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The Potential for Phosphorus Release from Floodplain Soils: Temporal Variability and Management Strategies

A thesis submitted in partial fulfillment of the requirements for the degree of Master of Science in Crop, Soil, and Environmental Sciences

by

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December 2017 University of Arkansas

This thesis is approved for recommendation to the Graduate Council.

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Abstract

Soils may contain large amounts of legacy phosphorus (P) due to past application of excess fertilizers, releasing P during runoff and flood events. As the level of P in soils increases, the release of P from the soils increases, but the temporal fluctuations of this release are not well known. Experiments were conducted to examine 1) the variability of soluble reactive P (SRP) release from the soil throughout a year and 2) the effect of repeated flooding and drying cycles on release rates. Soil cores were collected 7 times throughout a year from 2 sites within the Watershed Research and Education Center (WREC). Cores were inundated, water samples were analyzed for SRP, and release rates were calculated for each core. The soil was also collected and analyzed for Mehlich-III and water extractable P (WEP). SRP release rates changed significantly across seasons, but were not significantly related to Mehlich-III P (M3P) or WEP. The final set of cores was maintained in the lab for 73 days and flooded 5 times; there was no noticeable effect on SRP release.

Soils high in P should be managed so as not to become P sources to the watershed. Water treatment residuals (WTRs) have the ability to bind to P and reduce its solubility, but little research has been conducted on whether liquid WTRs would be more effective. Our objectives were to 1) determine an effective liquid WTR application rate and 2) the effectiveness of that rate when flooded repeatedly. Six liquid WTR treatments (220, 440, 1320, 2200, 3100, or 4400 kg ha⁻¹) and a control were applied in triplicate to cores from 3 sites within WREC. Soils were flooded and SRP was analyzed in the water samples. At a rate of 440 kg ha⁻¹, the liquid WTRs substantially reduced SRP release rates from the cores. When this rate was applied to another set of cores, release was controlled for 73 days when soil test P (STP) was less than 100 mg kg⁻¹. With higher STP, greater quantities or repeated applications of liquid WTRs may be required.

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Introduction

Soils are generally thought of as sinks for phosphorus (P), potentially adsorbing dissolved P from aqueous solution, but they also have the potential to be sources within watersheds. Soils within a watershed that receive inputs from typical P sources can become enriched and begin to act as sources. For example, soils can become elevated in P when manure was historically applied to meet the nitrogen needs of forages and crops (Sharpley et al., 1993). There have been past practices which has led to elevated soil test P (STP) within agricultural lands, i.e. legacy P in soil. Thus, legacy P issues across the agricultural landscape may be an important non-point source within the watershed (Sharpley et al., 1993; Abrams and Jarrell, 1995; Eghball and Power, 1999; Agyin-Birikorang et al., 2007).

While the P levels in soils influence release rates, P release may also be affected by temporal changes such as temperature (Chen et al., 2003). That is, warmer soils release P more readily than colder soils due to more active soil microbes (Holden and Armstrong, 1980; Habibiandekordi et al., 2015). However, there are also studies finding little to no effect of temperature on P release (e.g., see Kadlec and Reddy, 2001).

Another factor that might influence P dynamics in soils is moisture content, i.e., the wetting, drying, and rewetting in the environment. Several studies have found that reflooding dried soils results in a surge of P release if there is a large pool of labile P at the soil surface (Chepkwony et al., 2001; Venterink et al., 2002; Pant and Reddy, 2003; Aldous et al., 2005). P release may decrease following multiple flood cycles as that pool of labile P is reduced, but if soils are high in P, they may continue releasing for several years and act as a P source to the watershed (Pant and Reddy, 2003).

Because P is released from soils, and that release can fluctuate based on environmental factors, it's important to know how to manage that soil P. Water treatment residuals (WTRs) have been proposed as a mitigation technique for soils high in P. Many studies have shown that WTRs have high P sorption capacities, and that WTR addition to soils reduces available STP (Dayton and Basta, 2005; Makris et al., 2005; Agyin-Birikorang et al., 2008). The application of solid WTRs to small plots significantly reduced the soluble reactive P (SRP) concentration in the collected runoff as well as the availability of P in treated soils (Gallimore et al., 1999; Dayton et al., 2003; Habibiandekordi et al., 2015). The magnitude of the reduction in P availability in soils is dependent on the amount of WTRs incorporated (Novak and Watts, 2005). Thus, greater soil P levels require a larger quantity of solid WTRs to achieve the target reduction (Haustein et al., 2000).

Chapter 1: Temporal Variations in Phosphorus Release Rates from Floodplain Soils

Abstract

The release of legacy phosphorus (P) from soils historically fertilized with P above plant needs is a growing water quality concern. Understanding when these soils release P and the conditions associated with that release may improve the management of these areas and reduce P transport to streams and rivers. Two experiments were conducted to 1) examine the temporal changes in soluble reactive P (SRP) release rates and 2) to determine how multiple flooding and drying events affect SRP release rates from the soil. Cores were collected from two different soil series (Cleora fine sandy loam, CFSL and Leaf silt loam, LSL) at 7 different times throughout the year. One set of cores was maintained in the lab and flooded and drained 5 times over the course of 73 days in the summer. Cores were inundated with tap water for 7 hours and SRP concentrations were measured periodically. Release rates were calculated for each core and were separated by site for analysis. SRP release rates were not significantly related to water extractable P (WEP) at either site (P>0.05), but were significantly related with Mehlich-III P (M3P) (P<0.05) in LSL cores. Release rates did change significantly throughout the year (P<0.05), such that rates were least at the beginning of the year and greatest in the second half of the year. SRP release rates measured on the same cores did not change with flooding cycles over the course of the experiment. Factors such as plant growth or microbial activity may have been responsible for variable SRP release rates, but further research is needed to better understand how to manage these areas.

Introduction

The problems resulting from elevated phosphorus (P) in water bodies (i.e., streams, rivers, lakes, estuaries, etc.) have been well-documented (Daniel et al., 1998; Carpenter et al., 1998; Correll, 1998). Runoff from the landscape during rainfall events can transport P downslope where it eventually ends up in the water bodies. Soils have the ability to adsorb and store P, as well as release it into aqueous solution (Taylor and Kunishi, 1971). The release of P into waters (e.g., storm water runoff or floodwaters) increases with the amount of extractable P stored within the soil (Daniel et al., 1994; Young and Ross, 2001; Reavis and Haggard, 2016)

While the extractable P levels in soils influence release rates, P release may also be affected by temporal changes such as temperature (Chen et al., 2003). That is, warmer soils release P more readily than colder soils due to more active soil microbes (Holden and Armstrong, 1980;

Habibiandekordi et al., 2015). However, there are also studies finding little or no effect of temperature on P release from soils and sediments (e.g., see Kadlec and Reddy, 2001).

Another factor that might influence P dynamics in soils is the wetting, drying, and rewetting under natural conditions. Several studies have found that reflooding dried soils results in a surge of P release if there is a large pool of labile P at the soil surface (Chepkwony et al., 2001; Venterink et al., 2002; Pant and Reddy, 2003; Aldous et al., 2005). P release may decrease following multiple flood cycles as that pool of labile P is reduced, but if soils are high in P, they may continue releasing P for several years and act as a P source to the watershed (Pant and Reddy, 2003).

The fluctuations in P release when soil are subject to cycles of wet and dry are not well understood or consistent, as many factors may influence the SRP release rate from the soil. If soils are a potential P source, then it is important to understand if and how P release rates vary over time and with repeated wetting and drying events. The objectives of this study were 1) to observe whether SRP release rates vary temporally and 2) to evaluate whether repeated rainfall and flood events affect SRP release rates from soil.

Study Site Description

The University of Arkansas's Division of Agriculture Watershed Research and Education Center (WREC), in the headwaters of the Illinois River Watershed, encompasses 95 ha of land just north of the university in Fayetteville. The site, established in 2006 within the Arkansas Agricultural Research and Extension Center, contains wetlands, a stream, and pasture lands and includes a variety of soil series.

Soil series and management were different at the two sampling sites (Web Soil Survey, 2017). The soil at site 1 was mapped as a Leaf silt loam (LSL)— an active, thermic Typic Albaquult. It is a deep, poorly drained soil often found on floodplains. Nitrogen (N) and potassium (K) fertilizers were applied to the field on 27 May and 13 August of 2016. The soil at site 2 was mapped as Cleora find sandy loam (CFSL) — a mixed, active, thermic Fluventic hapludoll. It is a well-drained, course-textured soil also found on floodplains. N fertilizer was applied to this field on 26 August 2016 and 14 April 2017. In addition, cattle were grazed on site 2 fields for most of the year.

Both sites were in close proximity to the stream, Research Branch, which runs through WREC. The stream receives urban storm water and runoff from surrounding agricultural areas, and thus, WREC has been the focus of studies on the effects of land use (Brion et al., 2011), bioassessment (Longing and Haggard, 2010), and a watershed mass balance (Metrailor, 2012). The proximity to surrounding urban landscapes subjects the site to periodic floods throughout the year, inundating the soils and vegetation near the stream.

Methods

Soil core collection

Soil cores were collected from WREC in June, August, October, and December of 2016, and February, April, and May of 2017. Four cores were taken from each site during each collection. Plexiglas tubes (7.6 cm inner diameter) were pushed 10 cm into the ground and carefully twisted out, leaving an intact soil core inside. Cores were capped and returned to the lab where the caps were sealed with electrical tape to hold water.

Soil core incubation

Each soil core was inundated with tap water (having negligible P concentration, SRP = 0.002 mg L⁻¹) to an overlying volume of 1L. The overlying water was aerated to mimic flood waters and to prevent reductive dissolution of iron phosphate compounds under anaerobic conditions. Using a plastic syringe, 30 mL water samples were taken from the center of the overlying water in each core 0.5, 1, 2, 4, and 7 hours after flooding. Samples were filtered through 0.45 μ m membrane filters, acidified using 0.03 mL of concentrated HCl, and analyzed for SRP on the DU 730 Life Science UV/Vis spectrophotometer using the ascorbic acid method (APHA, 1999). The overlying water was replenished after each sampling round with an equal volume of tap water.

The cores collected during the final month (May 2017) were maintained in the laboratory for the entire summer to understand how cycles of rain events, floods, and dry periods affect soil P release. When rain events occurred at WREC, a volume of tap water equal to the depth of rainfall was added to each core within one day of the event, and water was allowed to infiltrate through and drain from the cores as in the field. Precipitation was estimated by averaging precipitation depths measured at two USGS gauge stations (USGS 071948095 Mud Creek and USGS 07048495 Town Branch) located north and south, respectively, of WREC. When precipitation depth of the two gauge stations averaged greater than 1.3 cm for one storm event and flooding was likely at WREC, cores were sealed at the bottom and inundated with 1 L overlying water. The overlying water was aerated, and 30 mL water samples were collected, filtered, acidified, and analyzed as previously described.

Soil moisture was measured throughout the summer experiment using the gravimetric method (DeAngelis, 2007). A sample of soil (\sim 10 g) was taken from one core at each site before each flood event and at least once per week throughout the experiment.

Following the experiment, the cores were drained, and the top 2 cm of soil were removed from each core. The soils were dried in an oven at 105 °C and ground with mortar and pestle to pass a 2mm sieve. Soils were analyzed by the University of Arkansas Soil Diagnostic Lab (Fayetteville, AR, U.S.A) for Mehlich-III and water extractable P, Al, and Fe.

SRP analysis and release rates

We accounted for the removal and addition of water and SRP to maintain a mass balance of P released into the overlying water. To estimate potential SRP release rate from the soil, the mass of SRP in the water at each sampling time was plotted against duration of inundation, and that slope (mg h⁻¹) was divided by the surface area of the cores (m²) to obtain the average potential P release rate (mg m⁻² h⁻¹). Rates were calculated individually for each core throughout the year.

Because the soil series and management regimes were so different between the two sites, the data were separated and analyzed by site rather than all together. For the year-long experiment, data were sorted into decimal time and SRP release rates were plotted against variables such as M3P, WEP, and time to determine significant relationships (α =0.05). In the summer experiment, SRP release rates were compared with soil moisture and inundation dates to determine if soil moisture or repeated flooding had any effect on SRP release rates.

Results

Seasonal Variability

Cleora Fine Sandy Loam

At this site, WEP averaged 22 mg kg⁻¹, though WEP within individual cores ranged widely from 8 to 50 mg kg⁻¹ over the course of the year. WEP content among cores collected at the same time during the year varied as much as 32 mg kg⁻¹, showing the variability of P content within an area. Unexpectedly, WEP was not significantly related to SRP release rates (P=0.547, Figure 1A). When the residuals of this model were plotted over time, they showed a significant seasonal pattern (r^2 =0.268, P=0.044, y=0.12–1.30*sin(2* π *time)+0.41*cos(2* π *time), Figure 1B) indicating that SRP release change throughout the year. WEP was generally greater in the beginning and end of the year compared to the middle, but there was no significant relationship between WEP and season (P=0.150, Figure 1C).



Figure 1. (A) Relationship between water extractable phosphorus (WEP) and soluble reactive P (SRP) release rates (r=0.132, P=0.547), (B) residuals of A plotted with time (r^2 =0.268, P=0.044, y=0.12-1.30*sin(2* π *time)+0.41*cos(2* π *time)), and (C) relationship between WEP and season (P=0.150).

M3P was significantly related to WEP content of the soils (r=0.556, P<0.001) and was also highly variable. While the average M3P content across all cores was 109 mg kg⁻¹, M3P ranged from as little as 25 to as much as 220 mg kg⁻¹. Cores were all collected within 3 meters of each other, but M3P varied as much as 50 mg kg⁻¹ within sampling dates.

Like WEP, M3P was not significantly related to SRP release rates as it was expected to be (P=0.838, Figure 2A), though there was a significant relationship between the reiduals and season (r^2 =0.264, P=0.047, y=0.12–1.3*sin(2* π *time) +0.56*cos(2* π *time), Figure 2B), further indicating that release rates change across seasons. We saw no significant pattern or relationship between M3P and season (P=0.716, Figure 2C).



Figure 2. (A) Relationship between Mehlich-III phosphorus (M3P) and soluble reactive P (SRP) release rates (-0.045, P=0.838), (B) relationship between residuals of A and season (r^2 =0.264, P=0.047, y=0.12–1.3* sin(2* π *time)+0.56*cos(2* π *time)), and (C) relationship between M3P and season (P=0.716).

SRP release rates were highly variable, ranging from 0.4 to as much as 9.0 mg m⁻² h⁻¹ in individual cores, though the average was 2.6 mg m⁻² h⁻¹. Though not related to M3P or WEP, SRP release rates, as the residuals of the relationships indicated, were significantly related to season (r^2 =0.269, P=0.044, y = 2.72–1.34*sin(2* π *time)+0.55*cos(2* π *time), Figure 3), which

accounted for 27% of the variability in SRP release. SRP release rates decreased from the beginning of the year through early summer, then increased during fall until reaching rates similar to those at the start of the year.



Figure 3. Significant relationship ($r^2=0.269$, P=0.044, y = 2.72–1.34*sin($2*\pi*time$) + 0.55*cos($2*\pi*time$)) between soluble reactive phosphorus (SRP) release rates and season.

Leaf Silt Loam

WEP averaged 44 mg kg⁻¹ at this site, and individual cores ranged from 17 to 90 mg kg⁻¹. WEP and SRP release were not significantly related (P=0.493), but there was a significant relationship between those residuals and season (r²=0.579, P<0.001, y=0.6943.68*sin(2* π *time)+0.55* cos(2* π *time), Figure 4). Residuals at the beginning of the year were generally less than zero while the residuals in the second half of the year were generally greater than zero, meaning that when variability related to WEP is accounted for, release rates in the beginning of the year are less than those in the second half of the year. WEP at this site was also significantly related to season (r²=0.310, P=0.024, y= 43.25+4.79*sin(2* π *time)+14.04*cos(2* π *time)), and was generally greater at the beginning and end of the year. Season accounted for roughly 31% of the variability in WEP.



Figure 4. (A) Relationship between water extractable phosphorus (WEP) and soluble reactive P (SRP) release rate (P=0.493), (B) relationship between residuals of A and season (r^2 =0.579, P<0.001, y=0.694–3.68*sin(2* π *time)+0.55*cos(2 * π *time)), and (C) relationship between WEP and season (r^2 =0.310, P=0.024, y=43.25+4.79 *sin(2* π *time)+14.04* cos(2* π *time)).

M3P ranged from 105 to 280 mg kg⁻¹ at this site and was significantly related to WEP (P<0.001). M3P was also significantly related to SRP release rates ($r^2=0.230$, P=0.020, y=0.03*x*-1.98, Figure 5A), accounting for more than 20% of the variability. As with WEP, residuals of this relationship were significant with season ($r^2=0.383$, P=0.007, y=0.499–2.77*sin(2* π *time)– 0.15*cos(2* π *time), Figure 5B). When M3P was plotted against time (Figure 5C), season on its own was not significant with M3P (P>0.05), but it became barely significant when the time was included in the model ($r^2=0.337$, P=0.045, y=261.02–58.01*sin(2* π *time)+11.20* cos(2* π *time)–84.16(time)). Time and season accounted for roughly 34% of the variability in M3P.



Figure 5. (A) Relationship between Mehlich-III phosphorus (M3P) and soluble reactive P (SRP) release ($r^2=0.230$, P=0.020, y=0.03x-1.98), (B) relationship between residuals of A and season ($r^2=0.383$, P=0.007, $y = 0.499-2.77*sin(2*\pi*time)-0.15*cos(2*\pi*time)$), and (C) relationship between M3P, time, and sesason ($r^2=0.337$, P=0.045, $y = 261.02-58.01*sin(2*\pi*time)+11.20*cos(2*\pi*time)-84.16(time)$).

SRP release rates across all cores from this site averaged 5.2 mg m⁻² h⁻¹, and the individual cores were quite variable, ranging from 0.6 to 12 mg m⁻² h⁻¹. Release rates were significantly related to M3P, but not to WEP. The residuals of those plots suggested that release rates change across seasons. Release rates from soils were least at the beginning of the year (generally <5 mg m⁻² h⁻¹) than they were at the end of the year (generally >4 mg m⁻² h⁻¹), and when plotted over time (Figure 6), release rates significantly changed across season (P<0.001, y = 5.87 – $3.81*\sin(2*\pi*time) + 0.16*\cos(2*\pi*time))$ which accounted for more than half of the variability

in SRP release ($r^2=0.577$).



Figure 6. Significant relationship between soluble reactive phosphorus (SRP) release rates and season ($r^2=0.577$, P<0.001, $y = 5.87 - 3.81*\sin(2*\pi*time) + 0.16*\cos(2*\pi*time))$.

Summer Experiments

There were 30 rain events over the course of the 73-day experiment (Figure 7), 7 of which were considered flood events because rainfall was greater than 1.3 cm. Flood events occurred on days 2, 9, 10, 24, 36, 44, and 73. Because the flood events happened so close together, events on days 9 and 10 were considered one event, and cores were only flooded once.

The average SRP release rates from the LSL were not significantly different among flood events (ANOVA, P>0.05), and did not increase or decrease over time (regression, P>0.05). The release rate after the first flood was 1.1 mg m⁻² h⁻¹, when soil moisture before flooding was around 20%. Soil moisture increased following 4 rain events to nearly 50%, and SRP release rates decreased to less than 1.0 mg m⁻² h⁻¹. Release rates reached a maximum of 1.5 mg m⁻² h⁻¹ during the third flood and soil moisture had decreased to 35%. Soil moisture decreased again along with release rates to 25% and 1.1 mg m⁻² h⁻¹, respectively. From there, release rates increased during the fifth

and sixth flood events and ended at $1.3 \text{ mg m}^{-2} \text{ h}^{-1}$. Soil moisture increased to nearly 70% but returned to about 35% at the end of the experiment.

The average SRP release rate from the CFSL was not significantly different among flooding events (ANOVA, P>0.05) and stayed around 0.4 mg m⁻² h⁻¹ throughout the experiment. The average release rate followed a pattern similar to that from LSL, except between floods 4 and 5 when rates from CFSL decreased while LSL increased. Fluctuations in soil moisture in these cores was also very similar to LSL, following nearly the same pattern throughout, though, CFSL generally had lower soil moisture content compared to CFSL.



Figure 7. Precipitation and soil moisture (SM) data at two sites (Cleora Fine Sandy Loam and Leaf Silt Loam) within the Watershed Research and Education Center over the summer of 2017 (*top*), and average soluble reactive phosphorus (SRP) release rate from the two sites with error bars denoting standard error about the means (*bottom*).

Discussion

The literature indicates that WEP should be significantly related to SRP release from soil and explain a substantial amount of the variability (Pote et al., 1996, 1999; Reavis and Haggard, 2016). However, SRP release rates were not significantly related to WEP in the soils at either

site, and there was a large amount of scatter within the data. WEP at both sites was larger at the beginning and end of the year compared to the middle. This may be because plants tend to take up less P when soil temperatures are cooler and they aren't growing, so P may accumulate in the soil. This may account for the greater amounts of soluble P in the soils at those times (Ye et al., 2014). Fertilizer applications in the spring may have boosted plant growth since WEP content in the soil declined in late spring and early summer, as the plants began growing. In addition to plant uptake, soil microbes can also be affected by soil temperature, and may slow mineralization of soil P in cooler temperatures while increasing mineralization in warmer months (Schmidt et al., 1999).

M3P, like WEP, is generally related to SRP release from soils, whether in runoff (Sharpley, 1995; Pote et al., 1996; Andrasky and Bundy, 2003) or flood events (Reavis and Haggard, 2016), but there was only a significant relationship at the LSL site. In the CFSL cores, several soil samples were unusual in that their M3P content was <60 but SRP release ranged as high as 9 mg $m^{-2} h^{-1}$ (Figure 2A). Those four samples all had extremely high M3 calcium (M3Ca) content (2100-2500 mg kg⁻¹, data not reported), as much as three times that in other cores at this site, indicating that the area may have been limed shortly before we collected soil cores; however, no lime had been applied on or near these fields in the past two years. Using a Mehlich-III extraction on calcareous soils, though, may have resulted in the low M3P numbers as it may not have extracted as much P as a method more suited to alkaline soils (Zbiral and Nemec, 2007). Removing those four soil samples from the analysis results in a significant relationship with SRP release, which is what we would have expected.

SRP release rates at both sites followed significant seasonal patterns throughout the year, as the SRP residuals suggested. Release rates did not follow the same patterns as WEP, which was

greater in the winter than summer, or M3P, which tended to be highly variable throughout the year; rather, rates were generally smaller in the first half of the year compared to the second half, especially from LSL soils. Fertlizer application in the spring may have boosted plant growth, resulting in smaller release rates in the late spring and early summer.

The summer experiment was conducted when SRP release rates should have been increasing. Rates during the year-long study ranged from 1 to 4 mg m⁻² h⁻¹ and 2.5 to 6 mg m⁻² h⁻¹ from CFSL and LSL sites, respectively. However, release rates never reached greater than 0.6 or 1.5 mg m⁻² h⁻¹ in the cores from the two sites, respectively, during the summer experiment. This may be because when cores were collected at the end of May, release rates, M3P, and WEP were generally at their lowest points throughout the entire year. Once in the lab, environmental factors such as plant growth did not influence P levels in the soil, and therefore SRP release rates remained unchanged throughout the summer.

Drying and rewetting soils has been shown to affect the amount of SRP released when reflooded (Chepkwony et al., 2001; Bostic and White, 2006). Desiccation of the soils can lead to microbial cell lysis or decreased affinity for P, resulting in a flush of P upon initial rewetting (Baldwin, 1996; Baldwin and Mitchell, 2000; Bostic and White, 2006). However, our soils did not reach the point of dryness that would result in microbial cells lysis or loss of affinity. Our soils were rewetted after only partially drying, and others cite that this may actually increase soil minerals' affinity for P, resulting in a smaller release of P when soils are flooded (Baldwin and Mitchell, 2000). While SRP release did occasionally vary with soil moisture in this manner, it was not consistent enough to attribute these fluctuations to soil moisture. We saw no clear relationship between soil moisture and SRP release from our cores at either site throughout the summer.

Conclusions

Soil P is highly variable across landscapes and over time, but its solubility to aqueous solution and tendency to be released throughout the year depend on various factors in the soil and environment.

- SRP release rates changed significantly across the seasons, and tended to be greatest in the fall and least in the late spring.
- Repeated wetting and drying events did not have any significant impact on SRP release from flooded soils.

Numerous factors such as temperature, microbial communities, soil mineral content, soil moisture, fertilization, etc., can play a role in SRP release from soils, making it difficult to understand which variables are responsible for SRP release in a specific area. However, understanding those variables would allow for better management of soil P and its potential for release.

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Chapter 2: Mitigating Soil Phosphorus Release Using Liquid Water Treatment Residuals

Abstract

Past application of fertilizers and manure to the landscape has led to a buildup of legacy phosphorus (P) within soils of some watersheds, leading to potential water quality concerns. Water treatment residuals (WTRs) have been land applied as solids (20% solids) to reduce P release from these soils, but using liquid WTRs (2% solids) may be more effective since it is difficult to get uniform coverage with solid WTRs. The objectives were 1) to show the variability of P release within a field treated with solid WTRs at a rate of 22,417 kg ha⁻¹ yr⁻¹, 2) to determine an more effective application rate for liquid WTRs, and 3) to evaluate the effectiveness of that application rate over several months. Six liquid WTR treatment rates (220, 440, 1320, 2200, 3100, or 4400 kg ha⁻¹) and a control were applied in triplicate to soil cores at varying soil test P (STP) levels. SRP was measured in each flooded core 5 times within 7 hours. P release rates were calculated for each core, and the top 2 cm of soil were analyzed for Mehlich-III (M3) and water extractable (WE) P, iron (Fe), and aluminum (Al). STP and release rates were highly variable (ranged 430-1400 mg M3P kg⁻¹ and 2-14 mg m⁻² h⁻¹, respectively) from the field treated with solid WTRs. Only a small application rate (440 kg ha⁻¹) of liquid WTRs was needed to significantly control P release from soil cores. This application rate was sufficient to control release over several months at STP <100, but greater application rates or repeated applications may be needed for long-term control on soils with greater STP. While there are challenges associated with using liquid WTRs (i.e., transportation costs), they are highly effective at reducing SRP release, and, if used as a field border in conjunction with solid WTR application to the field, they could be a valuable tool in managing P hotspots within watershed.

Introduction

Soils are generally thought of as sinks for phosphorus (P), potentially adsorbing dissolved P from aqueous solution, but they also have the potential to be sources within watersheds. Soils within a watershed that receive excessive P inputs can become enriched and begin to act as sources. For example, soils can become elevated in P when manure is applied to meet the nitrogen needs of forages and crops (Sharpley et al., 1993). Past practices have led to elevated soil test P (STP) within some agricultural lands today, i.e., legacy P in soil. Thus, legacy P issues across the agricultural landscape may be an important non-point source within the watershed (Sharpley et al., 1993; Abrams and Jarrell, 1995; Eghball and Power, 1999; Agyin-Birikorang et al., 2007).

Water treatment residuals (WTRs) have been proposed as a mitigation technique for soils high in P. They are a byproduct of the drinking water treatment process and contain all the sediments, organic material, and particulates from the raw source water, along with the coagulation chemicals from the treatment process. Alum [Al₂(SO₄)₃] is the most common coagulant used in the U.S. and, in addition to removing solids from source water, can strongly adsorb P and greatly reduce its aqueous solubility (Elliot et al., 1990). Typically, liquid WTRs (~2% solids) are centrifuged by drinking water facilities to a solid form (~20% solids) and disposed of in landfills, but land application to high STP soils to reduce P release to the environment could be both a more economical and a more environmentally sound alternative (Makris et al., 2005).

Many studies have shown that WTRs have high P sorption capacities, and that WTR addition to soils reduces available STP (Dayton and Basta, 2005; Makris et al., 2005; Agyin-Birikorang et al., 2008). The application of solid WTRs to small plots significantly reduced the soluble reactive P (SRP) concentration in the collected runoff as well as the availability of P in treated soils (Gallimore et al., 1999; Dayton et al., 2003; Habibiandekordi et al., 2015). The magnitude

of the reduction in P availability in soils is dependent on the amount of WTRs incorporated (Novak and Watts, 2005). Thus, greater soil P levels require a larger quantity of solid WTRs to achieve the target reduction (Haustein et al., 2000).

Solid WTRs have been applied and incorporated into soil in large fields before planting (Agyinbirikorang et al., 2007) and precisely applied to small-scale plots (Gallimore et al., 1999; Dayton et al., 2003; Habibiandekordi et al., 2015), ensuring that WTRs were evenly distributed across the soil surface. However, the clumpy nature of solid WTRs may make uniform surface application difficult to achieve and less effective in controlling P release (Oladji et al., 2007). Liquid WTRs, however, could be more evenly distributed across the landscape. The liquid WTRs would essentially act as a cap on the soil, sealing the surface and reducing P release when inundated. Therefore, the objectives of this experiment are three-fold: 1) to show the variability of P release in fields treated with solid WTRs, 2) to determine an effective application rate of liquid WTRs, and 3) to evaluate the effectiveness of that application rate in reducing P release over several months.

Study Site Description

The Fayetteville Biosolids Management Site (FBMS) is on the eastern side of Fayetteville, AR, adjacent to the White River. The White River is a primary inflow into Beaver Lake, the drinking water supply of Northwest Arkansas. Beaver Water District (BWD) takes in water from Beaver Lake, treats the source water, and produces the WTRs used in this study.

The composition of the residuals depends on the contents of the source water and the amount of chemicals added to the water (Environmental Protection Agency, 2011), so composition can vary

some from batch to batch. Phosphorus content was less than 2 mg kg⁻¹ in all analyzed WTRs, while Mehlich-III aluminum and iron (M3Al and M3Fe, respectively) were nearly 2400 and 500 mg kg⁻¹, respectively, resulting in a P saturation ratio (PSR) of 0.0005. The small P content and large Al content suggest that Al will react with P in the soil and form insoluble compounds (Dayton and Basta, 2005), thus reducing the amount of soluble P that can be released from the soil during inundation.

The FBMS is a 271-ha site that includes Midland Bermuda fields and operational facilities, and the site is managed by the Paul R. Noland Wastewater Treatment Facility. Historically, the facility applied the residual sludge or biosolids to the land within the FBMS for nearly 15 years (September 1988 to June 2003), increasing the P content of the soils. The site now implements a nutrient uptake program growing and harvesting hay as a way to remove P from the soil. In November 2012, BWD began sending the solid WTRs to the FBMS where they have been land applied in some areas, at no more than 22,417 kg ha⁻¹ yr⁻¹ (ADEQ Permit 0000-WG-WR), to mitigate P release during rainfall and flood events. Soil cores were collected from a field which had been treated with solid WTRs for 5 years on top of the historic biosolids application. The soil within the sampled field was mapped as Savannah fine sandy loam (fine-loamy, siliceous, semi active, thermic Typic Fragiudults) (Web Soil Survey, 2017).

Soil cores were also collected at the Watershed Research and Education Center (WREC), a 95-ha plot of land in the headwaters of the Illinois River Watershed and within the Arkansas Agricultural Research and Extension Center in Fayetteville, Arkansas. WREC, established in 2006 consists of pastures and wetlands, and a stream runs through it which receives urban storm water and runoff from agricultural lands (e.g., row crops and pastures) within its catchment (Brion et al., 2011; Metrailor, 2012). It has been the site of water quality research, including studies focusing on land use effects (Brion et al., 2011) and watershed mass balance (Metrailor, 2012). Soils sampled in this research include Leaf silt loam at site 1 (fine, mixed, active, thermic Typic Albaquults), Cleora fine sandy loam at site 2 (coarse-loamy, mixed, active, thermic Fluventic Hapludolls), and Pickwick silt loam at site 3 (Fine-silty, mixed, semiactive, thermic Typic Paleudults) (Web Soil Survey, 2017). Within the study site, the STP ranges from very high regions (nearly 400 ppm) to fairly low STP (<50 ppm). Previous studies (e.g., Rossetti et al., 2014) have sampled at sites across this range of STP levels, measuring P release and WTR effects. These results are summarized in Reavis and Haggard (2016).

Methods

FBMS Field and Lab Experiment

A total of 12 soil cores were collected randomly within the ~13 ha field at FBMS which had been treated regularly with solid WTRs. Cores were collected by pushing a Plexiglass tube (7.6 cm inner diameter) 10 cm into the ground then carefully removing the tube and capping the bottom to prevent soil loss. Cores were transported to the lab where the caps were then sealed with electrical tape in order to hold water. Tap water was added to each core to an overlying volume of 1L, and aquarium pumps connected to plastic tubes and straws taped inside each core aerated the overlying water to simulate flood conditions and to prevent anaerobic processes from altering P release mechanisms. After flooding the cores, 30 mL water samples were taken from the middle of the overlying water at 0.5, 1, 2, 5, and 8 hours, filtered through 0.45 μ m filters into plastic scintillation vials, and acidified using 0.03 mL trace HCl. The volume of overlying water was maintained at 1L throughout the experiment by adding tap water after sampling. Each sample was analyzed for SRP using the ascorbic acid method (APHA, 1999) on a spectrophotometer (DU 730 Life Science UV/Vis). The SRP mass released into the overlying water was corrected for the mass removed during sampling and the addition of tap water added during water replacement each round.

Cores were drained at the conclusion of the experiment and the top 8 cm of soil were removed from each core in 2cm segments and placed into aluminum pans. The soils were dried in an oven at 105 °C and ground to pass through a 2mm sieve. Each soil sample was analyzed for Mehlich III and water extractable P, Fe, and Al at the University of Arkansas Soil Diagnostic Lab (see Zhang et al., 2014 for methods). P saturation ratios were then calculated on a molar basis for each core, and $PSR = \frac{M3P}{(M3Al+M3Fe)}$ (Kleinman and Sharpley, 2002).

SRP mass did not always increase linearly in each core during inundation. Sometimes SRP increased linearly over the entire incubation, and sometimes the increase was linear over only the first few samples. Slopes were calculated for the linear portions of data for each core and were divided by the surface area of the core to determine SRP release rate from soil. If the slope was not significant (*P*>0.05), the rate was assumed to be zero. Release rates were compared across treatments and STP content using analysis of covariance (ANCOVA) to determine significant differences ($P \le 0.05$).

Liquid WTR Application Experiment

Three locations were sampled within the boundaries of WREC, with STP content ranging from 77 to 282 mg kg⁻¹. Twenty-one soil cores were collected in the same manner as the first experiment from each location between June and August 2016. Three cores were kept as experimental controls while each of the remaining 18 received one of 6 different WTR

application rates (220, 440, 1320, 2200, 3100, or 4400 kg ha⁻¹). Water treatment residuals were obtained from BWD and were roughly 2% solids (98% water). To maintain consistency across all cores and treatments, water was added to each core so that the total volume of liquid added across all cores was consistent.

The next day, cores were flooded with 1L overlying tap water and sampled as previously described. Water samples were analyzed for SRP, and release rates calculated for each core. Release rates were compared across treatment rates and STP content using ANCOVA analysis to determine significant differences among treatments. The top 2 cm of soil from each core were collected and analyzed as previously discussed.

Repeated Flooding and Drying Experiment

A summer-long experiment was set up in May 2017 to evaluate how repeated rainfalls and flooding events influence WTR effectiveness over time. Eighteen cores were collected in the manner previously described from each of the two sites at WREC, and WTRs were added to four cores from each set at a rate of 440 kg ha⁻¹. Precipitation was monitored via two USGS stations (USGS 071948095 Mud Creek and USGS 07048495 Town Branch) that lay north and south, respectively, of WREC.

A volume of tap water equal to the amount it rained at WREC was added to each core within one day of the rainfall. When average precipitation in one storm was greater than 1.3 cm and flood conditions at WREC were possible, all cores in the lab were flooded with 1L overlying water and sampled as previously described. SRP was analyzed in each sample and release rates were calculated as previously described for each core over the course of each flooding cycle. After flooding, bottom caps were removed and the cores were allowed to drain naturally.

The cores were subjected to a total of six flooding events over the course of 2.5 months. Following the final sampling round, the cores were drained to collect a soil sample. The top 2 cm of soil were collected in Al pans as before. All soil samples were dried in an oven at 105 °C, ground, and delivered to the Soil Diagnostic Lab to be analyzed as before for Mehlich III and water extractable P, Fe, and Al.

Results

FBMS

The amount of WEP in the soils from the WTR-treated field at FBMS was highly variable across the field and with depth (Figure 1A), ranging from 5 to 34 mg kg⁻¹ and averaging 15 mg kg⁻¹. As soil profile depth increased, WEP tended to increase as well (data not shown). At a depth of 8 cm, WEP was often nearly twice what it was in the top 2 cm.

WEP was not related to M3P in the soils, and it was a small fraction (<1-6%) of M3P extracted from the soils. M3P was also highly variable across the field and with depth (Figure 1B), ranging from 430 to over 1400 mg kg⁻¹ and averaging 1100 mg kg⁻¹. As with WEP, M3P increased as soil depth increased (data not shown). From 2 to 8 cm, M3P increased as much as 370 mg kg⁻¹ (data not shown).

WEP and M3P often account for a substantial amount of the variability in SRP release from soil (Pote et al., 1996, 1999; Reavis and Haggard, 2016) and were therefore expected to have a significant relationship with SRP release rates. However, neither WEP nor M3P were

significantly related to SRP release (P=0.178 and 0.921, respectively). Release rates were variable, ranging from 2 to more than 14 mg m⁻² h⁻¹, and averaging 8 mg m⁻² h⁻¹ (Figure 1C).



Figure 1. (A) Boxplots of water extractable phosphorus (WEP) (top 2 cm), (B) and Mehlich-III P (M3P) (top 2 cm), and (C) soluble reactive P (SRP) release rate data from the Fayetteville Biosolids Management Site (FBMS) field site treated with water treatment residuals (WTRs).

WREC WTR Treatment Rates

M3P in control cores from site 1 at WREC ranged 192-274 mg kg⁻¹ with an average of 241 mg kg⁻¹. SRP concentrations in the overlying water of the controls increased from an initial concentration of 0.002 mg L⁻¹ to nearly 0.25 mg L⁻¹ during inundation. By contrast, SRP concentrations measured in the overlying water of WTR-treated cores were less than those in the controls, reaching an average concentration of 0.02 mg L⁻¹ and a maximum concentration of no more than 0.08 mg L⁻¹ in any WTR-treated core.

SRP release rates for the control at site 1 averaged more than 6 mg m⁻² h⁻¹, and all WTR treatments were significantly less than the control, ranging from zero to nearly 3 mg m⁻² h⁻¹

(Figure 2). Release rates tended to decrease as WTR treatment rate increased. SRP release from cores treated with the lowest WTR rate (220 kg ha⁻¹) was 2.75 mg mg⁻² h⁻¹, a 57% reduction compared to the control. At 440 kg ha⁻¹, SRP release was less than 1 mg m⁻² h⁻¹, an 85% reduction compared to the control. Release rates from the remaining treatment applications (1300, 2200, 3100, and 4400 kg ha⁻¹, respectively) were generally significantly less than the control and the lowest WTR application rate (Figure 2).

In control cores from site 2, STP ranged from 222 to 247 mg kg⁻¹ which was not very different from site 1. SRP concentrations in the overlying water of control cores reached 0.25 mg L⁻¹ throughout inundation. In WTR-treated cores, SRP concentrations reached a maximum of 0.03 mg L⁻¹ with an average of 0.004 mg L⁻¹ (Figure 2, Site 2).

SRP release rates in the control cores from site 2 were similar to those from site 1, averaging 5.0 mg m⁻² h⁻¹. Rates from WTR-treated cores were again significantly less compared to the control cores. The lowest application rate of 220 kg ha⁻¹ was highly effective and reduced SRP release by 90% (0.5 mg m⁻² h⁻¹). At 440 kg ha⁻¹, SRP release was significantly different from both the control and the lowest application rate, reducing SRP release rates by 98% compared to the control. SRP release rates in the remaining treatments were not significantly different from the 440 kg ha⁻¹ application rate.

STP in control cores from site 3 ranged from 168 to 218 mg kg⁻¹, which was not significantly different from sites 1 and 2. SRP in the overlying water of the cores reached concentrations of only 0.13 mg L⁻¹ with an average of 0.08 mg L⁻¹ and an average release rate of 2.7 mg m⁻² h⁻¹ from the control. Concentrations in the water above WTR treatments increased to a maximum of 0.05 mg L⁻¹ across treatments with an average of 0.005 mg L⁻¹. Like in other trials, SRP release

rates from control cores were significantly greater than from those treated with WTRs. However, there were no significant differences among WTR treatment rates.



Figure 2. Soluble reactive phosphorus (SRP) release rate from soil cores collected at three sites within the Watershed Research and Education Center (WREC). Bars show standard error about each mean, and letters above each bar denote significant differences in SRP release rates within each graph.

Multiple Flooding and Drying Cycles

STP in cores from site 1 ranged from 105 to 233 mg kg⁻¹ with a mean of 177 mg kg⁻¹ which was significantly greater than STP in cores from site 2 which ranged 60 to 110 mg kg⁻¹ with a mean of 87 mg kg⁻¹ (Figure 3). Average SRP release rates were greatest from control cores at high STP, reaching more than 1.4 mg m⁻² h⁻¹. Release rates from WTR-treated cores at high STP were roughly 20-60% of control cores at the same STP level for most of the summer, but eventually increased to 1.5 mg m⁻² h⁻¹ which was not significantly different from the control.

The pattern of SRP release in cores with low STP was similar to that at high STP (Figure 3). Average SRP release rates from control cores throughout the summer at low STP were roughly half of those from cores at high STP. At low STP, WTR treatment was highly effective, as P release from the treated cores was not different from zero throughout the duration of the experiment.



Figure 3. Average soluble reactive phosphorus (SRP) release rate from the Watershed Research and Education Center (WREC) at high (177 mg kg⁻¹) and low (87 mg kg⁻¹) Mehlich-III P (M3P) levels under WTR-treated and control conditions. Bars show standard error about each mean.

Discussion

The WTRs used in our experiments and at FBMS were alum-based with a high P sorption capacity (based on the low PSR). The P adsorption capacity of WTRs increases with increasing Al content (Dayton et and Basta, 2005) and decreasing PSR (Elliot et al., 2002). This suggests that these WTRs should be effective at reducing extractable P in soils (e.g., Novak and Watts, 2004, 2005; Olaji et al., 2007), thereby reducing the potential for SRP release from soil during runoff (Gallimore et al., 1999; Haustein et al., 2000; Wagner et al., 2008) or flood events (Reavis and Haggard, 2016). The FBMS field had received solid WTRs for the last 5 years in an attempt to reduce STP that had accumulated with years of historical biosolids application. However, STP at the soil surface was still quite high and variable (260-1450 mg kg⁻¹). The application of solid WTRs was apparently not effective at reducing STP across the entire field. The potential for P release to flood water at relatively high rates (up to 15 mg m⁻² h⁻¹) still existed.

The literature suggests that WTRs are highly effective at reducing the solubility of P in soils and, thus, its release to either floodwater or runoff water (Gallimore et al., 1999; Dayton et al., 2003; Agyin-Birikorang et al., 2008; Habibiandekordi et al., 2015). Most of these studies have been conducted in lab settings or on small plots where residuals can be precisely and rather uniformly applied across the soil surface. However, based on the variability observed at FBMS, it is difficult to have uniform application of solid WTRs at the field scale. The solid WTRs are often clumpy in nature and, when applied, will clump together in some areas and leave other areas exposed, resulting in the variability observed in this field.

When applying WTRs at the field level, liquid WTRs may more easily and uniformly cover the soil surface. In our core experiments, liquid WTRs were highly effective at reducing SRP release at rates much smaller than the maximum permitted land application rate (22,417 kg ha⁻¹) at FBMS fields. These liquid WTRs were approximately 2% solids, and Haustein et al. (2000) observed that WTRs with 8% solids were able to uniformly cover the small plots. If more uniform coverage is achieved when applying liquid WTRs, then this might be a better management alternative than solid WTRs at P hot spots within a watershed.

While liquid WTRs were highly effective at reducing SRP release rates in our cores, there are some challenges to large-scale application. One of the biggest challenges would be transportation

because the liquid WTRs are mostly water; the WTRs used in this study were ~2% solids. For example, one company estimated that transporting residuals with 6% solids compared to 45% would quadruple the cost (Big Fish Environmental, 2009). The additional water transported to the fields might, however, have some benefit to forage growth (at FMBS), depending on application timing and antecedent conditions.

The amount of liquid WTRs needed to reduce SRP release may be significantly less than the amount of solids needed. Based on our experiments, substantial reduction in SRP release could be achieved with 440 kg ha⁻¹ liquid WTRs on soils with M3P less than 250 mg kg⁻¹, which is roughly 2% of the rate applied regularly at FBMS. This rate is smaller than any solid application rate seen in the literature and was just as effective at similar STP levels (Haustein et al., 2000; Wagner et al., 2008).

Despite this, the additional transportation costs might limit the use of liquid WTRs across large fields and throughout the watersheds. With a 11,356 L truck, application of liquid WTRs (2% solids) at 440 kg ha⁻¹ could cover roughly 0.5 ha. To cover a large area with liquid WTRs would be far more expensive and time consuming than covering the same area with solid WTRs. However, broadcast spreading of liquid WTRs across an entire field is not the only option for high soil P mitigation. Gallimore et al. (1999) found that a two-meter buffer strip of uniformly spread WTRs was successful in reducing runoff dissolved P in plot studies. Small plots, however, have uniform slopes and surfaces, and one potential drawback of field borders and buffers is preferential flow paths. These flow paths might need larger WTR application rates or broader areal coverage to effectively remove P from overlying waters.

An alternative, then, could be a hybrid approach of broadcasting solid WTRs and adding a buffer strip of liquid WTRs around the perimeter of the treatment area. This approach would keep the cost of transporting residuals down by using solids for the larger areas. The border of liquid WTRs would remove more dissolved P not captured by the solid residuals on the field.

This hybrid approach could also extend the effectiveness of WTRs over time. Liquid application at a fraction of typical rates was effective at controlling SRP release for more than 2 months at STP less than 100 mg kg⁻¹. At higher STP levels, slightly greater application rates of liquid WTRs would increase their sorption capacity and their longevity (Gallimore et al., 1999; Haustein et al., 2000; Novak and Watts, 2004). Increasing the application rate would not be practical on a large scale, but would be more feasible if applied as a buffer strip. Application of large quantities of solid WTRs has been shown to be effective for up to 7 years (Agyin-Birikorang et al. ,2007), thus, solid WTR application would still be required when the goal is to control SRP release over a large area, but because some P would be sorbed by the solid WTRs on the field before reaching the buffer strip, the liquid residuals in the buffer would also maintain their effectiveness for longer.

Conclusion

WTRs have often been used to mitigate P release from soils high in STP. Land-applied WTRs are in the solid form, but few studies have investigated the application of liquid WTRs.

• When examining a field treated with solid WTRs (~20% solids), we found that SRP release rates and STP were highly variable across the field.

- Liquid WTRs (~2% solids), however, covered the soil more evenly, and an application rate of only 440 kg ha⁻¹ was necessary to effectively control SRP release from flooded soils.
- Additionally, this small application rate effectively controlled SRP release for the entire experiment when STP was low, but was only effective for a couple months when STP was high, so application rates should increase as STP increases in order to maintain effectiveness.

There are challenges associated with using liquid WTRs (i.e., increased transportation costs), but our experiments found them to be highly effective at reducing SRP release rates. In conjunction with the use of solid WTRs, liquid WTRs may have a valuable role to play in mitigating P release from hotspots within watersheds.

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Conclusion

This research evaluated the temporal variability of P release from flooded soils and ways to manage that release using water treatment residuals. P release was found to change significantly over the year, rather than varying based on soil P content as expected. Release rates were greatest during the fall and smallest during spring. While the literature suggested that soil moisture plays a role in P flux, there seemed to be no relationship between soil moisture or flood events with P release rates from the soils.

We saw that there was P released from these soils throughout the year, and using WTRs is an inexpensive and convenient way to mitigate P release. Land applying solid WTRs can result in variable coverage across the soil surface, but applying liquid WTRs at even small amounts was shown to substantially reduce P release from the soils. Liquid WTRs would be valuable when used in smaller areas that are P hotspots within the watershed. Over larger areas, they can be applied in conjunction with solid WTRs to effectively control P release from soils with large amounts of P stored within them.