USGS

WATER RESOURCES RESEARCH GRANT PROPOSAL

Title: Phosphorus contamination potential of groundwater associated with land application of domestic and animal waste products in Florida.

Focus Categories: Nonpoint Pollution (NPP), Water Quality (WQL), Hydrogeochemistry (HYDGEO).

Keywords: Waste Disposal, Leaching, Adsorption, Soil Chemistry, Sludge.

Duration: Mar 1, 2000 – Feb 28, 2001.

Federal Funds Requested: $ 17,380.00

Non-Federal (matching) funds pledged: $ 40,000.00

Principal investigator(s) name(s), university, city, and water resource institute:

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Congressional district of the university: 5

Statement of critical regional or state water problem, and need for research

Nonpoint source pollution, especially from agricultural sources, has been identified as the major source of nutrients to, and degrader of, aquatic systems in USA (USEPA, 1995). Phosphorus is one of the major nutrients limiting the productivity of aquatic ecosystems, and Florida's ecosystems are extremely sensitive to anthropogenic P loads (Reddy et al., 1999). The phosphorus status of one system (such as agricultural land) can significantly impact the phosphorus status in another (such as a surface water or groundwater body). For example, agricultural drainage water discharged from the Everglades Agricultural Area into the Water Conservation Areas of the Everglades has shifted oligotrophic wetland areas into eutrophic conditions. Similarly, nutrient loads from the dairy industry on Lake Okeechobee seem to have shifted the lake toward hypereutrophic conditions (Reddy et al., 1999). Given the relatively high water table of the Florida aquifer systems and the uniqueness of Florida soils (sands) to allow significant P leaching (Harris et al., 1996), the groundwater bodies in several areas of the state are at risk of phosphorus contamination. This is particularly true in areas south of Orlando (the Lake Okeechobee basin), where soils with extremely low P retention capacity and high vulnerability to leaching surround P-sensitive water bodies.

On the other hand, agricultural lands need phosphorus for optimum crop production. One alternative to using the highly soluble (and leachable) P-fertilizers is the use of non-hazardous waste products, such as sewage sludge (hereafter referred to as biosolids), that can supply soluble phosphorus at a much slower rate, but in sufficient quantity to satisfy crop-P needs. Such a practice also promotes land application of non-hazardous wastes and the recycling of their nutrients. This is critical considering the fact that we are fast running out of other ways to dispose wastes. In Florida, pasturelands are promising recipients of domestic and animal wastes, considering their abundance in areas south of Orlando (the Lake Okeechobee basin). The major challenge lies in striking a balance between application of wastes (and maximizing agronomic benefits) while ensuring that the environmental quality of an ecosystem is not compromised. The primary concern in Florida is the potential eutrophication of aquatic regimes, groundwater in particular (caused by P leaching). At present, there is no P-based waste applications guideline in effect in the state of Florida. To develop one, a detailed and comprehensive study on the fate of phosphorus involving several typical Florida soils (including the Spodosols, one of the highly leachable, but most abundant soil types in the Lake Okeechobee basin) and a wide variety of biosolids and animal manures is needed. Leachability of phosphorus from waste materials and waste-amended soils as well as their sorption/desorption behavior as a function of soil/solution properties are matters of prime research interest.

Statement of results or benefits

The information derived from the proposed work will be of importance to state (e.g. Department of Environmental Protection) and local authorities (e.g. County governments in the Lake Okeechobee area) responsible for regulating land application of waste materials. Clearly, the widespread occurrence and land application potentials of domestic wastes and manures capable of supplying soluble phosphorus calls for a better understanding of the fate and transport of phosphorus in soils amended with these materials. This is particularly true in Florida considering the susceptibility of the water regimes to eutrophication caused by excess phosphorus. One of the areas that is most vulnerable in terms of phosphorus leaching potential is the Lake Okeechobee basin, south of Orlando, which hosts abundant pasturelands which are ideal settings for land application of wastes. Results obtained from this study will be important in developing best management practices to minimize non-point source P pollution, as well as in formulating environmentally safe P-based waste application guidelines. This study will also provide invaluable basic information on biogeochemical cycling of phosphorus in waste-amended soils (e.g., factors influencing P retention by sand). From a remedial viewpoint, such information will be of critical importance (e.g. methods to improve P retention by sand), as it will help improve remedial strategies and assessments of ecological risk driven by phosphorus.

Nature and Scope of Research

Beneficial reuse of biosolids and animal manures via land application is usually limited by nitrogen (N) considerations (Kaufman and Haith, 1986; Crohn, 1995). Application rates are determined by the N requirement of vegetation growing on a farm or watershed so as to minimize excess N available for leaching to ground waters. In most cases, applying biosolids and manures based on crop N needs simultaneously supplies excess phosphorus (P) to an ecosystem because the N:P ratio of both wastes is narrower than the N:P needs of the crops. This apparent excess of P could cause undesirable environmental effects, threatening surface or ground waters with eutrophication when the receiving waters are P-limited (McCoy et al., 1986). Two major environmental factors that govern the rate of application of wastes are the leachability and erosion potential of waste-borne phosphorus. Erosion and runoff related phosphorus contamination of surface water bodies are not major issues of concern in Florida due to its relatively flat topography. However, given the sandy texture (hence, low phosphorus retention capacity) of soils in Florida (particularly the pastureland soils south of Orlando in the Lake Okeechobee basin), application of biosolids and manures based on crop N needs may be risky in terms of groundwater phosphorus contamination potential.

Sound waste management in ecosystems or watersheds demands prudent consideration of P inputs (Pierzynski et al., 1994). We do know that (as with N) calculations based on total waste-P concentrations to determine allowable P loads to a soil can be grossly over conservative, as not all the (total) P may be soluble enough to leach. Such an overly conservative approach will result in very low allowable rates of biosolids (1-2 Mg/ha). This will likely severely reduce a farmer's interest in utilizing biosolids and a municipality's ability to dispose of a growing residual product. Phosphorus in biosolids and manures exists in a variety of forms (e.g. McCoy et al., 1986; Pierzynski et al., 1994) resulting in wide ranging solubilities (and hence, leachabilities). Inorganic P forms dominate both biosolids and manures (eg., Pierzynski et al., 1994; Sharpley, 1996), but the solubilities of P should be expected to vary with the waste. For example, McLaughlin (1984) reasoned that the availability of biosolids-P should depend on the residuals treatment processes used because they affect the forms of P in the biosolids. Pastene (1981) rightly warned that biosolids containing high levels of total P need not necessarily imply greater potential for P supply as had been suggested by Hinesly et al. (1976). This is especially true of biosolids produced through chemical treatment of wastewater for P removal. Pastene (1981) suggested that the molar ratio of (Al + Fe)/P is a good indication of the P supplying power of the biosolids: values <1 being characteristic of biosolids capable of supplying large quantities of soluble P. Soon and Bates (1982) found that biosolids treated with Ca salts yielded greater soluble, plant availabile P than Fe or Al treated biosolids. Given the interest in using Ca, Fe, and/or Al salts to treat lagooned manures or poultry bedding (Shrever et al., 1996), we should expect differential P solubilities and leachabilities in manures as well.

A further complication to predicting P leachabilities in biosolids or manure-amended soils is the P retention characteristics of the soil being amended. Barrow (1978) showed that phosphorus leaching is closely related to the retention and release properties of the soils. Different soils should be expected to convert soluble waste-P into soil forms (reaction products) of widely different solubilities. A given rate of soluble fertilizer-P results in different P solubilities and leaching tendencies in different soils, and reactions of waste-borne P should be similarly complex. Further, the wastes themselves may alter soil properties [e.g. pH (O'Connor et al., 1986), adsorption capacity - Fe and Al oxide content - (Shrever et al., 1996, O'Connor and Sarkar, 1999), organic P mineralization rates (White, 1981), and perhaps other factors such as soluble salts, soluble organic ligands, competing ions, etc.] that can alter P retention, and hence, leachability. We cannot hope to study all the possible effects in detail, but we intend to measure several of the routine soil chemical properties in amended and unamended soils to correlate with P retention. We also intend to select soils for study based on an index developed by Rhue et al. (1994) that distinguishes soils on the basis of their apparent P retention capacities.

In January, 2000, we will begin to study the forms, bioavailabilities, and mineralization rates of P in biosolids, fertilizers, and animal manures in a greenhouse setting. The work will be funded through a grant of $312,000/2y (total) from the Water Environment Research Foundation (WERF). We plan to evaluate the aforementioned P attributes in 3 Florida soils amended with 13 waste materials and 1 commercial fertilizer. Such a detail-oriented study will, nevertheless, remain partly incomplete if we ignore the important issues of leachability and retention characteristics of P due to budgetary constraints that limit our ability to hire sufficient research personnel. The entire amount budgeted in this proposal will be spent on a graduate research assistant and a senior undergraduate major who will focus their efforts on P retention and leaching issues.

Objectives of Research

1. Determine the solubility and leachability of phosphorus in biosolids, manures, and biosolids/manure-amended soils. Compare and contrast waste-P behavior with phosphorus solubility and leachability in untreated and P-fertilized soils. Quantify the relationship between various measures of P solubility (e.g. soil test P, "operationally defined" soluble forms of P) and P leachability.

2. Determine phosphorus retention/release characteristics of biosolids and manure amended soils. Compare and contrast these characteristics with P retention characteristics of untreated and P-fertilized soils. Identify factors influencing P retention in waste-amended soils.

Timeline of Activities

<u>Year 1, Months 1-6</u>: Purchase supplies; obtain soils, wastes, and plants; finish literature search; recruit student assistants; begin characterization of soils and waste products; setup, begin and finish first growing season (half of biosolids and manures) in the green-house; collect and begin analysis of soil and leachate samples; begin sorption studies.

<u>Year 1, Months 7-12</u>: Finish leachate P analysis; finish sorption studies; begin and finish desorption studies (with half of the biosolids and manures); analyze data; prepare and submit annual report.

***Year 2, Months 1-6*:** Begin and finish second growing season (remaining biosolids and manures) in the greenhouse; collect and analyze soil and leachate samples; collect and begin analysis of soil and leachate samples; begin batch sorption and desorption studies with remaining biosolids and manures.

***Year 2, Months 7-12*:** Finish leachate P analysis; finish batch sorption and desorption studies; finish data analysis; submit final report, graduate thesis (1), undergraduate special project report (1) and journal articles (2); present data in state, regional, and national meetings; prepare extension publication; recommend future work.
Only Year 1 (March, 1999 – Feb, 2000) work has been included in the current proposal following the protocols outlined in the RFP. We plan to submit a supplementary proposal in November, 2000, requesting funding for Year 2.

Materials

Various types of biosolids, animal manures, and a commercial fertilizer will be studied. We plan to evaluate a minimum of 14 materials. An overview of the experimental design (subject to modification) is given in Table 1.
Biosolids will be chosen on the basis of:

1. Pathogen reduction techniques: Raw, Class B, Class A

2. Biosolids treatment processes: Class A materials treated with Fe/Al, polymer addition, alkalinization (N-Viro), and biological P removal

3. Physical nature of end product: Dewatered, dried (cake *vs*. heat dried).

Animal manures will be chosen on the basis of:

1. Types: Dairy, poultry, swine, beef

A commonly used commercial fertilizer (concentrated super phosphate, CSP) will be used for comparison purposes.

Soils will be chosen on the basis of their apparent P retention capacities (Rhue et al., 1994): Low, medium, and high.

Bahiagrass (a common pasture grass grown on > 1 million ha in Fl) will be the test crop. Pastures are a common setting for land application of biosolids and manures because of large acreage, high N requirement, and isolated settings. Bahiagrass has P needs similar to most crops.

TABLE 1
Experimental Design [a]

Materials	**Soils**	**Rates**	**Reps**	**Total**
Raw, heat dried biosolid	3	2	3	18
Class B biosolid (cake)	3	2	3	18
Class B biosolid (heat dried)	3	2	3	18
Class A biosolid (heat dried)	3	2	3	18
Alkaline stabilized Class A biosolid (N-Viro)	3	2	3	18
Fe/Al-treated Class A biosolid	3	2	3	18
Polymer-treated Class A biosolid	3	2	3	18
Biosolid generated by biological P removal	3	2	3	18
Dairy manure	3	2	3	18
Poultry manure	3	2	3	18
Swine manure	3	2	3	18
Beef manure	3	2	3	18
Alkaline stabilized animal manure (N-Viro)	3	2	3	18
Concentrated Super Phosphate (CSP)	3	2	3	18
Controls	3	_	3	9
			Total	261

[a]Subject to modification based on materials availability and interim (year 1) results.

Methods, Procedures, and Facilities

Both greenhouse and laboratory studies will be conducted. We will begin with laboratory characterization of various biosolids and animal manures, focusing on P solubilities in each material (Objective 1) in addition to total elemental analyses using appropriate analytical procedures (EPA Publication SW-846, USEPA, 1986). Phosphorus solubilities will be determined using the first step of a sequential extraction procedure slightly modified from that used by Chang et al. (1983) to study P fractionation in biosolids-amended soils. We have successfully used the same technique to characterize P solubilities in three biosolids and two soils amended with biosolids (unpublished data). The characterization involves reacting material with 1 M KCl for 30 min, centrifuging, filtering, and measuring soluble reactive P (SRP) by the Murphy and Riley (1962) colorimetric procedure. KCl extractable P is defined as “soluble/exchangeable P” and is regarded as readily available to plants and leachable.

Additional biosolids- and manure-P characterization will involve extracts with water, citric acid (used for fertilizers, following Sikora and Mullins, 1995), and by Mehlich I solution (following Nelson et al., 1953, used successfully for acid soils and biosolids-amended soils in Florida). We hope that we can identify the most effective characterization of soluble (and hence, "leachable") biosolids- and manure-P.

We will also extract soils amended with biosolids, manures, and fertilizer using KCl and Mehlich I reagents. Soils, both freshly amended and equilibrated with amendments during 4 months of crop growth, will be extracted to suggest changes in soluble forms of P that could be expected in the field.

We will determine materials-P leachability in a greenhouse study, similar to that used in a related study (O'Connor and Villapando, 1998). The major objective will be to study the leaching characteristics of biosolids-borne P and manure-borne P in amended soils (including the Spodosols, one of the highly leachable, but most abundant soil types in the Lake Okeechobee basin) in comparison with controls. These studies will determine the relative impact of biosolids and animal manures on available P that can, when in excess, move into water. Each material (biosolids, manure, and fertilizer-CSP) will be mixed with each of the three soils (low, medium, and high P retention capacity) at 2 rates. Rates will be equivalent to 56 and 224 kg/ha, representing a medium "agronomic" rate of material (twice the crop P needs), and a high rate based on crop N needs. These rates were calculated using typical biosolids total N and P concentrations (~ 5 and 2%, respectively), and assuming that about 40% of the biosolids total N becomes available during the cropping period (O'Connor, 1997). Each treatment (approximately 14 materials, 3 soils, 2 rates) will be replicated 3 times in a randomized block design. Appropriate controls (no P) will also be included (Table 1).

All materials and nutrients will be mixed thoroughly with each soil and transferred to soil columns (PVC tubing 15 cm diameter and 45 cm long) constructed to contain 15 cm of treated soil over 25 cm of low P-sorbing sand and 2.5 cm of gravel. Each column will be fitted with a reservoir (PVC caps) equipped with a drainage hole and tubing to allow periodic sampling of leachate. Columns will be maintained at "field capacity" by periodic watering, and leached monthly with a uniform volume of water for each soil. Leachate volume and Soluble Reactive Phosphorus (SRP) will be measured (Objective 1). This water regime will mimic well-drained soil conditions subjected to periodic heavy rainfalls. A special blender is available that offers excellent mixing of small amounts (1-2% by weight) of amendments with large masses of soils. The blender also has a liquid addition "intensifier" for liquid amendment mixing. Bahiagrass (*Paspalum notatum Flugge.)* seed will be sown on the moistened columns, at several grams per pot to insure a thick and uniform grass coverage, usually within 2-4 weeks. Once a month, thereafter, grass will be harvested.

Soil samples from the original treatment mix (time zero) and from each column at season's end (time final) will be air-dried, and characterized for P solubility. In a recently concluded study, soluble-P values varied significantly with biosolids (anaerobically digested, dried, and pelletized) and fertilizer P sources. These values were consistent with the Mehlich-extractable P values of the materials (O'Connor and Sarkar, 1999). In the proposed study, extractable-P will be correlated with leachate P to quantify the relationship between various measures of P solubility and P leachability.

A similar approach will be adopted to fulfill objective (2). We will begin with laboratory characterization of P retention capacities of native soils. Traditional batch equilibration techniques will be utilized, in which a solution of known initial P concentration will be shaken with a given mass of soil for a specified time (Harris et al., 1996). Following shaking, the solution will be separated from the soil by centrifugation, filtered and analyzed for soluble P. The difference between P mass originally present and that remaining in solution after shaking will be assumed to have been sorbed. Desorption studies will be conducted immediately after the corresponding sorption study to determine the tendency of the soil to release retained phosphorus. The waste-equilibrated soil samples will be collected after the completion of the four-month cropping season and will be subjected to similar single-point P sorption studies. Differences in P sorption between native and treated soils will identify the effects of waste addition on soils. Factors influencing P retention in waste-amended soils will be identified. In a similar study involving two Florida soils and three heat-treated, pelletized biosolids, O'Connor and Sarkar (1999) noted significant influences of oxalate-extractable Fe and Al contents of amended soils on P retention. Phosphorus retention of capacity of soils was a direct function of their "reactive" Fe+Al contents. Biosolids with high Fe+Al content were able to increase the P retention capacity of the soils. The effect was more obvious in a soil with originally low P sorbing capacity, where biosolids addition resulted in a significant decrease in P mobility. The proposed investigation will allow us to study the effects of oxalate-extractable Fe+Al on P retention in a wider variety of soil-waste matrices. We also hope to study the relative effects of other factors on P retention, such as, changes in soil pH or ionic strength, presence of competing ions, organic ligands complexing Fe and Al, etc. associated with biosolids and manure applications. Appropriate desorption studies will be conducted to determine the possibility of leaching of retained P.

All column studies will be conducted in the greenhouses of the Soil and Water Science Department, University of Florida. These laboratory experiments will be conducted in the Soil Chemistry Lab (SCL) directed by Dr. O'Connor in the Soil and Water Science Department. Soil and water samples will be collected from the greenhouse using decontaminated collection equipment. Leachate samples will be collected, filtered, acidified, and stored in polyethelene or glass containers at 4^{o}C until analysis. Soil samples will be stored in air-tight polyethelene or glass containers until digestion. Soluble P concentrations will be measured by the colorimetric method of Murphy and Riley (1962) using a spectrophotometer at the SCL. The major ion analysis of samples will be done by inductively coupled plasma spectroscopy at UF's Analytical Research Lab (ARL) following standard protocols. Oxalate extractable Fe and Al will be measured using an atomic absorption spectrophotometer following the method of Sheldrick (1984). The pH and EC of water samples will be measured with an Accumet model 20 pH/conductivity meter. The QA/QC manual of the Soil Chemistry Lab will be followed to ensure proper quality control.

Related Research

There are numerous publications on fate of phosphorus in soil. There are relatively fewer (yet numerous) publications on fate of phosphorus in biosolids-amended soils. However, there are only a few published articles on P solubility, leachability, and retention in biosolids-amended soils of Florida. We are not aware of any research on biogeochemistry of phosphorus involving the specific soils and the array of biosolids (common to FL) we intend to use in our proposed project (Table 1). A research team at UF headed by Dr. O'Connor conducted one of the more recent studies that investigated phosphorus retention and leachability issues associated with land application of three exceptional quality, heat-treated, pelletized biosolids. Three typical Florida soils were used; at least one of them, the Spodosols (a major soil type in the Lake Okeechobee basin south of Orlando, the region of primary concern), will be used again in the proposed project. The major emphasis on this project, however, was on plant uptake (bioavailability) of biosolids-borne P; phosphorus leachability and retention were studied to obtain a more comprehensive understanding of P behavior in biosolids-amended soils. It was found that P leachability was a direct function of P retention capacity of the soils, which, in turn varied with the soil's "reactive" Fe+Al contents. In some cases, biosolids added sufficient Fe+Al oxides to the soils to enhance P retention capacity and decrease P mobility in soils. Peters and Basta (1996) made similar observations, as did Young et al. (1988). It needs to be mentioned, however, that due to time and budgetary constraints, the effect of only one type of biosolids applied to only one soil could be studied in detail. The pH of that soil did not vary much upon biosolids application; hence, the pH effect on P retention could not be comprehensively understood. O'Connor et al. (1986) observed significant pH effect in a biosolids-amended calcareous New Mexico soil. Leachability of P in biosolids-amended soils also depended upon the chemical forms of P; the KCl-extractable "exchangeable" P fraction was more susceptible to leaching; the NaOH-extractable "Fe/Al-bound" P fraction was resistant to leaching, and remained in the soil (Sarkar and O'Connor, submitted). Rydin (1996), however, showed that a significant portion of NaOH-extractable P may transform to inert phosphorus compounds by reacting with oxygenated rainwater; these inert phosphorus species are soluble and, hence, susceptible to leaching.

Fate of manure-borne phosphorus in soils has been of relatively recent interest compared to biosolids-borne P. Of particular interest is a study of phosphorus sorption capacities of wetland soils of Florida impacted by dairy effluent (Reddy et al., 1998). They found that, under aerobic conditions, about 87% of the P sorption variability could be accounted for by oxalate extractable Fe+Al; adding organic carbon to the regression improved the relationship by about 5%. Similar results were obtained by Nair et al. (1998) who studied dairy manure influences on P retention by a Florida soil. Application of manure added sufficient soluble P to exceed surface soil sorption capacities. This *temporarily retained* P had the potential to move downward through the soil profile through time; i.e. there is a possibility of groundwater contamination *via* P leaching. Soils in both studies were Spodosols of the Myakka/Immokalee series, a major soil type south of Orlando, the same soil type we intend to include in our proposed project. A detailed investigation, involving several other animal manures and possibly other soil types (typical to Florida), is necessary to fully understand the potential of groundwater pollution in Florida as a result of land application of manure. Though field studies (such as the ones cited above) are ultimately needed to fully characterize P biogeochemical processes, greenhouse studies offer the advantage of highly controlled environmental conditions and the opportunity to evaluate numerous treatments in a cost-effective manner. These studies are invaluable in providing the basic scientific information required to comprehensive understanding of the intricacies of an apparently simple process that could be later verified under field conditions. We believe that our proposed greenhouse study will be instrumental in enhancing our existing knowledge on P retention and release behavior in waste-amended soils of Florida.

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