

ABSTRACT

JAMESON, MOLLY CHRISTINE. Determination of Biosolids Phosphorus Solubility and Its Relationship to Wastewater Treatment. (Under the direction of Jeffrey G. White and Deanna Osmond).

Biosolids (BS) are the largely organic, often nutrient-rich, solid or semisolid residuals of wastewater treatment. Sewage sludge becomes BS when treated to meet federal and state standards for beneficial reuse via land application as a fertilizer and soil amendment. In North Carolina (NC) and elsewhere, land application is currently governed by BS plant-available N and the agronomic N needs of the crop on the receiving soil. Because of the differences between BS and crop N:P, N-based BS applications typically surpass crop P needs, create excess soil P, and increase surface water pollution potential. The USDA Natural Resources Conservation Service Nutrient Management Conservation Practice Standard mandates P budgets, and in many cases, P-loss risk assessments. Many states have P-based guidelines, and the NC Department of Environment and Natural Resources is considering guidelines for some nutrient-impaired watersheds. In NC, the P Loss Assessment Tool (PLAT) will likely be used to estimate BS P-loss risk. Forms and quantities of P in BS depend on wastewater treatment plant (WWTP) processes. We surveyed 26 NC WWTP to characterize BS production processes. In summer and fall 2013 and winter 2014, we sampled 28 BS from these 26 WWTPs and analyzed their total P (TP), water-extractable P (WEP), and percent DM, and quantified percent water-extractable P (PWEP) and nonsoluble P. The TP in BS varied considerably depending on type of BS and treatment process, 0.4 to 73.2 g kg⁻¹. The average TP in Class A alkaline stabilized BS was more than five times less than the average of the other BS, 5.0 vs. 26.6 g kg⁻¹, respectively. Total P varied seasonally: averaged over BS, winter TP was greater than both summer and fall TP, 31.3 vs. 17.8 and 21.8 g kg⁻¹,

respectively. We cannot definitively conclude that these differences were due to change in seasons, and this deserves further exploration. Biosolids WEP ranged from 0.02 to 9.1 g kg⁻¹ and PWEP from 0.2 to 35.2%. Averaged over BS, WEP and PWEP were 1.4 g kg⁻¹ and 5.0%, respectively. Biosolids PWEP was relatively low compared to other nutrient sources such as triple superphosphate (100%) and manures (e.g., 25 - 85%). Percent water-extractable P and WEP were strongly correlated ($r^2 = 0.89$). We developed BS categories for implementation in PLAT based on the relationships of P with treatment processes, including “Class A-alkaline,” “Class A-heat,” “Class B-slurry,” and “Class B-cake.” We concluded that stabilization processes used to meet pathogen reduction requirements reduced the soluble forms of P in BS substantially. Dissolved P in runoff has been found to be controlled by P solubility, so the potential for soluble P loss from land-applied NC BS is low. Understanding the potential environmental implications of land applying BS will be a function not only of the type of BS being applied, but also the application rate, method of application, and site-specific factors. Implementing our recommended BS categories in PLAT would allow the tool to be used when BS are applied, thus BS application rates could be based on both N and/or P as appropriate. Relative to N-based BS applications, basing them on P would likely decrease application rates, reduce the number of eligible receiving fields, and shorten the time soils can receive BS, thus making land application more costly. Hence, widespread adoption of a P-based approach may foster development of alternative beneficial uses of BS other than land application.

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Determination of Biosolids Phosphorus Solubility and Its Relationship to Wastewater
Treatment

by
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BIOGRAPHY

Molly Jameson was born in Savannah, GA; spent her early childhood in Lawrenceville, GA; and most of her adulthood in Tallahassee, FL. Early on, you could most likely find her outside playing in the woods, where she developed a deep appreciation for nature and the environment. In 2007, she pursued her undergraduate degree at the University of Florida in Horticultural Science with a specialization in Organic Crop Production. Upon graduation, she spent three years on a small family farm in Tallahassee called Turkey Hill, which cultivated her appreciation and passion for sustainable agriculture. It was through her work on the farm that she became aware of how important it is for a community to be connected to the food they eat, and to hold respect for soil, water, and species diversity. She subsequently got accepted into North Carolina State University, where she is pursuing her Master of Science degree in Soil Science.

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CHAPTER 1: LITERATURE REVIEW

Introduction: Defining Biosolids

Before the Clean Water Act (CWA) was amended in 1987, US cities disposed of their waste by discharging it directly into oceans and freshwater systems (USEPA, 1994a). These amendments prohibited this practice, making it necessary for Wastewater Treatment Plants (WWTPs) around the country to treat the waste so that it could be more responsibly managed (USEPA, 2002a). This meant that sewage sludge, defined as the untreated solid residuals that are separated from wastewater at WWTPs, needed to have set standards for treatment so that environmental impacts could be minimized when land applied. In 1993, these standards were developed as part of the US Environmental Protection Agency (EPA) Title 40 Code of Federal Regulation (CFR) Part 503 Rule revisions under Section 405 (d) of the CWA (USEPA, 1994a), aimed at reducing the level of pathogens, vector attractants, potentially toxic metals, and N loads within the sludge.

Once residuals are treated to meet these standards, they are commonly referred to as “biosolids” (BS). This term was coined in 1991 by the National Biosolids Partnership, which is operated by the Water Environment Federation (WEF). They define BS as, “the nutrient-rich organic materials resulting from the treatment of domestic sewage at a wastewater treatment facility” (WEF, 2005). The US EPA defined BS in 2005 as, “the nutrient-rich organic materials resulting from the treatment of sewage sludge (the name for the solid, semisolid or liquid untreated residue generated during the treatment of domestic sewage in a

treatment facility). When treated and processed, sewage sludge becomes biosolids which can be safely recycled and applied as fertilizer to sustainably improve and maintain productive soils and stimulate plant growth” (USEPA, 2005).

Due to their high organic matter content and high levels of nutrients, BS are considered a useful soil amendment and fertilizer source that can improve several soil properties, including increased water retention and decreased erosion rates. Government-sponsored research has been done on the land application of BS, including an extensive risk assessment study performed by the USEPA (USEPA, 1995). This risk assessment included the identification of chemicals likely to pose hazards, and included scientifically calculated concentration limits and loading rates of chemicals applied to land. In 2002, the National Research Council (NRC) of the National Academy of Sciences concluded that there “is no documented scientific evidence that the Part 503 Rule has failed to protect the public health.” They did, however, conclude that it was critical to update the scientific basis of the regulations to assure the public of their safety and to protect public health (National Research Council, 2002). In a 2012 article from the New England Water Environment Association journal about current BS use, it was noted that the National Academy of Sciences recent reviews still find that the land application of BS sensibly preserves public health and the environment, and the EPA has made no changes to 40 CFR Part 503. However, there have been discussions of minor changes to the rule, such as removing some options of pathogen reduction methods and updating BS analytical references. Even so, EPA reduced the budget

and staff for BS monitoring by the Office of Enforcement and Compliance Assurance (Beecher, 2012).

Unsurprisingly, there are still sectors of the public that oppose the land application of BS due to concerns over potentially toxic metals, pathogens, organic chemical pollutants, odors, and a lack of adequate risk assessment. The Sustainable Land Application conference in Orlando, Florida in 2004 addressed many of these concerns (Elliott and O'Connor, 2006). Since 2004, there have been multiple publications addressing such issues. For instance, Ippolito and Barbarick (2008) examined trace elements in BS, Overcash et al. (2005) examined concerns over organic chemicals, Godfree and Farrell (2005) looked at potential BS pathogens, Schiffman and Williams (2005) studied BS odors, Kester et al. (2005) administered a risk assessment, and Beecher et al. (2005) and Robinson et al. (2012) studied BS public perception. Although Robinson et al. (2012) notes successful BS application programs must involve stakeholders in decision-making processes, these scientific assessments dismissed many common negative perceptions concerning BS. Unfortunately, the hardest challenge facing the land application of BS may be something that in the past has been considered a major benefit: high nutrient levels.

Current Management: Uses and Challenges

Under the authority of the 40 CFR Part 503 Rule, BS land application rates have been traditionally regulated following the N requirements necessary to meet the optimum growth for crops, while aiming to avoid N runoff and leaching into the groundwater. The problem

with this regulation standard is that it largely ignores risks imposed by P. Biosolids that are land applied supply P in excess of crop needs for optimum growth. The N:P ratio of crops is in the range of 3:1 to 8:1. Contrastingly, BS N:P ratio is typically in the range of 2:1, 1:1, or even 1:2 (Shaffer et al., 2008), which makes BS an unbalanced nutrient source. Additionally, these ratios are lower than most manures (such as swine and broiler litter) and many inorganic fertilizers. Years of repetitive N-based applications of soil amendments with such imbalances of nutrients, such as BS, can lead to high P concentrations within the soil profile. Consequently, this results in BS applications greatly surpassing crop P needs, which increases the soil P levels, which in turn increases the potential for losses into the environment. Maguire et al. (2001) found that soils that received BS on an N-requirement basis had application rates of 135 kg P ha^{-1} , while the crop harvested only removed between 15 and 40 kg P ha^{-1} , suggesting that crop uptake is not enough to remove additions of P from BS. O'Connor et al. (2004) stated that most crops remove 10 to 40 kg P ha^{-1} , compared with 100 to 300 kg N ha^{-1} . Therefore, land applications of BS with a low N:P will mean much of the applied P will be left behind after crop removal.

This concern is further exacerbated as discharge limitations of effluent P from wastewater, which typically are around 1.0 mg L^{-1} (WEF, 2005), get increasingly more stringent, affording the likelihood of more P entering the sludge treatment processes (Stehouwer et al., 2000), and ultimately into the final BS product. Additionally, there will be a higher proportion of P rather than N in BS since N has the ability to denitrify to atmospheric nitrogen (N_2) and volatilize as ammonia (NH_3) during treatment processes such

as thermal drying (Smith and Durham, 2002), while P cannot readily change into gaseous forms.

As effluent limits get stricter and WWTPs channel more P loads to sludge treatment, it is important to understand what forms of P are at the greatest risk of being lost to the environment. These fractions will be most important to monitor when land applying BS, making it imperative to understand the basics of the P cycle, and how it affects P-loss risk. Phosphorus most often can be found as part of phosphate, or PO_4 . There are organic and inorganic P compounds that are soluble in water. Organic P is associated with C molecules, while inorganic P is not. Although both organic and inorganic P can be soluble or insoluble, soluble inorganic P such as PO_4^{3-} , HPO_4^{2-} , H_2PO_4^- , and H_3PO_4 is the predominant form that plants take up. If inorganic P is adsorbed to soil particles or it complexes with other minerals, such as with Fe or Al, it becomes insoluble (Hyland et al., 2005).

The typical form of P that can be lost to the environment is the soluble inorganic orthophosphate. When plants take up orthophosphate they convert it to organic P in their tissues, which eventually will be mineralized back to inorganic P by bacterial decomposition when the plant dies. Orthophosphates, such as PO_4^{3-} , HPO_4^{2-} , H_2PO_4^- , and H_3PO_4 species, will move via streams, lakes, and rivers, unless they are taken up by plants or adsorbed to soil that settles. This excess orthophosphate can lead to what is known as eutrophication.

Eutrophication is a natural phenomenon that occurs when dissolved nutrients such as N and P enter waterways, causing increased levels of algal and other aquatic plant growth

(WEF, 2006). Phosphorus is normally the nutrient in shortest supply in natural freshwater ecosystems. Therefore, even very small additions can have drastic effects. The transport mechanisms by which P can move into waterways include pathways such as P attached to particles of soil (sediment P), dissolved P (orthophosphate) in runoff moving across the soil surface, leached P (orthophosphate) moving down through subsurface drainage, or the direct loss of P, in multiple forms, from the nutrient source moving into water via runoff (Shaffer et al., 2008). In areas that are intensively farmed and receive excessive applications of BS or any other type of P-containing soil amendment, soluble P concentrations can exceed critical levels and are therefore capable of causing eutrophication of proximal water bodies.

The downward movement of P, especially in locations with shallow ground water and coarse-textured soils with low P sorption capabilities, can concentrate P, surpassing natural levels of the ecosystem (Elliott et al., 2002). Phosphorus introduced from point (e.g., wastewater treatment) and non-point sources (e.g., animal manures) can exacerbate the severity of eutrophication, which can lead to dense growths of algae and other aquatic vegetation. Once these die and are decomposed by microorganisms, organic P can be mineralized into inorganic orthophosphate. These processes require oxygen, depleting the aqueous concentration and subsequently leading to fish suffocation, as well as inhibiting productivity of other aquatic life (WEF, 2006).

According to EPA water quality criteria, total P (TP) should not exceed 0.01 to 0.03 mg L⁻¹ to avoid eutrophication risks (Osmond et al., 1995). Therefore, concentrations that

exceed this value can become a serious concern. The “National Manual of Good Practice” states that if soluble P concentrations exceed $0.01 \text{ mg L}^{-1} \text{ P}$, it can cause algal blooms in fresh water ecosystems (WEF, 2005). To go even further, it is noted in the WEF’s Manual of Practice for Biological Nutrient Removal (BNR) Operation in Wastewater Treatment Plants (2006), that soluble P concentrations, in the form orthophosphate, should not exceed 0.005 mg L^{-1} to limit the threat of eutrophication.

The National Pollutant Discharge Elimination System (NPDES) is a program established by the federal government for controlling point-source wastewater discharge in an effort to improve water quality within the US. The CWA requires states to monitor the health of their water bodies and report these assessments to the EPA every two years. These findings are called the "biennial water quality report," or the Threatened and Impaired Waters List (303(d)) (USEPA, 2012b). The NPDES permit program is therefore often administered on a state level, in which total maximum daily loads (TMDLs) are required for water bodies on the 303(d) list, with the EPA conducting facility compliance inspections. Therefore, there are no nationally specified effluent concentration limits, so maximum WWTP effluent limits vary state-to-state (USEPA, 2012a).

Effluent Limits in North Carolina

All effluent discharge concentration limits within the state of North Carolina (NC) are relative to the receiving water body. Discharge facilities within the state must be in compliance with effluent discharge permit requirements dictated by the NPDES, and also be

in compliance with effluent limits dictated by their respective watershed. Wastewater treatment plants are often grouped into associations that can influence the P discharge limits for the area (E. Hardee/NC DENR, personal communication, 2014). These limits can depend on location, nutrient sensitivity of surrounding water bodies, and flow rate of the facility. Many water bodies in the state have nutrient strategies, which enact certain N and P controls affecting facilities in the area. Total maximum daily loads and P concentration (mg/L) limits are then allocated to certain discharge facilities. For example, the NC Neuse River Sensitive Waters Management Strategy Rules that were adopted by the Environmental Management Commission in 1997 specified that WWTP P effluent limits should not exceed an average of 2 mg P L^{-1} , or 1 mg P L^{-1} if the WWTP is expanded, within the vicinity of this nutrient sensitive watershed (NC PLAT Committee, 2005).

In 2009, there were 1,314 active NPDES wastewater discharge permits in NC. The Triangle J Regional Council of Governments selected 38 discharge permits in the Neuse River watershed to estimate annual TP loads (Hanson, 2011). Twenty-nine of the 38 were municipal wastewater dischargers, and the remainders were industrial and commercial wastewater dischargers. They found that loading from individual dischargers ranged from 0.45 to $75,437 \text{ kg year}^{-1}$, and the TP load from the 38 dischargers was $150,721 \text{ kg year}^{-1}$. Since not all NPDES permitted dischargers are required to monitor and report nutrient concentrations, the total nutrients released from these sites remains unknown (NCDWQ, 2012).

Implications of Phosphorus-Based Biosolids Land Application

Traditional BS land application aims to avoid N runoff and leaching into groundwater by following USEPA land application regulations based on crop N requirements. This means land applied BS supplies P in excess of optimum growth of the crop, as the N:P ratio crops require is much higher than the BS N:P ratio (O'Connor et al., 2004). If BS nutrient management were to be based on crop P removal, this will involve decreasing total BS application rates, when compared to current management regulated strictly on an N basis. Additional commercial N fertilizer will be necessary to make up the difference and greater land area will likely be needed to receive BS, leading to increased transportation costs.

More states around the country have, or are currently developing, more stringent P-based nutrient management plans for BS land applications, which may lead to a decreased rate in allowable applications. The nutrient management plans could require changes to existing BS production practices, or to WWTPs abandoning their BS land application programs all together.

For example, when the Arkansas Department of Environmental Quality changed their regulations in 2003, the city of Fayetteville, Arkansas was forced to change its land application strategies to include P agronomic rate limitations. They therefore stopped land applying their Class B liquid BS and instead used landfills for disposal (City of Fayetteville, Arkansas, 2014). This exemplifies the fact that changes in nutrient management to include P-loss risk will cause WWTPs to rethink their BS disposal options. In examining the reason

Fayetteville disbanded their program, it is important to look at what changed in Arkansas's BS regulations. They acknowledged that P-based management was necessary, and application limits based on P were employed for manures and BS on sites with excessive nutrients. Nutrient management plans are not a requirement in Arkansas, but they do have stricter BS management practices that are not dictated by Part 503, such as increased surface water and drinking water set-backs, and elevated slope restrictions (Beecher, 2007). In 2010, Fayetteville installed a solar dryer system, where about 95% of all energy needed to dry their BS comes from thermal heat via the sun. They can now land-apply part of their Class A BS on a 670 acre Biosolids Management Site on site for hay production, and also sell to local farmers and residents (City of Fayetteville, Arkansas, 2014).

The variability of P solubility within BS has been shown to depend on production methods (Brandt et al., 2004) and can have a major influence on whether the BS pose an environmental risk. This leads to the importance of accounting for this variability when regulating land applications of BS, making it necessary to evaluate BS on an individual WWTP basis in order to understand what treatment processes affect P distributions. Waste water treatment plants that use certain treatment strategies that may reduce BS P-loss risk, such as lessening the mobility of P within the treated sludge employing a variety of biological, chemical, and physical processes, could therefore have lower soluble P concentrations after sludge treatment. Since addressing the loss of P in BS is becoming more imperative as concerns increase for high soil test P (STP) and subsequent water contamination, it may prove beneficial for WWTPs to employ these different strategies to

affirm their BS will be have low soluble P, and therefore remain a usable soil enhancement in land application. For instance, if BS production practices add large quantities of P-precipitating Al, Fe, and Ca, they may lessen the P losses to runoff or leaching (Maguire et al., 2001).

From an environmental standpoint, when BS applications are N-based, successive low N:P BS applications cause P to build up in soil. If BS land application rates become P-limited, it will limit land application of BS high in P (Elliott and O'Connor, 2006). In turn, if WWTPs use treatments to decrease the soluble P within their BS, land appliers may no longer find it economically feasible to use the BS, as they will need to seek a higher plant-available P source. It is in this regard that defining P availability within BS and developing the best research-based P nutrient management plans are imperative.

There are other options of BS disposal, but each involves their own set of disadvantages. These options include incineration, landfilling, and composting. There are certain differences in regulation for each disposal option. For instance, land applied incinerated ash or ash which is not disposed of in a landfill, will be subject to Part 257, which covers the criteria for classification of solid waste disposal, instead of the standards for the use or disposal of sewage sludge, Part 503. If sewage sludge or ash is destined for a landfill, it follows the criteria for municipal solid waste landfills, Part 258 (USEPA, 1994a), as opposed to Part 503. Although landfilling BS has in the past been a popular disposal method, it can be a cause of leachate pollution and can emit the greenhouse gas, methane. Methane

production in anaerobic digestion can provide useable energy for a WWTP, but involves careful management and capture of the gas (Wang et al., 2008). Incinerating BS can produce ash that has been used in the production of construction materials and fuels, but this process can consume more energy than it actually produces due to very high moisture levels within the BS, and can still be a potential nutrient concern when land applied. Composting BS falls under Part 503 and can be a beneficial option for BS disposal, including easy handling, storage, and nutrient and soil conditioning value. Disadvantages to composting BS can include potentially inadequate pathogen control, odor production, lack of consistency, need for a bulking agent, and substantial effort required for turning and curing (USEPA, 2002b).

Due to the complexities involved in any BS disposal option, it is also important for the producer to develop multiple reuse options, as regulatory pressure may dictate changes down the road. Because land applying BS can be a very efficient and affordable reuse option in recycling nutrients and organic C, it continues to be a preferred method of disposal, making proper management techniques, dictated by the BS source, even more imperative. Elliott and O'Connor (2006) proposed a set of principles for the long-term viability of recycling BS via land application limited by P. These principles include setting environmental risk STP benchmarks, protecting vulnerable watersheds using adequately monitored buffer zones, accounting for P-loss risk differences by BS type, and recognizing fertilizer value variability by BS type.

Understanding such principles and developing site specific nutrient management plans when land applying BS can potentially lower P-loss impact. Allowing the application of BS in accordance with soluble P values that fit site requirements and limiting how much BS P is applied based on soil test analyses and crop specifications will assist in optimizing plant P-use efficiency and minimize water pollution.

Phosphorus Index Concept

Due to over application of P-containing amendments and lack of P management for agricultural lands, states need to better address P-loss from agricultural fields into nutrient sensitive watersheds, and site assessment tools must be implemented (McDowell et al., 2001). Each state's United States Department of Agriculture Natural Resources Conservation Service (USDA-NRCS) office was required to develop the Nutrient Management Code 590 Practice Standard, in which an appropriate nutrient budget for N, P, and K had to be determined when writing a nutrient management plan. Development of the USDA-NRCS Nutrient Management Standard was in part driven by the Unified Strategy for Animal Feeding Operations to ensure balanced nutrient applications (USEPA, 1999). In addition, the USDA-NRCS 590 Nutrient Management Standard must be used to develop a nutrient management plan when state or federal cost-share funds are received. In NC, nutrient management plans that meet the USDA-NRCS 590 Standard are required for farm operations where liquid dairy or swine waste is collected and applied, and the animal populations are \geq 250 swine or 100 cows (Johnson et al., 2005). Phosphorus site assessment indices, or "P

indices,” was a tool designed for nutrient management planners to rank the relative susceptibility of agricultural lands to P loss. A P index evaluates factors that contribute to P-loss such as physical site characteristics and the potential for off-site P transport and P-loss potential that is a result of management practices and P-source characteristics (Coale et al., 2005), such as related to BS production methods. Phosphorus indices originated as an on-site scoring matrix, employing multiple source and transport factors to identify sites most vulnerable to runoff and leaching P-loss risk (Elliott and O’Connor, 2006). In some states, changes in Code 590 and inclusion of the P Index led to regulatory changes for BS; in addition to meeting USEPA standards, BS application had to be in accordance with the NRCS 590 Standard (Sharpley et al., 2003). These changes may dramatically alter application rates of BS.

Many states use the P Index to be in compliance with the Code 590 nutrient management standards. By examining the methods of soil and crop management and the corresponding P status of the receiving soil, the P index can describe the potential within the site for runoff, leaching, and erosion into a receiving watershed (Gburek et al., 2000). Therefore, accuracy of the P Index for predicting potential P loss relies on correct quantification of susceptibility to runoff and leaching problems within a particular area, taking into account local conditions, such as climate, geomorphology, and hydrology (Elliott and O’Connor, 2006). Stringent limits on the distance required from surface waters, slope controls, and soluble/TP data monitoring will help control P-loss when land applying BS (Beecher, 2007).

Maryland is an example of state-mandated BS application based on both N and P. Nutrient management and nutrient standards are stricter in this state than NRCS Code 590. Following the enactment of Maryland's 1998 Water Quality Improvement Act, it was mandated that after July 2005, all land-applied nutrients must include a nutrient management plan regulating both N and P, with guidance from a certified nutrient management planner. This legislation treated BS, fertilizers, and manures identically (Shober and Sims, 2003). For land-applied Class B BS, Maryland requires seasonal soil tests and soil tests preceding new nutrient management plans.

Florida is another state that passed legislation requiring that BS land application in specific nutrient sensitive locations be based on both N and P. In the Everglades Protection Area, they require the implementation of measures to prevent degrading water quality by cautious monitoring of STP, P concentrations within the applied source, crop P uptake, soil P retention, and slope calculations. There are other states that have varying levels of BS land application regulations, guidelines, or have researched P loss potential, but there are many inconsistencies and gaps in communication amongst state officials and those involved in research efforts to protect the environment (Shober and Sims, 2003). Since the 40 CFR Part 503 Rule, the federal framework of BS management has remained unchanged for over 20 years. Many states continue to enact additional BS guidelines independently, reducing their reliance on the EPA regulations (Beecher, 2012). This adds variability and complexity to land application strategies, as influenced by environmental risk and public perception.

Measuring Phosphorus Loss Potential – Water Extractable Phosphorus vs. Total Phosphorus

Current research suggests that measurements of TP in land applied residuals are not sufficient in determining the form of P that will be significant in contributing to P-loss risk and subsequent environmental degradation. Measuring concentrations of what is commonly referred to as water-extractable P (WEP) has been shown to be strongly correlated with runoff and leaching of P from soils amended with animal manures (Kleinman et al., 2002). Water-extractable P can therefore be used as a reliable indicator of P-loss into the environment from land applied BS (Kleinman et al., 2002). Water extractable P, which estimates dissolved inorganic orthophosphate, comprises the majority of P that is soluble. Although there are particulate P losses, the nonsoluble P in soil amendments are considered to be more likely to stay adsorbed to the surfaces of mineral soil particles, providing a long-term P supply (Johnson et al., 2005). It is possible, however, that nonsoluble P can contribute to runoff P if sufficient BS solids stays on the soil surface and runoff velocity is sufficient to transport them (NC PLAT Committee, 2005). As discussed by Elliott et al. (2006), runoff P concentrations can vary drastically, depending on the source of P that is land-applied, even when TP loading rates are equivalent. It was observed in soils treated with poultry litter that P runoff concentrations were almost three times lower when the same litter was treated with Al sulfate (alum), which reduced the water soluble portion of P within the litter (Moore et al., 2000). However, there has been limited research conducted on WEP from BS destined for

land application. Many of the studies included both BS and manure, and many did not include information concerning the source of the BS or how they were produced.

In order to distinguish nutrient fertility sources of P by their relative potential to be lost to the environment via runoff, a weighting factor, the P source coefficient (PSC), has been developed. In 2004, nutrient fertility faculty from the Mid-Atlantic Region and the Ohio River Valley decided to increase consistency among regional P index tools. Participants included the states of Delaware, Maryland, Pennsylvania, Virginia, West Virginia, as well as the District of Columbia, the Delaware River Basin Commission, and the Ohio River Valley Sanitation Commission. They created a table of consistent PSCs on a scale between zero and 1.0, where biological P removal (BPR) and BNR BS received a PSC of 0.8 and all other BS a value of 0.4. Inorganic fertilizer and swine manure PSCs were given values of 1.0, while other manures and alum-treated manures were given PSC of 0.8 and 0.4, respectively (Coale, et al., 2005). A minimum PSC of 0.1 was enforced for all amendments regardless of WEP, as all sources are positive contributions to P source within a P index system. Also, all land-applied amendments with WEP above 9 g P kg^{-1} were given a PSC of 1.0. Should a BS applicator choose not to rely on this “book value,” a uniform procedure was defined to determine a PSC based on the WEP of the BS to be applied (Coale et al, 2005). In the Pennsylvania P Index there are six PSC categories of BS, including BS produced by BPR, alkaline stabilization, conventional stabilization, composting, heat-drying, and advanced alkaline stabilization. These were given a PSC of 0.8, 0.4, 0.3, 0.3, 0.2, and 0.2, respectively. The PSCs were determined using runoff and laboratory studies, such as those conducted by

Brandt et al. (2004) and Elliott et al. (2005; 2006). Elliott et al. (2006) found that when Pennsylvania P Index PSCs were multiplied by the TP application rate, it represented the applied P that was susceptible for transport off-site. In the absence of the PSC for a particular BS, runoff dissolved P and TP were not correlated ($r^2 = 0.02$) with total applied P. But when the application rates of BS were multiplied by their respective PSC, dissolved and TP were strongly correlated ($r^2 = 0.71 - 0.80$) with total applied P (Elliott et al., 2006).

Elliott et al. (2006) described that using the generic Pennsylvania P Index PSCs in determining the potential for P runoff was not always consistent, as treatment processes from various WWTPs had WEP values that varied significantly. They, therefore, compiled a substantial amount of literature in order to show the role WEP plays in determining potential for P runoff from applied materials. This was done via a meta-analysis of known studies that were in accordance with National Phosphorus Research Project (NPRP) protocol guidelines (NPRP, 2006) from many types of manures and BS produced from many treatment processes, yielding materials with a wide range of runoff P potential. The literature compilation included WEP ranging from very low (such as BS with high Fe) to very high (swine slurry). As WEP for a soil amendment increased, so did runoff dissolved P, indicating there is a correlation between concentrations of WEP and measurements of potential P runoff (Kleinman et al., 2005). Elliott et al. (2006) then developed a regression equation, called the WEP-to-PSC conversion equation, linking measured WEP of soil amendments with their resultant PSC. Before Elliott et al. (2006) tested the equation on field runoff data where BS were applied on a N-based application rate, the TP loading rates were first multiplied by the

appropriate PSC from the Pennsylvania P Index. The regression relationship between measured WEP and the resultant PSC was $r^2 = 0.45$, which was not considered statistically significant. When the TP loading rates were instead multiplied by PSCs from the WEP-to-PSC conversion equation, the regression relationship rose to $r^2 = 0.86$. This evidence suggests that PSCs specific to WEP of a particular soil amendment are useful and is thought to allow for a more accurate prediction of potential P in runoff.

In the study by Brandt et al. (2004), TP varied widely across the poultry manure, triple superphosphate (TSP), dairy manure, and 41 BS examined. They therefore found it more meaningful to compare what they considered the “environmentally relevant phosphorus content of a material in relation to total phosphorus.” Therefore, they divided the WEP by TP for each soil amendment and then multiplied this fraction by 100 to derive Percent Water Extractable P (PWEP). By land applying BS based on TP, PWEP could then indicate the risk of P-loss, as a BS with a greater PWEP would likely have a greater P-loss risk.

Importance of Wastewater Treatment and Biosolids Production Relative to Total and Water-Extractable Phosphorus

Soluble P within all BS has been assumed to act similarly to that of manures, but the bioavailability, solubility, and transport pathways of P once land applied have been found to be very different amongst different types of BS and different from many livestock manures. Various WWTP production processes and chemicals can have dramatic effects on P in resultant BS (Elliott et al., 2006), as is hypothesized for NC BS. The extent of BS P solubility

can be a result of chemical additions and processes in many steps of wastewater alterations. Wastewater can have many different characteristics depending on source. As it enters the plant as influent, wastewater then undergoes many forms of primary, secondary, and tertiary treatments and clarifiers before effluent is discharged with dramatically lower P concentrations. As a result, the solids portion of the influent stream gets concentrated, as does its P, into sludge, which then typically undergoes additional treatment, such as thickening, stabilization, and dewatering, to meet disposal requirements. Different methods of treatment depend on the nature and quantity of influent, economic and geographical circumstances, and evolving wastewater technologies.

Therefore, if NC is going to develop methods of regulating BS land application on a P basis, it is imperative that WWTPs (i.e., BS sources), their methods of treatment and BS production, and the potential relationships of these to WEP and TP, be addressed for BS across the state. Interestingly, the Pennsylvania Index does not have a PSC for BS with high Al or high Fe, which have been shown to drastically reduce P solubility (Brandt et al., 2004). Therefore, using a PSC representing another qualitative category may not accurately depict the P-loss potential for BS containing high Al or Fe. Also, when it comes to BS that are treated with processes that would cause them to fit into multiple categories, it may make using a particular generic PSC ineffective. For instance, if the BS are both heat-dried and a result of a BPR production process, assigning a PSC of 0.2 (heat-dried) versus a PSC of 0.8 (BPR), would be a drastic difference. The PSC chosen for such BS would affect land application rates, and consequently, soluble P runoff potential (Elliot et al., 2006).

If qualitative categories and soluble and nonsoluble P fractions for NC BS were to be developed, they would likely need to be comprehensive enough to accommodate BS that are a result of many process combinations. In the next section, five important factors affecting BS P solubility will be examined: BS class distinctions, biological processes employed by WWTPs, chemicals used in the WWTP process, physical BS production techniques such as moisture reduction and thermal treatments (Elliott and O'Connor, 2006), and seasonal variation.

Biosolids Class Distinctions

Biosolids can be classified as either Class B, Class A, or Class A – Exceptional quality (EQ) depending upon their pathogen, vector attractant, and potentially toxic metal reduction requirements. These standards were developed as part of the 40 CFR Part 503 Rule (USEPA, 1994a), where it became necessary to distinguish the BS class standards. Class B BS are treated with a “Process to Significantly Reduce Pathogens” (PSRP), commonly by means of anaerobic or aerobic digestion, and require site use and access restriction permits. To be designated Class A, BS must be treated with a “Process to Further Reduce Pathogens” (PFRP), e.g., heat drying, beta ray irradiation, in order to meet more stringent pathogen limits. Both classes require subsequent fecal coliform testing, but Class A typically requires higher temperature treatments for longer time periods. It is only Class A BS and Class A EQ BS that are suitable for home gardens, public access sites, etc. It is therefore a combination of effluent P and N-reduction requirements and BS class standards that dictate what chemicals

and processes a WWTP will use throughout their wastewater treatment processes. The class distinction Class A EQ is used by some states, wherein BS must meet stricter metal concentration limits than Class A or Class B, but Class A EQ BS have the same pathogen and vector reduction requirements as Class A. Once Class A BS are deemed “EQ” BS, they can be used without further 40 CFR 503 restrictions, and be applied to lawns or home garden sites (NRC, 2002).

Biosolids classes can vary drastically in physical composition. Class B BS normally have very low or moderate solids content (slurry and cake, respectively). Class A BS typically have higher solids content (dewatered cake, pellets, or powders). The composition of the BS is dependent upon the character of the sewage influent, the type of WWTP, and the nature of the treatment processes by which they are produced. Sewage entering a plant is first screened to remove debris. After screening, it is composed of 99.9 percent water and 0.1 percent total solids. The heterogeneous solids portion is about 50 percent suspended and 50 percent dissolved. The organic components of the solids include proteins, carbohydrates, and fats. The inorganic portion includes metals, grit, and salts. After the primary settling stages, the solids still contain a large fraction of the influent organic components. These have not undergone decomposition, making them unstable, grayish in color, and granular. These solids are the largest constituent of the biochemical oxygen demand (BOD), which is the amount of oxygen needed by microbes to oxidize the organic components. In secondary settling stages at a WWTP, the solids begin to be decomposed, and are generally more stable, flocculated, and more homogenous. Biosolids are typically brown in color after a biologically activated

sludge process and darker in color if they undergo chemical precipitation (Spellman, 1997). They typically then undergo various dewatering and stabilization steps, which are dependent upon the specific WWTP, but typically aim at concentrating the solids for storage, transport, and pathogen reduction.

In linking BS class rating with BS P solubility, examining how different treatment processes influence the final product is important. Unfortunately, not many studies have been conducted on sludge before and after pathogen reduction, and defining clear boundaries to separate influences on pathogen reduction strategies are not always definitive. Brandt et al. (2004) examined WWTPs that used different PFRP processes and found that PFRP processes have significant influences on soluble P. This study examined WWTPs using PFRP strategies such as alkaline stabilization, composting, thermal heating, and pelletizing to achieve Class A status. The BS were analyzed before and after they underwent the aforementioned pathogen reduction processes to evaluate how the processes affected the soluble P concentrations. The Class A alkaline-stabilized BS made by aerobic digestion via BPR processes had nearly 0% PWEP, while BS that were composted saw a reduction in PWEP ranging from one half up to a tenfold decrease. Thermal heating to receive Class A status yielded PWEP values of less than 1% and pelletized aerobically digested cake from the BPR process saw a reduction from 24% before pelletization to a PWEP of about 11% after pelletization. Biosolids that were considered conventionally stabilized, which indicates they were anaerobically or aerobically digested, had PWEP averaging 2.2% and 2.7%, respectively (Brandt et al., 2004). This indicates that even without additional treatments such

as PFRP, PWEF values for BS can be quite low when compared to other organic fertilizer sources such as manures, or chemical fertilizers like TSP.

Richards et al. (1997) in Syracuse, NY sampled BS from a dewatered anaerobic digestion process that were either composted, alkaline stabilized, pelletized, or incinerated. When these samples were measured by a weak acetic acid extraction called a toxicity characteristic leaching procedure (TCLP), the initial TCLP P for the dewatered cake was 6% of TP, while composted BS were 2.4%, alkaline stabilized were 0.6%, and pellets were 0.9%. Although TCPL is not equivalent to WEP, it is a measurement aimed at determining the fraction of BS that can have the biggest negative impacts on the environment. While all BS in this study had TCPL P fractions of 6% and below, those that were treated with PFRP had the lowest values.

Due to the obvious complexities in wastewater treatment processes needed to meet strict effluent requirements and sludge pathogen reduction standards, simply examining BS P class rating is not enough to distinguish the role treatments play in influencing BS P, although knowing if a BS was treated with a particular PSRP or PFRP to meet regulatory standards for land application does give an indication of potential P concentrations. A closer examination of the chemical and biological processes that are involved in Class B PSRP and Class A PFRP to achieve the regulatory requirements is imperative in understanding what truly characterizes BS P.

Biological Treatments

In order to properly remove solids from a wastewater stream in an effort to discharge clean effluent, the most common WWTP practice is biological digestion. As previously mentioned, two forms of biological digestion include aerobic and anaerobic digestion, which are considered to be conventional treatment processes for a WWTP. The type of biological treatment can vary depending on how the plant manipulates their treatment processes and is influenced by influent conditions. Although wastewater influent can vary from plant to plant, wastewater treatment processes can generally be more effective if influent flow and loading rates are known. The types of organic and inorganic C, N, and P that make up wastewater are separated into particulate and soluble forms which will affect how the wastewater will be treated. The concentration of constituents comprising the wastewater stream will vary depending on the raw wastewater influent flow rate and the processing stage of the treatment plant. Typical influent total organic C is in the range of 80 to 260 mg L⁻¹; total N, 20 to 70 mg L⁻¹; and TP, 4 to 12 mg L⁻¹ (WEF, 2006). Since WWTPs typically use aerobic and/or anaerobic conventional wastewater treatment methods, it is important to have a general understanding of these processes.

Aerobic Digestion

The main distinction between aerobic and anaerobic conventional digestion processes at a WWTP is the reliance on oxygen. Aerobic digestion is a common method of meeting PSRPs. In aerobic digestion, BS are agitated with air in order to maintain aerobic conditions

in the range of 40 days at 20°C to 60 days at 15°C (USEPA, 1994a). In this method, organic matter, oxygen, and cellular material react to form digested sludge, carbon dioxide, and water. Microorganisms therefore use oxygen to break down and oxidize the organic matter within the wastewater. By monitoring timing and including multiple clarification steps, a properly aerated WWTP can decrease total C, N, and P loads, but also harvest the energy necessary to keep the digestion process active. Through respiration, the bacteria breakdown the organic components into relatively stable solids, substantially reduce pathogens and mass of solids, and also synthesize more bacteria to continue the processes (Wang et al., 2007).

A common method of measuring organic C content in a wastewater stream is by determining the BOD in mg L^{-1} . As organic solids are settled within the WWTP clarifying processes, the ratio (mg L^{-1}) of BOD to TP will tend to decrease. A proper ratio of BOD:P must be maintained in order for a WWTP to successfully decrease P in effluent. If the ratio is too low, such as below 20, organisms will not have enough C to uptake P. Similarly, BOD:organic N should be in the range of 4:1 to 5:1 if a WWTP is trying to remove N via denitrification, as organic C must be present for biological growth in treating wastewater. Therefore, although the goals of aerobic digestion are ultimately to remove solids and decrease BOD, proper timing and other manipulations of the system are imperative, as it is the products of oxidation that maintain biological activity and drive system biology (WEF, 2006).

Anaerobic Digestion

In anaerobic digestion, microorganisms are involved that do not require oxygen to decompose the organic matter in the wastewater. Anaerobic digestion is also a common method of meeting PSRPs. In anaerobic digestion, BS are treated without air in the range of 15 days at 35 to 55 °C to 60 days at 20 °C (USEPA, 1994a). Anaerobic digestion is a two-step process where first facultative acid-forming bacteria use the organic matter within the wastewater to form organic acids such as amino acids and fatty acids, stabilized solids, and more facultative microorganisms. In the second step, methane-forming microorganisms use these acids as energy to form methane gas, stabilized solids, carbon dioxide, water, and more methane-forming organisms. The characteristics and quantity of methane produced are influenced by influent properties, flow rate, pH, and temperature of the wastewater stream that is introduced into the digester. Anaerobic digestion reduces pathogens, decreases solids volume, and does not require high amounts of nutrients or oxygen. It has recently become more popular, as WWTPs can use the methane produced as a fuel source, either internally or by exporting it off site (Wang et al., 2007). As previously discussed in evaluating BS class distinctions, conventional sludge treatment can produce relatively low PWEF values, as it was found by Brandt et al. (2004) that anaerobically stabilized BS had an average PWEF of 2.2%, while aerobically stabilized BS PWEF averaged 2.7%. In another study by Sui et al. (1999), anaerobically digested sludge at an Ames, Iowa WWTP resulted in a PWEF of 1.1%, or 0.24 g kg⁻¹ WEP per 22.1 g kg⁻¹ TP.

Biological Nutrient Removal

In conventional digestion systems, final N effluent is typically around 20 mg L^{-1} and P effluent typically ranges between 3 to 4 mg L^{-1} . Therefore WWTPs generally use other means of further reducing their final effluent nutrient rate. In order to meet stringent effluent requirements, WWTPs can manipulate their systems to remove excess N and P present in their wastewater (WEF, 2006). Typically, with most current treatment processes, this concentrates nutrients in sludge. Many WWTPs therefore employ biological processes that remove a portion of the nutrients from the wastewater, known as biological nutrient removal, or BNR. These systems are modifications of activated sludge, where the bioreactor is divided into certain anaerobic, anoxic, aerobic, and mixed liquor reactivation zones, which are differentiated by their terminal electron acceptor. The manipulation of the zones can target the removal of both N and P. All BNR systems must have an aerobic zone, as nitrifying bacteria for N removal and P removing bacteria both require oxygen. For P removal, an anaerobic zone is additionally required, and for N removal an anoxic zone is additionally required. For N removal, nitrification first occurs in the aerobic zone and denitrification – where nitrate-N is converted to N gas – occurs in the anoxic zones (Grady et al., 1999).

Biological Phosphorus Removal

A form of BNR that is more specific for removing P is called biological phosphorus removal, or BPR. In such systems, P removal is reliant upon P-accumulating heterotrophic microorganisms in the wastewater stream. It is through proper design and system

maintenance that WWTPs can promote these microorganisms in their system. In anaerobic conditions, these phosphate accumulating organisms (PAOs) breakdown polyphosphate, which are comprised of many phosphate molecules, to acquire the energy to convert organic materials within the solids to C compounds referred to as poly-hydroxyalkanoates (PHAs). This process releases P, increasing the phosphate concentration in the anaerobic stage. When a WWTP then introduces aerobic conditions to the PAOs, they oxidize the stored PHAs to acquire energy. This energy is then used for maintenance and growth of the PAOs, and they are able to take up phosphate that was released in the anaerobic stage. Therefore, P is removed from the wastewater stream, as it is stored in the biomass, where it can subsequently enter sludge treatment, so as to not leave the WWTP in the effluent. In addition, some of the energy and C in the mixed liquor is recirculated to the anaerobic zones to repeat the process (WEF, 2006).

Since modifications to BNR processes play an important role in amount and type of nutrients that are removed, placement and timing of anoxic and anaerobic zones will dictate nutrients that are able to be removed from effluent streams. Some WWTP's BNR processes are designed specifically to reduce total N, while others are designed to reduce total N and TP. Unfortunately, N removing systems can have adverse impacts on BPR processes, as nitrification causes nitrate-N to get recycled to the anaerobic zone. The denitrifying bacteria in the anaerobic zone will compete with the PAOs for substrate, reducing the PAOs selective advantage, which terminally reduces the P that is able to be removed (Grady et al., 1999). How a plant configures their system is often dependent upon their targeted quality of

effluent, influent conditions, existing processes, climatic circumstances, and operator knowledge. A WWTP can achieve very low P concentrations in effluent, such as below 0.7 mg L⁻¹, by using a BPR system (Jeyanayagam, 2005). Composition of the influent wastewater stream such as varying degrees of oxygen, N, and organic matter levels can also have an impact on BPR effectiveness. By combining treatment processes, such as using BPR, chemicals added to precipitate metals, and final clarifier filtration, WWTPs can potentially get their effluent total P loads as low as 0.1 mg L⁻¹ (Jeyanayagam, 2005). Due to economic or other site restrictions, many WWTPs do not use BPR, and instead rely simply on conventional digestion or other means of nutrient reduction to reduce their P effluent loads.

In the study by Brandt et al. (2004), BS produced using BPR had the highest TP content among both the BS and manures, with a median equal to 34.4 g kg⁻¹. Interestingly, when comparing BS and manures, the study found that most of the BS examined contained the highest amount of TP, but the lowest amount of WEP, the only exception being from those produced from BPR. These BPR-produced BS had high TP and WEP, which is reflective of many studies examining BS produced by WWTPs employing the process of BPR. In greenhouse studies by O'Connor (2004), 12 BS were compared relative to TSP fertilizer for P phytoavailability. All but two of the BS were considered less than 75% phytoavailable compared to TSP. These two BS were produced using BPR where no Fe or Al salts were added to aid in P precipitation. This research suggests that BS produced in this manner can be expected to pose a greater P-loss risk, acting similarly to a highly soluble commercial fertilizer.

Chemical Treatments

Al and Fe Salts

As discussed by Brandt et al. (2004), the use of certain chemical additions in the wastewater treatment process can cause metal-phosphate precipitates to comprise the P fraction within BS. The use of these chemicals is a common process in wastewater streams to keep in compliance with NPDES permit requirements (USEPA, 2012a) limiting P in effluent. These Al- and Fe-containing chemicals, typically Al and Fe salts, remove soluble P from the wastewater by metal phosphate precipitation (Maguire et al., 2001). These reactions result in decreased levels of soluble P within the wastewater and increased solids production. The amounts of these chemical salts administered at a WWTP can vary significantly, depending on the use of other nutrient reduction strategies, and the nature of the wastewater. Biosolids Al and Fe phosphate precipitate concentrations will vary as a result. Traditionally, wastewater is treated with chemical precipitates to reduce P effluent loads, not to lower P solubility within resultant BS. The degree to which WWTPs are administering these salts specifically in sludge treatment is not well documented. Chemical feeds can be supplied into the wastewater within many of the treatment phases, including secondary clarification processes, during sludge stabilization, or in measures to control odor, which may affect P solubility within the BS. Additionally, the chemical composition of these salts can vary, which can also influence their ability to precipitate phosphates. Overall, Al and Fe compounds added to wastewater streams increase TP but lower soluble P concentrations

found in BS, and therefore lead to decreased potential for soluble P-loss runoff and leaching risks when the BS are land applied (Elliott et al., 2005). By comparison, Al and Fe salt concentrations are found to be much lower in manures tested in many studies. Studies such as performed by Elliott et al. (2002), where eight BS were examined, including those processed with Al and Fe, showed that these chemicals can substantially reduce P losses from applied BS. In all but one type of BS studied, it was found that the majority of inorganic P, which comprised 80 to 90% of all forms of P in the BS, was in Fe- and Al- associated compounds. It was concluded that Fe and Al P fixation can make the soluble P in BS immobile, reducing P losses from land applied soil amendments treated in this manner. The increased use of these salt precipitates in wastewater treatment, as opposed to other soil amendment sources, leads to the importance in distinguishing land applied BS from manures and synthetic fertilizers.

As noted by Brandt et al. (2004), BPR BS are typically low in Al and Fe, as these facilities do not rely on the salt P precipitation to meet their P effluent standard limits. The six BPR-produced BS in their study contained nearly half the concentrations of Al and Fe minerals compared to the other BS. If WWTPs were to combine nutrient reducing efforts by using BPR and Fe and Al chemicals, it could potentially lead to low effluent P rates and increased P precipitation within the resultant BS. Regulating BS on an N and P basis instead of just an N basis may lead to more facilities employing both treatments.

Aluminum and Fe metals have an inverse correlation with soluble P: the higher their concentration, the less mobile P becomes in receiving soils (Brandt et al., 2004). Brandt et al. (2004) compared PWEF values for inorganic fertilizers such as TSP, organic fertilizers (manures), and conventionally produced BS, and found that all the soil amendment types were different, and that the amendment type molar concentration of total Fe plus total Al was the most significant predictor of PWEF, other than amendment type itself. Therefore, as Al and Fe concentrations increased, the level of PWEF decreased. It was found that the Fe and Al content of BS varied from about 2 g kg⁻¹ to more than 83 g kg⁻¹ and about 4 g kg⁻¹ to more than 66 g kg⁻¹ dry weight equivalent, respectively.

Further evidence supporting the ability of Al and Fe chemicals to precipitate P can be found in the earlier study performed by Pierzynski et al. (1990). They reported that P water extractions on two sludges, with both high levels of Al and Fe amendments and rich with phosphate, had high resistance to P dissolution-desorption during the extraction. The high levels of Al and Fe within the sludges were considered to be the primary reason for this strong sorption capability. The elements were imperative in the partitioning of solid phase P within the wastewater residuals. In a study conducted by Maguire et al (2001), where they examined BS treated with many different processes, the BS containing Fe and Al had the lowest water-soluble P. Interestingly, in a study by Huang et al. (2008), in which freshly anaerobically digested BS were treated with one of three chemicals, either ferrous sulfate, quicklime (CaO), or alum, P speciation was found to be most related to the Al:P within the BS. At low Al:P, water-soluble P transformed into Al-P species. At high Al:P, which lowered

the pH, water-soluble P increased. This may be a result of unstable Ca-P species (hereafter, Ca-P) dissolving within the BS due to the low pH.

To develop weighting factors in a P index, DeLaune et al. (2004) examined factors that affect P runoff. They found a significant linear relationship ($r^2 = 0.92$) between the dose of alum applied to a poultry litter and the soluble reactive P (SRP) concentrations in runoff water. With alum added at 5%, 10%, and 20%, SRP within the runoff decreased 42%, 49%, and 97%, respectively. It was also noted in this study that the precise mechanisms responsible for reducing P solubility by alum treatments is still uncertain.

Alkalization

Alkalization is the process of using a liming agent, such as a Ca-containing compounds like CaO, hydrated lime, and lime kiln dust, to treat sludge to meet pathogen reduction requirements. This is achieved via pasteurization from the heat generated by such highly reactive chemicals, as well as the substantial increase in pH. Both Class A and Class B pathogen reduction standards can be met by adding an alkalizing agent, but the classes differ by duration and temperature requirements. Class B alkaline stabilization pathogen reduction can be met when the sludge and alkali mixture pH is more than 12 after 2 hours. For Class A BS, pH needs to be at least 12 for over 72 hours, with temperatures above 52°C for more than 12 hours, and overdosing with alkali is common to achieve these requirements. Class B BS therefore usually require less alkaline material and shorter mixing time (USEPA, 2000). Land

applying Class A alkaline stabilized BS is most often used when trying to adjust the pH of the soil, rather than for nutritional benefits, as nutrients per unit of material is often low.

Alkalizing chemicals high in Ca are thought to increase P precipitation and adsorption as pH increases and Ca phosphate formation often reduced soluble P (Brandt et al., 2004). However, in the study by Brandt et al. (2004), when BS were treated with alkaline chemicals, PWEF values exceeded those of BS produced from conventional digestion methods without the addition of alkaline chemicals.

One means of advanced alkaline stabilization is a patented method called the N-Viro process (N-Viro International Corporation, Toledo, OH), where dewatered sludge is combined with alkaline industrial byproducts which generate high pH, heat, and drying to eliminate pathogens. The product of this method is referred to as N-Viro Soil, which has physical characteristics similar to mineral soils and is used as a lime substitute (Logan and Harrison, 1995). This type of stabilization significantly dilutes the BS with solids that do not contain P and also reduces organic matter content of the BS (O'Connor et al., 2004). For BS that were treated with this method in the Brandt et al. (2004) study, TP, WEP, and PWEF levels were very low: 3.7 and 0.01 g P kg⁻¹ BS, and <0.5%, respectively. In the study by O'Connor et al. (2004), they had one BS that was a product of N-Viro stabilization, and it had TP concentration of 3.2 g kg⁻¹, which was substantially less than any other BS in their P phytoavailability study.

When determining environmental implications for sludge treated by advanced alkaline stabilization in the production of BS however, it is important to take caution in making conclusions concerning the degree of P-loss risk. Unlike other BS production practices that yield lower pH values, those from advanced alkaline stabilization can have pH values as high as 12 or more. Reactions controlling P solubility within this extremely high pH range would not reflect those at lower pH values that are typical for other methods of BS production or typical soil pH where BS are applied.

Interestingly, some research suggests that the use of alkalinized BS on soils can actually increase P solubility, although this research did not include alkalization such as the N-Viro process (Brandt et al., 2004). Maguire et al. (2001) found that when BS were treated with both lime and Al and Fe salts, WEP tended to increase in amended soils, compared to WEP of soil extracts in which metal salts were added, but were not limed. When Brandt et al. (2004) examined different alkaline-stabilized BS cakes, the BS with the highest concentrations of Al and Fe ($13.8 \text{ g kg}^{-1} \text{ Al}$ and $26.3 \text{ g kg}^{-1} \text{ Fe}$) had the lowest PWEF (3.32%) of all the alkaline-stabilized cakes tested. This suggests that although BS treated with both lime and metals salts may have the lowest P solubility, when these BS are applied to soils, soil pH may alter P reactions that control solid phases in limed BS, which may cause soil WEP to increase. Within the PSC table for the Pennsylvania P Index, alkaline stabilized BS were given a 0.4. This is the second highest PSC for BS, second only to BPR BS PSC of 0.8 (Elliott et al., 2006). This type of phenomenon suggests that “book-value” categorical PSCs

may not adequately predict the potential for P-loss risk, as the PSC may be an overestimate or underestimate.

In the study by Huang et al. (2008), the BS that were treated with ferrous sulfate transformed Ca-P to Fe-P species within the BS, which may explain the results of the Brandt et al. (2004) study for BS treated with both Fe salts and alkaline stabilization. As reported by Huang et al. (2008), when BS were stabilized with the alkalizing material CaO, water-soluble P transformed into Ca-phosphates, a phenomenon that increased with elevated CaO additions. Clearly, alkaline stabilization can have mixed effects on P solubility, which can be highly dependent on other chemical additions, soil pH, the nature of the alkalizing source, and the dose prescribed.

Physical Treatments

Moisture Reduction

A common practice of WWTPs sludge treatment is to physically decrease the moisture content of the sludge. This practice not only lowers transport costs, but also can be used to meet pathogen reduction requirements. During the dewatering stage in sludge treatment, centrifuges, belt presses, gravity thickeners, and other mechanical tools are utilized to remove some of the liquid portion of the sludge. This supernatant can encompass a majority of the soluble P within sludge (Brandt et al., 2004). By removing soluble P, PWEF and potential for P release in runoff from land-applied BS can be greatly reduced (Withers et al., 2001). In many WWTP systems, this liquid is returned to the head of the plant, where it

once again undergoes treatment processes such as primary clarification and secondary biological treatments.

In the study by Brandt et al. (2004), where the 41 BS and various animal manures were examined, the BS total solids content had a large range, between 6 to 93%, with a median of 21%. By comparison, the total solids content for poultry litter ranged from 26 to 92% with a median of 81%, but in the dairy manures was only 10 to 17%, with a median of 15%. The dairy manures had the largest PWEF among all manures and BS, averaging 52%. Withers et al. (2001) found that the PWEF for liquid cattle manure was 60%. Contrastingly, the PWEF for the dewatered sludge cakes were below 1%. In a study by Sharpley and Moyer (2000), PWEF was 26% for poultry manure and 51% for dairy manure. By comparison, the PWEF of TSP was found by Brandt et al. (2004) to be 85%. Withers et al. (2001) concluded that the potential release of P via runoff was the lowest for dewatered sludge cake, when compared with TSP, liquid cattle manure, and liquid digested sludge. It must be noted, however, that manures are influenced by digestive processes and nutrient-specific livestock diets, and that different concentrations of Fe and Al in soil amendments can affect P speciation (Brandt et al. 2004).

Thermal Treatments

A very effective method of moisture reduction is through thermal treatment of the sludge. This form of moisture reduction often involves a very large decrease in liquid content, often resulting in a pelletized product. When Brandt et al. (2004) compared PWEFs

for various BS production methods, thermally treated BS (without BPR) had an average PWEF of 0.48%. The only stabilization method with lower PWEF was from the advanced alkaline stabilized BS, such as the N-Viro stabilization, which had an average PWEF of 0.21%. Interestingly, thermally treated BS had an average TP of 26.1 g kg⁻¹, which is more than seven times as much TP than the N-Viro BS, at 3.7 g kg⁻¹. By comparison, BS that were composted, alkaline stabilized, or BPR cake had PWEF values of 3.1, 7.4, and 13.9%, and TP values of 17.5, 12.7, and 32.0 g kg⁻¹ BS, respectively.

The higher TP of thermally heated BS, when compared to another PFRP strategy such as advanced alkaline stabilization, can be attributed to the fact that when BS are treated thermally, water is evaporated from the solids, not removed in liquid form. Since gaseous forms of P are negligible, P becomes concentrated in the solids. By comparison, advanced alkaline stabilized BS are diluted with lime products, such as lime kiln dust, causing these BS to have very low P concentrations, by comparison.

In the study by Brandt et al. (2004), thermally treated BS were in a pellet form, which have been shown to have slow rates of physical decomposition, and therefore are less of an immediate environmental concern for P-loss risk. In their study, three of the pelleted BS samples that were conventionally stabilized were ground and sieved, but WEP did not change, or the changes were negligible. This may be a result of major changes of BS properties after heat treatment. Thermal treatments can convert forms of soluble P into insoluble forms, such as Ca-P minerals, which can significantly reduce the extractability of P

(Smith et al., 2002). The dissolution of some phosphate minerals formed from heat drying is very slow and they do not readily release P into solution once they are rewetted (Brandt et al., 2004). One of the BS in their study, a pelleted BPR treated BS, did have an increase in WEP from 2.6 to 3.9 g kg⁻¹ after grinding and subsequent sieving. This may have been due to increased surface area. In a greenhouse study by Elliott et al. (2002), heat dried pellets applied to a sandy soil had very low rates of P leaching. This research suggests that although thermally treating BS may not decrease TP, the release of soluble P from BS can be drastically reduced, in contrast to strictly conventional sludge digestion treatment (Smith et al., 2002).

Although these studies have shown that there is a major reduction in soluble P for pelletized BS, this might be a temporary phenomenon. O'Connor et al. (2004) pointed out that pelletizing BS reduced P phytoavailability during their 5-month greenhouse study, but that soluble P may increase over the long term, regardless of the pelletization process. Although, as previously noted by Johnson et al. (2005), surface-applied, highly nonsoluble P soil amendments, such as thermally dried BS, are considered to be more likely to remain on the surface of the soil, thus providing a P source over the long term. More field studies monitoring BS P release need to be conducted over a long-term period to confirm such speculation.

Seasonal Variation

Seasonal variation can have an effect on BS produced at a facility, as temperature and precipitation can alter biological activity within a wastewater stream. To a point, higher temperatures would normally promote more biological activity, which increases reaction rates. This would in turn be expected to yield increased P removal from wastewater streams, implying an increased concentration of P in BS. A study by de-Bashan et al. (2004) indicated that bacterial isolates grew well at 15° C and 25° C, but growth was limited at 5° C. In the study, at 15° C the P removal rate was 0.6 mg P L⁻¹ day⁻¹, but this rate could not be obtained at lower temperatures.

According to WEF (2006), studies on the effects of temperature on EBPR (enhanced biological P removal) have yielded contradictory results, with some studies reporting temperature did not affect EBPR, some reporting low temperatures adversely affecting the process, but many reporting that despite slow reaction rates at lower temperatures, EBPR efficiency was significantly greater. In systems where EBPR was improved by lower temperatures, WEF (2006) suggests that PAOs have a selective advantage, outcompeting mesophilic competitors for substrate in anaerobic zones, increasing PAOs population size. At higher temperatures there is more competition for substrates by non-PAOs. Some researchers also believe PAOs prefer to accumulate glycogen, rather than P, at higher temperatures, while others think that there is a shift in populations, from PAOs to glycogen-accumulating organisms (GAOs). Either way, the dependency on glycogen decreases EBPR efficiency.

Additionally, when two aerobic sludges, one from 5° C and one from 20° C, were compared under an electron microscope, almost all cells, PAOs and non-PAOs, from the 5° C sludge had the ability to store poly-P. The sludge at 20° C had only one group, the PAOs, storing poly-P.

Phosphorus Recovery

Another potential method for reducing soluble P concentrations in BS is by using a P-recovery mechanism to form phosphate complexes, which some WWTPs are already performing, and many others are considering or adopting pilot programs. Phosphorus recovery is the process of forming and harvesting crystalline products such as struvite and other groups of phosphate minerals such as apatite. Struvite is magnesium ammonium phosphate ($MgNH_4PO_4$), which can easily form in the wastewater treatment process, since all of these elements enter sludge digesters. Struvite scaling can therefore become a nuisance at WWTPs if not properly treated or harvested, as it can clog up the wastewater stream pipes. This problem was therefore the original driving force behind P recovery technologies (Boudeman et al., 2014). Although the formation of struvite can be minimized at WWTPs by adding ferric chloride and other precipitators that aid in the reduction of its formation, harvesting struvite can be useful if it is properly managed and if components are in proper ratios (WEF, 2006). If the formation and harvest of struvite is desired, it is essential to add adequate amounts of magnesium, maintain a pH above 7.5, and have low concentrations of

suspended solids, along with the presence of soluble P and ammonium (de-Bashan et al., 2004).

Struvite can be precipitated in a controlled fashion by using a dedicated reactor instead of spontaneous formation (de-Bashan et al., 2004). The struvite can then be harvested and used as a slow release P fertilizer or an ingredient in animal feed (Shaffer et al., 2008). This can be advantageous for a WWTP in that P-recovery systems can generate revenue by fertilizer production, decrease effluent P loads, reduce P returned to the influent of the plant, reduce struvite scaling, and lessen the reliance on other treatment methods for nutrient reduction, such as the addition of chemicals (Le Corre et al., 2009). By recovering P in the form of struvite, WWTPs can remove more than 70% of P from their system (Le Corre et al., 2009).

As P recovery technologies improve, WWTP will be able to recover more struvite and other P containing minerals. By employing P recovery mechanisms, not only would P concentrations in BS be reduced, but they could also reduce the reliance on phosphate rock being used as a fertilizer source. Increasing phosphate rock costs could also increase the demand for improving the purification of P recovery technologies, which may drive WWTPs in this direction for nutrient management. These P containing minerals harvested from wastewater treatment could be useful when a slow-release P fertilizer is desired, such as forest or pasture settings, with potentially low P runoff and leaching risks, making it a very cost-effective P recycling option (de-Bashan et al., 2004).

Biosolids Phosphorus Loss Risk in North Carolina

As mentioned previously, the NPDES was established at the federal level in order to control point-source wastewater discharge. In NC, the NC Department of Environment and Natural Resources (NCDENR) Division of Water Resources [DWR; formerly the division of Water Quality (DWQ)] administers the NPDES Permitting and Compliance Program. Most NPDES permits require WWTPs to monitor TP and total N in effluent, but currently in NC, there are no standards for these nutrients, and limits are placed in permits after water quality surveys are administered on receiving waterways (NCDENR, 2014a).

The DWR is responsible for issuing BS land application permits. Biosolids can be applied to either dedicated or non-dedicated fields. Dedicated fields are those receiving BS where applications either exceed agronomic rates or frequencies, are applied through irrigation systems, or are applied as a primary use of the land. The DWR issues three types of BS application permits including the Land Application of Residual Solids (LARS) for dedicated fields, Distribution of Residuals Solids for non-dedicated fields or for sale as a fertilizer to the public, and Surface Disposal of Residual Solids into monofills (NCDWQ, 2012).

Annually, LARS permit holders must submit a report to DWR which includes the amount of P and PAN that were applied and which fields received the BS. Distribution permit holders are not required to supply this information. Therefore, it is the LARS permit holders that provide useful information in determining potential nutrient loads from BS

dedicated field applications on a watershed basis. Based on the annual reports, DWR maintains a Basinwide Information Management System (BIMS) database containing coordinates for each permitted application field, including field acreage, lbs P ac⁻¹ applied, and lbs PAN ac⁻¹ applied. For 2010, there were 125 LARS permit holders who reported residual land applications out of 193 total permits active that year. These applications were across 10,747 ha (26,556 ac) within 12 of 17 of NC's watersheds and averaged 103 kg P ha⁻¹ (92 lbs P ac⁻¹) and 86 kg PAN ha⁻¹ (77 lbs PAN acre⁻¹). The Cape Fear River was within the watershed with the highest amount of total P [298,344 kg (657,737 lbs)] applied. When normalized by area size, the Neuse River watershed had 13 kg P km⁻² (73 lbs P sq mi⁻¹), which was the greatest concentration in this same year (NCDWQ, 2012). Although the BIMS database contains nutrient data as related to BS land applications from LARS permit holders, there is currently no established assessment of P-loss risk imposed by these applications to watersheds within NC.

Estimating Phosphorus Loss Risk at the Field Scale: the North Carolina Phosphorus Loss Assessment Tool

The NC Phosphorus Loss Assessment Committee developed a P-loss assessment known as the Phosphorus Loss Assessment Tool (PLAT) in order to meet the NRCS Code 590 Nutrient Management Standard for P and conform to NC environmental conditions and regulations (NC PLAT Committee, 2005).

Determining a PLAT P-loss risk rating is necessary for those who must utilize the NRCS Code 590 Nutrient Management Standard and then meet at least one of the following three criteria: when the rate of P applications surpasses land-grant university fertility rate guidelines; the field is within a P-impaired watershed; or if the water quality control authority of the NRCS and State has not determined the conditions in which risk of P-loss is low (NRCS, 2012). In one nutrient sensitive watershed in NC, the Jordan Lake, the Agricultural Rule allows BS to be applied on a P basis (NCDENR, 2014). Once appropriate P coefficients are developed for BS, PLAT can be augmented to include this form of organic nutrients. Thus, BS application in the Jordan Lake watershed may require the use of PLAT.

The NC PLAT takes into account four P-loss pathways in determining the potential overall P-loss for a particular field. These pathways include sediment P, which includes losses via erosion of P attached to soil; soluble P via surface runoff; soluble P via subsurface drainage or leaching; and P losses (particulate and soluble) from nutrient sources. The NC P-loss tool computes a PLAT rating for each potential P loss pathway and a total potential P loss rating is subsequently generated by adding the four potential pathways. The PLAT field program uses information such as field slope, soil texture, soil test P, source of P, P application rate, surface water proximity, crop type, and field conservation practices (Johnson et al., 2005). The NC PLAT software has been designed to incorporate at least 17 types of manures, including estimated total P and soluble P for each manure source (NC PLAT Committee, 2005). However, BS have not been addressed in PLAT, and no P fractions exist in the tool to account for potential differences in BS P solubility. Land application of

BS has instead strictly relied on applications based on agronomic N needs per USEPA Part 503 guidelines (USEPA, 1994a). As a consequence, land application of BS typically results in over application of P.

In the PLAT software, the estimated P loss values (in kg P ha⁻¹) are multiplied by 22.3 to convert to the final index value that has a scale system similar to the state soil test system, which is familiar to applicators of soil amendments (Johnson et al. 2005). Unfortunately, this similarity has created confusion: both PLAT output and the soil-test P rating are called the “P index.” Phosphorus loss ratings are placed into categories, low (0-25), medium (26-50), high (51-100), or very high (>101), allowing for an indication of the severity of P-loss potential from the particular site. A low or medium potential P loss index indicates that there is negligible or low potential for P-loss through all possible pathways, which would allow an applicator to follow an N-based application rate. If the rating for the field in question is high, the application rate limit relies on P removed from the crop at harvest. The consequence of a very high rating is that no addition of P would be allowed (Shaffer et al., 2008).

North Carolina agricultural land is estimated to lose about 25 cm of water yearly to surface and subsurface drainage. If this water contained 1 mg P L⁻¹, TP loss would be 2.5 kg ha⁻¹ (2.2 lb ac⁻¹). In relation to the NC Neuse River Sensitive Waters Management Strategy Rules, where WWTP effluent limits are 2 mg P L⁻¹, applying a concentration limit of 2 mg P L⁻¹ to drainage water would allow 4.9 kg P ha⁻¹ (4.4 lb P ac⁻¹) to be lost. Therefore, giving an

agricultural field a very high rating was set as more than $4.5 \text{ kg P ha}^{-1} \text{ yr}^{-1}$ ($4 \text{ lb P loss ac}^{-1} \text{ yr}^{-1}$); (NC PLAT Committee, 2005).

In a study by Johnson et al. (2005), sites in NC that were of highest concern for P-loss risk included those in the Coastal Plain and those using animal wastes with high levels of soluble P. Soluble P-loss via runoff was the greatest loss pathway in PLAT, which considers both the amount of soluble P estimated to be lost and the volume of runoff expected for a particular site. It was found that soils in most of the 100 counties within NC exceeded 53 mg kg^{-1} Mehlich-3 P, which is the STP optimum value for crop growth. Once a soil becomes P saturated, the risk of P-loss greatly increases. Different soils have varying degrees of P retention potential, which PLAT accounts for by creating P threshold groups. These thresholds are defined as “the STP value at which 1 mg L^{-1} of dissolved P is expected to be lost to the soil solution” (Johnson et al., 2005). The groups are assigned based on soil texture, depth to the Bt soil horizon, and drainage, but are referred to by their texture for simplicity. These include clays, loams, sands, and organics, with Mehlich-3 STP threshold values of 500, 200, 100, and 50 mg kg^{-1} , respectively. For each group, a distinctive equation was developed based on NC soil studies, allowing for estimation of soluble P for a particular STP (Johnson et al., 2005).

In nutrient-sensitive NC river basins and watersheds, such as the Neuse and Tar-Pamlico Rivers, and Lake Jordan, soluble P-loss risk can have severe environmental consequences, as soils are already so saturated in P that plants do not respond to additional P,

and P is lost to surface waters via the multiple loss pathways. In the study by Johnson et al. (2005), 92% of the 1379 fields studied fell into the low or medium PLAT index ratings, not requiring nutrient management plan alterations. The remaining 8% of the study sites were in the high and very high categories, which would result in the termination of N-based nutrient management plans for these locations.

In a sampling analysis that delineated agriculture within the Lake Jordan watershed in NC, BS were second only to poultry litter as the most-applied organic fertilizer source (Osmond, 2007). In this analysis, BS were primarily applied to pasture and hay fields and sorghum for silage. Of the 74 fields in which organic soil amendments were applied, 14 received BS. Seventeen of the 74 fields had STP index rating of very high (>100). Eleven of these 17 fields had PLAT index ratings of 100 to 200, while six fields were between 200 and 500 or above. Out of these 17 fields with very high STP index ratings, eight land applied BS, which represented 57% of all fields receiving BS.

Biosolids applicators can use PLAT to more efficiently strategize their nutrient management goals, while also decreasing environmental impact from potential P losses. Phosphorus-based restrictions on soil amendment application rates for sites with high and very high ratings can lead to additional consequences, such as insufficient bioavailable levels of N necessary for optimum crop growth (Shaffer et al., 2008). Meeting the high N demand of some crops, while following P-based regulations, could prove very costly to farmers. As

discussed in previous sections, the BS applicators may then need to purchase additional fertilizers to meet their crop-specific N needs if P rates dictate their BS application rates.

Implementing the Phosphorus Loss Assessment Tool for Biosolids Management

In order to use PLAT to estimate P loss from land-applied BS, it is important to determine whether WEP, TP, and PWEF differ depending on how the BS were produced. To do this, NC-produced BS must be categorized based on treatment processes and these P parameters. By obtaining this information on NC BS and analyzing descriptive statistics on treatment processes, it might be possible to determine whether single representative values in NC PLAT will suffice for each parameter, or if multiple values will be necessary to estimate potential P-source loss risk. This research is important in defining the potential range of P parameter values, their distribution, and whether and which WWTP treatment processes influence these values. If relationships between the P parameters and treatment processes can be determined, the latter might be used to estimate the former. Then P parameter estimates based on treatment processes could be entered into PLAT as a lookup table.

If the amount of P and P solubility for BS produced by NC WWTPs are known, these values can be incorporated into PLAT so as to base BS land applications on both P and N. The type of BS being applied affects P losses from the source pathway, and ultimately will be part of the overall field PLAT rating.

Conclusion

After Title 40 CFR Part 503 USDA was created (USEPA, 1994a), it became necessary to accurately monitor the N loads of BS. Biosolids land applications in many states are therefore regulated following the N requirements necessary to meet the optimum growth for crops, while aiming to avoid N runoff and leaching into the groundwater. Due to the low N:P in BS, it is an unbalanced nutrient source if applied on an N-rate basis, supplying P in excess of crop needs for optimum growth (Shaffer et al., 2008). In areas near nutrient sensitive watersheds that are intensely farmed and receive many BS applications, soluble P can therefore cause eutrophication. The USDA-NRCS Conservation Practice Standard for nutrient management (590) was put into place to determine the P-loss risk of agricultural fields (USEPA, 1999). Unfortunately, BS P regulation has been overlooked in NC, as it has in many other states throughout the country, likely due to a lack of federal BS P regulation.

In the study by Brandt et al. (2004), WEP was divided by TP and multiplied by 100 to achieve a PWEF value, which is meant to represent the fraction of P susceptible to P-loss risk when land applied. While PWEF provides a reasonable estimate of potential P loss, a more accurate estimate of runoff P loss has been derived using a weighting coefficient, the PSC (Coale et al., 2005). In the Elliott et al. (2006) study, to develop a P index, the applied P that was considered susceptible to runoff could be calculated by multiplying the PSCs developed for the Pennsylvania P Index by the TP application rate, which represented the applied P that was susceptible for transport off-site.

Biosolids treatment methods are dependent on the nature and quantity of the influent, economical and geographical conditions, and wastewater treatment technology. The specific WWTP treatment methods employed to meet strict effluent P limits and BS pathogen reduction requirements have been shown in studies to affect P solubility (Brandt et al., 2004), so we hypothesized this will also be the case for NC produced BS P. These methods affecting P include biological manipulations, chemical additions, and moisture reduction techniques.

In order to assess the differences amongst BS P as related to treatment processes, our research goals included classifying WWTPs based on biological, physical, and chemical treatment characteristics, concentrating on those most likely to affect the amounts and forms of BS P. Biosolids were collected from among the larger municipal WWTPs around the state. They were analyzed for TP, WEP, PWEF, nonsoluble P, and solids content. We then determined whether these were related to NC WWTP treatments, as we hypothesized. This would be an indication that different soluble and nonsoluble P fractions could be related to the WWTP processes and BS characteristics, and these fractions could be used in the NC PLAT software.

The NC PLAT currently calculates P-loss risk for manures using book values based on analyses of TP and water-soluble P fractions in the manures. We hypothesize that BS total and water extractable P and their ratio will be correlated with some treatment processes, which would make it possible to associate a range of WEP and TP to these processes to be used as book values within PLAT. In principle, this may produce better estimates of P-loss

risk from land applied BS compared to estimates based on a single “global” value (Coale et al., 2005). This knowledge will give NCDENR the scientific justification to incorporate BS P factors into NC PLAT for use within watersheds with nutrient sensitive water bodies such as Jordan Lake.

The use of PLAT in BS land applications may lead WWTPs to alter treatment strategies in their BS production in order to reduce total and WEP, thus decreasing P-loss risk. Reducing P-loss risk into nutrient sensitive watersheds is very important for the integrity of ecosystems and water quality throughout NC. Incorporating NC-produced BS P solubility fractions into NC PLAT is an important strategy for responsible nutrient management and land applications of this soil amendment.

CHAPTER 2: MATERIALS AND METHODS

Wastewater Treatment Plant Selection

In order to survey WWTP processes, we needed to sample among WWTPs throughout NC. The State of NC requires WWTPs to register with NCDENR the plant location, its Operator in Responsible Charge (ORC), and the permitted flow. This registration data was received from NCDENR and used to select WWTPs. We limited target WWTPs to “Large Municipal Discharge Sites,” (LMDSs) which are defined by NCDENR as WWTPs with permitted flow greater than 1.0 million gallons per day (MGD). There were 119 total facilities within NC defined as a LMDS. Of these 119 plants, 102 were sent the survey, which was delivered via e-mail to the ORC’s of each plant. The additional 17 WWTPs could not be contacted. Of the ORCs sent the survey via e-mail, 59 responded, and in most cases, only after repeated e-mails or phone calls. Therefore limitations in gathering WWTP surveys included some ORCs unwillingness to participate or lack of response. Wastewater treatment plants that do not land apply BS, those that compost or incinerate their BS, or those that do not generate a Class A or Class B product, were not included in the study. Sampling was therefore restricted to WWTP whose ORCs responded to the survey and those that land apply their Class A or Class B BS. Out of the 59 responders, 42 reported that they land apply. Sixteen of these 42 were not included in the study either because they could not supply a final Class A or B product, or they did not respond when asked to participate in BS sample collection. Therefore, there were 26 participating facilities in the study (Table 1).

The WWTPs sampled represented most of the largest cities that land-apply BS within the state, but also included smaller treatment plants for which the ORCs were willing to participate. We averaged each facilities effluent flow rates as reported to the NPDES for 2013 and 2014. The mean, minimum, and maximum effluent flow rates of the participating WWTPs were 15.0, 0.1, and 75 MGD, respectively. The WWTP with 0.1 MGD was the only facility in our study permitted for more than 1.0 MGD effluent discharge, but obviously the actual discharge was much less during 2013 and 2014. The WWTPs surveyed and sampled represented nearly 70% of all land-applied BS in NC, with estimated annual dry tons ranging from approximately 300 (smaller plants) to 17,000 (larger plants) (Fig. 1). By surveying and sampling WWTPs that produce varying amounts of BS, a variety of different types of BS were obtained. This included those that produce less than 500 dry annual tons, 500 to 1000 dry annual tons, and larger WWTPs that produce over 1000 or even over 5000 dry annual tons (Fig. 1). The BS differed by treatment processes and chemical additions. There were three facilities in which an unfinished product was sampled and analyzed for P and dry matter (DM) content, but data from these unfinished products were omitted from the study. These three sludges are all treated with ferrous sulfate and mixed together to become one Class A product by thermal drying and centrifugation for pelletization. This final Class A product was analyzed for P and DM content, and is included in the study.

Wastewater Treatment Plant Flowchart

We developed an online survey questionnaire to capture variations in WWTP processes, some of which were likely to affect the solubility of P within the produced BS. We first developed a generalized flowchart of a WWTP (Fig. 2) to aid the ORCs in understanding which step of the treatment process a corresponding survey question was referencing. In this manner, it was important to make the flowchart comprehensive, yet simple enough for the ORCs to follow visually to facilitate accurate survey responses. To develop the flowchart, information on treatment plant processes, designs, layouts, and terminology was compiled from books and scholarly articles, with assistance from several plant operators and civil engineers. The flowchart was developed using PowerPoint tools and includes the typical WWTP treatment steps, including different chemical feeds that may exist within the various stages of a treatment plant. Wherever there could potentially be a chemical feed within the WWTP system, the flowchart was marked with a “C” displayed in a pink oval. There were 12 chemical feed ovals identified. Representing each “zone” in the flowchart were roman numerals within boxes, diamonds, or along directional arrows. There were 13 treatment zones indicated by roman numerals. These roman numerals were labeled within the flowchart and also within the online survey. As treatment plants inevitably vary from one another, broad terms such as, “collection (Influent),” “preliminary treatment,” “primary treatment,” “secondary treatment,” “final clarifier,” “tertiary filter,” and “disinfection” were used to classify zones of treatment leading to the effluent stream of the WWTP. Terms such as “thickening,” “stabilization,” and “dewatering” were used to refer to

zones within “sludge treatment” that resulted in the formation of Class A and/or Class B BS, which were indicated in light green-filled boxes, representing products from the system. There were also arrows in the flowchart and questions indicating the potential existence and direction of liquid and solids back streams and backwash. These streams were accompanied by either small yellow circles marked with an “L” for liquid streams or small dark green circles marked with an “S” for solid streams. A “chemical treatment (P Recovery)” was also displayed as a treatment zone option on the flowchart representing potential P recovery systems and the recovered P product within a green-filled product box.

Online Survey Development and Implementation

We developed survey questions that corresponded to each potential process and chemical addition in the flowchart. The online survey (Appendix, Table 1) was generated using Qualtrics software, Version 2013 of the Qualtrics Research Suite (Qualtrics, Provo, UT, USA). This online survey tool was used to develop and administer the online survey and administer the flowchart via an e-mail “panel” which was developed in Qualtrics that generated a survey link sent to each ORC at each WWTP.

The online survey began by asking the respondent basic information, such as their name, their title, and name of the facility, in which they could write-in answers. It then asks if their facility produces BS and if they land apply the BS by clicking “yes” or “no” to each question. The survey then follows the format of the flowchart, beginning with potential chemicals and treatment zones within each treatment process. For each plausible chemical

feed (indicated by the 12 pink ovals marked “C” in the flowchart), the same chemical list was displayed in the form of multiple choice, including the option to write-in unlisted chemicals or to check that no chemicals are added at the treatment zone. The online survey also included the treatment zones indicated by roman numerals, which coordinated with the flowchart, along with a multiple choice list of potential processes within a zone, which varied depending on the treatment zone identified. For example, potential processes identified in the preliminary treatment zone included “bar screen(s),” “grinder(s),” “grit chamber(s),” and a choice to identify unlisted processes by a write-in. But for other treatment zones, such as primary treatment or secondary treatment, the list of processes differed.

The survey included the use of tools such as “logic skip” to steer the ORC in the proper direction to reflect their specific treatment plant. For example, if the ORC responded “no” to having a tertiary clarifier, they were then directed to the next treatment step, rather than to questions about chemical feeds in tertiary clarifiers. The logic skip feature also made it simple for ORCs from treatment plants that do not land apply BS to simply respond “no” to the question asking if they land apply, and they were immediately directed to the end of the survey. Another way in which logic skip was useful was that it was easy to identify which facilities use BNR in their secondary treatment zone. If the respondent marked “activated sludge with biological nutrient removal” they would then be asked what type of BNR is used, such as, “nitrification but no denitrification,” “nitrification and denitrification,” “phosphorus removal only,” “phosphorus and nitrogen removal,” or a choice to identify an unlisted type of BNR by a write-in response.

While the primary purposes of the flow chart and survey were to target processes and chemicals likely to affect BS P quantity and form (soluble/nonsoluble), we made no assumptions about these. Instead, we made the flowchart and survey very general and comprehensive, and the survey questionnaire streamlined and efficient to foster gathering of treatment plant information.

From Qualtrics, survey responses were downloaded into a single Excel Spreadsheet. This information was also accessible via HTML form, where the responses from a single participant could easily be examined. The first step in analyzing the survey responses was to identify the participants who indicated that the WWTP land applies BS. Once these WWTPs were identified, the ORC was contacted via both e-mail and by phone to further explain the research and solicit their further participation by allowing their BS to be collected and analyzed. Most ORCs who responded to the online survey were willing to participate in this aspect of the study, although as previously mentioned not all responders indicated that their facility produces BS, or that the BS they produce are land applied.

Converting Online Survey to Binary Responses

The online survey results were imported into an Excel file with WWTP as rows. Responses to questions that were not binary, e.g., multiple choice questions that might elicit multiple responses from the list provided plus any number of “Others,” were converted into binary (Yes-No) responses. For example, the question, “What chemicals are added at C5?” might elicit Chemicals A, F, G from among those on the standard chemical response choice

list, as well as two “Others,” written in as T and U. All such questions were broken down into binary elements based on the supplied standardized list supplemented by any “Other” responses. E.g.: “Q1: At C5 is Chemical A added?” response, yes (1); Q2: At C5 is Chemical B added; response, no (0); At C5 is Chemical T added, yes (1), etc. This resulted in 121 positive response columns, meaning a response was recorded.

Three positive responses consisted of “Name of respondent,” “Title of respondent,” and “Name of facility.” For anonymity, ORC information obtained from these questions are not included herein. One of the survey questions was a “write-in” answer that asked the participant, “What are your most recent dry tons annual biosolids production figures?” for each class, if applicable. Due to the nature of our study, only participants that answered “yes” to “Does your facility produce biosolids?” and, “Are the biosolids produced by your facility land applied?” were retained. There were 247 additional “binary” response possibilities, or negative responses, that did not receive a response, indicating these processes or chemicals were not used by any participating WWTP, so these questions were removed from the analysis.

Each WWTP survey row in the Excel file was converted so that the BS identification number (ID) represented a BS instead of a WWTP. This was necessary as two of the WWTPs surveyed and sampled produced both Class A and Class B BS, in which sludge treatment processes differed between the classes. For these WWTPs, ORCs were asked to fill

out two surveys, one for Class A BS and one for Class B BS. Therefore, the online survey responses are identified by BS ID instead of a WWTP ID.

Biosolids Sample Collection

To facilitate the collection of the BS samples, qualifying WWTP facility addresses were entered into an Excel spreadsheet. An online mapping tool, BatchGeo (BatchGeo, LLC., Seattle, WA) was used to visualize each location on a map. Once locations were identified, sampling routes were developed. Sample collection was carried out over three seasons: Summer 2013, Fall 2013, and Winter 2014 (hereafter: Summer, Fall, Winter), in order to account for seasonal variability that may exist in BS production methods and P solubility.

Sampling materials included on each sampling trip included a plastic bin, sterile rubber gloves, a large plastic funnel, wide-mouth 250 ml high-density polyethylene (HDPE) sample bottles, a plastic scooper, safety goggles, lab coats, Kem wipes, de-ionized (DI) water, coolers, frozen blue ice, gallon-size plastic bags, and Sharpie markers for labeling. The funnel and scooper were washed with P-free soap, acid washed, and rinsed with DI water prior to each trip.

Sampling methods varied depending on the consistency of the BS. Based on literature values for BS slurries, we calculated that 1 L (four 250 mL bottles) of BS would more than suffice for our analyses. If the BS being collected was liquid in consistency, the funnel was used for collection. In these cases, the ORC would normally lower a long handled scoop into

their BS basin, and carefully fill four wide-mouth 250 mL HDPE sampling bottles. After collection, the funnel would be rinsed first with regular water, and then rinsed with DI water. For BS that had the consistency of cake, pellets, or sand, we filled the plastic bottles by hand using sterile rubber gloves. Sample bottles were tightly sealed with polypropylene caps; exteriors rinsed at a WWTP sink; labeled with date, location, and class; placed into plastic Ziploc bags; sealed; and subsequently placed in coolers with frozen blue ice. The samples were then transported to NCSU William's Hall laboratory and refrigerated at 4°C for no more than 21 days before samples were prepared for analysis. In Fall, BS 5 was collected from the wrong location within the treatment stream, so the Fall data for this BS was omitted from the analysis. In the Winter, there was a chemical contamination at the WWTP that produced BS 23, so this sample could not be obtained.

Laboratory Analyses

Dry Weight Analyses

To determine the dry weight content of the BS samples, Peters et al. (2003) method of dry matter (DM) determination of manures was employed. The bottles containing liquid BS were mixed and bottles containing BS with the consistency of cake or dryer were poured into a plastic Ziploc bag and then mixed. Subsamples of 20 g were measured into tared aluminum drying boats. First, the aluminum drying boats were labeled and weighed separately and weights were recorded to three decimal places. The aluminum drying boats were then placed back on the scale and tared. Biosolids subsamples were then placed into the boats and

measured to within 20.0-20.09 g and weights were recorded. Samples were then placed into an air circulating oven at 105°C and dried for 24 hours. After 24 hours, samples were weighed and measurements recorded. In order to ensure samples had come to a stable weight, they were re-weighed after six hours, but no samples required additional drying. Percent DM could then be calculated by first subtracting the aluminum drying boat tare weight, and then dividing the BS weight after oven drying by the original subsample weight (20 g), and multiplying by 100 to obtain a percent.

Determination of Water-Extractable Phosphorus

Water-Extractable P analyses followed the 100:1 (solution:solids) extraction ratio protocol recommended by Kleinman et al. (2007). As discussed by Kleinman et al. (2007), this ratio was reasonably balanced, precise, and well adapted for use in laboratories. It also had the highest correlation with dissolved runoff P, relative to the other ratios tested, which included 10:1 and 200:1.

Four subsamples of each BS were analyzed. For each BS subsample, the “as is” amount equivalent to 0.5 g dry weight, as determined by dry weight analyses, was placed in a 50 mL plastic centrifuge tube. Deionized water was added using a squeeze bottle for a total volume of 50 mL to achieve the final extraction ratio of 100:1, where solution included liquid already in material and the added DI water. The centrifuge tube was then capped tightly. Each tube was labeled using a numerical and alphabetical system.

Centrifuge tubes were then placed on an oscillating shaker and agitated for 1 hour at 150 rpm. They were then centrifuged in a RC5C centrifuge (Sorvall, Thermo Scientific, Asheville, NC) at 1500 x g (3600 RPM) for 10 minutes using a carbon fiber rotor, model number F21-8x50y (Fiberlite, Thermo Scientific, Asheville, NC). Each of the four replications from one set of samples was centrifuged at the same time to achieve centrifugal balance. Sample supernatant was then filtered through a Whatman #1 filter paper folded within a small funnel directly into a plastic scintillation vial, which was labeled using the numerical and alphabetical (replications a, b, c, d) system. Scintillation vials were immediately capped and extract was stored in a freezer at -18°C. The P concentration of the extract was determined using a Perkin Elmer Model 2000 inductively coupled plasma optical emission spectrometer (ICP) (Perkin Elmer, Waltham, MA).

Determination of Total Phosphorus

To determine TP, it was first necessary to oven dry enough of each “as is” BS in an air-circulating oven in order to achieve three replications of 2.5 g dry subsamples. In order to achieve this, the amount of “as is” BS to achieve 7.5 g was calculated using dry weight measurements previously obtained. The “as is” BS were poured into acid washed and labeled glass beakers, placed into an air circulating oven at 105°C, and dried to stable weight. After 24 hours, most samples were nearly dry, but it took an additional 24 hours to achieve a stable weight for large samples (those that had low solids content). After the 48-hour period, the dry BS were weighed out into the three replications each of 2.5 g into tared, acid-washed, and

labeled (using high temperature resistant wax pencil) ceramic crucibles. Samples were then placed in a muffle furnace and ashed at 500°C overnight in order to destroy the organic, combustible portion of the BS by thermal decomposition. Samples were allowed to cool. To solubilize the ash and its trivalent metal oxides and to convert pyro and meta phosphates into orthophosphate required for ICP analysis (Greweling et al., 1976), 2 mL DI water and 4 mL 6N HCl was added to each sample, which was stirred then heated to approximately 110°C and evaporated to dryness (~1 hr). For the Summer samples, this was done on a hot plate due to the unavailability of steam. The Fall and Winter samples were heated on a steam plate. An additional 4 mL 6N HCL was added to each sample, which was heated for approximately 1 min then poured into a 50-mL acid washed and labeled glass volumetric flask with the assistance of an acid washed small funnel and DI water. The DI was used to wash the sides of the crucible into the volumetric flask in order to capture all residue. Crucibles were scraped using a Teflon scraping tool and DI water, to ensure all contents would be collected, which was especially important on samples where residue had a tendency to stick to the bottom of crucible. The 50-mL volumetric flask was brought to volume using additional DI water. Flasks were then covered with parafilm and sat overnight. They were then mixed thoroughly by hand-shaking, and immediately filtered using Whatman #2V filter papers and small funnels directly into labeled scintillation vials. Samples were refrigerated no more than 2 weeks before being analyzed for P concentration via ICP.

Total Phosphorus Laboratory Error

During Summer, the crucibles used during the TP analysis were too small, and sample from one replicate of one BS and two replicates from another BS (2c, 13a, 13b) fizzled over the rim during TP analysis, due to the presumed reaction of HCl with lime within the BS. These were omitted from the replicate mean calculations. In subsequent seasons, larger crucibles were used to account for this reaction, and this issue did not persist. A portion of sample from two BS replicates were spilled in Summer (21b) and Fall (19b), and thus omitted.

Statistical Analyses

Biosolids Parameters Analyses

The BS parameters analyzed included TP, WEP, PWEP, and percent DM. Water-extractable and TP data were converted from mass:volume (mg P L^{-1}) to mass:mass (g P kg^{-1} BS). Percent DM was calculated as $\text{DM} \times 100$. For percent DM and TP within each BS, the three analytical replicates were averaged to produce a “replicate mean,” as were the four replicates of WEP. Percent WEP was determined by dividing the WEP replicate mean by the TP replicate mean and multiplying by 100 to obtain a percent. The consistency of the biosolids varied widely, which may have affected sampling and analytical error. Thus, we conducted an error analysis for each of the BS parameters using the CV of each replicate mean as the dependent variable. All other analyses were conducted on the replicate means. Descriptive statistics (mean, median, standard deviation [Std Dev], skewness [Skew],

kurtosis, coefficient of variation [CV], maximum, and minimum); frequency distribution histograms; correlations; and standard least squares analysis of variance (ANOVA) were calculated using JMP Pro, Version 10.0 software for Windows (SAS Institute, Cary, NC). Summary of fit, least squares means, and means comparisons (Student's t , $\alpha = 0.05$) were calculated for each parameter, including the CVs between BS replications and CVs between seasons. Herein, "difference" and "different" are used only when means comparisons were statistically significantly different at the $\alpha = 0.05$ level. For the ANOVA, the sampling design provided no legitimate error term against which a BS X Season could be evaluated. The effects of season were determined using BS's as replicates, and the effects of BS using seasons as replicates. In any analyses in which Summer, Fall, and Winter parameters were evaluated together, the term "seasons combined" was used. In describing correlations, r^2 greater than 0.8 were considered "very strong," 0.6 to 0.8 "strong," 0.4 to 0.6 "moderate," 0.2 to 0.4 "weak," and 0.1 to 0.2 "very weak."

Devising Wastewater Treatment Plant and Biosolids Categories from Online Survey

Results

The goal of the flowchart and online survey was to better understand what chemicals and processes are used at NC WWTPs in an attempt to determine if discernable relationships existed between these processes and chemicals and the P fractions within the resultant BS. If categories could be created representing WWTP with similar process characteristics, these categories could be statistically analyzed in regards to the P fractions within the BS. If

differences existed among P fractions for each category, the P fractions could then be assigned to separate WWTP categories within PLAT, assisting BS land appliers in reducing the risk of P-loss. Due to the complexity of the online survey results, multiple methods of interpretation were attempted. These included a hierarchical cluster analysis of the online survey, key components of the online survey, and comparisons by BS class. The cluster analysis grouped WWTPs representing different BS based on their surveyed characteristics. The key components analysis grouped BS based on the WWTP process characteristics that were used to produce them. These components were based on WWTP processes and chemicals that were most likely to affect P in BS, as interpreted by past studies and review of existing literature. Subsequently, BS class differences were examined, including their PSRPs and PFRPs.

Hierarchical Cluster Analysis of Online Survey

The first method attempted for interpreting the online survey was hierarchical cluster analysis, which is a technique of grouping data. The hierarchical cluster analysis was performed using JMP Pro, Version 10.0 software for Windows (SAS Institute, Cary, NC). The cluster analysis was performed by the Ward's minimum variance method. Since the online survey was converted to consist of only yes or no questions, each in a column, a hierarchical cluster analysis supporting nominal response columns was used. In an analysis of nominal columns, column responses that are identical between rows (WWTPs) contribute a distance of zero, otherwise they contribute a distance of one. The rows are grouped into

independent clusters based on a multidimensional distance matrix of dissimilarity and similarity. The clustering starts with “clusters” of one row, here individual WWTPs. At each step, the two closest clusters are grouped together. This process continues until all of the rows are combined into a single cluster. Each level of the hierarchy is characterized by the number of clusters it contains. The hierarchy is portrayed as an inverted tree called a dendrogram, which is a visual representation of the clustering sequence. The individual “clusters of one” are the leaves, the intermediate cluster combinations the branches, and the ultimate cluster of all branches, the trunk. It therefore shows which cluster each WWTP was in when it entered its next cluster in the combining process.

Characterizing differences between clusters

We characterized differences between two clusters using a strategy based on the number of WWTPs in each cluster that had responded positively to each individual binary question. For each question, we expressed these numbers as the percentages of the total number of WWTPs in their respective clusters. For each question, we then calculated the cluster difference by subtracting the percentage from one cluster from that of the other cluster. We then sorted the questions by these differences in order from largest to smallest. We interpreted differences near or equal to zero to indicate that the clusters were similar for that survey question, which was thus unlikely to be a factor distinguishing the two clusters. The larger the absolute difference, the more different the two clusters were for a particular question, indicating questions likely to have been important in distinguishing the clusters.

Key Components of Online Survey

In order to avoid the problems that arose when trying to weigh each positive survey response equally, key components of the online survey were developed that focused on factors thought to most readily affect BS consistency and the forms and quantities of P. Each BS was therefore represented by some combination of the key components. These key components were then used to compare and distinguish differences among the BS. As described in Chapter 1, these included many processes and chemicals used during wastewater treatment steps.

The key component tables were constructed by first assembling positive survey responses into groups, based on their similarity. These chemicals and processes therefore represented a simplified version of the survey, in which similar survey questions were grouped together within a key component. For example, if the facility added polymers in multiple treatment zones, these questions were combined into one. The key components therefore did not necessarily convey the treatment zone(s) in which the chemical was added or process occurred, but they provided a look-up table of all the major treatment processes thought to affect P.

Additional Biosolids Class and Treatment Comparisons

The differences among Class A and Class B BS pathogen reduction methods and consistency were examined. Subsequently, standard least squares one-way ANOVAs and Student's t means comparisons were performed on the effect of Class A and Class B on BS

parameters. Analyses of variance and Student's t means comparisons were performed for the effects on BS parameters of separating two groups of Class B BS by consistency, and separating two groups of Class A BS by different PFRPs.

Analyzing Effluent Discharge Phosphorus Limits with Biosolids Phosphorus

We hypothesized that relationships may exist between a facility's influent TP, discharge effluent TP, BS TP, P effluent limits, and respective watershed in which they are located. Evaluating facilities influent TP, P effluent limits, and watershed were limited, as many facilities are not required to report influent TP concentrations, some facilities do not have specific P effluent limits, and for some watersheds we had a limited amount of participating facilities.

To evaluate the potential relationship between discharge effluent TP and BS TP, the average effluent flow rates (MGD) and effluent mg TP L^{-1} concentration, as reported to the NPDES for 2013 and 2014 for each permitted discharge facility in the study, were compiled and effluent mass TP time^{-1} (Mg TP yr^{-1}) was subsequently determined. The BS TP seasons average mass concentration (g TP kg^{-1}) was determined from the study for each BS and the annual dry tons BS produced by facility, as reported in the online survey, were used to calculate the mass TP time^{-1} (Mg TP yr^{-1}) produced by BS for each facility.

Effluent mg TP L^{-1} may be influenced by influent mg TP L^{-1} , but the latter was known for only six facilities. The correlation between influent and effluent mg L^{-1} concentration for the six facilities was examined.

Each participating WWTP discharges effluent into a water body contained within a certain watershed. In order to see if the watersheds in which these facilities were located influenced effluent mg TP L^{-1} concentrations a standard least squares one-way ANOVA for the effects of watershed on effluent mg TP L^{-1} was performed.

CHAPTER 3: RESULTS AND DISCUSSION

Biosolids Parameters Analyses

Effects of Season on Sampling and Analytical Error Expressed as the CV of the Means of the Three Analytical Replicates

The summary statistics for sampling and analytical error of TP are shown in Table 2. The ANOVA test of seasonal effects on the CV of the analytical replicate means of TP had $F = 0.1$ (Table 3). Error was greater in Summer than Winter (CV = 12.9 versus 5.2%) but no other differences were detected. As previously discussed, errors occurred related to crucible size during the Summer TP dry digestion analysis, which may have affected the g TP kg^{-1} BS determined for these replications. The summary statistics for sampling and analytical error and effect of season on sampling and analytical error for WEP, PWEF, and percent DM were also examined (Tables 4 - 9). No differences among seasons were detected for CVs of these parameters. The mean CVs were 6.7, 12.0, and 1.6%, respectively.

Total Phosphorus

The frequency distribution histogram mean and median were similar for each season and combined over seasons (Fig. 3), indicating that TP was quasi-normally distributed. For Summer, Fall, and Winter combined, the mean, median, minimum, and maximum TP were 23.4, 23.6, 0.4, and 73.2 g kg^{-1} BS, respectively (Table 10). The skewness and kurtosis were close to 0, at 0.8 and 2.4, respectively. The frequency distribution histograms of the CV

within BS TP for all seasons and seasons combined were right skewed (Fig. 4) and the minimum and maximum for Winter were both outliers. The variability in TP within seasons, expressed as CV, ranged from 46.2% (Summer) to 48.4% (Fall) (Table 10).

Averaged over seasons, TP varied widely among BS (Table 11). Total P also varied among seasons (Table 12), although the proportion of total variance explained by the ANOVA model was low ($r^2 = 0.20$). Summer and Fall TP were similar (17.8 and 21.8 g kg⁻¹, respectively), but Winter TP was about twice as large (31.3 g kg⁻¹).

These TP values were similar to other studies. Although Brandt et al. (2004) did not compare P based on season, the TP values of the 41 BS within their study varied from 3.7 to 40.2 g kg⁻¹ with a mean of 24.4 g kg⁻¹ and median of 24.8 g kg⁻¹. So although our minimum and maximum had greater spread, the mean and median values were comparable. Similarly, the BS that were used in the runoff P study by Kleinman et al. (2007) had TP that varied from 16.5 to 40.4 g kg⁻¹ BS, although this range only included five BS. In the study by Maguire et al. (2001), in which eight BS were examined, TP concentrations ranged from 10.5 to 30.0 g kg⁻¹, with a mean of 21.3 g kg⁻¹.

Seasonal Total Phosphorus Variability

In attempting to understand why there was TP seasonal variation observed among the WWTPs, the literature discussed in Chapter 1 highlighted the complexity of the relationship between P, temperature, and WWTP processes. These factors can affect biological activity at WWTPs, which may result in increased or decreased P removal. As WEF (2006) reported,

PAOs may have a selective advantage over competitors for substrate accumulation at lower temperatures, which allows them to store more phosphate. Also, they reported that in one study, PAOs and non-PAOs could store poly-P at lower temperatures, as opposed to only the PAOs at higher temperatures. Although the WWTPs within our study did not indicate the specific use of EBPR, some did indicate the use of BNR that removes P and N, which could be an indication of anaerobic, aerobic, and anoxic zones that affect biological activity of PAOs. Understanding the specific biological treatment design and bacterial strains at the facilities in the study was not within the scope of this research, but given the complexity of the relationships between microorganisms, P, temperature, and WWTP processes, a degree of seasonal variation was expected. Averaged over BS, Winter TP was 9.5 g kg^{-1} more than Fall TP and 13.5 g kg^{-1} more than Summer TP (Table 12). Due to analytical error, it is unwise to definitively conclude that these differences were due to temperature and precipitation differences of seasons, although seasonal variation amongst BS differences is an area of research that deserves further exploration over multiple years.

Water Extractable Phosphorus

The frequency distribution histograms for WEP for each season and seasons combined were right skewed (Fig. 5). The skewness and kurtosis of the seasons combined were 2.4 and 5.6, respectively. The seasons combined mean, median, minimum, and maximum WEP were 1.4, 0.6, 0.02, and 9.1 g kg^{-1} BS, respectively (Table 13). The frequency distribution histograms of the CV within BS WEP for all seasons and seasons

combined were right skewed (Fig. 6). The three highest WEP values for each season were all outliers, which identified by ID number, were BS 3, 4, and 17 for Summer and Winter and BS 3, 17, and 30 for Fall (not shown). This contributed to the fact that median WEP was lower than the mean for each season and seasons combined, which indicated typical BS analyzed had even lower WEP than their average suggests. Seasonal average WEP varied widely among BS (Table 14) and BS 17 had the highest WEP value when averaged over seasons, 6.9 g kg^{-1} . The Summer, Fall, and Winter WEP means were 1.4, 1.0, and 1.7 g kg^{-1} BS (Table 15). In contrast to TP, no differences in WEP were detected among seasons. This may have been due to the substantial variability in WEP within seasons. Expressed as CV, these ranged from 132.2% (Winter) to 164.7% (Fall) (Table 13).

In comparison, the WEP values from seven BS analyzed by Kleinman et al. (2007) using the 100:1 extraction ratio, were all below 2 g kg^{-1} , although the range was not identified. Out of the 41 BS examined by Brandt et al. (2004), their data were also right skewed. The mean WEP in the Brandt study was 1.3 g kg^{-1} , which was only 0.1 g kg^{-1} less than the mean of BS combined in our study, and the medians were both 0.6 g kg^{-1} . The minimum WEP of the Brandt et al. (2004) study was 0.01 g kg^{-1} , which is 0.01 g kg^{-1} less than our study minimum for BS combined. Also, their maximum WEP was 8.86 g kg^{-1} , which was only 0.24 g kg^{-1} less than our maximum. In the study by Elliott et al. (2002), in which eight BS were examined, water-soluble P mean was approximately 2.2 g kg^{-1} , which included an N-Viro BS in which WEP was below the method detection limit of 0.04 mg kg^{-1} .

The minimum WEP was therefore the undetectable N-Viro BS and the maximum was 8.86 g kg⁻¹.

Percent Water-Extractable Phosphorus

The frequency distribution histograms (Fig. 7) for PWEP of each season and seasons combined paralleled those of WEP, in that they were also right skewed. The skewness and kurtosis were 2.6 and 7.5, respectively, for seasons combined. The mean, median, minimum, and maximum PWEP were 5.0, 2.9, 0.2, and 35.2%, respectively (Table 16), for seasons combined.

The frequency distribution histograms of the CV within BS PWEP for all seasons and seasons combined were right skewed (Fig. 8). The highest PWEP values for each season were outliers, which were BS 3, 4, and 17 for Summer, BS 17 and 30 for Fall, and BS 4 and 17 for Winter (not shown). These BS remained outliers when seasons were combined. Similar to WEP, this contributed to the fact that median PWEP was lower than the mean for each season and seasons combined, which indicates the average is an overestimation of the typical BS PWEP values. For example, the average PWEP value for the outlier BS 17 is 23.9%. This is almost fivefold greater than the seasons-combined average and more than eightfold greater than the median of seasons combined. The variability in PWEP across seasons, expressed as CV, ranged from 118.9% (Winter) to 143.7% (Fall) (Table 16). Averaged over seasons, PWEP varied widely among BS (Table 17). Interestingly, the BS with highest WEP value when averaged over seasons, BS 17, also had the highest PWEP

value averaged over seasons. There were no differences among seasons PWEF detected (Table 18), which was similar to WEP but contrary to TP.

Brandt et al. (2004) PWEF data for their BS were similarly right skewed, and their PWEF mean was the same as ours (seasons combined) and had a very similar median, 2.8%. Interestingly, the average over seasons of BS ID 17 (Table 17) was 23.9%, which was very close to the Brandt et al. (2004) maximum PWEF of 24.0%. The minimum PWEF in their study was 0.03%, which was even lower than the minimum for our study.

Percent Dry Matter

The percent DM of seasons combined was somewhat bimodal (Fig. 9), with the lower mode appearing skewed right and the higher mode skewed left, although there were more than twice as many BS with percent DM values below 20% than there were above 80%. This may have been an indication of the variety of treatment processes used in reducing BS moisture content during pathogen reduction steps, which result in BS with low or high percent solids. The skewness and kurtosis for seasons combined was 1.2 and -0.2, respectively. The mean, median, minimum, and maximum percent DM were 28.0, 15.2, 1.4, and 95.9%, respectively (Table 19), for seasons combined.

The frequency distribution histograms of the CV within BS percent DM for all seasons and seasons combined were right skewed (Fig. 10). There was only one outlier for percent DM among all seasons, which was BS ID 20 for Winter (not shown). Similar to WEP

and PWEP, median percent DM were lower than means for each season and seasons combined. Unlike WEP and PWEP, there was only one outlier, for one season, which indicates the average is not an overestimation of the typical BS percent DM, but that percent DM varied widely amongst the BS in the study. The variability in percent DM across seasons, expressed as CV, ranged from 114.3% (Fall) to 121.0% (Winter) (Table 19). Averaged over seasons, percent DM ranged widely (Table 20), but the ANOVA detected no differences among seasons (Table 21), which was similar to effect of season on WEP and PWEP, but contrary to TP.

Brandt et al. (2004) mean, median, maximum, and minimum total percent solids were 34.7, 21.0, 6.0, and 93.4%, which were all higher values than our study, except for the maximum percent DM value. In the study by Elliott et al. (2002), in which eight BS were analyzed, the mean, minimum, and maximum percent DM were 54.3, 14.2, and 93.2%, respectively. Unlike many of the BS in our study, which were considered “slurries,” these two studies considered their BS “cakes,” “pellets,” or a form of similar consistency.

Correlations

Correlations were examined, including WEP by TP, PWEP by TP, PWEP by WEP, TP by DM, WEP by DM, and PWEP by DM for each season and seasons combined (Figs. 11 - 16). Water-Extractable P was found to be weakly linearly correlated to TP for all three seasons ($r^2 = 0.25 - 0.26$), and for seasons combined ($r^2 = 0.23$) (Fig. 11). Percent Water-Extractable P was found to be very weakly linearly correlated to TP for Summer (Fig. 12, r^2

= 0.17) and for seasons combined (Fig. 12, $r^2 = 0.07$). However, PWEF was not found to be linearly correlated to TP for Fall or Winter (Fig. 12). These very weak or non-existent correlations suggest that TP alone cannot be used to estimate PWEF. Percent water-extractable P was strongly linearly correlated with WEP for each season and seasons combined (Fig. 13). Summer, Fall, Winter, and seasons combined r^2 were 0.97, 0.92, and 0.92, 0.89, respectively. Since WEP is a parameter within PWEF and they are strongly correlated suggests PWEF can be estimated solely by WEP and that TP may not be needed.

There were no linear correlations for Summer, Fall, or Winter between TP and percent DM (Fig. 14), WEP and percent DM (Fig. 15), or PWEF and percent DM (Fig. 16), but the scatterplots of each suggested curvilinear relationships. Therefore, for seasons combined, quadratic models were examined and the polynomial fit full models were significant (Figs. 14 - 16). For seasons combined, TP, WEP, and PWEF were all found to be very weakly related ($r^2 = 0.16, 0.15, \text{ and } 0.13$, respectively) with percent DM. The correlations of percent DM with the P parameters, although very weak, may have been an indication that treatment techniques at WWTPs, such as mechanical moisture reduction and thermal heat drying, may influence the total amount of P and P solubility within the BS product.

Unfortunately, not many other studies have evaluated correlations between BS TP, WEP, PWEF, and percent DM. Brandt et al. (2004) examined WEP by TP in BS and manures, but did not indicate the strength of this correlation, nor did they examine

correlations between PWEP and WEP. Therefore, we evaluated the potential correlations using their data and determined that there were correlations between some parameters. Their WEP and TP were very weakly correlated ($r^2 = 0.15$), and PWEP and WEP were strongly correlated ($r^2 = 0.86$) (not shown). We also observed curvilinear relationships in some scatterplots of their data, so we performed quadratic regressions of both TP and WEP on DM, which revealed weak ($R^2 = 0.35$) and very weak relationships ($R^2 = 0.16$), respectively (not shown).

Online Survey Responses

There were 121 positive responses in the online survey (Table 22) and 247 negative responses (Table 23). Out of the 121 columns of responses 100% of participants answered “yes” to measuring total P effluent, as all discharge facilities within NC report and monitor effluent P to the NPDES. There were 23 response columns in which 50% or more ORCs marked that they use a certain treatment process or chemical for their respective BS. The questions with the highest frequency of responses (not including questions with 100% response described above), 92.9%, indicated the use of grit chambers in preliminary treatment and that liquid from sludge thickening is returned to primary treatment. Out of these same 23 most frequently answered questions, nine relate to various treatment zones in which no chemicals are added. Polymers and gravity belt thickeners are used in sludge thickening zones by 67.9 and 60.7%, respectively. In the dewatering zone, polymers are used

and belt presses are used by 60.7 and 50.0%, respectively. Also, only 67.9% measure TP in influent, likely due at least in part because doing so is not required by all facilities.

Of the 26 participating facilities, two produced both Class A and Class B BS. Total, there were eight Class A BS and 20 Class B BS, which represent 28.6 and 71.4% of participants, respectively. There were 37 questions that received only one response, in which there were many “write-in” answers concerning specific chemicals and processes. For example, chemical additions such as sodium hypochlorite and sulfur dioxide (which are used in disinfecting and dechlorination), were used by two WWTPs, but at separate treatment zones.

All 28 of the BS analyzed came from WWTPs that used some form of biologically activated sludge process, but only 12 used a form of BNR. Of the 42.9% that use some form of BNR, 3.6% manipulate their systems to maximize nitrification, 14.6% for nitrification and denitrification, and 25.0% reported that they are trying to remove both N and P. Although the online survey asked what type of BNR was used, it did not elaborate on specific manipulations, other than these main processes.

One facility, BS 17, was unique in that it was the only one that uses what is known as wet air oxidation (WAO) to assist in stabilizing the sludge and removing the solids from the supernatant. This is a physicochemical process where, under high temperature and pressure, oxygen reacts with organic matter, with the goal of converting the substances to simpler forms, or complete oxidation to CO₂ and water (Khan et al., 1999).

Hierarchical Cluster Analysis of Online Survey

Once our hierarchical cluster analysis was performed, we needed to decide which cluster combinations to consider. Since there were 28 surveys, there were 27 cluster combinations created. When performing a cluster analysis, one important decision that needs to be made is deciding on the number of clusters to use to characterize the subjects. As Qualtrics (2014) notes, clusters should identify distinct separations. Unfortunately, when examining our cluster analysis dendrogram and its associated distance graph (Fig. 17), interpreting cluster characteristics and separations proved difficult. The greatest separation apparent in the distance graph of the cluster dendrogram was between the two penultimate clusters. Therefore, we started by examining these two clusters, which we call the “Two-Cluster Model,” to see if we could recognize patterns amongst responses. This model split the online surveys representing each BS’s WWTP treatment processes into two groups (Fig. 18).

Differences Between Two Clusters

In examining the percentage differentials in the Two Cluster Model (Appendix, Table 2), we found that many survey responses were similar between clusters. There were nine positive survey responses that were most similar between the two clusters, all with differentials of one percentage point. These responses were therefore unlikely to distinguish the clusters. These included: lime added at primary treatment, centrifugation during sludge thickening before stabilization, heat drying stabilization, pasteurization stabilization,

dewatering sludge using a drying bed, not having a dewatering process, ferric chloride added at dewatering, consistency of cake for Class A BS, and dusty/powdery consistency of Class A BS. Additionally, there were 52 more positive responses with absolute differentials less than 10 percentage points, indicating that these positive responses would also be unlikely to distinguish the BS.

The five Cluster 1 characteristics that most distinguished it from Cluster 2 included no chemicals added at sludge thickening, no tertiary filter, influent TP not measured, liquid from stabilization returned to influent, and gravity sludge thickening before stabilization, with absolute differentials of 69.2, 62.6, 61.5, 52.3, 48.2 percentage points, respectively. The five Cluster 2 characteristics that most distinguished it from Cluster 1 included filter backwash discharged into influent, polymers added at sludge thickening, influent TP measured, no chemicals added to filter backwash, and a tertiary filter, with absolute differentials of 70.3, 69.2, 69.2, 62.6, and 62.6 percentage points, respectively.

After cluster differentials had been performed and described, the next challenge was to interpret the results in the context of WWTP processes, e.g., what did the clusters “mean,” and what, if any, relationships might they have with the P parameters?

As discussed by Hennig and Liao (2013), one challenge when defining clusters is related to a phenomenon coined “substantial aspect.” This concept concedes that the researcher may have knowledge concerning the subject matter that “may suggest that some variables are more or less important for clustering.” In their article, they clustered socio-

economic stratification data. Characterizing this data by discrete variation can seem warranted, but can ultimately produce noticeably artificial cluster differences. For example, regarding substantial aspect in Hennig and Liao's socio-economic study, "owns" and "pays rent" were their strongest categories. They therefore weighted these two categories within their data more heavily as means to convey the important of owning or renting, as related to socio-economic status. Obviously, this type of weighting system introduces bias into the clustering procedure and creates a challenging inference problem. This preprocessing of variables within the set of data could be deemed logical, despite the introduced bias, but changing the weight of some variables can completely alter the cluster combinations, potentially leading to additional concerns regarding substantial aspect and other challenges.

In our hierarchical cluster, each question was weighted equally. While this may have been a well-constructed unbiased approach, the results may indicate that substantial aspect should be considered. Without factors that discriminated amongst the importance of individual questions, creating categories summarizing WWTP characteristics was a difficult task. In the future, dropping responses deemed unimportant and performing a new cluster analysis may be warranted, although deciding which questions to drop would take careful consideration.

In our study, there were many positive responses in which cluster differentials were near zero, implying the responses could therefore not be a characteristic that distinguished one cluster from another. For example, heat drying stabilization was one of the positive

responses in which the differential between clusters was 1.0. But as discussed in Chapter 1, thermal treatments are a common practice to meet PFRP and substantially alter the consistency of the BS. Not differentiating the survey responses by a characteristic such as thermal heating may ultimately prove our cluster categories artificial.

Similar variables within the online survey also made distinguishing their relative importance quite challenging. For example, the use of sludge thickening polymers was a major variable distinguishing Cluster 2 from Cluster 1. Out of the 15 surveys in Cluster 2, 100.0% of them used polymers in sludge thickening. Only 23.1% of Cluster 1 used polymers at sludge thickening. However, 78.6% of all facilities added polymers somewhere in their treatment stream, but not necessarily during sludge thickening. Problems arose in deciding if some treatment zones were “more important” than others in adding polymer. Complicating this matter, quantities and types of polymers added at each treatment zone were unknown.

As discussed by Qualtrics (2014), cluster procedures are “pre-classificatory” in that the researcher has not partitioned the rows of data using prior judgment. It is assumed, however, that there is some level of heterogeneity in the data, or that “clusters” exist based on independent variables having commonalities. Since no formal categorical definitions had been assessed prior to the clustering, we experienced major difficulties in forming summary measures of each cluster in a descriptive sense to adequately define the clusters; therefore, drawing inference on their statistical significance was a challenge. Differentials between

responses were examined, but many of the largest differences between clusters were hard to explain in a relevant context.

While the cluster characteristics may be relevant in the context of mapping treatment systems, they proved difficult summary measures for actually discriminating and defining our cluster categories. Despite the characterization of the Two Cluster Model, we have not yet been able to ascribe real meaning to the clusters. This inability to summarize positive responses of each cluster in an appropriate descriptive sense made defining clusters too difficult from the perspective of distinguishing the BS by categories to be used in PLAT. Therefore, it became imperative to try other approaches for determining discernible differences amongst positive survey responses for categorizing BS by treatment processes and their potential correlations with BS TP, WEP, PWEF, and percent DM.

Analysis of Biosolids Parameters Between Two Clusters

Although we found it difficult to adequately characterize the cluster combinations, we did test for the effects of cluster on the BS parameters. We used a two-way ANOVA to test whether there were interactions between seasons and clusters at the level of two clusters, but found none, so the reduced one-way ANOVAs were used to examine differences in BS parameters between clusters. In the ANOVA of TP with two clusters as model effect, there were differences, although the proportion of total variance explained by the model was low (Table 24, $r^2 = 0.09$). Least squares means differences (Student's t , $\alpha = 0.05$) determined that Cluster 1 TP was less than that of Cluster 2.

In the ANOVAs of WEP, PWEF, and percent DM, with two clusters as model effect, differences were not detected (Tables 25 - 27). Also, in examining the unprotected LSD (Student's t , $\alpha = 0.05$), no difference was detected between the cluster means for WEP, PWEF, and percent DM.

Although there was a difference between clusters for TP, due to the aforementioned difficulty associated with summary measures in defining clusters and the substantial aspect problems that arose, we decided to pursue other methods of evaluating our online survey positive responses.

Key Components of Online Survey

The key components, as adapted from the online survey positive responses, were biological treatments, metal salt treatments, physicochemical treatments, physical dewatering mechanisms, alkalization, and thermal stabilization. Each key component included at least one sub-component.

The biological key component included the sub-components activated sludge, BNR, aerobic stabilization, and anaerobic stabilization (Table 28), as these were the biological survey positive responses thought to most likely affect P. All WWTPs indicated that they had an activated sludge process, which is not surprising, as taking advantage of microbial reactions to flocculate and stabilize sludge can be a very effective method of removing solids from the supernatant in preparation for effluent discharge. All but three BS were a result of anaerobic or aerobic sludge stabilization, which are very common methods of meeting

PSRPs requirements, and are thought to affect P. Additionally, 42.9% of the BS were a result of a BNR process, which is also thought to affect P within a BS, although as discussed in Chapter 1, the method of BNR can greatly influence the amount of P removal.

The metal salts sub-components identified from the positive survey responses included the use of ferric sulfate, ferrous sulfate, ferric chloride, sodium aluminate, alum, polyaluminum chloride, and polymers (Table 28). The Al- and Fe-based chemicals were identified as most likely to affect P, as they commonly form metal phosphate precipitates, removing soluble P from the wastewater stream (Maguire et al., 2001), which may decrease BS WEP. Polymers were also included, as these are commonly used to aid in the precipitation of P with these metal salts and to assist with coagulation of solids. They were used by 78.6% of facilities somewhere in the treatment process.

We included the physicochemical sub-component WAO stabilization as a process thought to affect P (Table 28), although not discussed in Chapter 1. The processes involved in this method introduce high temperature and pressure, causing O₂ to react with the sludge, oxidizing organic matter to CO₂, water, and intermediate products, and reducing total solids content. The nature of, and the degree to which, solids are produced depends on specific system manipulations of time, temperature, and pressure. Phosphorus is converted into inorganic and soluble phosphates. To remove this dissolved P, along with other aqueous compounds, biological treatments often accompany WAO (Khan et al., 1999). Therefore, the degree to which soluble P will be removed from the wastewater stream depends on additional

treatments after WAO. The only WWTP that used WAO in our study was the WWTP that produced BS 17. This WWTP had an activated sludge process and lime chemical feeds in preliminary, primary, and secondary treatment zones. However, they did not report the use of BNR or aerobic or anaerobic sludge stabilization, and they did not report using any metal salts precipitates or other sludge stabilization techniques. Interestingly, this BS was an outlier with one of the highest WEP and PWEF for seasons combined; when averaged over seasons, it had the highest WEP and PWEF in our study.

Dewatering methods were also included in the key components (Table 29), because as discussed in Chapter 1, physical moisture reduction can greatly reduce the soluble P in the BS final product (Brandt et al., 2004). Dewatering can therefore be an important process in decreasing the potential for P-loss via runoff, as concluded by Withers et al. (2001). Sub-components of dewatering included the use of a centrifuge, gravity, gravity belt thickener, belt press, drying bed, and decanting from an aerobic digester. Processes such as centrifugation, which was performed by four facilities in the dewatering zone, could therefore have influences on BS WEP.

The sub-components of alkalization included Class A alkaline stabilization, Class B alkaline stabilization, and additions of bases, including lime, magnesium hydroxide, sodium hydroxide, and sodium bicarbonate (Table 29). As discussed in Chapter 1, Class A alkaline stabilization, which was performed by 14.3% of WWTPs in the study, is a common method of reaching Class A PFRP reduction requirements. Class B alkaline stabilization was

performed by 7.1% of WWTPs in the study. These processes are thought to exert influence on BS P by Ca-phosphate precipitation and adsorption (Brandt et al., 2004) and/or by diluting the BS with a product such as lime kiln dust (O'Connor et al. 2004), both of which increase BS pH and can affect WEP and TP. The other alkaline chemicals included as alkalization sub-components are often used as alkalization supplements. There were 12 WWTPs identified as using such chemicals in the study.

Heat stabilization was the sub-component identified as a thermal drying key component used by 14.3% of WWTPs in the study. As described in Chapter 1, this process is a common method of PFRP reduction requirements. It can greatly reduce the liquid content of sludge, as water is evaporated from the solids, and can alter the forms of P, potentially decreasing BS PWEF (Brandt et al., 2004).

The key components of the online survey gave a better visual understanding of processes performed and chemicals used at each participating WWTP in the study. They also indicated WWTP processes thought to most affect P in BS. However, these components were still very complicated and variable among and within BS. Developing clear categories to summarize these BS production processes was still not accomplished. The key components were instead used as a guide going forward in distinguishing BS by major pathogen reduction methods that substantially alter the BS, as we pursued means of distinguishing categories for the BS in our study.

Biosolids Class A and Class B Differences

Due to the complexity of treatment processes used in producing BS, Brandt et al. (2004) categorized BS by “final treatment process.” Their research influenced our strategy going forward to differentiate the BS in our study, in that we placed more emphasis on certain zones of BS treatments.

Interestingly, we found a trend existed, in which class of BS was related to consistency. Class A BS seemed to have higher solids content than Class B BS. Class A were described in the survey as “dry cakes” and “pellets”, while Class B BS were described as the “slurries” and “wet cakes.” Instead of distinguishing the BS by multiple treatment processes, we hypothesized that grouping BS based on their pathogen reduction class – either Class A or Class B – could be a useful strategy in devising BS categories in PLAT. The Part 503 Rule differentiates BS class by PSRP and PFRP, in which Class A BS must undergo processes that are more stringent, including longer durations of high heat and pH, which may have different effects on P and BS consistency than less stringent processes used to produce Class B BS. The relationship between class and BS parameters in this study were therefore examined.

The mean TP of Class A and Class B were 16.7 and 26.1 g kg⁻¹, respectively, which were different (Table 30), but the strength of this relationship was very weak ($r^2 = 0.12$). The mean WEP of Class A and Class B were 0.8 and 1.6, respectively (Table 31), but no difference was detected, perhaps due to substantial variability (CV = 150%). The mean PWEF of Class A and Class B were 3.4 and 5.6, respectively, between which there was also

no difference detected (CV = 132%) (Table 32). The mean percent DM (Table 33) of Class A and Class B were 75.3 and 9.3, respectively, which were different and had a very strong relationship ($r^2 = 0.85$).

Although TP and percent DM differed between Class A and Class B BS, within Class A BS, TP also appeared to be related to DM content. This led us to reexamine the correlation between TP and percent DM (Fig. 14). However, this reexamination needed to be in the context of treatment processes that might explain why TP within BS that were Class A appeared to be related to percent DM.

Comparing Class A Alkaline Stabilization, Class A Thermal Heat Drying, Class B-slurry, and Class B-cake Biosolids Types

As a first step in reexamining the relationship between TP and DM, we wanted to understand major processes that might differentiate DM content among BS. As described in Chapter 1, the PFRP to meet Class A pathogen reduction requirements can vary greatly, which can have profound impacts on P fractions and final BS consistency. Using this knowledge, Class A BS in the study were divided into groups based on their pathogen reduction processes: alkaline stabilization (Class A-alkaline) and thermal heat drying (Class A-heat). These categories were similar to the BS categories used by Brandt et al. (2004), “BPR N-Viro” and “Heat dried.” Although “alkaline stabilization” for Class A and Class B was not formally distinguished on the online survey, those that used “alkaline stabilization” that resulted in a Class A BS were considered Class A alkaline stabilized, implying they used

PFRP. Thermal stabilization included any Class A BS that listed “heat drying” as their stabilization method.

As discussed previously, all but three BS in our study used aerobic or anaerobic sludge stabilization (ID numbers 15, 17, and 27). Biosolids 15 relied on Class A alkaline stabilization to reduce pathogens and had very low P parameter values. Biosolids 27, which had P values close to average for Class B BS, relied on Class B alkaline stabilization. Interestingly, BS 17, which is a Class B BS, had the highest WEP and PWEF values when averaged over seasons. This suggests that aerobic and anaerobic digestion to stabilize sludge may be important factors in reducing WEP and PWEF if alkaline stabilization is not used.

Besides these differences, Class B BS were hard to distinguish based on specific treatment processes. Most Class B BS that were considered cakes used anaerobic stabilization and most considered slurries used aerobic stabilization, but there were some exceptions. Additionally, the use of alkaline materials, metal salts, and dewatering techniques varied greatly among these groups. Most Class B cakes were produced from belt presses and most Class B slurries were from gravity belt thickeners, but there were exceptions or some from more than one dewatering mechanical tool. Since the result of dewatering techniques is most exemplified by their final consistency, rather than the specific mechanical tools used in dewatering, the two groups were divided by their differences in consistency. There was a gap between 6.3 and 12.6% solids, separating the Class B slurries from the Class B cakes.

Therefore, Class B slurries (Class B-slurry) were in the lower percent DM range, 1.4 to 6.3%, and Class B cakes (Class B-cake) were in the higher percent DM range, 12.6 to 24.3%.

In our study, we had 32 BS that were Class B-slurry, 28 Class B-cake, 12 Class A-heat, and 11 Class A-alkaline for seasons combined. Therefore, any relationships that were explored were in the context of unequal sample sizes, with about two to three times more Class B BS than either Class A BS types. To explore the relationships of the BS P parameters with Class A-alkaline, Class A-heat, Class B-slurry, and Class B-cake, SLS one-way ANOVAs of these effects were performed.

The TP of Class A-alkaline, Class A-heat, Class B-slurry, and Class B-cake (Table 34) were 5.0, 27.5, 25.9, and 26.4 g kg⁻¹, respectively, among which there were differences. The Class A-heat, Class B-slurry, and Class-B cake TP values were more than five times greater than that of Class A-alkaline.

The WEP of Class A-alkaline, Class A-heat, Class B-slurry, and Class B-cake were 0.07, 1.4, 2.2, and 0.9 g kg⁻¹, respectively, among which there were differences (Table 35). Class B-slurry was not different from Class A-heat, but was greater than Class B-cake and Class A-alkaline. No difference was detected between Class A-heat and the other BS types.

The mean PWEF of Class A-alkaline, Class A-heat, Class B-slurry, and Class B-cake (Table 36) were 1.9, 4.8, 7.5, and 3.3%, respectively, among which there were differences. Similar to WEP, Class B-slurry was not found to be different from Class A-heat, but was

greater than Class B-cake and Class A-alkaline. No difference was detected between Class A-heat and the other BS types.

The mean percent DM of Class A-alkaline, Class A-heat, Class B-slurry, and Class B-cake (Table 37) were 57.5, 91.6, 3.2, and 16.8%, respectively, among which all were different. Class B-slurry percent DM was about 5 to 29 times less than that of the other BS types.

The linear correlation of WEP with TP for seasons combined (Fig. 11) was reexamined, with markers used to designate the four BS types to illustrate differences (Fig. 19). This graph illustrates the ANOVA (Table 34), in that TP mean of Class A-heat, Class B-slurry, and Class B-cake were not different, but Class A-alkaline was different from the other BS types. Additionally, it illustrates why there were no differences between Class A-heat and the other BS types found in the ANOVA (Table 35) for WEP, although Class B-slurry was greater than Class B-cake and Class A-alkaline.

The quadratic relationship of TP and percent DM for seasons combined was also reexamined, with markers used to designate the four BS types to illustrate differences (Fig. 20). Examining the x-axis in the fit model illustrates the nature of the DM differences among the BS types (Table 37) in the ANOVA. It also illustrates the ANOVA (Table 34) for effects of BS type on TP, in which Class A-alkaline was much less than all other BS types. The fit model plotted with markers illustrated that although BS with different treatments varied by

solids content, the TP of Class B-slurry, Class B-cake, and Class A-heat were similar, suggesting PSRP and PFRP can yield similar TP.

The quadratic relationship of PWEF with percent DM for seasons combined was also reexamined, with markers used to designate the four BS types to illustrate differences (Fig. 21). Just as with the quadratic regression of TP and percent DM, examining the x-axis in this correlation illustrated why percent DM was different in the ANOVA (Table 37) for effects of BS type on percent DM. It also illustrated the ANOVA (Table 36) for effects of BS type on PWEF, in which Class B-slurry and Class A-heat were not different but Class B-slurry was more than Class A-alkaline and Class B-cake. The fit model plotted with markers illustrated that although BS with different treatments varied by solids content, PSRP and PFRP can yield similar TP. It also suggests that if the Class B-slurry outliers were not considered, Class B-slurry would not be more than other BS types.

Additionally, reexamining the quadratic relationships of PWEF and TP with percent DM for seasons combined using markers further validated our decision to categorize Class A BS by treatment type instead of by consistency. We discovered that a Class A alkaline stabilized BS from Summer had 15.9 to 21.3% higher percent DM than for the same BS in Fall or Winter, and about 30% higher percent DM than any other Class A alkaline stabilized BS for any season. According to the ORC at this facility, we subsequently determined that during the summer when this BS was sampled, the WWTP's alkalizing equipment unintentionally added extra lime kiln dust. Although our study determined that mean percent

DM for Class A-alkaline was 57.5%, this incident confirms that alkaline stabilization is capable of producing BS with much higher percent DM, although to meet PFRP they do not need to add this extra alkaline material and economically it would be impractical. This reinforces our decision to categorize Class A BS based on treatment process, rather than DM consistency, as placing this BS in a category based on percent DM would highly overestimate TP, as lime kiln dust substantially diluted the BS P.

Differences Within Biosolids Class

We also examined the differences in P parameters between Class A BS (Tables 38 - 41) and between Class B BS (Tables 42 - 45). For TP, there were strong differences between Class A-alkaline and Class A-heat (Table 38), which suggested that Class A alkaline stabilization yields much lower TP than heat drying, although both achieve Class A status. In the comparison of WEP between Class A-alkaline and Class A-heat, Class A-heat was 20 times greater than Class A-alkaline (Table 39), although both had relatively low WEP compared to Class A-heat TP. Therefore, Class A alkaline stabilization can not only produce BS with low TP relative to heat drying, but also can produce even lower WEP than heat drying. The two Class A BS groups also were different for PWEF, as Class A-heat was more than twice as large as Class A-alkaline (Table 40). Despite these differences, our findings showed that Class A alkaline stabilization and Class A heat drying were capable of producing PWEF values of less than 5%. The Class A alkaline stabilized BS had about 30% less dry matter than Class A thermally heated BS (Table 41).

No differences in TP were detected between Class B-slurry and Class B-cake (Table 42). However, WEP of Class B-cake was more than twice as large as that of Class B-slurry (Table 43), although for both Class B groups, WEP was low in relation to TP. The P_{WEP} of Class B-slurry was more than double that of Class B-cake (Table 44). There were also strong differences between mean percent DM of Class B-slurry and Class B-cake BS (Table 45), as expected, since the two Class B groups were separated based on consistency. The mean percent DM of Class B-cake was about five times greater than Class B-slurry.

Relationship of Effluent Discharge Phosphorus Limits with Biosolids Phosphorus

We examined relationships between a facility's influent TP, discharge effluent TP, BS TP, P effluent limits, and the watershed in which it discharges. Influent TP, discharge effluent TP, and BS TP are thought to be related, as they would be expected to be mass balanced. However, evaluation of some of these parameters, including influent TP, P effluent limits, and watershed, were limited, as some facilities were not required to report influent TP to the NPDES, some did not have specific regulated P effluent limits, and we had a limited number of WWTPs per watershed.

Testing whether BS TP was related to P effluent limits proved difficult, as nutrient reduction strategies varied considerably by water body and watershed, affecting whether effluent limits of any type are even imposed on facilities. Therefore, actual average effluent TP concentrations – rather than P effluent limits – were used to test whether relationships existed between effluent P and BS TP.

The BS Mg TP yr⁻¹ was found to be very weakly correlated with effluent Mg TP yr⁻¹ (Fig. 22). Although effluent mg TP L⁻¹ and BS TP concentrations will be influenced by influent mg TP L⁻¹, the latter was unknown for many of the WWTPs, as they were not regulated to monitor or report this information. Of the six facilities for which data was gathered, the mean, minimum, and maximum influent P were 5.3, 4.3, and 6.0 mg P L⁻¹, respectively, indicating relatively low variability. There was no correlation between influent and effluent TP concentration for the six facilities (not shown).

Each participating facility discharged its effluent into a water body contained within a certain watershed (Fig. 1). We were therefore interested to see if effluent TP differed among watersheds, as some watersheds differ by effluent P limits. Watersheds that did not have more than one WWTP were not included in the analysis. These included Broad, New, Tar Pamlico, Little Tennessee, French Broad, and Pasquotank, with effluent TP concentrations of 2.8, 1.2, 1.0, 1.0, 1.0, and 0.2 mg L⁻¹, respectively. The ANOVA for the effects of watershed on effluent mg TP L⁻¹ (Table 46) determined that there were differences amongst watersheds ($r^2 = 0.68$). However, every watershed shared a similarity with at least one other watershed. No difference was detected between the Cape Fear and the Yadkin, in which means were 3.1 and 2.4 mg P L⁻¹, respectively. There were no differences detected among Yadkin, Lumber, or Catawba. There were also no differences detected between Lumber, Catawba, and Neuse. Lumber and Catawba had effluent concentrations of 1.5 and 1.0 mg L⁻¹, respectively. Neuse had effluent concentration limits below 1 mg L⁻¹. Evaluating the facilities' effluent

concentration limits and nutrient strategies may shed light on why some effluent concentrations appeared to be lower than others. Although, as previously discussed, some facilities lack effluent limits or nutrient strategies.

The Neuse watershed was the only one with set effluent concentration limits for all the discharge facilities. The mean effluent TP for the seven WWTPs surveyed in the Neuse was 0.7 mg L^{-1} , which was 1.3 mg L^{-1} below their 2 mg L^{-1} effluent concentration limit. Two participating facilities (in the Catawba and Cape Fear) had mass TP day^{-1} or mass TP summer^{-1} TMDL restrictions on effluent TP. The Yadkin watershed nutrient strategies are currently being developed (M. Templeton/NCDENR, personal communication, 2014). Due to the inconsistencies in nutrient reduction limits and strategies between watersheds and between facilities within the same watershed, we could not conclude that such reduction strategies necessarily influenced discharge effluent TP or BS TP.

Considerations for Future Studies

Surveying Wastewater Treatment Plant Production Processes

In future studies concerning P solubility and its relationship to WWTP processes, deviations from survey methods of this study would be recommended. As discussed, although it seemed appropriate initially to request detailed online survey responses, this made analyzing the survey responses cumbersome and impractical from the perspective of creating discrete categories based on treatment processes directly from survey hierarchical cluster

analysis. Instead, we recommend a simpler survey, based on what is likely to affect P and BS consistency, which would likely be more easily analyzed in relation to creating BS categories from WWTP processes. From our study, this could be done by re-analyzing something similar to our key components in a hierarchical cluster analysis, although difficulties such as substantial aspect and separating cluster combinations may still arise and some questions may need to be modified.

Additionally, since the majority of WWTPs in our study relied on aerobic or anaerobic sludge stabilization to meet Class B PSRP and alkaline stabilization or heat drying as PFRP for Class A BS, it would be of interest for future studies to examine more pathogen reduction strategies allowed under Part 503. Future studies might further examine relationships between and within PSRP and PFRP strategies in producing the different classes of BS, including before and after pathogen reduction processes and how different processes are related.

Total Phosphorus Analysis

It is possible that TP was underestimated in this study due to analytical error and possible short-comings of the TP HCl dry digestion method. If so, actual PWEF would be lower than determined, and soluble P-loss risk would have been overestimated. The dry ashing-HCl digestion used in this study was based on a method designed for elemental analysis of plant tissue (Greweling et al., 1976), which is used routinely by the Environmental and Agricultural Testing Service, Dept. of Soil Science, NC State University

for the analysis of P in both solid and liquid wastes. Other studies such as Brandt et al. (2004) and Maguire et al. (2001) used USEPA-approved elemental analysis such as methods 3050 and 3051 for digestion, although P is not listed as one of the elements to be determined by these methods. In the 3050 method, which is the conventional USEPA method, BS are digested in nitric acid and hydrochloric acid (USEPA, 1986). In the 3051 method, BS are digested in nitric acid and subsequently microwaved (USEPA, 1994b).

In the study by Maguire et al. (2001), different methods of BS P analysis were compared for eight different BS. This included a nitric-perchloric acid digestion, USEPA 3050 acid-peroxide digestion, and USEPA 3050 acid ammonium oxalate P extraction, where P means were 21.3, 16.5, and 21.2 g kg⁻¹, respectively. Thus, the acid-peroxide digestion yielded 80% of the other methods, although all three methods of measuring P were correlated very strongly with one another. This study found that most of the P in BS is in a form that is easily extracted by the oxalate method, which is known for removing P from Fe and Al oxides in soil. However, when lime was added to BS, oxalate was not as effective as the other two methods, which they speculated may have been due to precipitation of Ca oxalate.

In our study, we speculate that the HCl may not have been strong enough or may not have been in enough quantity to fully solubilize the TP in the BS, and/or other compounds may have interfered with total TP digestion. Although as described, methods used by other studies may also not be accounting for all P that is within their given BS. As previously discussed, our BS mean and median TP g kg⁻¹ were both only about 1 g kg⁻¹ less than the

mean and median in the Brandt et al. (2004) study, and our maximum TP was actually 33 g kg⁻¹ larger than their maximum. Similar results were found in comparisons with other studies. Although the treatment processes that produced the BS in these studies differed, the fact that our summary statistics were comparable suggests that we may have in fact accounted for most of the TP within our BS. A consistent and reliable standardized BS TP digestion method needs to be developed in order to facilitate direct comparisons.

CHAPTER 4: CONCLUSIONS

Phosphorus Solubility in North Carolina Produced Biosolids

Applying BS based on agronomic N rates will most likely surpass the P needs of the crops. This study, however, determined that the amount of WEP and PWEF within BS are relatively low, when compared to other soil amendments, such as TSP and manures, examined from other studies. Since dissolved P in runoff has been found to be controlled by P solubility, the potential for soluble P loss from land-applied BS could be mitigated by WWTP processes that reduce PWEF. We found that processes used to meet nutrient effluent discharge limits and processes to meet BS pathogen reduction requirements reduced the soluble forms of P in BS substantially. We also found a strong correlation between PWEF and WEP. If an estimate of PWEF is desired in order to estimate soluble P-loss potential, our research indicates that determining WEP alone may be sufficient with no need to determine TP. Further study is needed to confirm the PWEF-WEP relationship and determine whether it is generalized over a wider range of BS than in our study.

We determined that TP in BS varies, depending on type of BS and treatment process. Therefore, predicting TP P-loss risk is a function of WWTP processes, and is especially influenced by methods of Class A pathogen reduction. Understanding the potential environmental implications will be a function not only of the type of BS being applied, but also the method of application and amount of BS applied. We also determined that seasonal variability, as influenced by microorganisms, temperature, and precipitation, may alter

amounts of TP within BS. In our study we determined that Winter TP was different than both other seasons when averaged over BS, in which it almost 10 g kg^{-1} more than Fall and nearly doubled that of Summer (Table 12). Due to TP analytical error, we cannot definitively conclude that these differences were due to change in seasons, although this is an area of research that deserves further exploration.

Deriving Parameters for PLAT

Total Phosphorus and Percent Dry Matter

One of the main goals of this research was to determine if P fractions could be correlated with treatment processes in order to update the PLAT software to include BS P content, represented in terms of treatment process categories. Due to the overall varied TP and percent DM within this study, and their relation to class and pathogen reduction processes, we suggest that more than one TP and percent DM value be used to represent different categories of BS.

As outlined in Table 47, we examined two categorical options for deriving the different TP and percent DM values to be used in PLAT. One option, which we call “Option 1,” would be to simply use “Class A” and “Class B” as categories to encompass all NC BS. What we call “Option 2” would use “Class A-alkaline,” “Class A-heat,” “Class B-slurry” and “Class B-cake” as categories. We decided to include an option separating Class A BS based on different PFRP and Class B BS based on consistency, as these factors were found to most

differentiate each class. Additionally, application rates between Class B slurries and Class B cakes will differ based on consistency of the BS.

In examining the two options, it was determined that Option 1 would overestimate TP for Class A alkaline stabilized BS, as this process dilutes TP in the BS considerably. It would also underestimate TP for Class A thermally heat dried BS, as this process tends to concentrate the TP within the BS, as opposed to dilution. This would overestimate percent DM for alkaline stabilized BS and underestimate percent DM for heat dried BS. Additionally, this would underestimate percent DM for BS cakes.

Evidence to support these claims can be seen when making comparisons between the two options. As can be seen in Table 47, the mean and median of BS TP for “Class A” were 16.7 and 17.8 g kg⁻¹, respectively. However, when Class A alkaline stabilized BS were viewed separately from Class A heat dried, the mean and median BS TP values for “Class A-alkaline” were 5.0 and 2.8 g kg⁻¹, respectively. Therefore, if Class A alkaline stabilized BS were to be represented strictly by “Class A,” median TP would be overestimated six-fold compared to when Class A alkaline was separated from Class A heat dried. The “Class A-heat” mean and median BS TP were 27.5 and 26.3 g kg⁻¹, respectively, which was about 10 g TP kg⁻¹ BS more than when represented solely by the “Class A” category, which would thus underestimate TP in these BS.

As stated, categorical Option 1 would overestimate percent DM for alkaline stabilized BS and underestimate percent DM for heat dried BS. Although the median percent DM value

for “Class A” BS was only 2.5 percentage points lower than the median for “Class A-heat,” the mean for “Class A” was 16.3 percentage points lower than the mean for “Class A-heat.” The mean percent DM for “Class A-alkaline” was 57.5%, which was 17.8 percentage points lower than the mean for “Class A.” Concerning median values, this difference was even more drastic, as “Class A-alkaline” percent DM was 33.2 percentage points less than that of “Class A.” Additionally, the mean percent DM for “Class B-cake” was 16.8%, which was 7.5 percentage points higher than the mean for “Class B.” The “Class B” median percent DM also underestimates “Class B-cake” by 12.2 percentage points.

Due to all of the aforementioned examination of TP and percent DM concerning the two categorical options, we recommend Option 2, containing the “Class A-alkaline,” “Class A-heat,” “Class B-slurry,” and “Class B-cake” categories. In making this recommendation, we acknowledge that the Part 503 Rule permits additional alternatives besides alkaline stabilization and thermal heat drying stabilization to meet Class A PFRP reduction requirements (USEPA, 1994a). We feel, however, that industry personnel involved in BS land application will have the knowledge required to choose the most appropriate category to represent their BS, if neither alkaline stabilization or heat drying are used to meet the Class A standards. Additionally, we acknowledge that Class B BS may have a consistency somewhere between “slurry” and “cake,” but in such cases, method of application should dictate which category to choose.

Water-Extractable and Percent Water-Extractable Phosphorus

Similarly to TP, we determined that Option 1 would overestimate WEP and PWEF for Class A alkaline stabilized BS and underestimate WEP and PWEF for Class A thermally heat dried BS (Table 47). Similarly to TP and percent DM, there were no mean or median “Class A-alkaline” WEP or PWEF values that were above their respective values when comparing to “Class A” from Option 1. Conversely, there were no mean or median “Class A-heat” WEP or PWEF values that were below their respective values in “Class A.”

Notably, the “Class A” median WEP was more than seven times as large as its “Class A-alkaline” counterpart, and the “Class A” mean WEP was more than 11 times the “Class A-alkaline” mean WEP. The “Class A” mean and median WEP were about a two to four-fold underestimate of Class A heat dried BS.

For Class B BS, “Class B” was not a drastic overestimation or underestimation for WEP or PWEF for “Class B-cake” or “Class B-slurry,” although all mean and median WEP and PWEF values for “Class B” would be a slight underestimation for “Class B-slurry.” Although “Class B” would slightly overestimate “Class B-cake” mean and median PWEF, as well as mean WEP, median WEP for both categories was the same.

Input Parameters for the Phosphorus Loss Assessment Tool

Phosphorus content is presently organized by “animal type” and “manure type” within the PLAT software. These headings would need to be adjusted to include the four

types of BS categories, or be recorded in a separate table. For each animal manure currently within the PLAT software database, total lbs P per unit of application (lbs ton⁻¹, lbs 1000 gal⁻¹, or lbs ac in⁻¹) “book values” exist. These include total P, soluble P (soluble P = total P x soluble fraction), and nonsoluble P (nonsoluble P = total P - soluble P), on a wet weight basis, and their respective soluble P fractions, which were based on previous research (NC PLAT Committee, 2005).

Table 48 was adapted from the NC PLAT Committee Table II.19 (NC PLAT Committee, 2005), and includes the four BS categories and their respective lbs P unit⁻¹ parameters. Mean values for P parameter g P kg⁻¹ BS and PWEF and DM percentages were used to represent these categories, although many were slightly conservative estimates when compared to their respective median values. We converted the mean g P kg⁻¹ BS dry weight mass concentrations into their respective lbs P unit⁻¹ on a wet weight basis, using the mean percent DM values for each category. For Class B-slurry we used lbs P 1,000 gal⁻¹, based on manures currently in PLAT with similar low percent solids, and for Class B-cake, Class A-alkaline, and Class A-heat, we used lbs P ton⁻¹.

As discussed in Chapter 3, our TP analytical method was not a USEPA-approved digestion method, and it may not have accounted for all BS TP. Thus, our TP mean may have been an underestimate. Subsequently, the PLAT “book values” we have determined for our four categories could also be underestimating TP. Therefore, implementing TP “book values” based on our study should be done with caution. That said, if TP was underestimated, our

PWEP would have been overestimated. Implementing them in PLAT would overestimate soluble P and thus be conservative in estimating P-loss risk.

Sensitivity of Phosphorus Loss Assessment Tool on Source Phosphorus

In order to have the scientific justification to guide BS applications using PLAT, P solubility needed to be determined for NC BS. This information can then be added to the PLAT database to improve the prediction of P-loss risk when land applying BS. In determining the potential affects this may have on BS land applications, the influence of BS particulate and soluble source P on the PLAT overall index rating needs to be assessed.

As discussed in Chapter 1, PLAT accounts for four P-loss pathways in determining potential P-loss for a particular site. These pathways include sediment P (losses via erosion of P attached to soil), soluble soil P via surface runoff, soluble P via subsurface drainage or leaching, and P losses from particulate and soluble P from the source (Johnson et al., 2005). The pathway that this research aimed to address was source P loss, i.e., losses from the BS themselves. In addition to the P source characteristics, many other model inputs influence estimation of P loss via this pathway. These include the application rate and method, and site conservation practices. Therefore, the P fractions of a source contributes only a portion to the source P loss, which in turn, is only one of four pathways influencing the final PLAT value for predicting P-loss risk for a site.

Currently in PLAT, animal waste is applied on an N-rate basis if the site PLAT index value is low (0 to 25) or medium (26 to 50). High PLAT index values (51 to 100) limit the application rate to the P that is removed by the harvested crop. Very high index values (>101) limit any additional P applications, except as a starter fertilizer (Shaffer et al., 2008). Therefore, for low or medium PLAT index ratings, the potential P loss risk from source cannot be characterized without first knowing the N rate. For example, two BS could be applied that have the same amount of P, but different amounts of N. If applying on an N basis, more P would therefore be applied in the BS with less N. Conversely, if the PLAT value is high, then the land applicator must apply BS based on crop P removal, which would limit overall P applications. Estimating the potential P loss risk when using PLAT is therefore a function of many site-specific factors that influence the four pathways and overall PLAT rating, which in turn influence how much BS can actually be applied.

Since runoff P losses are controlled by P solubility, and we determined that soluble P in NC BS is relatively low, we predict that the influence of soluble source P losses on the overall PLAT rating will be low. In the PLAT software, the soluble P fraction cannot be altered by the user. Therefore, depending on which BS category is selected, the soluble P fraction will never change. We suggest that due to the overall low WEP and PWEP of BS within this study, the complexity of treatment processes that produced these P fractions, and the relative insensitivity of PLAT to PWEP, single WEP and PWEP values might be

adequate for all four of our BS categories. To confirm this, however, a PLAT sensitivity analysis would need to be conducted using these categories.

Soil test P relates to the amount of dissolved and particulate P in runoff, and buildup of P in the soil can cause increased P-loss risk (Johnson et al., 2005). Soil test P therefore has a much heavier relative “weight” in PLAT, as it is used in calculating P-loss risk within the sediment P, soluble surface runoff, and soluble P leaching pathways. Theoretically, if STP is low for a site, the land applicator could apply large amounts of BS with very high P, and still obtain an overall low PLAT index rating. Conversely, if the STP is high for a site, this may mean the overall PLAT index rating could be high or very high, which would limit the BS application rates based on P.

Additionally, if BS are applied to a site repeatedly over multiple years, STP will rise, which can increase the overall PLAT rating, curtailing BS applications allowed for the site. As discussed in Chapter 1 in the analysis delineating agriculture in the Neuse watershed, BS were second only to poultry litter as the most-applied organic fertilizer source (Osmond, 2007). Of the 74 fields in which organic soil amendments had been applied, 14 had received BS and 17 had very high (>100) STP. Out of the 17 fields with very high STP, eight had received BS. This indicates that 57% of the BS-receiving fields had very high STP. The P-loss risk from these BS-receiving fields was therefore much higher than for fields with low STP.

Although the soluble P fraction cannot be altered by the user, the lbs total P unit⁻¹ soil amendment can be manually changed in the PLAT software. Once the user enters their own total P, the soluble and nonsoluble P portions are automatically calculated using the fixed soluble P fraction for the respective soil amendment. Although the four-category Option 2 may adequately represent NC BS and serve as a predictor for BS total P, if actual total P values are known, the land applicators should enter these values into the software instead of using the total P “book values” derived from our research. Since the residuals land application permit requires total P testing, this information should be available (E. Hardee/NC DENR, personal communication, 2014). This would likely improve the accuracy of PLAT in predicting P-loss risk. Land application of BS based on both P and N may decrease overall application rates, reduce the number of fields eligible to receive BS, and shorten the time soils can receive biosolids, thus potentially making land application more costly. Hence, the widespread adoption of a P-based approach may foster development of alternative beneficial uses of BS other than land application.

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TABLES

Table 1. Biosolids identification number (BS ID), biosolids class (BS Class), and participating facilities (WWTP, wastewater treatment plant; WRF, water reuse facility).

BS ID	BS Class	Facility Name
1	B	Franklin County WWTP
2	A	Neuse River WWTP
3	B	Neuse River WWTP
4	B	Little Creek WRF
5	B	Big Buffalo WWTP
6	B	Leith Creek WWTP
7	B	Lumberton WWTP
8	B	Cross Creek WWTP
9	B	Wilson WWTP
10	B	Kinston Regional WRF
11	B	Havelock WWTP
12	A	Archie Elledge WWTP
13	A	Fourth Creek WWTP
14	B	Fourth Creek WWTP
15	A	Lower Creek WWTP
16	A	Jimmy Smith WWTP
17	B	Corpening Creek WWTP
19	A	Waynesville WWTP
20	A	Whittier Sanitary District WWTP
21	B	Spindale WWTP
22	B	Irwin Creek WWTP
23	B	McAlpine Creek WWTP
24	B	McDowell Creek WWTP
26	B	North Durham WRF
27	B	Elizabeth City WWTP
28	B	Southside WWTP
29	B	James A. Loughlin (Northside) WWTP
30	A	South Cary WRF

Table 2. Summary statistics for sampling and analytical error of total P expressed as the CV of the means of the three analytical replicates. Summer 2013, Fall 2013, Winter 2014, and seasons combined.

Three-replicate CV for biosolids total P									
Season	N	Mean	Median	Minimum	Maximum	SD	CV	Skew	Kurtosis
		-----%-----							
Summer	27	12.9	8.3	0.4	41.8	11.2	86.8	1.1	0.5
Fall	27	7.8	1.9	0.6	71.2	17.9	230.1	3.1	9.1
Winter	26	5.2	2.2	0.3	39.2	8.1	155.0	3.3	12.7
Seasons Combined	80	8.7	3.3	0.3	71.2	13.3	153.6	2.8	9.0

Table 3. Effect of season on sampling and analytical error of total P expressed as the CV of the means of the three analytical replicates (F, Fall 2013; S, Summer 2013; W, Winter 2014).

<u>Summary of Fit</u>				
R ²			0.06	
RMSE			13.1	
CV (%)			150.6	
Observations			80	
<u>Analysis of Variance</u>				
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	2	818.1	409.0	2.4
Error	77	13232.3	171.8	Prob > F
Total	79	14050.4		0.1
<u>Least Squares Means</u>				
Season		CV		Std Error
		-----%-----		
F		7.8ab†		2.5
S		12.9a		2.5
W		5.2b		2.6
Mean		8.7		

†Means followed by the same letter are not significantly different (Student's t, $\alpha = 0.05$).

Table 4. Summary statistics for sampling and analytical error of water-extractable P expressed as the CV of the means of the four analytical replicates. Summer 2013, Fall 2013, Winter 2014, and seasons combined.

Season	N	Four-replicate CV for biosolids water-extractable P							
		Mean	Median	Minimum	Maximum	SD	CV	Skew	Kurtosis
		-----%-----							
Summer	28	6.1	5.3	0.5	20.2	4.9	79.6	1.2	1.3
Fall	27	5.9	5.4	0.9	17.5	3.9	65.1	1.4	2.4
Winter	26	8.0	4.9	1.1	26.8	7.0	87.7	1.3	1.1
Seasons Combined	81	6.7	5.4	0.5	26.8	5.4	81.0	1.5	2.5

Table 5. Effect of season on sampling and analytical error of water-extractable P expressed as the CV of the means of the four analytical replicates (F, Fall 2013; S, Summer 2013; W, Winter 2014).

<u>Summary of Fit</u>				
R ²			0.03	
RMSE			5.4	
CV (%)			80.6	
Observations			81	
<u>Analysis of Variance</u>				
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	2	69.8	34.9	1.2
Error	78	2261.8	29.0	Prob > F
Total	80	2331.6		0.3
<u>Least Squares Means</u>				
Season	CV	Std Error		
		-----%-----		
F	5.9a†	1.0		
S	6.1a	1.0		
W	8.0a	1.1		
Mean	6.7			

†Means followed by the same letter are not significantly different (Student's t, $\alpha = 0.05$).

Table 6. Summary statistics for sampling and analytical error of percent water-extractable P expressed as the CV of the means of the three analytical replicates. Summer 2013, Fall 2013, Winter 2014, and seasons combined.

Three-replicate CV for biosolids percent water-extractable P									
Season	N	Mean	Median	Minimum	Maximum	SD	CV	Skew	Kurtosis
		-----%-----							
Summer	27	14.6	12.4	2.2	33.1	9.5	65.2	0.5	-1.1
Fall	27	11.3	6.5	1.2	56.4	13.8	122.1	2.1	4.3
Winter	26	9.9	7.2	0.5	42.9	9.6	96.8	1.8	4.5
Seasons Combined	80	12.0	8.2	0.5	56.4	11.2	93.6	1.6	3.0

Table 7. Effect of season on sampling and analytical error of percent water-extractable P expressed as the CV of the means of the three analytical replicates (F, Fall 2013; S, Summer 2013; W, Winter 2014).

<u>Summary of Fit</u>				
R ²				0.03
RMSE				11.2
CV (%)				93.3
Observations				80
<u>Analysis of Variance</u>				
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	2	305.5	152.8	1.2
Error	77	9585.8	124.5	Prob > F
Total	79	9891.4		0.3
<u>Least Squares Means</u>				
Season	CV	Std Error		
		-----%-----		
F	11.3a†	2.1		
S	14.6a	2.1		
W	9.9a	2.2		
Mean	12.0			

†Means followed by the same letter are not significantly different (Student's t, $\alpha = 0.05$).

Table 8. Summary statistics for sampling and analytical error of percent dry matter expressed as the CV of the means of the three analytical replicates. Summer 2013, Fall 2013, Winter 2014, and seasons combined.

Three-replicate CV for biosolids percent dry matter									
Season	N	Mean	Median	Minimum	Maximum	SD	CV	Skew	Kurtosis
-----%-----									
Summer	28	1.4	0.6	0.02	13.8	2.6	189.4	4.4	21.4
Fall	27	1.8	0.7	0.02	14.8	3.1	174.7	3.3	12.1
Winter	26	1.6	0.8	0.02	6.7	2.0	122.4	1.5	1.1
Seasons Combined	81	1.6	0.7	0.02	14.8	2.6	162.9	3.5	13.9

Table 9. Effect of season on sampling and analytical error of percent dry matter expressed as the CV of the means of the three analytical replicates (F, Fall 2013; S, Summer 2013; W, Winter 2014).

<u>Summary of Fit</u>				
R ²			0.004	
RMSE			2.6	
CV (%)			162.5	
Observations			81	
<u>Analysis of Variance</u>				
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	2	2.4	1.2	0.2
Error	78	528.8	6.8	Prob > F
Total	80	531.1		0.8
<u>Least Squares Means</u>				
Season	CV	Std Error		
		-----%-----		
F	1.8a†	0.5		
S	1.4a	0.5		
W	1.6a	0.5		
Mean	1.6			

†Means followed by the same letter are not significantly different (Student's t, $\alpha = 0.05$).

Table 10. Biosolids total P summary statistics for Summer 2013, Fall 2013, Winter 2014, and seasons combined.

Season	N	Biosolids total P								
		Mean	Median	Minimum	Maximum	SD	CV	Skew	Kurtosis	
		-----g kg ⁻¹ -----					%			
Summer	28	17.8	18.8	0.4	31.0	8.2	46.2	-0.5	-0.3	
Fall	27	21.8	22.7	1.0	45.3	10.5	48.4	-0.1	0.0	
Winter	26	31.3	31.4	1.3	73.2	14.6	46.8	0.7	1.9	
Seasons Combined	81	23.4	23.6	0.4	73.2	12.6	53.6	0.8	2.4	

Table 11. Effect of biosolids (BS) on total P (TP).

<u>Summary of Fit</u>				
R ²			0.73	
RMSE			8.1	
CV (%)			34.6	
Observations			81	
<u>Analysis of Variance</u>				
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	27	9164.9	339.4	5.2
Error	53	3468.4	65.4	Prob > F
Total	80	12633.2		<0.0001
<u>Least Squares Means</u>				
BS	TP	Std Error		
		-----g kg ⁻¹ -----		
13	0.9j†	4.7		
2	1.7ij	5.7		
19	6.2hij	4.7		
15	10.0ghij	4.7		
21	14.7fghi	4.7		
8	14.9fghi	4.7		
22	16.5efghi	4.7		
5	18.5defgh	5.7		
7	19.1defgh	4.7		
6	19.7defg	4.7		
27	20.2defg	4.7		
12	21.3cdefg	4.7		
9	22.0cdefg	4.7		
29	22.6cdefg	4.7		
28	23.9cdef	4.7		
14	24.2cdef	4.7		
10	27.1bcdef	4.7		
23	27.1bcdef	4.7		
16	27.4bcdef	5.7		
17	28.3bcde	4.7		
1	29.2bcde	4.7		
20	30.2bcd	4.7		
30	30.9bcd	4.7		
4	31.4bcd	4.7		
24	34.3bc	4.7		
11	37.9ab	4.7		
26	39.9ab	4.7		
3	48.6a	4.7		
Mean	23.4			

†Means followed by the same letter are not significantly different (Student's t, $\alpha = 0.05$).

Table 12. Effect of season on total P (TP; F, Fall 2013; S, Summer 2013; W, Winter 2014).

<u>Summary of Fit</u>	
R ²	0.20
RMSE	11.4
CV (%)	48.7
Observations	81

<u>Analysis of Variance</u>				
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	2	2580.7	1290.4	10.0
Error	78	10052.5	128.9	Prob > F
Total	80	12633.2		0.0001*

<u>Least Squares Means</u>		
Season	TP	Std Error
	-----g kg ⁻¹ -----	
F	21.8b†	2.2
S	17.8b	2.1
W	31.3a	2.2
Mean	23.4	

†Means followed by the same letter are not significantly different (Student's t, $\alpha = 0.05$).

Table 13. Biosolids water-extractable P summary statistics for Summer 2013, Fall 2013, Winter 2014, and seasons combined.

Biosolids water-extractable P									
Season	N	Mean	Median	Minimum	Maximum	SD	CV	Skew	Kurtosis
		-----g kg ⁻¹ -----					%		
Summer	28	1.4	0.6	0.02	9.7	2.2	162.8	2.7	7.7
Fall	27	1.0	0.3	0.02	7.1	1.7	164.7	2.5	6.3
Winter	26	1.7	1.1	0.02	9.1	2.3	132.2	2.1	4.0
Seasons Combined	81	1.4	0.6	0.02	9.1	2.1	151.6	2.4	5.6

Table 14. Effect of biosolids (BS) on water-extractable P (WEP).

<u>Summary of Fit</u>				
R ²				0.82
RMSE				1.1
CV (%)				78.6
Observations				81
<u>Analysis of Variance</u>				
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	27	284.6	10.5	8.7
Error	53	64.3	1.2	Prob > F
Total	80	349.0		<.0001
<u>Least Squares Means</u>				
BS	WEP	Std Error		
		-----g kg ⁻¹ -----		
13	0.02e†	0.6		
19	0.03e	0.6		
8	0.05e	0.6		
2	0.06de	0.8		
9	0.09de	0.6		
7	0.1de	0.6		
15	0.2de	0.6		
21	0.2de	0.6		
23	0.2de	0.8		
29	0.2de	0.6		
12	0.3de	0.6		
1	0.3de	0.6		
28	0.4de	0.6		
22	0.6cde	0.6		
5	0.8cde	0.8		
10	0.8cde	0.6		
16	1.0cde	0.6		
20	1.0cde	0.6		
6	1.1cde	0.6		
11	1.1cde	0.6		
27	1.3cde	0.6		
26	1.6bcde	0.6		
14	1.9bcd	0.6		
24	2.3bc	0.6		
30	3.3b	0.6		
3	5.8a	0.6		
4	5.9a	0.6		
17	6.9a	0.6		
Mean	1.4			

†Means followed by the same letter are not significantly different (Student's t, $\alpha = 0.05$).

Table 15. Effect of season on water-extractable P (WEP; F, Fall 2013; S, Summer 2013; W, Winter 2014).

<u>Summary of Fit</u>				
R ²				0.02
RMSE				2.1
CV (%)				150.0
Observations				81
<u>Analysis of Variance</u>				
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	2	6.5	3.3	0.7
Error	78	342.4	4.4	Prob > F
Total	80	349.0		0.5
<u>Least Squares Means</u>				
Season	WEP		Std Error	
	-----g kg ⁻¹ -----			
F	1.0a†		0.4	
S	1.4a		0.4	
W	1.7a		0.4	
Mean	1.4			

†Means followed by the same letter are not significantly different (Student's t, $\alpha = 0.05$).

Table 16. Biosolids percent water-extractable P summary statistics for Summer 2013, Fall 2013, Winter 2014, and seasons combined.

Season	N	Biosolids percent water-extractable P								
		Mean	Median	Minimum	Maximum	SD	CV	Skew	Kurtosis	
		-----g kg ⁻¹ -----					%			
Summer	28	6.2	3.8	0.5	35.2	8.2	131.4	2.5	6.2	
Fall	27	3.9	2.1	0.2	26.0	5.6	143.7	2.8	9.2	
Winter	26	4.8	3.2	0.2	24.9	5.7	118.9	2.4	6.1	
Seasons Combined	81	5.0	2.9	0.2	35.2	6.6	132.8	2.6	7.5	

Table 17. Effect of biosolids (BS) on percent water-extractable P (PWEP).

<u>Summary of Fit</u>				
R ²				0.76
RMSE				4.0
CV (%)				80.0
Observations				81
<u>Analysis of Variance</u>				
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	27	2662.9	98.6	6.2
Error	53	840.8	15.9	Prob > F
Total	80	3503.6		<.0001
<u>Least Squares Means</u>				
BS	PWEP		Std Error	
		-----%-----		
8	0.4g†		2.3	
9	0.4g		2.3	
7	0.6g		2.3	
19	0.6g		2.3	
23	0.6efg		2.8	
29	1.0fg		2.3	
1	1.2efg		2.3	
21	1.3efg		2.3	
12	1.4efg		2.3	
15	1.5efg		2.3	
28	1.8efg		2.3	
13	2.5efg		2.3	
10	2.8efg		2.3	
11	3.1efg		2.3	
2	3.4defg		2.8	
20	3.4efg		2.3	
22	3.6efg		2.3	
16	3.8efg		2.3	
26	4.1defg		2.3	
5	4.2defg		2.8	
6	6.2defg		2.3	
27	6.5defg		2.3	
24	7.1def		2.3	
14	7.7de		2.3	
30	10.4cd		2.3	
3	14.8bc		2.3	
4	18.9ab		2.3	
17	23.9a		2.3	
Mean	5.0			

†Means followed by the same letter are not significantly different (Student's t, $\alpha = 0.05$).

Table 18. Effect of season on percent water-extractable P (PWEP; F, Fall 2013; S, Summer 2013; W, Winter 2014).

<u>Summary of Fit</u>	
R ²	0.02
RMSE	6.6
CV (%)	132.0
Observations	81

<u>Analysis of Variance</u>				
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	2	76.8	38.4	0.9
Error	78	3426.8	44.0	Prob > F
Total	80	3503.6		0.4

<u>Least Squares Means</u>		
Season	PWEP	Std Error
	-----%-----	
F	3.9a†	1.3
S	6.2a	1.3
W	4.8a	1.3
Mean	5.0	

†Means followed by the same letter are not significantly different (Student's t, $\alpha = 0.05$).

Table 19. Biosolids percent dry matter summary statistics for Summer 2013, Fall 2013, Winter 2014, and seasons combined.

Biosolids percent dry matter										
Season	N	Mean	Median	Minimum	Maximum	SD	CV	Skew	Kurtosis	
		-----g kg ⁻¹ -----						%		
Summer	28	28.4	16.2	1.6	95.9	33.3	117.2	1.2	0.0	
Fall	27	28.8	15.2	1.4	95.0	32.9	114.3	1.1	-0.3	
Winter	26	26.9	15.1	1.7	94.7	32.6	121.0	1.3	0.1	
Seasons Combined	81	28.0	15.2	1.4	95.9	32.5	116.0	1.2	-0.2	

Table 20. Effect of biosolids (BS) on percent dry matter (DM, %).

<u>Summary of Fit</u>				
R ²				1.0
RMSE				3.0
CV (%)				10.7
Observations				81
<u>Analysis of Variance</u>				
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	27	84103.4	3114.9	353.3
Error	53	467.2	8.8	Prob > F
Total	80	84570.6		<.0001
<u>Least Squares Means</u>				
BS	DM	Std Error		
		-----%-----		
6	1.7j†	1.7		
17	2.3j	1.7		
1	2.4j	1.7		
4	2.6j	1.7		
21	3.2j	1.7		
3	3.3j	1.7		
7	3.3j	1.7		
8	3.5j	1.7		
27	3.6j	1.7		
11	4.0j	1.7		
5	5.8j	2.1		
24	13.2i	1.7		
28	15.2hi	1.7		
14	15.9ghi	1.7		
26	16.1ghi	1.7		
10	16.5ghi	1.7		
29	17.6ghi	1.7		
22	18.1ghi	1.7		
23	18.9gh	2.1		
9	20.6g	1.7		
15	38.0f	1.7		
19	52.5e	1.7		
2	59.7d	2.1		
13	80.6c	1.7		
16	89.1b	1.7		
12	89.3b	1.7		
30	92.7ab	1.7		
20	95.2a	1.7		
Mean	28.0			

†Means followed by the same letter are not significantly different (Student's t, $\alpha = 0.05$).

Table 21. Effect of season on percent dry matter (DM, %; F, Fall 2013; S, Summer 2013; W, Winter 2014).

<u>Summary of Fit</u>				
R ²				0.0006
RMSE				33.0
CV (%)				117.9
Observations				81
<u>Analysis of Variance</u>				
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	2	52.1	26.1	0.02
Error	78	84518.5	1083.6	Prob > F
Total	80	84570.6		1.0
<u>Least Squares Means</u>				
Season	DM	Std Error		
		-----%-----		
F	28.8a†	6.3		
S	28.4a	6.2		
W	26.9a	6.5		
Mean	28.0			

†Means followed by the same letter are not significantly different (Student's t, $\alpha = 0.05$).

Table 22. Online survey questions with responses; Column 1, processes and chemical additions; Column 2, number (n) of positive responses (out of 28); Column 3, positive responses as percentage (%) of total; Columns 4 and 5, number (n) of positive responses in Cluster 1 and Cluster, respectively.

Process/Chemical Addition	Positive Responses			
	Full Survey		Cluster 1	Cluster 2
	n	%	n	n
Produce biosolids	28	100.0	N/A	N/A
Land apply biosolids	28	100.0	N/A	N/A
<u>Prior to preliminary treatment</u>				
calcium/sodium nitrate	5	17.9	2	3
chlorine	2	7.1	2	0
ferric sulfate	2	7.1	0	2
ferrous sulfate	2	7.1	0	2
hydrogen peroxide	1	3.6	0	1
lime	1	3.6	1	0
magnesium hydroxide	2	7.1	0	2
sodium hydroxide	1	3.6	0	1
liquid oxygen*	1	3.6	1	0
bioxide*	1	3.6	0	1
sulfur dioxide*	1	3.6	1	0
no chemicals added	14	50.0	7	7
<u>Measure total phosphorus</u>				
Influent - yes	19	67.9	4	15
Influent - no	8	28.6	8	0
Effluent - yes	28	100.0	N/A	N/A
<u>Preliminary treatment</u>				
grinder(s)	3	10.7	0	3
grit chamber(s)	26	92.9	11	15
drip degreaser(s)*	2	7.1	2	0
<u>Primary Treatment</u>				
chemical scrubbing	2	7.1	0	2
sedimentation tank(s)	11	39.3	3	8
biological odor control*	1	3.6	0	1
conventional primary clarifier*	2	7.1	0	2
no primary treatment*	1	3.6	1	0
lime	2	7.1	1	1
sodium hydroxide	1	3.6	1	0

Table 22. Continued

Process/Chemical Addition	Positive Responses			
	Full Survey		Cluster 1	Cluster 2
	n	%	n	n
<u>Primary Treatment, continued</u>				
sodium aluminate*	1	3.6	0	1
no chemicals added	21	75.0	8	13
<u>Secondary treatment</u>				
activated sludge	18	64.3	10	8
activated sludge with BNR	12	42.9	4	8
trickling filters	1	3.6	0	1
denitrifying filter*	1	3.6	0	1
secondary clarifiers*	2	7.1	2	0
BNR - nitrification but no denitrification	1	3.6	0	1
BNR - nitrification and denitrification	4	14.3	1	3
BNR - phosphorus and nitrogen removal	7	25.0	3	4
acetic acid	1	3.6	0	1
activated carbon	1	3.6	0	1
aluminum sulfate, (alum)	4	14.3	1	3
chlorine	1	3.6	1	0
lime	4	14.3	2	2
magnesium hydroxide	4	14.3	1	3
methanol	4	14.3	0	4
polymers	3	10.7	1	2
sugar water	1	3.6	0	1
sodium hypochlorite*	1	3.6	1	0
polyaluminum chloride*	1	3.6	0	1
sulfur dioxide	1	3.6	1	0
no chemicals added	9	32.1	5	4
<u>Final Clarifier</u>				
no final clarifier	1	3.6	1	0
ferric chloride	1	3.6	0	1
sodium aluminate*	1	3.6	0	1
polyaluminum chloride*	1	3.6	0	1
no chemicals added	22	78.6	11	11
<u>Tertiary filter</u>				
has tertiary filter	18	64.3	4	14
no tertiary filter	10	35.7	9	1

Table 22. Continued

Process/Chemical Addition	Positive Responses			
	Full Survey		Cluster 1	Cluster 2
	n	%	n	n
methanol	2	7.1	0	2
sugar water	2	7.1	0	2
no chemicals added	16	57.1	4	12
<u>Filter Backwash Discharge</u>				
discharges into influent or primary treatment	17	60.7	3	14
filter not in operation*	1	3.6	1	0
no chemicals added	18	64.3	4	14
<u>Sludge Thickening</u>				
centrifugation	2	7.1	1	1
gravity	10	35.7	8	2
gravity belt thickener(s)	17	60.7	4	13
decanting water from aerobic digester*	3	10.7	2	1
ferric chloride	1	3.6	1	0
polymers	19	67.9	4	15
no chemicals added	9	32.1	9	0
liquid goes back to influent or primary treatment	26	92.9	11	15
liquid goes to sludge stabilization*	2	7.1	2	0
polymers added to liquid returned	1	3.6	1	0
sodium aluminate* added to liquid returned	1	3.6	0	1
no chemicals added to liquid returned	25	89.3	11	14
<u>Sludge Stabilization</u>				
aerobic digestion	14	50.0	7	7
air drying	1	3.6	1	0
alkaline (lime)	6	21.4	4	2
anaerobic digestion	11	39.3	3	8
heat drying	4	14.3	2	2
pasteurization	3	10.7	2	1
wet air oxidation	1	3.6	1	0
polymers	5	17.9	2	3
sodium bicarbonate*	1	3.6	0	1
no chemicals added	20	71.4	10	10
liquid goes back to influent or primary treatment	18	64.3	12	6
no liquid produced*	5	17.9	0	5
liquid goes to dewatering*	1	3.6	0	1

Table 22. Continued

Process/Chemical Addition	Positive Responses			
	Full Survey		Cluster 1	Cluster 2
	n	%	n	n
liquid leaves facility*	3	10.7	1	2
sodium aluminate* added to liquid returned	1	3.6	0	1
no liquid returned*	3	10.7	0	3
no chemicals added to liquid returned	23	82.1	13	10
<u>Sludge Dewatering</u>				
belt press(es)	14	50.0	5	9
centrifugation	4	14.3	1	3
drying bed(s)	2	7.1	1	1
sludge lagoon(s)	1	3.6	1	0
gravity belt thickening*	3	10.7	2	1
no dewatering processes*	2	7.1	1	1
ferric chloride	2	7.1	1	1
polymers	17	60.7	6	11
no chemicals added	8	28.6	7	1
liquid goes back to primary treatment	23	82.1	12	11
liquid land application*	1	3.6	1	0
no liquid produced*	2	7.1	0	2
ferric chloride added to liquid returned	1	3.6	0	1
ferrous sulfate added to liquid returned	1	3.6	0	1
Polymers added to liquid returned	3	10.7	1	2
sodium aluminate* added to liquid returned	1	3.6	0	1
no chemicals added to liquid returned	21	75.0	12	9
<u>Class and Consistency</u>				
Class A	8	28.6	5	4
Class B	20	71.4	8	12
Class A cake	2	7.1	1	1
Class A pellet	3	10.7	2	1
Class A very dry cake*	2	7.1	1	1
Class A dry cake*	3	10.7	2	1
Class B cake	9	32.1	1	8
Class B slurry	11	39.3	7	4

Table 23. Online survey questions without responses, subsequently removed from analysis. Processes and chemicals grouped by treatment zone. Column 1: Prior to Preliminary Treatment, Primary Treatment, Secondary Treatment, Final Clarifier, Class and Consistency, and Phosphorus Analysis in Effluent. Column 2: Tertiary Filter, Filter Backwash, and Sludge Thickening. Column 3: Sludge Stabilization and Sludge Dewatering.

<u>Prior to Preliminary Treatment</u>	<u>Tertiary Filter</u>	<u>Sludge Stabilization</u>
acetic acid	acetic acid	beta ray irradiation
activated carbon	activated carbon	blending
aluminum sulfate (alum)	aluminum sulfate (alum)	gamma ray irradiation
chlorine dioxide	calcium/sodium nitrate	thermophilic aerobic digestion
ferric chloride	chlorine	acetic acid
glycerine	chlorine dioxide	activated carbon
methanol	ferric chloride	aluminum sulfate (alum)
ozone	ferric sulfate	calcium/sodium nitrate
phosphates	ferrous sulfate	chlorine
polymers	glycerine	chlorine dioxide
rosin amine salts	hydrogen peroxide	ferric chloride
sugar water	lime	ferric sulfate
sulfuric acid	magnesium hydroxide	ferrous sulfate
	ozone	glycerine
<u>Primary treatment</u>	phosphates	hydrogen peroxide
Activated primary clarifier	polymers	magnesium hydroxide
chemical feeder(s)	rosin amine salts	methanol
flash mixing unit(s)	sodium hydroxide	ozone
flocculation tank(s)	sulfuric acid	phosphates
acetic acid	Other, please specify: - TEXT	rosin amine salts
activated carbon		sodium hydroxide
aluminum sulfate, (alum)	<u>Filter Backwash</u>	sugar water
calcium/sodium nitrate	acetic acid	sulfuric acid
chlorine	activated carbon	liquid returned - acetic acid
chlorine dioxide	aluminum sulfate (alum)	liquid returned - activated carbon
ferric chloride	calcium/sodium nitrate	liquid returned - aluminum sulfate (alum)
ferric sulfate	chlorine	liquid returned - calcium/sodium nitrate
ferrous sulfate	chlorine dioxide	liquid returned - chlorine
glycerine	ferric chloride	liquid returned - chlorine dioxide

Table 23. Continued

<u>Primary treatment (continued)</u>	<u>Filter Backwash (continued)</u>	<u>Sludge Stabilization (continued)</u>
hydrogen peroxide	ferric sulfate	liquid returned - ferric chloride
magnesium hydroxide	ferrous sulfate	liquid returned - ferric sulfate
methanol	glycerine	liquid returned - ferrous sulfate
ozone	hydrogen peroxide	liquid returned - glycerine
phosphates	lime	liquid returned - hydrogen peroxide
polymers	magnesium hydroxide	liquid returned - lime
rosin amine salts	methanol	liquid returned - magnesium hydroxide
sugar water	ozone	liquid returned - methanol
sulfuric acid	phosphates	liquid returned - ozone
	polymers	liquid returned - phosphates
<u>Secondary Treatment</u>	rosin amine salts	liquid returned - polymers
intermittent sand filter(s)	sodium hydroxide	liquid returned - rosin amine salts
lagoon(s)	sugar water	liquid returned - sodium hydroxide
rotating biological contactors (RBC)	sulfuric acid	liquid returned - sugar water
stabilization pond(s)	Other, please specify: - TEXT	liquid returned - sulfuric acid
Other, (not specified):		
BNR - phosphorus removal only*	<u>Sludge Thickening</u>	<u>Sludge Dewatering</u>
BNR - TEXT	dissolved air floatation (DAF)	pressure filtration
calcium/sodium nitrate	acetic acid	rotary press(es)
chlorine dioxide	activated carbon	vacuum filtration
ferric chloride	aluminum sulfate (alum)	acetic acid
ferric sulfate	calcium/sodium nitrate	activated carbon
ferrous sulfate	chlorine	aluminum sulfate (alum)
glycerine	chlorine dioxide	calcium/sodium nitrate
hydrogen peroxide	ferric sulfate	chlorine
ozone	ferrous sulfate	chlorine dioxide
phosphates	glycerine	ferric sulfate
rosin amine salts	hydrogen peroxide	ferrous sulfate
sulfuric acid	lime	glycerine
	magnesium hydroxide	hydrogen peroxide
<u>Final Clarifier</u>	methanol	lime
acetic acid	ozone	magnesium hydroxide
activated carbon	phosphates	methanol
aluminum sulfate (alum)	rosin amine salts	ozone

Table 23. Continued

<u>Final Clarifier (continued)</u>	<u>Sludge Thickening (continued)</u>	<u>Sludge Dewatering (continued)</u>
calcium/sodium nitrate	sodium hydroxide	phosphates
chlorine	sugar water	rosin amine salts
chlorine dioxide	sulfuric acid	sodium hydroxide
ferric chloride	Other, please specify: TEXT	sugar water
ferric sulfate	liquid returned - acetic acid	sulfuric acid
ferrous sulfate	liquid returned - activated carbon	Other, please specify:- TEXT
glycerine	liquid returned - aluminum sulfate (alum)	liquid returned - acetic acid
hydrogen peroxide	liquid returned - calcium/sodium nitrate	liquid returned - activated carbon
lime	liquid returned - chlorine	liquid returned - aluminum sulfate (alum)
magnesium hydroxide	liquid returned - chlorine dioxide	liquid returned - calcium/sodium nitrate
methanol	liquid returned - ferric chloride	liquid returned - chlorine
ozone	liquid returned - ferric sulfate	liquid returned - chlorine dioxide
phosphates	liquid returned - ferrous sulfate	liquid returned - ferric sulfate
polymers	liquid returned - glycerine	liquid returned - glycerine
rosin amine salts	liquid returned - hydrogen peroxide	liquid returned - hydrogen peroxide
sodium hydroxide	liquid returned - lime	liquid returned - lime
sugar water	liquid returned - magnesium hydroxide	liquid returned - magnesium hydroxide
sulfuric acid	liquid returned - methanol	liquid returned - methanol
	liquid returned - ozone	liquid returned - ozone
<u>Class and Consistency</u>	liquid returned - phosphates	liquid returned - phosphates
Class A liquid	liquid returned - rosin amine salts	liquid returned - rosin amine salts
Class A slurry	liquid returned - sodium hydroxide	liquid returned - sodium hydroxide
Class B pellet	liquid returned - sugar water	liquid returned - sugar water
<u>Phosphorus Analysis in Effluent</u>	liquid returned - sulfuric acid	liquid returned - sulfuric acid
none analyzed		

Table 24. Effect of two clusters on total P (TP; F, Fall 2013; S, Summer 2013; W, Winter 2014).

<u>Summary of Fit</u>	
R ²	0.09
RMSE	12.1
CV (%)	51.7
Observations	81

<u>Analysis of Variance</u>				
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	1088.4	1088.4	7.4
Error	79	11544.8	146.1	Prob > F
Total	80	12633.2		0.008

<u>Least Squares Means</u>		
Cluster	TP	Std Error
	-----g kg ⁻¹ -----	
1	19.6b†	1.9
2	27.0a	1.9
Mean	23.4	

†Means followed by the same letter are not significantly different (Student's t, $\alpha = 0.05$).

Table 25. Effect of two clusters on water-extractable P (WEP; F, Fall 2013; S, Summer 2013; W, Winter 2014).

Summary of Fit

R ²	0.002
RMSE	2.1
CV (%)	150.0
Observations	81

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	0.8	0.8	0.2
Error	79	348.1	4.4	Prob > F
Total	80	349.0		0.7

Least Squares Means

Cluster	WEP	Std Error
	-----g kg ⁻¹ -----	
1	1.5a†	0.3
2	1.3a	0.3
Mean	1.4	

†Means followed by the same letter are not significantly different (Student's t, $\alpha = 0.05$).

Table 26. Effect of two clusters on percent water-extractable P (PWEP; F, Fall 2013; S, Summer 2013; W, Winter 2014).

<u>Summary of Fit</u>	
R ²	0.02
RMSE	6.6
CV (%)	132.0
Observations	81

<u>Analysis of Variance</u>				
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	54.3	54.3	1.2
Error	79	3449.3	43.7	Prob > F
Total	80	3503.6		0.3

<u>Least Squares Means</u>		
Cluster	PWEP	Std Error
	-----%-----	
1	5.8a†	1.1
2	4.2a	1.0
Mean	5.0	

†Means followed by the same letter are not significantly different (Student's t, $\alpha = 0.05$).

Table 27. Effect of two clusters on percent dry matter (DM%; F, Fall 2013; S, Summer 2013; W, Winter 2014).

Summary of Fit

R ²	0.004
RMSE	32.7
CV (%)	116.8
Observations	81

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	302.6	302.6	0.3
Error	79	84268.0	1066.7	Prob > F
Total	80	84570.6		0.6

Least Squares Means

Cluster	DM (%)	Std Error
	-----%-----	
1	30.0a†	5.2
2	26.2a	5.0
Mean	28.0	

†Means followed by the same letter are not significantly different (Student's t, $\alpha = 0.05$).

Table 28. Positive responses (√) for biological, metal salt, and physicochemical key components of online survey for each biosolids with sum (out of 28) and percentage (%) of positive responses.

<u>Key Components</u>	Biosolids (ID, see Table 1)																														N	%	
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	19	20	21	22	23	24	26	27	28	29	30					
<u>Biological</u>																																	
Activated sludge	√	√	√	√	√	√	√	√	√	√	√	√	√	√	√	√	√	√	√	√	√	√	√	√	√	√	√	√	√	√	√	28	100.0
BNR	√	√	√	√					√	√					√					√			√	√			√	√			12	42.9	
Aerobic stabilization	√	√	√	√	√	√	√			√	√		√	√		√			√										√		14	50.0	
Anaerobic stabilization								√	√			√						√			√	√	√	√	√		√	√			11	39.3	
<u>Metal Salts</u>																																	
Ferric sulfate		√	√																													2	7.1
Ferrous sulfate																									√					√		2	7.1
Ferric chloride												√											√	√								3	10.7
Sodium aluminate										√																						1	3.6
Alum				√					√	√															√							4	14.3
Polyaluminum chloride											√																					1	3.6
Polymers		√	√		√			√	√	√	√	√	√	√	√	√		√	√			√	√	√	√	√	√	√	√	√	√	22	78.6
<u>Physicochemical</u>																																	
Wet air oxidation stabilization																		√														1	3.6

Table 30. Effect of biosolids class on total P (TP; A, Class A; B, Class B).

<u>Summary of Fit</u>				
R ²				0.12
RMSE				11.9
CV (%)				50.9
Observations				81
<u>Analysis of Variance</u>				
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	1457.7	1457.7	10.3
Error	79	11176.5	141.5	Prob > F
Total	80	12634.2		0.0019
<u>Least Squares Means</u>				
Class		TP		Std Error
		-----g kg ⁻¹ -----		
A		16.7a†		2.5
B		26.1b		1.6
	Mean	23.4		

†Means followed by the same letter are not significantly different (Student's t, $\alpha = 0.05$).

Table 31. Effect of biosolids class on water-extractable P (WEP; A, Class A; B, Class B).

<u>Summary of Fit</u>				
R ²				0.03
RMSE				2.1
CV (%)				150.0
Observations				81
<u>Analysis of Variance</u>				
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	12.2	12.2	2.8
Error	79	336.8	4.3	Prob > F
Total	80	349.0		0.1
<u>Least Squares Means</u>				
Class	WEP		Std Error	
	-----g kg ⁻¹ -----			
A	0.8a†		0.4	
B	1.6a		0.3	
Mean	1.4			

†Means followed by the same letter are not significantly different (Student's t, $\alpha = 0.05$).

Table 32. Effect of biosolids class on percent water-extractable P (PWEF; A, Class A; B, Class B).

<u>Summary of Fit</u>				
R ²				0.02
RMSE				6.6
CV (%)				132.0
Observations				81
<u>Analysis of Variance</u>				
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	81.2	81.2	1.9
Error	79	3422.4	43.3	Prob > F
Total	80	3503.6		0.2
<u>Least Squares Means</u>				
Class		PWEF		Std Error
		-----%-----		
A		3.4a†		1.4
B		5.6a		0.9
Mean		5.0		

†Means followed by the same letter are not significantly different (Student's t, $\alpha = 0.05$).

Table 33. Effect of biosolids class on percent dry matter (DM, %; A, Class A; B, Class B).

<u>Summary of Fit</u>				
R ²				0.85
RMSE				12.8
CV (%)				45.7
Observations				81
<u>Analysis of Variance</u>				
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	71711.4	71711.4	440.6
Error	79	12859.2	162.8	Prob > F
Total	80	84570.6		<.0001
<u>Least Squares Means</u>				
Class		DM		Std Error
		-----%-----		
A		75.3a†		2.7
B		9.3b		1.7
Mean		28.0		

†Means followed by the same letter are not significantly different (Student's t, $\alpha = 0.05$).

Table 34. Effect of biosolids type (Class A-alkaline, Class A-heat, Class B-slurry, Class B-cake) on total P (TP).

<u>Summary of Fit</u>				
R ²				0.35
RMSE				10.4
CV (%)				44.4
Observations				81
<u>Analysis of Variance</u>				
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	4360.8	1453.6	13.5
Error	77	8273.4	107.5	Prob > F
Total	80	12634.2		<.0001
<u>Least Squares Means</u>				
BS Type	TP	Std Error		
		-----g kg ⁻¹ -----		
Class A-alkaline	5.0b†	3.1		
Class A-heat	27.5a	3.0		
Class B-slurry	25.9a	1.8		
Class B-cake	26.4a	2.0		
Mean	23.4			

†Means followed by the same letter are not significantly different (Student's t, $\alpha = 0.05$).

Table 35. Effect of biosolids type (Class A-alkaline, Class A-heat, Class B-slurry, Class B-cake) on water-extractable P (WEP).

<u>Summary of Fit</u>				
R ²				0.13
RMSE				2.0
CV (%)				142.9
Observations				81
<u>Analysis of Variance</u>				
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	45.8	15.3	3.9
Error	77	303.2	4.0	Prob > F
Total	80	349.0		<.0123
<u>Least Squares Means</u>				
BS Type	WEP	Std Error		
		-----g kg ⁻¹ -----		
Class B-slurry	2.2a†	0.4		
Class A-heat	1.4ab	0.6		
Class B-cake	0.9b	0.4		
Class A-alkaline	0.07b	0.6		
Mean	1.4			

†Means followed by the same letter are not significantly different (Student's t, $\alpha = 0.05$).

Table 36. Effect of biosolids type (Class A-alkaline, Class A-heat, Class B-slurry, Class B-cake) on percent water-extractable P (PWEP).

<u>Summary of Fit</u>				
R ²				0.12
RMSE				6.4
CV (%)				128.0
Observations				81
<u>Analysis of Variance</u>				
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	376.3	125.4	3.1
Error	77	3127.3	40.6	Prob > F
Total	80	3503.6		<.0320
<u>Least Squares Means</u>				
BS Type	PWEP		Std Error	
		-----%-----		
Class B-slurry	7.5a†		1.1	
Class A-heat	4.8ab		1.8	
Class B-cake	3.3b		1.2	
Class A-alkaline	1.9b		1.9	
Mean	5.0			

†Means followed by the same letter are not significantly different (Student's t, $\alpha = 0.05$).

Table 37. Effect of biosolids type (Class A-alkaline, Class A-heat, Class B-slurry, Class B-cake) on percent dry matter (DM, %).

<u>Summary of Fit</u>				
R ²				0.96
RMSE				6.8
CV (%)				24.3
Observations				81
<u>Analysis of Variance</u>				
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	81055.1	27018.4	591.8
Error	77	3515.5	45.7	Prob > F
Total	80	84570.6		<.0001
<u>Least Squares Means</u>				
BS Type	DM		Std Error	
		-----%-----		
Class A-heat	91.6a†		2.0	
Class A-alkaline	57.5b		2.0	
Class B-cake	16.8c		1.3	
Class B-slurry	3.2d		1.2	
Mean	28.0			

†Means followed by the same letter are not significantly different (Student's t, $\alpha = 0.05$).

Table 38. Effect of biosolids Class A type (Class A-alkaline and Class A-heat) on total P (TP).

<u>Summary of Fit</u>				
R ²				0.83
RMSE				5.4
CV (%)				32.3
Observations				23
<u>Analysis of Variance</u>				
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	2899.6	2899.7	101.2
Error	21	601.6	28.7	Prob > F
Total	22	3501.2		<.0001
<u>Least Squares Means</u>				
Class A Types	TP	Std Error		
		-----g kg ⁻¹ -----		
Class A-alkaline	5.0b†	1.6		
Class A-heat	27.5a	1.5		
Mean	16.7			

†Means followed by the same letter are not significantly different (Student's t, $\alpha = 0.05$).

Table 39. Effect of biosolids Class A type (Class A-alkaline and Class A-heat) on water-extractable P (WEP).

<u>Summary of Fit</u>	
R ²	0.34
RMSE	1.0
CV (%)	125.0
Observations	23

<u>Analysis of Variance</u>				
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	10.2	10.2	10.9
Error	21	19.6	0.9	Prob > F
Total	22	29.8		0.0034

<u>Least Squares Means</u>		
Class A Types	WEP	Std Error
	-----g kg ⁻¹ -----	
Class A-alkaline	0.07b†	0.3
Class A-heat	1.4a	0.3
Mean	0.8	

†Means followed by the same letter are not significantly different (Student's t, $\alpha = 0.05$).

Table 40. Effect of biosolids Class A type (Class A-alkaline and Class A-heat) on percent water-extractable P (PWEP).

<u>Summary of Fit</u>	
R ²	0.20
RMSE	3.04
CV (%)	89.4
Observations	23

<u>Analysis of Variance</u>				
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	47.4	47.4	5.1
Error	21	194.2	9.2	Prob > F
Total	22	241.5		0.0344

<u>Least Squares Means</u>		
Class A Types	PWEP	Std Error
	-----%-----	
Class A-alkaline	1.9b†	0.9
Class A-heat	4.8a	0.9
Mean	3.4	

†Means followed by the same letter are not significantly different (Student's t, $\alpha = 0.05$).

Table 41. Effect of biosolids Class A type (Class A-alkaline and Class A-heat) on percent dry matter (DM, %).

<u>Summary of Fit</u>	
R ²	0.67
RMSE	12.5
CV (%)	16.6
Observations	23

<u>Analysis of Variance</u>				
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	6669.3	6669.3	42.5
Error	21	3293.4	156.8	Prob > F
Total	22	9962.8		<.0001

<u>Least Squares Means</u>		
Class A Types	DM	Std Error
	-----%-----	
Class A-alkaline	57.5b†	3.8
Class A-heat	91.6a	3.6
Mean	75.3	

†Means followed by the same letter are not significantly different (Student's t, $\alpha = 0.05$).

Table 42. Effect of biosolids Class B consistency (Class B-slurry, Class B-cake) on total P (TP).

Summary of Fit

R ²	0.000447
RMSE	11.7
CV (%)	44.8
Observations	58

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	3.4	3.4	0.03
Error	56	7671.9	137.0	Prob > F
Total	57	7675.3		0.9

Least Squares Means

Class B Consistency	TP	Std Error
	-----g kg ⁻¹ -----	
Class B-slurry	25.9a†	2.3
Class B-cake	26.4a	2.1
Mean	26.1	

†Means followed by the same letter are not significantly different (Student's t, $\alpha = 0.05$).

Table 43. Effect of biosolids Class B consistency (Class B-slurry, Class B-cake) on water-extractable P (WEP).

Summary of Fit

R ²	0.08
RMSE	2.3
CV (%)	143.8
Observations	58

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	23.4	23.4	4.6
Error	56	283.6	5.1	Prob > F
Total	57	307.0		0.04

Least Squares Means

Class B Consistency	WEP	Std Error
	-----g kg ⁻¹ -----	
Class B-slurry	2.2a†	2.2
Class B-cake	0.9b	0.9
Mean	1.6	

†Means followed by the same letter are not significantly different (Student's t, $\alpha = 0.05$).

Table 44. Effect of biosolids Class B consistency (Class B-slurry, Class B-cake) on percent water-extractable P (PWEP).

<u>Summary of Fit</u>	
R ²	0.08
RMSE	7.2
CV (%)	128.6
Observations	58

<u>Analysis of Variance</u>				
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	247.7	247.7	4.7
Error	56	2933.2	52.4	Prob > F
Total	57	3180.9		0.03

<u>Least Squares Means</u>		
Class B Consistency	PWEP	Std Error
	-----%-----	
Class B-slurry	7.5a†	1.3
Class B-cake	3.3b	1.4
Mean	5.6	

†Means followed by the same letter are not significantly different (Student's t, $\alpha = 0.05$).

Table 45. Effect of biosolids Class B consistency (Class B-slurry and Class B-cake) on percent dry matter (DM, %).

<u>Summary of Fit</u>	
R ²	0.92
RMSE	2.0
CV (%)	21.5
Observations	58

<u>Analysis of Variance</u>				
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	2674.3	2674.3	674.3
Error	56	222.1	4.0	Prob > F
Total	57	2896.4		<.0001

<u>Least Squares Means</u>		
Class B Consistency	DM	Std Error
	-----%-----	
Class B-slurry	3.2a†	0.4
Class B-cake	16.8b	0.4
Mean	9.3	

†Means followed by the same letter are not significantly different (Student's t, $\alpha = 0.05$).

Table 46. Effect of watershed on effluent total P concentration (mg TP L⁻¹ effluent). Watersheds with only a single WWTP were excluded from the analysis.

<u>Summary of Fit</u>				
R ²			0.68	
RMSE			0.8	
CV (%)				
Observations			20	
<u>Analysis of Variance</u>				
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	19.4	4.9	8.0
Error	15	9.1	0.6	Prob > F
Total	19	28.5		0.0011
<u>Least Squares Means</u>				
Watershed	TP	Std Error	Number of WWTPs	
	-----mg L ⁻¹ -----			
Cape Fear	3.1a†	0.4	4	
Broad	2.8††		1	
Yadkin	2.4ab	0.6	2	
Lumber	1.5bc	0.6	2	
New	1.2		1	
Catawba	1.0bc	0.3	5	
Tar Pamlico	1.0		1	
Little Tennessee	1.0		1	
French Broad	1.0		1	
Neuse	0.6c	0.3	7	
Pasquotank	0.2		1	
Mean	1.5			

†Means followed by the same letter are not significantly different (Student's t, $\alpha = 0.05$).

††Means without letters were not included in analysis.

Table 47. The two options considered for creating biosolids categories for implementation into the Phosphorus Loss Assessment Tool. Biosolids (BS) parameters for each category include total P, water-extractable P, percent water extractable P, and percent DM (TP; WEP; PWE; DM, %).

BS Parameters	Option 1		Option 2			
	Class A	Class B	Class A-alkaline	Class A-heat	Class B-slurry	Class B-cake
TP (g kg⁻¹ BS)						
Median	17.8	24.4	2.8	26.3	24.1	26.3
Mean	16.7a†	26.1b	5.0b	27.5a	25.9a	26.4a
WEP (g kg⁻¹ BS)						
Median	0.3	0.7	0.04	1.2	1.0	0.7
Mean	0.8a	1.6a	0.07b	1.4ab	2.2a	0.9b
PWE (%)						
Median	2.2	3.0	1.8	3.5	3.8	2.5
Mean	3.4a	5.6a	1.9b	4.8ab	7.5a	3.3b
DM (%)						
Median	88.4	4.5	55.2	90.9	3.3	16.7
Mean	75.3a	9.3b	57.5b	91.6a	3.2d	16.8c

†Means within an option row followed by the same letter are not significantly different (Student's t, $\alpha = 0.05$).

Table 48. Phosphorus content in selected biosolids types. Total P; soluble P fraction weight basis (soluble P fraction = soluble P/total P); percent solids (% Solids); soluble P (soluble P = total P*soluble fraction), nonsoluble P (nonsoluble P = total P - soluble P), and units of application in pounds P per 1000 gal or ton, calculated on a wet weight basis.

Biosolids Type	Total P	Soluble Fraction (wt. basis)	% Solids	Soluble P	Nonsoluble P	Pounds P Per
Class B slurry†	6.92	0.085	3.2	0.588	6.332	1000 gal
Class B cake	8.87	0.034	16.8	0.302	8.568	ton
Class A alkaline	5.75	0.014	57.5	0.081	5.669	ton
Class A heat dried	50.38	0.051	91.6	2.565	47.815	ton

†Approximate density calculated as 8.34 lbs gal⁻¹

FIGURES

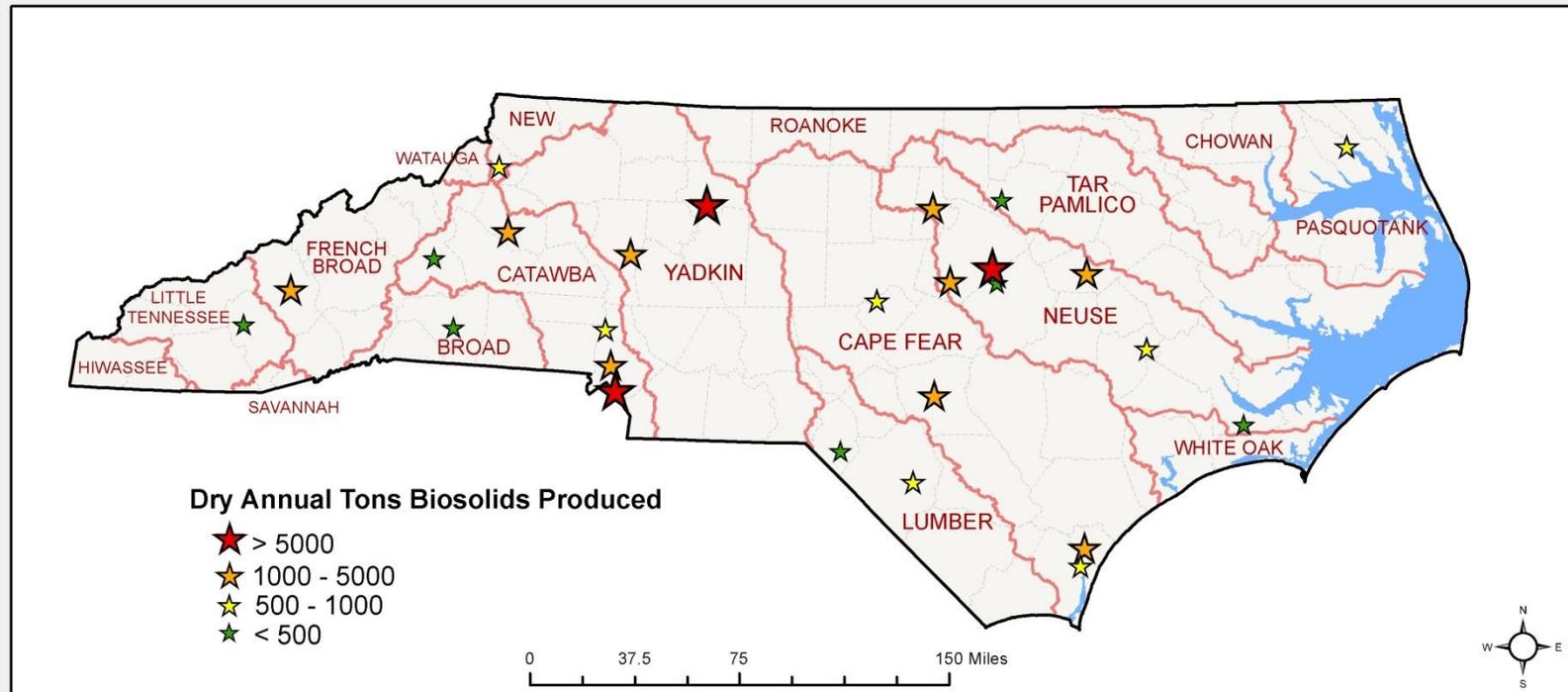


Fig. 1. Locations of the participating wastewater treatment plants (WWTPs). Stars represent the amount of dry annual tons biosolids produced by each WWTP.

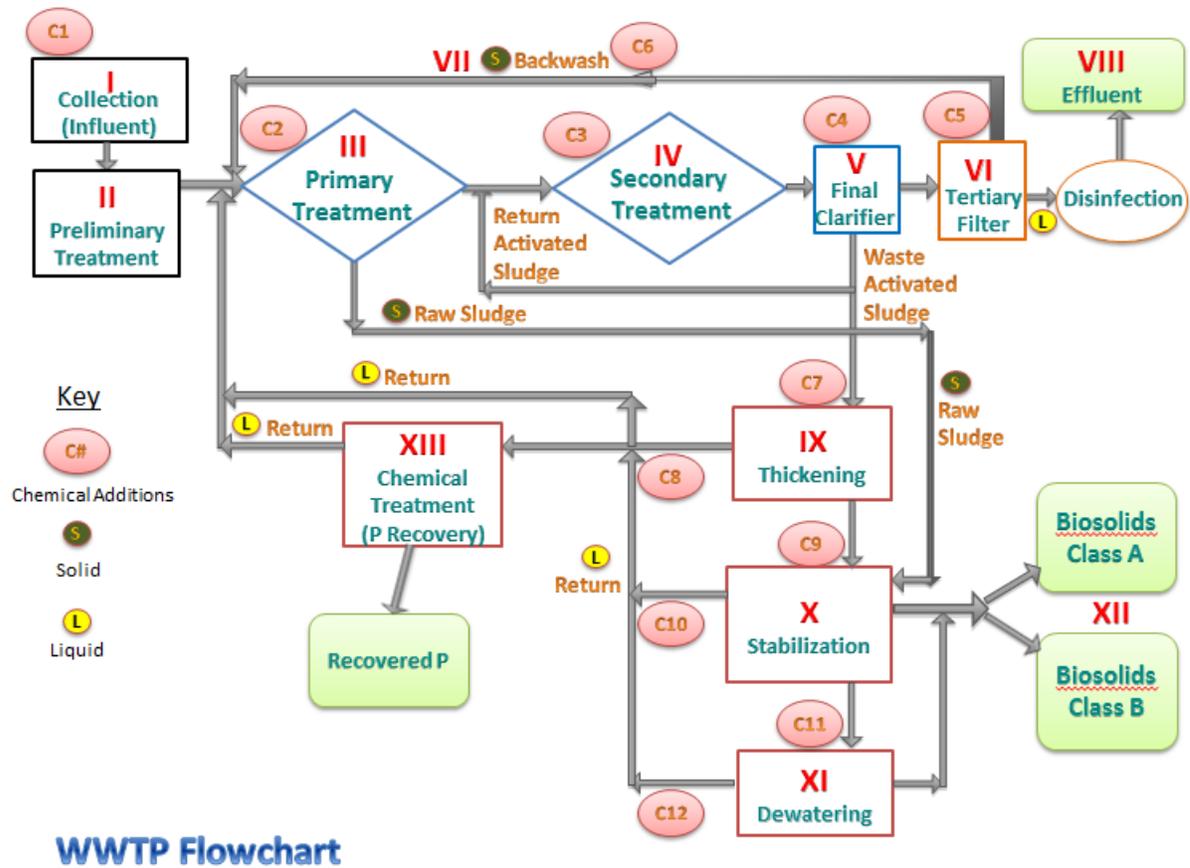


Fig. 2. Wastewater treatment plant (WWTP) flowchart that served as the basis of the online survey questionnaire and included with same when the survey was administered to Operators in Responsible Charge (ORCs).

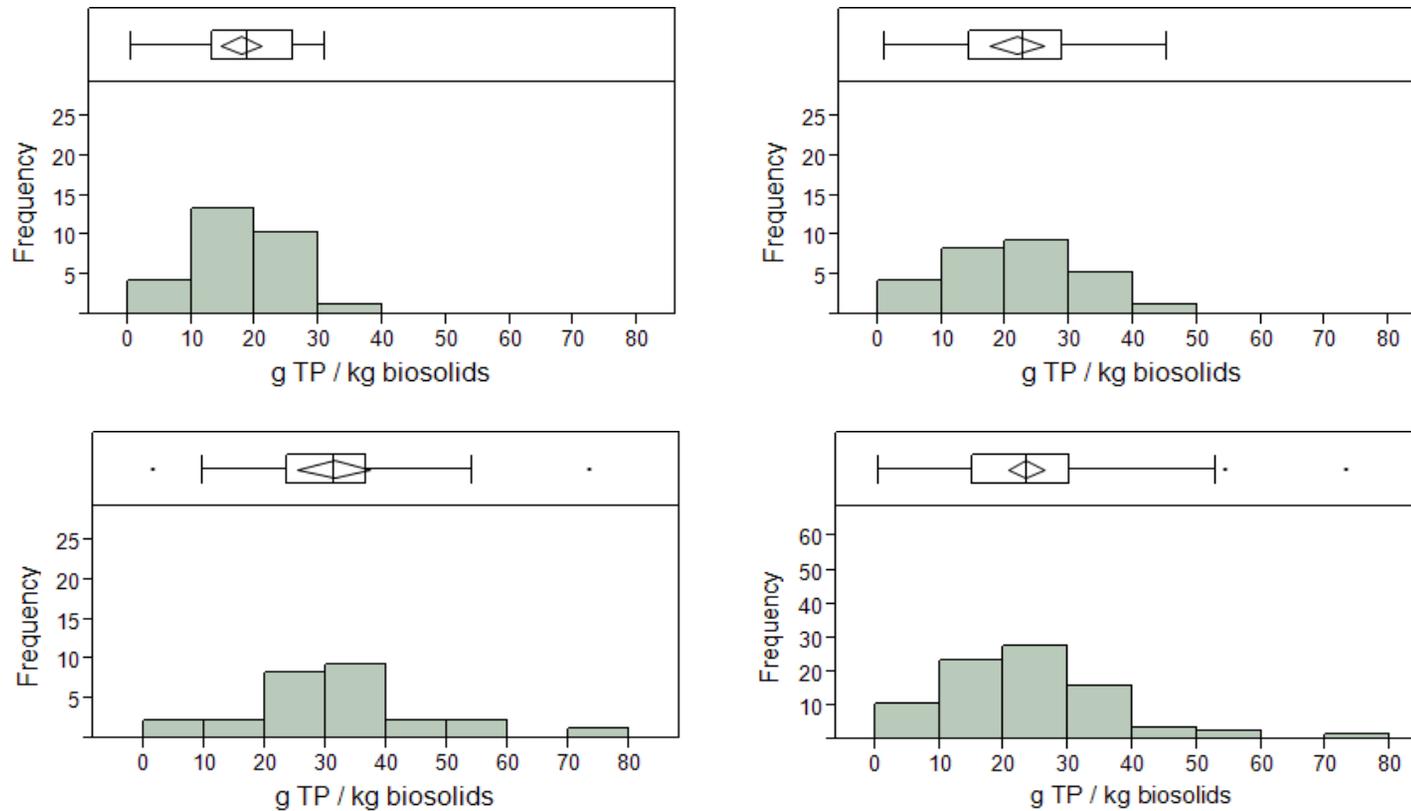


Fig. 3. Frequency distributions of biosolids total P (TP); for all seasons and seasons combined. Top left, Summer 2013; top right, Fall 2013; bottom left, Winter 2014; bottom right, seasons combined. Boxplots: the vertical line within a box is the median; the ends of the box are the 1st and 3rd quartiles, the difference between which is the interquartile range (IQR); the whiskers extend to the furthest point within 1.5*IQR from the box; points beyond the whiskers are potential outliers. The middle of the confidence diamond is the mean and left and right points of the diamond represent the upper and lower 95% of the mean.

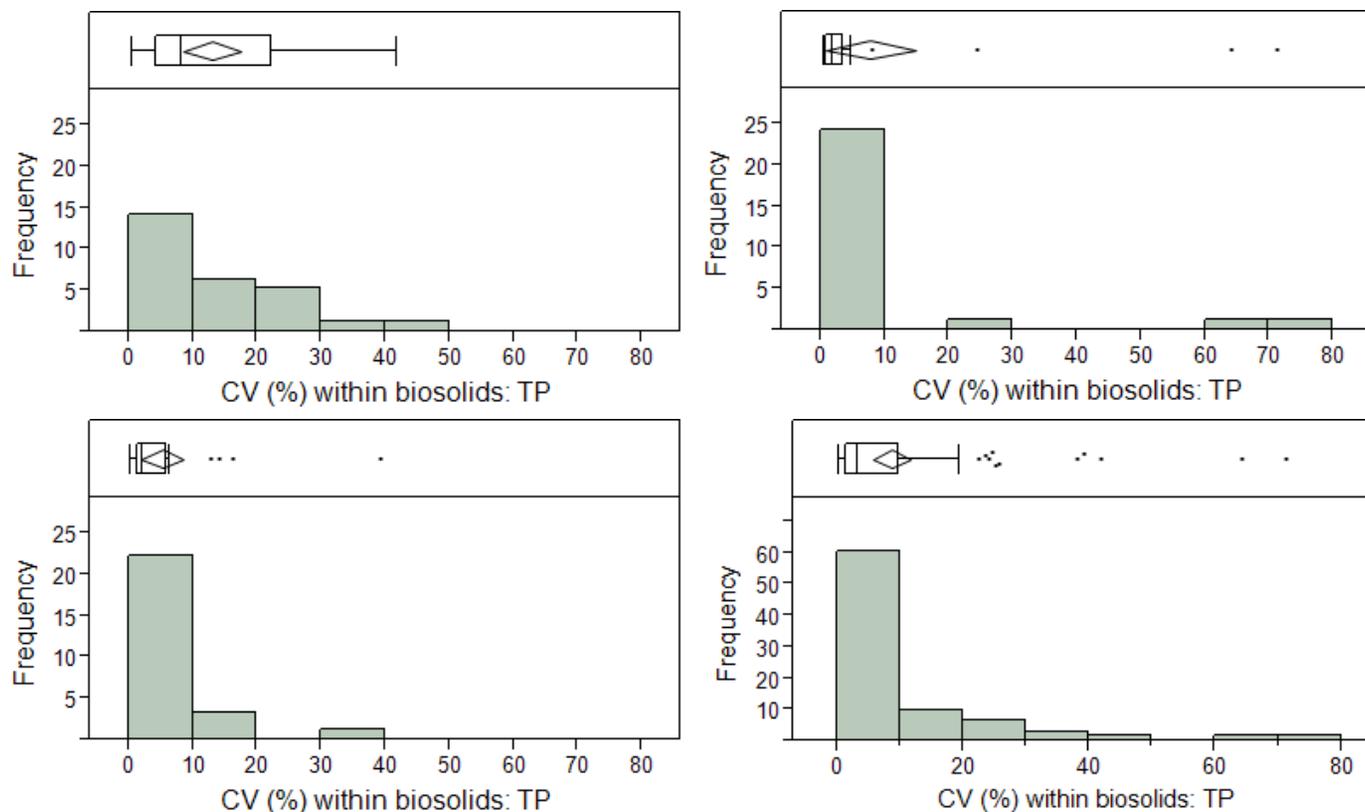


Fig. 4. Frequency distributions of the coefficient of variation ($100 \times \text{standard deviation} / \text{mean}$) (CV) (%) within biosolids total P (TP) for all seasons and seasons combined. Top left, Summer 2013; top right, Fall 2013; bottom left, Winter 2014; bottom right, seasons combined. Boxplots: the vertical line within a box is the median; the ends of the box are the 1st and 3rd quartiles, the difference between which is the interquartile range (IQR); the whiskers extend to the furthest point within $1.5 \times \text{IQR}$ from the box; points beyond the whiskers are potential outliers. The middle of the confidence diamond is the mean and left and right points of the diamond represent the upper and lower 95% of the mean.

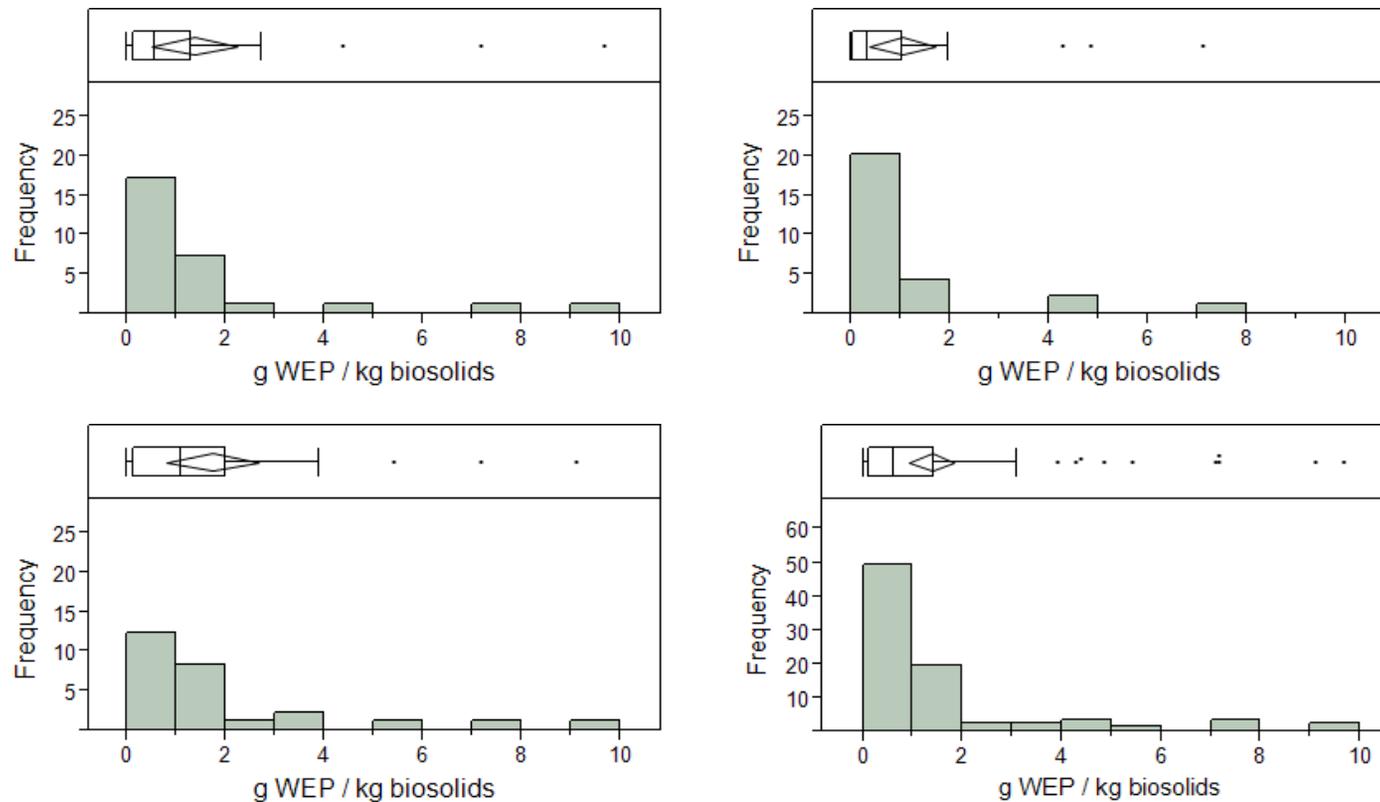


Fig. 5. Frequency distributions of biosolids water-extractable P (WEP); for all seasons and seasons combined. Top left, Summer 2013; top right, Fall 2013; bottom left, Winter 2014; bottom right, seasons combined. Boxplots: the vertical line within a box is the median; the ends of the box are the 1st and 3rd quartiles, the difference between which is the interquartile range (IQR); the whiskers extend to the furthest point within 1.5*IQR from the box; points beyond the whiskers are potential outliers. The middle of the confidence diamond is the mean and left and right points of the diamond represent the upper and lower 95% of the mean.

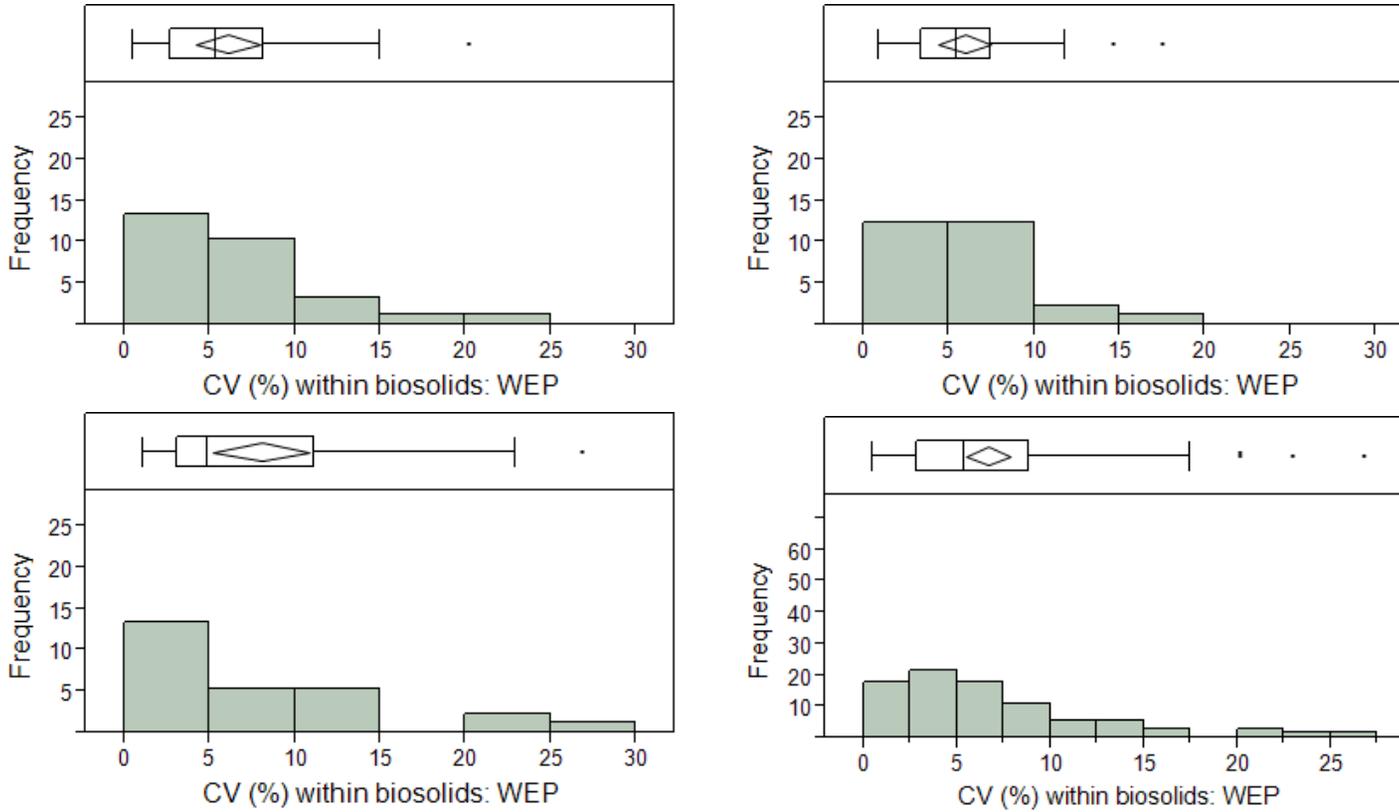


Fig. 6. Frequency distributions of the coefficient of variation ($100 \times \text{standard deviation} / \text{mean}$) (CV) (%) within biosolids water-extractable P (WEP) for all seasons and seasons combined. Top left, Summer 2013; top right, Fall 2013; bottom left, Winter 2014; bottom right, seasons combined. Boxplots: the vertical line within a box is the median; the ends of the box are the 1st and 3rd quartiles, the difference between which is the interquartile range (IQR); the whiskers extend to the furthest point within $1.5 \times \text{IQR}$ from the box; points beyond the whiskers are potential outliers. The middle of the confidence diamond is the mean and left and right points of the diamond represent the upper and lower 95% of the mean.

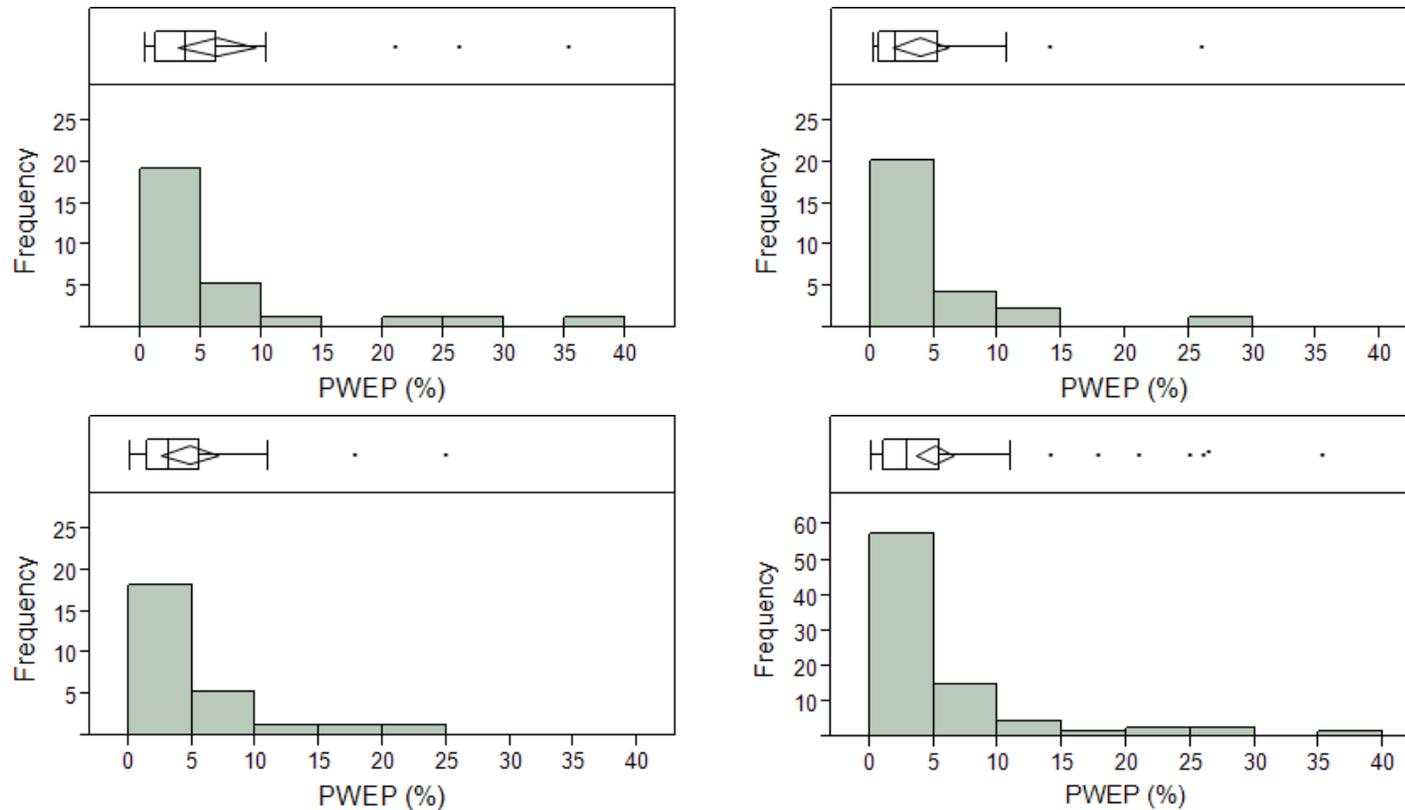


Fig. 7. Frequency distributions of biosolids percent water-extractable P ($PWE = 100 * WEP / TP$); for all seasons and seasons combined. Top left, Summer 2013; top right, Fall 2013; bottom left, Winter 2014; bottom right, seasons combined. Boxplots: the vertical line within a box is the median; the ends of the box are the 1st and 3rd quartiles, the difference between which is the interquartile range (IQR); the whiskers extend to the furthest point within $1.5 * IQR$ from the box; points beyond the whiskers are potential outliers. The middle of the confidence diamond is the mean and left and right points of the diamond represent the upper and lower 95% of the mean.

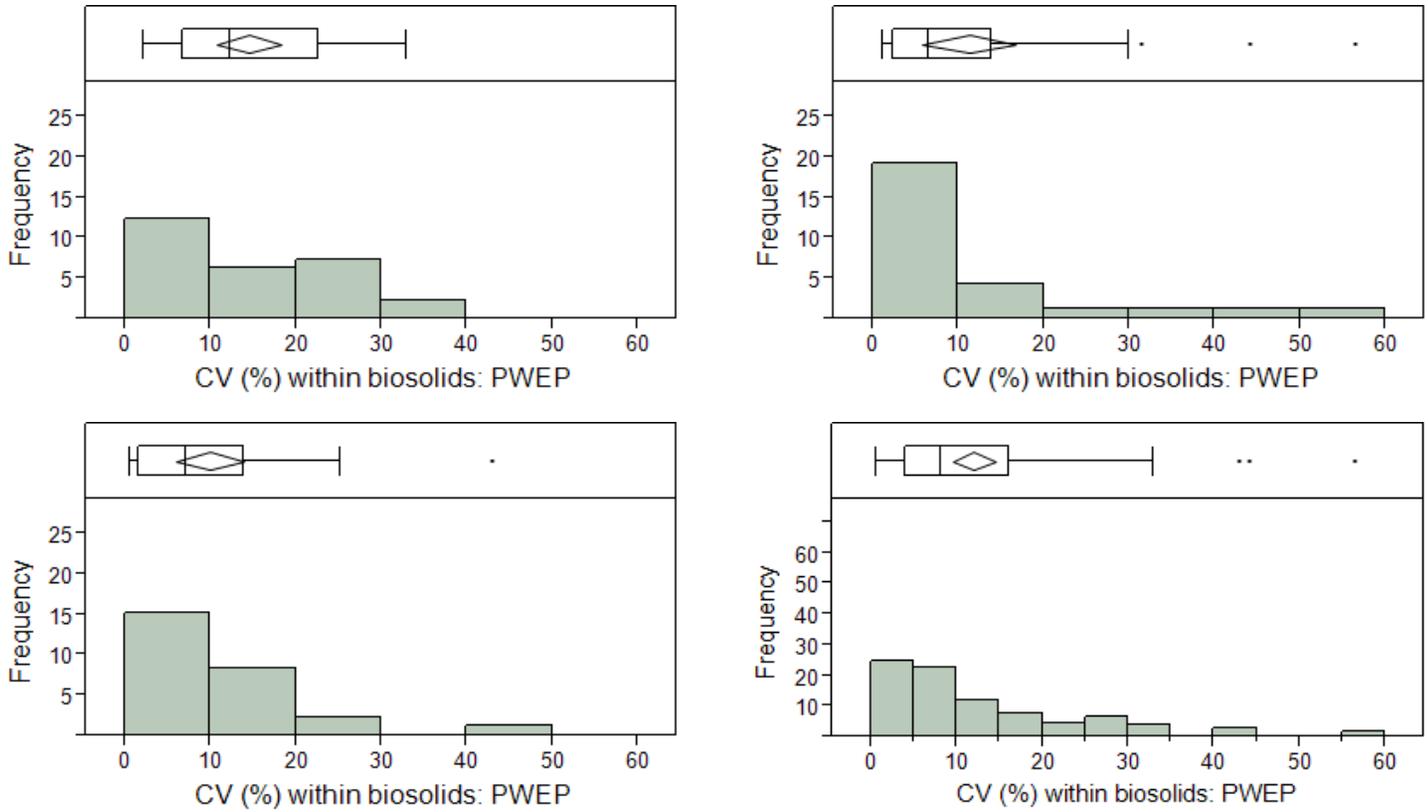


Fig. 8. Frequency distributions of the coefficient of variation ($100 \times \text{standard deviation} / \text{mean}$) (CV) (%) within biosolids percent water-extractable P ($\text{PWEP} = 100 \times \text{WEP} / \text{TP}$) for all seasons and seasons combined. Top left, Summer 2013; top right, Fall 2013; bottom left, Winter 2014; bottom right, seasons combined. Boxplots: the vertical line within a box is the median; the ends of the box are the 1st and 3rd quartiles, the difference between which is the interquartile range (IQR); the whiskers extend to the furthest point within $1.5 \times \text{IQR}$ from the box; points beyond the whiskers are potential outliers. The middle of the confidence diamond is the mean and left and right points of the diamond represent the upper and lower 95% of the mean.

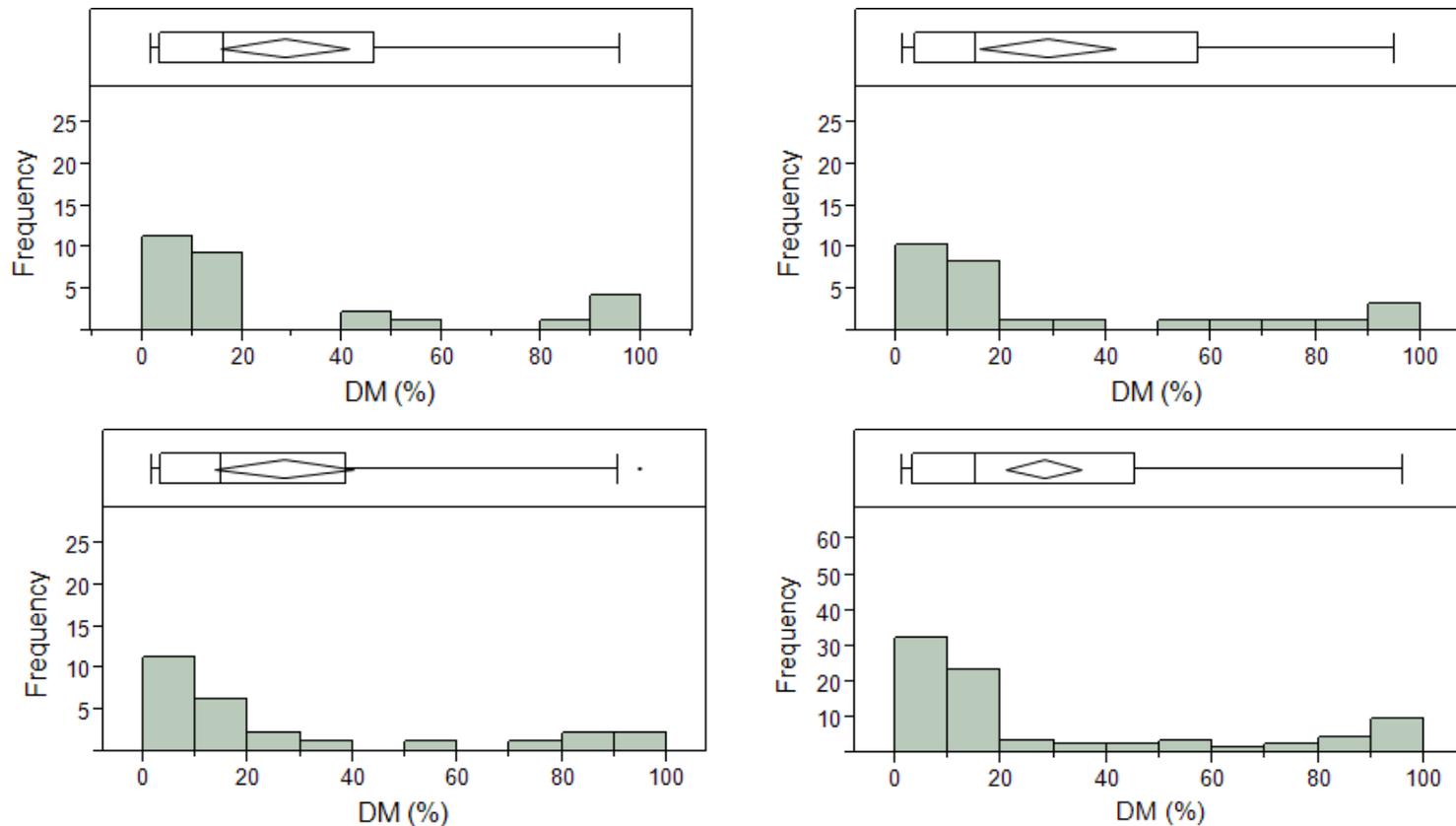


Fig. 9. Frequency distributions of biosolids percent dry matter (DM, %); for all seasons and seasons combined. Top left, Summer 2013; top right, Fall 2013; bottom left, Winter 2014; bottom right, seasons combined. Boxplots: the vertical line within a box is the median; the ends of the box are the 1st and 3rd quartiles, the difference between which is the interquartile range (IQR); the whiskers extend to the furthest point within 1.5*IQR from the box; points beyond the whiskers are potential outliers. The middle of the confidence diamond is the mean and left and right points of the diamond represent the upper and lower 95% of the mean.

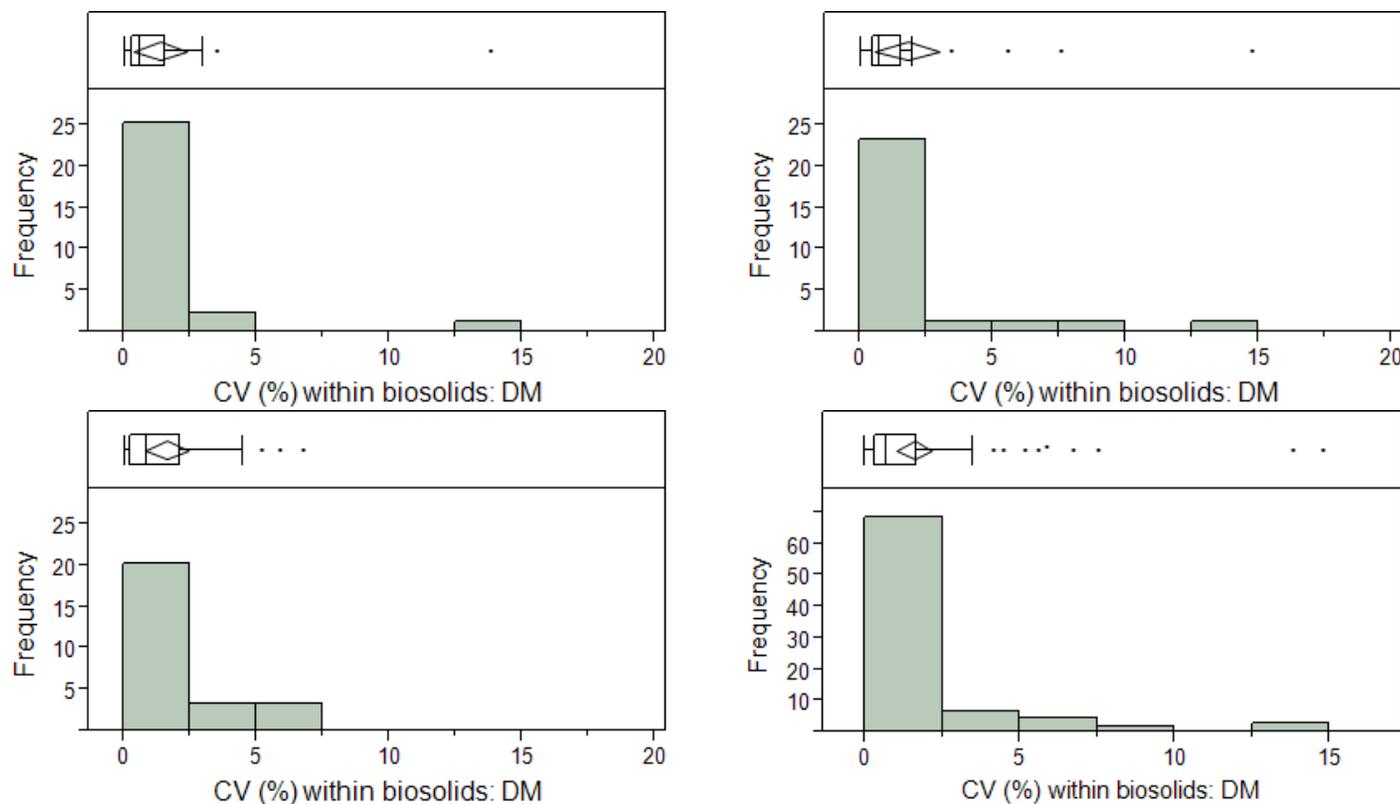


Fig. 10. Frequency distributions of the coefficient of variation ($100 \times \text{standard deviation} / \text{mean}$) (CV) (%) within biosolids percent dry matter (DM, %) for all seasons and seasons combined. Top left, Summer 2013; top right, Fall 2013; bottom left, Winter 2014; bottom right, seasons combined. Boxplots: the vertical line within a box is the median; the ends of the box are the 1st and 3rd quartiles, the difference between which is the interquartile range (IQR); the whiskers extend to the furthest point within $1.5 \times \text{IQR}$ from the box; points beyond the whiskers are potential outliers. The middle of the confidence diamond is the mean and left and right points of the diamond represent the upper and lower 95% of the mean.

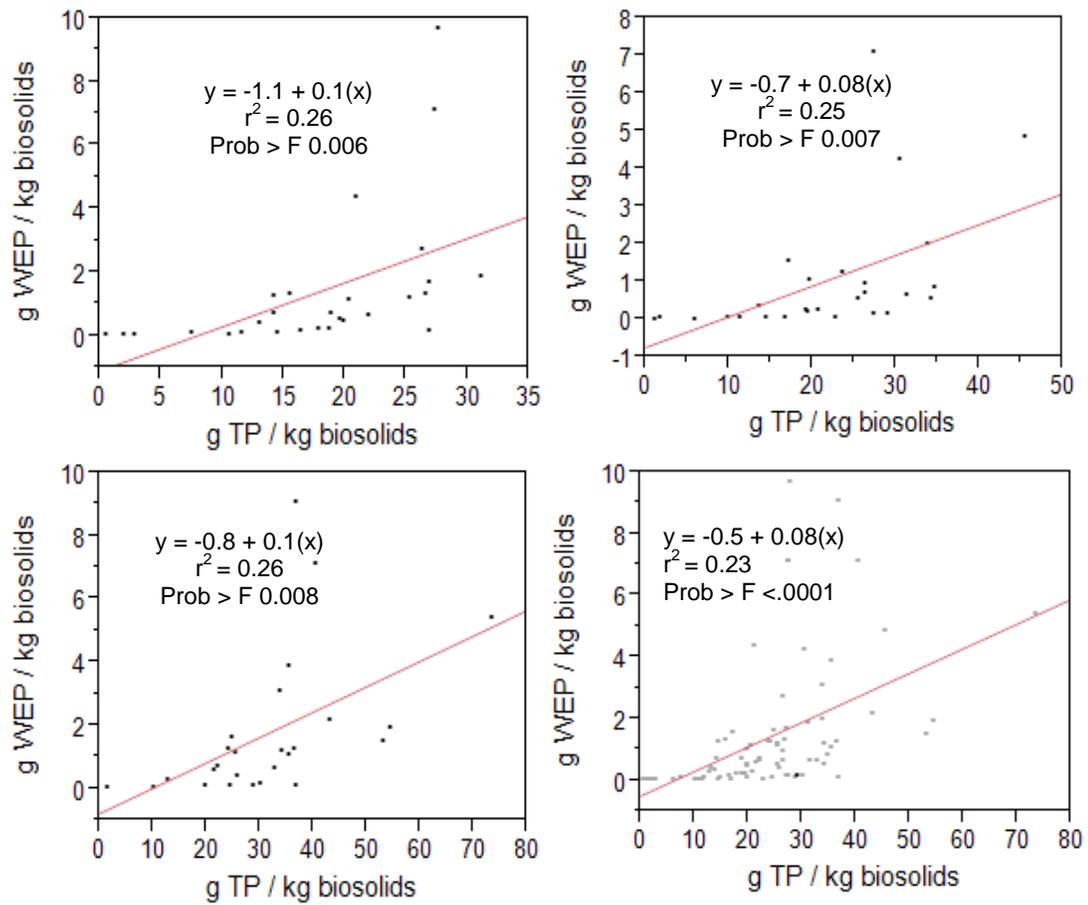


Fig. 11. Bivariate linear fit correlations for water-extractable P (WEP) by total P (TP) for all seasons and seasons combined. Top left, Summer 2013; top right, Fall 2013; bottom left, Winter 2014; bottom right, seasons combined.

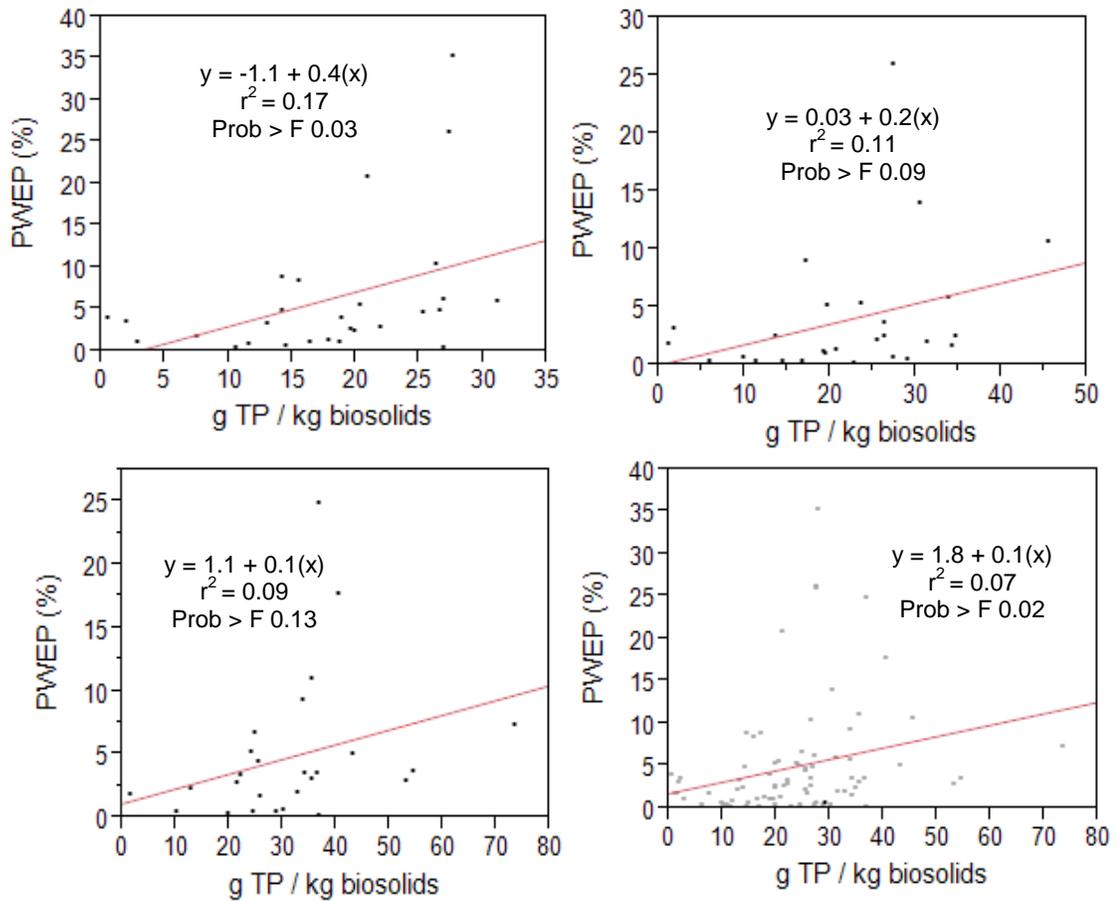


Fig. 12. Bivariate linear fit correlations for percent water-extractable P (PWEP = 100*WEP/TP) by total P (TP) for all seasons and seasons combined. Top left, Summer 2013; top right, Fall 2013; bottom left, Winter 2014; bottom right, seasons combined.

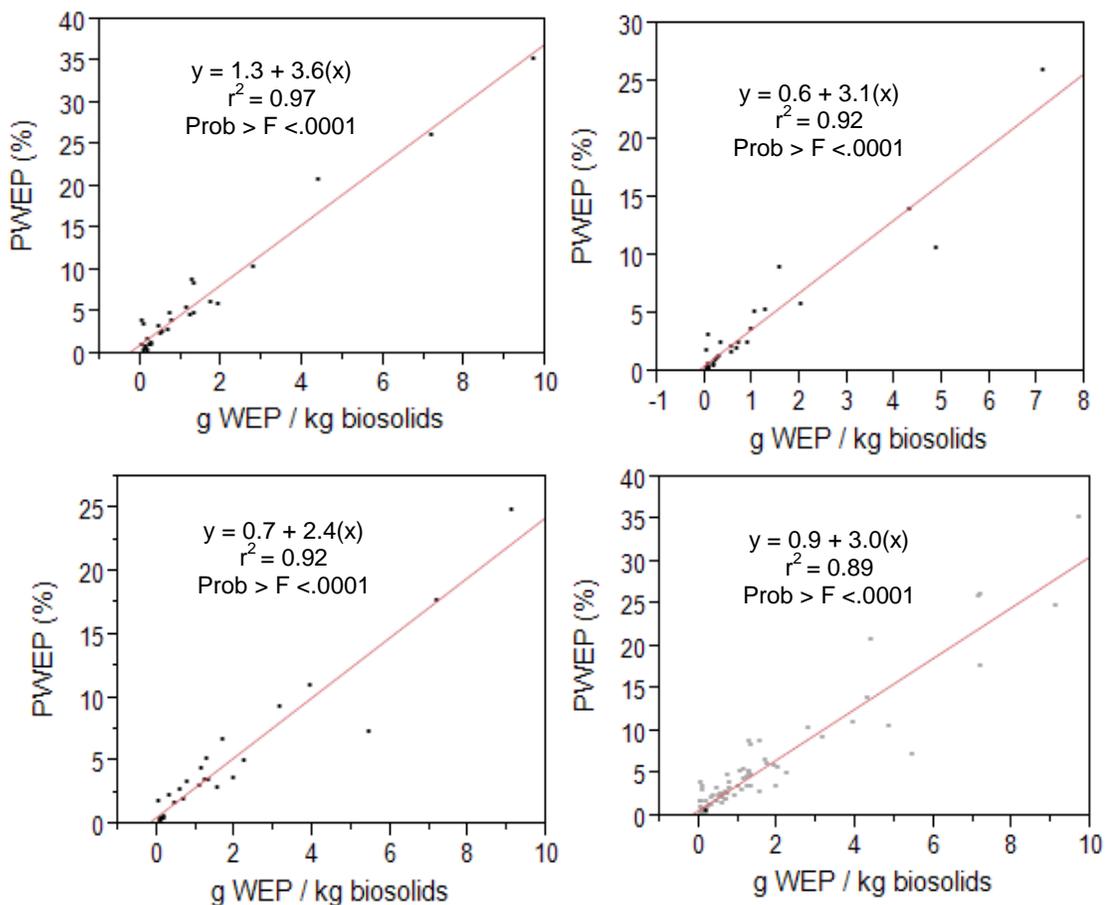


Fig. 13. Bivariate linear fit correlations for percent water-extractable P (PWEP = $100 \cdot \text{WEP} / \text{TP}$) by water-extractable P (WEP) for all seasons and seasons combined. Top left, Summer 2013; top right, Fall 2013; bottom left, Winter 2014; bottom right, seasons combined.

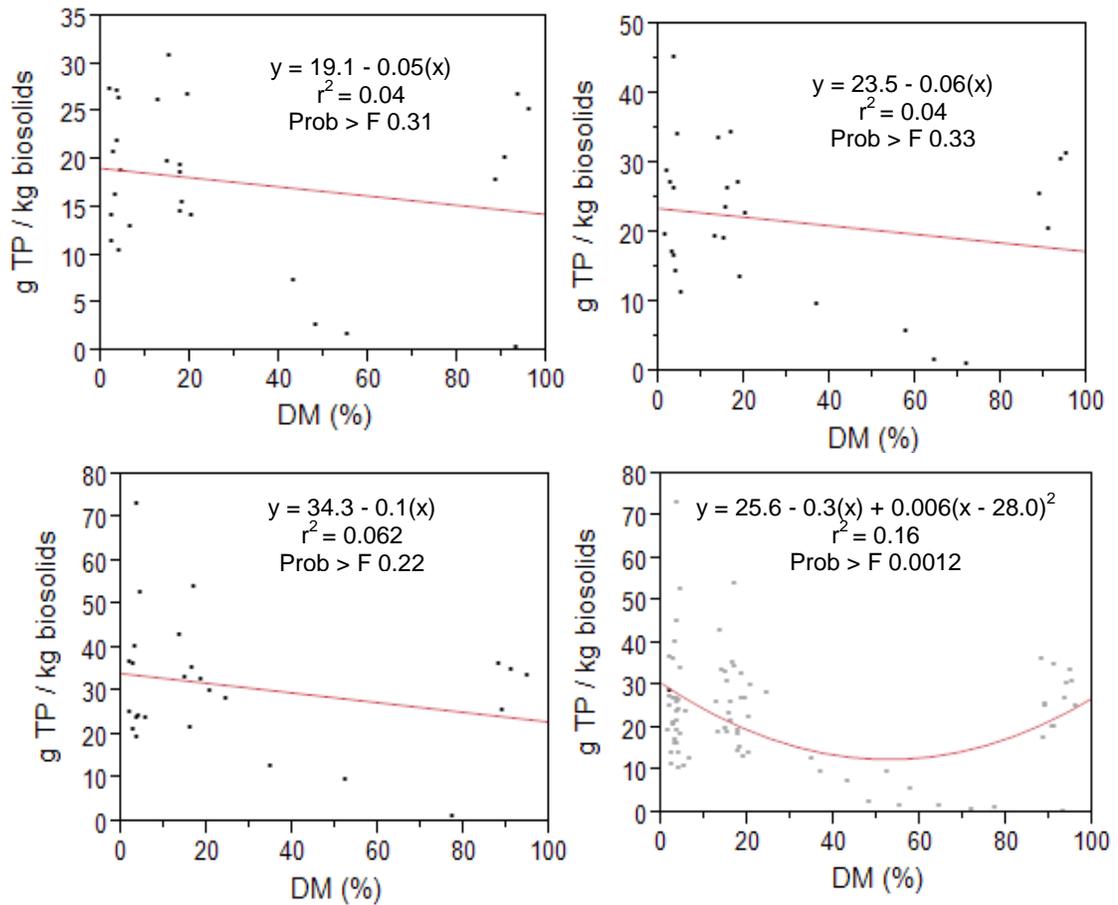


Fig. 14. Bivariate linear fit correlations for total P (TP) by percent dry matter (DM, %) for all seasons. Top left, Summer 2013; top right, Fall 2013; bottom left, Winter 2014. Bottom right, bivariate quadratic regression for TP by percent DM for seasons combined.

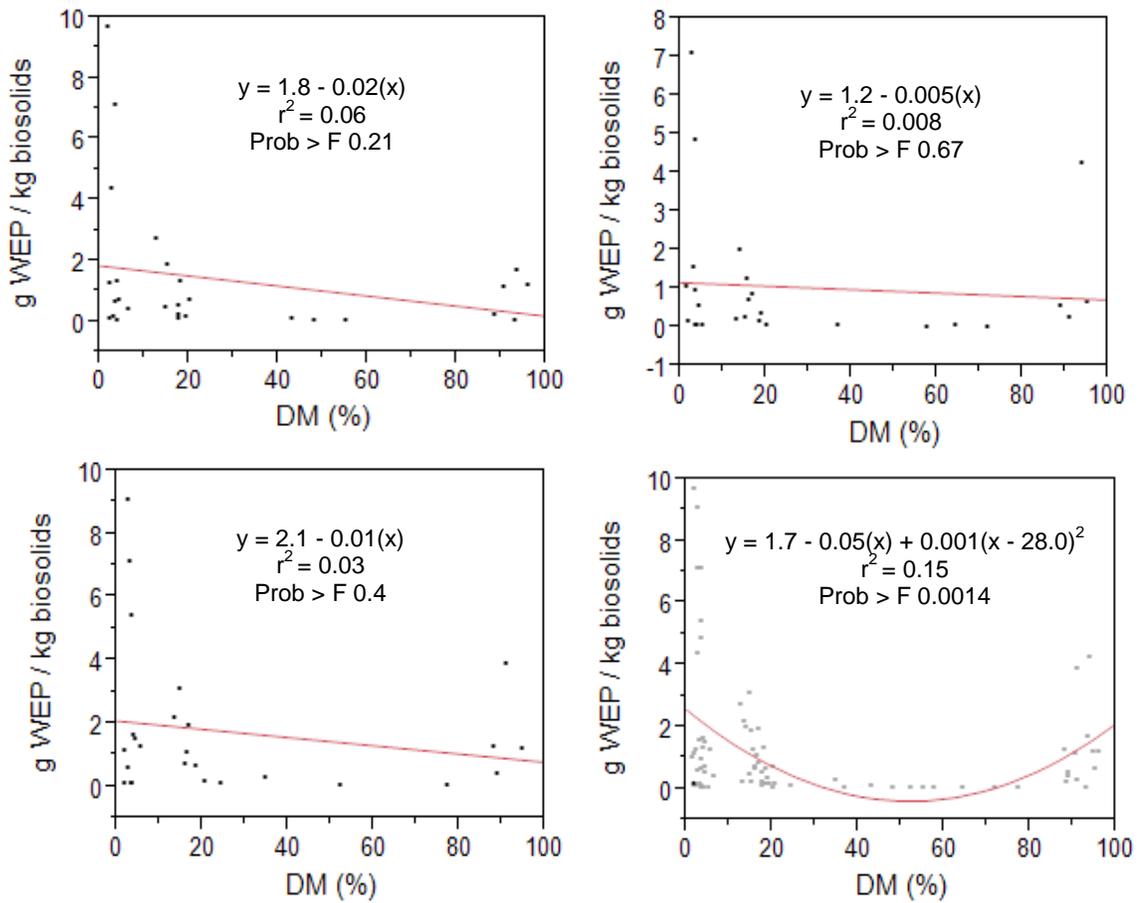


Fig. 15. Bivariate linear fit correlations for water-extractable P (WEP) by percent dry matter (DM, %) for all seasons. Top left, Summer 2013; top right, Fall 2013; bottom left, Winter 2014. Bottom right, bivariate quadratic regression for WEP by percent DM for seasons combined.

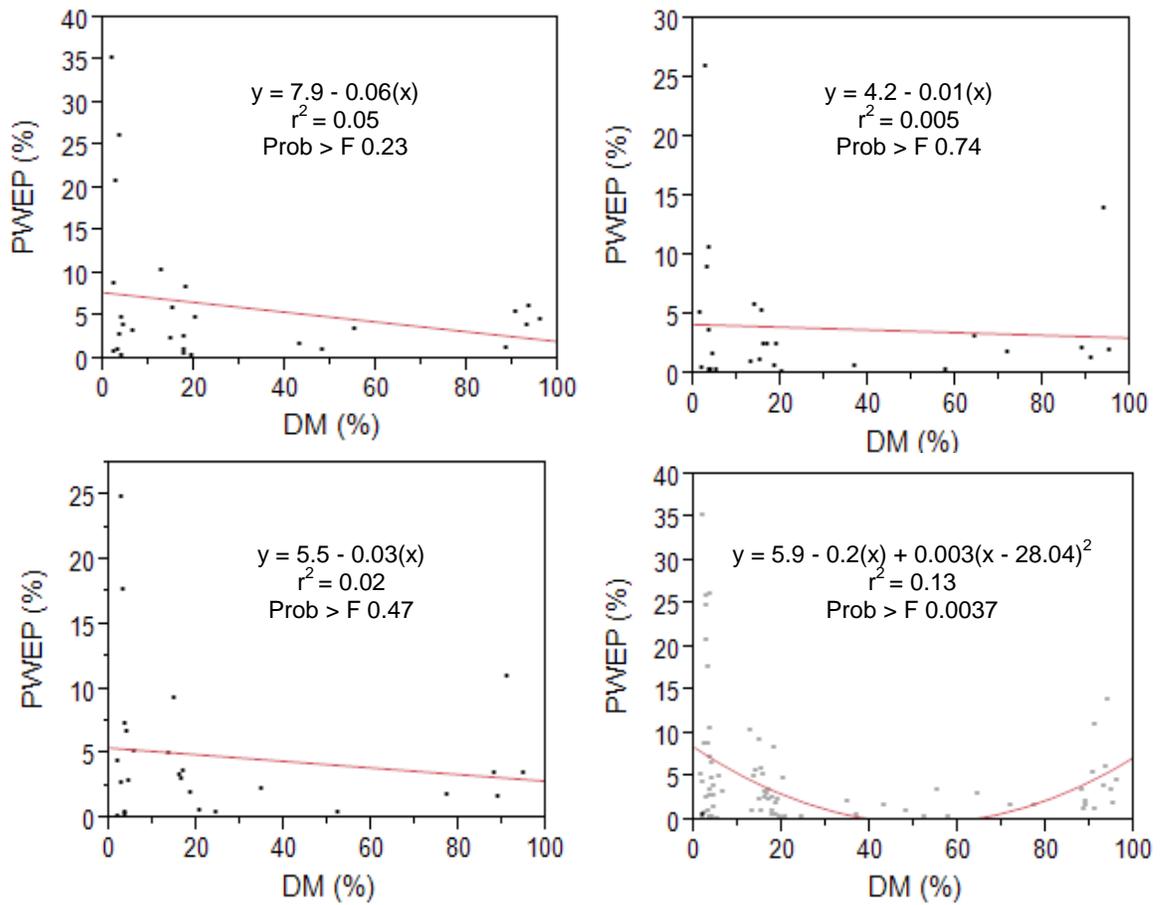


Fig. 16. Bivariate linear fit correlations for percent water-extractable P (PWE) by percent dry matter (DM, %) for all seasons. Top left, Summer 2013; top right, Fall 2013; bottom left, Winter 2014. Bottom right, bivariate quadratic regression for PWE by percent DM for seasons combined.

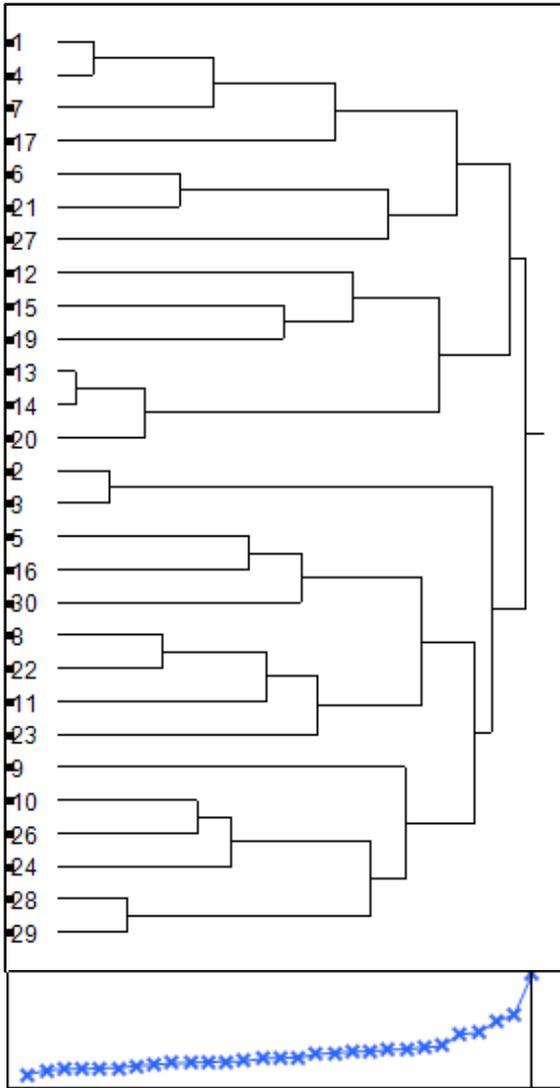


Fig. 17. Online survey hierarchical cluster analysis dendrogram and distance graph (bottom).

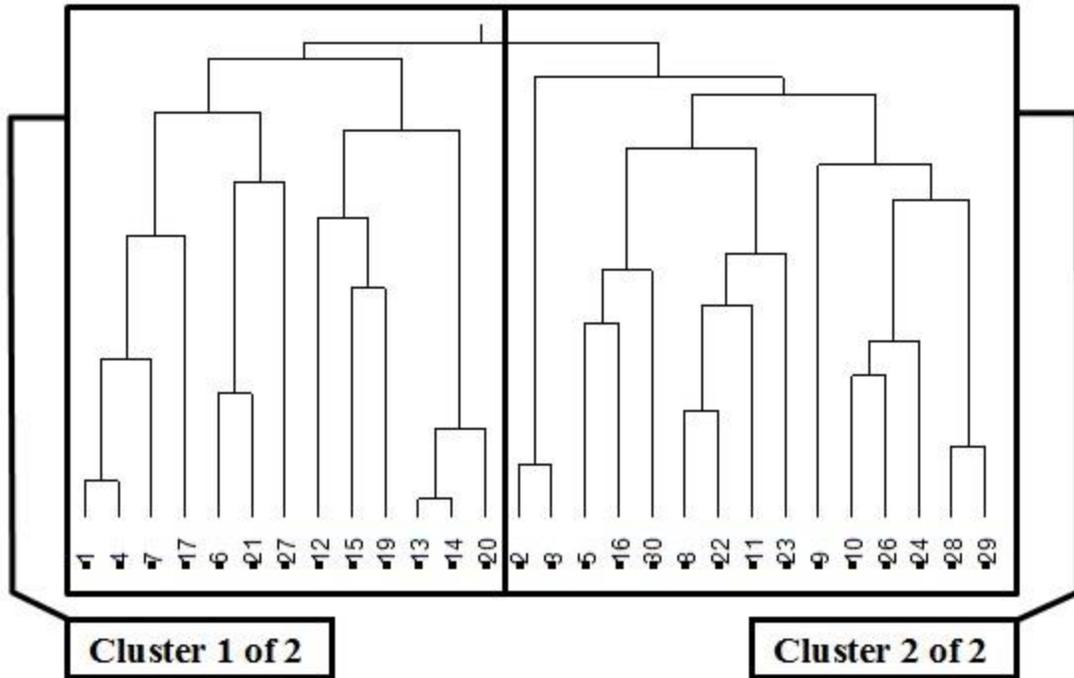


Fig. 18. Dendrogram of online survey hierarchical cluster analysis, displaying the two penultimate clusters. The numbers refer to the biosolids identification numbers (BS IDs, see Table 1).

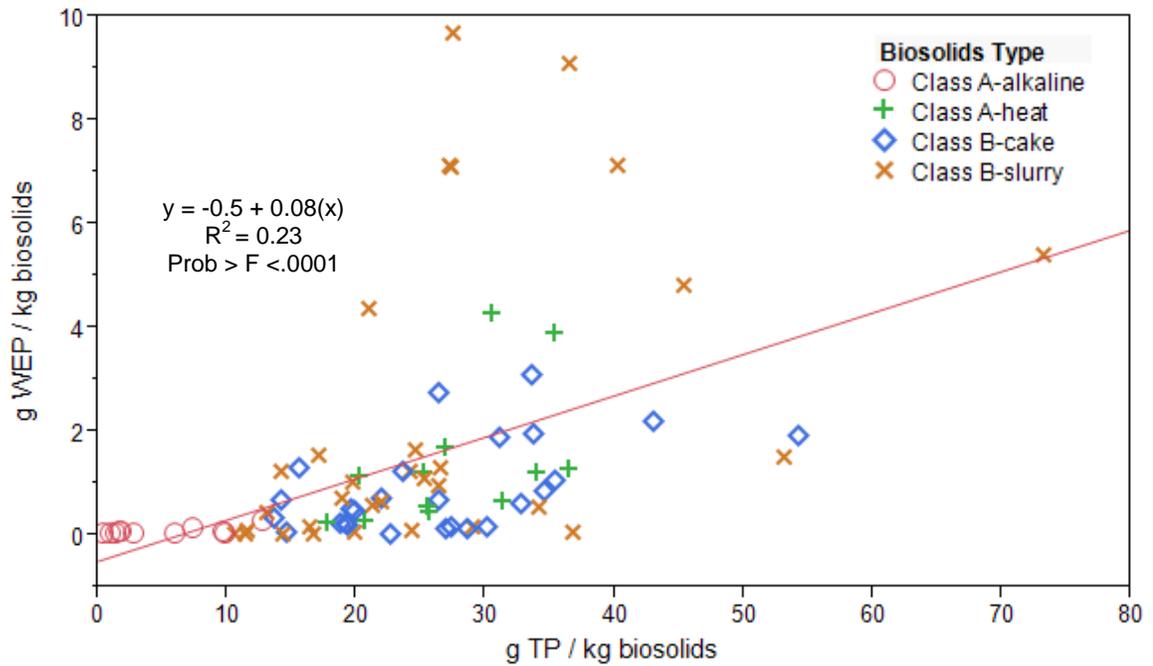


Fig. 19. Bivariate linear fit correlation for biosolids water-extractable P by total P (WEP by TP); seasons combined with markers for Class A alkaline stabilization (Class A-alkaline), Class A thermal heat drying (Class A-heat), Class B consistency of slurry (Class B-slurry), and Class B consistency of cake (Class B-cake).

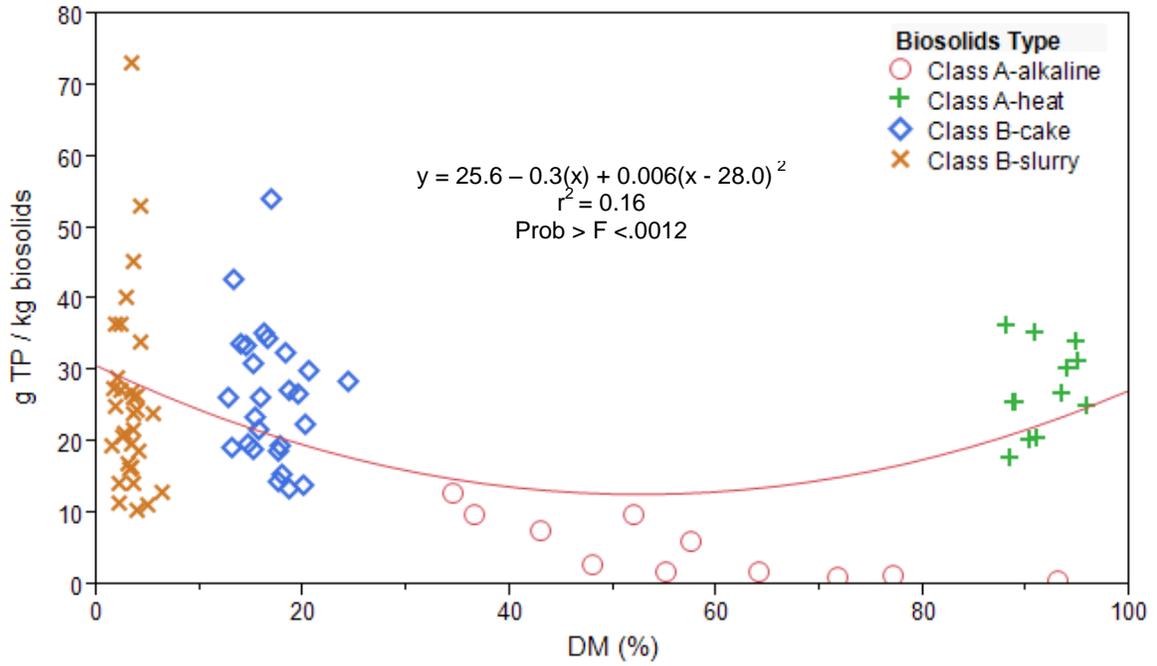


Fig. 20. Bivariate quadratic regression for biosolids total P by percent dry matter (PWEF by DM, %); seasons combined with markers for Class A alkaline stabilization (Class A-alkaline), Class A thermal heat drying (Class A-heat), Class B consistency of slurry (Class B-slurry), and Class B consistency of cake (Class B-cake).

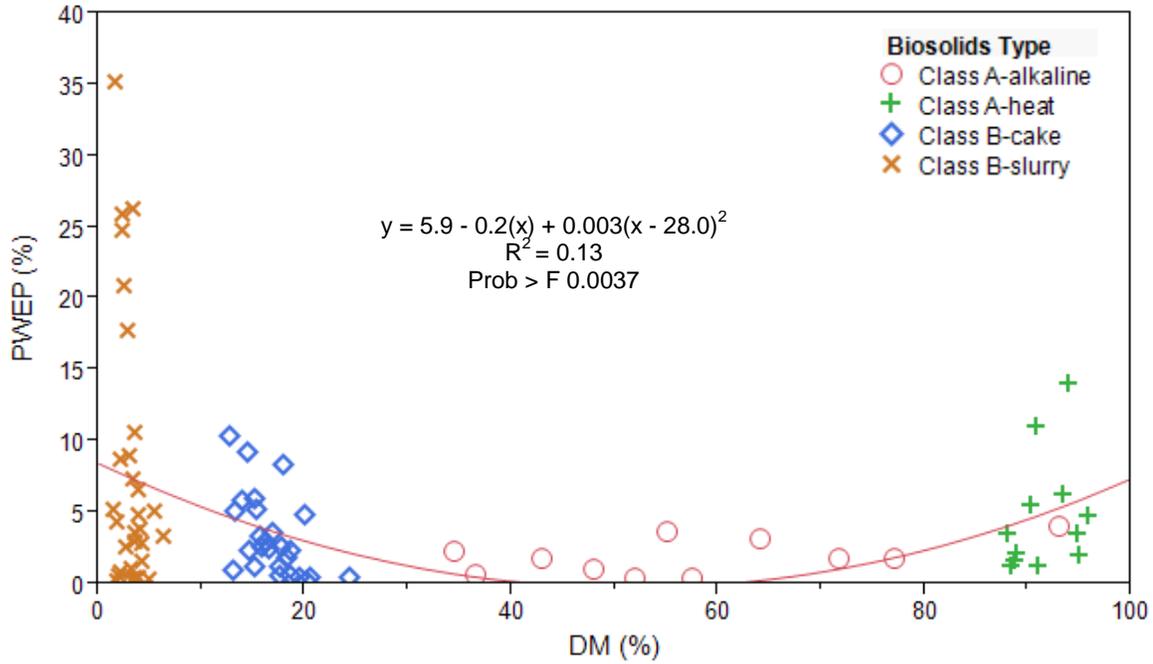


Fig. 21. Bivariate quadratic regression for biosolids percent water-extractable P by percent dry matter (PWE by DM, %); seasons combined with markers for Class A alkaline stabilization (Class A-alkaline), Class A thermal heat drying (Class A-heat), Class B consistency of slurry (Class B-slurry), and Class B consistency of cake (Class B-cake).

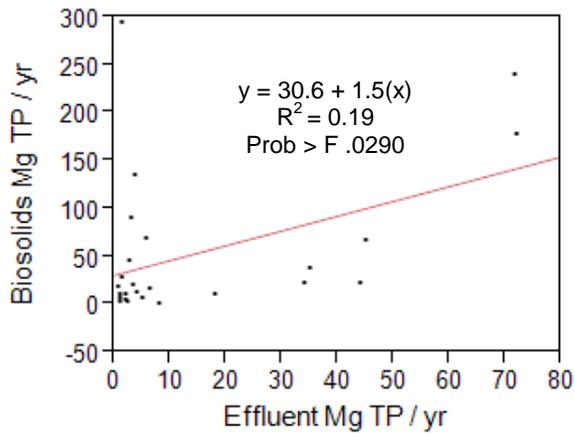


Fig. 22. Bivariate linear fit correlation for average of Summer 2013, Fall 2013, and Winter 2014 biosolids Mg TP year⁻¹ by average of 2013 and 2014 effluent Mg TP year⁻¹.

APPENDIX

Table 1. Online survey questions.

Name of respondent:

Please specify: _____

Title of respondent:

Please specify: _____

Name of facility:

Please specify: _____

Does your facility produce biosolids?

- Yes
 No

(If No Is Selected, Then Skip To End of Survey)

Are the biosolids produced by your facility land applied?

- Yes
 No

(If No Is Selected, Then Skip To End of Survey)

C1) Please check all chemicals you feed in the collection system or prior to Preliminary Treatment (and specify any chemicals not listed):

- acetic acid
 activated carbon
 aluminum sulfate, (alum)
 calcium/sodium nitrate
 chlorine
 chlorine dioxide
 ferric chloride
 ferric sulfate
 ferrous sulfate
 glycerine
 hydrogen peroxide
 lime
 magnesium hydroxide
 methanol
 ozone
 phosphates
 polymers
 rosin amine salts
 sodium hydroxide
 sugar water
 sulfuric acid
 Other, please specify: _____
 No chemicals added

Table 1. Continued

I) Do you measure Total Phosphorus in the influent?

- Yes
- No

II) Please check all processes performed in preliminary treatment (and specify any processes not listed):

- bar screen(s)
- grinder(s)
- grit chamber(s)
- Other, please specify: _____

III) Please check all processes performed in primary treatment (and specify any processes not listed):

- activated primary clarifier (fermentation)
- chemical feeder(s)
- chemical scrubbing
- flash mixing unit(s)
- flocculation tank(s)
- sedimentation tank(s)
- Other, please specify: _____

C2) Please check all chemicals added in primary treatment (and specify any chemicals not listed): (Refer to "C1)" chemical list)

IV) Please check all processes done in secondary treatment (and specify any process not listed):

- activated sludge
- activated sludge with biological nutrient removal
- intermittent sand filter(s)
- lagoon(s)
- rotating biological contactors (RBC)
- stabilization pond(s)
- trickling filters
- Other, please specify: _____

(Answer If) Please check all processes done in secondary treatment (a... biological nutrient removal Is Selected)

IVa) What type of biological nutrient removal is used? (please specify if not listed)

- nitrification but no denitrification
- nitrification and denitrification
- phosphorus removal only
- phosphorus and nitrogen removal
- Other, please specify: _____

C3) Please check all chemicals added in secondary treatment (and specify any chemicals not listed): (Refer to "C1)" chemical list)

V) Is there a final clarifier in secondary treatment?

- Yes
- No

(Answer If Yes Is Selected) Is there a final clarifier in secondary treatment?

Table 1. Continued

C4) Please check all chemicals added in the final clarifier (and specify any chemicals not listed): (Refer to "C1)" chemical list)

VI) Is there a tertiary filter?

- Yes
- No

(Answer If Yes Is Selected) Is there a tertiary filter?

C5) Please check all chemicals added in tertiary filter (and specify any chemicals not listed): (Refer to "C1)" chemical list)

(Answer If Yes Is Selected) Is there a tertiary filter?

- influent or primary treatment
- Other, please specify: _____

(Answer If Yes Is Selected) Is there a tertiary filter?

C6) Please check all chemicals added to filter backwash (and specify any chemicals not listed): (Refer to "C1)" chemical list)

VIII) Do you measure Total Phosphorus in the effluent?

- Yes
- No

IX) Please check all processes performed in sludge thickening before stabilization (and specify any processes not listed):

- centrifugation
- dissolved air floatation (DAF)
- gravity
- gravity belt thickener(s)
- Other, please specify: _____

C7) Please check all chemicals added in sludge thickening (and specify any chemicals not listed): (Refer to "C1)" chemical list)

IXa) Where does the liquid from sludge thickening process go?

- back to influent or primary treatment
- phosphorus recovery process
- another location, please specify: _____

C8) Please check all chemicals added to the liquid returned from sludge thickening process (and specify any chemicals not listed):
(Refer to "C1)" chemical list)

Table 1. Continued

- aerobic digestion
- air drying
- alkaline stabilization
- anaerobic digestion
- beta ray irradiation
- blending
- composting
- gamma ray irradiation
- heat drying
- incineration
- lime stabilization
- pasteurization
- thermophilic aerobic digestion
- wet air oxidation
- Other, please specify: _____

C9) Please check all chemicals used in stabilization process (and specify any chemicals not listed): (Refer to "C1)" chemical list)

Xa) Where does the liquid from stabilization process go?

C10) Please check all chemicals added to the liquid returned from stabilization process (and specify any chemicals not listed):
(Refer to "C1)" chemical list)

XI) Please check all processes performed in dewatering sludge (and specify any processes not listed):

- belt press(es)
- centrifugation
- drying bed(s)
- pressure filtration
- rotary press(es)
- sludge lagoon(s)
- vacuum filtration
- Other, please specify: _____

C11) Please check all chemicals added in dewatering process (and specify any chemicals not listed): (Refer to "C1)" chemical list)

XIa) Where does the liquid from dewatering process go?

C12) Please check all chemicals added to the liquid returned from the dewatering process (and specify any chemicals not listed):
(Refer to "C1)" chemical list)

XII) What class of biosolids are produced?

- class A
- class B

XIIa) What are your most recent dry tons annual biosolids production figures?

- class A quantity, please specify: _____
- class B quantity, please specify: _____

(Answer If class A Is Selected) XII) What class of biosolids are produced?

Table 1. Continued

XIIb) What is the consistency of the class A biosolids produced?

- cake
- liquid
- pellet
- slurry
- Other, please specify: _____

(Answer If class B Is Selected) XII) What class of biosolids are produced?

XIIb) What is the consistency of the class B biosolids produced?

- cake
- liquid
- pellet
- slurry
- Other, please specify: _____

XIIc) Do you conduct phosphorous analysis on the final biosolids product?

- Yes
- No

XIII) Is there phosphorus recovery from sludge or sidestreams?

- Yes
- No

(Answer If Yes Is Selected) Is there phosphorus recovery from sludge or sidestreams?

XIIIa) What is the phosphorus recovery process?

- Please specify: _____
-

Table 2. Online survey with Two Cluster Model percentage differential.

Online Survey Positive Responses	Survey Total	Cluster 1	Cluster 2	Differential
	-----%-----			
Sludge thickening -No chemicals added	32.1	69.2	0.0	69.2
Tertiary filter? -no	35.7	69.2	6.7	62.6
Measure Total phosphorus in the influent? -no	28.6	61.5	0.0	61.5
Liquid from stabilization process -back to influent or Primary treatment	64.3	92.3	40.0	52.3
Sludge thickening before stabilization -gravity	35.7	61.5	13.3	48.2
Dewatering process -No chemicals added	28.6	53.8	6.7	47.2
Liquid returned from stabilization process -No chemicals added	82.1	100.0	66.7	33.3
Liquid returned from the dewatering process -No chemicals added	75.0	92.3	60.0	32.3
Secondary treatment -activated sludge	64.3	76.9	46.7	30.3
Consistency of the class B biosolids produced? -slurry	39.3	53.8	26.7	27.2
Liquid from dewatering process -back to influent or Primary treatment	82.1	92.3	73.3	19.0
Stabilization process -alkaline (lime)	21.4	30.8	13.3	17.4
Prior to Preliminary Treatment -chlorine	7.1	15.4	0.0	15.4
Preliminary treatment -drip degreaser(s)	7.1	15.4	0.0	15.4
Secondary treatment -Secondary clarifiers	7.1	15.4	0.0	15.4
Where does liquid from sludge thickening go? -to sludge stabilization	7.1	15.4	0.0	15.4
Secondary treatment -No chemicals added	32.1	38.5	26.7	11.8
What class of biosolids are produced -class A	32.1	38.5	26.7	11.8
Final clarifier -No chemicals added	78.6	84.6	73.3	11.3
Stabilization -No chemicals added	71.4	76.9	66.7	10.3
Sludge thickening before stabilization -decanting water from aerobic digester	10.7	15.4	6.7	8.7
Dewatering sludge -gravity belt thickening	10.7	15.4	6.7	8.7
Consistency of the class A biosolids produced? -pellet	10.7	15.4	6.7	8.7
Consistency of the class A biosolids produced? -dry cake	10.7	15.4	6.7	8.7

Table 2. Continued

Online Survey Positive Responses	Survey Total	Cluster 1	Cluster 2	Differential
	-----%-----			
Prior to Preliminary Treatment -lime	3.6	7.7	0.0	7.7
Prior to Preliminary Treatment -liquid oxygen	3.6	7.7	0.0	7.7
Prior to Preliminary Treatment -sulfur dioxide	3.6	7.7	0.0	7.7
Primary treatment -No Primary treatment	3.6	7.7	0.0	7.7
Primary treatment -sodium hydroxide	3.6	7.7	0.0	7.7
Secondary treatment -chlorine	3.6	7.7	0.0	7.7
Secondary treatment -sodium hypochlorite	3.6	7.7	0.0	7.7
Secondary treatment -sulfur dioxide	3.6	7.7	0.0	7.7
Final clarifier in Secondary treatment? -no	3.6	7.7	0.0	7.7
Where does the filter backwash discharge? -filter not in operation	3.6	7.7	0.0	7.7
Sludge thickening -ferric chloride	3.6	7.7	0.0	7.7
Liquid returned from sludge thickening process -polymers	3.6	7.7	0.0	7.7
Stabilization process -air drying	3.6	7.7	0.0	7.7
Stabilization process -wet air oxidation	3.6	7.7	0.0	7.7
Dewatering sludge -sludge lagoon(s)	3.6	7.7	0.0	7.7
Liquid from dewatering process -liquid land application	3.6	7.7	0.0	7.7
Prior to Preliminary Treatment -No chemicals added	50.0	53.8	46.7	7.2
Stabilization process -aerobic digestion	50.0	53.8	46.7	7.2
Secondary treatment -lime	14.3	15.4	13.3	2.1
Primary treatment -lime	7.1	7.7	6.7	1.0
Sludge thickening before stabilization -centrifugation	7.1	7.7	6.7	1.0
Stabilization process -heat drying	7.1	7.7	6.7	1.0
Stabilization process -pasteurization	7.1	7.7	6.7	1.0
Dewatering sludge -drying bed(s)	7.1	7.7	6.7	1.0
Dewatering sludge -No dewatering sludge processes	7.1	7.7	6.7	1.0
Dewatering process -ferric chloride	7.1	7.7	6.7	1.0
Consistency of the class A biosolids produced? -cake	7.1	7.7	6.7	1.0

Table 2. Continued

Online Survey Positive Responses	Survey Total	Cluster 1	Cluster 2	Differential
	-----%-----			
Consistency of the class A biosolids produced? -dusty/powdery	7.1	7.7	6.7	1.0
Type of biological nutrient removal used -phosphorus and nitrogen removal	25.0	23.1	26.7	-3.6
Prior to Preliminary Treatment -calcium/sodium nitrate	17.9	15.4	20.0	-4.6
Stabilization -polymers	17.9	15.4	20.0	-4.6
Secondary treatment -polymers	10.7	7.7	13.3	-5.6
Liquid from stabilization process -leaves facility	10.7	7.7	13.3	-5.6
Liquid returned from the dewatering process -polymers	10.7	7.7	13.3	-5.6
Prior to Preliminary Treatment -hydrogen peroxide	3.6	0.0	6.7	-6.7
Prior to Preliminary Treatment -sodium hydroxide	3.6	0.0	6.7	-6.7
Prior to Preliminary Treatment -bioxide	3.6	0.0	6.7	-6.7
Primary treatment -biological odor control	3.6	0.0	6.7	-6.7
Primary treatment -sodium aluminate	3.6	0.0	6.7	-6.7
Secondary treatment -trickling filters	3.6	0.0	6.7	-6.7
Secondary treatment -denitrifying filter	3.6	0.0	6.7	-6.7
Type of biological nutrient removal is used -nitrification but no denitrification	3.6	0.0	6.7	-6.7
Secondary treatment -acetic acid	3.6	0.0	6.7	-6.7
Secondary treatment -activated carbon	3.6	0.0	6.7	-6.7
Secondary treatment -sugar water	3.6	0.0	6.7	-6.7
Secondary treatment -polyaluminum chloride	3.6	0.0	6.7	-6.7
Final clarifier -ferric chloride	3.6	0.0	6.7	-6.7
Final clarifier -sodium aluminate	3.6	0.0	6.7	-6.7
Final clarifier -polyaluminum chloride	3.6	0.0	6.7	-6.7
Liquid returned from sludge thickening process -sodium aluminate	3.6	0.0	6.7	-6.7
Stabilization -sodium bicarbonate	3.6	0.0	6.7	-6.7
Liquid from stabilization process -dewatering	3.6	0.0	6.7	-6.7
Liquid returned from stabilization process -sodium aluminate	3.6	0.0	6.7	-6.7

Table 2. Continued

Online Survey Positive Responses	Survey Total	Cluster 1	Cluster 2	Differential
	-----%-----			
Liquid returned from the dewatering process -ferric chloride	3.6	0.0	6.7	-6.7
Liquid returned from the dewatering process -ferrous sulfate	3.6	0.0	6.7	-6.7
Liquid returned from the dewatering process -sodium aluminate	3.6	0.0	6.7	-6.7
Liquid returned from sludge thickening -No chemicals added	89.3	84.6	93.3	-8.7
Type of biological nutrient removal is used -nitrification and denitrification	14.3	7.7	20.0	-12.3
Secondary treatment -aluminum sulfate, (alum)	14.3	7.7	20.0	-12.3
Secondary treatment -magnesium hydroxide	14.3	7.7	20.0	-12.3
Dewatering sludge -centrifugation	14.3	7.7	20.0	-12.3
Prior to Preliminary Treatment -ferric sulfate	7.1	0.0	13.3	-13.3
Prior to Preliminary Treatment -ferrous sulfate	7.1	0.0	13.3	-13.3
Prior to Preliminary Treatment -magnesium hydroxide	7.1	0.0	13.3	-13.3
Primary treatment -chemical scrubbing	7.1	0.0	13.3	-13.3
Primary treatment -conventional Primary clarifier	7.1	0.0	13.3	-13.3
Tertiary filter -methanol	7.1	0.0	13.3	-13.3
Tertiary filter -sugar water	7.1	0.0	13.3	-13.3
Liquid from dewatering process -no liquid produced	7.1	0.0	13.3	-13.3
Preliminary treatment -grit chamber(s)	92.9	84.6	100.0	-15.4
Where does liquid from sludge thickening go? -back to influent	92.9	84.6	100.0	-15.4
What class of biosolids are produced? -class B	71.4	61.5	80.0	-18.5
Preliminary treatment -grinder(s)	10.7	0.0	20.0	-20.0
Liquid returned from stabilization process -no liquid is returned	10.7	0.0	20.0	-20.0
Dewatering sludge -belt press(es)	50.0	38.5	60.0	-21.5
Secondary treatment -activated sludge with biological nutrient removal	42.9	30.8	53.3	-22.6
Primary treatment -No chemicals added	75.0	61.5	86.7	-25.1
Secondary treatment -methanol	14.3	0.0	26.7	-26.7
Dewatering process -polymers	60.7	46.2	73.3	-27.2

Table 2. Continued

Online Survey Positive Responses	Survey Total	Cluster 1	Cluster 2	Differential
	-----%-----			
Primary treatment -sedimentation tank(s)	39.3	23.1	53.3	-30.3
Stabilization process -anaerobic digestion	39.3	23.1	53.3	-30.3
Liquid from stabilization process -none produced	17.9	0.0	33.3	-33.3
Consistency of the class B biosolids produced? -cake	32.1	7.7	53.3	-45.6
Tertiary filter -No chemicals added	57.1	30.8	80.0	-49.2
Sludge thickening before stabilization -gravity belt thickener(s)	60.7	30.8	86.7	-55.9
Tertiary filter? -yes	64.3	30.8	93.3	-62.6
Filter backwash -No chemicals added	64.3	30.8	93.3	-62.6
Measure Total phosphorus in the influent? -yes	67.9	30.8	100.0	-69.2
Sludge thickening -polymers	67.9	30.8	100.0	-69.2
Where does filter backwash discharge? -influent	60.7	23.1	93.3	-70.3

Table 3. Raw water-extractable P, total P, and percent dry matter data of analytical replicates for each season (S, Summer 2013; F, Fall 2013; W, Winter 2014) organized by biosolids identification (ID) number.

BS ID	rep	Water-extractable P			Total P			Percent Dry Matter		
		S	F	W	S	F	W	S	F	W
		-----g kg ⁻¹ -----						-----%-----		
1	a	0.6	0.2	0.1	21.8	29.4	36.8	3.5	1.9	1.7
1	b	0.6	0.2	0.1	23.2	28.8	37.2	3.4	1.9	1.7
1	c	0.6	0.2	0.1	20.6	28.8	36.2	3.4	1.9	1.9
1	d	0.7	0.2	0.1	N/A	N/A	N/A	N/A	N/A	N/A
2	a	0.1	0.1	0.1	2.3	1.1	0.0	54.4	63.8	55.2
2	b	0.1	0.1	0.1	1.3	3.0	0.0	55.8	64.5	56.1
2	c	0.1	0.1	0.1	N/A	0.9	0.1	55.4	64.2	55.8
2	d	0.1	0.1	0.1	N/A	N/A	N/A	N/A	N/A	N/A
3	a	6.9	5.1	5.4	27.2	45.4	75.4	3.4	3.4	3.3
3	b	6.9	4.9	5.5	28.6	45.6	75.0	3.4	3.4	3.4
3	c	7.3	4.8	5.5	25.8	45.0	69.2	3.4	3.4	3.0
3	d	7.6	4.6	5.4	N/A	N/A	N/A	N/A	N/A	N/A
4	a	9.0	1.0	7.2	28.0	25.4	41.2	1.6	3.5	2.7
4	b	9.3	0.9	7.2	27.2	26.6	40.0	1.6	3.5	2.7
4	c	9.8	1.0	7.1	27.2	27.0	39.6	1.6	3.5	2.8
4	d	10.6	1.0	7.2	N/A	N/A	N/A	N/A	N/A	N/A
5	a	0.5	N/A	1.2	13.6	N/A	23.6	6.5	N/A	5.4
5	b	0.5	N/A	1.3	13.9	N/A	24.0	6.1	N/A	5.4
5	c	0.4	N/A	1.3	11.5	N/A	24.6	6.2	N/A	5.4
5	d	0.4	N/A	1.2	N/A	N/A	N/A	N/A	N/A	N/A
6	a	1.3	1.0	1.1	14.8	19.5	26.0	2.0	1.4	1.7
6	b	1.2	1.1	1.1	15.0	20.2	26.2	2.0	1.4	1.7
6	c	1.3	1.1	1.1	12.9	19.3	23.4	2.0	1.5	1.7

Table 3. Continued

BS ID	rep	Water-extractable P			Total P			Percent Dry Matter		
		S	F	W	S	F	W	S	F	W
		-----g kg ⁻¹ -----						-----%-----		
6	d	1.2	1.1	1.1	N/A	N/A	N/A	N/A	N/A	N/A
7	a	0.2	0.05	0.1	17.0	16.4	24.0	3.5	3.4	3.5
7	b	0.1	0.05	0.1	16.9	17.0	24.4	2.8	3.4	3.5
7	c	0.2	0.04	0.1	15.2	16.5	24.2	2.8	3.2	3.5
7	d	0.2	0.04	0.1	N/A	N/A	N/A	N/A	N/A	N/A
8	a	0.1	0.05	0.1	10.6	13.7	19.7	3.9	3.5	3.2
8	b	0.0	0.05	0.1	10.9	14.5	20.0	3.8	3.5	3.2
8	c	0.0	0.04	0.1	10.2	14.8	19.6	3.8	3.5	3.3
8	d	0.1	0.05	0.1	N/A	N/A	N/A	N/A	N/A	N/A
9	a	0.1	0.04	0.1	12.0	23.4	26.6	17.7	20.2	23.5
9	b	0.1	0.1	0.1	14.2	22.8	26.0	17.4	19.9	25.4
9	c	0.1	0.1	0.1	17.3	21.8	33.2	17.6	20.1	23.9
9	d	0.1	0.1	0.1	N/A	N/A	N/A	N/A	N/A	N/A
10	a	0.6	0.7	1.0	19.2	25.8	34.4	17.7	15.6	16.0
10	b	0.5	0.7	1.0	20.4	26.4	36.2	17.5	16.1	16.1
10	c	0.5	0.7	0.9	19.0	26.8	35.6	17.7	15.9	16.3
10	d	0.5	0.6	1.5	N/A	N/A	N/A	N/A	N/A	N/A
11	a	1.3	0.5	1.5	25.8	34.2	52.2	3.9	3.9	4.1
11	b	1.3	0.6	1.6	27.6	34.4	53.6	3.9	4.2	4.1
11	c	1.3	0.6	1.6	26.2	33.8	53.4	3.9	4.4	4.1
11	d	1.3	0.6	1.5	N/A	N/A	N/A	N/A	N/A	N/A
12	a	0.3	0.3	0.5	17.9	20.6	24.0	88.4	91.0	89.1
12	b	0.2	0.2	0.4	18.6	20.4	26.0	88.3	90.9	89.1
12	c	0.2	0.3	0.4	17.0	20.8	26.8	88.4	91.0	87.7

Table 3. Continued

BS ID	rep	Water-extractable P			Total P			Percent Dry Matter		
		S	F	W	S	F	W	S	F	W
		-----g kg ⁻¹ -----						-----%-----		
12	d	0.2	0.3	0.4	N/A	N/A	N/A	N/A	N/A	N/A
13	a	0.02	0.02	0.02	N/A	1.7	0.8	92.3	72.4	76.5
13	b	0.02	0.02	0.02	N/A	0.6	1.3	93.0	71.2	77.5
13	c	0.02	0.02	0.02	0.4	0.6	1.9	93.8	71.5	77.2
13	d	0.02	0.02	0.03	N/A	N/A	N/A	N/A	N/A	N/A
14	a	1.3	1.4	3.5	15.8	23.0	33.8	18.0	15.1	14.5
14	b	1.3	1.2	3.0	16.5	23.8	33.0	18.0	15.2	14.4
14	c	1.3	1.3	3.0	14.2	24.0	33.8	17.8	15.3	14.4
14	d	1.3	1.2	2.9	N/A	N/A	N/A	N/A	N/A	N/A
15	a	0.1	0.1	0.3	7.8	7.0	14.5	43.5	37.1	34.2
15	b	0.1	0.1	0.3	7.4	11.4	12.3	42.8	36.1	34.8
15	c	0.1	0.1	0.3	7.1	10.9	11.3	42.4	36.4	34.3
15	d	0.1	0.1	0.2	N/A	N/A	N/A	N/A	N/A	N/A
16	a	1.2	0.6	1.3	20.0	25.4	36.6	90.4	88.9	88.2
16	b	1.1	0.5	1.3	21.2	25.0	36.6	90.3	88.9	88.2
16	c	1.1	0.6	1.3	19.6	26.0	36.0	90.3	89.0	87.7
16	d	1.2	0.6	1.3	N/A	N/A	N/A	N/A	N/A	N/A
17	a	4.4	7.0	9.2	20.6	27.0	36.2	2.4	2.3	2.4
17	b	4.3	7.3	9.1	21.2	27.6	36.8	2.4	2.3	2.4
17	c	4.4	7.2	9.1	21.0	27.4	36.6	2.4	2.3	2.2
17	d	4.4	7.0	9.0	N/A	N/A	N/A	N/A	N/A	N/A
19	a	0.03	0.02	0.04	4.1	6.2	8.2	47.2	49.1	52.7
19	b	0.03	0.02	0.04	N/A	3.7	11.3	47.5	57.6	51.3
19	c	0.03	0.02	0.04	1.9	5.6	10.0	49.1	66.1	51.7

Table 3. Continued

BS ID	rep	Water-extractable P			Total P			Percent Dry Matter		
		S	F	W	S	F	W	S	F	W
		-----g kg ⁻¹ -----						-----%-----		
19	d	0.03	0.02	0.04	N/A	N/A	N/A	N/A	N/A	N/A
20	a	1.2	0.6	1.2	24.4	31.2	33.2	95.9	95.3	94.7
20	b	1.3	0.6	1.3	19.7	32.2	34.8	95.9	94.9	94.7
20	c	1.2	0.7	1.2	31.4	30.6	34.0	95.9	94.9	94.8
20	d	1.2	0.6	1.2	N/A	N/A	N/A	N/A	N/A	N/A
21	a	0.1	0.04	0.7	10.5	11.2	21.4	2.1	4.8	2.7
21	b	0.1	0.04	0.6	N/A	11.9	20.6	2.0	5.0	2.6
21	c	0.1	0.04	0.6	12.6	10.8	21.8	2.0	4.9	2.6
21	d	0.1	0.04	0.5	N/A	N/A	N/A	N/A	N/A	N/A
22	a	0.7	0.3	0.8	14.1	13.5	21.6	19.8	18.5	15.6
22	b	0.7	0.3	0.7	11.4	13.5	22.2	20.4	18.7	15.7
22	c	0.6	0.3	0.7	16.9	13.6	22.0	19.8	18.5	15.7
22	d	0.7	0.4	0.7	N/A	N/A	N/A	N/A	N/A	N/A
23	a	0.1	0.2	N/A	24.6	26.8	N/A	19.3	18.1	N/A
23	b	0.1	0.2	N/A	21.4	27.2	N/A	19.4	18.8	N/A
23	c	0.1	0.1	N/A	34.6	28.0	N/A	19.3	18.5	N/A
23	d	0.2	0.2	N/A	N/A	N/A	N/A	N/A	N/A	N/A
24	a	2.7	1.9	2.8	24.4	33.6	43.0	12.6	15.1	13.1
24	b	2.8	1.6	2.2	21.2	34.0	42.8	12.6	13.0	13.3
24	c	2.8	2.2	1.7	33.4	33.6	43.0	12.5	13.6	13.2
24	d	2.7	2.2	2.0	N/A	N/A	N/A	N/A	N/A	N/A
26	a	2.0	0.8	2.0	28.0	34.8	54.2	15.2	16.7	17.4
26	b	2.0	0.8	1.9	25.2	34.0	52.2	15.0	16.5	16.0
26	c	1.8	0.9	2.1	39.8	34.8	56.2	14.9	16.5	17.1

Table 3. Continued

BS ID	rep	Water-extractable P			Total P			Percent Dry Matter		
		S	F	W	S	F	W	S	F	W
		-----g kg ⁻¹ -----						-----%-----		
26	d	1.7	0.9	1.7	N/A	N/A	N/A	N/A	N/A	N/A
27	a	0.7	1.5	1.7	17.8	17.4	26.4	4.1	2.9	3.7
27	b	0.7	1.6	1.7	15.2	16.8	23.8	4.0	2.9	3.7
27	c	0.7	1.6	1.6	23.4	17.2	23.6	4.0	2.9	3.8
27	d	0.8	1.5	1.6	N/A	N/A	N/A	N/A	N/A	N/A
28	a	0.4	0.2	0.7	20.2	19.5	32.0	14.9	13.0	18.4
28	b	0.5	0.2	0.6	17.3	19.3	32.8	14.6	12.9	18.2
28	c	0.6	0.2	0.6	21.8	19.2	33.2	13.9	13.0	17.9
28	d	0.5	0.2	0.6	N/A	N/A	N/A	N/A	N/A	N/A
29	a	0.2	0.2	0.1	18.6	19.1	29.8	17.4	14.9	20.8
29	b	0.2	0.3	0.2	15.9	18.9	30.6	17.5	15.0	20.3
29	c	0.3	0.3	0.2	21.6	19.1	30.0	17.5	15.0	20.1
29	d	0.2	0.2	0.2	N/A	N/A	N/A	N/A	N/A	N/A
30	a	1.7	3.9	3.7	27.0	30.6	34.6	93.4	93.8	90.9
30	b	1.7	4.3	4.0	27.0	30.6	36.4	93.3	93.9	90.9
30	c	1.6	4.4	4.1	26.8	30.2	35.0	93.4	93.9	90.8
30	d	1.7	4.4	3.9	N/A	N/A	N/A	N/A	N/A	N/A