

11-5-2019

Using Poultry Litter Ash as a Fertilizer Source for Bermudagrass (*Cynodon dactylon*) Establishment and Loblolly Pine (*Pinus taeda*) Plantation

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USING POULTRY LITTER ASH AS A FERTILIZER SOURCE FOR
BERMUDAGRASS (*Cynodon dactylon*) ESTABLISHMENT AND
LOBLOLLY PINE (*Pinus taeda*) PLANTATION

A Dissertation

Submitted to the Graduate Faculty of the
Louisiana State University and
Agricultural and Mechanical College
in partial fulfillment of the
requirements for the degree of
Doctor of Philosophy

in

The School of Plant, Environmental, & Soil Sciences

by
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B.S. Alcorn State University, 2013
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December 2019

Acknowledgements

There are several people that I would like to thank for their continuous support and effort towards my dissertation work. First and foremost, my major two advisors, Dr. Lewis Gaston and Dr. Jeffrey Beasley; thank you two for seeing something in me that I could not have seen for myself. Thanks for continuing to push me and believing in me when I did not want to go on. I will forever be grateful for all the knowledge and wisdom you two have instilled in me.

I would also like to personally thank Dr. Michael Blazier for always taking time out of your schedule to accommodate me and teaching me things that I had no clue I would even experience, even if it was just shooting a shotgun, you taught me; and for that I am grateful. I would also like to thank Dr. Christopher Clark for serving as a member of my committee. It has been such a pleasure and honor to attend Louisiana State University. Life takes you places that you cannot even imagine. Lastly, I would like to genuinely thank Wanda LaBlanc for your assistance with XRD and XRF, your generosity has made a big contribution to my research.

Throughout my entire doctoral program, my family and friends have been the most supportive. Mom and Dad for all the sacrifices you have made, I appreciate you more than you will ever know. To my loving and wonderful husband, thanks for being so understanding and my backbone, I couldn't have done any of this without your tough love. To my little Cayden, you have motivated me in ways that your little brain couldn't even fathom, and I thank you for that reminder to keep going every day.

Lastly, I would like to thank God because none of this would be possible without you.

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Abstract

Lower solubility of P in poultry litter ash (PLA) than conventional phosphorus (P) fertilizers such as triple superphosphate (TSP) may reduce P losses in drainage and runoff; however, lower P solubility may reduce its efficacy as a P source. A laboratory experiment was conducted to examine the effect of acidity on P release and the effect of different combustion temperatures on P solubility as a prerequisite to the following studies. The first research objective was to determine the effect of soil pH on the efficiency of PLA relative to TSP for establishment of common bermudagrass (*Cynodon dactylon* L.) and P loss on representative coastal plain soil and. In the study, yields and tissue concentrations were greater where fertilized. Yields were minimally greater with TSP but there were no consistent differences in tissue concentrations between PLA and TSP. Leaching of P was negligible regardless of P fertilizer solubility, however, P runoff losses significantly decreased TSP>PLA>control. The second research objective was to evaluate PLA compared to triple superphosphate in ratios of PLA:TSP of 0:100, 25:75; 50:50; 75:100 as a pre-plant fertilizer incorporated into a 90:10 (v/v) sand and peat mixture seeded with bermudagrass. Compared to TSP, PLA reduced P, K, Ca, and Mg leaching losses during the first two weeks after planting. As the percentage of PLA increased relative to TSP, nutrient leaching decreased incrementally. The objective of the third study was to determine whether P supplied as PLA produced an effect equal to that of TSP on loblolly pine (*Pinus taeda* L.) growth and nutrient uptake at mid-rotation of two genotypes (eastern and western) on a coastal plain soil. Effects on soil parameters and runoff P were also examined. Through 2+ years, P fertilization and source had no effect on tree growth, but foliar P concentrations were greatest with TSP, and short-term increase in stem diameter was greater for the eastern genotype. There was numerically less P in runoff with PLA than TSP, however,

variability among replicates precluded significance. Based on the data from these studies, PLA has the potential to reduce P loss and serve as an alternative to conventional P fertilizers.

Chapter 1. Literature Review

1.1. Poultry Production

The poultry industry began to expand in the mid-1930s. In fact, during the years of 1929-1936 when the United States experienced economic depression, many rural families depended on the dynamics of the poultry farm for financial assistance (Moreng & Avens, 1985). Even after these years, the poultry industry continued to provide economic stability for rural families; however development of the commercial poultry industry did not begin until the early 1940's throughout World War II (Moreng & Avens, 1985). During the war, red meat was limited but poultry meat was readily available, thus increasing consumption and leading to an overflow in production (Dicks, 2010). Without rationing and other limitations during World War II, poultry meat and eggs were quickly adapted worldwide. Worldwide consumption of poultry meat will continue to increase (Nahm, 1999) because poultry meat is favored by most societies due to its low cholesterol content and price (Bolan, et al., 2010).

The poultry industry is recognized as one of the largest and most rapidly growing agricultural systems in the world (Bolan et al., 2010). In 2017, the total farm value of poultry production in the United States was over \$42.7 billion, making the U.S. the largest poultry producing country (Ma et al., 2019). In the southeastern U.S., poultry production is one of the major agricultural industries (Paudel & McIntosh, 2005). For instance, in 2016 the state of Louisiana had approximately \$1.6 billion in combined farm revenue from poultry (Ma et al., 2019). Although, Louisiana has a small number of birds compared to other southerneastern states, the poultry industry has had a steady production increase since the 1990's (US Poultry, 2017). A single poultry operation is capable of producing half a million birds a year, and loads of waste must be properly disposed from each operation (Paudel et al.,2002; Paudel, Adhikari, & Martin Jr, 2004;

Paudel & McIntosh, 2005; MacDonald et al., 2009). Although the poultry industry has a positive economic impact, poultry waste management is a gradually increasing environmental issue (Ma et al., 2019) particularly for air (Ritz, Fairchild, & Lacy, 2004) and water (Sims & Wolf, 1994) quality.

1.2. Environmental Impacts of Poultry Waste

1.2.1. Air Quality

The release of ammonia (NH_3) from nitrogenous waste in poultry litter (Reece, Lott, & Deaton, 1980) is a major environmental concern for air quality. For instance, high concentrations of NH_3 in poultry houses negatively impact the health of animals and stockmen (Ritz, Fairchild, & Lacy, 2004). Ammonia emissions from poultry production can also contribute to ecological damage and environmental pollution, such as soil acidification and particulate pollution in the air (Kangas & Sanna, 2001), which adversely affect plant biodiversity (McGinn & Janzen, 1998) and human respiratory problems (Ritz, Fairchild, & Lacy, 2004).

There are a variety of environmental (Thompson & Meisinger, 2004; He et al., 1999; Lui, Li, & Alva, 2007) and management (Sharpe et al., 2004; Paul et al., 1998; Todd et al., 2005; Todd, Cole, & Clark, 2006) factors that affect NH_3 volatilization loss. Environmental factors that affect NH_3 loss are soil pH, soil types, temperature, wind speed, and soil moisture (Paramasivami et al., 2009). A key component to successfully managing poultry waste is good nutrient management (Nahm, 2000). One management strategy is to alter the animal diet by minimizing volatile nitrogen (N) compounds in excreta, thus minimizing the exposure to volatile N compounds (Paramasivami et al., 2009). Periodic removal of poultry litter (PL) creates a transient source of NH_3 and its subsequent reuse as a fertilizer degrades water quality.

1.2.2 Water Quality

Several studies indicate that land application of PL results in excessive accumulation of nutrients, in particular P, and non-point source pollution of water via leaching and run-off (Dutta et al., 2010; He et al., 2009; Kingery et al., 1994; Mitchell & Tu, 2006; Moore et al., 1995; Sauer & Meek, 2003; Sauer et al., 1994). Application of PL to agricultural land has been linked to eutrophication of water bodies (Mozaffari & Sims, 1994; Sharpley et al., 1996) and groundwater contamination with nitrate (Bitzer & Sims, 1988). Although an initial spike in P concentration may have little effect, long-term application of PL greatly increases the concentration of soil P, thus the capacity of the soil to retain P from PL (Hooda et al., 2000). For instance, manure application with high P content can result in saturation of P sorption sites, decreasing the P sorption capacity of soils, thus increasing the rate of P release or desorption (Labosky & Lamb, 2004). Long-term loading also increases P leaching, especially in sandy soils (high hydraulic conductivity) or where fields are artificially drained (Sims & Wolf, 1994). Sandy soils also have fewer adsorption sites, thus tend to saturate with P more easily, which increases the threat of P loss to the environment (Siemens et al., 2004).

Although soil P build-up might be avoided if a mass of P was removed in harvest equal to that applied in PL, in practice since PL is typically applied at a rate based on its N concentration. However, the ratio of P to N in PL is greater than in the harvested biomass, soil P concentration increases over time (Bolan et al., 2010). Best management practices, however, prescribe adoption of P-based rather than N-based application of PL depending on the soil P concentration and risk of off-site P loss as estimated by a P-index (which varies state-by-state; Osmond et al., 2005). The latter may recommend no further P application and active soil remediation. Once under this constraint or to avoid it, there are alternative ways to manage the excess PL generated.

Poultry producers need to adopt better management techniques that protect water quality by reducing nutrient losses from poultry litter, while making the valuable nutrients more available for plant uptake (Pote et al., 2009).

1.3. Alternatives to Land-Application of Poultry Waste

1.3.1. Use with Crops and in Forestry

Although over-application of PL raises environmental concerns, PL has been typically used as an inexpensive fertilizer to enhance soil quality and agricultural productivity of forage crops (Kingery et al., 1993; Beavis and Mott, 1996; He et al., 2009; Harmel, Haney, and Smith, 2011). Cattle production is normally an integral part of poultry production, with PL reused as fertilizer for the pasture. It is this scenario, particularly a grazed, not hay pasture, long-term application of PL may lead to excessive loss of P in runoff and drainage. Provided that there is agricultural land nearby, any excess PL can be sold off-farm. However, the wet mass and low concentration of nutrients in PL result in high transportation cost on a per nutrient mass basis, thus limiting the range of sale. Composting (Kelleher et al., 2002a), pelletizing (McMullen et al., 2005) or other means (below) whereby nutrients are concentrated decreases transportation cost, making the alternative more feasible. Since poultry production in the southern US is concentrated mostly in forest land, use of PL for forest fertilization would avoid some of the transportation cost. Although several studies have shown that, for example, fertilization of loblolly pine (*Pinus taeda*) with PL is beneficial to growth (Samuelson et al., 1999; (Friend et al., 2006; Blazier et al., 2008), tree density limits maneuverability of spreading equipment, thus also limits common use of PL for forest fertilization (Blazier et al., 2008).

1.3.2. Biomass Energy and Residual Fertilizer/Soil Amendment Materials

While composting reduces the mass of PL and concentrates nutrients, potentially usable energy is lost to the environment. Anaerobic digestion, combustion and pyrolysis are processes that directly or indirectly use PL for energy and produce nutrient-rich byproducts. Anaerobic digestion releases biogases, including CH₄, which can be used directly or purified of NH₃ and H₂S for sale, leaving a sludge or liquid residue that can be used as an agricultural fertilizer (Kelleher et al., 2002) or nutrient source for industrial biological processes (Singh, Reynolds, & Das, 2011). However, high water content of the byproduct may limit off-farm sales. Heat from combustion can be used to displace fuel for heating poultry houses and / or for power (Kelleher et al., 2002a), and leaves non-volatile nutrients concentrated in the poultry litter ash (PLA). Anoxic combustion of biomass, including PL, yields synthesis gases, principally H₂ and CO (which can be directly used or upgraded to other organics via Fisher-Tropes), bio-oils (which can be refined), and solid biochar (a high-value soil amendment; Song and Guo, 2012).

1.3.3. Composition of PLA

Furnace design and operation control combustion temperature, which in turn affects the composition of PLA. The mass of poultry litter dry matter lost during combustion increases with temperature, thus the concentrations of non-volatile elements increase; however, ratios of the residual elements vary with temperature (Faridullah et al., 2008). The greatly increased concentrations of Ca, Mg, K and especially P is the basis for research into the potential use of PLA as a liming agent (Yusiharni, Ziadi, and Gilkes, 2007) and fertilizer source of P (e.g. Codling, Chaney, and Sherwell, 2002).

Although the CaCO₃ equivalent of PLA increases simply by increasing the concentration of bases with increasing combustion temperature, the form in which the bases, particularly Ca,

exist is expected to change with temperature. At less than calcination temperature ($\sim 850^\circ\text{C}$; $\text{CaCO}_3 \rightarrow \text{CaO} + \text{CO}_2$) calcite is expected and above it, CaO. However, whereas Komiyama, Kobayashi, and Yahagi (2013) found CaCO_3 in layer manure ash produced at 700°C , it was absent from broiler litter ash. Furthermore, x-ray diffraction (XRD) data of PLA made at 800 to 1000°C showed the presence of CaCO_3 and $\text{CaC}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ but not CaO (Acharya et al., 2014). Apparently, composition of the starting material affects that of the ash but given greater solubility of the oxide than carbonate, PLA generated above 850°C might be more effective as a liming agent despite inclusive data on CaO formation. On the other hand, fusion and solidification of relatively low melting point constituents (Acharya et al., 2014) may affect dissolution kinetics should CaO form. For example, Wells (2013) reported formation of vitreous material when PL was combusted at 1000°C , however the effect, if any, on Ca solubility was not examined.

There has been more research on PLA as a P source. Soluble inorganic P and organic P in PL appear to be converted to hydroxy apatite, $\text{Ca}_5(\text{PO}_4)_3\text{OH}$ (Komiyama, Kobayashi, and Yahagi, 2013). However, Acharya et al. (2014) instead found XRD evidence for only struvite, $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$, though their elemental analysis did not show the presence of N or $\text{Mg} \geq \text{P}$. Regardless of the mineral form in PLA, solubility of P is greatly increased in acid (e.g. 1.5% H_2O - compared with 82% HCl-soluble; Codling, 2006). Therefore, efficacy as a P fertilizer source should be greater in acid soils (Codling, Chaney, & Sherwell, 2002), though it may be low relative to TSP, superphosphate or even rock phosphate (Yusiharni, Ziadi, & Gilkes, 2007).

Since PL contains appreciable quantities of trace elements (Bolan et al., 2010), these are concentrated in PLA (Faridulla et al., 2009c; Komiyama, Kobayashi, and Yahagi, 2013). Although there is a little effect of combustion temperature on H_2O -extractability of Cu, Mn, Ni,

Pb and Zn, all become more soluble in acid (Faridulla et al., 2009c). Thus, whereas reduced solubility of P from PLA may be an environmental benefit provided that it is an adequate P fertilizer source, soil conditions that favor P-availability also increase metal solubility.

1.4. Use of PLA with Agronomic and Horticultural Crops

1.4.1 Effects of PLA on Soil Chemical Parameters

Codling, Chaney, and Sherwell (2002) found that PLA, compared to KH_2PO_4 (supplemented to match PLA composition), increased soil pH, H_2O -extractable P and DPTA-extractable Cu but either did not affect or decreased Mehlich 3 P, Ca, Mg and K, and DPTA-extractable Fe, Mn and Zn in a greenhouse study with wheat (*Triticum aestivum*). Similarly, Bachmann and Eichler-Löbermann (2010) found that PLA increased bioavailable P (resin) comparable to KH_2PO_4 . Despite much higher H_2O -extractable P in PL than PLA (Codling, 2006), soil P (H_2O -extractable besides HNO_3^- , EDTA- and neutral salt-extractable) was higher when PLA rather than PL that had been incorporated (Faridulla et al., 2013). Similarly, Faridulla et al. (2009a) earlier found higher amounts of soil P extracted with Bray 1, DPTA, Mehlich 1 and Olsen with PLA than PL. In a related study with turkey manure ash, Pagliari, Strock, and Rosen (2010) found little difference been measures of soil P availability with respect to TSP.

Thus, on the basis of soil analyses, PLA would seem an adequate P source. Moreover, PLA would seem to have a net environmental benefit in that its lower P solubility should lead to reduced off-site losses, as shown by Wells et al. (2017) with significantly less P leaching compared to super phosphate used to fertilize containerized lantana (*Lantana camara*) and verbena (*Verbena canadensis*). Furthermore, despite moderately high concentration of acid-extractable trace metals in PLA (Faridulla et al., 2009c), mobility of Cu, Mn, Ni and Zn was greater with PL than PLA, perhaps due to complexation with dissolved organic matter with PL

and lower pH (Faridulla et al., 2012). Although leaching losses of metals in Faridulla et al. (2012) were greater than without PLA, Codling (2013) found no effect of As uptake by corn (*Zea mays*), soybean (*Glycine max*) and wheat compared to control, thus no indirect evidence for increased As mobility with PLA (containing 12.9 mg As kg⁻¹).

1.4.2. Effects of PLA on Nutrient Uptake and Yield

Poultry litter ash is a suitable P source for buckwheat (*Fagopyrum esculentum*; Bachmann and Eichler-Löbermann, 2010), corn (Faridulla et al., 2009a; Codling, 2013; Faridulla et al., Faridulla, Eneji, and Mahmood, 2013), Japanese mustard spinach (*Brassica rapa*; Faridulla et al., 2009b), lantana and verbena (Wells et al., 2013), oil radish (*Raphanus sativus*; Lopez et al., 2009; Bachmann and Eichler-Löbermann, 2010), peanut (*Arachis hypogaea*; Codling, Lewis, and Watts, 2015), phacelia (*Phacelia tanacetifolia*; Bachmann and Eichler-Löbermann, 2010), ryegrass (*Lolium multiflorum*; Lopez et al., 2009; Bachmann and Eichler-Löbermann, 2010), soybean (Codling, 2013) and wheat (Codling, Chaney, and Sherwell, 2002; Codling, 2013). Among these crops, responses of the grasses are probably more relevant to two of the studies later described.

Concentrations of non-N macronutrients in corn shoots were not significantly different when corn was fertilized with PLA or PL, but concentrations of Cu, Mn and Zn were greater with PLA (Faridulla et al., 2009a; 2013). However, the lower tissue concentrations of P, K, Ca and Mg largely reflected dilution into the significantly greater biomass with PLA. Codling (2013) compared uptake and yields with PLA and KH₂PO₄ and found generally no difference in shoot or root P between treatments nor consistently greater shoot or root biomass with KH₂PO₄. Lopez et al. (2009) found no difference in ryegrass tissue P, K, Ca or Mg and yields between fertilization with PLA and KH₂PO₄, nor did Bachmann and Eichler-Löbermann (2010). Wheat

tissue P was greater with PLA than KH_2PO_4 and there was no treatment effect on tissue Ca, but tissue K and Mg were greater with KH_2PO_4 (Codling, Chaney, and Sherwell, 2002).

Furthermore, P source had no effect on wheat yields. Codling (2006) later found numerically small but significantly greater wheat shoot and root tissue P with TSP than PLA but no treatment effect on either shoot or root biomass. Thus, given small to no apparent difference between soluble inorganic P and PLA with respect to P availability and grass productivity, the environmental advantage would seem to favor PLA.

1.5. Niche Uses for PLA

Poultry diet includes soluble inorganic P as a supplement due to inefficient utilization of phytate P in feed grain (e.g. Vieira, Stefanello & Sorbara, 2014). Akpe, Waibel, & Morey (1984) and Muir, Leach, Jr., & Heinrichs (1990) examined the efficiency with which turkey and poultry litter ash, respectively, could be used in place of an inorganic P supplement, and found both ash materials could be used with only ~ 20% loss in assimilation compared with soluble inorganic P.

Besides well-recognized sources of lead pollution including industrial processes, and earlier leaded gasoline and paint, total soil lead from shot / bullets at shooting ranges, dove fields, etc. may be as high as 10 g kg^{-1} (Levonmaki, Harikainen, & Kairesalo, 2006). These sites pose an ecological threat to wild birds through ingestion of shot (US EPA, 2005) for as long the shot remain at or near the soil surface before moving deeper by bioturbation, besides lasting risk to soil and water quality through Pb mobility. Pb solubility and mobility are reduced by raising soil pH as by liming, precipitation with phosphate as tertiary Pb phosphate, $\text{Pb}_3(\text{PO}_4)_2$, and especially chloropyromorphite $\text{Pb}_5(\text{PO}_4)_3\text{Cl}$ (Sposito, 1989). The efficacy of PLA as the phosphate source for precipitating Pb was examined by Hashimoto, Taki, & Sato (2009), who found it reduced solution concentrations by up to 97% in soil from a shooting range. Chromium

is also a pollutant for which abatement by a material related to PLA (fly ash from combustion of PL) has been examined (Kelleher et al., 2002b). Those authors found that the ash could adsorb up to 60 g Cr (III) kg⁻¹ from simulated wastewater.

Chapter 2. Preliminary Experiments

2.1. Introduction

Previous research has examined the effect of the temperature at which PLA is produced on P release from PLA, generally finding it greatest for about 550°C (Yusiharni, et al. 2007; Faridullah et al., 2008; Faridullah et al., 2009; Faridullah, Eneji, & Mahmood, 2013; Wells, 2013). However, given the alkalinity and Ca content of PLA (Yushiharni et al., 2007), P solubility is increased with acidity of the medium (Codling, 2006). While these generalities likely hold irrespective of the source PL from which the ash is made, differences in bedding material, management of the litter while it was in the poultry house, diet and other factors lead to wide variation in the composition of PL (Bolan et al., 2010), thus PLA composition and nutrient release. The source of the PLA used in the studies reported in this dissertation was the same as in Wells (2013), but the nutrient composition guaranteed by the supplier (North American Fertilizer, Benson, MN) is a practical time-average over various PL sources and conditions in the large-scale furnace. Visual examination of the PLA indicated substantial heterogeneity in particle size and completeness of combustion (e.g. evident unburned PL). Although the laboratory and greenhouse studies undertaken in this dissertation attempted to homogenize (grinding, sieving and mixing) a large subsample so as to best obtain comparable results, variability among the gram-scale quantities used with experimental units was likely. Even so, an attempt was made to quantify the kinetics of P release from the PLA and develop an empirical relationship describing P release as dependent on total available acidity. Release kinetics have not been reported in the literature but would be expected to affect uptake by plants, leaching and loading into surface runoff. An examination of the effect of acidity on P release was prerequisite to the study on effect of soil pH on P uptake, leaching and runoff. Although effect of

combustion temperature on P solubility has been previously studied (Yusiharni, et al. 2007; Faridullah et al., 2008; Faridullah et al., 2009; Faridullah, Eneji & Mahmood, 2013; Wells, 2013), material produced under different combustion conditions was examined in an attempt to better understand factors affecting P solubility.

2.2. Materials and Methods

2.2.1 Effect of Combustion Temperature on PLA Composition and Nutrient Solubility

Six replicates of air-dried PL (broiler) were heated in a muffle furnace from ambient to 500°C over four hours, 750°C over six hours and 1000°C over eight hours and maintained at these temperatures for two hours. Heating to 500°C was either with the furnace closed as in normal operation or partially opened to increase air supply. Mass loss during combustion was recorded, and the six PLA replicates for each condition were combined and well-mixed. The elemental composition ($Z \geq 9$) of the PLA samples was determined by x-ray fluorescence (XRF) of fusion pellets (LSU Shared Instruments Facility; SIF). Mineralogical composition was determined by powder x-ray diffraction (XRD) using Cu-K α radiation ($\lambda = 1.5406 \text{ \AA}$) at 45 kV and 40 mA, programmed for 4 to $70^\circ 2\theta$ in 0.026° steps at $0.02^\circ \text{ s}^{-1}$ (SIF). Results were interpreted using HighScore Plus software (Malvern Pananalytical, Malvern, UK; Degen et al., 2014). Three replicates of each PLA and the source PL were extracted by shaking with deionized water (2 g to 20 mL) for 1, 2, 6 and 24 h in 50 mL centrifuge tubes, filtered ($0.45 \mu\text{m}$) and the filtrate was analyzed colorimetrically for dissolved reactive P (DRP; Pote & Daniel, 2009). The filtered PLA extracts were also analyzed by inductively coupled plasma (ICP) spectrometry (ICP SPECTRO ACRCOS Model FH E12, Kleve, Germany) by the Louisiana State University Agricultural Center (LSU AgCenter) Soil Testing and Plant Analysis Laboratory (STPAL), and the pH was measured.

2.2.2 Kinetics of P Release from Test PLA

Random subsamples of PLA were combined and ground to pass a 2 mm sieve. (Chemical data on the PLA provided by the LSU AgCenter Department of Agricultural Chemistry are given in Table 1.) Triplicate samples were extracted as above except shorter (1/2 h) and longer (72 h) extraction times were included. Solution concentration of P was measured by ICP (STPAL). The time-course release of P from commercial triple superphosphate (TSP) and rock phosphate (sample courtesy of the Potash and Phosphate Institute, Norcross, GA) was also determined for comparison.

Table 2. 1. Selected macronutrient composition and CaCO₃ equivalency (CCE) of PLA.

P	K	Ca	Mg	CCE
----- g kg ⁻¹ -----				%
43.5	74.7	159.0	20.3	22.44

2.2.3. Effect of Total Acidity on P Release from Test PLA

Duplicate 1 g samples of PLA were shaken for 24 h with 20 mL solutions (HCl) of over a range of increasing total H⁺ to PLA ratios from 0 to 10 mmole H⁺ / g PLA. The final pH of each sample was measured, and an aliquot filtered at 0.45 μm for colorimetric DRP analysis.

2.2.4. Statistical and Other Analyses

The proc GLM procedure of SAS (2012) was used to determine statistically significant differences among means obtained in the combustion study, and where differences existed, Tukey was used for means separation at $P \leq 0.05$. The REG procedure was used for linear regressions.

Possible control of P release by mineral solubility was examined using PLA produced under the above conditions. Measured concentrations, [X], were converted to activities, (X), by use of the Davies model for single-ion activity coefficients,

$$\log \gamma_{\pm} = 0.5 z_{\pm} (I / [1^{\frac{1}{2}} + I^{\frac{1}{2}}] - 0.3 I) \quad [1a]$$

where γ_{\pm} is activity coefficient, z_{\pm} is ionic charge and I is ionic strength, given by

$$I = \frac{1}{2} \sum [X]_i z_i^2 \quad [1b]$$

Thus,

$$(X) = \gamma [X] \quad [2]$$

Control by dissolution / precipitation of P minerals detected by XRD was examined by taking ratios of the ion products to the corresponding solubility products, K_{sp} , of Ca-phosphates, for example with hydroxyapatite,

$$\Omega = (Ca^{2+})^5(OH^{-})(PO_4^{3-})^3 / 10^{-58.40} \quad [3]$$

where the K_{sp} s were taken from Chow (2001).

2.3. Results and Discussion

2.3.1 Effect of Combustion Temperature on PLA Composition and Nutrient Solubility

Increasing maximum combustion temperature and O₂ supply decreased final mass of the PLA (Table 2) and altered elemental (Table 3) and mineralogical composition (Table 4).

Relative to the mass at 500° C, increasing the final combustion temperature or venting at 500° C

Table 2. 2. Effects of increasing temperature and air supply on loss of poultry litter (PL) mass.

Combustion Condition	Fractional Reduction PL Mass
500 °C	0.803 D [†]
500 °C + Air	0.843 C
750 °C	0.858 B
1000 °C	0.892 A

[†] Data followed by different letters are significantly different (Tukey, $P < 0.05$).

Table 2. 3. Effects of increasing temperature (T) and air-supply on the elemental composition (Z ≥ 9) of poultry litter ash. Data exclude elements not present at $\geq 1\%$ in at least one PLA sample.

T	Na	Mg	Al	Si	P	S	Cl	K	Ca	Mn	Fe
°C	----- % -----										
500	6.49	6.41	0.60	3.33	14.20	0.33	6.11	31.51	28.85	0.85	1.57
500 [†]	6.84	7.04	0.75	3.56	15.41	5.22	1.07	27.60	28.85	0.88	1.69
750	6.94	6.50	0.77	3.32	13.76	3.61	7.50	28.98	26.06	0.79	1.48
1000	7.97	8.74	1.38	6.43	19.82	0.84	0.00	15.42	35.51	1.07	2.17

[†] Vented.

Table 2. 4. Effects of increasing temperature (T) and air-supply on minerals in poultry litter ash.

T	Amorphous	HA [†]	Other P [‡]	KCl	K ₂ SO ₄	CaCO ₃	Na ₂ SO ₄	SiO ₂	Mg ₂ SiO ₄
°C	----- % -----								
500	46.1	7.0	10.2	9.7	12.2	13.8	0.0	1.0	0.0
500 ^{††}	42.0	12.7	8.8	1.8	18.0	0.0	0.0	0.0	0.0
750	50.0	16.9	6.4	10.8	12.0	3.6	0.0	0.0	0.0
1000	44.4	15.0	0.0	0.0	3.6	0.0	29.8	0.3	6.9

[†] Hydroxyapatite, Ca₅(PO₄)₃(OH).

[‡] Ca₉FeH(PO₄)₇ at 500° C, and Ca₉MgK(PO₄)₇ at 500° C vented and 1000° C.

^{††} Vented.

decreased the mass of all elements except Al and Si (ignoring S, for which the low XRF mass at 500° C may be in error; based on the data the elemental composition for S is not in typical range in as opposed to other elements), particularly for K from 750 to 1000° C (Table 5). Thus, more

thorough combustion conditions than in the fully closed muffle furnace at 500° C resulted in net loss of macronutrients with loss in PLA mass.

Table 2. 5. Effects of increasing temperature (T) and air-supply on the elemental composition (Z \geq 9) of poultry litter ash (PLA) relative to PLA produced at 500° C in an un-vented furnace. Data exclude elements not present at \geq 1% in at least one PLA sample.

T	Na	Mg	Al	Si	P	S	Cl	K	Ca	Mn	Fe
°C	----- % -----										
500 [†]	84.0	87.5	99.6	85.2	86.5	1260.6	14.0	69.8	79.7	82.5	85.8
750	77.1	73.1	92.5	71.9	69.8	788.5	88.5	66.3	65.1	67.0	67.9
1000	67.9	75.4	127.2	106.8	77.2	140.8	0.0	27.1	68.1	69.7	76.5

[†] Vented.

Up to half of the PLA consisted of non-crystalline substances irrespective of combustion temperature, and there was general consistency in the mineralogy at < 1000° C (Table 4). In particular, hydroxyapatite, $\text{Ca}_9\text{X(II)Y(I)(PO}_4)_7$, KCl, K_2SO_4 and (generally) CaCO_3 were present, though the percentage of hydroxyapatite increased and CaCO_3 decreased with increasing temperature. Non-apatite P, KCl and CaCO_3 were absent at 1000 °C, and there was substantially less K_2SO_4 , consistent with loss of K from 750 to 1000° C (Table 5). Analysis of the diffraction scans for PL burned at 1000° C indicated that Na_2SO_4 was the dominant mineral and there was appreciable Mg_2SiO_4 . However, the presence of 29.8 % Na_2SO_4 and 3.6% K_2SO_4 implies the presence of several times the amount of S shown by the XRF data. The 500° C data also have a similar inconsistency in amounts of S present, however, the data for S (and Cl) appear more variable than for other elements (Table 3).

The solubility of P was greatly reduced compared to PL, but there was little time-dependency in P release either for PL or any of the PLA materials and increasing the temperature of combustion did not consistently reduce P solubility (Fig. 1). Rather, solubility was somewhat

greater at 750° C than 500° C but much greater at 500° C than 1000° C. Combustion at 500° C under normal furnace operating conditions or with increased air supply reduced P solubility almost an order of magnitude relative to PL. However, beyond the apparent local maximum at 750° C, solubility was reduced more than five orders of magnitude when burned at 1000° C. Concurrently, there was a large increase in Ca²⁺ release at 1000° C (Fig. 2), suggesting that precipitation of a Ca-phosphate might control P solubility. However, the ratio of ion activity product to K_{sp}, Ω, for the only crystalline P mineral present in the 1000° C PLA, hydroxyapatite, indicated super-saturation increasing to nearly 17 orders of magnitude with time (data not shown). Solubility data for the mixed Ca-phosphates, Ca₉X(II)Y(I)(PO₄)₇, were not found in a literature search, however, given the expected low solubility of Ca-phosphates with Ca / P ratios > 1 (Chow, 2001), it seems unlikely that these controlled P solubility for PLA produced at ≤ 750° C. Rather, the source of solution P, irrespective of combustion temperature, was probably the amorphous material, which by difference with mineral forms contained 77% (500° C), 73% (500° C vented), 68% (750° C) and 86% (1000° C) of the P in these materials.

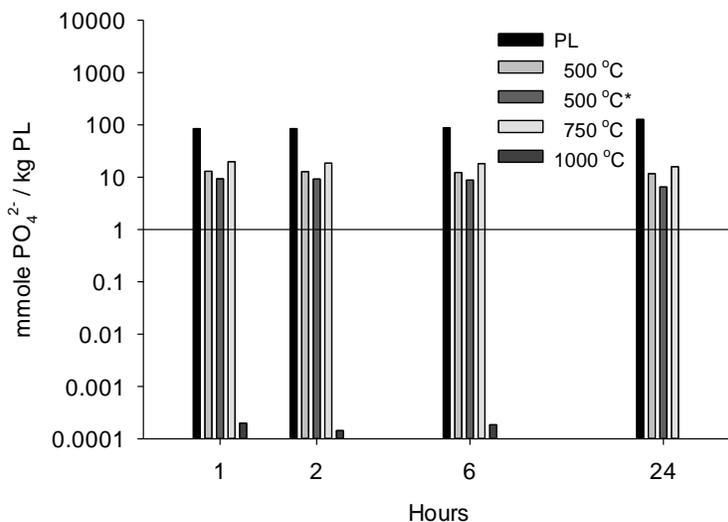


Figure 2. 1. Kinetics of P release from poultry litter (PL) and PL combusted at different temperatures expressed relative to an equivalent mass of PL. The 500° C* burn was vented.

While combustion greatly reduced P solubility relative to PL (Fig. 1), it similarly left little to almost none of the K, Ca and Mg in the PLA water-soluble. Greatest solubility of P with the 750 °C PLA was only 2.57% of the mass estimated by XRF, and at 1000 °C, essentially none. Similarly, the greatest percentage of Ca (1.45%) was released from the 1000 °C PLA (Fig. 2), and at lower temperatures only about 0.01%. The release of Mg (about 0.01%) was unaffected by combustion conditions but release of K decreased from 11.27% at 500 °C and 12.84% when vented to 2.54% at 750 °C and < 0.01% at 000 °C (data not shown).

2.3.2. Kinetics of P Release from Test PLA

Very little of the 1400 mmole P kg⁻¹ (Table 1) was released from PLA and there was no significant linear trend in release over time up to 72 h extraction (Fig. 3). The average amount of P released (0.045 mmole kg⁻¹) was only somewhat greater than released from the 1000° C PLA (0.012 mmole kg⁻¹; Fig. 1). In contrast, up to 85% of the P in TSP went into solution beyond ½ h extraction time. Although P release from rock phosphate slightly decreased with time (mmole P kg⁻¹ = 0.415 – 0.0019 hours; R² = 0.85, *P* < 0.01), its solubility was about 10-times that of PLA

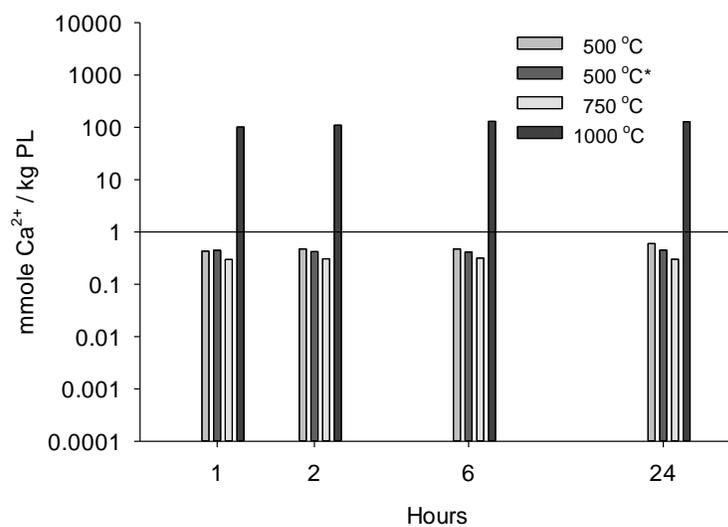


Figure 2. 2. Kinetics of Ca release from poultry litter (PL) combusted at different temperatures expressed relative to an equivalent mass of PL. The 500 °C* burn was vented.

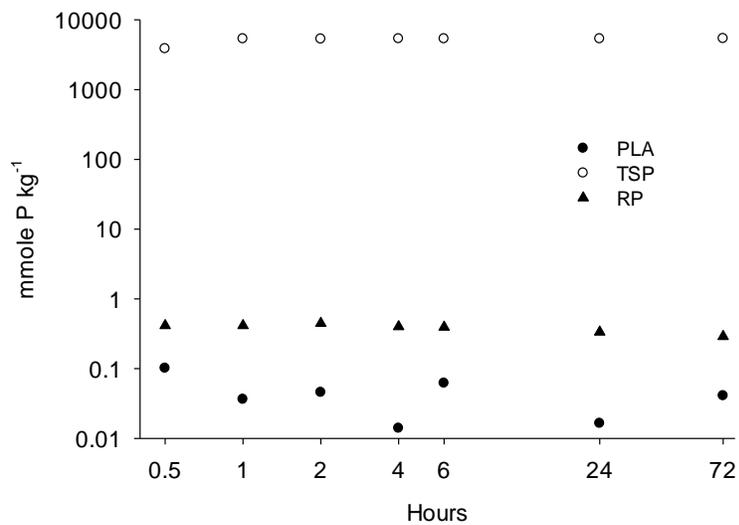


Figure 2. 3. Kinetics of P release from poultry litter ash (PLA), triple superphosphate (TSP) and rock phosphate (RP).

2.3.3. Effect of Total Acidity on P Release from Test PLA

Little P was released with increasing initial H^+ until addition of 5 mmol H^+ per g, beyond

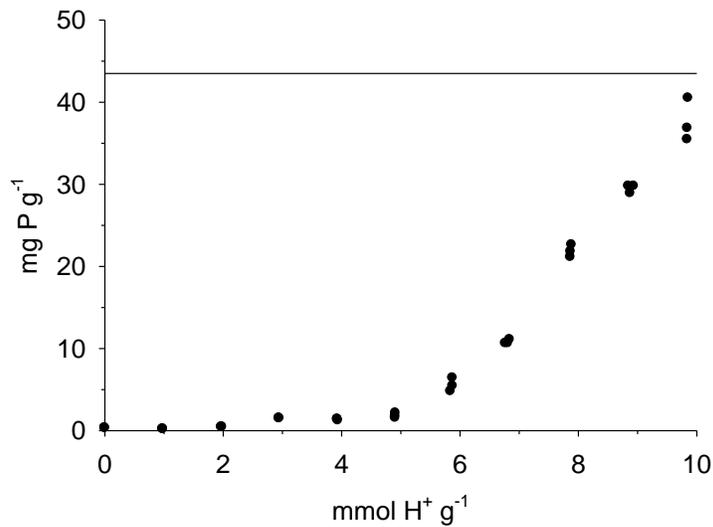


Figure 2. 4. Effect of total acidity on P release from poultry litter ash (PLA) after 24 h shaking. The straight line indicates the total amount of P in the PLA sample.

which P release increased to about 90% of the total amount present (Table 1) at 10 mmol H⁺ g⁻¹ (Fig. 4). Release of P was accompanied by almost total consumption of the added acid –the pH of the supernatant steadily decreased from ~11 at no added H⁺ to only ~2 when reacted with undiluted 0.5 M HCl. Since the CaCO₃ equivalent was only 22.4% or 2.24 mmol kg⁻¹, appreciable release of P occurred only after it was neutralized with ~5 mmol H⁺.

2.4. Conclusions

Combustion / heating PL at 1000° C, reduced the mass relative to PL somewhat more than combustion at lower temperatures, however, the 1000° C temperature presumably converted CaCO₃ to CaO (though not detected by XRD), thereby increasing the solubility and release of Ca. Heating at 1000° C reduced the solubility of P by more than five orders of magnitude relative to 750° C but as with the lower temperature PLA materials, ion products indicated super-saturation with respect to hydroxyapatite. Therefore, the source of water-soluble P was the large fraction of amorphous material present regardless of combustion

temperature or condition. However, no more than ~3% (750° C PLA) of the P in PLA was water-soluble. Except for the ~12% water-soluble K in lower temperature PLA materials, the solubility of bases was low (Ca ~1%, 1000° C PLA) to very low. Moreover, P release from commercially available PLA was even lower than from rock phosphate. Thus, reported positive responses to P fertilization with PLA equal to fertilization with soluble commercial P fertilizer (e.g. Codling, Chaney, & Sherwell, 2002) indicate that soil acidity, including in the rhizosphere, must be responsible for P release. However, an amount of total soil acidity greater than the CaCO₃ equivalent of the PLA is apparently needed to affect the release of a substantial fraction of the total P in PLA. Many horticultural/agricultural crops cannot thrive in acidic soils because of limit nutrient availability, however based on the data from this study, PLA may be a suitable fertilizer for acidic soils acting as a liming agent with the possibility of increasing the solubility of nutrients, thus making nutrients available for plant uptake.

Chapter 3. Bermudagrass Establishment on Ruston Soil

3.1. Introduction

Agricultural use of Coastal Plain soils of the southeastern and midsouth US is primarily forestry and pasture (USDA-NRCS, 2006; Karki, Goodman, & Sladden, 2009). The latter includes integrated poultry-cattle production in which poultry waste (litter; PL, a mix of bedding material such as wood shavings, spilled feed and excreta) is recycled as pasture fertilizer of reasonably high nutrient content (Bolan et al., 2010). Long-term use of PL as pasture fertilizer, however, has in some cases overly enriched the soil with N and especially P (McDowell & Sharpley 2002), such that loss to downstream water bodies is a threat to water quality (Meinikmann, Hupfer, & Lewandowski, 2015; McDowell et al., 2015). In general, however, Coastal Plain soils are natively infertile and/or were degraded by previous agriculture, so their productivity is increased by fertilization, including P.

Long-term pasture management includes renovation of low-quality and -yielding areas in which bahiagrass (*Paspalum notatum* Flüggé) or common bermudagrass (*Cynodon dactylon* L.) Pers.) is seeded or hybrid bermudagrass is sprigged in a fertilized, prepared soil surface (Newman et al., 2009; Obour et al., 2009; Silveira et al., 2013). Furthermore, where low-fertility forest is converted to pasture, fertilization is critical for successful grass establishment (Hawke, 2004). Besides commercial inorganic fertilizers, potential options include incorporation of PL (Pote et al., 2003; Nichols, Daniel, & Edwards, 1994) or poultry litter ash (PLA), the residue of combusting PL for heat and power (Polesek-Karczewska et al., 2018). Several studies have examined the use of PLA as a fertilizer source for non-volatile nutrients and found it adequate for several agronomic (Codling, Chaney, & Sherwell, 2002; Bachmann & Eichler-Lobermann, 2010) and horticultural (Wells, 2013) plants. Although PL is not currently burned for heat

and/or power in the Coastal Plain region, space-heating poultry houses might cut production costs (Costello, 2017), leaving nutrient-dense PLA as an easily transported and odor-free, marketable by-product (Habetz & Echols, 2006; Polesek-Karczewskan et al., 2018). Thus, use of PLA for pasture and forest fertilization is a future possibility, and its adequacy as a nutrient source for pasture grasses and possible environmental benefit compared to inorganic P fertilizers warrant study. The latter is based on its comparatively low P-solubility, thus reduced P mobility in the environment (Wells et al., 2017).

Similar to rock phosphate, the P-solubility of PLA is promoted by soil acidity (Yushiarni, Ziadi, & Gikes, 2007); however, since PLA is alkaline (Codling, Chaney, & Sherwell, 2002), incorporating it into an acid soil may shift soil pH toward neutral, optimizing nutrient availability (Saha, Adhikari, & Mandal, 1999) while maintaining plant-available P sufficiency. However, the effect of soil pH on the uptake of P by pasture grass from sandy soil fertilized by incorporated PLA is not known, nor are effects on P loss in leaching and runoff. Therefore, a greenhouse study was conducted to evaluate the effect of soil pH on the efficacy of PLA relative to highly-soluble triple superphosphate (TSP) in supplying P for bermudagrass establishment on a typical coastal plain soil from Louisiana, and to determine the effect of PLA relative to TSP on reducing P in leachate and runoff. Preliminary characterization of the PLA was prerequisite to design of the study.

3.2. Materials and Methods

The PLA was obtained from North American Fertilizer, Benson, MN. Results of elemental and other analyses of a well-mixed subsample by the LSU AgCenter Department of Agricultural Chemistry are given in Table 1. Inasmuch as the literature are inconsistent on mineral forms of P in PLA (Komiyama, Kobayashi, & Yahagi, 2013; Acharya et al., 2014), the

material was analyzed by powder x-ray diffraction (XRD) using Cu-K α radiation ($\lambda = 1.5406 \text{ \AA}$) at 45 kV and 40 mA, programmed for 4 to 70° 2 θ in 0.026° steps at 0.02° s⁻¹ (LSU Shared Instruments Facility). Results were interpreted using HighScore Plus software (Malvern Pananalytical, Malvern, UK; Degen et al., 2014). The effect of total acidity on release of P and bases from PLA was approximated by shaking 1 g of PLA with 20 mL of HCl (ratios ranging from 0 to 10 mmol H⁺ g⁻¹) in triplicate for 24 h, with colorimetric analysis for dissolved reactive P (DRP) in the supernatant as described by Pote & Daniel (2009) and bases determined by inductively couple plasma (ICP) optical emission spectroscopy by the LSU AgCenter Soil Testing and Plant Analysis Laboratory (STPAL).

Table 3. 1. Selected macronutrient composition and CaCO₃ equivalency (CCE) of PLA.

P	K	Ca	Mg	CCE
----- g kg ⁻¹ -----				%
43.5	74.7	159.0	20.3	22.44

The test soil was a low-fertility sandy loam Ruston (fine-loamy, siliceous, semi active, thermic Typic Paleudults). Characterization data include texture by the hydrometer method (Gee and Bauder, 1986), organic C by wet digestion (Nelson and Sommers, 1982), bases extractable with 1 N NH₄OAc (Thomas, 1982), total acidity as determined by titration of BaCl₂ + triethanolamine extracts (Thomas, 1982), oxalate-extractable Al, Fe and P (Al_{ox}, Fe_{ox} and P_{ox}; Schoumans, 2009), and pH determined at a 1:1, water to soil ratio (Table 2). All parameters were determined in triplicate except once for texture. The soil was also analyzed for Mehlich 3 extractable macronutrients by STPAL on which recommendations for bermudagrass establishment were made.

Table 3. 2. Selected properties of the Ruston soil.

Clay	Sand	OC	Ca	Mg	K	Na	Total Acidity	CEC [†]	pH	Al _{ox} +Fe _{ox}	P _{ox}
---	%	---	g kg ⁻¹				----- cmol(+) kg ⁻¹ -----			mmol kg ⁻¹	
4.8	82.7	2.3	0.64	0.35	0.02	0.01	2.12	3.14	5.80	22.0	1.0

[†] Cation exchange capacity expressed as sum of cations.

3.2.1. Design and Initiation of the Greenhouse Experiment

Containers (10 cm diameter x 20 cm depth PVC pipe fitted with end caps drilled for drain lines) were filled to 18 cm depth with 2.300 kg of the Ruston soil that had been amended with S to lower the pH to 5, amended with Ca(OH)₂ to raise the pH to 7 or without pH adjusted from initial pH ~6 (Table 2). Rates of S (0.17 g kg⁻¹) and Ca(OH)₂ (0.76 g kg⁻¹) were determined by titration with H₂SO₄ and Ca(OH)₂, respectively as per STPAL. The upper 10 cm of the soil was amended with triple superphosphate (TSP) or PLA at rates of 48 mg P per container (59.4 kg P ha⁻¹) or not amended with P fertilizer. The P rate was based on the STPAL recommendation for bermudagrass establishment on a fine sandy loam having a Mehlich 3 P concentration of 1.9 mg kg⁻¹ (rated very low). Potassium, Ca, Mg and CaCO₃ equivalency in PLA (Table 1) were matched for the TSP treatment by adding 159 mg KCl, 207 mg CaCl₂ • 2H₂O + 203 mg CaCO₃, and 75 mg MgCl₂ • 6H₂O + 43 mg MgCO₃ along with the 243 mg TSP added to each container. The K added in PLA or with TSP supplied 91% of the amount recommended by STPAL.

Four replicate containers of each soil pH x fertilizer treatment were arranged in a split-plot randomized complete block design with containers held on a wooden frame that allowed leachate to drain into capped jars (hole in lid for drain line) below. (The design was intended to control for likely variation in light and especially temperature in the no air-conditioned greenhouse.) Soil in containers was wetted from the bottom, seeded with common bermudagrass at 79 mg per container (97.6 kg ha⁻¹) in mid-June, and irrigated daily with 100 mL (1.25 cm) of

deionized water, an amount sufficient to produce drainage. Three weeks after planting (WAP) bermudagrass was fertilized with NH_4NO_3 109 mg N per container ($134.5 \text{ kg N ha}^{-1}$), and at 91 mg N per container ($112.0 \text{ kg N ha}^{-1}$) following the first of two harvests.

3.2.2. Leachate Collection and Analysis

The volume of leachate collected over each of eight two-week periods was determined by mass. Total P (TP) in leachate sample was determined by digestion with potassium persulfate + H_2SO_4 followed by colorimetric analysis (Pote, Daniel, & DeLaune, 2009). A subsample was also filtered for colorimetric determination of DRP.

3.2.3. Biomass and Tissue Concentrations

Bermudagrass was harvested at 3 cm stubble height 8 and 16 WAP and dried at 55°C for 72 hours for biomass yield. Samples were milled to pass a 0.5 mm sieve, and 0.5 g subsamples were digested in 5 ml of concentrated HNO_3 for 4 hours at 130°C and filtered (Whatman 42) before being brought to a final volume of 50 ml using deionized water. Concentration of P, K, Mg and Ca were determined by ICP (STPAL).

3.2.4. Simulated Runoff

Following the second bermudagrass harvest, containers were drilled at the soil surface, tapped and fitted with 90° plastic elbows through which surface-ponded water would drain. The frame with containers was placed 2 m below a 2HH-SS30WSQ nozzle (Spraying Systems Co., Wheaton, Illinois) connected to a deionized water supply. Tubing was run from the elbows to plastic bottles below containers through holes in caps, and simulated rain was pumped onto the set of containers at 7.6 cm h^{-1} for $\frac{1}{2}$ h. Volume of simulated runoff collected was measured by mass, and concentrations of DRP and TP determined as above.

3.2.5. Statistical Analysis

Repeated measures data (bermudagrass yield, tissue concentrations, and leachate DRP and TP) were analyzed using the MIXED procedure of SAS 9.4 (SAS Institute, Cary NC), with initial soil pH, fertilizer treatment and sampling time as fixed effects. Rep, rep x pH and rep x pH x treatment were random effects. Runoff data were also analyzed with the MIXED procedure. Means were separated following Tukey at $\alpha = 0.05$.

3.3. Results and Discussion

3.3.1. Mineralogical Composition of PLA and Acid-Induced Nutrient Release

The XRD analysis found that greater than half (53.9%) of the PLA was amorphous, and hydroxyapatite (14.8%), quartz (12.0%), calcite (9.5%), arcanite (K_2SO_4 ; 6.0%) and apthitalite ($K_{2.25}Na_{1.75}(SO_4)_2$; 4.0%) were the major crystalline forms. Compared to the elemental composition of PLA (Table 1), hydroxyapatite accounted for $\sim (27.3 \text{ g kg}^{-1} / 43.5 \text{ g kg}^{-1}) \times 100 = 63\%$ of the P present. Similarly, crystalline forms accounted for 61% and 42% of the Ca and K, respectively, but none of the Mg. Accordingly, the solubility of these PLA macronutrients likely was not controlled by mineral solubilities, especially for P and hydroxyapatite, as shown in Chapter 2 for other examples of PLA.

Rather, P solubility depends on acidic dissolution (Codling, Chaney, & Sherwell, 2002; Yushiarni, Ziadi, & Gikes, 2007). This effect is shown in Fig. 4 of Chapter 2 with little change in P release over a wide range of increasing total acidity, then sharp increase to approximately 90% total solubilization at about 10 mmol H^+ per g of PLA. Those data are extended in Fig. 1, which shows that release of Ca and Mg were also H^+ -dependent, however more gradual than P. In contrast, K was highly soluble regardless of total initial acidity. Although diffusion of acid in soil would greatly slow the kinetics of P, Ca and Mg release compared to conditions in the batch

study, total acidity in the pH-unaltered Ruston soil was about twice that necessary to effect solubilization of probably all of the P in the incorporated PLA. Specifically, $48 \text{ mg P} / (43.5 \text{ mg kg}^{-1}_{\text{PLA}}) = 1.10 \text{ g}$ of PLA (Table 1) were incorporated into 1.28 kg of the Ruston soil, thus it was exposed to 25 mmol total acidity per g in the pH-unaltered soil (Table 2). The added S would have potentially increased total acidity to 32 mmol kg^{-1} , and reaction with Ca(OH)_2 would have reduced it to about 1 mmol kg^{-1} .

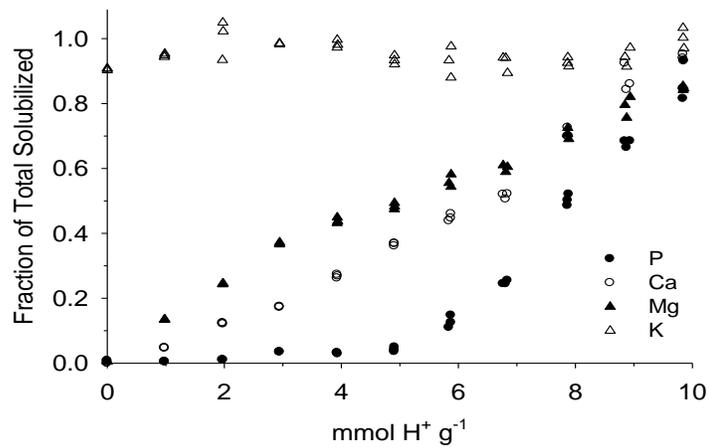


Figure 3. 1. Effect of increasing mass ratio of HCl to poultry litter ash on the release of P and bases.

3.3.2. Bermudagrass Yields and Tissue Concentrations

The presumed advantage of PLA compared to TSP is reduced P leaching and runoff due to lower solubility (Wells et al, 2017). However, the lower solubility of PLA may be inadequate to meet the nutritional needs of bermudagrass, especially at establishment (Codling, 2006; Pagliari et al., 2010) and with increasing soil pH. Although averaged over soil pH and harvests yield was significantly greater with TSP than PLA (14%; Table 3), the yield with PLA was nearly 500% greater than without fertilization. Further, there was no significant fertilizer

treatment x pH interaction and no decreasing trend in yield for PLA with increasing initial soil pH (data not shown). The latter, however, may be a consequence of some residual acidity at pH 7 and especially acidification in the rhizosphere. Late-season regrowth over two months following the first harvest was poor, resulting in a significantly lower yield.

Fertilizer increased tissue concentrations of all elements relative to control but the effects of PLA and TSP varied by element (Table 3). Tissue P was higher with TSP than PLA, reflecting the high solubility of TSP. Potassium did not differ between fertilizers, consistent with the high solubility of K in PLA (Fig. 1), but Ca and Mg were higher with PLA than TSP. Continued acid-induced release of Ca and Mg from PLA (Fig. 1) may have occurred after relatively high initial base leaching from all fertilizer treatments amended with $\text{Ca}(\text{OH})_2$, i.e. at high base saturation of the CEC. Thus, Ca and Mg in PLA may have been retained to a greater extent than in the TSP treatment. Although bases in the leachate were not measured, the parallel experiment reported in Chapter 4 found greater base leaching with TSP than PLA. Initial soil pH also had a significant effect on concentrations except for K. Numerically greatest tissue P at pH 5 but curvilinear trend (pH 5 = pH 7 > pH 6) may reflect increasing release of P from PLA with decreasing pH. Increasing Ca and Mg concentrations with pH is consistent with expected increasing availability. All tissue concentrations were significantly higher in the second harvest and affected by fertilizer treatment. The effect of harvest presumably reflects concentration into the smaller biomass from the second harvest for which uptake (= concentration x biomass) was significantly less (data not shown). All interactions significantly affected tissue P, but whereas there was no significant fertilizer x pH interaction with PLA on yield, tissue P was significantly greater for pH 5 with PLA than pH 6 and 7 (data not shown), likely due to acid-induced release (Fig. 1). Significant fertilizer x harvest interactions for K and Ca reflected numerically or

Table 3. 3. Effects of P source (none, triple superphosphate, TSP, or poultry litter ash, PLA), initial soil pH and harvest on yield and tissue concentrations of P, K, Ca and Mg in bermudagrass.

Effect	Yield	P	K	Ca	Mg
	g container ⁻¹	----- mg g ⁻¹ -----			
P Source					
None	0.17 C	0.93 C	7.02 B	5.87 B	0.86 B
TSP	1.13 A	1.73 A	9.66 B	5.66 B	0.88 B
PLA	0.99 B	1.48 B	10.35 A	7.30 A	1.33 A
Soil pH					
5	0.82	1.56 A	9.29	5.54 B	0.86 B
6	0.76	1.23 B	9.44	5.76 B	0.93 B
7	0.70	1.36 AB	8.29	7.53 A	1.28 A
Harvest					
1	1.15 A	1.07 B	8.14 B	5.86 B	0.76 B
2	0.37 B	1.69 A	9.88 A	6.69 A	1.29 A

† Numbers within a column for an effect are significantly different if followed by different letters (Tukey; $\alpha = 0.05$).

statistically greatest concentrations with PLA, together with higher concentrations in the second harvest. Since tissue Mg was greatest for PLA and for the highest initial soil pH, the fertilizer x pH interaction was significant.

Although tissue P was significantly greater with TSP than PLA, the numerical difference was small (0.25 mg g⁻¹), and even tissue P for TSP was below the range typical for bermudagrass (Mills & Jones, 1996). However, previous research on P uptake by bermudagrass from a Ruston soil showed that tissue concentrations increase with increasing soil test P (Gaston & Kover, 2015; Ryan, Gaston, & Felicien, 2016). The average concentration of tissue K for PLA, however,

approached the minimum of the typical range, and concentrations of Ca and Mg were well within it (Mills & Jones, 1996).

3.3.3. Phosphorus Leaching

Concentration and mass of DRP and TP lost in leaching were very low and not affected by either adjustment of soil pH or P source (Table 4). The only significant effect was time in the sampling sequence, for which DRP concentration and load were greater for the first than subsequent events. There were no significant interactions. Negligible P loss presumably reflects low initial P saturation ($P_{ox} / [Al_{ox} + Fe_{ox}] = 0.045$; from Table 2) of the appreciable sorption capacity ($[Al_{ox} + Fe_{ox}] = 22.0 \text{ mmol P kg}^{-1}$; Table 2; e.g. Hooda et al., 2004). Thus, leaching from the upper 10 cm to lower soil was likely small, and since the P sorption capacity of the lower soil ($1.15 \text{ kg} \times 22.0 \text{ mmol P kg}^{-1} \times 0.955 = 24.16 \text{ mmol P}$) was much greater than the total P incorporated into the upper 10 cm ($48 \text{ mg P} = 1.55 \text{ mmol P}$), negligible leaching would be expected. Thus, the hypothetical advantage of PLA relative to TSP on reducing P leaching was not demonstrated. On the other hand, P transport to surface water bodies by leaching and

Table 3. 4. Effects of P source (none, triple superphosphate, TSP, or poultry litter ash, PLA), initial soil pH and leaching event on average concentrations of dissolved reactive phosphate [DRP] and total phosphorus [TP], and DRP and TP losses in leachate.

Effect	[DRP]	[TP]	DRP Loss	TP Loss
	----- mg L ⁻¹ -----		----- mg container ⁻¹ -----	
P Source				
None	0.006	0.005	0.003	0.002
TSP	0.003	0.037	0.001	0.002
PLA	0.005	0.007	0.002	0.003
Soil pH				
5	0.006	0.009	0.002	0.004
6	0.003	0.005	0.002	0.002
7	0.004	0.035	0.002	0.002
Sample				
1	0.015 A [†]	0.016	0.006 A	0.004
2	0.000 B	0.004	0.000 B	0.003
3	0.001 B	0.001	0.000 B	0.001
4	0.001 B	0.045	0.000 B	0.003

[†] Numbers within a column for an effect are significantly different if followed by different letters (Tukey; $\alpha = 0.05$).

lateral subsurface flow is principally an environmental issue where soils are highly enriched with P and are artificially drained or have an impervious subsoil horizon. The more serious threat to water quality in other situations is P transport in runoff (He et al., 2006; Bergstrom, et al., 2015; Sims & Joern, 1998).

3.3.4. Phosphorus in Simulated Runoff

Concentrations and loads of DRP and TP in the simulated runoff experiment increased 0 P < PLA < TSP, with values for TSP about 3-times greater than for PLA (Table 5). Despite

numerically least concentration and load for initial soil pH 7, high variability among replicates precluded a significant pH effect. The only significant P source x initial pH interaction was for load of DRP, which increased with pH for TSP but decreased for no P fertilization, such inconsistency suggesting that the effects were not meaningful. Previous runoff studies have shown high DRP concentrations associated with application of water-soluble P fertilizers (Kleinman et al., 2002), including significantly higher concentrations in runoff P with superphosphate compared with less water-soluble fertilizers (Weatherley et al., 2011).

Similarly, Roberts & Israel (2017) found that loss of P in runoff during simulated rainfall onto boxes of coastal plain soil was about 10-fold greater with surface-applied diammonium phosphate than PL. The effect of P solubility on P concentration in runoff also occurs when the fertilizer is incorporated into the soil, however concentrations in runoff are substantially reduced (Wang et al., 2013). Therefore, when a highly soluble P source is incorporated into the soil, its dissolution apparently loads adsorbent surfaces with a greater amount of desorbable P than does a less-soluble P source as suggested by DRP concentrations in Table 5 and Wang et al. (2013). However, solubility may have little effect on the loss of particulate P –the numerical difference between concentrations of TP and DRP for PLA and TSP was comparable and there was no difference in particulate P loads (Table 5), similar to the results of Wang et al. (2013). Thus, the benefit of PLA on reducing P loss in runoff (71% compared to TSP) is in reduced loss of the more bioavailable DRP.

Table 3. 5. Effects of P source (none, triple superphosphate, TSP, or poultry litter ash, PLA), and initial soil pH on average concentrations of dissolved reactive phosphate [DRP] and total phosphorus [TP], and DRP and TP loads in runoff.

Effect	[DRP]	[TP]	DRP Loss	TP Loss
	----- mg L ⁻¹ -----		----- kg ha ⁻¹ -----	
P Source				
None	0.055 C [†]	0.120 C	0.011 C	0.023 C
TSP	2.285 A	2.359 A	0.424 A	0.446 A
PLA	0.726 B	0.874 B	0.108 B	0.130 B
Soil pH				
5	1.068	1.254	0.217	0.248
6	1.132	1.239	0.185	0.202
7	0.866	0.862	0.140	0.148

[†] Numbers within a column for an effect are significantly different if followed by different letters (Tukey; $\alpha = 0.05$).

3.4. Conclusions

Although bermudagrass yield and tissue P concentrations were significantly greater with TSP than PLA, absolute differences were numerically small. Furthermore, PLA increased yield about 500% compared to no fertilization, and tissue concentrations of K, Ca and Mg for fertilization with PLA were numerically or statistically greater than fertilization with TSP supplemented with KCl, and Ca and Mg carbonates and chlorides. Tissue concentrations of K, Ca and Mg approached or attained typical values only with PLA. Thus, PLA is apparently comparable to commercial fertilizer carriers as a nutrient source for the establishment of bermudagrass on a low fertility soil despite its lower solubility, especially of P. No advantage of PLA compared to TSP was seen with P leaching, reflecting high P retention by the Ruston soil. On the other hand, the effect of lower P solubility with PLA was seen in the simulated runoff study –although P runoff was nearly an order of magnitude greater for soil fertilized with PLA

than without P fertilization, P loss with PLA was < 30% of that with TSP. Thus, PLA with respect to TSP had neutral effects on bermudagrass and P leaching but greatly reduced P runoff loss. These tentative conclusions must be confirmed in field plot studies on bermudagrass establishment.

Chapter 4. Incorporating Poultry Litter Ash as a Pre-plant Fertilizer to Reduce Nutrient Leaching during Bermudagrass Establishment

4.1. Introduction

Adjustments in soil fertility using pre-plant fertilizers to supply nutrients such as P are especially important during turfgrass propagation (Chang, Xiong, and Deying 2014; Soldat and Petrovic 2008) to accelerate growth (Randall and Hoelt 1988) and establishment. Increasing the initial P concentration in the soil stimulates root development for robust root growth versus delaying P fertilization (Kristoffersen, Bakkegard, and Hoel 2005) that can result in thinning swards (Kussow 2007). However, managing P fertility is particularly challenging when establishing turfgrass in coarse-textured soils that have low cation exchange capacity (CEC), low water holding capacity (Pathan, Aylmore, and Colmer 2003), and high hydraulic conductivity. Under these conditions, nutrients are prone to leaching below the developing turfgrass root zone, especially in areas that receive high irrigation, annual rainfall, or both.

Incorporating water-soluble nutrient sources as pre-plant fertilizers increases nutrient leaching. For example, Shuman (2002) reported that in sand-based golf greens dissolved P concentrations in leachate increased from 0.5 to 1 mg L⁻¹ due to the application of soluble inorganic fertilizers. Although environmental factors greatly affect nutrient uptake and movement (Kristoffersen, Bakkegard, and Hoel 2005; Mahler 1985), using slowly available P sources can be an effective practice for increasing nutrient retention.

¹ This chapter was previously published as Crystal Vance, "Incorporating Poultry Litter Ash as a Pre-plant fertilizer to Reduce Nutrient Leaching during Bermudagrass Establishment," *Communications in Soil Science and Plant Analysis* 50 (2019): 2190-2197. Reprinted by permission of Taylor and Francis, LLC

Suitable slow-release fertilizer sources must provide adequate amounts of readily available nutrients that correspond to plant requirements to support growth. One slowly available and renewable P source is poultry litter ash (PLA), a by-product of poultry litter (PL; a combination of bedding material, spilled feed, and excreta) combustion that has been used to generate heat and power (Kelleher et al. 2002; Martin and Lefcort 2002). During the combustion process, non-volatile nutrients, in particular P, are more concentrated in PLA as opposed to actual poultry litter (Bolan et al. 2010). Also, combusting PL decreases biomass > 80%, and reduces water-soluble P to 1.5% in PLA from >50% in PL (Wells et al. 2013). Since poultry production in the southeastern United States generates more than 20 million metric tons of PL per year (Feng et al. 2015), there is abundant raw material for adoption of this process in the region.

Compared to water-soluble nutrient sources commonly utilized as pre-plant fertilizers, PLA may be a suitable P source with the added benefit of limiting nutrient leaching. Application of PLA has been shown to be an effective P source for other horticultural (Faridullah et al. 2009; Wells et al. 2013) and agronomic crops (Codling et al. 2002). Establishing turfgrass in coarse-textured soils raises agronomic and environmental concerns regarding nutrient availability and movement. Therefore, the objective of this study was to evaluate PLA as pre-plant fertilizer source during bermudagrass (*Cynodon dactylon* L.) establishment as a best management practice on coarse-textured soils.

4.2. Materials and Methods

4.2.1. Soil Mixture, and Experimental Treatments, Design and General Protocol

A six-week greenhouse study to evaluate PLA as a fertilizer source for bermudagrass establishment in sand was initiated in February 2018 and repeated in April 2018 at the Louisiana State University Hill Farm, Baton Rouge, LA. The substrate was 90:10 (v/v) sand:peat moss.

The pH of the mixture was determined using a 1:2 solid to water ratio, exchangeable bases by extraction with neutral 1 N NH₄OAc with analysis by ICP (Thomas 1982), total acidity by reaction with BaCl₂ + triethanolamine and titration (Thomas 1982) and saturated hydraulic conductivity by constant head (Klute and Dirksen 1986). These data are given in Table 1.

Ten-cm diameter PVC sections were filled to 20 cm depth with 2,725g of the mixture, the upper 10 cm of which had been uniformly mixed with P at a nominal surface application rate of 59 mg P dm⁻² (or 59 kg ha⁻¹). Poultry litter ash (PLA; 4.4% P) and/or triple superphosphate (TSP; 19.4% P) in PLA:TSP P ratios of 0:100, 25:75, 50:50, 75:25, 100:0, i.e. 1,094 mg PLA: 0 mg TSP to 0 mg PLA: 243 mg TSP per container. . The P fertilization rate was based on the recommendation from the Louisiana State University Soil and Plant Testing Laboratory (LSU STPAL) for bermudagrass on loamy sand. . Since the PLA also contained 7.5% K, 15.9% Ca, and 2.0% Mg, and had a CaCO₃ equivalent of 24.4% (analysis by the LSU AgCenter Department of Agricultural Chemistry), and TSP contained 15.0% Ca, 156 mg KCl, 203 mg CaCO₃, 43 mg MgCO₃, 207 g CaCl₂·2H₂O and 75 mg MgCl₂ · 6H₂O per 253 mg TSP were added to balance nutrient composition among the treatments.

Table 4. 1.Exchangeable bases, total acidity, cation exchange capacity (CEC), pH and saturated hydraulic conductivity (K_{Sat}) of the sand-peat mixture. Parameters were determined in triplicate.

Ca [†]	Mg	K	Na	Total Acidity [‡]	CEC ^{††}	pH	K _{Sat} ^{‡‡}
----- cmol(+) kg ⁻¹ -----							cm min ⁻¹
0.05	0.03	0.04	0.01	0.93	1.06	5.6	2.9

[†] Neutral 1 N NH₄OAc; analysis by ICP

[‡] BaCl₂ + triethanolamine

^{††} Sum of cations

^{‡‡} Constant head method

Three replicates of each treatment were arranged in a completely randomized design, with containers held in a wooden frame to allow leachate to flow by gravity into glass jars located below each container. Containers were seeded at 98 mg dm^{-2} with bermudagrass ‘Sahara’ and irrigated daily for the first five weeks to replace 100% evapotranspiration (ET) loss based on the weight loss of three designated containers, and every other day for the final week. Once a week 100 mL of water above ET losses were applied to each container to ensure that leaching would occur. Beginning two weeks after planting (WAP) bermudagrass was fertilized weekly with N at 12.2 mg dm^{-2} with NH_4NO_3 .

4.2.2. Bermudagrass Growth and Leachate Analyses

Bermudagrass percent visual coverage was recorded at three and six WAP. At the end of six weeks, plants were harvested, and shoots and roots were separated. Roots were washed free of sand and peat particles, and shoots and roots were dried at 55°C for 72 hours before biomass was recorded. Dry shoot tissue samples were milled to pass a 0.05- mm screen, and 0.5-g subsamples were analyzed for nutrient concentrations. Tissue subsamples were digested in 5 mL of concentrated nitric acid for 4 hours at 130°C and filtered (Whatman 42) before being brought to a final volume of 50 mL (Havlin and Soltanpour 1980). Tissue concentrations of P, K, Ca, and Mg were determined using inductively coupled plasma (ICP) optical emission spectroscopy (STPAL). Leachate samples of nutrients were collected weekly, and volumes were recorded. Subsamples were analyzed for P, K, Ca, and Mg by ICP (STPAL) with weekly and cumulative mass losses for each nutrient calculated.

4.2.3 Statistical Analysis

Treatment containers were completely randomized. All data were analyzed using the MIXED procedure of SAS 9.4 (Littell et al. 1996 for the fixed effect of treatment. Experimental

run and treatment replicate were random variables. Means were separated following Tukey's HSD post-hoc procedure at $P = 0.05$.

4.3. Results and Discussion

Phosphorus is required for maintaining healthy turfgrass and is essential for the growth and development of turfgrass seedlings (Carrow and Rieke 1972). Phosphorus fertilizer therefore is recommended as a pre-plant fertilizer (Horgan and Rosen 2010) to accelerate growth of germinated seeds (Chang, Jin, and Li 2014; Soldat and Petrovic 2008). Following the STPAL recommendation for P resulted in bermudagrass groundcover increasing from 28 to 83% between three and six WAP with no differences among treatments at either date. Treatment also had no effect on shoot, root, and total plant biomass at the end of the six weeks, although all tended to numerically increase as the percentage of PLA increased relative to TSP (Table 2).

The similarity in bermudagrass growth between P sources proved the initial concern that the less water-soluble PLA-P could limit bermudagrass growth due to restricted availability of P was unfounded. Availability of nutrients from PLA may be similar to fertilization with insoluble P sources such as rock phosphate, in which soil and especially rhizosphere acidity promotes P dissolution for increased plant uptake (Khasawneh and Doll 1978; Yusharni, Ziadi, and Gilkes 2007;). Given the positive growth response of bermudagrass to P fertility and lack of differences in plant growth among treatments, PLA alone was an effective P source as has been reported for wheat (Codling et al. 2002), Japanese mustard spinach (*Brassica rapa* L.) (Faridullah et al. 2009), and lantana (*Lanata camera* L.) (Wells et al. 2017). Yet, tissue concentrations of P, K, and Ca were consistently below the reported typical ranges for common bermudagrass (Table 3) (Mills and Jones 1996) for all treatments. Only Mg tissue concentrations were within the typical range.

Deficiencies in P can lead to poor seed emergence and vigor (Christians 1996) that if not corrected through supplemental fertilization can cause stunted growth, thinning turf, and poor establishment (Tisdale et al. 1993). However, at no time during the experiments did bermudagrass exhibit poor growth or leaf purpling, a common symptom of P deficient plants (Taiz and Zeigler 2003). Colding, Chaney, and Sherwell (2002) found a similar response when evaluating PLA. They reported that wheat fertilized with PLA had lower P tissue concentrations but yield was unaffected compared to wheat fertilized with more readily available potassium phosphate. Bermudagrass might have benefited from subsequent applications of P, K, Ca, and Mg, but the focus of the study was to evaluate the use of PLA as a pre-plant fertilizer during the initial weeks of establishment, a time prone to nutrient loss from higher irrigation and greater drainage.

Table 4. 2. No effect of increasing the percentage of poultry litter ash (PLA) relative to triple superphosphate (TSP) on bermudagrass biomass. The pre-plant P rate was 135 kg P₂O₅ ha⁻¹.

Treatment	Shoot	Root	Total Biomass	Root:Shoot
	----- g -----			
0:100 PLA:TSP	5.28 A [†]	2.75 A	8.02 A	0.53 A
25:75 PLA:TSP	5.99 A	3.12 A	9.09 A	0.50 A
50:50 PLA:TSP	5.85 A	2.81 A	8.65 A	0.46 A
75:25 PLA:TSP	6.18 A	4.58 A	10.76 A	0.68 A
100:0 PLA:TSP	6.89 A	4.41 A	11.03 A	0.56 A

[†] Means within a column not followed by the same letter are significantly different at $P \leq 0.05$ using Tukey's HSD.

Table 4. 3. Effect of increasing the percentage of poultry litter ash (PLA) relative to triple superphosphate (TSP) on bermudagrass tissue concentrations at six weeks after planting. The pre-plant P rate was 135 kg P₂O₅ ha⁻¹.

Treatment	P	K	Ca	Mg
	----- % -----			
0:100 PLA:TSP	0.106 A [†]	0.402 A	0.260 A	0.262 A
25:75 PLA:TSP	0.125 A	0.344 B	0.267 A	0.233 A
50:50 PLA:TSP	0.136 A	0.365 AB	0.274 A	0.234 A
75:25 PLA:TSP	0.113 A	0.303 AB	0.247 A	0.216 A
100:0 PLA:TSP	0.137 A	0.439 AB	0.280 A	0.262 A

[†] Means within a column not followed by the same letter are significantly different at $P \leq 0.05$ using Tukey's HSD.

The clear benefit of PLA is reduced nutrient leaching with each 25% increase in PLA relative to TSP, nutrient leaching was incrementally reduced (Fig. 1). Leaching was greatest during the first two weeks and declined sharply thereafter for all fertilizer treatments. Whereas K, Ca, and Mg losses tended to consistently decrease over time with increasing leaching, P losses peaked with the second leaching event, reflecting sorption of phosphate in the sand-peat mixture below the depth of incorporation but limited retention of the bases due to low CEC (Table 1).

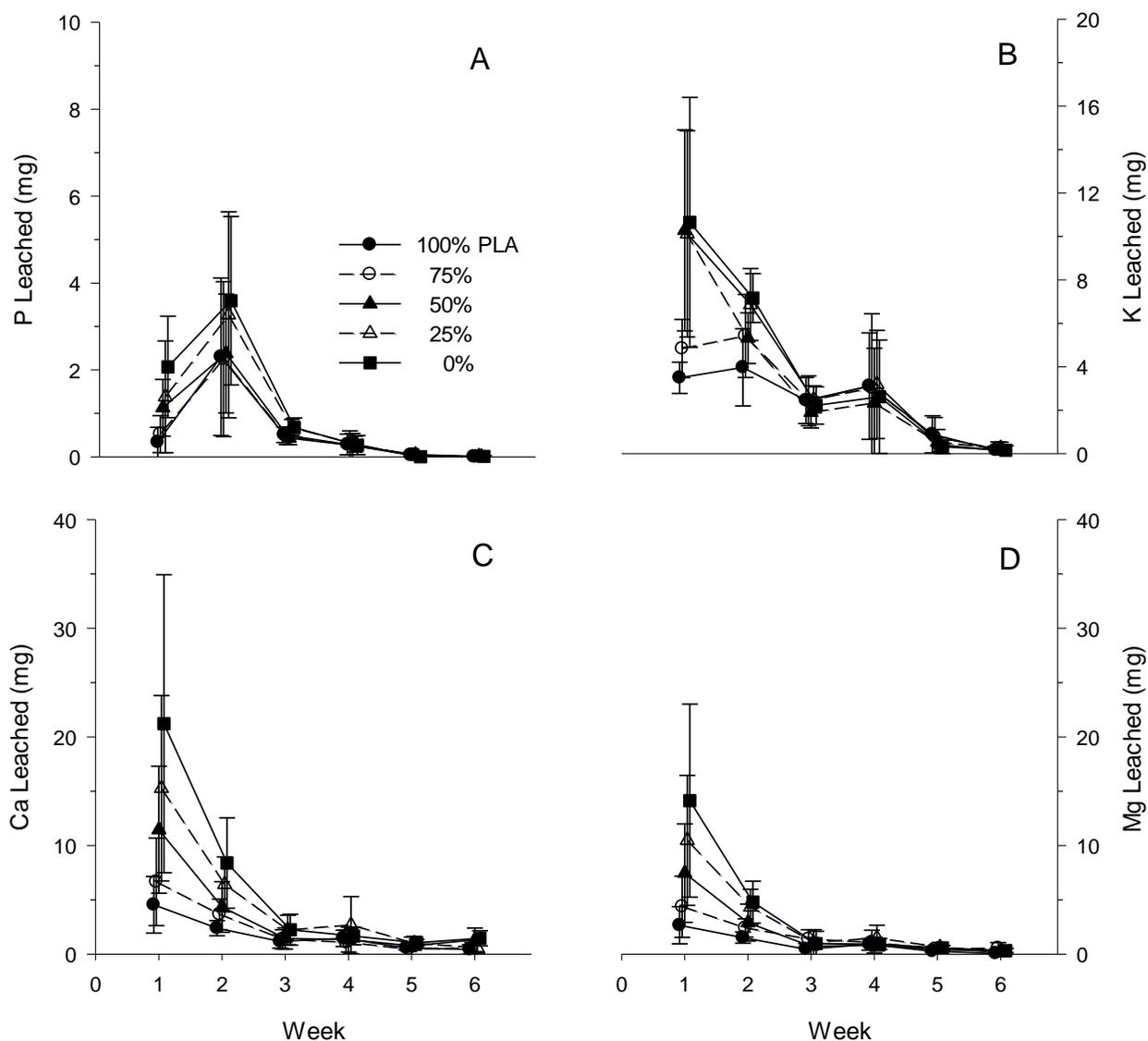


Figure 4. 1. Effect of increasing the percentage of poultry litter ash (PLA) relative to triple superphosphate on average mass of A) phosphorus, B) potassium, C) calcium and D) magnesium lost per weekly leaching event. Data are displaced along week axis for better clarity. Standard deviations are shown. The pre-plant P rate was 135 kg P₂O₅ ha⁻¹.

High leaching losses of nutrients following fertilizer application are common in many horticultural production systems (Cookson, Rowarth, and Cameron 2000) due to the application of highly water-soluble sources and low adsorption capacity of the medium (Nelson, Parsons, and Mikkelsen 2005; Sims, Simard, and Joern 1998). Using a pre-plant fertilizer composed of

75 to 100% PLA in the soil mixture not only curbed nutrient losses the first two weeks during bermudagrass establishment but decreased cumulative P, K, Ca, and Mg leaching > 46%, 27%, 61% and 60%, respectively, versus 100% TSP over the six-week experimental period (Fig. 2). These findings were similar to Wells et al. (2013), who also reported reduced P leaching for PLA compared to superphosphate from containerized lantana (*Lantana camera* L.) when evaluating ratios of the two fertilizers.

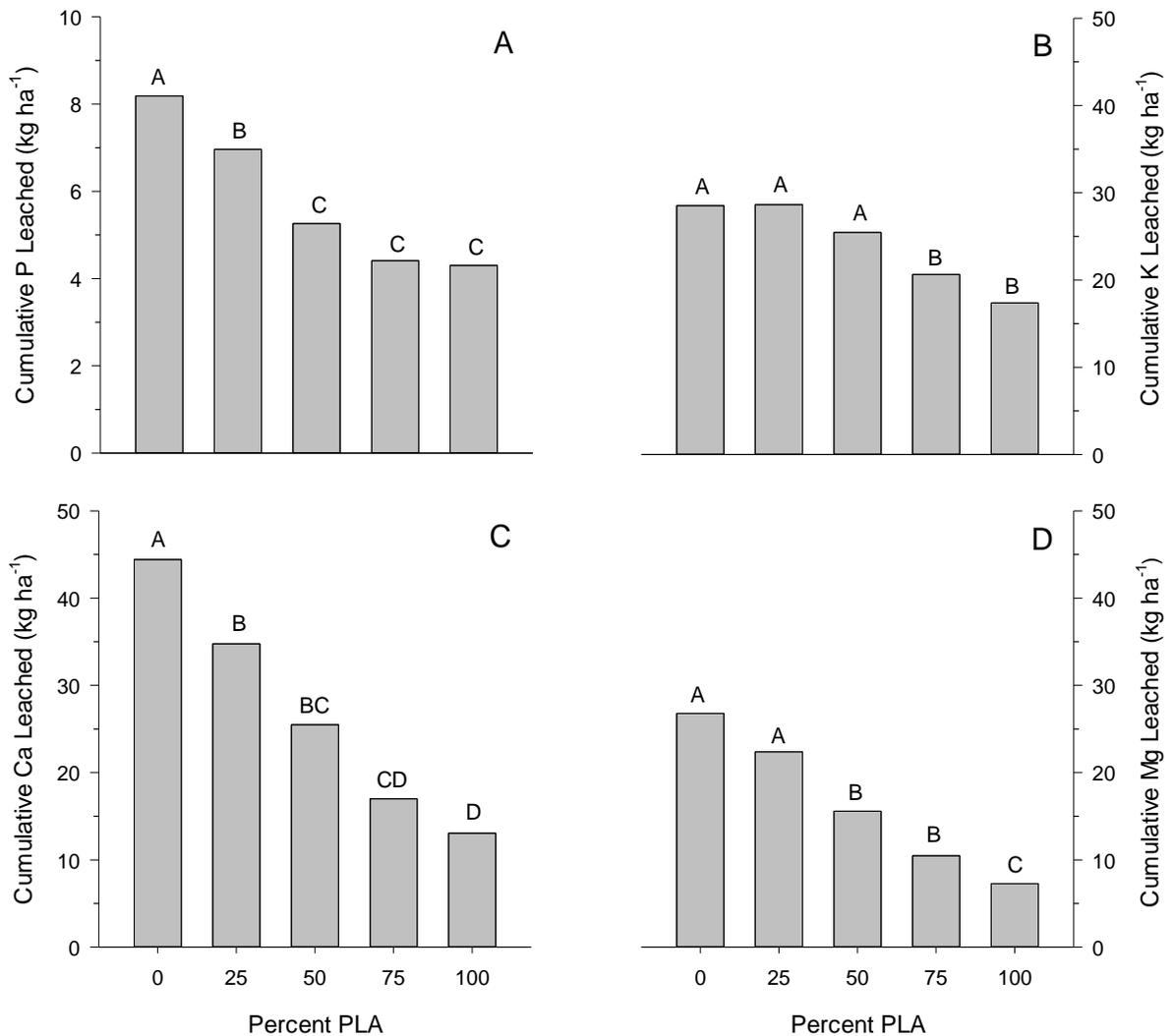


Figure 4. 2. Effect of increasing the percentage of poultry litter ash (PLA) relative to triple superphosphate on mean cumulative leaching loss of A) phosphorus, B) potassium, C) calcium and D) magnesium. Means within data for an element not shown with the same letter are significantly different at $P \leq 0.05$ using Tukey's HSD. The pre-plant P rate was $135 \text{ kg P}_2\text{O}_5 \text{ ha}^{-1}$.

High hydraulic conductivity and low sorption capacity greatly limit nutrient retention (Soldat and Petrovic 2008) in a sand-peat medium. However, greater water solubilities of TSP and K, Ca and Mg chlorides compared to PLA, given the limited root development and nutrient uptake of seedlings during the first two WAP, was the primary factor affecting nutrient leaching.

With respect to P, only 1.5% of the total P in PLA is water-soluble (Wells 2013) as opposed to > 80% in TSP (Schulte and Kelling 1996). Combustion of PL at high temperatures results in formation of water-insoluble di- and tri-calcium phosphates (Wazer 1958) and appears to reduce the solubility and mobility of the other macronutrients. As bermudagrass develops a more robust root system, differences in nutrient leaching losses due to fertilizer solubility would presumably be reduced through increased plant interception and uptake (Bowman et al. 1998).

Reducing early nutrient losses during establishment is especially important when evaluating P. Concerns with P losses are not only agronomic in terms of plant fertility but also represent an environmental issue regarding the fate of applied P fertilizers. Concentrations as low as 0.10 mg L⁻¹ correspond to increased incidence of eutrophication in water bodies (Faucette and Ferver 2010). The application of 100% PLA resulted in a 53% reduction in P leached during the first two WAP compared to the highly water-soluble 100% TSP; and over the six weeks 100% PLA resulted in a 47% reduction in cumulative P leached (Fig. 2). Therefore, PLA can reduce P leaching in a soil mixture that is commonly recommended for athletic field construction but would also be expected to reduce nutrient leaching in coarse-textured native soils. Further research is needed to evaluate PLA as a nutrient source that is broadcast applied to established turfgrass.

4.4. Conclusions

Using 100% PLA as a pre-plant fertilizer is an effective source for P, K, Ca, and Mg for bermudagrass establishment in a sand-based athletic field or native soil. There was no benefit to bermudagrass growth when mixing the more water-soluble TSP with the less water-soluble PLA but increasing PLA relative to TSP did reduce nutrient leaching. Therefore, PLA is a renewable nutrient source that can be used as an effective pre-plant fertilizer to support turfgrass growth

and reduce nutrient leaching during the initial weeks of establishment when planting into coarse-texture soils.

Chapter 5. Mid-Rotation Phosphorus Fertilization of Loblolly Pine

5.1. Introduction

Loblolly pine (*Pinus taeda* L.) is the most commercially valuable and planted tree species in the southeastern United States (Schultz, 1997; US Department of Agriculture, 2016; Raymond, Fox, & Strahm, 2016) due to greater growth rates (35 m³ ha⁻¹ year; 18-year rotation) and economic value represented by an internal return of 24% (Cubbage et al., 2007). However, growth rates and biomass production in loblolly pine stands are dependent on site factors such as fertility and water availability (Albaugh et al., 1998; Dougherty, Whitehead, & Vose, 1994; Allen, Dougherty, & Campbell, 1990). Plantation productivity is also commonly influenced by silvicultural practices such as site preparation and fertilization that enhance resource availability (Miller, Boyd, & Edwards, 1999; Jokela & Martin, 2000; Haywood et al., 2003). Therefore, understanding the impacts of forest management practices on plantation productivity requires extensive assessment of relationships between silvicultural practices and environmental limitations on tree growth (Tang et al., 2004).

Tree species growth is dependent on plant-available nitrogen (N) and phosphorus (P) in the soil (Allen, 1987; Fox et al., 2007). However, in loblolly pine plantations throughout the southern United States, levels of N and P are relatively low (Shoulders & Tiarks, 1983), thus nutrient deficiencies reduce aboveground biomass production (Vose & Allen, 1988; Retzlaff et al., 2001; Samuelson et al., 2001). Jokela & Martin (2000) reported that annual fertilizer application increased the above-ground biomass of loblolly pine by 197%. Usually commercial fertilizers such as diammonium phosphate (DAP) and urea are used to supply N and P to increase tree productivity in loblolly pine plantations (Ross, McKee, & Mims, 1995).

An alternative soil amendment used to supply N and P, and C in agricultural ecosystems is poultry litter (PL; Tabler & Berry, 2003), a by-product of poultry production that consists of bedding material such as peanut hulls and wood shavings, spilled feed and excreta (Blazier, Liechty, & Ellum, 2015). Besides N and P, it is a source of other macronutrients and micronutrients (Bolan et al., 2010), and it is used to fertilize major crops, pasture, and hayfields (Nyakatawa & Reddy, 2002; Pederson, Brink, & Fairbrother, 2002). Furthermore, PL has been shown to be an adequate fertilizer for loblolly pine (Samuelson et al., 1999; Friend et al., 2006; Blazier et al., 2008). Yet, PL application to loblolly pine plantations can be of environmental concern, principally nutrient runoff and leaching losses, especially of P, potentially leading to eutrophication of freshwater systems (Sheng et al., 2014; Bohara et al., 2018). Therefore, it is important to develop strategies to mitigate environmental impacts of PL.

One strategy is to combust it as a renewable fuel to generate electricity (Codling, 2013) and thereby reduce excess waste (Costello, 2017; Pagliari, Rosen, & Strock, 2009). The resulting poultry litter ash (PLA) contains high amounts of Ca, Mg, K and P, and is a source of Cu, Zn and Mn that may be utilized for crop production (e.g. Codling, Chaney, & Sherwell, 2002). Although N, C, and S are lost due to volatilization, the solubility of P is greatly reduced (Codling, 2013), compared to its solubility in PL Codling (2006). However, reduced solubility does not necessarily reduce plant uptake of P. For example, Codling, Chaney, & Sherwell, (2002) reported that there were higher P concentrations in wheat tissue where PLA rather than potassium phosphate was used as the P fertilizer source. Whether the same holds for mid-rotation loblolly pine is unknown. Therefore, a study was conducted to: 1) assess the adequacy of PLA relative to triple superphosphate (TSP) as a P source for mid-rotation loblolly pine, 2) quantify

the effects of PLA relative to TSP on selected soil properties and genotypes, and 3) quantify the expected reduction in runoff P with PLA compared to TSP.

5.2. Materials and Methods

A field study was conducted to evaluate the efficacy of PLA as an effective P source for a loblolly pine plantation on Wolfpen soil (loamy, siliceous, semiactive, thermic Arenic Paleudalfs) at the Louisiana State University Agricultural Center Hill Farm Research Station, Homer, LA (32.7522° N, 93.0431° W). The study was begun in December 2016, at which time the trees were 11 years old. The plantation consists of 30 plots, each 8.4 m along a row centered about a measurement a tree and 9.6 m across to the adjacent rows with five trees in each plot. There were two genotypes included in the study: eastern (North Carolina; 18 plots) and western (Louisiana; 12 plots). Phosphorus was applied at 0 and 56 kg P ha⁻¹ as either PLA or TSP, randomly assigned within each genotype. The P fertilization rate was based on Jokela (2004). Baseline soil characterization data are from the LSU AgCenter Soil Testing and Plant Analysis Laboratory (LSU AgCenter STPAL) for 1:1, water to soil pH, total organic C and total N by Leco C / N analyzer, and analysis of Mehlich 3 extracts by inductively coupled plasma (ICP) optical emission spectroscopy, and are given in Table 1. The PLA was obtained from North American Fertilizer, Benson, MN. Selected results of elemental and other analyses of a well-mixed subsample by the LSU AgCenter Department of Agricultural Chemistry are given in Table 2. Precipitation data were obtained from a nearby US National Oceanographic and Atmospheric Administration weather station, Minden 2.2 NE, LA.

Table 5. 1 Initial soil pH, total organic C (OC), total N (TN) and Mehlich 3 nutrients.

Depth	pH	OC	TN	P	K	Ca	Mg	S
15	5.4 ± 0.1 [†]	9.7 ± 2.3	0.6 ± 0.3	58 ± 14	162 ± 7	276 ± 89	20 ± 5	5 ± 1
30	5.1 ± 0.1	3.0 ± 0.7	0.3 ± 0.1	73 ± 24	174 ± 5	123 ± 33	6 ± 4	3 ± 1

Standard deviation.

Table 5. 2. Selected macronutrient composition and CaCO₃ equivalency (CCE) of PLA.

P	K	Ca	Mg	CCE
----- g kg ⁻¹ -----				%
43.5	74.7	159.0	20.3	22.44

5.2.1. Tree Growth and Tissue Composition

The diameter at breast height (DBH) and height of plot-center trees were measured using an electronic hypsometer (Haglof, Inc.) and foliar samples collected January 2017, March 2018 and September 2019. Foliar samples were dried at 60° C for two days before biomass was recorded. These were then ground and subsamples analyzed for nutrient concentration following digestion with nitric acid for 4 hours at 130° C and filtering (Whatman 42) into a final volume of 50 ml. Composition of digests were determined using inductively coupled plasma (ICP) optical emission spectroscopy (ICP SPECTRO ACRCOS Model FH E12, Kleve, Germany) by the LSU Agricultural Center Soil Testing and Plant Analysis Laboratory (LSU AgCenter STPAL).

5.2.2. Soil Sampling and Analyses

Soil samples were taken randomly to 30 cm within plots and divided into approximate A and E horizons (0 – 15 and 15 – 30 cm) in December (baseline) 2016, May and August 2017, and September 2019. Samples were bulked per plot and stored at 4 °C until analyzed for acid and alkaline phosphatase activity (*p*-nitrophenyl phosphate method; Tabatabai, 1994). Subsamples

were air-dried, ground and sieved at 2 mm, and analyzed for Mehlich 3 extractable nutrients, and for baseline samples, also pH, total organic C and total N.

5.2.3. P Runoff

Runoff subplots were stainless steel with covered collection troughs measured as 0.50 x 0.50 m x 0.15 m. Subplots were installed to 7.5 cm in three replicates of the fertilizer treatments all located in plots of the eastern genotype (N = 9). Runoff was drained through PVC pipes and collected into 20 L plastic collection buckets. Following rainfall > 2 cm, buckets were checked for runoff and volume measured by net mass. If there was sufficient volume for analysis (0.2 L), up to 0.5 L was taken for analysis during 2017 (16 events). Runoff subsamples were filtered for the colorimetric determination of dissolved reactive P (DRP) as described by Pote & Daniel, (2009). Total P (TP) was also determined on an unfiltered subsample by persulfate + H₂SO₄ digestion followed by colorimetric analysis (Pote, Daniel, & DeLaune, 2009).

5.2.4. Statistical Analyses

Tree growth, tissue composition and soil data were analyzed using the GLIMMIX procedure of SAS (2012). Fertilizer P treatment, genotype and treatment x genotype were fixed effects for growth increments from December 2016 to August 2018 and September 2019. Fertilizer, genotype, date of sampling and all interactions were fixed effects for the tissue composition data, and similarly for the soil data, which were analyzed separately by the two depths. Fertilizer P treatment was the fixed effect for cumulative loads of DRP and TP.

5.3. Results and Discussion

5.3.1. Tree Growth and Tissue Composition

In the southeastern United States, fertilizer is applied to more than 150,000 ha of plantation loblolly pine each year to increase tree productivity (Zhang & Allen, 1996). For

example, Scott and Bliss (2012) found that use of TSP increased loblolly pine height by 3 m and diameter at breast height (DBH) by 2.7 cm relative to no fertilizer control. However, at more than one and two years after P fertilizer application, neither increment in height nor DBH increased with fertilizer P, although genotype had a significant effect on DBH increase from December 2016 to August 2018 (Table 3). The latter difference, 1.79 compared to 1.07 cm increase, represents a 60% greater increase in basal area for the eastern compared with western genotype; however, the numerically similar difference from December 2016 to September 2019, 3.21 compared to 2.52 cm (35% greater basal area), was significant only at $P < 0.08$. Growth response to fertilization has been shown to differ by family, particularly for loblolly pine (Li, Allen, & McKeand, 1991; Samuelson, 2000).

There were significant effects of P fertilizer on tissue concentrations of P and Mn, of genotype on concentrations of B, Cu and Mn, and of year on P, K, S, B and Fe (Table 4). Specifically, tissue concentration of P and Mn was greater with TSP than for no P fertilization, as was Mn (Table 5). Furthermore, P concentration increased with time following application of fertilizer, thus possibly indicating a slow response to fertilization. However, B concentration also increased from 2018 to 2019, an effect that cannot be explained by the small amount added with PLA (189 mg kg⁻¹; not shown in Table 2), particularly since PLA did not increase the tissue concentration of B. Furthermore, tissue K decreased over time despite application of K with PLA. Tissue concentrations of B, Cu and Mn were all higher in the western than eastern loblolly genotype. Similarly, Crawford, Lockaby, & Somers (1991) found differences in accumulation of N, P and K among genotypes. In general, relationships between genetic selections and silvicultural practices are expected to have a positive impact on loblolly pine yield in the future. (Lambeth, McKeand, & Rousseau, 2005).

Table 5. 3. Analysis of variance results for effects of P fertilizer (F), genotype (G) and F x G on loblolly pine height and DBH increase from December 2016 to August 2018 and to September 2019.

Effect	Date	Δ Height	Δ DBH	Date	Δ Height	Δ DBH
F	08-2018	NS [†]	NS	09-2019	NS	NS
G		NS	*		NS	NS
F x G		NS	NS		NS	NS

[†] * = $P < 0.05$, ** = $P < 0.01$, *** $P < 0.001$ and NS = not significant.

Table 5. 4. Analysis of variance results for effects of P fertilizer (F), loblolly pine genotype (G) and sampling year (Y), and all interactions on tissue concentrations of selected macro- and micro-nutrients.

Effect	P	K	Ca	Mg	S	B	Cu	Fe	Mn	Zn
F	** [†]	NS	NS	NS	NS	NS	NS	NS	**	NS
G	NS	NS	NS	NS	NS	***	*	NS	***	NS
F x G	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
Y	**	**	NS	NS	*	**	NS	**	NS	NS
F x Y	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
G x Y	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
F x G x Y	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS

[†] * = $P < 0.05$, ** = $P < 0.01$, *** $P < 0.001$ and NS = not significant.

Table 5. 5. Effects of fertilizer P, F (control, C; poultry litter ash, PLA; or triple superphosphate, TSP), genotype, G (eastern, E; or western, W), and sampling year, Y (2016, 2018 or 2019), on loblolly pine height foliar tissue concentrations.

Effect	Level	P	K	B	Cu	Fe	Mn
		----- g kg ⁻¹ -----		----- mg kg ⁻¹ -----			
F	C	1.32 B	5.05	12.13	3.02	229	269 B
	PLA	1.37 AB	4.99	12.90	3.10	426	286 B
	TSP	1.44 A	4.98	12.60	3.08	460	353 A
G	E	1.40	4.86	9.86 B	2.93 B	448	225 B
	W	1.36	5.16	15.23 A	3.20 A	295	381 A
Y	2016	1.34 B	5.38 A	11.92 B	2.93	817	303
	2018	1.34 B	4.92 B	11.75 B	3.02	426	286
	2019	1.45 A	4.72 B	13.96 A	3.25	229	319

† Numbers in a column for an effect followed by different letters are significantly different ($\alpha = 0.05$).

5.3.2. Mehlich 3 P

The effect of P fertilization and sampling data on soil Mehlich 3 P was significant for the A horizon but not the E (Table 6). Specifically, concentrations decreased as TSP > PLA, control, and increased with time from pre-fertilization = May 2017 < August 2017, which were greater than September 2019 (Table 7). Lower concentration of Mehlich 3 P in the A horizon with PLA than TSP presumably reflects the lower solubility of PLA and likely physical retention of PLA particles by the O horizon, which in turn may account for maximum concentration in the A horizon in August rather than May 2017. Lower concentrations more than a year later in September 2019 coincide with greatest foliar tissue P (Table 4), thus to some extent may reflect P uptake from the soil. Although the effects of neither fertilization nor sampling date were significant in the E horizon, numerical trends were similar (Table 7). Numerically lower Mehlich 3 P in the A horizon

of eastern than western genotype loblolly pine and significantly lower concentrations in the E horizon may be due to soil spatial variability that was not controlled in the experimental design, however, since the concentration of P in eastern genotype foliage was numerically greater than in the western genotype (Table 5), P uptake may have also affected concentrations of soil Mehlich 3 P.

Table 5. 6. Analysis of variance results for effects of P fertilizer (F), genotype (G) and sampling date (D), and all interactions on Mehlich 3 P in the A and B horizons.

Horizon	F	G	F x G	D	F x D	G x D	F x G x D
A	***†	NS	NS	***	NS	NS	NS
E	NS	***	NS	NS	NS	NS	NS

† * = $P < 0.05$, ** = $P < 0.01$, *** $P < 0.001$ and NS = not significant.

5.3.3. Soil Phosphatase Activity

Genotype significantly affected acid phosphatase activity in A and E horizons and alkaline phosphatase activity in the E horizon (Table 8). Specifically, whereas acid phosphatase activity was greater in soil under the eastern genotype in the A horizon, alkaline phosphatase activity was greater under the western genotype in both horizons (Table 9). Fertilizer P did not affect phosphatase activity, and neither acid nor alkaline phosphatase in either the A or E horizons appeared related to soil moisture as inferred from antecedent rainfall and temperature as found by Sardans et al. (2008) and Huang et al. (2011), except acid activity in the A horizon. The latter was lowest for the September 2019 sampling for which the combination of summer temperature

Table 5. 7. Effects of fertilizer P, F (control, C; poultry litter ash, PLA; or triple superphosphate, TSP), genotype, G (eastern, E; or western, W), and sampling date, D (December 2016, May 2017, August 2017 or September 2019), on Mehlich 3 P in the A and E horizons.

Effect	Level	A Horizon	E Horizon
		----- mg kg ⁻¹ -----	
F	C	64.1 B	79.1
	PLA	68.6 B	83.3
	TSP	83.4 A	85.9
G	E	68.9	73.1 B
	W	75.2	92.3 A
D	December 2016	58.7 C	76.8
	May 2017	69.1 BC	82.5
	August 2017	87.2 A	91.3
	September 2019	73.3 B	80.5

† Numbers in a column followed by different letters are significantly different ($\alpha = 0.05$).

and comparatively little antecedent rainfall (3 and 11 cm, the preceding one and two months, compared in particular with 11 and 37 cm for the August 2017 sampling; respective soil water contents, 0.036 and 0.073 at sampling) may have suppressed activity due to droughty soil conditions. The genotype x sampling date interaction for acid phosphatase in the A horizon largely reflects higher activity in soil under the eastern genotype, whereas the fertilizer P x genotype interaction for alkaline phosphatase may indicate that effects of fertilizer source depend on genotype, at least for the eastern loblolly pine (activity with PLA > control, TSP). In general, however, effects on alkaline phosphatase activity were small compared to acid phosphatase, and activity of acid phosphatase in the A horizon was about twice that in the E horizon.

Table 5. 8. Analysis of variance results for effects of P fertilizer (F), genotype (G) and sampling date (D), and all interactions on acid and alkaline phosphatase activity in the A and E horizons.

Phosphatase	Horizon	F	G	F x G	D	F x D	G x D	F x G x D
Acid	A	NS [†]	***	NS	***	NS	*	NS
Alkaline		NS	NS	**	***	NS	NS	NS
Acid	E	NS	*	NS	***	NS	NS	NS
Alkaline		NS	**	NS	***	NS	NS	NS

[†] * = $P < 0.05$, ** = $P < 0.01$, *** $P < 0.001$ and NS = not significant.

Table 5. 9. Effects of fertilizer P, F (control, C; poultry litter ash, PLA; or triple superphosphate, TSP), genotype, G (eastern, E; or western, W), and sampling date, D (December 2016, May 2017, August 2017 or September 2019), on acid and alkaline phosphatase in the A and E horizons.

Effect	Level	A Horizon		E Horizon	
		Acid	Alkaline	Acid	Alkaline
----- $\mu\text{mol g}^{-1} \text{h}^{-1}$ -----					
F	C	2.35	0.14	0.97	0.14
	PLA	2.43	0.16	0.96	0.13
	TSP	2.48	0.14	1.05	0.13
G	E	2.70 A	0.14	0.93	0.12 B
	W	2.15 B	0.15	1.06	0.15 A
D	December 2016	2.32 B	0.22 A	0.81 C	0.24 A
	May 2017	2.62 A	0.12 B	0.74 C	0.06 C
	August 2017	2.79 A	0.06 C	0.96 B	0.06 C
	September 2019	1.97 C	0.19 A	1.46 A	0.18 B

[†] Numbers in a column followed by different letters are significantly different ($\alpha = 0.05$).

5.3.4. Runoff P

Transport and P sources are two key factors that influence P movement in runoff

(Sharpley, 2000; McDowell et al., 2004; Sharpley, McDowell, & Kleinman, 2001). The effect of

source was seen in mean cumulative load of TP through nearly one year after P fertilizer application, being numerically greatest from the TSP plots, total P load of PLA plots was only slightly greater than from controls (Figure 1; DRP similar but not shown). However high variability precluded statistically significant difference. The increase due to TSP with respect to the control was 0.14 kg ha^{-1} compared with 0.01 kg ha^{-1} for PLA over about one year of runoff, and largely reflected initial losses. A similar difference between P in runoff with highly water-soluble TSP and less water-soluble P sources was shown by Shigaki, Sharpley, & Prochnow (2007).

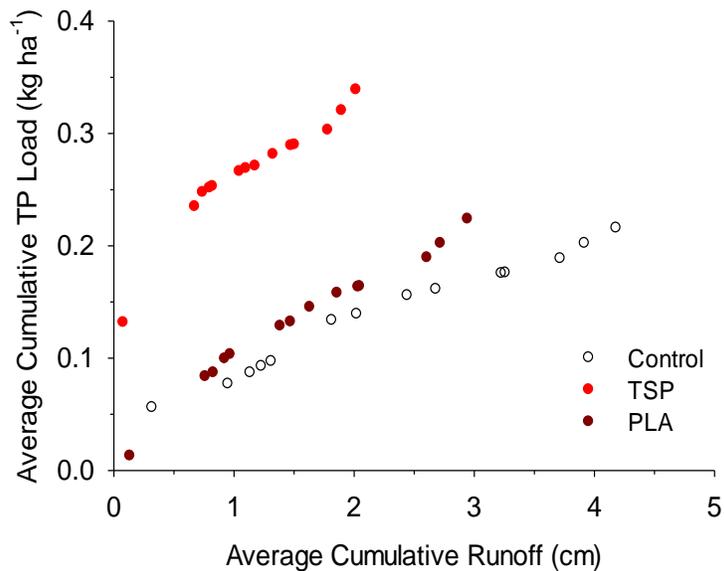


Figure 5. 1. Average cumulative loads of P from plots fertilized with 56 kg P ha^{-1} as poultry litter ash (PLA) or triple-superphosphate (TSP) and without P fertilization.

5.4. Conclusions

As of over two years post-application, neither PLA nor TSP had increased measures of loblolly pine growth or increased uptake of P compared to the unfertilized control averaged over eastern and western genotypes. Rather, the only significant effects on tree growth parameters

were of genotype, greater increase in DBH from December 2016 to August 2018 for the eastern than western genotypes. However, fertilization with TSP increased foliar concentration of P compared to PLA and control, and foliar P tended to increase over time. Similarly, Mehlich 3 P in the A horizon was significantly greater for TSP than PLA, which was only numerically greater than the control, and increased to a maximum for the second post-application sampling in August 2018, then declined. Differences in Mehlich 3 P in the E horizon followed the same pattern but were not significant. Fertilizer P source did not affect soil phosphatase activity, whereas genotype and sampling date did. However, the effect of genotype varied with acid or alkaline phosphatase assay and horizon, and comparatively wet / dry soil conditions over time were only weakly related to lower activity (possibly acid phosphatase in the A horizon). Consistent with expected effect of P solubility, P losses were less with PLA than TSP, however, variability among replicates precluded statistical significance. Beyond the first one or two runoff events, the difference in cumulative P losses between PLA and TSP was negligible. Follow-up measurements of tree growth may be necessary to conclude whether P fertilization of mid-rotation loblolly pine under initial soil conditions at the Wolfpen site was beneficial, and discern any longer-term differences between PLA and TSP.

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Vita

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