

ALUMINUM WATER TREATMENT RESIDUAL EFFECTS ON SOIL PHOSPHORUS  
RETENTION AND FORAGE YIELD AND NUTRITIVE VALUE

By

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To Beau Roberts, for being my mentor and challenging me in every facet of life;  
and to Uncle Paul, for giving me a love and respect for agriculture.

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Abstract of Thesis Presented to the Graduate School  
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The health of aquatic systems is being threatened by the addition of phosphorus (P) which causes increased eutrophication. Many Florida soils have naturally low capacities to retain P, allowing for P loss to local waterways by leaching. In addition, P is often over-applied when animal manures are used as fertilizers, which further increases the chances of P loss. Water treatment residuals (WTRs) are by-products of the water purification process which contain high amounts of amorphous aluminum (Al) or iron, giving them the capacity to retain P. Land application of WTRs has been investigated mainly in greenhouse experiments to test the P sorbing capacities of WTRs and to evaluate potential Al toxicity to plants.

The purpose of this project was to evaluate the effects of an Al-WTR on soil P retention, soil microbial population, groundwater soluble reactive P (SRP) concentrations and forage yield and nutritive value. Three WTR application rates (0, 1.5 and 3% dry weight by mass) and two application methods (soil incorporation and surface applied) were tested on a bahiagrass (*Paspalum notatum*) pasture in Ona, FL. Soils on the site had low P levels (A horizon = 2.1 mg Mehlich-1 P kg<sup>-1</sup>) so triple superphosphate was added at 224 kg ha<sup>-1</sup> to simulate high P loadings.

The addition of WTR showed a trend toward slightly higher pH. Water extractable P concentrations decreased as WTR application rate increased, suggesting less P will be lost to

leaching. Mehlich-1 Al increased from 22 mg kg<sup>-1</sup> to 390 mg kg<sup>-1</sup> (3% WTR rate) in the A horizon, giving the soil a greater P retention capacity which was observed in the increasing soil P storage capacity (SPSC) values which occurred with increasing WTR rate as well. The SPSC values increased on plots with WTR even after heavy loads of P were applied and some remained high enough that additional P should be retained. Potential nitrification rates increased with higher WTR application rates, indicating no adverse effects occurred on soil microbial populations because of WTR application. Groundwater SRP concentrations decreased with the addition of WTR by 78% in the surface applied treatments. There were no differences in SRP concentrations among the incorporated treatments which was likely a temporary result of the tilling process. Forage yield, crude protein and neutral detergent fiber were not affected by WTR application. Tissue Al did not increase and tissue calcium and magnesium uptake was not restricted. Tissue P decreased as WTR application rate increased. Tissue P levels were higher in the incorporated treatments than the surface applied treatments. However, tissue levels did not fall below the bahiagrass limiting value (0.15%).

Based on this study, the use of WTRs is recommended as an amendment to soils with low P retention capacities that have been heavily impacted with P or will receive high amounts of P e.g., a dairy sprayfield.

## CHAPTER 1 INTRODUCTION

Nutrient enrichment was the leading cause of water degradation in the 1990s, polluting approximately 45% of waters in the United States (USEPA, 2000). Nitrogen (N) and phosphorus (P) are the main nutrients of concern; while they are naturally present in the environment, they are also added as fertilizers in agriculture and home lawns and can enter aquatic systems through runoff or leaching. Eutrophication occurs when increased levels of nutrients in aquatic systems cause an influx of aquatic organisms and ultimately a decline in dissolved oxygen (Brady and Weil, 2002). Under some eutrophic conditions, fishery, recreation and industrial water use is put under restrictions and surface waters may no longer be used for drinking water (Sharpley et al., 2001). Although N is necessary for the algae that cause the damages associated with eutrophication, environmental concerns have been focused on P since it has been shown as the limiting nutrient that most often causes accelerated impairment in aquatic systems (Sharpley et al., 1994).

Florida has a large number of important water resources which contribute to many facets of life: groundwater supplies 90% of the drinking water in the state, sport fishing and bird watching bring millions annually to the economy and aesthetic beauty can be appreciated by all. In Florida's unique ecosystem, P from urban and agricultural activities has impacted many aquatic systems. The transfer of P downstream to other systems has created far reaching impacts (Reddy et al., 1999). For example, drainage water enriched with P from the Everglades Agricultural Area drains into the Water Conservation Areas and has caused eutrophic conditions. The movement of P into freshwater bodies may lead not only to eutrophication but also generally unhealthy biological conditions.

The main sources of P in Florida are fertilizers, animal manures, municipal biosolids and effluents and composts. Application of manures and biosolids often occur at N agronomic rates resulting in P levels which are higher than necessary (McCoy et al., 1986). Long histories of high P applications have caused an accumulation of P to the point where many crops do not benefit from added P. Although there is often little or no crop response from P fertilizers, many farmers in Florida still apply higher amounts than necessary. In 1996, over 50% of P fertilizers purchased in Florida were used in the South Florida Water Management District which is especially susceptible to P loading in aquatic systems (Reddy et al., 1999).

Florida has more than 14 million hectares of acidic soils most of which are sandy (He et al., 2005). These sandy soils are less capable of retaining P because unlike finer textured soils they do not contain large quantities of mineral components such as metal oxides and secondary phyllosilicates which aid in P retention (Sample et al., 1980). A study conducted on 96 Florida sandy soils showed more than 10% of the soil P was water soluble suggesting high leaching potential (Zhang et al., 2002). Harris et al. (1996) contend the main reasons for low P retention in Alaquods are (i) very low amounts of aluminosilicate and metaloxide clays in the E horizon and (ii) a seasonal shallow water table that results in the transport of P laterally into connected drainage ways.

In 2007 Florida had 18,900 cattle farms and 440 dairy farms (USDA, 2008). Livestock operations across the nation are intensifying their practices and creating Concentrated Animal Feeding Operations (CAFOs). With this intensification comes more manure production per acre of land. Before USEPA regulations, CAFOs applied manure at N agronomic rates resulting in excessive P levels and possible P loss scenarios (USDA-USEPA, 1999). The risk of P loss can be exacerbated with the poor sorbing soils found in Florida.

A number of best management practices (BMPs) have been designed to help control P loss from agricultural soils. Fertilizer application rates, animal density, irrigation management, runoff and wetland retention systems are being managed to help reduce eutrophication of aquatic systems (Bottcher and Rhue, 1984). While these measures are helping to reduce P contamination in Florida's aquatic systems, increasing populations and intensively managed agricultural operations will place pressure to continually reduce the amount of P entering aquatic systems.

Controlling erosion, lowering levels of fertilizers in agricultural production and implementing BMPs are approaches used to help prevent eutrophication (Troeh et al., 2004). Several physical means of reducing surface runoff to minimize P losses are currently in practice: conservation tillage, buffer strips, runoff water impoundments and riparian zones (Novak and Watts, 2004). Recently, a variety of chemical soil amendments have been suggested as means of increasing P retention by the soil and thereby reducing P losses.

Aluminum (Al) and iron (Fe) salts ( $\text{Al}_2(\text{SO}_4)_3$ ,  $\text{FeCl}_3$ ) have been shown to lower the amounts of labile P in the soil but often have high costs which detract from their potential widespread use (Moore and Miller, 1994). Water treatment residuals (WTRs) are by-products of the drinking water purification process that contain amorphous Fe or Al oxides or calcium carbonate as well as sediment and humic substances from the treated water (Makris et al., 2004). The Fe and Al compounds in WTRs contain reactive hydrous oxides with substantial P-fixing capabilities (Elliott et al., 2002). The current methods of WTR disposal are expensive for water treatment municipalities and include storage in landfills and onsite lagoons and discharge into sanitary sewer systems (Dayton and Basta, 2001). Thus, other methods of disposal and utilization such as land application are being evaluated. Ippolito et al. (2003) list some of the possible benefits of WTR land application: increased N and organic carbon availability for

plants, improved aggregate stability and water retention, better aeration and drainage capacity and increased cation exchange capacity for coarse textured soils.

Numerous studies have looked at the characteristics and capabilities of WTRs as a means of controlling P loss from leaching. Most of these studies have taken place in a laboratory or a greenhouse under controlled conditions. These methods are a good way to make initial evaluations of the tested material. There is concern in the agricultural sector of using an amendment containing Al and the effects it may have on crop growth and production. There is a strong need to determine the effects WTR will have on soil P movement, plant P uptake and possible Al toxicity problems under field conditions. This study will address these concerns under field conditions.

The objectives of this study are to:

1. Evaluate the effects of Al-WTR application method and rate on P forms in the soil
2. Determine the effects of Al-WTR application on the soil microbial population
3. Examine P leaching through groundwater monitoring
4. Evaluate forage yield and nutritive value as affected by Al-WTR application method and rate

## CHAPTER TWO LITERATURE REVIEW

### Phosphorus

Phosphorus (P) is a macronutrient required by plants to provide and produce energy and is the second most limiting nutrient. In most plants it comprises up to 0.2% of dry weight (Schachtman et al., 1998), while legumes can have higher concentrations in the range of 0.4-0.6% (Lush, 1934). It is essential to many cell components such as nucleic acids (DNA and RNA), phospholipids and adenosine triphosphate (ATP) (Schachtman et al., 1998). ATP is required for most processes within the plant that need energy including the uptake and transport of nutrients. Phosphorus improves various plant physiological aspects such as nitrogen (N) fixation, photosynthesis and root development. Phosphorus deficiency can be observed by stunted, thin-stemmed and spindly plants. Some plants develop a purple color while extremely deficient P levels may result in the yellowing of leaves. Deficiencies also cause delayed maturity, sparse flowering and poor seed quality (Brady and Weil, 2002).

The movement of P in the environment is strongly affected by the form of P. Inorganic P is normally found bound with aluminum (Al), iron (Fe), calcium (Ca) and magnesium (Mg) (Graetz and Nair, 1999). Inorganic P is the most common form taken up by plants and is rarely higher than 10  $\mu\text{M}$  in soil solution. The form of inorganic P changes according to pH. The dissociation pKs of  $\text{H}_3\text{PO}_4$  are 2.1 and 7.2 for  $\text{H}_2\text{PO}_4^-$  and  $\text{HPO}_4^{2-}$ , respectively. Most P uptake occurs in a pH range of 5.0-6.0 making  $\text{H}_2\text{PO}_4^-$  the most readily available form of inorganic P to plants (Schachtman et al., 1998).

The Langmuir Equation is a mathematical model used to predict anion adsorption onto reactive surfaces. This equation shows that the availability of phosphate is often controlled by the amount of saturation at the sorption sites. As the sorption sites become saturated with anions

the concentration of the anion in solution increases. Desorption of phosphate creates a condition of hysteresis in which desorption is much slower than adsorption to such an extent that it does not affect soil processes. Hysteresis has important environmental implications because it allows the soil to hold onto phosphate it has sorbed even once its concentration decreases. This ultimately results in less leaching; however, it is only effective when soils have the ability to sorb phosphate or when the soil has not been oversaturated (McBride, 1994).

Soils can become loaded with high concentrations of P through agricultural management practices. Manure from local livestock productions is often applied as a fertilizer based on plant N requirements which results in P application rates that exceed plant needs (Sharpley et al., 1994). Excessive fertilizer usage and biosolids application can also load soils with excessive P (O'Connor et al., 2002). Graetz and Nair (1999) reported that up to 80% of total P has the potential to be transported in heavily manure-impacted upland soils. Repeated additions of P over time reduce the soil's capacity to retain P (Reddy et al., 1999).

Soils in the eastern Coastal Plain are characteristically highly weathered and coarse textured soils which have a lowered affinity to sorb P. When these soils with low P retention capacity are coupled with high loads of P, the ability to bind P is significantly lowered (Novak and Watts, 2004). Florida has many low P sorbing soils and hydraulic conditions that allow for P to easily leach into bodies of water causing environmental problems (O'Connor et al., 2002).

Soil test P (STP) and degree of P saturation (DPS) are indicators often used to assess environmental risks associated with P application. Neither indicator however accounts for the soil's capacity to retain P. Although STP and DPS values may be low, applying high amounts of additional P could result in excess P not bound by the soil and induce risks to local water systems. Nair and Harris (2004) proposed using a calculation to determine the soil P storage

capacity (SPSC) which would account for both previous P loadings onto a soil and the capacity of the soil to retain additional P. They found that some Spodosols in Florida had low STP and DPS values but also low sorption capacities so loading these soils with P based on these two factors could pose environmental risks. The SPSC value would show potential risk of P application for soils with low sorption capacities.

### **Phosphorus Retention by WTR**

The ability of WTRs to sorb P has been tested to determine if it can be used as an agent to prevent P runoff. Gallimore et al. (1999) applied WTR to plots fertilized with two poultry litters at a rate of 6.72 Mg P ha<sup>-1</sup>. The WTR was applied at 44.8 and 11.2 Mg ha<sup>-1</sup> on broadcast plots and 44.8 Mg ha<sup>-1</sup> on a buffer strip. After simulated rain events, surface runoff was collected, analyzed and compared to control plots. Dissolved P was reduced 42.7% in the broadcast plots at the 44.8 Mg ha<sup>-1</sup> WTR rate and 45.9% in the buffer strips when compared to the control. Land application of WTR did not increase soluble Al or sediment in the surface runoff.

Novak and Watts (2004) evaluated the use of WTRs for P retention on sandy coastal plains that have a high susceptibility to P leaching. Soils obtained from North and South Carolina were combined with several WTRs in blends of 0, 2.5, 5.0, 7.5 and 10.0% dry weight by mass. A calcium chloride solution with P concentrations ranging from 5,000 to 30,000 mg P L<sup>-1</sup> was added to the soil and WTR mixtures. The WTRs proved to sorb 100% of P when the solution contained between 5,000 and 10,000 mg P L<sup>-1</sup>. The P<sub>max</sub> value determined for the soils alone was less than 1 mg P g<sup>-1</sup> while the P<sub>max</sub> when WTR was incorporated with the soil ranged from 1.7 to 8.5 mg P g<sup>-1</sup> depending on the composition of the WTR used. From this study it was concluded that WTR can increase a soil's P sorption values and prospectively help P from moving to bodies of water.

Alum ( $\text{Al}_2(\text{SO}_4)_3$ ) has been used to reduce P in a variety of environmental conditions. It has been applied in wetlands when sediments have been known to release P (Ann et al., 1999) and also to soils amended with poultry litter high in P (Shreve et al., 1995). The P retaining capacities of alum were compared to an Al-WTR because of the high cost of alum. The Al-WTR was nearly as effective as alum at reducing the amount of P released when combined with a poultry litter. The advantages of the WTR include lower costs and less dissolved Al released than alum (Makris et al., 2005).

Other amendments have also been studied for their ability to reduce P movement. Haustein et al (2000) compared the efficiency of a WTR to a by-product of the commercial alum production process, HiClay Alumina (HCA). An Al-WTR and HCA were applied at rates of 0, 2.2, 9.0, and 18  $\text{Mg ha}^{-1}$  to plots with high to excessive soil test P levels (150 to 300  $\text{mg P kg}^{-1}$  soil). Rainfall was simulated to produce runoff one day, one month, and four months after application. The WTR was able to sorb P at a rate 20 times higher than HCA most likely because WTR contained three times as much Al and more clay particles. This study also looked at Al concentrations in the runoff. The rain event after one day showed greater amounts of total Al for the two highest WTR application rates than the control since the residue contained 47  $\text{g Al kg}^{-1}$ . Runoff Al concentrations after one and four months did not differ from the controls. Thus, controlling when WTR is applied may prevent greater levels of Al in runoff.

Elliott et al. (2002) examined the ability of Al-WTR, Fe-WTR and Ca-WTR to prevent P leaching in a sandy soil amended with biosolids and triple superphosphate (TSP). Soil columns were set up with bahiagrass grown on an A horizon of Immokalee sand and an E horizon of a Myakka series. WTR was applied at 56  $\text{Mg ha}^{-1}$ . The controls had 21% of total P leach from TSP and 11% of total P leach from the Largo cake biosolid over four months. When WTR was

applied, the amount of leached P reduced to 3.5%, 2.5% and less than 1% of total applied P for Fe-WTR, Ca-WTR and Al-WTR respectively, making the Al-WTR the most effective in decreasing soluble P of the materials tested.

Miyittah-Kporgbe (2004) investigated the effects of application rate and the level of mixing an Al-WTR with the soil on P leaching. Leaching rates determined through a column study showed a reduction by 87 to 99.7% when Al-WTR was mixed completely with 15 cm of soil; partial mixing with 7.5 cm of soil only resulted in 40 to 58% less leaching than control columns with no applied WTR. More P retention was accredited to more contact with the amendment. Runoff from rainfall simulations showed that when Al-WTR was surface applied, soluble P concentrations were less than the critical threshold of  $0.03 \text{ mg L}^{-1}$ . The leachate concentrations when WTR was surface applied were above the critical threshold because of limited contact with the amendment. These studies showed that higher surface application rates can be as effective as using lower quantities of Al-WTR mixed throughout the soil profile. Therefore, Al-WTR should be applied and distributed according to anticipated P losses. Surface applications will help prevent runoff losses and mixing the amendment will help prevent leaching.

In a study by Silveira et al. (2006) the efficiency of WTR mixed within the entire profile was evaluated by observing soluble reactive P (SRP) concentrations in leachate. WTR was added to a manure-impacted Immokalee soil at rates of 0, 25, 50 and  $100 \text{ g kg}^{-1}$ . The WTR in the soil columns was either left unincorporated or mixed within the top 7.5 or 15 cm. Water extractions through the columns were analyzed for SRP. Control samples had an average SRP concentration of  $38 \text{ mg L}^{-1}$ . In columns with WTR in the top 7.5 cm, SRP decreased to  $19 \text{ mg L}^{-1}$  and when WTR was in the entire 15 cm SRP further declined to  $2 \text{ mg L}^{-1}$ . Thus, results

showed deeper incorporation of WTR increased P immobilization. Rew (2006) showed similar results in a rainfall simulation involving the effects of an Al-WTR on manure impacted soils with a fluctuating high water table. Boxes were filled with manure-impacted soils high in P and either mixed with WTR or had WTR applied at the surface at a rate of 2.5% dry weight by mass. Soluble P in runoff was more effectively reduced on the surface applied treatments while incorporating the WTR was better at reducing soluble P in leachate and subsurface flow. Both of these studies imply that incorporating the WTR allows for more contact with the impacted soil and so better reduces downward movement of P than surface application. It was suggested that to reduce P in runoff and leaching, WTR would need to be surface applied after being incorporated (Rew, 2006).

Ippolito et al. (2003) reviewed the relationship between time and P desorption from a WTR. Water treatment residual samples with a given load of P were shaken in a calcium chloride solution for various lengths of time and analyzed to observe how much P was desorbed from the amendment. After a linear decrease in the amount of P sorbed (approximately 28 days), the concentrations of P being desorbed quickly leveled off. The quick decline in P desorption may represent P that was adsorbed to variable charged surfaces and the leveling off of desorption may be a result of more strongly precipitated calcium phosphates.

The long term effects of P bound by WTR have not been extensively studied. In one study by Agyin-Birikorang et al. (2007) the P bound by an Al-WTR was found to be stable after 7.5 years. A rainfall simulation demonstrated that WTR reduced dissolved and bio-available P by more than 50% compared to controls. Therefore, it was shown that after 7.5 years the P associated with WTR should not contaminate local waterways by runoff or leaching.

### WTR-Induced P Deficiency in Plants

Phosphorus deficiency in plants is a concern when applying an Al based WTR. Because of its chemical makeup and high concentration of Al, WTRs may sorb the P available to and needed for plants. Codling et al. (2002) applied WTR to three different soils at rates of 0, 10, 25 and 50 g kg<sup>-1</sup>. After seven weeks, three cycles of wheat (*Triticum aestivum* L.) were grown. At the 10 g kg<sup>-1</sup> WTR application rate, biomass yield was not influenced for all the soils. Two of the three soils showed lower biomass yields at the 25 and 50 g kg<sup>-1</sup> WTR application rates. These two soils had a greater sand content than the unaffected soil. Tissue P concentration fell to deficient levels (<1.5 g kg<sup>-1</sup>) at the 25 and 50 g kg<sup>-1</sup> application rate in the second and third cropping cycles although no deficiency symptoms were visual. Results from the study suggest WTRs can be applied at rates up to 10 g kg<sup>-1</sup> without reducing yield production. Heil and Barbarick (1989) reported increased sorghum-sudangrass (*Sorghum bicolor*) growth above the control when WTR was applied at 5 and 10 g kg<sup>-1</sup>. When application rates increased to 15 and 25 g kg<sup>-1</sup>, there was a 1.4 to 4.3 fold decline in plant yields.

Ippolito et al. (2002) studied the effects of combining WTR with biosolids on two range grasses. Before testing it was assumed P availability would be reduced because of the chemical composition of WTR and thus P was supplemented through the application of biosolids. However, even when the two amendments were co-applied, P availability was reduced for blue grama (*Bouteloua gracilis*) and western wheatgrass (*Agropyron smithii*) observed by the lowering of tissue P. When WTR was applied alone, results showed increased Al uptake but no effects on dry matter yield or P uptake.

Cox et al. (1997) performed a greenhouse study to determine the effects of alum sludge application on P availability to wheat (*Triticum aestivum* L.) in a fine-loamy, Ultisol. Alum sludge is a byproduct of the drinking water purification process using Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> \* 14 H<sub>2</sub>O and

which has low solids content (1-2%). Surface applied alum sludge had little effect on P availability while incorporating the sludge into the entire soil volume did show a decrease in P availability in the significantly decreased dry matter yields and plant P uptake. When the sludge was incorporated, Al compounds retained soil P that was otherwise loosely bound soil P and available to the wheat. It was suggested that higher P fertilizer requirements will be needed on soils amended with alum sludge.

An Fe-WTR was amended with P to determine if adding P could prevent P deficiencies in plants that may be caused by the WTR. The Fe-WTR was also amended with a cationic polymer, Magnafloc 572C, before being applied to sorghum-sudangrass. The amount of P needed to make the Fe-WTR a supplier of P was only 14.6 to 19.4 g P kg<sup>-1</sup>. This low P rate was partially a result of P release from the cationic polymer. The cost of adding P to WTRs to make them a supplier of P will vary depending on the amount of Fe or Al, but in this study it was more economical than placing the WTR in a landfill (Hyde and Morris, 2004).

In a rainfall simulation, the effects of an Al-WTR at a 2.5% dry weight by mass application rate were observed for stargrass (*Cynodon* spp). Shoot growth, root biomass, and forage P, N and Al concentrations were not affected by the WTR (Rew, 2006). An Al-WTR was applied in combination with poultry litter manure under field conditions to determine if the WTR amendment would affect the yield and nutrient concentration of corn (*Zea mays* L.). The poultry litter was amended with WTR at rates of 0, 5.6 and 11.2 Mg ha<sup>-1</sup> before being applied to the soil. The two sites used for the study were classified as an Ultisol and an Entisol. Corn grain yield and mineral nutrients (Zn, Mn, Fe, Ca, Mg, and K) in the leaves and grain were not affected by the addition of WTR. Tissue P levels remained above adequate growth levels for corn (2.0 g kg<sup>-1</sup>) and were similar to control plot leaf P concentrations, indicating adequate levels of

available P. Leaf Al concentrations were not affected by WTR and did not reach toxic levels (Codling et al., 2007). A similar study was conducted by Codling et al. (2002) on wheat grown in a greenhouse where P deficiencies were observed when the WTR application rate was 25 and 50 g kg<sup>-1</sup>. The differences in P uptake between greenhouse and field conditions were attributed to the confining volume of growing pots. Under greenhouse potting conditions, the volume of soil to supply P is limited (Codling et al., 2007).

A study by Oladeji et al. (2006) evaluated the effects of an Al-WTR on bahiagrass (*Paspalum notatum*) in a glasshouse study. The WTR was applied at three rates (0, 10, and 25 g kg<sup>-1</sup>) on P deficient soils in combination with various P sources applied at N and P agronomic rates. Bahiagrass tissue P concentrations fell between 1.5 to 6.0 g kg<sup>-1</sup>, right around the critical P value of approximately 2.0 g kg<sup>-1</sup> used in this study. Dry matter yields were not affected by WTR application. From this study it was concluded that in order to avoid agronomic deficiencies, WTR application rates should be based upon the Al, Fe and P composition of the soil, WTR, and P-source. Applying WTRs so soil P storage capacity is at zero allows for P uptake in plants to be minimally impacted and increased environmental benefits such as less P leaching.

### **Aluminum**

Aluminum makes up 7% of the earth's crust by mass and is the most abundant element in the crust (Delhaize and Ryan, 1995). It is found in all plants and some more tolerant species may contain more than 0.1% of Al by dry weight. Tissue samples from grass tops contained 50-3410 µg Al g<sup>-1</sup> by dry weight (Kabata-Pendias and Pendias, 2001). Although the physiological function of Al in plants is not clearly known it may be beneficial to some plants in small quantities in its ability to activate some enzymes and control plasma and membrane permeability.

Aluminum toxicity is one of the major problems and growth limiting agents in acidic soils. Toxicity occurs when the pH falls below 5.5 and becomes increasingly damaging below 5.0 (Foy et al., 1978). The trivalent form of Al ( $\text{Al}^{+3}$ ) dominates solutions when the pH is below 5.0;  $\text{Al}(\text{OH})^{+2}$  and  $\text{Al}(\text{OH})_2^+$  become more prevalent as pH increases. The trivalent form is assumed to be the phytotoxic form of Al because other trivalent cations are also toxic to plants and because toxicity is associated with acidic soil conditions (pH less than 5.0) (Delhaize and Ryan, 1995). Taking into consideration that WTRs are often buffered at 7.0 and agricultural fields are often maintained above pH 5.5 for optimal growing conditions, Codling et al. (2007) contend Al toxicity should not issue problems since  $\text{Al}^{+3}$  solubility decreases as pH approaches 5.2. Soils high in organic carbon content may form Al complexations further reducing toxicity possibilities (Hsu, 1976).

Acidification increases Al and manganese solubility, thereby decreasing root growth (Jarvis, 1987). The process of soil acidification is mainly caused by heavy rains which strip major cations from the soil. It can also result from using acidifying fertilizers (Mora et al., 1999).

Aluminum toxicity causes less P uptake into the plant and also disables its movement within the roots (Edwards, 1991). Damage due to Al toxicity arises from decreased nutrient uptake and transport of other vital nutrients, especially Ca, potassium and Mg, as well as from imbalanced cation to anion ratios (Kabata-Pendias, 2001). Nichol et al. (1993) observed the effects on cation uptake at Al levels of  $100\mu\text{M}$ . Calcium uptake was inhibited by 69%, ammonium by 40% and potassium by 13%. Possible mechanisms of reducing cation uptake include the  $\text{Al}^{+3}$  ions blocking or binding to negative charges in proteins of the plasma membrane. Even in micromolar concentrations, Al can cause stress in plants observable by the

restriction of root growth which later inhibits plant growth (Delhaize and Ryan, 1995). The obstruction of root growth can cause the plant to experience less water and nutrient uptake since roots do not penetrate the soil as extensively (Strid, 1996).

Aluminum toxicity can be minimized by adding various solutes to the solution. Hydroxyl ions or alkaline salts reduce Al activity instead of Al concentrations but prove to be helpful nonetheless. Raising the solution pH also decreases Al toxicity (Parker et al., 1989). While Al toxicity is strongly affected by pH, the amount of extractable Al in soil and soil solution is partially determined by mineralogy and the amount of weathering (Fox et al., 1991). For instance, solution Al in an Ultisol had toxic values around  $0.62 \text{ mg L}^{-1}$  while Al concentrations of an Oxisol were around  $0.05 \text{ mg L}^{-1}$  under the same growing conditions. Mineralogical structure helps to explain these differences in Al solubility. More highly weathered soils normally have lower Al solubility.

Edwards (1991) studied interactions between soil acidity, liming and P additions to spring barley, winter wheat, woilseed rape and grass common to Scotland. Yield reductions were witnessed with increases in acidity. By maintaining P soil levels adequate for crop growth, the effects of Al toxicity were noticeable at pH values below the optimal growing conditions of 5.5.

In a study conducted by Dayton and Basta (2001), WTR was considered as a substitute for the top layer of soil. Many properties were analyzed including cation exchange capacity, nutrient levels, plant available water and potential toxicities. The only possible toxicity problems pertained to levels of nitrite which if greater than  $10 \text{ mg kg}^{-1}$  are considered toxic to plants. Water soluble Al levels fell below the regulatory value of  $1.0 \text{ mg L}^{-1}$  in all of the 14 WTRs tested.

## **Phosphorus and Water Movement in Spodosols**

There are approximately 4.5 million acres of Spodosols in Florida (Li et al., 1997). Spodosols are characteristically nearly level, poorly drained, sandy soils with high seasonal water tables and an organic Bh horizon. Naturally these soils are very infertile but can be highly improved for crops or pasture if managed correctly (Soil Survey Staff, 1996).

Spodosols are formed from intensive acid leaching in moist areas. Acid organic compounds bind Al or Fe and carry them down the soil profile by percolating waters. A subsurface spodic horizon (Bh) forms when Al and Fe precipitate beneath an albic horizon. The organic compounds make this horizon black in color (Brady and Weil, 2002). Spodosols can cause a perched water table to exist. This can result in surface and subsurface runoff losses because P does not come into contact with the Bh horizon where the Al and Fe have the capability of forming precipitates and preventing P loss (Burgora, 1989).

Spodosols found in the Myakka series in Florida have an average water table depth of 76 cm (Allen, 1987). Nair et al. (1999) looked at how P retention was affected by the changing aerobic and anaerobic conditions that occur with a fluctuating water table in Myakka soils. The sandy upper A and E horizons allow P to move into the spodic horizon where it can become trapped due to higher contents of Al and Fe. Anaerobic conditions led to equilibrium P concentrations which were 30% greater than aerobic conditions and a P sorption maximum that was less than 25% of aerobic conditions. These statements suggest P retention decreases when the water table conditions are high (above the spodic horizon). Villapando and Graetz (2001) similarly found that even though most of the P content of the Bh horizon was associated with Al and Fe, a significant percentage was still in a highly mobile form which could be transported to nearby waterways with lateral movement above the spodic. Conditions in which P could move laterally above the Bh horizon depend on the P loading of the spodic: non-manure impacted soils

such as pasture soils have less P and so the possibility of P moving out of the system is less than a highly impacted soil (Nair et al., 1999).

### **Soil Microorganisms**

Microorganisms encompass only 1-3% of soil organic matter (Jenkinson, 1988) yet there can be as many as  $10^9$  bacterial cells per gram of soil (Torsvik et al., 1990). Microorganisms have many vital roles in the soil environment. They decompose organic matter thereby releasing substantial amounts of nutrients. They are major constituents in biogeochemical cycles (carbon, nitrogen, sulphur) and also help in environmental processes such as the degradation of pollutants (Curtis and Sloan, 2005).

Soil microorganisms are considered an excellent biological soil quality indicator because they respond quickly to environmental changes (Turco et al., 1994). They also play a vital role in the cycling of N and C (Nannipieri et al., 1990). Specifically, nitrification is a functional parameter in assessing soil quality because it is carried out by specialized bacteria which are very sensitive to environmental changes, stress and toxic substances (Bollmann, 2006).

Nitrification, the reduction of N to nitrate ( $\text{NO}_3^-$ ), is normally a two-stage process with ammonia being oxidized to nitrite, and then nitrite quickly being oxidized to  $\text{NO}_3^-$  (Prosser, 2007). Nitrification is affected by a number of factors which can interact to affect the overall process. Nitrifiers must be present, and because they are aerobic organisms oxygen must also be available. Nitrification is optimal when soils are near field capacity or when 60% of pore spaces are filled with water. The most important regulating factor is substrate availability, namely ammonium (Myrold, 1999). Soil pH is another influence on nitrification. Most soils below pH 4.5 have negligible nitrification rates because nitrifiers generally work best at neutral pH. It has been shown however that nitrification can occur at pH below 4.0 in forest soils. One possible explanation for nitrification under these low pH conditions is that N rich material in microsites

release ammonium, increasing the pH within the microsite. Another is the presence of more acid tolerant nitrifiers. The best possible explanation is that the lack of Al toxicity in organic and some forest soils allows nitrification at even lower pHs (Paul and Clark, 1996). Temperature, water potential, salinity and nutrient availability also affect nitrification rates (Myrold, 1999).

Aluminum negatively affects soil bacterial growth and overall numbers of rhizobium (Glenn and Dilworth, 1991). The mechanisms which specifically harm the bacteria are unknown. Soybeans and cowpeas have shown decreased levels of N fixing capabilities when grown in combination of low pH and toxic levels of Al (Mookherji and Floyd, 1991). A study on forest soils reported decreased nitrification rates as potassium chloride extractable Al increased (Gilliam et al., 2005).

Alexander (1980) proposed that acid precipitation affects nitrification more than other N cycle phases. The effects of nitrification as impacted by acid precipitation were studied by Bitton et al. (1985). Acid rain of pH 3.7 and 3.0 along with a control of 4.6 was applied to 75 m<sup>2</sup> plots on a typical Florida sandy soil. Nitrification rates proved to be the most sensitive to the acid rain and were reduced from 0.16 mg NO<sup>-3</sup>-N kg<sup>-1</sup> d<sup>-1</sup> on control plots to 0.08 and 0.06 mg NO<sup>-3</sup>-N kg<sup>-1</sup> d<sup>-1</sup> for acid rain with a pH of 3.7 and 3.0, respectively.

There is little information on the impact that WTR application has on soil microorganisms. In one study, six WTRs from South Africa were added to two soils at various rates and soil respiration was monitored. In general, the soils amended with WTR showed significant increases in soil respiration. The effects were less visible after time, indicating increased microbial activity may not be a long-term result. Based on soil microbial indicators alone, WTR could be applied at a 300 Mg ha<sup>-1</sup> rate without negatively impacting soil quality (Pecku et al., 2006).

Sikora (2004) investigated the effects of Al-WTR on plant residue decomposition. The WTR was applied to two Ultisols at rates of 0.5, 2.5 and 10 g kg<sup>-1</sup>. Carbon labeled soybean leaves and stems were added. Lime (CaO) was also added to evaluate the effects of raising the pH. There was no effect on soybean residue decomposition by WTR or CaO addition. Salinity increases were negligible and did not affect the other soil characteristics studied. It was recommended that the WTR application rate should have the same metal molar concentration as soil P molar concentration.

### **Bahiagrass**

Bahiagrass is a warm season perennial common throughout Florida. It can sustain high temperatures and low periods of rain which often limit the growth of other grasses. In Florida, bahiagrass covers approximately 2.5 million acres making it the highest single used grass pasture species (Chambliss and Adjei, 2006). It is used extensively in Florida because of its ability to tolerate a wide range of soil conditions, produce moderate yields even when the soil has low fertility, establish easily from seed, withstand close grazing and is not regularly affected by insects and disease. Mylavarapu et al. (2007) determined the limiting value of bahiagrass to be 1.5 g kg<sup>-1</sup>. The limiting value is defined as the tissue concentration above which added P will have no effect on plant yield.

Aluminum has no known biological value to animal nutrition, thus it is almost never added to the diet. It can be ingested through soil or grass. In some animals, Al at certain levels can cause P deficiencies. Toxicity is altered by the solubility of Al and quantity of P in the diet. Thompson et al. (1959) proposed ruminant P levels were not as negatively impacted by the consumption of excess Al. Based on results from numerous studies, the Board on Agriculture (1980) set the maximum tolerable level for Al for sheep and cows at 1,000 ppm.

Since bahiagrass is such an important and widely used forage in Florida cattle production, it is vital to consider the impact WTRs may have on forage yield and tissue nutrient concentrations, namely Al. Phosphorus levels should not drop below levels which would decrease yields. Aluminum can affect animal dietary needs and should also be monitored, not only in the amount consumed in the forage but also in the soil.

CHAPTER THREE  
MATERIALS AND METHODS

**Site Description and Experimental Design**

The study was performed on an established bahiagrass (*Paspalum notatum*) pasture at the Range Cattle Research and Education Center (RCREC) in Ona, FL (27°26' N, 81°55' W). Main plots (15.24 X 6.10 m) were divided into subplots (3.05 X 6.10 m) and separated by 3.05 m buffer strips (Fig. 3-1). A randomized block, split-plot design used application method (soil incorporation and surface applied) as the main plot and WTR rate (0, 35 and 70 Mg ha<sup>-1</sup>) as the subplot. Each treatment was replicated four times. Aluminum WTR was incorporated in the top 10-15 cm of soil in the designated plots by disking. The bahiagrass was allowed to grow in from the existing tilled-in material. Elliott et al. (2002) determined practical and effective WTR application rates to be between 20 and 100 Mg ha<sup>-1</sup> through column leaching studies and thus rates for this study were chosen within this range.

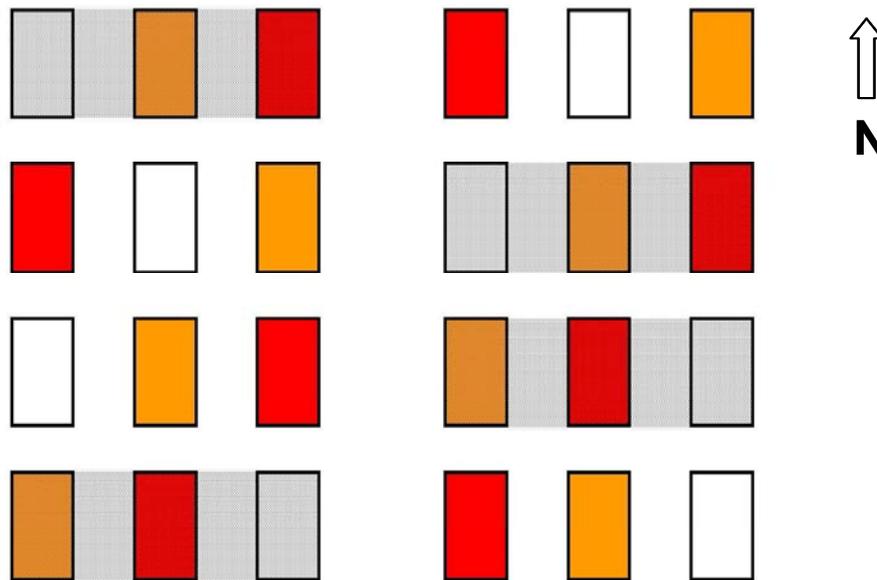


Figure 3-1. Plot design and layout. The shaded regions indicate surface applied treatments and non-shaded regions indicate incorporated treatments. White boxes are the controls, light gray boxes are the 35 Mg ha<sup>-1</sup> WTR application rate and the darker gray boxes are the 70 Mg ha<sup>-1</sup> WTR application rate.

The soils on the site are from the Myakka soil series (sandy, siliceous, hyperthermic aeric alaquod). This soil series is found extensively throughout Florida, covering more than 1.5 million acres. In Hardee County, it makes up more than 55,000 acres which is more than 13% of the county's soils (Soil Survey Staff, 1996).

The study site had very low initial P concentrations (Mehlich-1 P = 2.1 mg kg<sup>-1</sup> for the A horizon). Additional P was surface applied in the form of triple superphosphate (TSP) with a hand-held fertilizer spreader to all plots at a rate of 224 kg P ha<sup>-1</sup>. Applying TSP at high levels allowed for the movement of P throughout the soil profile to be evaluated. It also simulated circumstances when P would be applied at high levels to the soil surface, such as in the application of manures or biosolids at N agronomic rates. A timeline of the various activities on the plots is shown in Table 3-1.

Table 3-1. Dates and descriptions of field activities.

Aug. 15-17, 2006	Laid plot perimeters and collected soil samples.
Sept. 9, 2006	WTR delivered from Manatee County Water Treatment Plant. WTR sieved to 0.64 cm.
Jan. 4, 2007	Initial forage samples taken. Moisture content of sieved WTR determined.
Jan. 5, 2007	Plots mowed and forage removed. WTR weighed into bags for application.
Jan. 11, 2007	Al-WTR applied to plots with a drop fertilizer spreader
Jan. 12, 2007	WTR incorporated into necessary plots. Fertilizer combination spread (34-0-60).
March 26, 2007	Wells placed in the middle of plots 60 cm deep.
April 23, 2007	Nitrification soil samples collected and then TSP applied at 224 kg ha <sup>-1</sup> per plot.
May 24, 2007	Forage harvested. Fertilizer combination spread (34-0-60).
June 20, 2007	Forage harvested. Fertilizer combination spread (34-0-60).
July 20, 2007	Forage harvested. Fertilizer combination spread (34-0-60).
Aug. 20-21, 2007	Second soil samples collected. Forage harvested. Fertilizer combination spread (34-0-60).
Sept. 20, 2007	Nitrification soil samples and groundwater samples collected. Forage harvested.
Oct. 2, 2007	Groundwater samples collected.

### **Water Treatment Residual**

An Al-WTR was selected for field testing because of its effectiveness in controlling P leaching when soluble P was in contact with the WTR. Aluminum WTR was acquired from the Manatee County Water Treatment Plant in Bradenton, FL. Previous studies compared an Al-WTR from this plant to an Al-WTR from Okeechobee, FL, and to other various soil amendments. The ability of each amendment to sorb P and prevent leaching and runoff was evaluated. The other amendments were not recommended for an assortment of reasons including ineffective P sorbing capacities, high concentrations of potentially detrimental nutrients, threats of soil salinity and high cost. The Manatee WTR was recommended over the Okeechobee WTR for field evaluation because of its drier condition, ease of handling and the large quantities

produced yearly (about 4,000 Mg yr<sup>-1</sup>). The recommended rate of application was 2.5% dry weight by mass (O'Connor, 2005); the rates of application for this study were 1.5 and 3.0% dry weight by mass.

The initial size of the WTR particles varied largely from clay size upwards to 12 cm so they were passed through a 0.64 cm mesh to obtain better uniformity. The moisture content of the WTR was not high enough to cause a problem when sieving and so it was not previously air dried. Sieved WTR was stored at the study site under visqueen tarps until application. Five random samples were taken the day prior to WTR application to determine the average moisture content of the AI-WTR. Given the 30.3% moisture content, the amount of WTR needed for the 35 and 70 Mg ha<sup>-1</sup> application rates was 93 and 186 kg per plot, respectively. The WTR was applied with a drop fertilizer spreader to assure an even distribution.

### **Soil Sampling**

Two soil samplings were performed during the course of the study. An initial sampling before any treatment was applied was taken in August 2006 and the second sampling was taken in August 2007. Two holes were augured to a depth of one meter for each plot. The profile was carefully laid out in rain gutters and depths noted at each change in horizon (Table 3-2). One composite sample from each horizon was then taken from both profiles. The A horizon was split into two depth increments, one from the top 8 cm and one from below 8 cm. This technique was followed to better quantify differences due to the incorporation process. All soil samples were placed in plastic bags and kept on ice until they were transported to Gainesville, FL, for laboratory analysis. All samples were air-dried before analysis.

Table 3-2. Depths to the bottom of the soil horizons sampled. The A horizon was sampled from 0-8 cm and 8 cm to the bottom of the horizon.

Soil Horizon	Average Depth (cm)
A	8
A	21
E	54
Bw	75
Bh	83
Bt	≥ 100

Soil samples were collected on April 23, 2007 and September 20, 2007 to determine nitrification potential. In these samplings, the top 5 cm of the A horizon were placed in sampling bags and stored in a cooler. A 1.3 cm diameter stainless steel probe was pre-marked to 5 cm and two samples from each quartile of the plot were collected.

### **Forage Sampling**

An initial forage sampling was conducted in January 2007 before any treatments were applied. Two 51cm<sup>2</sup> tracts were used to take forage samples from each plot. The grass was cut to approximately 3 cm above the ground with hand shears. Plots were harvested every 30 days from May to September, 2007, with a flail mower and a dump wagon attachment to collect and weigh harvested forage. Hand drawn samples at each harvest were dried at 105°F, moisture content calculated and sieved to 2 mm for analysis. After each harvest, plots were fertilized with ammonium nitrate and potassium chloride at rates of 89.7 kg N ha<sup>-1</sup> and 44.9 kg K<sub>2</sub>O ha<sup>-1</sup>, respectively.

### **Groundwater Sampling**

A 60 cm PVC well was placed above the spodic horizon in the center of all plots. There were slits in the lower 10 cm to allow water to enter the well. The goal was to collect groundwater samples when a perched water table existed to observe concentrations of P leaching. Due to an unusually dry early summer, samples were not able to be collected until late

in August and September. The RCREC received 7.4 cm of rain in May through August of 2007, while the average amount of rain for the previous 4 years was 13.85 cm (FAWN).

Water samples were collected using a battery operated vacuum pump. Prior to sampling, standing water in the wells was purged to assure that fresh water was being sampled. After approximately one hour, the wells were recharged and 200 mL samples were collected into Nalgene bottles. Samples were preserved with H<sub>2</sub>SO<sub>4</sub> (1 drop per 20 mL sample). Samples were placed on ice before refrigerated in the laboratory.

The groundwater table depth was measured using a pressure transducer to measure the potential of water at varying depths (modeled from the design of Schumann and MunozCarpena, 2002). The transducer was calibrated by using a bucket of water at known water levels and recording the voltage. A regression was then made from several values and an equation was produced to convert voltage readings to water depth. A Hobo datalogger was set to record voltage readings in the field every 15 minutes.

### **Analytical Methods**

#### **Soil**

All soil samples were air dried before analysis. Water extractable P, Mehlich-1 P, Fe and Al, pH and the soil P storage capacity were determined for all soil samples.

Water extractable P (WEP) was determined using a 1:10 soil to solution ratio (Kuo, 1996). Three grams of soil was weighed into 50mL centrifuge tubes and 30mL of distilled deionized water (DDI) was added. The tubes were placed on a mechanical shaker for one hour and then passed through a 0.45 µm filter with a vacuum. After extraction the samples were refrigerated. Analysis with a Technicon<sup>TM</sup> Autoanalyzer was performed within 48 hours.

Mehlich-1 extractable P, Fe and Al were determined on all soil samples (Mehlich, 1953). Five grams of soil was weighed into a 50 mL centrifuge tube and 20 mL of Mehlich-1 solution

(0.05 N HCl and 0.025 N H<sub>2</sub>SO<sub>4</sub>) was added. The tubes were shaken for five minutes and immediately filtered through Whatman #41 filter paper. Samples were analyzed on a Technicon™ Autoanalyzer.

The pH was determined by weighing 5 grams of soil and adding 10 mL DDI water giving a 1:2 soil to solution ratio. The solution was stirred gently and after 30 minutes a reading was taken with an Orion pH electrode.

### **Nitrification Potential**

The nitrification potential of the soils was determined using the shaken soil slurry method (Hart et al., 1996). The method was performed the day after soil collection. Fifteen grams of field moist soil was added to a 250 mL Erlenmeyer flask. One hundred milliliters of a solution containing 1.5 mM NH<sub>4</sub><sup>+</sup> and 1.6 mM PO<sub>4</sub><sup>-3</sup> was added to each flask. The original procedure called for 1.0 mM PO<sub>4</sub><sup>-3</sup> but this was increased to account for the low soil acidity. The buffer solution was adjusted to pH 7.2 by adding NaOH dropwise. Parafilm with a slit cut on top was placed securely over each flask to allow oxygen exchange but minimize evaporation. Samples were placed on a shaker at 1,000 rpm in an incubator at 25°C. Approximately 10 mL of slurry was collected at 2, 4, 8, 20 and 24 hours and filtered through Whatman #40 filter paper. Samples were stored in scintillation bottles and acidified with one drop of concentrated H<sub>2</sub>SO<sub>4</sub>. Analysis for nitrate-N was performed on a rapid flow analyzer using EPA Method 353.2. The rate of nitrate-N production (mg NO<sub>3</sub><sup>-</sup>-N L<sup>-1</sup> h<sup>-1</sup>) was calculated by linear regression of solution concentration versus time. The gravimetric soil water content was determined for the same soil samples (mass of water / mass of dry soil) and used to calculate the dry soil potential nitrification rate.

## **Groundwater**

Groundwater samples were analyzed for soluble reactive P (SRP) within 48 hours of collection. Twenty milliliters of the sample was filtered through a 0.45 $\mu$ m filter before analysis. Samples were analyzed using a Technicon<sup>TM</sup> Autoanalyzer by EPA Method 365.1.

## **Forage**

All forage samples were dried and then ground to 2 mm with a Wiley mill. Neutral detergent fiber content (NDF), N (crude protein), P, Al, Ca and Mg were determined for forage samples.

Neutral detergent fiber estimates the plant's cell wall contents and is the best indicator of how much forage an animal will potentially consume. To determine the NDF, 0.50 g of grass was weighed into a bag, the weight was recorded and the bag sealed (Golding et al., 1985). The sample was then boiled in a neutral detergent solution for one hour leaving the cell wall constituents. The bags were soaked in acetone and placed in an oven for 2-3 hours to complete the drying process. The dry bags were weighed and the NDF was reported on a dry matter basis.

Tissue nutrient content (N, P, Al, Fe, Mg and Ca) was determined using a modification of the total Kjeldahl method (Gallaher et al., 1975). Samples were weighed to 0.25 g and placed in 75 mL volumetric glass tubes. A catalyst (containing 9:1 K<sub>2</sub>SO<sub>4</sub>:CuSO<sub>4</sub>), 6 mL of concentrated H<sub>2</sub>SO<sub>4</sub> and 2 mL of H<sub>2</sub>O<sub>2</sub> were added and samples were digested at 375°C for at least 4 hours. After samples cooled they were brought to volume with DDI water, mixed thoroughly, filtered and stored in 20 mL scintillation vials. Nitrogen and P in the digestate was determined by semiautomated colorimetry using a Technicon<sup>TM</sup> Autoanalyzer by TKN Method 351.2. Crude protein was calculated by multiplying the amount of N in the sample by 6.25. Aluminum, Fe, Mg and Ca were determined by atomic adsorption spectrophotometry.

### **Quality Assurance and Quality Control**

Quality assurance and quality control (QA/QC) were met with 10% repeats and blanks for each procedure. Quality control was determined by certified external standards and standard calibration curves. A standard deviation of 10% was required for all repeats and quality control checks.

### **Statistical Analysis**

Statistical analysis was executed using SAS version 8.2 1999-2001 (SAS Institute Inc., Cary, North Carolina, USA). The Waller-Duncan procedure was used to compare the mean values for application method and application rate. ANOVA was used to determine the individual variable responses (application method and rate) and interactions between the variables. A single degree of freedom was used to explore the general mean contrast between application rates.

## CHAPTER FOUR RESULTS AND DISCUSSION

### Soil Properties

#### Initial Soil Characteristics

The soil on the experimental site was classified as Myakka fine sand based on soil survey information (USDA-Soil Conservation Service, 1984). This was substantiated during the sampling process. Twenty-two of the 24 plots had a spodic horizon within the one-meter depth sampled.

Initial soil WEP and Mehlich-1 P average concentrations were relatively low and decreased with depth through the A and E horizons and then Mehlich-1 P slightly increased in the B horizons whereas WEP remained low (Table 4-1). An anomaly was found in the A horizon (0-8 cm) between WEP and Mehlich-1 P concentrations which were 9.0 and 2.1 mg kg<sup>-1</sup>, respectively. It is uncommon for WEP concentrations to be higher than Mehlich-1 P concentrations because extracting P with an acid solution should extract more P than the water extract. There is no logical explanation for this observation and it was not observed in post-treatment sampling. Mehlich-1 P concentrations in the B horizons were approximately 30-fold greater than in the A and E horizons. Mehlich-1 P concentrations less than 10 mg kg<sup>-1</sup> in the A horizon are considered very low for agronomic crops as well as bahiagrass and P fertilization would be recommended for this site (Kidder et al., 2002). These concentrations are slightly lower to those found in forage fields in South Florida (Graetz and Nair, 1995).

Table 4-1. General characteristics in soil samples taken prior to amendment application (August 2006) averaged for all plots.

Soil Horizon	Soil pH	Water Extractable P	Mehlich-1 P	Mehlich-1 Al	Mehlich-1 Fe	Al:Fe Ratio
-----mg kg <sup>-1</sup> -----						
A (0-8 cm)	4.59	9.0 ± 3.8	2.1 ± 1.2	22	9.9	4.6
A (8-21 cm)	4.42	3.0 ± 1.6	1.4 ± 2.7	35	14.7	5.0
E	4.36	0.8 ± 0.8	0.7 ± 1.9	20	9.0	4.5
Bh	5.62	1.3 ± 1.4	29.1 ± 16.7	334	71.7	9.7
Bw	5.40	1.9 ± 3.3	36.1 ± 16.6	287	54.1	11.0
Bt	6.37	2.0 ± 0.9	38.4 ± 19.5	131	25.4	10.7

Mehlich-1 Al and Fe concentrations were approximately 10 and 4 fold greater, respectively, in the B horizons than in the A and E horizons (Table 4-1). This accumulation of metals is a result of spodic horizon formation when metals and carbon are redistributed from one zone to another (Franzmeier et al., 1965). The Al:Fe ratios in the A-E and B horizons were approximately 5 and 11, respectively. It is typical for Fe concentrations to be lower than Al concentrations in poorly drained Spodosols in Florida (Sodek et al., 1990). These spodic Al and Fe values are similar to those found in a South Florida beef pasture by Nair and Graetz (2002).

#### **Aluminum Water Treatment Residual Characteristics**

The moisture content and pH of the Al-WTR were 30.3% and 6.8, respectively (Table 4-2). Mehlich-1 Al and Fe concentrations were 1,007 and 17 mg kg<sup>-1</sup>, respectively. A recent study using material from the Manatee County Water Treatment Plant found that nearly all the Al and Fe was oxalate extractable (O'Connor, 2005). Oxalate extracts amorphous Al and Fe which are highly reactive to P (McKeague et al., 1971). Another study also using material from the same source at an earlier date found that approximately 80% of the Al was amorphous (Miyittah-Kporge, 2004) and the total Al concentration was 89.1 g kg<sup>-1</sup> (O'Connor et al., 2002). The WTR had a substantial total P content (1,300 mg kg<sup>-1</sup>). Although this will result in a large amount of P added to the soil, there is little chance of P release from the WTR since WEP

concentrations were below the detection limit. Most of this P is bound by the extensive amount of Al. Desorption of P from an Al-WTR was reported to be less than 1% (Makris et al., 2004).

Table 4-2. General characteristics of the Manatee County Al-WTR.

Moisture Content	pH*	Mehlich-1 Al	Mehlich-1 Fe	Mehlich-1 P	Total P**	Water Extractable P
30.3%	6.8	1007	17	2	1300	BDL***

\* Soil:Solution ratio of 1:2

\*\* Total P determined by ignition method (Anderson 1976)

\*\*\* Detection limit was 0.02 mg L<sup>-1</sup>

### Soil pH

Soil pH eight months after WTR application was affected by application method for the A (0-8 cm) and A (8-21 cm) horizons and nearly affected by WTR application rate in the A (0-8 cm) horizon (Table 4-3). There were no effects of WTR application method or rate in the E and B horizons. In the A (0-8 cm) and A (8-21 cm) horizons differences resulted from application method in which the incorporated treatments had a higher pH than the surface applied treatments (Table 4-4).

Table 4-3. The p-values for the pH data using ANOVA and general mean contrast (single degree of freedom).

Soil Horizon	Method	Rate	Method*Rate	Control vs. Treatments	35 Mg ha <sup>-1</sup> vs. 70 Mg ha <sup>-1</sup>
A (0-8 cm)	<i>0.009</i>	0.0564	0.9094	<i>0.0126</i>	0.2683
A (8-21 cm)	<i>0.0083</i>	0.1034	0.6941	<i>0.0403</i>	0.6156
E	0.9536	0.6706	0.5785	0.7623	0.6168
B	0.9805	0.1316	0.3168	0.5134	<i>0.0456</i>

† Values italicized are significant at  $\alpha = 0.05$ .

Table 4-4. Soil pH averages for incorporated and surface applied treatments 8 months after WTR application.

Application Method	A (0-8 cm)	A (8-21 cm)	E	B
Incorporated	4.89 a	4.73 a	4.89 a	5.98 a
Surface Applied	4.71 b	4.43 b	4.93 a	6.18 a

† Treatments with similar letters show no significant difference for that column by Waller-Duncan ( $p < 0.05$ ).

Soil pH is important when applying Al-WTRs to agricultural land because if the pH drops below 4.0, Al becomes more soluble and toxicity implications become more serious. The target pH for bahiagrass in Florida is 5.5 (Mylavarapu et al., 2007). The WTR used in this study had positive effects on soil pH, showing an increasing trend with WTR application, but the soil pH values even after WTR application suggest that liming of this soil should be recommended. There may have been a slight acidifying effect from the ammonium nitrate fertilizer (89.7 kg N ha<sup>-1</sup>) applied after each harvest. However all treatments, including the controls, received the same fertilization so the relative effect of WTR application rate on soil pH can still be observed.

The pH of WTRs from different treatment plants can vary from 5.3 to 8.0 based on their chemical makeup, how the WTR was produced and the characteristics of the water being purified (Silveira et al., 2006, Codling et al., 2002, and Ippolito et al., 2003). Independent of the exact pH of the material, WTRs have not expressed a strong effect on the soil pH in recent studies. Ippolito et al. (2003) looked at changes in pH when WTR was shaken with a potassium chloride solution. Subsamples were taken throughout a 215 day study and results showed the initial pH of the WTR solution was 7.6 and increased to 8.15 after approximately 135 days. In another study, soil samples from the top 2.5 cm with WTR applied at rates up to 18 Mg ha<sup>-1</sup> showed no increase in pH after four months (Haustein et al., 2000). The pH of the WTR in this study was 5.8 and the pH of the control plots was 5.5. In the same study, a HiClay Alumina (HCA) amendment was compared to the WTR. The HCA had a pH of 3.4 and reduced the soil pH from 5.53 (control) to 5.27 at an application rate of 18 Mg ha<sup>-1</sup>. Codling et al. (2007) applied combinations of poultry litter and WTR at rates up to 11.2 Mg ha<sup>-1</sup> and observed minimal changes in pH due to WTR application.

Tillage may cause pH to be higher than if the soil was left unmanaged. Godsey et al. (2006) reported that over many years no-till plots of wheat, soybean and sorghum had a lower average pH than conventionally tilled plots. No-tillage systems can increase the organic matter (OM) of the soil. Shertz (1994) showed an example in which a farm in Illinois had an increase in soil OM from 1.87% to 4.0% in fifteen years of no till. Higher soil OM levels can potentially lead to an increase in soil acidity (Coleman and Thomas, 1967). Carbon dioxide is produced when OM decomposes, which releases  $H^+$  when it reacts with water. Organic matter also contains carboxylic and phenolic groups which act as weak acids (Havlin et al., 2005). This acidifying action of soil OM has a minimal effect on the overall soil pH in mineral soils but can still be a factor. No-till systems also have higher rates of water infiltration and thus leach cations such as  $Ca^+$  which can be replaced by  $H^+$ , causing higher rates of soil acidity over time (Powers and McSorley, 2000).

### Water Extractable Phosphorus

Water extractable P is the form of P most easily lost to leaching and subsurface transport. Application method affected WEP concentrations in the A (8-21 cm) and E horizons while application rate influenced WEP concentrations in the A (0-8 cm), A (8-21 cm) and E horizons (Table 4-5). The interactions between WTR application method and rate were not significant.

Table 4-5. The p-values for the WEP data using ANOVA and general mean contrast (single degree of freedom).

Soil Horizon	Method	Rate	Method*Rate	Control vs. Treatments	35 Mg ha <sup>-1</sup> vs. 70 Mg ha <sup>-1</sup>
A (0-8 cm)	0.5962	<i>&lt;.0001</i>	0.9011	<i>&lt;.0001</i>	<i>0.0341</i>
A (8-21 cm)	<i>0.0319</i>	<i>0.0004</i>	0.223	<i>0.0001</i>	0.5329
E	<i>0.0066</i>	<i>0.0004</i>	0.9758	<i>&lt;.0001</i>	<i>0.5543</i>
B	0.9731	0.447	0.5473	0.616	0.2504

† Values italicized are significant at  $\alpha = 0.05$ .

Water extractable P concentrations are shown by horizon for the incorporated and surface applied treatments in Figs. 4-1 and 4-2, respectively. Recall that 14 weeks after WTR was applied to the appropriate treatments, a blanket application of P as triple superphosphate (TSP) was surface applied at a rate of 224 kg P ha<sup>-1</sup> to all plots including the controls. Concentrations of WEP increased in the controls compared to the pretreatment concentrations due to this application of P. Thus, data are also presented in these figures for WEP concentrations prior to the application of P.

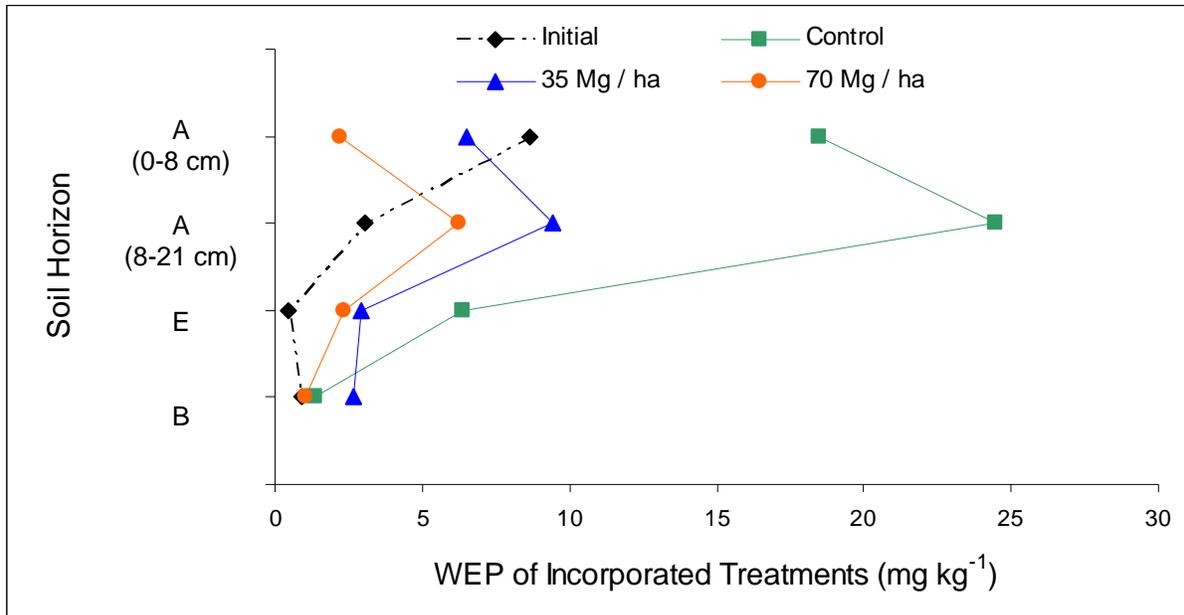


Figure 4-1. The WEP concentrations of incorporated treatments by soil horizon. The dashed line represents the average WEP concentration of all plots from the 2006 sampling before amendment application. The other lines represent the WEP concentrations from the 2007 soil sampling after amendment application.

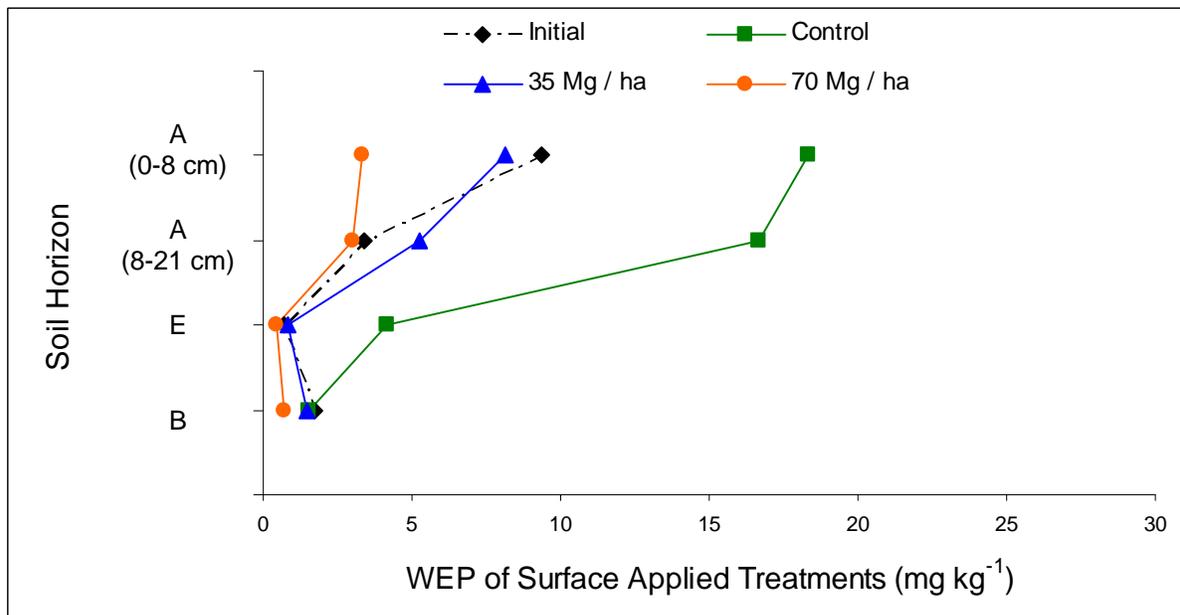


Figure 4-2. The WEP concentrations of surface applied treatments by soil horizon. The dashed line represents the average WEP concentration of all plots from the 2006 sampling before amendment application. The other lines represent the WEP concentrations from the 2007 soil sampling after amendment application.

There was considerable movement of WEP downward in the soil profile in the control treatments four months after TSP application. Increases in WEP concentrations were observed in all horizons except the B horizon (Figs. 4-1 and 4-2). It is not possible to determine if the lack of response in the B horizon was due to the P bonding strength of the B horizon or to the lack of movement to this depth. Application of WTR decreased the WEP concentrations relative to the control in all horizons except the B horizon where there was no effect. Differences between the 35 and 70 Mg ha<sup>-1</sup> WTR application rates were only significant in the A (0-8 cm) horizon (Table 4-5).

The WEP concentrations in the incorporated treatments of the A (8-21 cm) and E horizons were greater than the surface applied treatments (Table 4-5) suggesting there may be a potential effect of tillage on P transport in the soil. Tilling destroys macropores which allow for more rapid water movement (Hill and Mannering, 1995). Thus, no-tillage systems have greater water movement which may result in faster P transport and may help explain the WEP concentrations between the incorporated and surface applied treatments. This P may not be observable in the B horizon due to extensive bonding by Al in the spodic horizon.

These results are similar to previous results reported in the literature. Codling et al. (2000) studied the effects of WTRs and iron rich residues (IRR) when added to poultry litter and soils. Each amendment was added to the litter and soils at rates up to 10% dry weight by mass. Samples were incubated for 7 weeks and subsamples taken at 2, 4 and 7 weeks. The litter and WTR combination showed a decrease in water soluble P (WSP) as WTR rate increased. The WTR reduced WSP more than the IRR. Reductions in WSP compared to the controls at the highest application rate were 87% and 42% for the fourth week for WTR and IRR, respectively. Similar results were shown when a WTR was added to three different soils at rates up to 5% dry

weight by mass in a wheat growth chamber experiment (Codling et al., 2002). Soil WSP decreased as WTR rate increased for all three soils and was reduced 98 to 99% at the 5% application rate when compared to the controls.

### Mehlich-1 Extractions

Mehlich-1 P concentrations were affected by application method in the A (8-21 cm) horizon (Table 4-6). The distribution of Mehlich-1 P in the soil profile for incorporated and surface applied WTR treatments is shown in Figs. 4-3 and 4-4, respectively. As noted earlier for WEP concentrations, the blanket application of TSP is responsible for the considerable increases in Mehlich-1 P in 2007 compared to 2006.

Table 4-6. The p-values for the Mehlich-1 data using ANOVA and general mean contrast (single degree of freedom).

Response Variable	Soil Horizon	Method	Rate	Method*Rate	Control vs. Treatments	35 Mg ha <sup>-1</sup> vs. 70 Mg ha <sup>-1</sup>
Mehlich-1 P	A (0-8 cm)	0.2794	0.4589	0.8492	0.2202	0.9433
	A (8-21 cm)	<i>&lt;.0001</i>	0.0696	0.5926	0.0617	0.1762
	E	0.0811	0.1607	0.8637	0.0616	0.8085
	B	0.1635	0.3327	0.286	0.4761	0.1217
Mehlich-1 Al	A (0-8 cm)	<i>0.0099</i>	<i>&lt;.0001</i>	0.1267	<i>&lt;.0001</i>	<i>0.0026</i>
	A (8-21 cm)	<i>0.0005</i>	<i>&lt;.0001</i>	<i>0.0174</i>	<i>&lt;.0001</i>	<i>0.0076</i>
	E	0.4174	<i>0.038</i>	0.1199	<i>0.0375</i>	0.0825
	B	0.7486	0.8271	0.6664	0.5454	0.9947
Mehlich-1 Fe	A (0-8 cm)	0.0742	0.1745	0.5916	0.1465	0.227
	A (8-21 cm)	0.7233	0.8367	0.7284	0.6436	0.6983
	E	0.5292	0.2455	0.613	0.114	0.5795
	B	0.6607	0.0669	0.9241	0.0557	0.146

† Values italicized are significant at  $\alpha = 0.05$ .

The only difference observed for Mehlich-1 P concentrations was in the A (8-21 cm) horizon where the WTR incorporated treatments had greater concentrations than the surface applied treatments (Table 4-7). This may be a result of the incorporation process since tilling did not occur exactly at 8 cm where the A horizon was split for two samples. Mehlich-1 P concentrations followed the same general trend for all treatments in both the incorporated and

surface applied treatments (Figs. 4-3 and 4-4). This trend shows less Mehlich-P in the A (0-8 cm) horizon of the controls than WTR amended treatments but more Mehlich-1 P in the A (8-21 cm) and E horizons of the controls than WTR amended treatments. This suggests P is being retained near the surface in the WTR treated soils and not moving lower down the soil profile. This trend may be more significant with time as P will continually move downward in the controls.

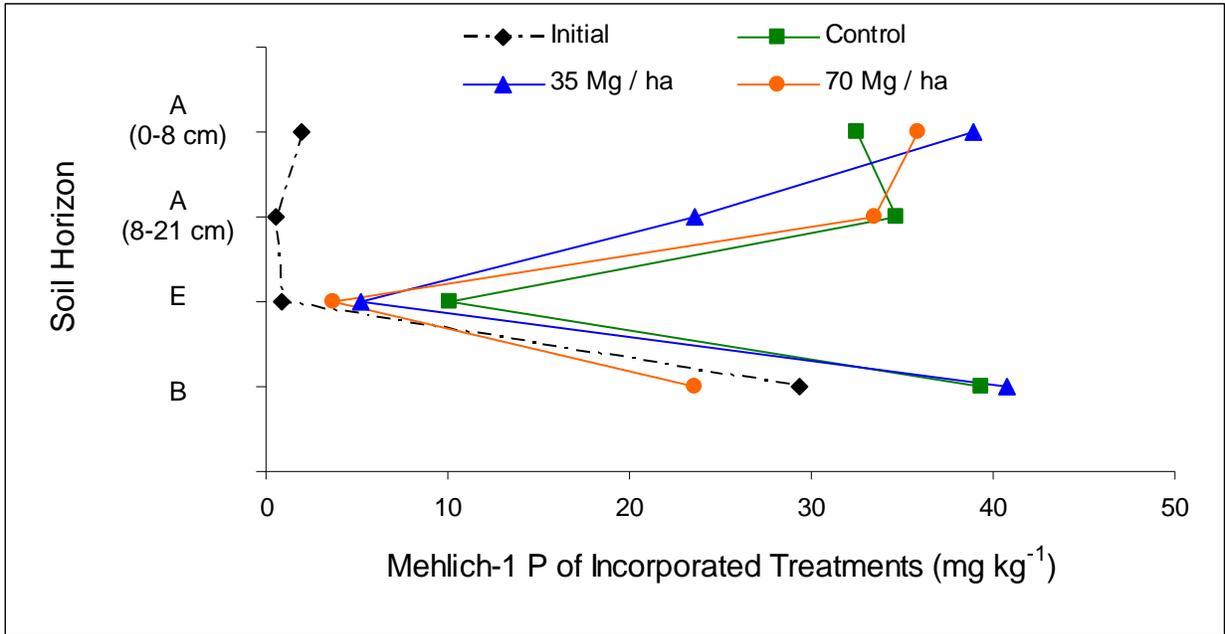


Figure 4-3. Mehlich-1 P concentrations of incorporated treatments by soil horizon. The dashed line represents the average concentration of all plots from the 2006 sampling before amendment application. The other lines represent the WEP concentrations from the 2007 soil sampling after amendment application.

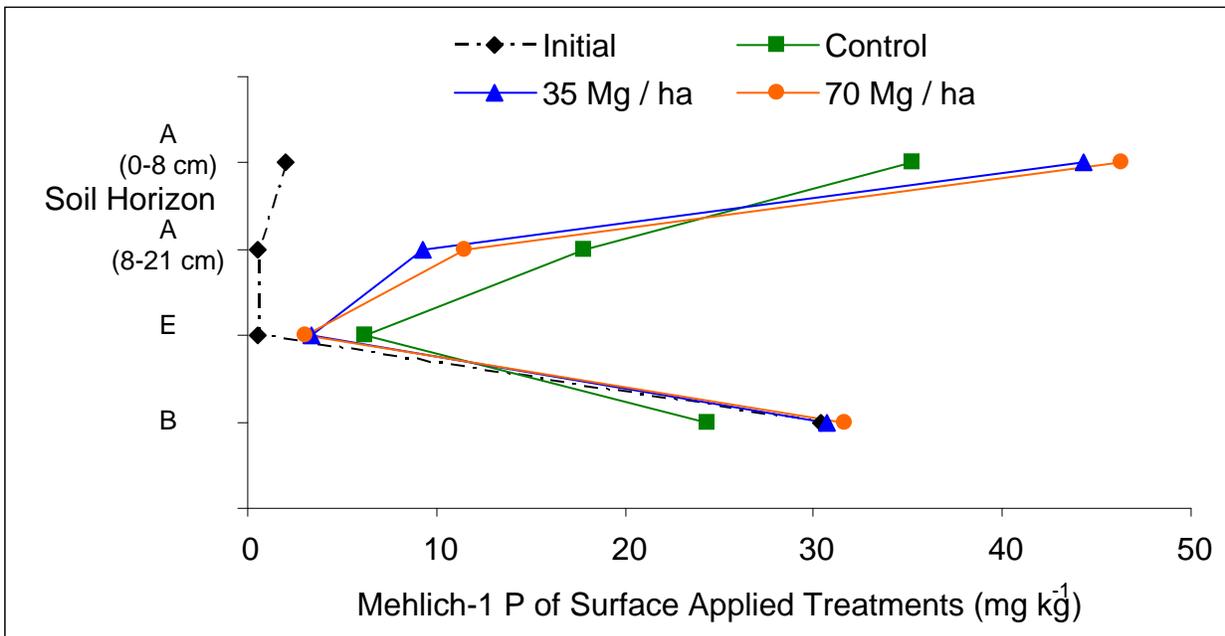


Figure 4-4. Mehlich-1 P concentrations of surface applied treatments by soil horizon. The dashed line represents the average concentration of all plots from the 2006 sampling before amendment application. The other lines represent the WEP concentrations from the 2007 soil sampling after amendment application.

Rew (2006) did not observe changes in soil Mehlich-1 P before or after rainfall simulations in which an Al-WTR was applied at rates up to 2.5% dry weight by mass. The soil was impacted with heavy loads of manures and so had initially high P concentrations. Before the simulations began, some treatments had the WTR mixed throughout the top 10 or 20 cm and no Mehlich-1 P differences occurred due to this application method.

Mehlich-1 Al concentrations were higher in the A (0-8 cm) and A (8-21 cm) horizons when the WTR was incorporated compared to surface applied (Table 4-6; Figs. 4-5 and 4-6). Application rate of WTR affected Mehlich-1 Al concentrations in the A (0-8 cm), A (8-21 cm) and E horizons. Additions of WTR did not change the soil Fe concentration due to the low Fe concentrations in the Al-WTR (Table 4-6).

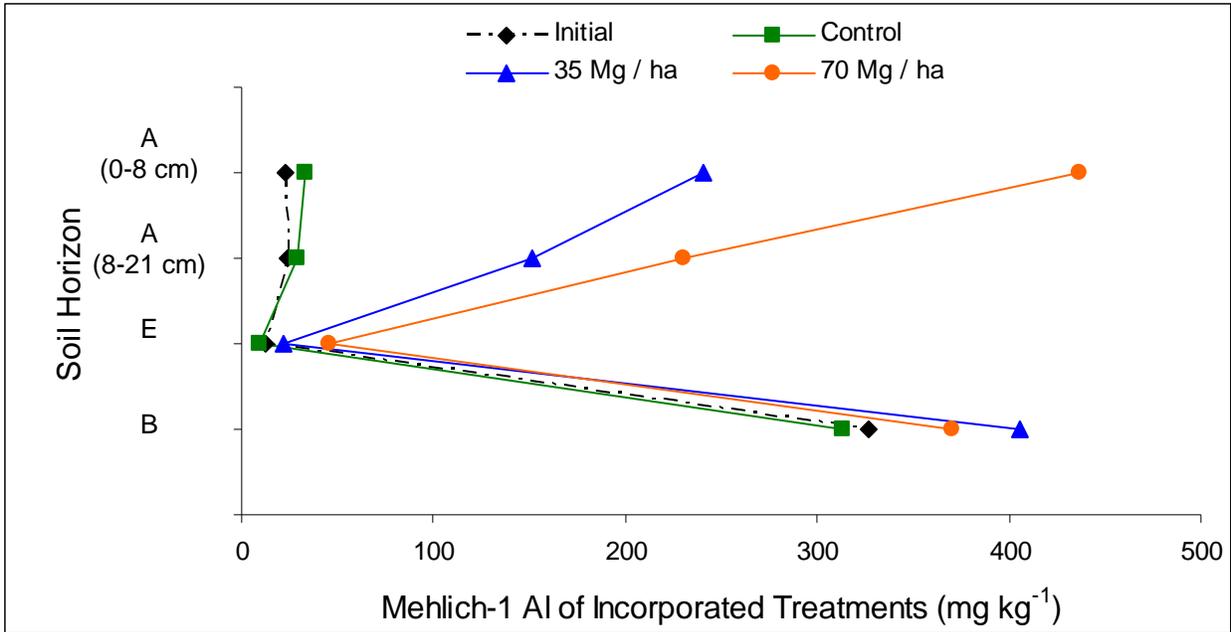


Figure 4-5. Mehlich-1 Al concentrations of incorporated treatments by soil horizon. The dashed line represents the average concentration of all plots from the 2006 sampling before amendment application. The other lines represent the WEP concentrations from the 2007 soil sampling after amendment application.

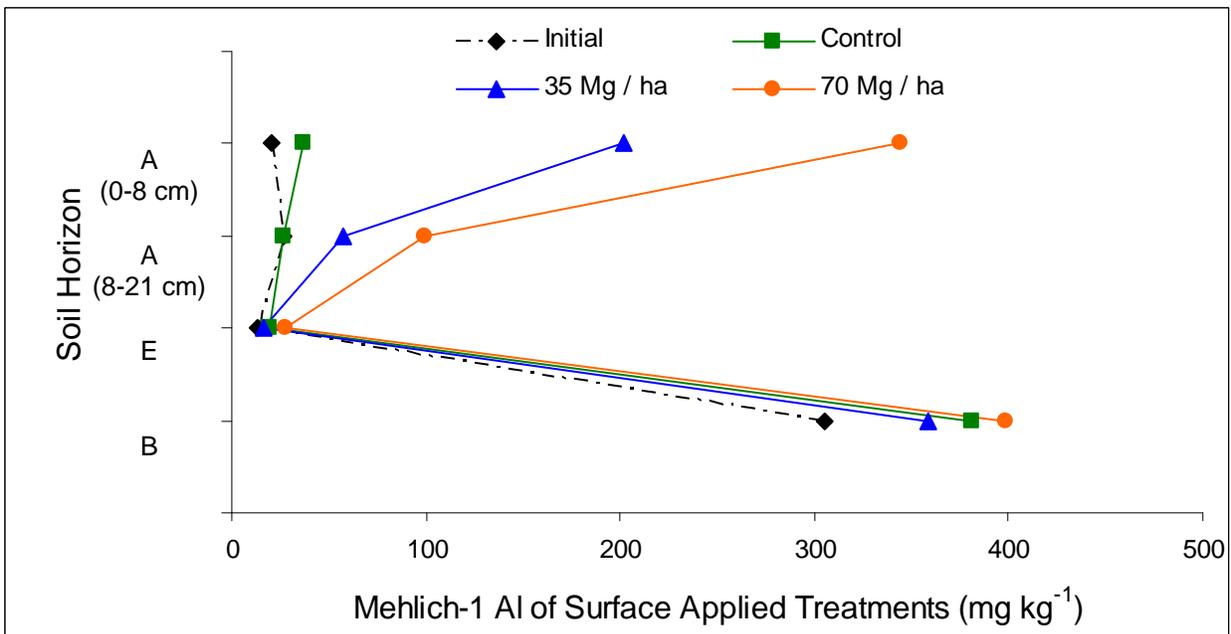


Figure 4-6. Mehlich-1 Al concentrations of surface applied treatments by soil horizon. The dashed line represents the average concentration of all plots from the 2006 sampling before amendment application. The other lines represent the WEP concentrations from the 2007 soil sampling after amendment application.

Higher Al concentrations suggest a greater potential of the soil to retain P. It also results in a greater need to be aware of possible Al toxicity to plants. These potential effects will be discussed in the forage section of this chapter.

### **Soil Phosphorus Storage Capacity**

The soil P storage capacity (SPSC) was estimated from the following calculation determined by Nair and Harris (2004):

$$\text{SPSC (mg P kg}^{-1}\text{)} = (0.15 - \text{PSR}) (\text{Al} + \text{Fe}) \times 31$$

where  $\text{PSR} = (\text{P}) / (\text{Al} + \text{Fe})$  and all elements are expressed as mmoles. The P, Al and Fe values were Mehlich-1 extractable concentrations. The 0.15 value is the threshold for Florida soils suggested by Nair and Harris (2004) and 31 is the atomic mass of P. When SPSC values are greater than 0 the soil is acting as a P sink and when SPSC values are less than 0 the soil is acting as a P source. The true SPSC calculation uses oxalate extractable concentrations instead of Mehlich-1 concentrations. Therefore the SPSC values shown here do not give exact values but are instead approximations in which relative changes can still be analyzed. There have been other instances when Mehlich-1 values for P, Al and Fe were used to calculate SPSC values (Nair and Harris, 2004).

The SPSC factor is unique in that it takes into account not only how much P the soil contains from past loadings but also how much P retention capacity remains in the soil. Soil test P (STP) concentrations are often used to determine environmental risks due to P, but a low STP does not necessarily indicate that the soil has the capacity to retain added P (Nair and Harris, 2004).

The average SPSC values prior to application of any treatment for the A (0-8 cm), A (8-21 cm), E and B horizons were 2, 4, 3 and 44 mg kg<sup>-1</sup> respectively. The B horizon showed a higher SPSC value due to higher concentrations of Fe and Al. All horizons showed positive

values suggesting the soils could retain additional P. The A and E horizons had low SPSC values close to zero, or equilibrium, implying the soils could easily become P sources. Even though these soils had low initial P concentrations, they also contained very little Al and Fe to give them any significant P retention capacity.

The SPSC was affected by the addition of P and WTR. Application method affected SPSC only in the A (0-8 cm) horizon while rate affected SPSC in all horizons except the B horizon (Table 4-7). Both of the control treatments became significantly negative as a result of adding P to a soil which had an initially low SPSC (Figs. 4-7 and 4-8). These soils have lost the ability to retain P and prevent leaching. SPSC values increased as WTR application rates increased at every horizon except the B horizon where P and Al levels were unaffected.

Table 4-7. The p-values for the SPSC data using ANOVA and general mean contrast (single degree of freedom).

Soil Horizon	Method	Rate	Method*Rate	Control vs. Treatments	35 Mg ha <sup>-1</sup> vs. 70 Mg ha <sup>-1</sup>
A (0-8 cm)	<i>0.0004</i>	<i>&lt;.0001</i>	<i>0.019</i>	<i>&lt;.0001</i>	<i>0.0006</i>
A (8-21 cm)	0.1036	<i>0.0012</i>	0.1043	<i>0.0003</i>	0.5023
E	0.2506	<i>0.018</i>	0.7193	<i>0.0065</i>	0.4346
B	0.4223	0.7748	0.6761	0.4874	0.9717

† Values italicized are significant at  $\alpha = 0.05$ .

In the A horizon (0-8 cm), the incorporated treatments with WTR have greater SPSC values than the original soils suggesting that the applied WTR increased the soil's ability to retain P even after a large amount of P was added. However, Figure 4-8 shows that the surface applied treatments with WTR were only slightly positive at the 70 Mg ha<sup>-1</sup> application rate and dropped below zero at the 35 Mg ha<sup>-1</sup> application rate.

The E horizon soils treated with WTR continued to have SPSC values near zero similar to the pre-amendment soils. While the WTR did not increase the soil's capacity to retain P at

this depth, it did prevent the soil from becoming a P source as was observed in the negative values of the controls.

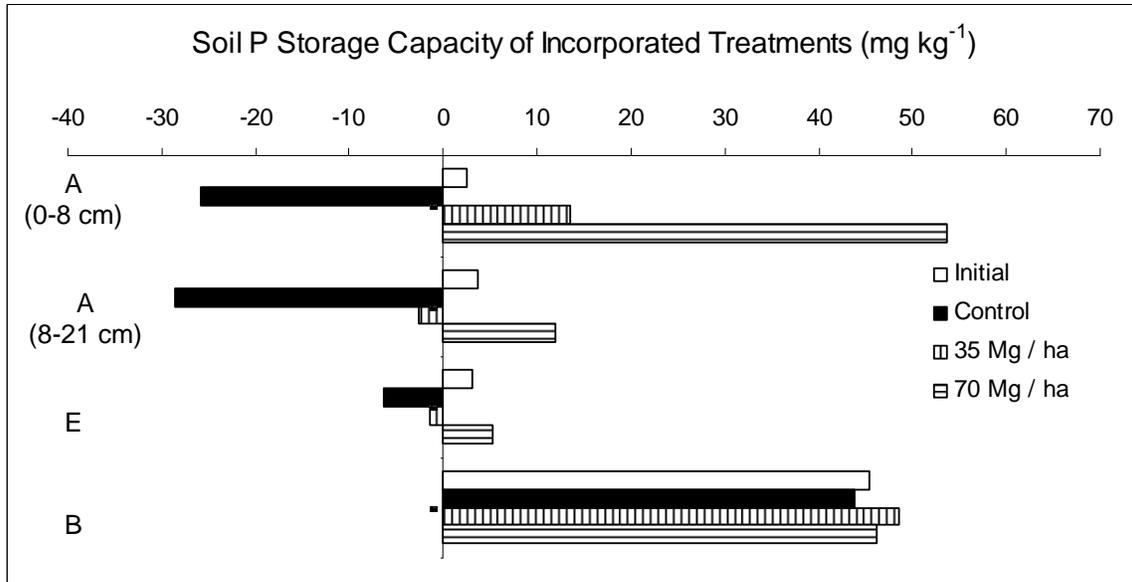


Figure 4-7. Soil P storage capacity of incorporated treatments by soil horizon. The initial values are the average SPSC values of all plots from the 2006 sampling before amendment application. The control and WTR amended values are from the 2007 soil sampling after amendment application.

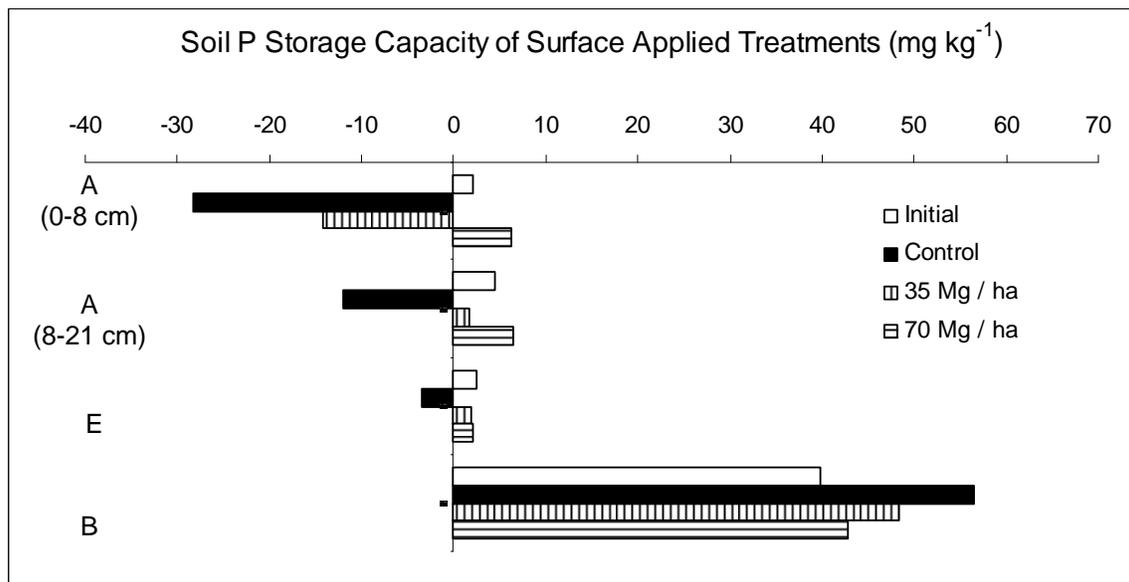


Figure 4-8. Soil P storage capacity of surface applied treatments by soil horizon. The initial values are the average SPSC values of all plots from the 2006 sampling before amendment application. The control and WTR amended values are from the 2007 soil sampling after amendment application.

Figure 4-9 helps explain the differences in SPSC values of the incorporated and surface applied treatments. The incorporated treatments had Al (from the WTR) mixed throughout the top 10-15 cm of the soil profile by the tilling process. The A horizon soil samples were taken above and below 8 cm. Samples were not mixed before being collected but rather taken at regular increments along the length of the profile sampled (8 cm in this case) so the samples from the surface applied treatments contained soil that did not include WTR. Recall that the soil before WTR amendment had very low amounts of Fe and Al. Since this poor P retaining soil, coupled with a larger amount of P from TSP application, was used to calculate the SPSC, the value dropped below zero even though WTR was present. The soil sampling process involved taking intact cores and placing them in sequence in a rain gutter. Subsequent subsampling from each increment was done by compositing random samples taken with a spatula. This subsampling protocol may have resulted in a non-uniform sampling of the given soil horizon thereby explaining differences in SPSC between incorporated and surface applied treatments.

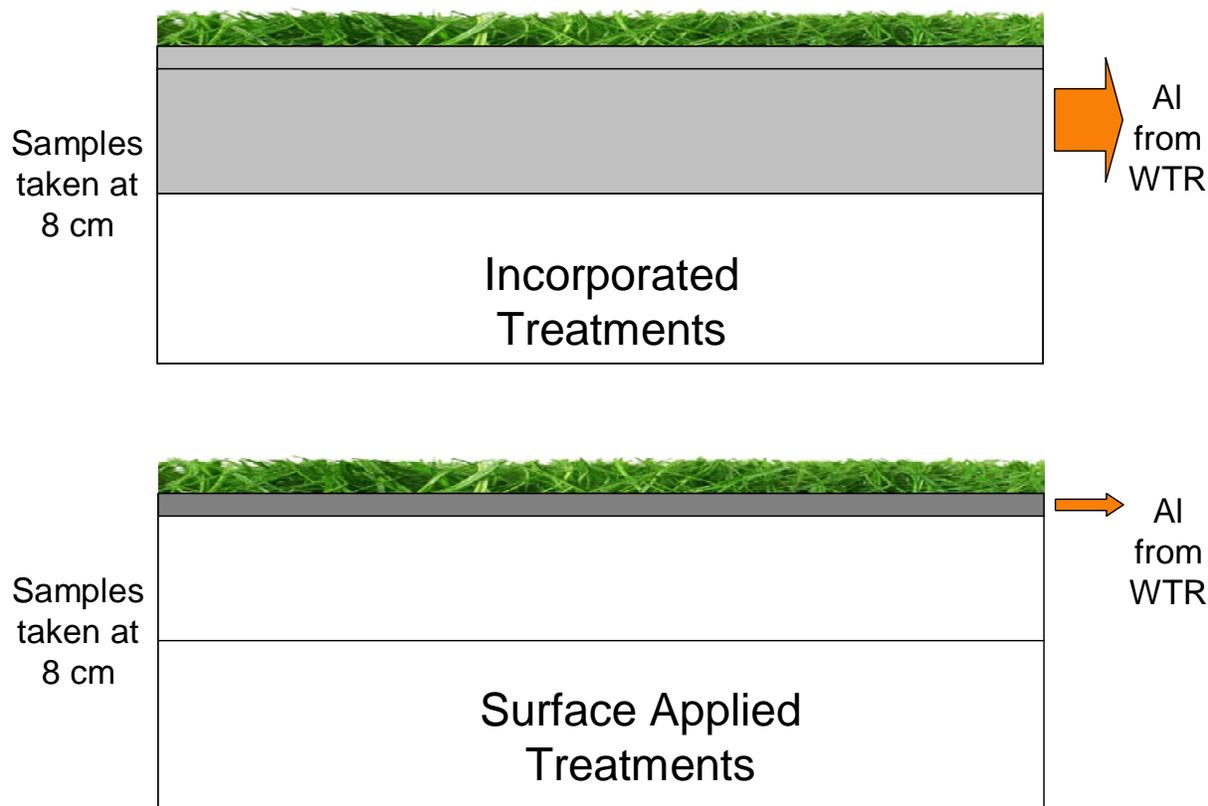


Figure 4-9. Differences in SPSC for incorporated and surface applied treatments.

Oladeji et al. (2006) also observed SPSC values in a bahiagrass field where WTR was surface applied at rates up to 2.5% dry weight by mass. Various P sources were added at N and P agronomic rates. It was found that independent of the P source or rate, SPSC increased as WTR rates increased. The SPSC values in this study were calculated using oxalate extractable concentrations. When P was applied with TSP at values greater than plant requirements which were meant to mimic manure applications ( $88 \text{ kg P ha}^{-1}$ ), the SPSC values were approximately  $20 \text{ mg kg}^{-1}$ . In comparison, the SPSC values determined from Mehlich-1 concentrations in our study showed values with an average of  $6 \text{ mg kg}^{-1}$  (3% dry weight WTR, surface applied and under high TSP additions). Considering some differences existed between the two studies, SPSC

values did not greatly vary and showed similar trends. Thus, the Mehlich-1 extractable concentrations used can give a good indication of the SPSC values.

### **Soil Nitrification Potential**

Since the microbial nitrification process in soils is sensitive to a variety of environmental factors, it was used as an indicator to evaluate any adverse effect that the AI-WTR might have on the soil microbial population. The assay used a solution of  $(\text{NH}_4)_2\text{SO}_4$  buffered at pH 7.2. Preliminary studies showed essentially no nitrate production when this test was first used. Because the soil had a relatively low initial pH (4.6), it was hypothesized that the buffered  $(\text{NH}_4)_2\text{SO}_4$  solution added to the soil did not have enough buffering capacity to overcome the low soil pH and the pH of the solution dropped to levels where microorganisms could not reach their optimum nitrification potential. This was confirmed by a subsequent test that showed the initial solution pH of 7.2 drop to 5.2 after addition of the soil. The pH of the soil was considered to be a limiting factor in this study so the assay was rerun with newly collected soil and the added solution contained a higher concentration of phosphate buffer to prevent the pH from dropping.

After increasing the buffer strength of the solution, nitrification potential increased as the application rate of WTR increased, especially in the surface applied treatments (Table 4-8 and Fig. 4-10). The interactions between WTR application method and rate were not significant.

These results may be indicative of some of the possible benefits of WTR application Ippolito et al. (2003) observed, including increased N and organic carbon availability and greater aggregate stability. Potential nitrification rates were lower in the incorporated treatments because the WTR was spread throughout the top 10-15 cm while the soil samples for this study were collected only from the top 5 cm.

Table 4-8. The p-values for the nitrification potential data using ANOVA and general mean contrast (single degree of freedom).

Sampling Date	Method	Rate	Method*Rate	Control vs. Treatments	35 Mg ha <sup>-1</sup> vs. 70 Mg ha <sup>-1</sup>
September 20, 2007	<i>&lt;.0001</i>	<i>0.0504</i>	0.1386	0.1687	<i>0.0369</i>

† Values italicized are significant at  $\alpha = 0.05$ .

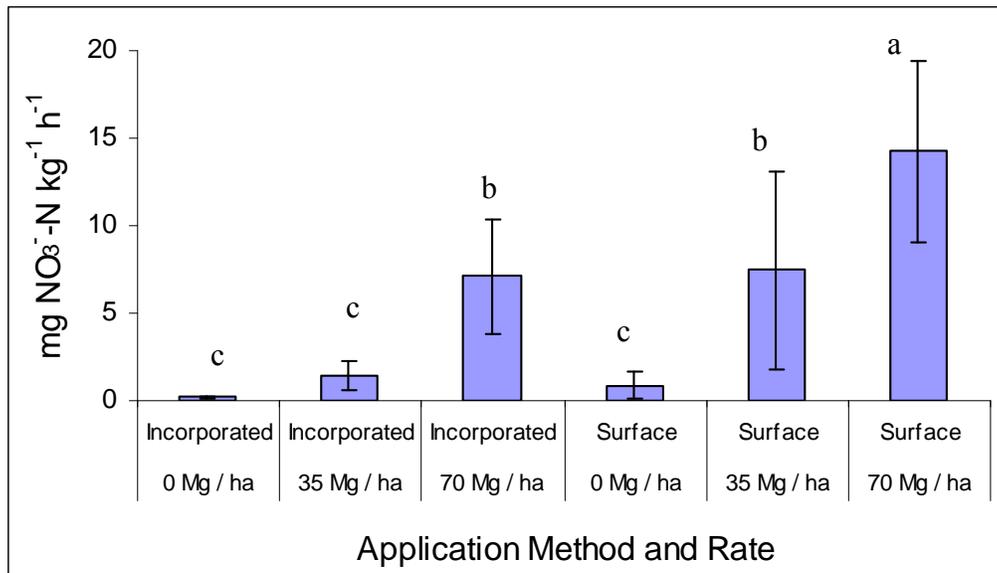


Figure 4-10. Nitrification potential of treatments for soil samples taken on September 20, 2007. † Treatments with similar letters show no significant difference by Waller-Duncan ( $p < 0.05$ ).

It is important to note that the values determined by this study are not actual field nitrification rates but rather potential nitrification values. Factors which regulate nitrification rates such as temperature,  $\text{NH}_4^+$  supply and pH were controlled for optimal results.

In a study by Pecku et al. (2006), the effects of WTRs on soil respiration and microbial community structure were evaluated. Six different WTRs were added to two soils at rates up to 500 Mg ha<sup>-1</sup>. Soil microbial respiration increased as WTR application rate increased. This increase was influenced not only by the application rate but also by the type of WTR and its chemical characteristics. Respiration rates decreased with time (10 days) indicating WTR may not enhance microbial activity for extended periods. Two WTRs had lower respiration rates when combined with the soils. The lower rates were considered to be a result of the acidic

conditions present in the soil (pH < 5.2 and 6.2). Microbial community structure was analyzed with denaturing gradient gel electrophoresis and diversity was not found to be adversely affected by WTR application. This study concluded that WTRs could be applied to these specific soils at rates up to 300 Mg ha<sup>-1</sup> without negatively impacting the microbial community.

In another study highlighting the effects of Al-WTRs on plant decomposition, C-labeled soybean leaves and stems were added to two soils amended with WTR at rates up to 1% dry weight by mass. The Al-WTR did not affect decomposition of either the plant material or soil organic matter. In comparison, an iron rich residue (not Fe-WTR) decreased decomposition rate, likely because of pH or salinity changes caused by the amendment (Sikora, 2004).

Both of the above studies showed similar results as found in our study: Al-WTRs did not cause adverse effects on the biological functions of soil as measured by soil microbial functions. These two studies were performed under laboratory conditions while the Ona study employed soil and WTR samples taken from field conditions.

### **Soluble Reactive Phosphorus in Groundwater**

Because of drought conditions during the study period, wells contained sufficient groundwater for sampling only during a period from mid-September to mid-October (Figure 4-11). Samples were obtained at two dates during this period: September 20, 2007, and October 2, 2007.

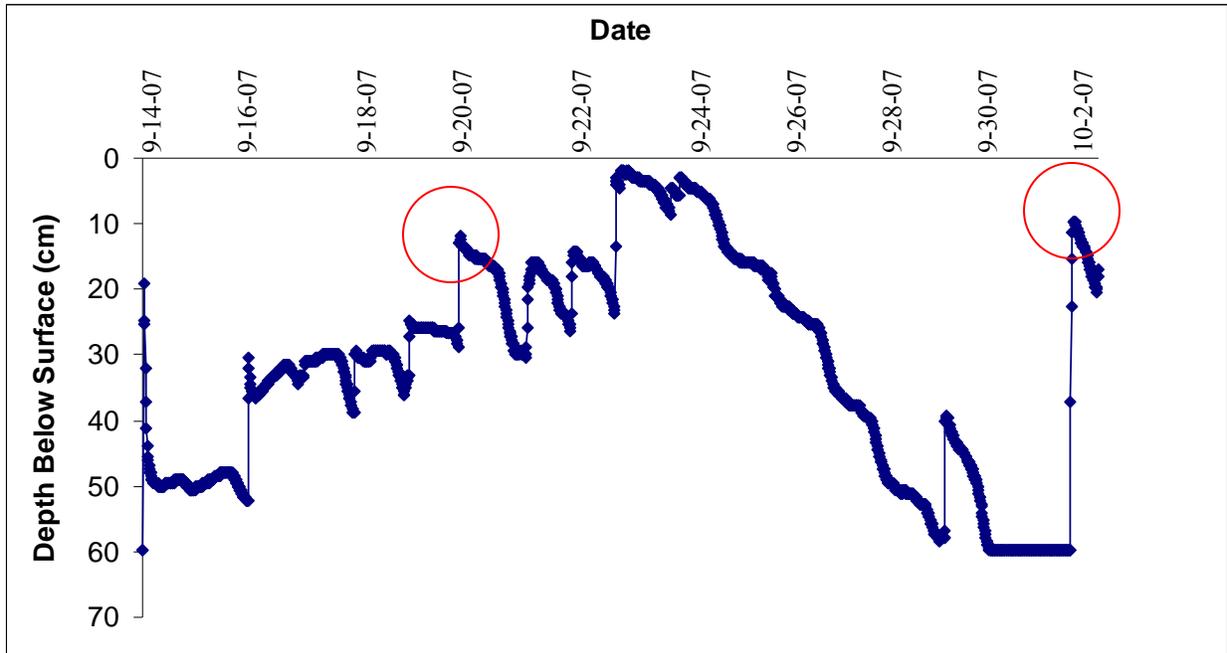


Figure 4-11. Water depth data from the pressure transducer. The ground surface is located at zero on the y axis. The two sampling dates are circled and show they were collected recently after rain events.

Many factors need to be considered when analyzing groundwater soluble reactive P (SRP) concentrations. The water table from which the samples were collected was above the spodic horizon. Nair et al. (1999) showed that lateral movement above the spodic horizon is possible and it cannot be ruled out under the field conditions present in this study. Since SRP concentrations did not change between the two sampling dates (Fig. 4-12) and there was a difference due to WTR application in the surface applied treatments (Table 4-9), it is believed that subsurface lateral flow did not cause substantial variations in groundwater SRP concentrations. When the SRP concentrations were looked at on a plot map there were no trends seen relating concentrations with subsurface movement. Sampling quickly after rain events may have helped to prevent subsurface lateral flow effects from obstructing the results from WTR application.

Triple superphosphate was added in April and groundwater samples were not collected until September. The rate of P movement between these time frames is unknown. The study procedure represents a situation in which high loads of P would be applied after WTR application. If the soil had been initially loaded with P throughout the upper soil profile, groundwater data may have given different results especially visible in the incorporated treatments.

Groundwater SRP concentrations were reduced by the surface application of WTR but were not affected when the WTR was incorporated (Figure 4-12). The standard deviation of the control surface treatment was very large in the first sampling but decreased considerably in the second sampling while not having different SRP concentrations. The second sampling gave a more stabilized and representative value of the SRP concentrations in these control treatments and was still higher than treatments with WTR. There were no differences between the two WTR application rates.

Table 4-9. The p-values for the groundwater SRP data using ANOVA and general mean contrast (single degree of freedom).

Sampling Date	Method	Rate	Method*Rate	Control vs. Treatments	35 Mg ha <sup>-1</sup> vs. 70 Mg ha <sup>-1</sup>
September 20, 2007	0.5805	0.0695	0.104	<i>0.0301</i>	0.504
October 2, 2007	0.8413	<i>0.026</i>	<i>0.0305</i>	<i>0.0123</i>	0.336

† Values italicized are significant at  $\alpha = 0.05$ .

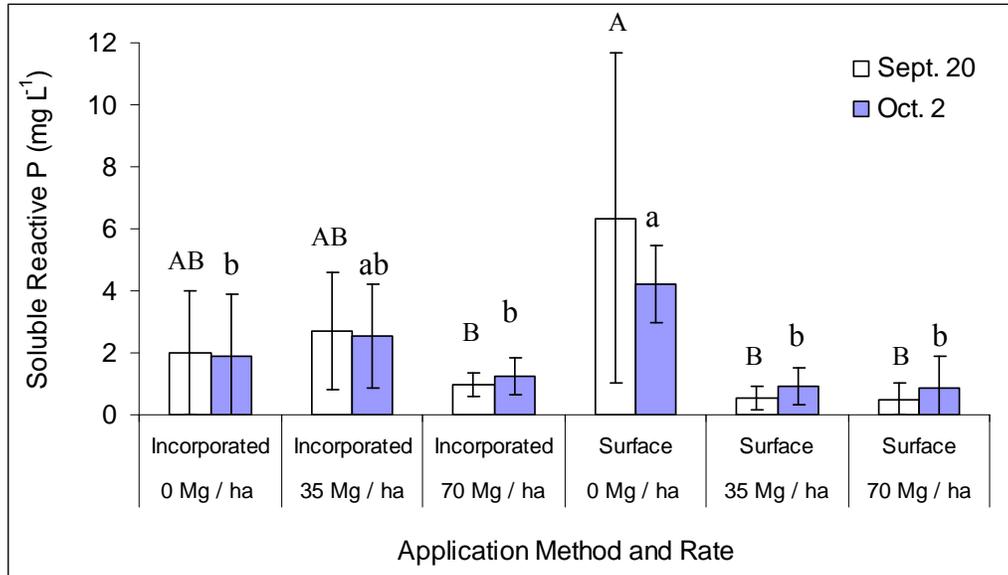


Figure 4-12. Water SRP concentrations across treatments for two sampling dates in 2007. †Treatments with similar letters and casing show no significant difference by Waller-Duncan ( $p < 0.05$ ).

The process of incorporation impacted the groundwater SRP concentrations. The surface applied control treatments had almost twice as high SRP concentrations than all of the incorporated treatments, including the incorporated control treatments. As previously mentioned, tilling destroys macropores so water movement is greater through no-till systems. Shipitalo et al. (2000) studied the leaching of anions and cations in tilled and no-till systems by using strontium bromide hexahydrate ( $\text{SrBr}_2 \cdot 6\text{H}_2\text{O}$ ) as a tracer. The  $\text{Br}^-$  was leached more quickly in the no-till systems initially but overall anion loss after 2 years was not different between tilled and no-till systems. Thus there may have been greater leaching of SRP in the surface applied treatments, possibly explaining why the surface applied control had greater concentrations than the incorporated control.

The SRP concentrations for the incorporated treatments showed no differences. The effects due to tilling seem to have prevailed over any possible effects resulting from WTR application that were seen in the surface applied treatments. However, the conditions brought

about by tilling may only be temporary. Future sampling may be needed to show how groundwater SRP is affected by WTR application. The incorporated treatments with WTR did not have lower SRP concentrations than the surface applied treatments with WTR. This is likely because the main P source was applied only at the surface and was not present throughout the soil profile.

Silveira et al. (2006) showed that by incorporating WTR in the top 15 cm of manure impacted soil, SRP concentrations in leachate were reduced more than if the WTR was incorporated in the top 7.5 cm. Control samples had SRP concentrations of 38 mg L<sup>-1</sup>, while the 7.5 and 15 cm incorporated samples had SRP concentrations of 19 and 2 mg L<sup>-1</sup> respectively. Rew (2006) observed that WTR applied on the surface better controlled P in runoff, but that incorporating WTR better controlled P in subsurface flow and leachate concentrations. Both of these studies dealt with highly impacted soils which contained large amounts of P throughout the profile due to excessive loads of manure, while the soils at the Ona study site were naturally low in P and had a P source applied to the surface.

### **Forage Yield and Nutritive Value**

The P sorbing characteristics of Al-WTRs help prevent P loss from leaching and runoff but also raise concern for P availability to plants and potential Al toxicity effects. Thus, forage yield and several forage quality parameters were closely observed to evaluate the effects of WTR application on bahiagrass.

#### **Forage Yield**

Forage was harvested in January 2007 prior to application of any treatment to evaluate uniformity of the study area. No differences across treatments in forage yield were observed before WTR was applied. Yield averaged 5.8 tons ha<sup>-1</sup> (Table 4-11).

Table 4-10. The p-values for the forage yield data using ANOVA and general mean contrast (single degree of freedom).

Month	Method	Rate	Method*Rate	Control vs. Treatments	35 Mg ha <sup>-1</sup> vs. 70 Mg ha <sup>-1</sup>
January	0.9283	0.3292	0.4687	0.1426	0.9874
May	<i>&lt;.0001</i>	0.2742	0.4541	0.136	0.5674
June	<i>&lt;.0001</i>	0.1417	0.9699	0.077	0.3685
July	0.089	0.6099	0.6195	0.3596	0.7245
August	<i>0.03</i>	0.0799	0.0578	0.1147	0.0936
September	0.9674	0.3422	0.0845	0.3745	0.2444

† Values italicized are significant at  $\alpha = 0.05$ .

†† January data was collected before any amendments were applied. May through September data was collected after WTR and TSP application.

Treatment effects were evaluated on five harvests taken during May through September, 2007. The incorporation process caused yield reduction for the first two harvests (May and June) because the grass was not completely reestablished from tilling (Table 4-11). In the subsequent harvests, there were no yield differences observed except for August when yield was greater in the incorporated than in the surface applied treatments. Differences did not occur in the previous and following months so the August forage yield differences may be an anomaly. However, it may take additional yield measurements in the following year to confirm this.

Table 4-11. Forage yield averages for incorporated and surface applied treatments.

Application Method	January	May	June	July	August	September
	----- Tons ha <sup>-1</sup> -----					
Incorporated	5.77 a	0.05 b	0.45 b	2.62 a	4.27 a	2.25 a
Surface Applied	5.74 a	0.41 a	1.44 a	2.91 a	3.90 b	2.25 a

† Treatments with similar letters show no difference for that column by Waller-Duncan ( $p < 0.05$ ).

In this field study, WTR application rates up to 3% dry weight by mass did not indicate any trends toward negatively impacting bahiagrass yield. Oladeji et al. (2006) evaluated bahiagrass yields in a glasshouse pot experiment. An AI-WTR was applied on a dry weight basis at rates of 0, 1 and 2.5% dry weight by mass to a P deficient soil fertilized at N and P based rates. Bahiagrass yields were reduced in the first harvest likely due to germination difficulties. In the second crop, yields were not affected at any WTR application rate. In a study using stargrass

grown in a P impacted soil with WTR at rates of 2.5% dry weight by mass, no effects on yield were observed (Rew, 2006).

In a study by Codling et al. (2007), corn grain yields were not adversely affected when poultry litter was mixed with an Al-WTR at rates of 0, 5.6 and 11.2 Mg ha<sup>-1</sup> and applied to plots before corn was planted. However, wheat biomass yields were impacted at certain WTR rates in a study by Codling et al. (2002). Three soils of various textures including a silt loam, fine sandy loam and sand were mixed with WTR at rates of 0, 1, 2.5 and 5% dry weight by mass. Three cycles of wheat were grown and analyzed in a greenhouse pot experiment. The fine sandy loam and sand soils showed yield reductions at the 2.5 and 5% application rates but no effects were found for the silt loam soil.

### Neutral Detergent Fiber

Neutral detergent fiber (NDF) is often used as a forage quality indicator and measures the amount of hemicellulose, cellulose and lignin in the forage. These polymers are not directly composed of P, the element of concern for plant availability with WTR application. However, observing no effect on NDF percentage does suggest that WTR application does not interfere with reactions in the plant.

Table 4-12. The p-values for the forage NDF data using ANOVA and general mean contrast (single degree of freedom).

Month	Method	Rate	Method*Rate	Control vs. Treatments	35 Mg ha <sup>-1</sup> vs. 70 Mg ha <sup>-1</sup>
January	0.9063	0.2741	0.1166	0.2185	0.2953
May	0.0553	0.6835	0.6908	0.4498	0.6787
June	<i>0.0014</i>	0.2438	0.6354	0.1529	0.3743
July	0.7035	0.9367	0.2993	0.8907	0.7426
August	<i>0.0311</i>	0.6239	0.8157	0.7243	0.3726
September	0.3016	0.7375	0.3985	0.7636	0.4787

† Values italicized are significant at  $\alpha = 0.05$ .

†† January data was collected before any amendments were applied. May through September data was collected after WTR and TSP application.

Neutral detergent fiber percentages did not change due to WTR application rate over the course of the study (Table 4-12). Values were only affected by application method in the June and August harvests. In June, the surface applied treatments had higher NDF percentages than the incorporated treatments. However, the control was not different than the treatments receiving AI-WTR suggesting that WTR was not a factor in causing the decrease in NDF. The August NDF averages for incorporated and surface applied treatments differed less than 1%, and were not different when analyzed using the Waller-Duncan test (Table 4-13). Overall, NDF was not impacted by WTR application rate.

Table 4-13. Forage NDF percentage averages for incorporated and surface applied treatments.

Application Method	January	May	June	July	August	September
	----- Neutral Detergent Fiber %-----					
Incorporated	76.18 a	68.85 a	68.15 b	70.23 a	70.03 a	73.95 a
Surface Applied	76.14 a	67.58 a	70.84 a	70.63 a	70.68 a	74.84 a

† Treatments with similar letters show no difference for that column by Waller-Duncan (p<0.05).

The NDF percentages found in this study were similar to those found for bahiagrass in other studies in Florida. A study examining bahiagrass NDF based on organic matter percentage at the RCREC in Ona, FL, showed values of 76% for both 4 and 10 week maturity periods (Arthington and Brown, 2005). Samples of coastal bermudagrass taken in October and November from dairies in the Suwanee River Valley showed NDF content ranging from 78 to 85% (Staples et al., 2003).

### Crude Protein

There were no pretreatment (January) differences among treatments in bahiagrass crude protein percentage (Tables 4-14). There were also no differences in crude protein content of forage due to WTR application rate in post-treatment harvests. There was an effect due to application method as surface applied treatments had higher percentages than the incorporated treatments in May; in June and July the incorporated treatments had higher percentages than the

surface applied treatments. In general, less mature grasses have higher crude protein levels than more mature grasses (Arthington and Brown, 2005). While values were lower in May, the less mature grasses on the incorporated treatments almost doubled their crude protein content by June, possibly explaining why values were higher than in the surface applied treatments.

Table 4-14. The p-values for the forage crude protein data using ANOVA and general mean contrast (single degree of freedom).

Month	Method	Rate	Method*Rate	Control vs. Treatments	35 Mg ha <sup>-1</sup> vs. 70 Mg ha <sup>-1</sup>
January	0.5604	0.4889	0.1951	0.4049	0.3948
May	<i>0.0001</i>	0.8384	0.9865	0.8705	0.5747
June	<i>&lt;.0001</i>	0.4749	0.6777	0.2871	0.5645
July	<i>0.0058</i>	0.982	0.5071	0.8514	1
August	0.0949	0.8471	0.7832	0.8514	0.5924
September	0.4913	0.1407	0.2947	0.0514	1

† Values italicized are significant at  $\alpha = 0.05$ .

†† January data was collected before any amendments were applied. May through September data was collected after WTR and TSP application.

Table 4-15. Crude protein percentage averages for incorporated and surface applied treatments.

Application Method	January	May	June	July	August	September
	----- Crude Protein %-----					
Incorporated	6.10 a	8.28 b	15.25 a	12.42 a	10.19 a	13.19 a
Surface Applied	6.30 a	9.85 a	13.58 b	11.13 b	9.82 a	13.06 a

† Treatments with similar letters show no difference for that column by Waller-Duncan ( $p < 0.05$ ).

The crude protein percentage of bahiagrass grown in the summer ranged from 6.8 to 10.3%. Percentages dropped as maturity times increased from 4 to 6 weeks and 6 to 8 weeks (Sollenberger et al., 2006). These values were lower than the bahiagrass values seen in our study but may be indicative of an unfertilized system.

## Tissue Nutrient Effects

### Phosphorus

The pretreatment forage harvest showed no differences in P tissue concentration (0.12 – 0.15%) across all plots. Pretreatment concentrations were very low because of the initial low soil P concentrations (See Table 4-1). Tissue P concentrations were affected by application

method for all harvests (Tables 4-16). Surface applied WTR resulted in lower tissue P concentrations than when WTR was incorporated. The surface applied treatments have nearly all of the applied WTR still present close to the ground surface where most of the bahiagrass roots are located. This higher concentration of WTR may be adsorbing P that would otherwise be available for plant uptake. Plant maturity may also partially explain why the less mature plants of the incorporated treatments showed higher tissue P. Less mature plants take up nutrients at a quicker rate than more mature plants. For instance, in sorghum when half the plant mass had been produced 60% of P, 70% of N and 80% of K had already been taken up (Vanderlip, 1993). The surface applied controls may have been affected by the unusually large amount of P applied and thus were not lower than the incorporated controls of less maturity. Application rate was also significant at each harvest. However, there were differences between the 35 and 70 Mg ha<sup>-1</sup> rates for only the July and August harvest dates.

Table 4-16. The p-values for the forage tissue P data using ANOVA and general mean contrast (single degree of freedom).

Month	Method	Rate	Method*Rate	Control vs. Treatments	35 Mg ha <sup>-1</sup> vs. 70 Mg ha <sup>-1</sup>
January	0.4676	0.7058	0.4934	0.5668	0.5519
May	<i>0.0123</i>	<i>&lt;.0001</i>	0.0743	<i>&lt;.0001</i>	0.1707
June	<i>0.0032</i>	<i>0.0002</i>	0.8144	<i>&lt;.0001</i>	0.1113
July	<i>0.0029</i>	<i>&lt;.0001</i>	<i>0.0194</i>	<i>&lt;.0001</i>	<i>0.0156</i>
August	<i>0.014</i>	<i>&lt;.0001</i>	<i>0.0004</i>	<i>&lt;.0001</i>	<i>0.0098</i>
September	<i>0.0208</i>	<i>&lt;.0001</i>	<i>0.039</i>	<i>&lt;.0001</i>	0.1068

† Values italicized are significant at  $\alpha = 0.05$ .

†† January data was collected before any amendments were applied. May through September data was collected after WTR and TSP application.

Table 4-17. Tissue P content. WTR application rate in  $\text{Mg ha}^{-1}$  is represented by 0, 35 and 70. Application treatment is represented by I (incorporation) and S (surface application).

Treatment	January	May	June	July	August	September
	-----Tissue Phosphorus %-----					
0 I	0.14 a	0.86 a	0.90 a	0.48 a	0.42 ab	0.43 a
35 I	0.13 a	0.57 b	0.63 b	0.36 ab	0.32 b	0.32 b
70 I	0.15 a	0.52 b	0.46 c	0.31 bc	0.26 c	0.29 bc
0 S	0.14 a	0.89 a	0.71 b	0.50 a	0.49 a	0.46 a
35 S	0.12 a	0.37 c	0.33 cd	0.26 bc	0.22	0.24 cd
70 S	0.12 a	0.21 d	0.22 d	0.19 c	0.17	0.20 d

† Treatments with similar letters show no difference for that column by Waller-Duncan ( $p < 0.05$ ).

Tissue P concentrations were highest during May and June and then decreased to relatively stable concentrations for the remainder of the harvests. The decreased P concentration for July and August may be due to the higher yields during this time resulting in dilution of the tissue P (Table 4-11). In addition, P concentrations in the soil solution may have decreased due to leaching as 25 cm of rainfall occurred after TSP application (FAWN). The treatment most impacted as measured by tissue P% was the surface applied 70  $\text{Mg ha}^{-1}$  treatment. Tissue P concentrations were low and relatively constant for this treatment throughout the entire study and ranged from 0.17 to 0.22%. The August average of 0.17% was just slightly above the limiting value of tissue P concentration of 0.15% (Mylavarapu et al., 2007).

Oladeji et al. (2006) observed decreasing plant P tissue concentrations with WTR application rates at 1 and 2.5% dry weight by mass when fertilizers (biosolids, manures and TSP) were applied at P agronomic rates of 44  $\text{kg P ha}^{-1}$ . However, when fertilizers were applied at N agronomic rates (P application ranged from 88-280  $\text{kg P ha}^{-1}$  depending on the N:P ratio in the source), treatments amended with WTR had higher tissue P concentrations than the controls at P agronomic rates. This suggests that under N agronomic rates, WTRs do not cause the tissue P to drop below critical values even though tissue P is lowered. These results are similar to our study where P was applied at high rates (224  $\text{kg P ha}^{-1}$ ) to a WTR amended soil and tissue P levels

decreased but did not fall below the critical value. Rew (2006) observed no differences in shoot P of stargrass when WTR was surface applied or mixed in the top 10 cm and 20 cm. Stargrass was planted in boxes after the WTR and soil were mixed.

In a study by Codling et al. (2007) with WTRs, tissue P levels for corn grain and ear leaves stayed above the minimum level needed for adequate growth ( $2 \text{ g kg}^{-1}$ ) at WTR rates of 0, 5.6 and  $11.2 \text{ Mg ha}^{-1}$ . Wheat showed deficient amounts of P ( $< 1.5 \text{ g kg}^{-1}$ ) when WTR was applied at rates of 2.5 and 5% dry weight by mass. The wheat in this study was grown in pots so deficiencies may have been exacerbated because roots may have been constricted in the pots. In the field, roots may have grown to depths below WTR application to obtain adequate P for plant tissue.

### **Aluminum**

Tissue Al for January, May and June showed concentrations below  $370 \text{ } \mu\text{g g}^{-1}$  (data not shown) which is within normal levels ( $50$  and  $3410 \text{ } \mu\text{g g}^{-1}$ ) as reported by Kabata-Pendias and Pendias (2001). Concentrations did not increase with WTR application rate and no differences occurred between treatments. Forage samples for July through September were analyzed with a different instrument than the January, May and June samples because the original instrument failed and could not be repaired in time to analyze the later samples. The substitute instrument had a detection limit of  $1.0 \text{ mg L}^{-1}$ , and the July through September samples had Al tissue concentrations below this limit.

While Al tissue concentrations do not appear to have been affected by WTR application, it is unusual for Al to be transported from the roots to the leaves so low Al concentrations in the forage may not be indicative of signs of Al stress or toxicity. Cation uptake by roots is restricted when Al toxicity is affecting plants. Thus, calcium (Ca) and magnesium (Mg) levels were also evaluated.

## Calcium and Magnesium

Table 4-18. The p-values for the forage tissue Ca and Mg data using ANOVA and general mean contrast (single degree of freedom).

Nutrient	Month	Method	Rate	Method*Rate	Control vs. Treatments	35 Mg ha <sup>-1</sup> vs. 70 Mg ha <sup>-1</sup>
Calcium	July	0.9331	<i>0.0185</i>	0.7056	<i>0.0159</i>	0.0958
	August	<i>0.0015</i>	0.2582	0.7376	0.1923	0.31
	September	<i>0.005</i>	<i>0.0472</i>	0.5771	<i>0.0153</i>	0.816
Magnesium	July	0.6282	0.7173	0.1722	0.5842	0.5539
	August	0.0657	<i>0.0304</i>	0.0668	<i>0.0163</i>	0.2267
	September	0.6684	0.8677	0.1439	0.6497	0.7927

† Values italicized are significant at  $\alpha = 0.05$ .

†† July through September data was collected after WTR and TSP application.

Tissue Ca concentrations for July through September samples showed little variation ranging from 0.34% to 0.40% (Table 4-19) but did experience some effects due to application method (August and September) and rate (July and September) (Table 4-18). Surface applied WTR treatments had slightly higher tissue Ca concentrations than incorporated treatments. Treatments amended with WTR had higher tissue Ca concentrations. Decreased Ca uptake was not observed due to WTR application. Magnesium concentration was only affected in August by application rate (Table 4-18). The surface applied control treatment was higher than all other treatments and may be an anomaly (Table 4-20). It does not appear cation uptake is being negatively affected by WTR application rate but may need to be monitored in the future.

Table 4-19. Tissue Ca percentages for July through September.

Treatment	July	August	September
	-----Tissue Ca %-----		
0 I	0.353 a	0.353 c	0.333 b
35 I	0.360 a	0.355 bc	0.365 ab
70 I	0.385 a	0.373 abc	0.368 a
0 S	0.343 a	0.383 abc	0.373 a
35 S	0.370 a	0.393 ab	0.392 a
70 S	0.388 a	0.395 a	0.385 a

† Treatments with similar letters show no difference for that column by Waller-Duncan ( $p < 0.05$ ).

Table 4-20. Tissue Mg percentages for July through September.

Application Rate	July	August	September
	-----Tissue Mg %-----		
0 Mg ha <sup>-1</sup>	0.32 a	0.35 a	0.28 a
35 Mg ha <sup>-1</sup>	0.30 a	0.33 b	0.28 a
70 Mg ha <sup>-1</sup>	0.32 a	0.32 b	0.27 a

† Treatments with similar letters show no difference for that column by Waller-Duncan (p<0.05).

Codling et al. (2007) saw no changes in leaf or grain concentrations of Al, Ca or Mg at WTR rates up to 16.8 Mg ha<sup>-1</sup>. Other minerals were also studied, including zinc, manganese, iron and potassium. Similarly, there were no impacts from WTRs on these minerals. Rew (2006) monitored stargrass tissue Al concentrations below 40 µg g<sup>-1</sup> in a rainfall simulation experiment with WTR application rates up to 2.5% dry weight by mass. The Al concentration of roots were also examined and showed no increases due to WTR. The Ca and Mg content in forage averaged over flatwoods and fresh marsh sites in South Florida was 0.19% and 0.09%, respectively (Kalmbacher and Ezenwa, 2006).

## CHAPTER 5 CONCLUSIONS

The use of Al-WTRs has been proposed as a means to help reduce P runoff and leaching from agricultural land where soils are loaded with high amounts of P or have a low capacity to retain P. Many laboratory and greenhouse studies have been conducted to evaluate the efficiency of WTRs to retain P and to ascertain the potential for Al toxicity to plants. This field study evaluated the effects on soil P retention and movement when applying an Al-WTR followed by a heavy application of P to a soil with a low capacity to retain P. High amounts of P were added in the form of triple superphosphate (TSP) to simulate high P loadings that may result from biosolid or manure application. An Al-WTR was added at 1.5 and 3.0% of the dry weight of soil to a bahiagrass pasture in South Florida. Half of the plots were disked in order to incorporate the amendment in the top 10-15 cm. Soil samples were taken from each horizon to a depth of one meter 8 months after WTR application. Soil pH, water extractable P, Mehlich-1 P, Al and Fe, and the soil P storage capacity (SPSC) were measured. The soil microbial population was monitored with a nitrification assay. Groundwater samples were collected from wells placed above the spodic horizon in each plot. Forage samples were collected every 30 days for 5 months to monitor for toxicity effects. Yield, neutral detergent fiber (NDF), crude protein and tissue analysis including P, Al, Ca and Mg content were observed.

Soil pH increased slightly with WTR application, thereby creating better conditions for bahiagrass growth and also decreasing the threat of Al toxicity. The addition of WTR reduced water extractable P concentrations in the A and E horizons. Mehlich-1 P concentrations showed no increases due to WTR application, but increased from previous conditions only because of the large amount of P added as TSP. Mehlich-1 Al concentrations and SPSC increased as WTR application rate increased. The soil's capacity to retain P increased even after heavy P loadings

which should reduce P movement through the soil. Long term studies are needed to establish the continual benefits of WTRs on soil P retention. The soil microbial population was not adversely affected as nitrification potentials increased as WTR application rate increased. The analysis of soluble reactive P (SRP) in groundwater showed a decrease in the surface applied treatments with WTR application but no changes among the incorporated treatments. Tillage seems to have affected SRP concentrations but future sampling may show results without interference from the incorporating process. Regular groundwater sampling in the future may also help to validate current results.

Forage yield, NDF and crude protein were not affected by WTR application rate for the duration of the study. Tissue analysis did not show additional uptake of Al and the uptake of Ca and Mg was not restricted, suggesting Al has not interfered with cation uptake by roots. Tissue P declined with added WTR but concentrations appear to have stabilized over the five harvests and did not drop below the critical level for bahiagrass. Again, future analysis is needed to assure tissue P does not drop any further. One of the issues associated with Al toxicity is the restriction of root growth. This study did not attempt to measure root biomass because this would have required complete removal of the bahiagrass on the study site. The field plots were preserved so future research can be continued. Decreased root growth can result in less nutrient and water uptake, affecting growth and yield. This situation has the potential to develop with time so future sampling is needed to assure toxicity effects do not present themselves.

Based on this study, the use of WTRs is recommended as an amendment to soils with low P retention capacity that have been heavily impacted with P or will receive high amounts of P, e.g., a dairy sprayfield. The WTRs have shown to lower WEP concentrations below those of untreated soil. The SPSC values have also shown an increase, even with high loads of applied P,

suggesting even more P can be retained if added to the soil. Incorporation of the WTR is recommended based on two main factors: bahiagrass tissue P concentration was less affected under these conditions and the SPSC benefited greater in the A horizon. However, the physical incorporation process is not always practical. If applying WTR to a large area, surface application may be easier and less destructive when considering extra time is needed for regrowth after disking. Surface application is still very effective at P retention, especially when the P source is being applied after WTR application. No adverse effects were observed by forage yield or nutritive value at any rate. Therefore, the 70 Mg ha<sup>-1</sup> application rate is recommended since it adds a greater amount of Al to the soil, further increasing the capacity of the soil to retain P than lower application rates. Forage yield and tissue P concentrations need to be analyzed in the future to assure toxicities do not develop with time.

The prevention of P loss from agricultural fields is essential for the health of aquatic systems. The application of Al-WTRs has the potential to achieve these goals but the quality of the crop must also be seriously regarded. Different crops may have varying sensitivities to Al in the soil or to how much tissue P is required for normal plant growth and function. Application method and rate are important factors to consider when applying WTRs. Future analysis of the soil and forage on this study site would be useful to assess long term implications of WTR usage.

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## BIOGRAPHICAL SKETCH

Julie Lauter Driscoll was born in 1984. She is the third daughter of Michael and Connie Driscoll and was born and raised in Fort Pierce, FL. Coming from a long line of Gators, she began her undergraduate degree in August 2002 at the University of Florida where she decided to major in soil and water science. She worked at the Indian River Research and Education Center in Fort Pierce for three summers where she completed an internship and an independent work study in a soil toxicology lab. It was these experiences where she discovered a desire to protect agriculture and the environment and decided to pursue her Master of Science in the summer of 2006. Julie worked to prevent phosphorus leaching from impacting surface and ground waters at a field study in Ona, FL.