee A horizon soil (Immokalee fine sand, sandy, siliceous, hyperthermic Arenic Alaquods; Table 2-1) and enough water (40 mL) to reach field capacity. The biosolids-amended soil was incubated for 2 weeks in zip-lock plastic bags. The bags of soil were mixed and opened daily during the initial

incubation to avoid anaerobic conditions. As in the glasshouse study (below), ammonium nitrate was added to the soil to supply additional N. Originally, we intended to equalize varying N levels among the materials at both P rates to 300 kg N ha<sup>-1</sup> plant available N (PAN). We initially applied 75 kg N ha<sup>-1</sup> to the biosolids-amended soil at the start of the 2-week equilibration, with the intention of split applying the remaining N after leachings. However, the columns of soil were near field capacity for the duration of the experiment, and applying AN in solution would have induced unintentional leaching; applying AN in crystalline form would not allow for N distribution throughout the soil column. Thus, the remaining 225 kg N ha<sup>-1</sup> was not applied.

Incubation columns were constructed of 17 cm x 5 cm sections of polyvinyl chloride (PVC) tubing with screening in the bottom to prevent soil loss. At the end of the initial 2-week bag incubation, 400 g biosolids-amended soils were packed into the incubation columns to a depth of 13 cm and a bulk density of 1.51 g cm<sup>-3</sup>. A total of 75 columns was used (12 materials x 2 rates x 3 reps + 3 controls = 75). The columns were positioned vertically in wooden racks for leaching events.

Preliminary experiments were conducted to determine a representative column pore volume (PV). Two methods were used to determine PV, gradual wetting and flood wetting. Four incubation columns were filled with 400 g of dry soil and weighed. Tap water was gradually added to two of the columns until 1 or 2 drops of water exited the bottom (gradual wetting). The retained water volume (~118 mL) was assumed to be 1 PV. The other two columns were flooded with 250 mL of tap water and allowed to drain overnight. When drainage was complete, the columns were re-weighed and the difference in weight (~120 g) was assumed to be 1 PV. One PV was taken to be 120 mL.

Every 2 weeks, sufficient tap water (adjusted to pH 5.0) was added to the columns to result in 60 mL (1/2 PV) of drainage. We adjusted the pH of the water to 5.0 to simulate the pH of rainfall in south Florida. Laboratory tap water was analyzed for soluble reactive (inorganic) P (below detection limit; 0.001 ppm). For the first 2 leachings, P analysis of the leachates included total phosphorus (TP), total dissolved phosphorus (TDP) and soluble reactive phosphorus (SRP); pH and EC was also determined. Total P in the leachate was determined by digesting 5 mL of unfiltered leachate with 0.35 g of potassium persulfate and 1 mL of 5.5 M H<sub>2</sub>SO<sub>4</sub> on a digestion block at 125° C until only 5 mL of solution remained (USEPA, 1993). The temperature was then increased to 150° C until 0.5 mL of solution remained. Reflux caps were placed on top, and the temperature was further increased to 380° C and digestion continued until the solution was clear. TDP was determined in the same way, however the leachates were vacuum filtered through a 0.45 µm- filter to remove particulates before digestion. The digested leachate was analyzed for P via the molybdenum blue method (Murphey and Riley, 1962). SRP was determined by vacuum filtering leachate through a 0.45 µm- filter. The filtered leachate (undigested) was analyzed for P using the molybdenum blue method. Organic P was determined by subtracting SRP from TDP. Results from the first 2 leachings, revealed that the SRP represented the majority (>80%) of the total P leached from the amended soils, and that organic P was minimal (<10% of TP). Thus in subsequent leaching events, TP and TDP were measured only for select (highly colored or cloudy) samples: Lakeland NS, OCUD S, OCUD E cake and OCUD E dry. TDP and TP values were determined on these samples to ensure organic P and particulate P was minimal. Leachate pH and EC was measured directly on unfiltered/undigested leachate.

## Glasshouse Study

Seven biosolids and TSP were chosen for the glasshouse experiment for a total of 8 materials. Two biosolids were selected to represent (expected) low-P availability biosolids: Milorganite, and GreenEdge (PWEP  $\leq 1.1\%$ ). Four biosolids were selected as high P-availability biosolids (Boca Raton, GRU, Lakeland NS, and OCUD S), which are BPR or BPR-like residuals (PWEP  $\geq 15\%$ ). One biosolids selected (Disney) had a moderate PWEP (8.4%) and biosolids-P was expected to be moderately plant available. Biosolids were also selected based on PSI and PWEP values. BPR and BPR-like biosolids typically have PSI and PWEP values greater than traditionally produced residuals. We chose biosolids with high PSI and PWEP values to expand the existing relative P phytoavailability database. Much work has been done on the phytoavailability and lability of conventionally produced biosolids, including some Florida materials, and some BPR materials (O'Connor et al., 2004; Elliott et al., 2002), but the database is limited. Materials were mixed with 4 kg of A horizon Immokalee soil (Table 2-1) at 3 rates: 56, 112, and 224 kg P ha<sup>-1</sup>. The 56 and 224 kg P ha<sup>-1</sup> rates represent P-based and N-based application rates, respectively. The 112 kg P ha<sup>-1</sup> rate was added to better define the relative P phytoavailability response curve of the materials. Immokalee fine sand was chosen to represent a typical, P-deficient, low-P sorbing Florida soil. A P-deficient and low P-sorbing soil is necessary to represent a “worst-case” scenario of biosolids land application, and to maximize plant response to P additions. The Immokalee soil does not retain P and can allow P movement to ground and surface waters.

The biosolids-amended soils were equilibrated (at field capacity) in zip-lock plastic bags for 2 weeks in the laboratory prior to use in the glasshouse. Ammonium nitrate (AN) was added to the amended soils to equalize varying amounts of N supplied by the biosolids. N was applied at an equivalent rate of 300 kg ha<sup>-1</sup> plant available nitrogen (PAN). The GreenEdge material

supplied the most PAN (240 kg ha<sup>-1</sup>) at the high P rate. We opted to supply an additional 60 kg ha<sup>-1</sup> of N to ensure N limitations would not affect P release or uptake. This rate is excessive for Bahiagrass (179 kg N ha<sup>-1</sup> recommended; Kidder et al., 1998), but could not be avoided due to the N:P ratio of the materials and the high targeted P rate of 224 kg ha<sup>-1</sup>. N mineralization was assumed to be 40% of the total N in all of the biosolids, based on previous experience (O'Connor and Sarkar, 1999). The total supplemental AN needed was split-applied, with ¼ applied prior to incubation, and the remaining split applications added throughout the growing season (following the first 3 leaching events). AN is fully soluble in water and therefore is immediately available for plant uptake. Thus, AN was split-applied to ensure sufficient N was available for uptake throughout the growing season. A blend of potassium-magnesium sulfate (“sul-po-mag”; 22% S, 18% K, 11% Mg) was added (0.91 g, equivalent to ~444 kg ha<sup>-1</sup>) to supply adequate and uniform S, K, and Mg.

Containers for the glasshouse study were constructed of 15 cm diameter X 45 cm long sections of polyvinyl chloride (PVC) tubing. A screen was fitted to the bottom to prevent soil loss. A PVC cap fitted with plastic tubing was attached to the bottom of the column to allow leachate collection. A total of 100 columns was used (8 materials X 4 replicates X 3 rates + 4 controls = 100). The columns were arranged in a randomized complete block design in wooden racks holding 6 columns each. The experiment was blocked to minimize glasshouse positioning effects. The columns were rotated one position within each block weekly to further minimize glasshouse effects on grass growth. O'Connor et al. (2004) used the same container and experimental design to quantify P uptake and leaching for 12 biosolids products in the same soil.

Thirty cm of sand (~8.5 kg) was packed into the columns as a support layer (TP = 12 g kg<sup>-1</sup>, WEP = 0.12 mg kg<sup>-1</sup>, pH = 5.1, RPA = 8.6%). The sand was included to provide additional

rooting depth for the grass. The sand was flooded and allowed to freely drain to wet the layer to field capacity and to remove soluble constituents. Following the 2-week laboratory incubation, 4 kg of biosolids-amended soil was placed on top of the sand layer in each column, and 5 g of Bahiagrass seed was planted. Five grams of Bahiagrass seed per pot is a seeding rate equivalent to  $\sim 28.6 \text{ Mg ha}^{-1}$ , the recommended seeding rate is  $\sim 10.9 \text{ Mg ha}^{-1}$  (Chambliss et al., 2001). We increased the seeding rate to ensure sufficient and rapid soil surface coverage. The seeds were covered with a layer of sand ( $\sim 0.5 \text{ cm}$ ) and misted every 3-4 hours until germination. The grass was allowed to grow for approximately 7 weeks before the first harvest. Subsequent harvests occurred at approximately 4-week intervals.

The Bahiagrass was harvested to a height of 3.8 cm using scissors. The wet clippings were placed in pre-weighed paper bags and dried at  $68^\circ \text{ C}$  to constant weight to represent grass yields. The dried tissue was ground to pass a #20 sieve with a Wiley mill, digested (Andersen, 1976) and analyzed for P via the molybdenum blue method (Murphey and Riley, 1962).

Phosphorus uptake was calculated as yield times tissue P concentration. Individual harvest P uptake masses were summed to give cumulative P uptake mass. Tissue N content of plants from the  $224 \text{ kg P ha}^{-1}$  rate for all treatments was also measured for all 4 harvests. Tissue N was determined by grinding the tissue to a fine powder, and analyzing for N by combustion at  $1010^\circ \text{ C}$  using a Carlo Erba (Milan, Italy) NA-1500 CNS analyzer.

Following each harvest, sufficient tap water (adjusted to pH 5.0) was applied to each column to yield  $\sim 500 \text{ mL}$  ( $\sim 0.25$  pore volume) of leachate. Soluble reactive (inorganic) P (SRP) of glasshouse tap water was measured (below detection limit; 0.001 ppm) via the molybdenum blue method (Murphey and Riley, 1962). Leachate was analyzed for SRP using the molybdenum blue method. Leachate volume times leachate P concentration yielded mass of P leached.

Leachates from the Lakeland NS material (high P rate) were highly colored. TP was determined on the leachates to ensure organic P loss from the material was not significant.

### **Statistical Analysis**

Cumulative P leached from the laboratory incubation was subjected to a time-series analysis (SAS Institute, 1989). Data were tested for normality using PROC UNIVARIATE. To normalize yield and P uptake data, logarithmic transformations were needed. P leaching data were normalized with a square transformation. Transformed cumulative yield, P uptake, and P leached from the glasshouse study were statistically analyzed using PROC GLM. Means separation of treatment differences was by Tukey test ( $p \leq 0.05$ ) on transformed data. Relative phytoavailability was calculated by fitting a linear regression to P uptake as a function of P applied data using a slope- ratio approach. All P sources were regressed through a common intercept of 8.48 mg, the average value of P uptake for the control, resulting in a response proportional to the rate of P application.

Table 2-1. Selected properties of Immokalee fine sand.

<b>Parameter, units</b>	<b>Value</b>
Sand, %	95 <sup>a</sup>
pH	4.8
Organic matter, g kg <sup>-1</sup>	7.0 <sup>a</sup>
Oxalate-extractable P, mg kg <sup>-1</sup>	13.1 <sup>a</sup>
Oxalate-extractable Fe, mg kg <sup>-1</sup>	85.6 <sup>a</sup>
Oxalate-extractable Al, mg kg <sup>-1</sup>	40.1 <sup>a</sup>
PSI <sup>c</sup>	0.14 <sup>a</sup>
RPA <sup>d</sup> , %	2.0
Total P, mg kg <sup>-1</sup>	15.5
Mehlich 1-extractable P, mg kg <sup>-1</sup>	1.47
KCL-extractable P, mg kg <sup>-1</sup>	1.9 <sup>a</sup>
NaOH-extractable P, mg kg <sup>-1</sup>	3.3 <sup>a</sup>
HCl-extractable P, mg kg <sup>-1</sup>	0.9 <sup>a</sup>
Sequenced sum, mg kg <sup>-1</sup>	6.1 <sup>a</sup>

<sup>a</sup>Data from O'Connor et al. (2004). <sup>c</sup>Phosphorus Saturation Index. <sup>d</sup>Relative phosphorus adsorption [fraction of 400 mg P kg<sup>-1</sup> soil load sorbed (Harris et al., 1996)].

## CHAPTER 3 RESULTS AND DISCUSSION

### **Laboratory Characterization**

The laboratory characterization was key to determining which materials warranted further study in the dynamic laboratory incubation and glasshouse studies. Twenty-one biosolids were received and analyzed (Table 3-1). As detailed below, both basic chemical properties and P characteristics were important for material selection. Recall that a goal of both the glasshouse and dynamic laboratory incubation experiments was to include materials not previously studied, specifically BPR and BPR-like products.

Total nitrogen (TN) concentrations ranged from 16 to 70 g kg<sup>-1</sup> (1.6 to 7%). Biosolids C:N ratios were generally low (6-15), with the exception of the West Palm Beach Compost material (C:N of 26) (Table 3-2). The TN and C:N ratios are typical of U.S. produced biosolids (USEPA, 1995). C:N ratios are important for predicting N mineralization from biosolids. In general, when the C:N ratio of an organic material is less than 20:1, N will be released into the soil. If the C:N ratio exceeds 30:1, N can be immobilized by soil microbes, and unavailable for plant uptake. Thus, we selected materials with C:N ratios below 20:1. The Disney material was chosen as the representative compost residual to avoid possible complications with N immobilization from high C:N ratio of the West Palm Beach biosolids.

Biosolids pH varied with source and product form (Table 3-2). Cakes were circum-neutral to alkaline, and thermally dried products were slightly acidic. Lime stabilized products had the highest pH values, as lime stabilization increases the pH of the final product (pH > 12). We excluded materials with pH values > 9 from the glasshouse study as Bahiagrass prefers an acidic environment (pH ~5.0; Chambliss and Adjei, 2006). O'Connor et al. (2004) reported

severely limited growth of Bahiagrass when biosolids with high pH (>12) values were applied to the Immokalee soil at N-based rates.

TP concentrations of biosolids (Table 3-3) ranged from 7.9 to 33 g kg<sup>-1</sup> (0.79 to 3.3%), typical of TP concentrations of biosolids produced conventionally nationwide (~20 to 40 g kg<sup>-1</sup>), and  $\geq 40$  g kg<sup>-1</sup> for BPR materials (USEPA, 1995). The difference in TP values results from the differences in wastewater characteristics and treatment practices (Brandt et al., 2004). TP concentrations were not used to include/exclude materials, but TP concentration was an important measurement, as the laboratory determined TP values were used to calculate the mass of biosolids needed to attain selected P application rates.

TP values for the materials were determined using 3 methods: Andersen (Andersen, 1976), EPA 3050A (USEPA, 1995) and perchloric acid (Association of Official Analytical Chemists, 1990; The Fertilizer Institute, 1982) (Table 3-4). The Andersen and EPA 3050A analyses were performed at UF. The perchloric acid analysis was conducted at an independent laboratory for additional TP verification. The ability to recover P from the biosolids differs among the techniques, which is reflected in the variability of the reported TP concentrations. UF researchers were not provided information on how the individual WWTPs analyzed materials for TP, which could explain the variability between determined and producer-supplied P values. A standard reference biosolids material (National Institute of Standards and Technology, Standard 2781: Domestic Sludge) was used to gauge the ability of digestion methods to recover P. All three methods had excellent P recovery percentages (88-96%).

Researchers chose to use the TP values determined by the Andersen (1976) method for this study, primarily because of its ease of use. The EPA 3050A method is tedious and time consuming, making the method impractical for the large number of samples involved in this

study. The perchloric acid analysis requires special facilities to handle the explosive nature of perchloric acid, which were not available in the UF laboratory. In contrast, the Andersen (1976) and EPA 3050A methods use hydrochloric and nitric acid, respectively, which are much safer to handle. In a preliminary experiment, the Andersen-TP concentrations were used to calculate the quantity of biosolids necessary to reach a target P rate of 224 kg P ha<sup>-1</sup>. A small quantity (200g) of Immokalee soil was amended with the calculated masses of biosolids. When the biosolids-amended soils were analyzed for TP using the Andersen (1976) method, the target P rate was reached, indicating that the TP values obtained from the Andersen (1976) method were adequate measures of the total P in the biosolids.

Total Fe, Al, and Ca concentrations were measured on the Andersen-TP extracts via ICP (Table 3-2). Total metal concentrations were representative of biosolids produced nationally and reflected the individual biosolids treatment processes. Total Fe ranged from 2.2 to 60 g kg<sup>-1</sup> (0.2 to 6%), Al ranged from 1.7 to 24 g kg<sup>-1</sup> (0.17 to 2.4%) and Ca ranged from 13 to 310 g kg<sup>-1</sup> (1.3 to 31%). The Lakeland Glendale material is lime stabilized, which is reflected in the high total Ca (310 g kg<sup>-1</sup>). The greater a residual's Fe and Al content, the better the material is able to retain P, leading to lower phytoavailability and lower leaching risks (Elliott et al., 2002). As discussed below, Milorganite (41 g kg<sup>-1</sup> Fe, 2.7 g kg<sup>-1</sup> Al) has relatively high amounts of Fe and Al, and resulted in decreased P release and P phytoavailability.

Water-extractable P (WEP) ranged from 0.04 to 14 g kg<sup>-1</sup> (Table 3-3). Brandt et al. (2004) demonstrated that WEP is a good measure of the environmentally relevant portion of P in biosolids. WEP is used to calculate percent water-extractable P (PWEP = WEP/ TP\*100). Most of the non-BPR materials analyzed had PWEP values < 5%, which is typical of non-BPR biosolids produced in the U.S. (Brandt et al., 2004). BPR materials, (Boca Raton, OCUD E cake

and dry) had PWEF values  $\geq 11\%$ . Brandt et al. (2004) determined the average PWEF for various BPR biosolids sampled nationwide to be  $\geq 14\%$ . PWEF was high for both Lakeland NS (47%) and GRU (26%). These materials are not produced via BPR processes, but personal communication with plant operators suggests that P removal is likely occurring in both systems. We have categorized these materials as “BPR-like”.

Both the Lakeland NS and GRU materials are low percent solids (3 and 5%, respectively, Table 3-2), and we decided to analyze the liquid and solid phases of both materials to determine the concentration of TP. Phosphorus in the liquid phase would be expected to be highly labile, and we suspected that the high PWEF values were due in part to high concentrations of P in the liquid phase. To determine the TP concentrations in the separated liquid and solid phases of each residual, we centrifuged sub-samples at 12,000 rpm for 15 minutes to separate the liquid phase from the solid phase. The separated phases were then analyzed for TP, the solid phase via the Andersen (1976) method, and the liquid phase via persulfate digestion (USEPA, 1993). Analysis showed that 50% and 20% of TP was in the liquid phase for the Lakeland NS and GRU materials, respectively. The smaller concentration of P in the liquid phase for GRU than Lakeland NS is reflected in the smaller PWEF value for GRU (26%) versus Lakeland NS (47%).

Disney is a BPR biosolids, however the PWEF (8.4%) is below average for a BPR residual. The Disney material is a composted mixture of biosolids, food, and yard waste. Brandt et al. (2004) reported that composting an anaerobically digested cake decreased WEP by 10-fold. The yard waste itself could decrease PWEF; the Disney compost was sieved to pass through a 2 mm sieve prior to analysis, however small pieces of wood were obvious. The yard waste composted with the Disney biosolids decreases the mass of biosolids per kg of land applied finished product (composted biosolids + yard waste, even after sieving), thus reducing labile P.

Oxalate-extractable Fe, Al, and P concentrations (Table 3-3) were used to calculate PSI values for the materials. PSI can be used *a priori* to gauge the lability of P in many biosolids in sandy soils (Elliott et al., 2002). PSI relates the moles of oxalate-extractable P to the moles of extractable Fe and Al (PSI = moles P / [moles Fe + moles Al]). Biosolids with high concentrations of Fe and Al tend to have less labile P. PSI has no meaning for biosolids whose P chemistry is controlled by Ca (lime stabilized materials). Elliott et al. (2002) suggested a critical PSI value of 1.1 for non-lime stabilized materials. That is, if the PSI of a material exceeds the critical value, appreciable P leaching may occur from amended, sandy soils with limited P retention capacity. Biosolids with PSI values  $\leq 1.1$  resulted in minimal P leaching in the Elliott et al. (2002) study. Most of the samples analyzed in the current study were BPR or BPR-like materials, and the PSI values exceed the critical value proposed by Elliott et al. (2002). We would expect significant leaching when these materials were land applied to Immokalee soil that retains P poorly.

### **Dynamic Laboratory Incubation**

Eleven biosolids and TSP were individually mixed with 13 cm (400 g) samples of Immokalee soil for the dynamic incubation study. Soil columns were leached a total of 8 times (~4 total PV) over 5.5 months. The first 7 leachings were conducted bi-weekly to attain ~60 mL (1/2 PV) of drainage each time. Significant P release (leaching) ceased after 5 leachings (2.5 months). A time-series analysis (SAS Institute, 1989) showed no difference in cumulative P released for leachings 5-7. We waited 2 additional months to conduct the final leaching to allow for P dissolution and distribution through the individual columns of soil and to confirm P release from all materials was maximized. Indeed, P loss from all materials during leaching 8 was minimal (< 1mg). Data from the final leaching was added to the time-series analysis and, again, showed no change in cumulative P released between leachings 5-8. Three months passed

between leachings 5 and 8. Because cumulative P release did not change over the 3-month time frame, we assumed that P release from the materials had ceased, and the experiment was terminated. SRP in leachate was taken to represent total P released, as SRP constituted the majority (>85% of TP) of P lost to leaching. To calculate P released as a percentage of applied P, the mean mass of P released from the control treatment was subtracted from the mass of P released for all other treatments, and the difference divided by the appropriate mass of P originally applied.

One objective of the study was to quantify water soluble P (mass of P released) from various biosolids representing a range of chemical characteristics. Biosolids with lower quantities of water soluble P are less likely to negatively affect the environment. The quantity of water soluble P is governed by several factors, including biosolids treatment processes [especially heat drying (Smith et al., 2002; Maguire et al., 2001)], and biosolids chemical composition (especially the quantity of Fe and Al oxides in the material). Residuals high in Fe and Al oxides retain P, and result in less P leaching (Brandt et al., 2004). Thus, we expected significantly more P release from BPR biosolids low in Fe and Al oxides than thermally dried residuals high in Fe and Al.

ANOVA showed significant source (P-source), rate (P-rate) and source by rate interactions. As a result, comparisons of cumulative P released and cumulative P leached as a percent of applied P are discussed within each P application rate and not across P application rates. Cumulative P release was greatest from BPR and BPR-like materials (Lakeland NS, GRU, Boca Raton, and OCUD E dry and cake) (Table 3-5). Figures 3-1a and b show cumulative P release (percentage of applied P) in bar graph form for the 56 and 224 kg P ha<sup>-1</sup> rates, respectively. P release from Lakeland NS, GRU, Boca Raton, and OCUD E cake biosolids was

equal to TSP at both the 56 and 224 kg P ha<sup>-1</sup> application rates (Figures 3-1a and b). The OCUD E dry material was produced by thermally drying the OCUD E cake material. The OCUD E cake is undigested biosolids and is not lawfully land applied, but was included for scientific interest. Heat drying the OCUD E cake created a class AA material that may be land applied. Heat drying typically decreases labile P (Smith et al., 2002), and P release was less for the OCUD E dry biosolids than for the OCUD E cake biosolids. However, P release from the OCUD E dry biosolids at the 56 kg P ha<sup>-1</sup> application rate was equal to that from TSP, indicating the OCUD E dry biosolids still had high quantities of labile P.

P release from the Disney biosolids was less than TSP and most BPR and BPR-like biosolids at both the 56 and 224 kg P ha<sup>-1</sup> rates. At the 56 kg P ha<sup>-1</sup> rate, P release from Disney was equal to GRU. As previously mentioned, Disney is a composted mix of biosolids, yard, and food waste, which results in a PWEF (8.4%) less than the other BPR and BPR-like biosolids (≥ 15%) in this study. The PSI (0.45) of Disney reflects relatively large quantities of Fe and Al, which likely reduced P release.

At 56 kg P ha<sup>-1</sup>, P release from Milorganite and GreenEdge was significantly lower than from TSP. Both Milorganite and GreenEdge are thermally dried. Thermal drying typically lowers quantities of labile P. Milorganite also has relatively high total Fe + Al concentrations (44 g kg<sup>-1</sup>) compared to typical biosolids total Fe + Al concentrations (20 g kg<sup>-1</sup>), which we expected to further reduce P lability. P release from Milorganite and GreenEdge was not significantly different, suggesting that P release from Milorganite and GreenEdge could be controlled by physical properties (pellet dissolution). When the laboratory incubation was dismantled, pellets of Milorganite and GreenEdge were still apparent, suggesting that even though the columns were near field capacity for the duration of the experiment, the length of time was not sufficient for

complete pellet dissolution. Milorganite Greens Grade, which is the same product as Milorganite, but passed through a smaller sieve, has a slightly greater PWEP value (0.85%) than Milorganite (0.58%). We delayed the final leaching for 2 months following leaching 7 to allow dissolved P to diffuse through soil pores (a time limited reaction), and the columns of soil were maintained near field capacity during the 2-month break. Despite the elapsed time and sufficient moisture, P release from all materials was < 1 mg in leaching 8. Time series analysis also indicated that P release was minimal over the last 3-month period (leachings 5-8), suggesting that P release from the P sources was maximized.

We expected GreenEdge to release a greater quantity of P than Milorganite based on the biosolids PSI values, total Fe and Al concentrations, and PWEP values. GreenEdge has greater PSI (1.0) and PWEP (1.1%) values than Milorganite, (PWEP: 0.58%, PSI: 0.55) and Milorganite has greater Fe and Al ( $44 \text{ g kg}^{-1}$ ) concentrations than GreenEdge ( $\text{Fe} + \text{Al} = 23 \text{ g kg}^{-1}$ ). Based on the P chemistry, we expected a greater quantity of P release from GreenEdge, however the data did not support our *a priori* assumption. We hypothesized that calcium was influencing P lability, however the Ca concentration of GreenEdge is not great enough to suggest that Ca is controlling P solubility. We then ground a sample of GreenEdge with a mortar and pestle and determined PWEP on the finely ground biosolids. The PWEP of GreenEdge did not increase after grinding, (1.3%, compared to 1.1% on un-ground biosolids) indicating that pellet size was not influencing P solubility. We currently have no explanation for the equal quantities of P released from GreenEdge and Milorganite. Butkes et al. (1998) studied P retention of water treatment residuals (WTRs) and suggested that cationic polymers added during the water treatment processes can retain P. During the dewatering of GreenEdge, a polymer (Ciba Zetag

8849FS) is added, and it is possible that the dewatering polymer has the capacity to sorb P, thus reducing P lability.

Cumulative mass of P released from the OCUD S material was equal to TSP at the 56 kg P ha<sup>-1</sup> application rate and greater than TSP at the 224 kg P ha<sup>-1</sup> rate (Figures 3-1a and b). The OCUD S material is a conventionally produced biosolids; however, the PWEP (21%) and the PSI (2.9) values of the OCUD S material are high, and consistent with the high quantity of P leached. Given the treatment process of the OCUD S biosolids (anaerobically digested) we would not expect PWEP and PSI values to be so high. However the total Fe + Al concentrations of the OCUD S material are low (11g kg<sup>-1</sup>), resulting in the high PSI value and quantities of P released equal to TSP.

As previously mentioned, biosolids-TP is not a good indication of P lability. However, PWEP can be used to gauge the environmental impact a residual will have once land-applied. A logarithmic relationship exists between cumulative P released (% of applied P) and biosolids-PWEP at both rates (Figures 3-2a and b). The correlation was similar for the 56 kg P ha<sup>-1</sup> rate ( $r^2 = 0.65$ ) and the 224 kg P ha<sup>-1</sup> application rate ( $r^2 = 0.69$ ). The correlation between cumulative P released (% of applied P) and biosolids-PWEP was not strong enough to indicate that PWEP can be used to predict the amount of P release that will occur when a biosolids is land applied. However, Brandt et al. (2004) demonstrated that PWEP is a superior measure of the environmentally relevant portion of P in biosolids and manures than biosolids-TP or soil test P. Biosolids-PWEP can be used *a priori* to gauge the potential of a residual to negatively affect the environment. PWEP is a measure of the water-soluble P in biosolids, thus materials with PWEP values  $\geq 14\%$  (vertical line in Figures 3-2a and b) should be assumed to have a larger negative environmental impact than biosolids with PWEP values  $< 14\%$ . Recall that Brandt et al. (2004)

reported an average PWEF value for BPR biosolids sampled nationwide to be  $\geq 14\%$ . Given the ease with which WEP/PWEF can be determined, PWEF can be used as a quick and efficient gauge of a residual's potential to negatively impact the environment once land applied. Elliott et al. (2006) used source WEP to improve P source coefficient assignments ( $PSC = 0.102 \times WEP^{0.99}$ ) for state P-indices, and runoff dissolved P was well correlated ( $r^2 = 0.80$ ) with source-WEP.

Figures 3-3a and b show P released (% of applied P) as a function of PSI for the 56 and 224 kg P ha<sup>-1</sup> rates, respectively. Elliott et al. (2002) demonstrated that for biosolids with PSI values  $\leq 1.1$ , no appreciable leaching occurred from another sample of the Immokalee soil amended with biosolids. Several materials used in this study had PSI values above the suggested critical point of 1.1, which would portend significant P losses measured from the BPR and BPR-like materials. Data from the current study indicate that a critical PSI value of  $\sim 2.0$  better separates biosolids where leaching was greatest, suggesting that the critical PSI value be raised from 1.1 to  $\sim 2.0$ . However, the Elliott et al. (2002) study differed from the current study in 2 ways: 1) the Elliott et al. (2002) study utilized much larger soil columns, where  $\frac{1}{4}$  of a pore volume was 500 mL, and 4 monthly leachings were necessary to reach 1 pore volume of drainage; and 2) Bahiagrass was grown in the Elliott et al. (2002) soil columns, reducing the quantity of P available for leaching. Data published separately (O'Connor et al., 2004) showed that Bahiagrass took up 29-57% of applied P at the 56 kg P ha<sup>-1</sup> application rate. In the dynamic laboratory incubation,  $\sim 4$  pore volumes of drainage were collected versus 1 pore volume of leachate collected in the Elliott et al. (2002) study. The small columns in the current study included no plants and were flushed with more pore volumes of water than the large glasshouse columns, which would encourage P release and movement downward (and out) of the small

columns. Given the differences in experimental design between the current study and Elliott et al. (2002) study, raising the critical value from 1.1 to ~2 is probably not justified. While there is no clear mathematical relationship between cumulative P release and PSI, it is evident that P release increases when  $PSI > 2.0$ . P release appears to be maximized (~80% of applied P) at PSI values  $> 2.0$

The second major objective of dynamic laboratory study was to examine the kinetics of P release. Figures 3-4a and b show cumulative P released as a percentage of P applied per leaching event for the 56 and 224 kg P ha<sup>-1</sup> rates, respectively. While the ultimate quantity of P released is important, the rate at which P is released is also important. When large amounts of P are released from a material quickly, more P is in the soil solution at any given time and is subject to leaching in periodic rain events, increasing the risk of ground and surface water impairment. Slow rates of P release provide plants more time to take up biosolids-P, decreasing the amount of P subject to loss from the soil profile.

Materials with high PWEF values (OCUD E cake, Lakeland NS, GRU, OCUD S) released large quantities of P quickly (within the first 3 leachings). At the 224 kg P ha<sup>-1</sup> application rate (Figure 3-4b), the thermally dried materials (Milorganite, GreenEdge, and Tallahassee) and the conventionally produced Broward cake released  $< 7\%$  of applied P during the first leaching event and the remaining materials released  $>15\%$  of applied P. The quantity of P released from Milorganite and GreenEdge was ~55% less than P released by the highly-P soluble materials during the first leaching event. Phosphorus release from the cake and slurry materials began to decrease (slow) in leaching 4, but P release from Milorganite and GreenEdge continued to increase through leaching 5. As discussed above, high rates of P release from BPR and BPR-like materials increase the quantity of P in the soil solution at any given point in time.

The increase in soil solution P suggests that BPR and BPR-like biosolids could be excellent fertilizers, but also makes BPR and BPR-like residuals more likely to negatively affect the environment, when soil solution P is washed through the soil profile into ground and surface waters.

We can roughly convert total PVs leached to residence time in a field setting. One PV of leachate represents ~5.64 cm drainage. Cumulative leaching (~480 mL) represented 22.6 cm of drainage. Yearly rainfall in south Florida averages ~140 cm yr<sup>-1</sup> (Obeysekera et al., 2004) and evapotranspiration is ~70% of rainfall (Nachabe et al., 2005). Subtracting evapotranspiration from rainfall (140 cm yr<sup>-1</sup> – 100 cm yr<sup>-1</sup>) yields 40 cm yr<sup>-1</sup> drainage. Assuming 40 cm drainage yr<sup>-1</sup>, we can calculate the number of PVs leached per year (40 cm yr<sup>-1</sup>/5.64 cm PV<sup>-1</sup> = 7.09 PV yr<sup>-1</sup>). Thus, the 4 PVs leached in this study equate to ~7 months in the field. This study represents an extreme case of biosolids land application where the soil used had minimal P sorbing capacity and no plants were grown to take up applied P. This experiment was key to understanding biosolids P leaching characteristics in a representative “worst-case” scenario.

### **Glasshouse Study**

A glasshouse study was run concurrently with the laboratory leaching study. Seven biosolids were used and TSP was included as a reference. Biosolids were mixed with 13 cm (4 kg) samples of Immokalee soil and placed on top of 30 cm of base sand. Two biosolids evaluated had low P<sub>WEP</sub> (<1.1%) values: Milorganite, and GreenEdge, 1 biosolids had a moderate P<sub>WEP</sub> (Disney, 8.4%), and 4 biosolids had high P<sub>WEP</sub> (≥15%) values: Lakeland NS, OCUD S, GRU, Disney, and Boca Raton. Bahiagrass was grown and harvested monthly for 4 months. After each harvest, columns were leached to attain ~500 mL (1/2 pore volume) drainage. Statistical analysis (ANOVA) showed significant treatment (biosolids), rate (P-rate), and rate X treatment effects on

yield, P uptake, and P leaching (SAS Institute, 1999; Tables A-1, 2, and 3, respectively). Thus, results are discussed within each P application rate and not across P application rates.

## **Yields**

We provided excessive N and other macronutrients (S, K, Mg) to isolate P as the only nutrient variable. The lowest rate of P application ( $56 \text{ kg P ha}^{-1}$ ) provides more than adequate P for Bahiagrass. As a result, yields (Appendix Table A-4) were expected to be equal across all materials and application rates. Equal yields are critical since harvest yields are used to calculate P uptake. At the  $56 \text{ kg P ha}^{-1}$  application rate, there were no significant differences in cumulative yield between biosolids source treatments, and all treatment yields were significantly greater than the control (Figure 3-5a). At the  $112 \text{ kg P ha}^{-1}$  application rate, all treatment yields were greater than the control, but the cumulative yield for OCUD S biosolids was greater than yields for TSP, Disney, and GreenEdge (Figure 3-5b). At the  $224 \text{ kg P ha}^{-1}$  application rate (Figure 3-5c), all the yields for all materials except for Lakeland NS and Disney were different from the control. The cumulative yield for the Lakeland NS material was smaller than yields for all other materials with the exception of the Disney compost. Yields at the  $224 \text{ kg P ha}^{-1}$  application rate for Bahiagrass grown in soil amended with Lakeland NS were highly variable, and Bahiagrass growth was inexplicably reduced in 2 of the 4 soil columns. At the high P rate, the cumulative yield of the OCUD S biosolids was not different from the yields of the other materials (Figure 3-5c). Because ANOVA showed rate and treatment interactions affecting yield, we cannot statistically compare yields for treatments across rates. Figure 3-6 shows that most cumulative yields approached the average yields across treatments (except controls) ( $\sim 28 \text{ mg}$ ; horizontal line on Figure 3-6).

To validate that Bahiagrass N uptake was sufficient, we analyzed tissue from all four harvests at the highest rate of P application (Figure 3-7). We analyzed only tissue from the 224 kg P ha<sup>-1</sup> rate because most of the N at this rate was assumed to come from N mineralization from the biosolids. If we misjudged the N mineralization rate to be 40%, we would anticipate N deficiencies to be more apparent at the 224 kg P ha<sup>-1</sup> rate. Recall that N was equalized across P application rates and treatments using ammonium nitrate. More ammonium nitrate was needed at the 56 and 112 kg P ha<sup>-1</sup> rates than at the 224 kg P ha<sup>-1</sup> rate. The ammonium nitrate would be immediately available to the Bahiagrass, thus we would not expect N deficiencies due to slower (or smaller) than expected N mineralization from the biosolids. Tissue N concentration was used to calculate yield-weighted N concentrations for each treatment over the entire growing season using equation 3-1.

where

trt = treatment

H1 = harvest

H2 = harvest 2

$$\frac{\text{trt yield}_{H1} \times N_{H1} + \text{trt yield}_{H2} \times N_{H2} + \text{trt yield}_{H2} \times N_{H2}}{\text{cumulative trt yield}} \quad (3-1)$$

Measured N concentrations were sufficient for grazing beef cattle (minimum 1.12 g N g<sup>-1</sup>; NRC, 1996). Based on the N content per harvest (Figure 3-7), it appears we underestimated the quantity of N that would mineralize from the biosolids initially (first harvest), and overestimated N mineralization throughout the rest of the growing season. Figure 3-8 shows that yield-weighted N concentrations for all biosolids treatments were above the minimum N concentrations required for beef cattle. The yield-weighted tissue N concentrations were sufficient for us to believe N was not affecting yields. Micronutrients were also considered as the source of yield variation. Micronutrients were not supplied during the growing season, however

research has shown micronutrient deficiencies in Bahiagrass are rare and do not affect yields (Chambliss and Adjei, 2006).

### **P Uptake and Relative Phytoavailability**

The 224 kg P ha<sup>-1</sup> rate of P application resulted in the greatest cumulative P uptake (Figures 3-9a – c; Appendix Table A-2). P uptake ranged from 23-52 % of P applied at the low application rate (Figure 3-9a). In a similar study by O'Connor et al. (2004), P uptake ranged from 29-57% of applied P at the same low P application rate. At the 224 kg P ha<sup>-1</sup> rate, P uptake in the current study ranged from 7-28% (Figure 3-9c), whereas O'Connor et al. (2004) reported P uptake ranging from 11-29% of applied P at the 224 kg P ha<sup>-1</sup> rate. Recall that yields for Bahiagrass grown in two replicates of soil amended with the Lakeland NS biosolids were reduced at the highest rate of P application; therefore, average P uptake at this rate was lowered. Cumulative yield-weighted tissue P concentrations ranged from 0.5 g kg<sup>-1</sup> to 4.5 g kg<sup>-1</sup> (Table 3-7). Bahiagrass grown in soil with no added P (control) accumulated the least P, and soil amended with TSP at 224 kg P ha<sup>-1</sup> resulted in the highest yield-weighted P concentration. Adjei and Rechcigl (2002) suggested that a sufficient tissue P concentration for Bahiagrass is 2.0 g kg<sup>-1</sup>. However, the critical concentration (P concentration necessary for survival) for Bahiagrass is likely lower, and may even be closer to 1.0 g kg<sup>-1</sup> (personal communication, Dr. Jerry Sartain, 2007). Milorganite applied at the 56 and 112 kg P ha<sup>-1</sup> resulted in tissue P concentrations slightly below the hypothesized critical value of 1.0 g kg<sup>-1</sup>. However, cumulative yields of Milorganite and GreenEdge treatments were not different from yields in the fertilizer-P (TSP) treatment (Figures 3-5a-c). Thus, the low tissue P concentrations in Milorganite and GreenEdge treatments did not limit above ground growth. Bahiagrass grown in control columns (no added P) yielded less than all other treatments. Over the course of the 4-month growing season, no above ground P

deficiencies were noted for any treatment. Plant roots were not examined for P-deficiency as the glasshouse study is ongoing.

Relative phytoavailability (RPP) was estimated using a slope-ratio approach. Cumulative P uptake was plotted as a function of P applied for each biosolids (Figure 3-10). Linear regressions were fit to the data for each P source with a common intercept of 8.48 mg (mean cumulative P uptake for the control columns). To calculate RPP, the slope of the regression line for each material was divided by the slope of the regression line for TSP (Table 3-7). O'Connor et al. (2004) used the same approach to estimate RPP for 12 biosolids on the Immokalee soil. Table 3-7 also lists RPP values for biosolids produced or marketed in Florida determined by O'Connor et al. (2004). We attempted to fit both linear and 2<sup>nd</sup> degree polynomials to the data from the current study. The polynomials resulted in slightly greater  $r^2$  values, but greater coefficients of variability (CV) (Table 3-8). Comparison of polynomials by comparing slopes is also difficult, leading us to choose linear regressions to determine RPP. While a linear regression through only the 56 and 112 kg P ha<sup>-1</sup> rates had slightly higher  $r^2$  and lower CV values than linear regressions through all 3 rates,  $r^2$  and CV values for regressions including all 3 rates were acceptable and significant at  $p \leq 0.05$  (Table 3-8). Recall the purpose of including 3 rates of P application was to better define the RPP curve; therefore, we opted to include all rates for regression fit.

O'Connor et al. (2004) proposed 3 groupings of relative phytoavailability: high (>75% of TSP), moderate (25-75% of TSP), and low (<25% of TSP). With the exceptions of Disney, all of the BPR and BPR-like materials fit into the high RPP category. The Disney biosolids is a composted mixture of BPR biosolids and yard waste, with PWEP (8.4%) and PSI (0.43) values less than the PWEP and PSI of the other BPR biosolids included in the glasshouse study. The

PSI value for the Disney biosolids indicates a greater quantity of Fe and Al than the BPR or BPR-like biosolids. Stratful et al. (1999) reported that BPR biosolids contained more phytoavailable P compared to conventionally produced residuals.

The OCUD S material was 110% as phytoavailable as TSP. Interestingly, the OCUD S material is not a BPR material, but has a high PWEF (21%), exceeding to the average PWEF value (14%), for BPR materials (Brandt et al., 2004). To calculate the regression slope for Lakeland NS biosolids, 2 P uptake replicates were excluded at the 224 kg P ha<sup>-1</sup> rate. Recall that Bahiagrass yields were inexplicably reduced in 2 replicates of the Lakeland NS amended soil at the high P rate, reducing P uptake. Excluding data points reduces power of statistical comparison; however when cumulative yields from all 4 replicates were used for regression, the linear correlation was very poor ( $r^2 = 0.09$ ). Thus, the regression coefficient and  $r^2$  values for Lakeland NS listed in Table 3-7 reflect only 2 replications.

The linear regression model described the data for Milorganite poorly ( $r^2 = 0.51$ ). We, therefore, estimated RPP values for Milorganite via point estimates (Table 3-9) calculated using equation 3-2.

$$\text{Point Estimate RPP} = \frac{(\text{P uptake}_{\text{source}} - \text{P uptake}_{\text{control}}) / \text{P applied}_{\text{source}}}{(\text{P uptake}_{\text{TSP}} - \text{P uptake}_{\text{control}}) / \text{P applied}_{\text{TSP}}} \quad (3-2)$$

At the 56, 112, and 224 kg P ha<sup>-1</sup> rates, P in Milorganite was 38%, 31%, and 23% as phytoavailable as TSP, respectively. Thus, on average, Milorganite is ~31% as phytoavailable as TSP, placing it in the lower end of the moderate category of RPP values proposed by O'Connor et al., (2004). Milorganite would be assumed *a priori* to be in the low category of RPP values, based on PWEF (0.58%) and PSI (0.55) values, however the data support a moderate RPP for Milorganite. We also fit the P uptake data for Milorganite to a plateau model (Equation 3-2, Figure 3-10).

where

a = concentration of P in control (no added P)

b = P concentration plateau increment above the background (mg)

c = rate constant for change in uptake for a given change in the cumulative amount of P applied

P applied = rate of P application

$$\text{Uptake (mg)} = a + b(1 - e^{-c \cdot P_{\text{applied}}}) \quad (3-2; \text{Barbarick et al., 1995})$$

The good fit of the data to the plateau model confirms the unique P characteristics of Milorganite. We can calculate a similar RPP for Milorganite by dividing the mean cumulative P uptake for Milorganite (30.9 mg) by the mean cumulative P uptake for TSP (106 mg) at the 224 kg P ha<sup>-1</sup> application rate: 30.9 mg / 106 mg \* 100 = 29% RPP. Even at the high P loads (224 kg P ha<sup>-1</sup>) associated with N-based application rates, Milorganite is only ~29% as phytoavailable as TSP (consistent with point estimate).

The regression equation for the Lakeland NS uptake data resulted in a weak r<sup>2</sup> value (0.54), so we confirmed the RPP calculated via the slope-ratio approach using point estimates (Table 3-9). Averaging the point estimates for 56, 112, and 224 kg P ha<sup>-1</sup> resulted in a RPP of 90% for the Lakeland NS biosolids (consistent with slope-ratio approach).

We attempted to correlate RPP with biosolids-PWEP, but the correlation did not adequately explain the relationship between RPP and biosolids-PWEP (r = 0.64). However, when RPP is plotted as a function of biosolids-PWEP, (Figure 3-12) a logarithmic relationship exists (r<sup>2</sup> = 0.79). The logarithmic correlation is not strong enough to predict RPP using PWEP. However, it appears that biosolids-PWEP ≥ 14% segregates biosolids with greater RPP values from biosolids with lesser RPP values. Recall the same trend was noted with the P release data from the dynamic laboratory incubation; biosolids with PWEP values ≥ 14% could be assumed to result in a larger negative environmental impact than biosolids with PWEP values < 14%. In the glasshouse study, biosolids with PWEP values < 14% had RPP values below 60% (moderate

RPP category), compared to biosolids with PWEF values  $\geq 14\%$ , which had RPP values  $\geq 90\%$  (high RPP category).

The glasshouse study conducted here, and earlier work by O'Connor et al. (2004), demonstrates the wide range of RPP values possible for various biosolids. Knowledge of biosolids-RPP is key to adjusting application rates, should P-based limitations on biosolids land application be imposed. Results of the glasshouse study indicate that no change in biosolids application rate is needed (or justified) for BPR and BPR-like biosolids with RPP values in the high RPP category. However, application rates for Disney and GreenEdge could be approximately doubled that necessary to meet crop P needs. Based on the plateau model, Milorganite could be applied at rates  $\geq 224 \text{ kg P ha}^{-1}$ , and will only be about 1/3 as available as TSP. The low RPP of Milorganite does not necessarily imply that Milorganite is a poor P-fertilizer; Bahiagrass grown in Milorganite-amended soil showed no signs of above ground P deficiency during the course of the experiment and Bahiagrass yields were equivalent to TSP treatment yields for each application rate.

### **P Leaching**

A similar glasshouse study by Elliott et al. (2002) used a base sand with minimal P-sorbing capacity. The base sand used in this study had moderate P-sorbing capacity (RPA = 8.6%). Elliott et al. (2002) showed that a Florida sand with even moderate P-sorbing capacity (RPA=15.3%) can limit P leaching. Minimal P leaching occurred in the current glasshouse. Only TSP and BPR materials applied at the highest rate resulted in significant P leaching (Appendix Table A-3). The base sand is likely retarding the advance of P through the soil column. This study is on going, and it is possible that P retained in the column through the first growing

season will eventually leach during subsequent croppings. Given the deep rooting of Bahiagrass, the P sorbed to the base sand is also accessible for plant uptake.

Table 3--1. Materials received for laboratory characterization

Material	Class	Stabilization	BPR (Y/N)	Final Treatment	WWTP
<b>Milorganite</b>	AA	Aerobic	N	Thermally dried	Milwaukee, WI
Broward	B	Anaerobic	N	Thickened, dewatered	North District
Jacksonville (JEA) Cake	AA	Anaerobic	N	Thickened, dewatered	Buckman
<b>GreenEdge</b>	AA	Anaerobic	N	Thermally dried	Buckman
Lakeland Glendale	AA	Aerobic	N	Thickened, dewatered, Bioset™ system	Glendale
<b>Lakeland North Side</b>	AA	Aerobic, ATAD system	N	Thickened, dewatered	North side
Clay Fleming	B	Aerobic	N	Thickened, bio-N removal	Fleming Island
Clay Miller	B	Anaerobic/Lime Stab. <sup>a</sup>	N	Thickened, dewatered	Miller Street
<b>OCUD S Cake</b>	B	Anaerobic	N	Thickened, dewatered, bio-N removal	South
OCUD E Cake	NA <sup>b</sup>	Un-stabilized	Y	Thickened, dewatered	Eastern
OCUD E Dry	NA	Un-stabilized	Y	Thermally dried	Eastern
Tampa	AA	Anaerobic	N	Thermally dried	Howard F. Curren
West Palm Beach Central	AA	Aerobic	N	Composted material	East Central Region
<b>Disney Compost</b>	AA	Composted	Y	Composted material	Reedy Creek
Pinellas Cake	B	Aerobic	N	Thickened, dewatered	South Cross Bayou
Pinellas Dry	AA	Aerobic	N	Thermally dried	South Cross Bayou
Tallahassee Smith	AA	Anaerobic/aerobic	N	Thickened, dewatered	Thomas P. Smith
Orlando City	B	Lime Stabilized	N	Thickened, dewatered	Iron Bridge
<b>Boca Raton</b>	B	Anaerobic	Y	Thickened, dewatered	Glades Road
<b>Gainesville Regional Utilities (GRU)</b>	B	Aerobic	N	Thickened	Kanapaha
Ocala	AA	Anaerobic	N	Thermally dried	City of Ocala #3

<sup>a</sup>Time stabilized. <sup>b</sup>NA = not applicable. Bolded materials used in glasshouse study.

Table 3-2. Selected chemical and physical properties of biosolids

P Source	C	N	N	C to N ratio	Solids	LOI <sup>b</sup>	pH	EC	Fe	Al	Ca
	Average		Producer <sup>a</sup>	%		g kg <sup>-1</sup>					
Milorganite <sup>g</sup>	360	60	60	6.0	95	72	6.3	2900	41	2.7	15
Milorganite Greens Grade	370	62	60	5.9	95	74	6.0	3630	39	3.0	19
Broward County	390	53	69	7.3	13	74	7.9	3570	16	6.6	39
Jacksonville (JEA) Cake	390	57	60	6.8	20	76	8.2	3170	11	7.1	24
GreenEdge <sup>g</sup>	350	50	52	7.0	93	71	6.9	2600	15	8.4	25
Lakeland Glendale	170	30 <sup>c</sup>	24	5.7	35 <sup>c</sup>	17	12 <sup>c</sup>	6200	2.2 <sup>c</sup>	3.16 <sup>c</sup>	310 <sup>c</sup>
Lakeland North Side <sup>g</sup>	380	59	70	6.4	3	75	8.5	6550	4.5	11	22
Baltimore	300	43	52	7.1	87	61	5.9	2300	60	24	15
Clay Fleming	340	55	44	6.2	2	69	7.8	245	51	9.0	13
Clay Miller	350	54	NP <sup>d</sup>	6.3	19	64	9.3	932	6.6	6.8	119
Orange County South <sup>g</sup>	380	60	71	6.3	11	76	7.8	2750	5.4	5.6	24
Orange County East Cake	430	70	82	6.2	16	84	5.9	2700	2.2	7.7	17
Orange County East Dry	430	70	NP	6.2	95	84	5.9	1069	1.9	7.5	17
Tampa	410	56	52	7.4	96	76	7.1	743	8.8	8.8	42
West Palm Beach Compost	420	16	NP	26	64	56	6.5	2200	ND <sup>e</sup>	ND	ND
Disney Compost <sup>g</sup>	410	28	22	15	66	77	5.7	5190	37	10	16
Pinellas Cake	310	43	54	7.3	24	68	7.5	1173	27	15	27
Pinellas Dry	320	48	62	6.8	92	69	6.9	1157	28	19	35
Tallahassee Smith	430	65	41	6.6	98	20	5.9	1673	8	13	26
Orlando City	280	42	51	6.6	20	49	13	7730	2.6	3.5	159
Boca Raton <sup>g</sup>	370	62	50	6.0	13	76	7.5	3390	12	6.4	25
GRU <sup>g</sup>	400	64	62	6.2	5	80	6.4	1108	7.7	5.5	14
Ocala	ND	ND	NP	ND	93	ND	5.9	1833	11	9.8	20
TSP <sup>fg</sup>	ND	ND	ND	ND	92	21	5.9	ND	16	10	140

<sup>a</sup>Producer supplied data. <sup>b</sup>LOI = loss on ignition. <sup>c</sup>Data from Brandt and Elliott, 2005. <sup>d</sup>NP = data not provided. <sup>e</sup>ND = not determined.

<sup>f</sup>TSP data from O'Connor et al. (2004). <sup>g</sup>Materials used in glasshouse study.

Table 3-3. Selected phosphorus properties of materials

P Source	Total P		Fe-Strip		Oxalate Extractable		PSI		
	Determined	Producer <sup>a</sup>	P	WEP	PWE <sup>b</sup>	P		Al	Fe
Milorganite <sup>g</sup>	21	23	0.08	0.12	0.58	16	1.2	25	0.55
Milorganite Greens Grade	20	23	ND	0.19	0.85	15	1.4	24	0.53
Broward County	20	19	0.08 <sup>b</sup>	1.3 <sup>b</sup>	6.7	18	6.0	5.0	2.0
Jacksonville	15	20	0.05	0.31	2.0	13	10	5.0	1.2
GreenEdge <sup>g</sup>	17	19	0.12	0.19	1.1	13	5	13	1.0
Lakeland Glendale	11 <sup>b</sup>	ND <sup>c</sup>	ND	0.08 <sup>†</sup>	0.92 <sup>†</sup>	1.9	0.4	0.4	NA <sup>d</sup>
Lakeland NS <sup>g</sup>	29	29	0.35	14	47	22	3.2	8.3	2.0
Baltimore	23	27	ND	0.04	0.15	21	12	52	0.5
Clay Fleming	31	18	ND	0.47	1.4	32	8.1	52	0.83
Clay Miller	16	ND	ND	0.04	0.23	4.2	1.4	1.8	1.6
Orange County South <sup>g</sup>	23	30	0.14	4.8	21	23	5.0	4.4	2.9
Orange County East Cake	20	23	0.34	8.0	41	17	1.3	3.1	4.1
Orange County East Dry	23	23	1.4	2.7	11	22	4.5	1.1	3.8
Tampa	21	25	0.29	0.07	0.29	17	5.6	6.0	1.8
West Palm Beach Compost	7.9	ND	ND	0.71	8.1	9.1	1.8	6.5	1.6
Disney Compost <sup>g</sup>	11	27	0.95	0.02	0.22	11	7	20.0	0.43
Pinellas Cake	31	41	ND	0.27	0.78	30	11	12	1.6
Pinellas Dry	33	41	0.11	0.16	0.44	27	14	32	0.79
Tallahassee Smith	21	31	ND	1.4	6.1	19	6.5	5.2	1.8
Orlando City	17	20	0.003	0.04	0.23	13	1.7	2.4	NA
Boca Raton <sup>g</sup>	26	39	0.02	3.9	15	33	14	7.3	2.1
GRU <sup>g</sup>	31	48	1.7	7.9	26	21	6.4	3.7	2.1
Ocala	21	28	0.74	0.63	3	20	11	5.6	1.5
TSP <sup>g</sup>	190	210	ND	170	85	186	11	6.8	NA

<sup>a</sup>Producer supplied data. <sup>b</sup>Data from Brandt and Elliott, 2005. <sup>c</sup>ND = not determined. <sup>d</sup>NA = not applicable. <sup>e</sup>Materials used in glasshouse study.

Table 3-4. Comparison of biosolids-TP concentrations determined by 3 digestion methods

P Source	EPA3050A	Anderson	Perchloric	Producer <sup>a</sup>
	g kg <sup>-1</sup>			
Milorganite	ND	21	24	23
Broward	23	20	26	19
JEA Cake	18	15	20	20
GreenEdge	ND	17	ND	19
Lakeland NS	37	29	38	29
OCUD S	26	21	32	30
OCUD E Cake	24	20	29	23
Disney	ND	11	ND	27
Orlando	18	17	19	18
Boca Raton	34	26	39	39
GRU	ND	31	ND	48
Standard Reference	22 (91%)	21 (88%)	23 (96%)	24

<sup>a</sup>Producer-supplied data. <sup>b</sup>ND: not determined.

Table 3-5. Dynamic laboratory incubation: cumulative net SRP released and SRP released as a percentage of applied P. Means  $\pm$  1 standard error, n = 3.

P Source	Rate kg P ha <sup>-1</sup>	Cumulative P released	Cumulative P released % of applied P
Control	0	0.67 $\pm$ 0.062	NA
TSP	56	8.33 $\pm$ 0.318	80.9 $\pm$ 3.08
TSP	224	30.5 $\pm$ 1.11	74.0 $\pm$ 2.69
Milorganite	56	3.96 $\pm$ 1.11	38.5 $\pm$ 10.8
Milorganite	224	7.84 $\pm$ 1.54	19.0 $\pm$ 3.73
GreenEdge	56	4.12 $\pm$ 0.442	40.0 $\pm$ 4.29
GreenEdge	224	11.3 $\pm$ 0.842	27.4 $\pm$ 2.04
Disney	56	5.66 $\pm$ 0.252	42.0 $\pm$ 1.34
Disney	224	21.6 $\pm$ 2.42	39.9 $\pm$ 1.80
GRU	56	6.90 $\pm$ 0.239	67.0 $\pm$ 2.32
GRU	224	28.2 $\pm$ 1.08	68.5 $\pm$ 2.63
Boca Raton	56	9.03 $\pm$ 0.505	87.7 $\pm$ 4.90
Boca Raton	224	32.7 $\pm$ 0.756	79.3 $\pm$ 1.84
Lakeland NS	56	9.27 $\pm$ 0.228	90.0 $\pm$ 2.21
Lakeland NS	224	25.7 $\pm$ 0.770	62.4 $\pm$ 1.87
OCUD E Cake	56	9.56 $\pm$ 0.347	92.8 $\pm$ 2.15
OCUD E Cake	224	32.9 $\pm$ 0.275	79.9 $\pm$ 1.14
OCUD E Dry	56	7.16 $\pm$ 0.221	69.5 $\pm$ 2.05
OCUD E Dry	224	25.7 $\pm$ 0.469	62.4 $\pm$ 1.05
OCUD S	56	10.0 $\pm$ 0.212	97.3 $\pm$ 3.37
OCUD S	224	33.0 $\pm$ 0.434	80.0 $\pm$ 0.667
Broward	56	6.33 $\pm$ 0.753	61.4 $\pm$ 7.31
Broward	224	13.0 $\pm$ 0.856	31.5 $\pm$ 2.08
Tallahassee	56	5.43 $\pm$ 0.138	52.8 $\pm$ 1.34
Tallahassee	224	16.6 $\pm$ 0.742	40.3 $\pm$ 1.80

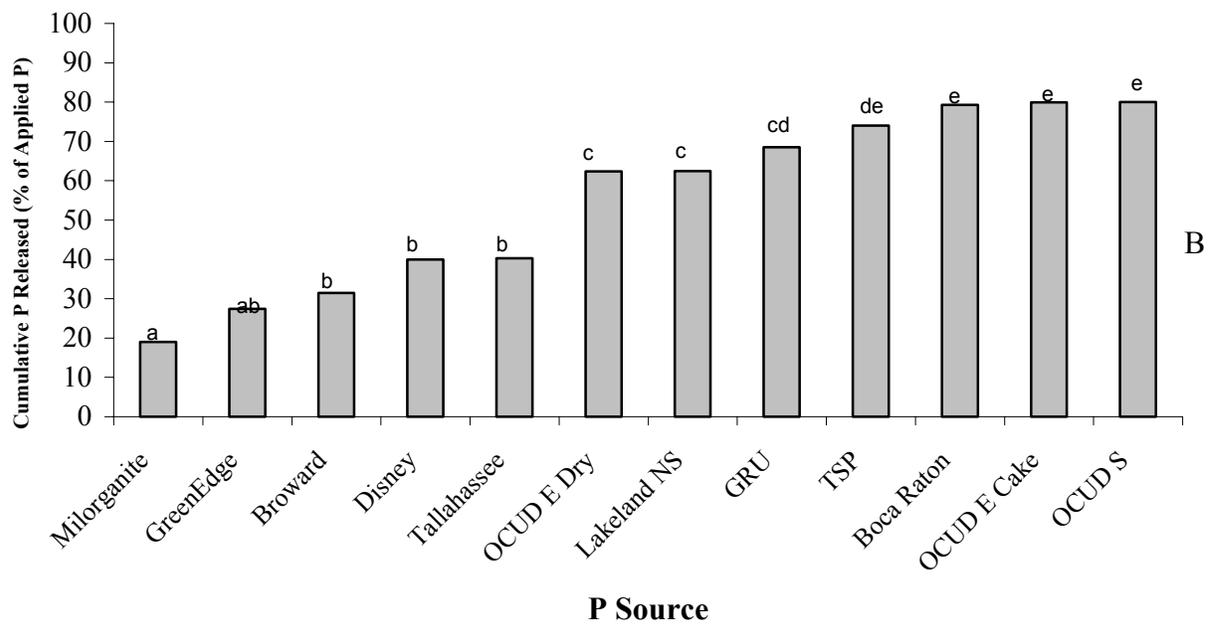
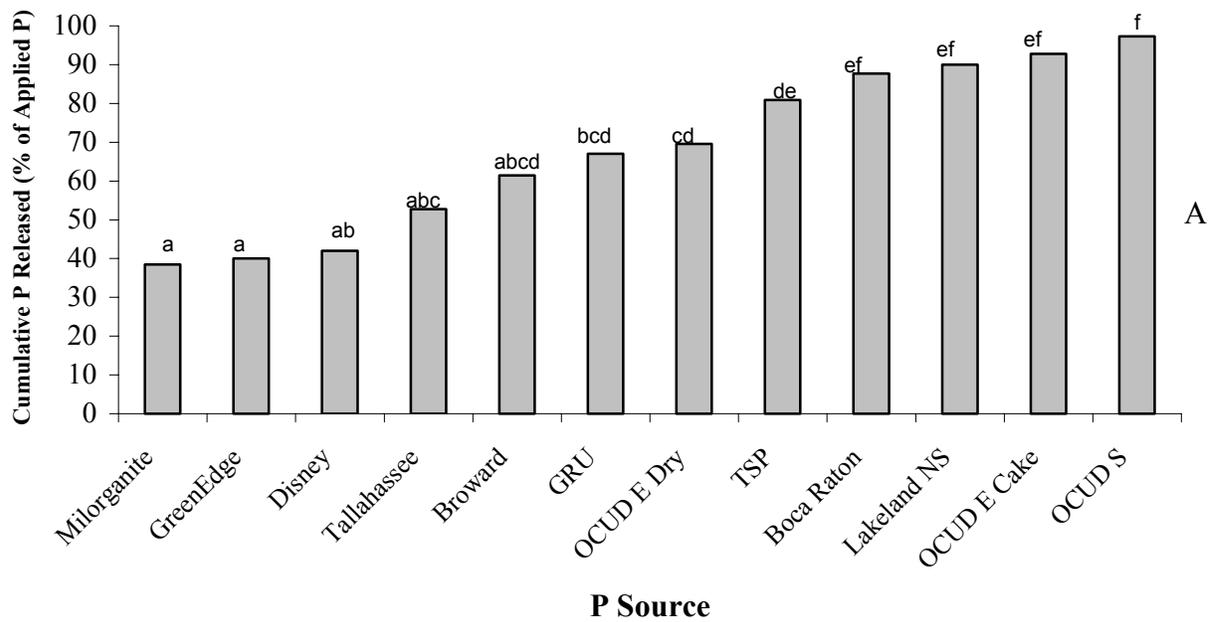


Figure 3-1. Cumulative SRP released (% of applied P) as a function of P source. Means capped with the same letter are not different (Tukey's Test,  $p \leq 0.05$ ). A) P-rate: 56 kg P ha<sup>-1</sup>. B) P-Rate: 224 kg P ha<sup>-1</sup>.

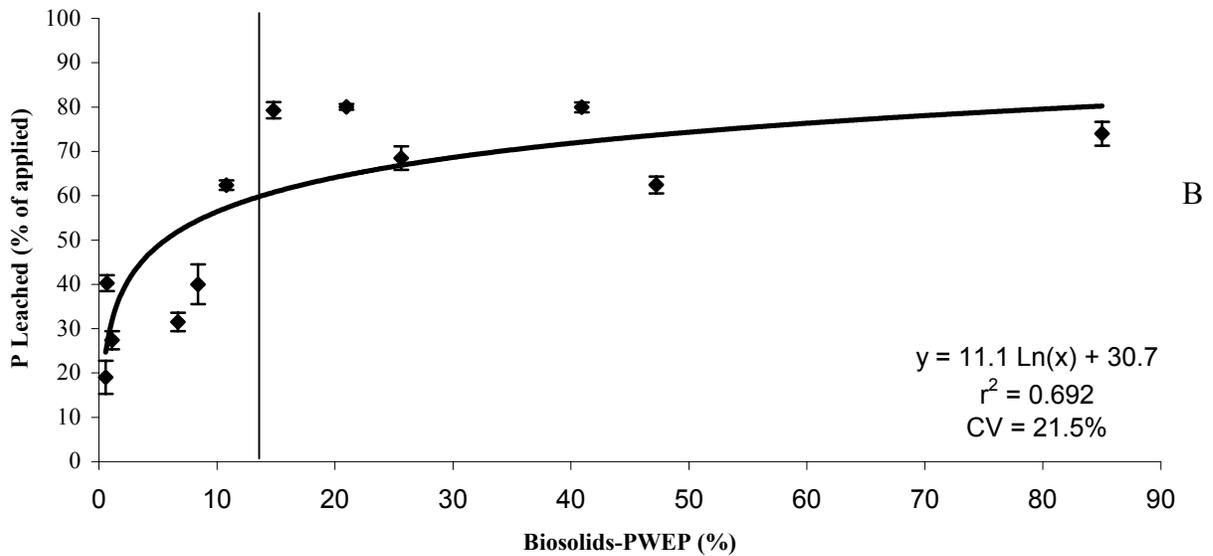
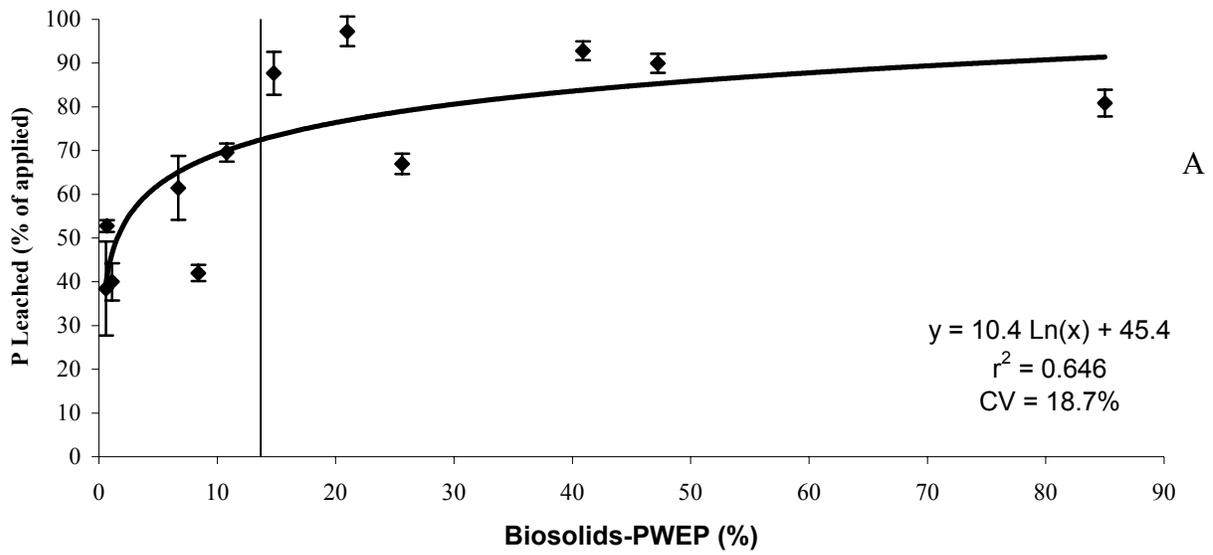


Figure 3-2. Dynamic laboratory incubation: cumulative P released (% of applied P) as a function of biosolids-PWEP. Error bars represent 1 standard error. Vertical line at biosolids-PWEP = 14% indicates when increased negative environmental impact may occur. A) P-Rate: 56 kg P ha<sup>-1</sup>. B) P-Rate: 224 kg P ha<sup>-1</sup>

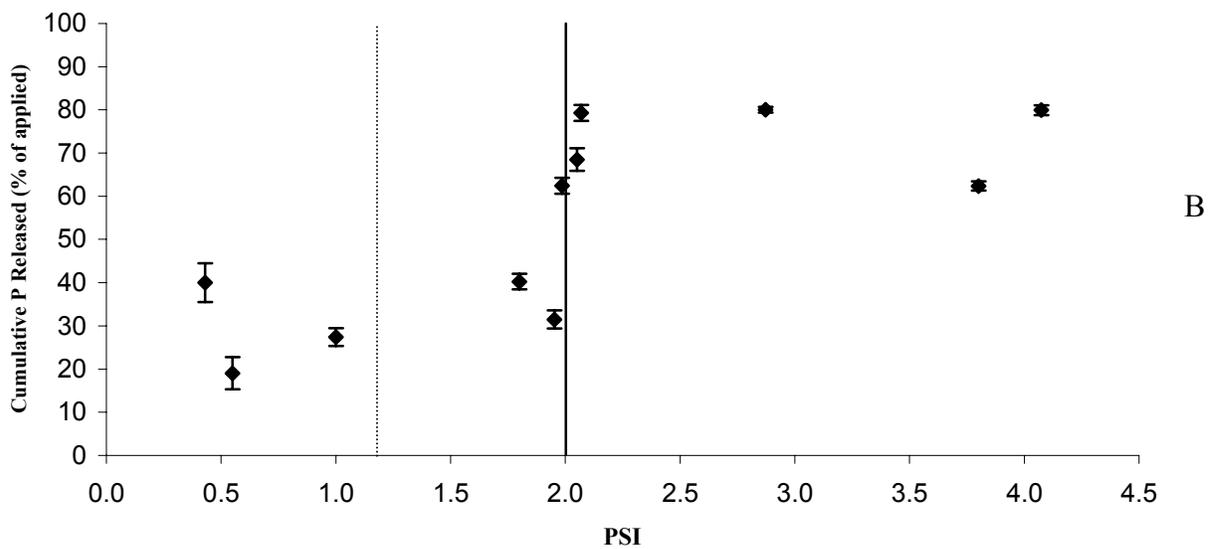
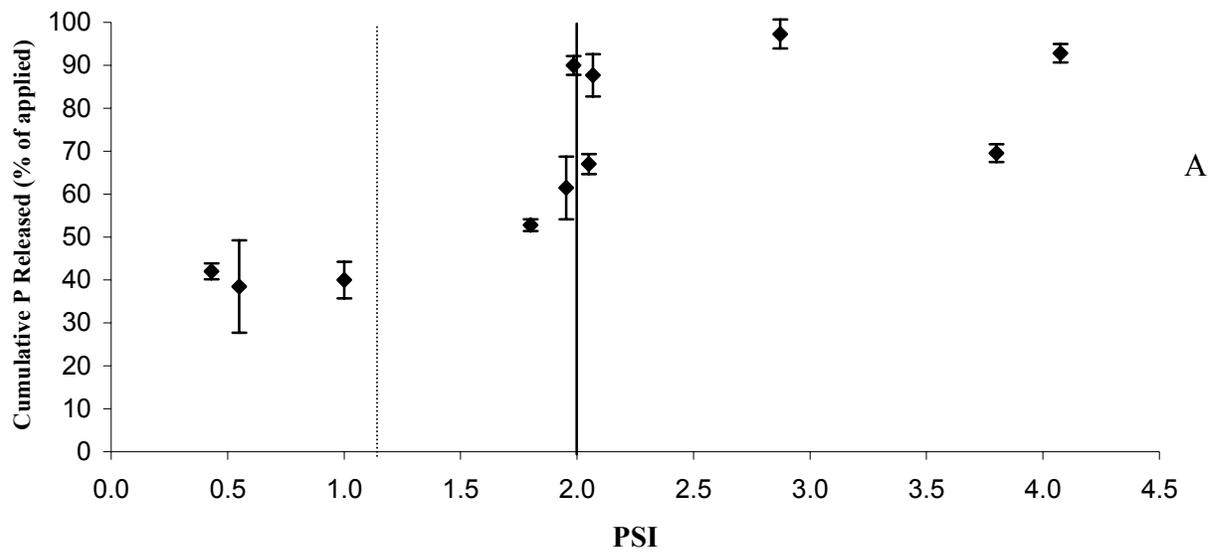


Figure 3-3. Dynamic laboratory incubation: cumulative P released as a function of PSI. Dashed vertical line at 1.1 PSI represents change point proposed by Elliott et al. (2002). Solid vertical line at 2.0 PSI represents increased critical PSI value suggested by data in the current study. A) P-Rate: 56 kg P ha<sup>-1</sup>. B) P-Rate: 224 kg P ha<sup>-1</sup>.

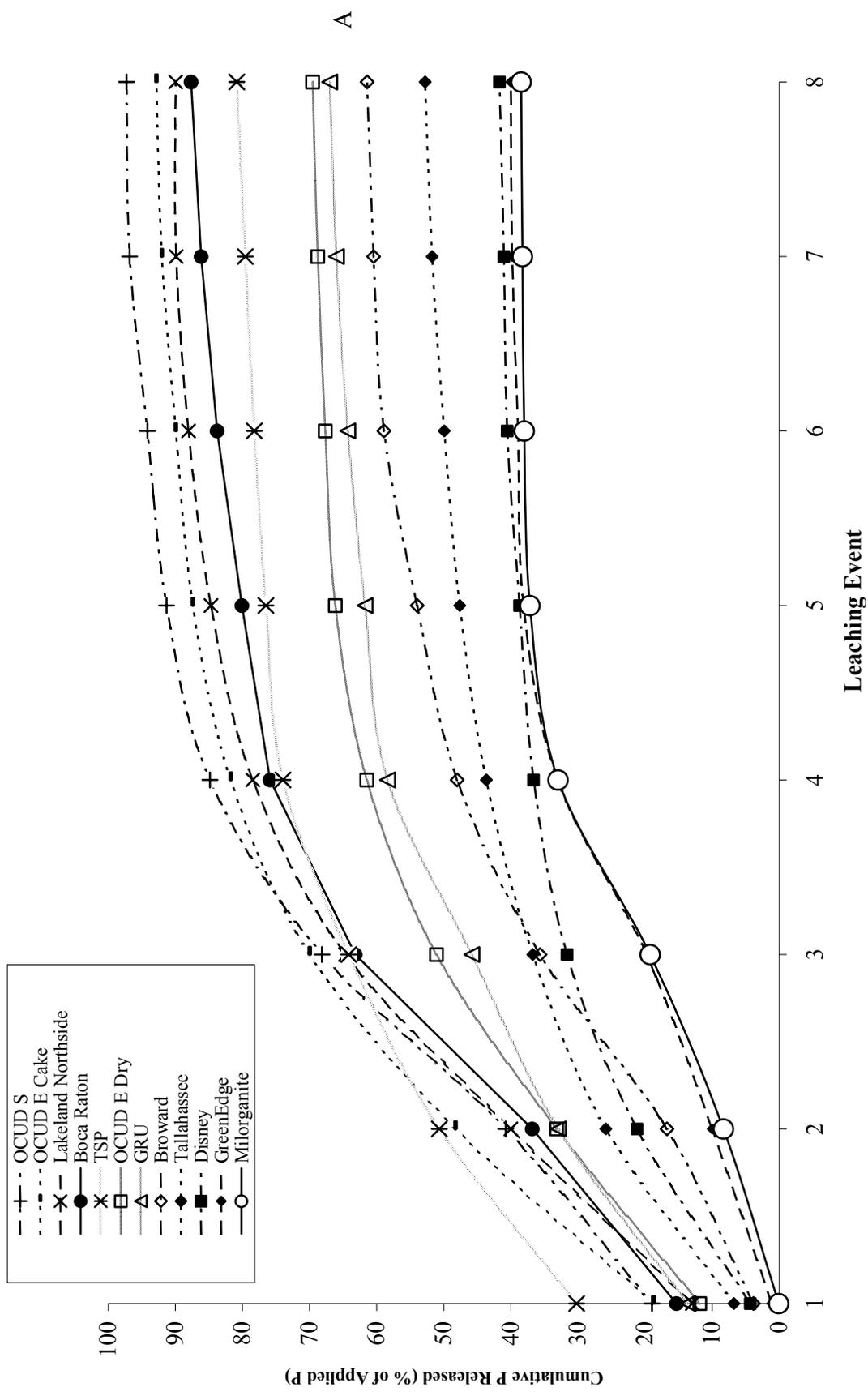


Figure 3-4. Dynamic laboratory incubation: cumulative P released (% of applied P) per leaching event. A) P-Rate: 56 kg P ha<sup>-1</sup>. B) P-Rate: 224 kg P ha<sup>-1</sup>.

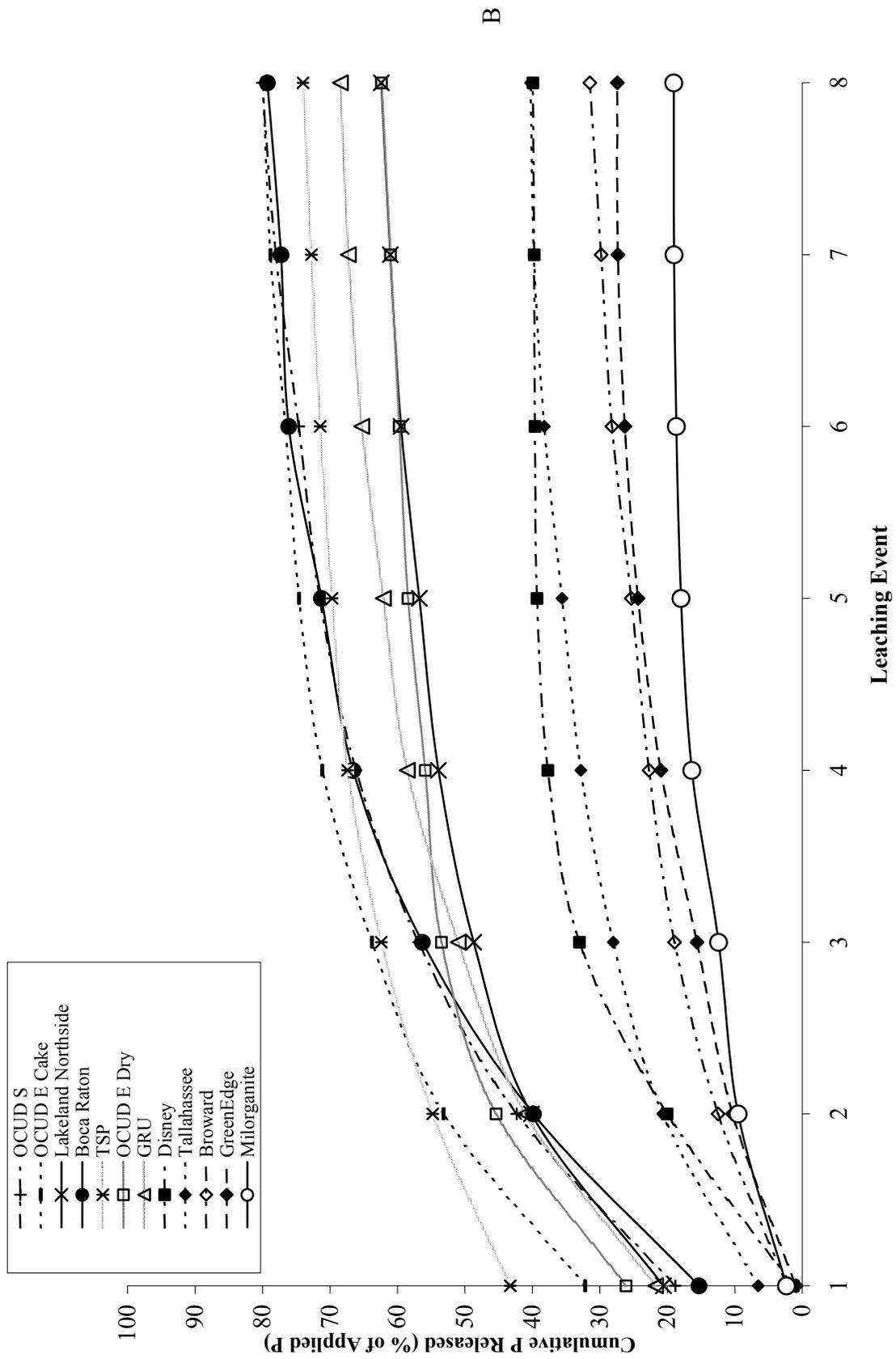


Figure 3-4. Continued

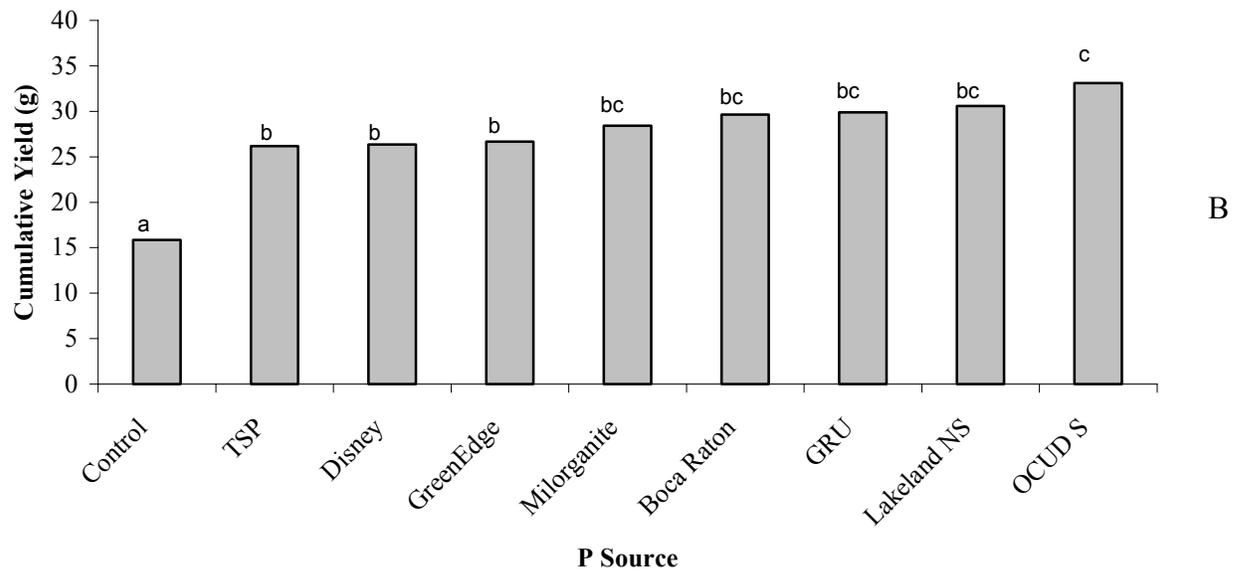
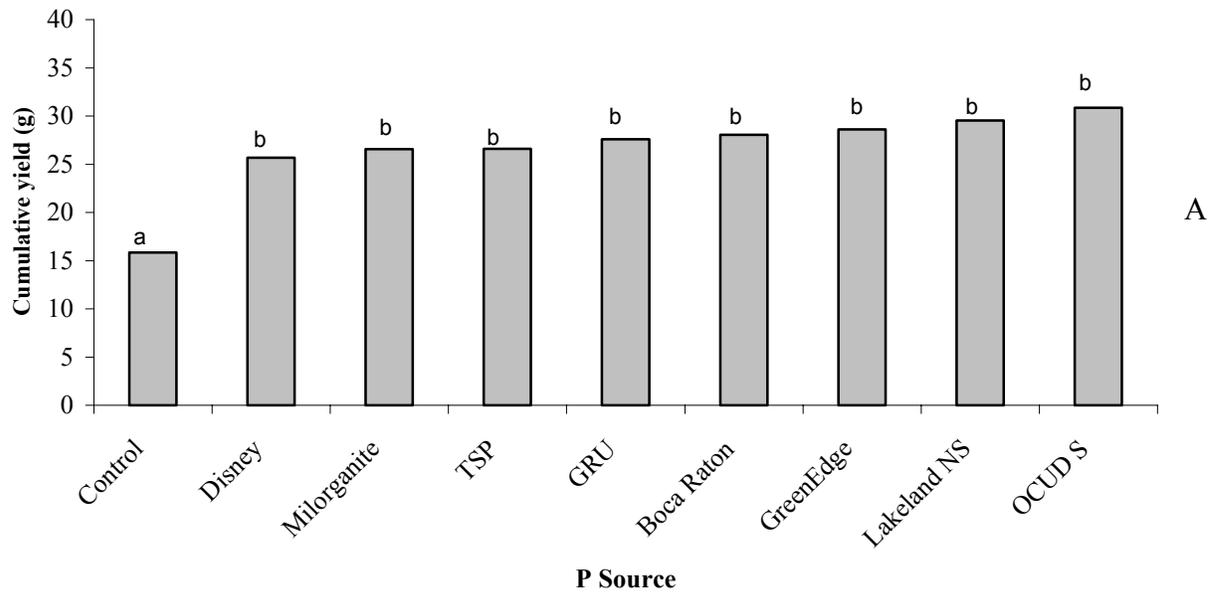


Figure 3-5. Cumulative Bahiagrass yields after 4 harvests. P-rate: 112 kg P ha<sup>-1</sup>. Means capped with the same letter are not different (Tukey Test, p≤0.05). A) P-Rate: 56 kg P ha<sup>-1</sup>. B) P-Rate: 112 kg P ha<sup>-1</sup>. C) P-Rate: 224 kg P ha<sup>-1</sup>.

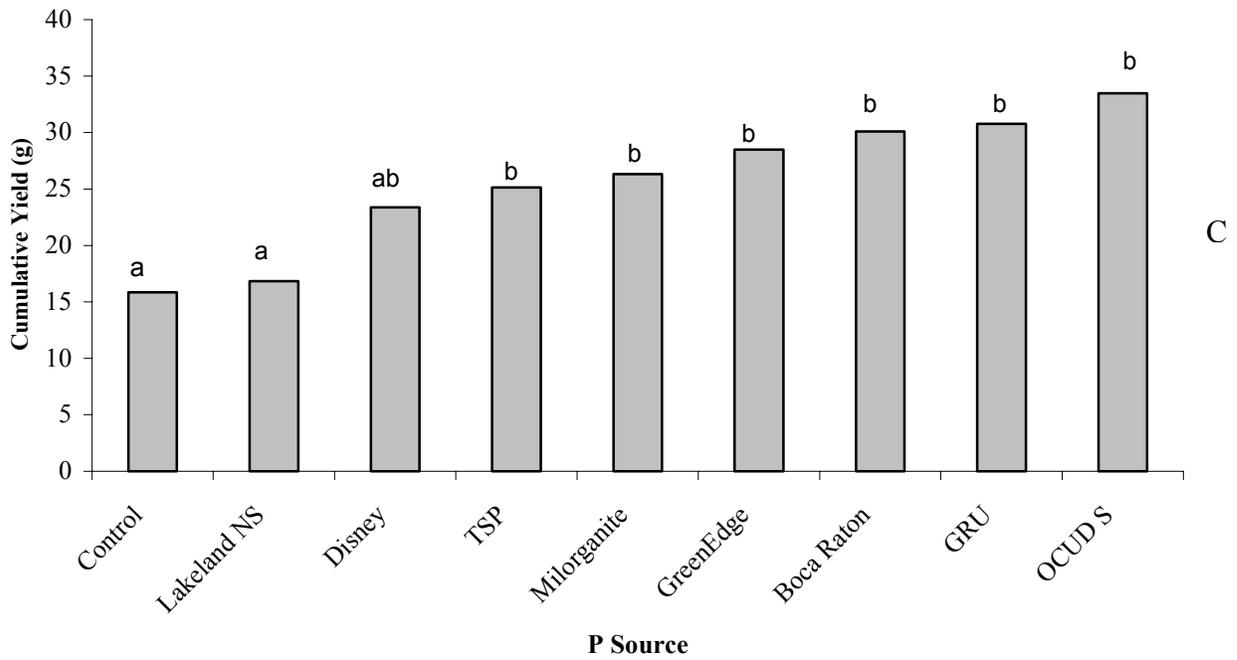


Figure 3-5. Continued

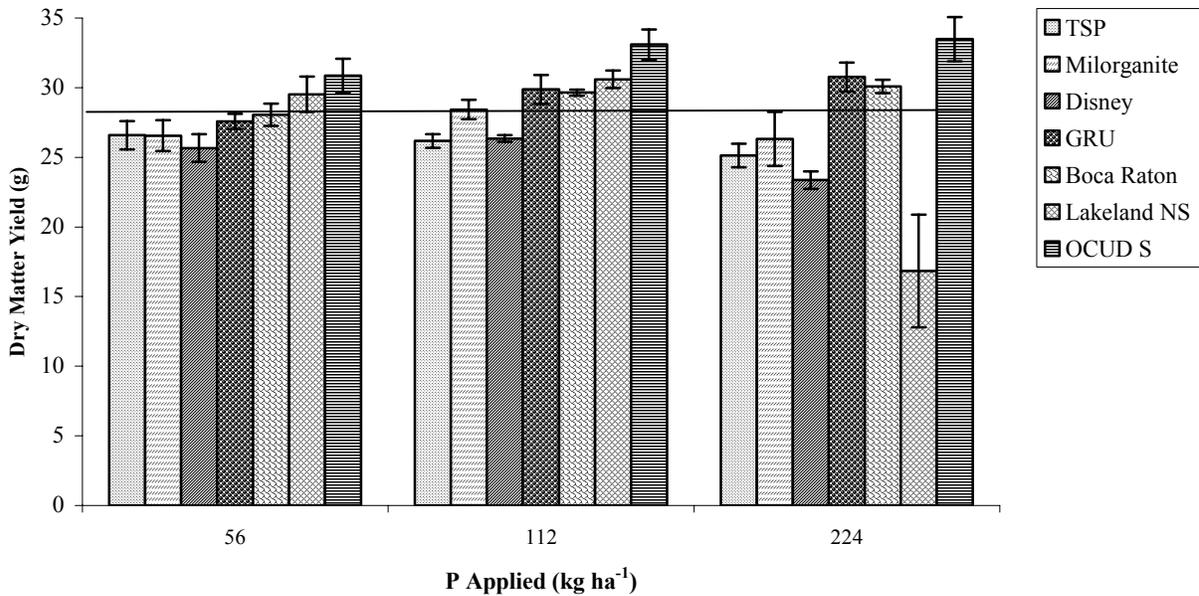


Figure 3-6. Cumulative Bahiagrass yields after 4 harvests for all P application rates. Horizontal line at ~28 g dry matter yield represents average dry matter yield across rates and treatments. Error bars represent 1 standard error.

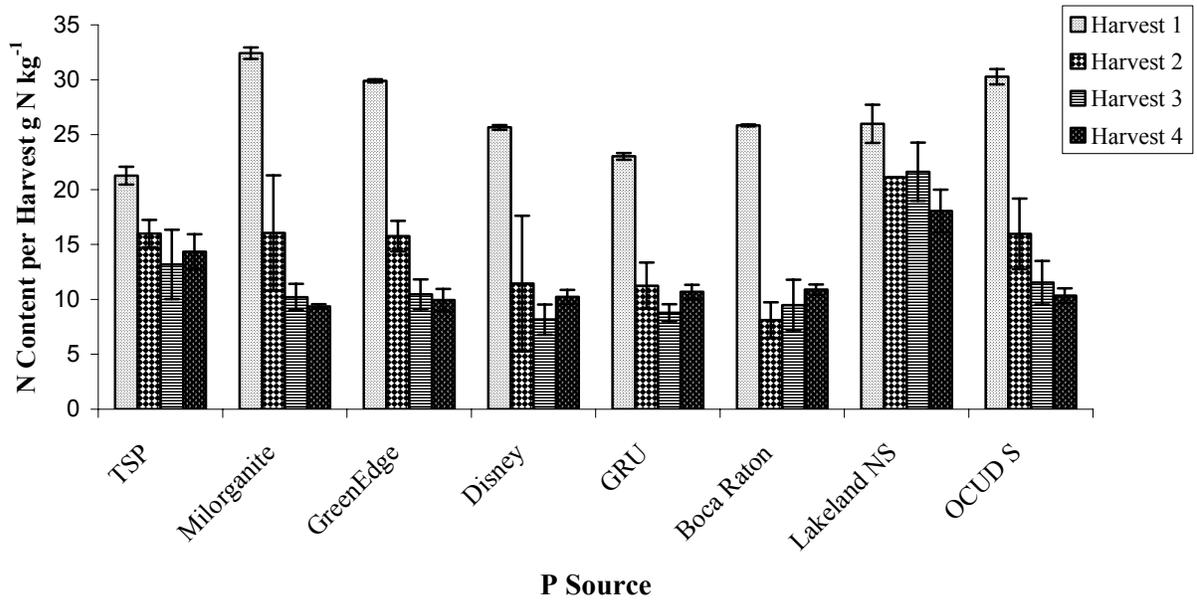


Figure 3-7. Bahiagrass N tissue concentration per harvest. P-rate: 224 kg P ha<sup>-1</sup>. Error bars represent 1 standard error.

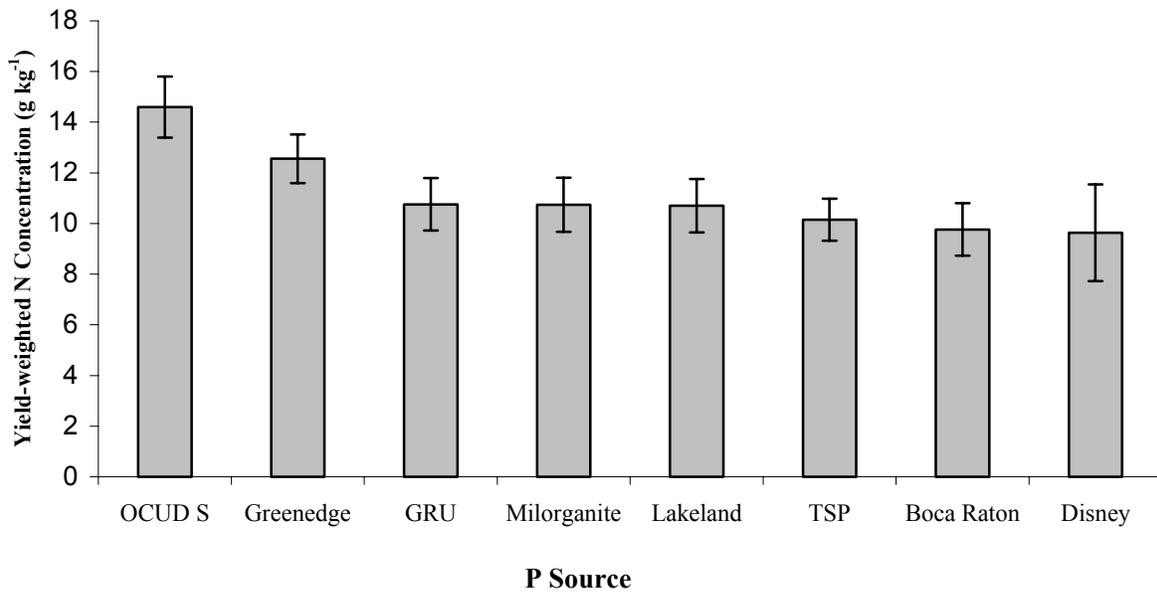


Figure 3-8. Bahiagrass yield-weighted N concentration after 4 harvests. P-rate: 224 kg P ha<sup>-1</sup>. Error bars represent 1 standard error.

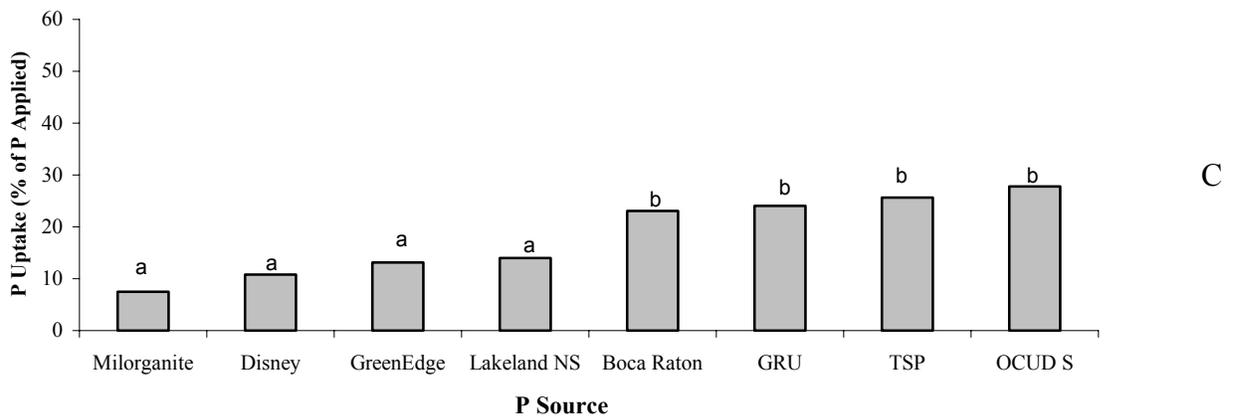
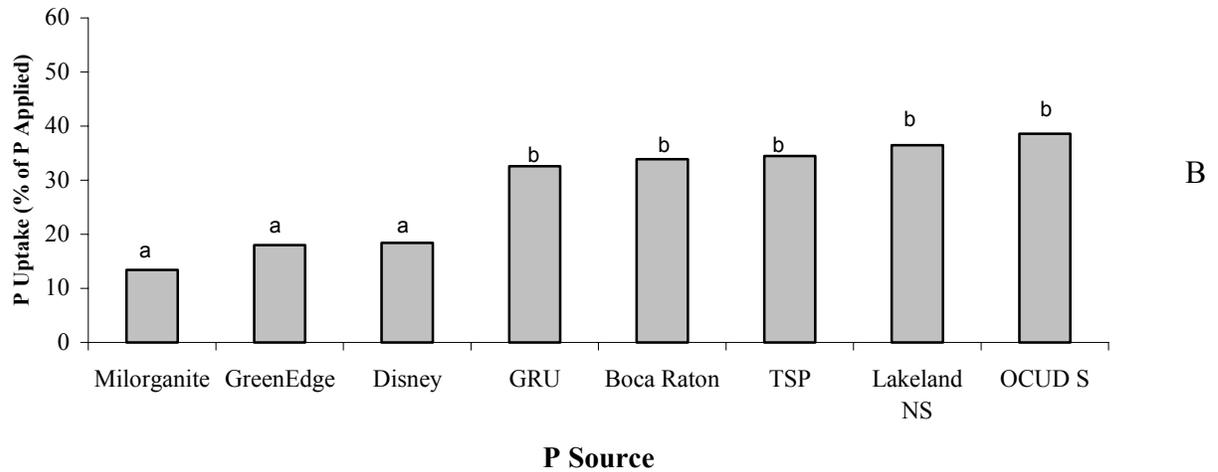
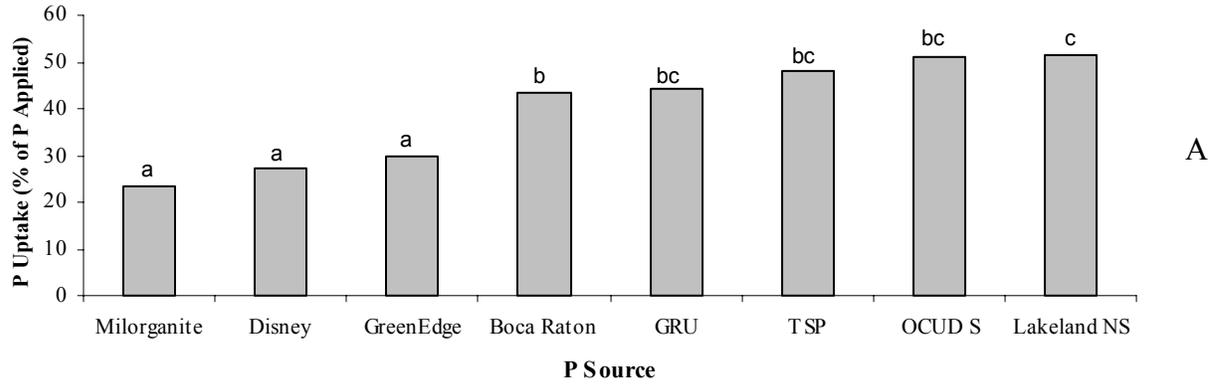


Figure 3-9. Cumulative Bahiagrass P uptake after 4 harvests. P-rate = 112 kg P ha<sup>-1</sup>. Means capped with the same letter are not different (Tukey Test, p≤0.05). A) P-Rate: 56 kg P ha<sup>-1</sup>. B) P-Rate: 112 kg P ha<sup>-1</sup>. C) P-Rate: 224 kg P ha<sup>-1</sup>.

Table 3-6. Cumulative Bahiagrass yield-weighted tissue P concentrations after four harvests.

P Source	Rate	Yield-Weighted Tissue P concentration g kg <sup>-1</sup>
Control	0	0.531 ± 0.06
TSP	56	1.86 ± 0.09
TSP	112	2.71 ± 0.21
TSP	224	4.20 ± 0.40
Milorganite	56	0.91 ± 0.06
Milorganite	112	0.97 ± 0.10
Milorganite	224	1.18 ± 0.08
GreenEdge	56	1.07 ± 0.17
GreenEdge	112	1.39 ± 0.7
GreenEdge	224	1.90 ± 0.03
Disney	56	1.43 ± 0.11
Disney	112	1.90 ± 0.05
Disney	224	2.50 ± 0.10
GRU	56	1.65 ± 0.13
GRU	112	2.25 ± 0.09
GRU	224	3.22 ± 0.26
Boca Raton	56	1.59 ± 0.16
Boca Raton	112	2.35 ± 0.24
Boca Raton	224	3.16 ± 0.49
Lakeland NS	56	1.81 ± 0.11
Lakeland NS	112	2.45 ± 0.04
Lakeland NS	224	3.50 ± 0.21
OCUD S	56	1.71 ± 0.09
OCUD S	112	2.41 ± 0.14
OCUD S	224	3.42 ± 0.16

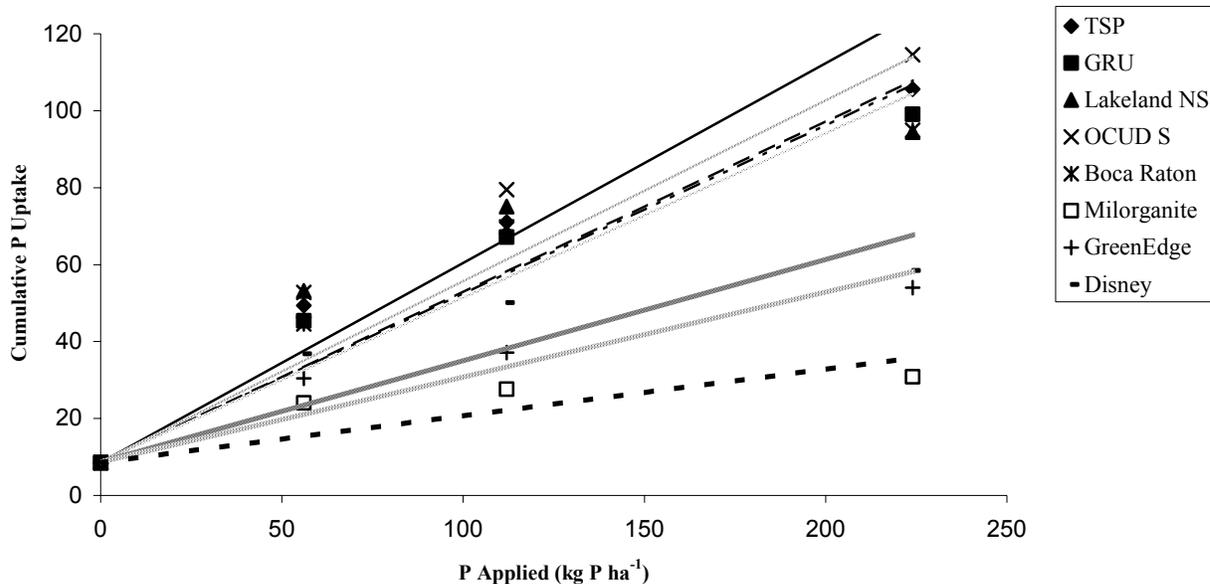


Figure 3-10. Cumulative Bahiagrass P uptake as a function of P applied

Table 3-7. R phosphorus phytoavailability of biosolids to TSP

P Source	r <sup>2</sup>	Linear Regression Coefficient	RPP (%)	Category	Reference Study
TSP	0.89	0.472	100	high	Current
OCUD S	0.90	0.519	110	high	Current
GRU	0.91	0.439	93	high	Current
Lakeland NS	0.54	0.435	92	high	Current
Boca Raton	0.86	0.429	91	high	Current
GreenEdge	0.88	0.222	47	moderate	Current
Disney	0.70	0.220	47	moderate	Current
Regression equation: $y = \text{---}x + 8.48$					Current
Milorganite			31*	moderate	Current
TSP		0.400	100	—	O'Connor et al. (2004)
Largo Cake		0.297	74	moderate	O'Connor et al. (2004)
Largo Pellets		0.193	48	moderate	O'Connor et al. (2004)
Baltimore Cake		0.136	34	moderate	O'Connor et al. (2004)
Tarpon Springs Cake		0.124	31	moderate	O'Connor et al. (2004)
Regression equation: $y = \text{---}x + 20.544$ ; $R^2 = 0.91$					O'Connor et al. (2004)

<sup>a</sup>Determined via point estimates.

Table 3-8. Various RPP estimate model fit statistics

P Source	2 Rates Linear		2 Rates Polynomial (2°)		All Rates Linear		All Rates Polynomial (2°)	
	r <sup>2</sup>	CV	r <sup>2</sup>	CV	r <sup>2</sup>	CV	r <sup>2</sup>	CV
TSP	0.94	14.0	0.98	22.1	0.89	19.3	0.96	29.4
GRU	0.96	12.1	0.98	19.2	0.91	17.5	0.98	23.0
Lakeland NS	0.90	18.8	0.95	37.2	0.54	38.3	0.97	78.8
OCUD S	0.97	10.2	1.0	12.4	0.90	18.8	0.98	22.7
Boca Raton	0.96	12.4	0.96	27.2	0.86	22.1	0.95	33.0
Milorganite	0.80	19.2	0.93	32.4	0.51	27.6	0.88	35.3
Disney	0.93	14.4	0.95	17.8	0.7	26.7	0.97	23.7
GreenEdge	0.87	17.1	0.97	20.4	0.88	17.5	0.97	12.6

Table 3-9. Point estimates of RPP for Milorganite and Lakeland NS biosolids

P Source	Rate	RPP %	Average
TSP	56	100	100
TSP	112	100	
TSP	224	100	
Milorganite	56	38	31
Milorganite	112	31	
Milorganite	224	23	
Lakeland NS	56	108	90
Lakeland NS	112	106	
Lakeland NS	224	55	

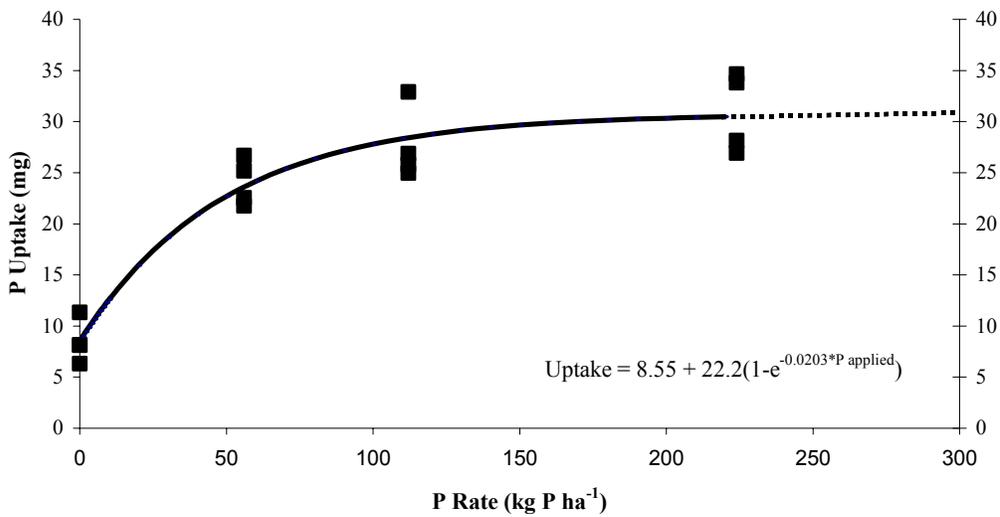


Figure 3-11. Plateau model: P uptake of Milorganite as a function of P applied

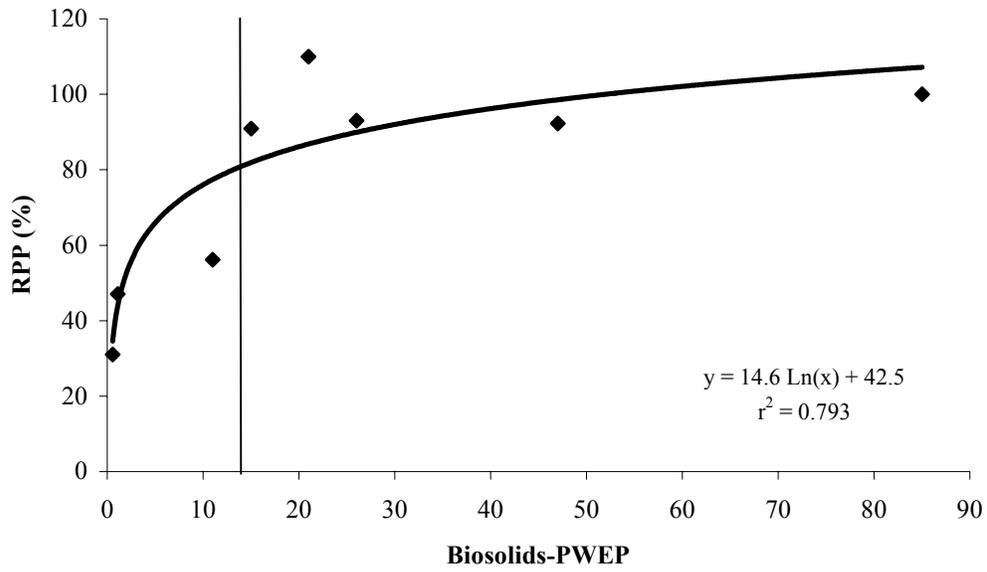


Figure 3-12. Relative phosphorus phytoavailability (RPP) as a function of biosolids-PWEP. Vertical line at 14% PWEP indicates when increased negative environmental impact may occur.

## CHAPTER 4 CONCLUSIONS

### **Dynamic Laboratory Incubation**

The objectives of the dynamic laboratory incubation were to quantify soluble P release, study kinetics of P release, and evaluate the leaching hazard of various biosolids sources. The experiment was designed to mimic an extreme situation of biosolids land application; the soil used had minimal P-sorbing capacity and no plants were grown to utilize supplied P. We accept our second hypothesis that P leaching would be significantly greater from BPR and BPR-like products than conventionally treated materials. P release from BPR and BPR-like biosolids was equal to TSP, thus we reject our third hypothesis that P lability from all biosolids would be less than TSP. This experiment is key to understanding the environmental hazard specific residuals may pose.

Biosolids-PWEP is an excellent indication of how a biosolids will impact the environment when land applied to sandy, low P-sorbing soils. Biosolids with high PWEP values, including BPR and BPR-like materials yielded the greatest cumulative P leached. Biosolids with PWEP values  $\geq 14\%$  should be assumed to have a larger potential negative environmental impact than biosolids with PWEP values  $<14\%$ . PSI can also be used to gauge the environmental impact of biosolids land applied to sandy soils with minimal P-sorbing capacity. Given the observed trends in PWEP and PSI values based on biosolids treatment process, (i.e. PWEP and PSI increase for BPR and BPR-like biosolids) environmental hazard can be roughly gauged by a biosolids treatment process. While exceptions exist, much research has shown that BPR and BPR-like materials have a greater risk of P loss compared to conventionally produced and pelletized biosolids. In this study, the differences in cumulative P mass leached between the dried and BPR or BPR-like materials appear to reflect both physical and chemical controls on P

solubility. The pellets of thermally dried materials did not completely dissolve over the course of the experiment, leading to smaller quantities of P released. Relatively high concentrations of Fe and Al in the Milorganite material also apparently decreased P release.

Thermally dried and conventionally produced residuals have a slower rate of P release than BPR and BPR-like products. Knowledge of the kinetics of P release is important to understanding the effects a residual will have on the environment. A slower rate of P release is desirable, because opportunity for plant uptake is increased and there is less P in the soil solution at any given moment. The less P in the soil solution at any given time means less P is available for leaching through and out of the soil profile, risking impairment of water bodies.

The dynamic laboratory incubation demonstrates that biosolids land application should not be regulated by assuming all biosolids have equal amounts of labile P. Measurements such as PWEF and PSI, should be considered in regulating biosolids land application. Assuming all biosolids to have equal amounts of labile P, and requiring P-based application rates without considering a residual's individual environmental hazard would unfairly burden municipalities facing disposal problems or unfairly advantage WWTP producing BPR products. Blanket regulation of biosolids land application is also unwise given the benefits biosolids can have to soil and crops when land applied. Biosolids land application can decrease chemical fertilizer inputs. Chemical fertilizers designed to be water-soluble and provide instant plant nutrition are a greater environmental hazard than conventionally produced or thermally dried residuals. While BPR and BPR-like biosolids treatment processes are environmentally beneficial to reduce P in wastewater effluent, these materials likely pose a greater environmental hazard due to more P and greater P lability when land applied.

## Glasshouse Study

If biosolids are to be applied to agricultural land under P-based restrictions, the quantity of P that will be available to the crop becomes critical. If a residual has low phytoavailability, and is applied at a P-based rate, the crop will be N and P deficient, requiring additional mineral fertilizer input. For BPR or BRP-like materials, P-based application to crops would provide sufficient P, but require supplemental N fertilizer to meet crop needs. Three BPR or BPR-like materials, and 1 conventionally produced biosolids (OCUD S) examined herein, fit into the high category (>75% of TSP) proposed by O'Connor et al. (2004). One BPR (Disney) and 2 thermally dried materials (Milorganite and GreenEdge) were in the moderate RPP category (25-75% of TSP). Milorganite fits into the moderate category proposed by O'Connor et al. (2004), despite expectation that it would be a low RPP material. Based on determined RPP values for BPR and BPR-like biosolids, we accept our first hypothesis that RPP would be greater from BPR and BPR-like biosolids than conventional biosolids [with the exception of the conventionally digested OCUD S biosolids (RPP = 110% of TSP)]. Materials in the high category for RPP also had the greater cumulative P leached in the laboratory incubation. Materials with high water-soluble P have more P available for plant uptake. Materials with high RPP make excellent fertilizers, but can also pose a greater environmental risk for P loss.

The 1995 U.S. EPA design manual (USEPA, 1995) suggests the average “relative effectiveness” for biosolids-P to be 50% of mineral fertilizer. While the relative effectiveness factor admits not all P in biosolids is phytoavailable, this study, as well as a similar study by O'Connor et al. (2004), shows the wide range in the relative phytoavailability of biosolids. Again, P phytoavailability and leaching hazard are linked to biosolids treatment processes. The Milorganite biosolids, which is relatively high in Fe and Al, showed the lowest relative phytoavailability (31%). The moderate RPP of the Disney compost is likely due to fact that the

material is a mixture of composted yard waste and biosolids, lowering the quantity of soluble/available P. Materials with the greatest PWEF values showed the greatest relative phytoavailability. If and when biosolids P-based regulation of biosolids land application is imposed, it would be wise to consider individual residual's P characteristics (PWEF and PSI). Given the range of biosolids P characteristics, regulating all biosolids as if residuals were all the same could unnecessarily limit beneficial biosolids land application. Most states use a P-index approach to predicting P loss from a material, and most states do not differentiate biosolids by treatment process or characteristics such as PWEF. Currently, Florida uses the same source coefficient for all biosolids (0.015). Elliott et al. (2006) suggested calculating PSC values by multiplying by the WEP ( $PSC = 0.102 \times WEP^{0.99}$ ), based on runoff P studies.

Research not detailed in this thesis used 4 biosolids (Milorganite, OCUD S, Lakeland NS, and Disney) and TSP in rainfall simulations and measured TP, TDP, and biologically (algae) available P (BAP) in runoff and leachate from Immokalee soil. Flow-weighted TP, TDP, and BAP were all highly correlated to biosolids-PWEF values. With the exception of Lakeland NS, BAP losses from all biosolids and TSP was predominantly via leaching. The low solids content (3%), of the Lakeland NS biosolids, however, resulted in extensive soil surface coverage with a layer of fine material that was particularly susceptible to rain drop impact and runoff loss. Cumulative BAP in runoff and leachate for all biosolids treatments was significantly less than from TSP treatments. Thus, in the short term, biosolids-P (even with very high PWEF values) is much less of an environmental threat than fertilizer-P. The results of the rainfall simulation confirm that leaching is the predominant P loss mechanism in typical Florida sands.

### **Land-Applying BPR or BPR-Like Residuals**

Municipalities using BPR biosolids treatment processes have several options to reduce the impact the residuals will have on the environment; however, each option has advantages and disadvantages. If supported by the P-Index, biosolids can be land applied at P-based rates, which will reduce the labile P in the soil solution at any given time, decreasing the chance of negative environmental impact. P-based application rates of BPR and BPR-like biosolids will provide sufficient crop P, but will require additional mineral fertilizer input of N. While applying residuals at a P-based rate is environmentally sound, more land area is required for disposal and disposal costs will increase. Farmers will also face increased costs from purchasing additional N fertilizer, which may discourage them from utilizing biosolids as fertilizer. Soil incorporation can also reduce P loss, especially in soils with sufficient P sorbing capacity.

Biosolids can also be co-applied with WTRs. O'Connor and Elliott (2000) suggested that co-applying biosolids with water treatment residuals (WTR) increases soil P retention capacity and reduce P mobility. Agyin-Birikorang et al. (2007) demonstrated that P sorbed to WTRs is retained long-term.

The dynamic laboratory incubation experiment demonstrated the benefits of high Fe and Al concentrations in limiting P loss, and WWTPs could add Fe and Al salts to BPR and BPR-like products to reduce environmental risk, however this would significantly increase biosolids mass, leading to transportation and disposal problems.

The simplest approach is to apply biosolids to soils with sufficient P-sorption capacity (easily determined). Elliott et al. (2002) demonstrated that even soils with moderate P-sorbing capacity could prevent significant P loss. Applying biosolids to land with sufficient P-sorbing capacity may require transporting biosolids longer distances, increasing transportation costs.

Lastly, thermally drying biosolids can reduce labile P and significantly reduce biosolids mass, decreasing the negative environmental impact. The trend for thermal drying to reduce P lability was not noted when the OCUD E cake material was dried, however the OCUD E cake material is un-digested, and therefore may not be land applied, which may produce unique P characteristics.

GLASSHOUSE ANOVA AND CUMULATIVE DATA TABLES

Table A-1. Cumulative Bahiagrass P uptake after 4 harvests.

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	27	30.6	1.13	52.5	<.0001
Error	72	1.56	0.022		
Corrected Total	99	32.2			
R-Square	Coeff	Var	Root MSE	loguptake Mean	
0.952	3.75	0.147		3.92	
Source	DF	Type I SS	Mean Square	F Value	Pr > F
Treatment (source)	7	24.3	3.04	141	<.0001
Block	3	0.024	0.008	0.37	0.772
P-Rate	2	4.81	2.40	111	<.0001
Treatment*Rate	14	1.45	0.104	4.81	<.0001
Source	DF	Type III SS	Mean Square	F Value	Pr > F
Treatment (source)	7	10.8	1.55	71.3	<.0001
Block	3	0.024	0.008	0.37	0.772
P- Rate	2	4.81	2.40	111	<.0001
Treatment*Rate	14	1.45	0.104	4.81	<.0001

Table A-2. ANOVA for cumulative Bahiagrass yield after 4 harvests.

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	27	3887782	143991	9.77	<.0001
Error	72	1061590	14744		
Corrected Total	99	4949373			
R-Square	Coeff Var	Root MSE	sqyield Mean		
0.786	15.8	121	771		
Source	DF	Type I SS	Mean Square	F Value	Pr > F
Treatment (source)	7	2658619	3323274	22.5	<.0001
Block	3	49493	16497	1.12	0.3472
P-Rate	2	1 246	62078	4.21	0.0187
Treatment*Rate	14	1055512	75393	5.11	<.0001
Source	DF	Type III SS	Mean Square	F Value	Pr > F
Treatment (source)	7	1554892	222127	15.1	<.0001
Block	3	494934	16497	1.12	0.3472
P-Rate	2	124156	62078	4.21	0.0187
Treatment*Rate	14	1055512	75393	5.11	<.0001

Table A-3. ANOVA for cumulative P leached in glasshouse experiment.

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	27	87.8	3.25	45.5	<.0001
Error	72	5.14	0.071		
Corrected Total	99	92.9			
R-Square	Coeff Var	Root MSE	lgleached Mean		
0.945	49.2	0.267	0.543		
Source	DF	Type I SS	Mean Square	F Value	Pr > F
Treatment	7	22.6	2.82	39.5	<.0001
Block	3	0.600	0.199	2.80	0.046
Rate	2	36.9	18.5	258	<.0001
Treatment*Rate	14	27.7	1.98	27.7	<.0001
Source	DF	Type III SS	Mean Square	F Value	Pr > F
Treatment	7	21.5	3.08	43.0	<.0001
Block	3	0.599	0.199	2.80	0.05
Rate	2	36.9	18.46	258	<.0001
Treatment*Rate	14	27.7	1.98	27.7	<.0001

Table A-4. Glasshouse study: cumulative Bahiagrass yields after 4 harvests. Means  $\pm$  1 standard error, n = 4.

Material	Rate kg P ha <sup>-1</sup>	Cumulative Yield g
Control	0	15.9 $\pm$ 1.3
TSP	56	26.6 $\pm$ 1.0
TSP	112	26.2 $\pm$ 0.49
TSP	224	25.1 $\pm$ 0.84
Milorganite	56	26.6 $\pm$ 1.1
Milorganite	112	28.4 $\pm$ 0.70
Milorganite	224	26.3 $\pm$ 2.0
GreenEdge	56	28.6 $\pm$ 1.0
GreenEdge	112	26.7 $\pm$ 0.23
GreenEdge	224	28.5 $\pm$ 0.63
Disney	56	25.7 $\pm$ 0.85
Disney	112	26.4 $\pm$ 0.88
Disney	224	23.4 $\pm$ 0.62
GRU	56	27.6 $\pm$ 0.55
GRU	112	29.9 $\pm$ 1.0
GRU	224	30.8 $\pm$ 1.0
Boca Raton	56	28.1 $\pm$ 0.79
Boca Raton	112	29.6 $\pm$ 0.21
Boca Raton	224	30.1 $\pm$ 0.48
Lakeland NS	56	29.5 $\pm$ 1.3
Lakeland NS	112	30.6 $\pm$ 0.63
Lakeland NS	224	16.8 $\pm$ 4.0
OCUD S	56	30.9 $\pm$ 1.2
OCUD S	112	33.1 $\pm$ 1.1
OCUD S	224	33.5 $\pm$ 1.6

Table A-5. Glasshouse study: cumulative P uptake for Bahiagrass after 4 harvests. Means  $\pm$  1 standard error, n = 4.

Material	Rate	Cumulative P Uptake
		mg
Control	0	8.48 $\pm$ 1.0
TSP	56	49.4 $\pm$ 1.1
TSP	112	71.0 $\pm$ 3.6
TSP	224	105 $\pm$ 6.6
Milorganite	56	24.0 $\pm$ 1.1
Milorganite	112	27.6 $\pm$ 1.8
Milorganite	224	30.9 $\pm$ 2.0
GreenEdge	56	30.5 $\pm$ 1.5
GreenEdge	112	37.1 $\pm$ 0.72
GreenEdge	224	54.0 $\pm$ 88
Disney	56	36.8 $\pm$ 2.3
Disney	112	50.1 $\pm$ 1.2
Disney	224	58.5 $\pm$ 1.7
GRU	56	45.4 $\pm$ 2.0
GRU	112	67.1 $\pm$ 2.5
GRU	224	99.0 $\pm$ 4.7
Boca Raton	56	44.6 $\pm$ 2.9
Boca Raton	112	69.8 $\pm$ 3.4
Boca Raton	224	94.9 $\pm$ 7.0
Lakeland NS	56	53.2 $\pm$ 1.7
Lakeland NS	112	75.1 $\pm$ 6.4
Lakeland NS	224	58.3 $\pm$ 14
OCUD S	56	52.8 $\pm$ 1.3
OCUD S	112	79.5 $\pm$ 1.6
OCUD S	224	115 $\pm$ 6.0

Table A-6. Glasshouse experiment: cumulative P leached after 4 leachings. Means  $\pm$  1 standard error, n = 4.

Material	Rate kg P ha <sup>-1</sup>	Cumulative	Cumulative
		P Leached mg	P Leached % of P Applied
Control	0	0.044 $\pm$ 0.006	NA
TSP	56	0.000 $\pm$ 0.005	0.042 $\pm$ 0.005
TSP	112	0.895 $\pm$ 0.373	0.46 $\pm$ 0.181
TSP	224	33.6 $\pm$ 4.88	8.17 $\pm$ 1.18
Milorganite	56	0.011 $\pm$ 0.11	0.053 $\pm$ 0.011
Milorganite	112	0.008 $\pm$ 0.006	0.025 $\pm$ 0.003
Milorganite	224	0.008 $\pm$ 0.008	0.012 $\pm$ 0.002
GreenEdge	56	0.005 $\pm$ 0.006	0.046 $\pm$ 0.005
GreenEdge	112	0.028 $\pm$ 0.011	0.034 $\pm$ 0.005
GreenEdge	224	0.042 $\pm$ 0.009	0.021 $\pm$ 0.002
Disney	56	0.002 $\pm$ 0.006	0.004 $\pm$ 0.002
Disney	112	0.002 $\pm$ 0.002	0.001 $\pm$ 0.000
Disney	224	0.008 $\pm$ 0.015	0.001 $\pm$ 0.002
GRU	56	0.028 $\pm$ 0.009	0.069 $\pm$ 0.009
GRU	112	0.423 $\pm$ 0.106	0.226 $\pm$ 0.051
GRU	224	13.6 $\pm$ 3.73	3.30 $\pm$ 0.906
Boca Raton	56	0.009 $\pm$ 0.004	0.051 $\pm$ 0.004
Boca Raton	112	0.261 $\pm$ 0.199	0.147 $\pm$ 0.096
Boca Raton	224	5.10 $\pm$ 1.24	1.25 $\pm$ 0.301
Lakeland NS	56	0.030 $\pm$ 0.005	0.070 $\pm$ 0.005
Lakeland NS	112	0.480 $\pm$ 0.118	0.254 $\pm$ 0.057
Lakeland NS	224	6.33 $\pm$ 2.13	1.55 $\pm$ 0.518
OCUD S	56	0.035 $\pm$ 0.012	0.076 $\pm$ 0.011
OCUD S	112	0.554 $\pm$ 0.246	0.290 $\pm$ 0.119
OCUD S	224	5.20 $\pm$ 0.538	1.27 $\pm$ 0.131

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## BIOGRAPHICAL SKETCH

Sarah Chinault was born in Lakeland, Florida, on November 24, 1981 to Edward and Linda Chinault. Sarah has an older brother (Chris, 28) and a younger sister (Amanda, 23). Sarah obtained her B.S. degree from the University of Florida in 2004, majoring in environmental science. Sarah's career goals include working for an environmental advocacy or animal rights group. Always an animal lover, Sarah has 5 small dogs that she considers children: Mocha (10), Mini-Me (7), Maynard (4), Hank (11), and Bauer (1). Mitch (11) recently passed from cancer and will be missed.