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INCREASING THE PHOSPHORUS SORPTION CAPACITY OF SOUTHEASTERN COASTAL PLAIN SOILS USING WATER TREATMENT RESIDUALS

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Long-term animal manure applications to many sandy Coastal Plain soils has resulted in the accumulation of excess soil phosphorus (P). When soils contain excessive P concentrations, dissolved phosphorus (DP) can be desorbed with water and transported into streams and rivers via runoff and leaching. The addition of DP into nutrient-sensitive Coastal aquatic ecosystems can then stimulate aquatic weed and algae growth and accelerate eutrophication. Augmenting a soil's P sorption capacity using alum-based water treatment residuals (WTRs) may be a new chemical-based method for increasing the soil's capacity to retain P. Laboratory experiments were conducted to determine if WTRs mixed into Autryville (Loamy, siliceous, subactive, thermic Arenic Paleudults) and Norfolk (Fine-loamy, kaolinitic, thermic Typic Kandiudults) soils could significantly increase their P sorption capacities. Water treatment residuals were obtained on two different occasions from a North Carolina municipal surface water treatment facility. Both WTRs (G1 and G2) were composed of fine-sized river sediments that were flocculated with liquid alum [$Al_2(SO_4)_3$]. Phosphorus sorption isotherms were determined on the WTRs, the soils, and soil + WTR mixtures of 2.5, 5.0, 7.5, and 10.0% (w/w). The P sorption maximums (P_{max}) were determined from the linear form of the Langmuir equation. The P_{max} values for G1 and G2 (175 and 85 mg P g^{-1}, respectively) were significantly higher than the P_{max} values for the Autryville or Norfolk soils (<1.0 mg P g^{-1}). Mixing WTRs into soils increased their P_{max} values several-fold (between 1.7 to 8.5 mg P g^{-1}) relative to soils with no WTR addition. This experiment demonstrates the feasibility of using alum-based WTRs to increase a sandy soil's ability to sorb more P. Our results suggest that WTR incorporation into sandy soils has the potential to be a new chemical-based best management practice (BMP) for reducing off-site P transport. (Soil Science 2004;169:206–214)

Key words: Coastal Plain soils, phosphorus sorption, alum, water treatment residuals, best management practices.

LIVESTOCK production and manure generation in the eastern Coastal Plain region are frequently concentrated in specific geographic areas (Kellogg et al., 2000). Because transporting manure away from a production facility is expensive, manure is typically applied onto nearby fields (Sharpley, 1999). Many Coastal Plain soils contain excess soil P concentrations as a result of repeated manure applications onto the same fields (Sharpley and Halvorson, 1994; Barker and Zublena, 1995; Sims et al., 1998; Hansen et al., 2002). Coale (2000) reported that more than 70% of the soils used for manure disposal in the Maryland portion of the Chesapeake Bay region have high to excessive soil test P contents. Sims et

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al. (2000) reported that in Sussex Co., Delaware, which alone produces 230 million broilers and 52,000 head of swine per year, approximately 92% of soils were rated as optimum to excessive in soil test P. It is not uncommon for Coastal Plain topsoil's with a long-history of manure application to contain several hundred kg of plant-available P per ha (Sims et al., 1998; Novak et al., 2000). Soils with excess soil P concentrations are a water quality concern because even a small amount of P (1 to 2 kg ha^{-1} yr^{-1}) transported into lakes can accelerate eutrophication (Lennox et al., 1997; Sharpley and Rekolainen, 1997). Eutrophication increases the cost of purifying surface water and restricts water usage by recreational and industrial sectors.

Many of the sandy soils in the eastern Coastal Plain region used for manure disposal are highly weathered Entisols, Alfisols, and Ultisols. Extreme mineral weathering, erosion, and high temperatures in these soils have reduced the quantity of secondary minerals (e.g., metal oxides, and secondary phyllosilicates) and organic phases (humate complexed cations). Coarse-textured soils with these characteristics generally have lower P sorption values than finer textured soils (Sample et al., 1980; Harris et al., 1996; Hansen et al., 2002). In addition, when soils are P saturated, their ability to bind additional P declines substantially (Beauchemin et al., 1996; Sharpley et al., 2002; Whalen and Chang, 2002). This means that sandy soils with low P sorption capacities are less likely to retain P, and continual application of manure can accelerate off-site P transport. Recent reports have shown that off-site P transport, particularly by leaching, has occurred in soils located in Delaware (Mozaffari and Sims, 1994), Florida (Graetz and Nair, 1995), and North Carolina (King et al., 1990; Novak et al., 2000).

Because of environmental concerns and regulatory pressure to reduce P loadings to surface water systems, research is now focused on developing and evaluating BMPs that minimize off-site P transport. Current BMPs used to reduce P transport into surface water systems include conservation tillage, crop residue management, cover crops, buffer strips, runoff water impoundment, and riparian zones (Sharpley and Halvorson, 1994; Dosskey et al., 2002). These types of BMPs focus on physically reducing P losses through minimizing runoff. On the other hand, there are few chemical-based BMPs available to reduce P leaching and runoff.

Recent investigations have reported a decrease in runoff P losses (Daniels et al., 1999a; Codling et al., 2002; Dayton et al., 2003) and in soil extractable P concentrations (Peters and Basta, 1996; Basta and Storm, 1997) from manure-treated soils after applying chemical amendments. The chemical amendments used in these studies were WTRs, a byproduct produced during drinking water purification of ground and surface water sources. Typically, a flocculating agent such as alum or an Fe-salt is added to the untreated water to settle out sediments (residuals). The use of alum or Fe-salts causes the residuals to contain varying concentrations of amorphous Al- and Fe-oxides and hydroxides (ASCE, 1996; Dayton et al., 2003). Aluminum and Fe-oxides and hydroxides are particularly effective at binding P. Dayton et al. (2003) reported that the P_{max} sorption values for 21 examples of Al-based WTRs from Oklahoma ranged between 0.30 and 5.14 mg P kg^{-1}.

Water treatment municipalities in many southeastern states discard WTRs through landfill disposal or discharge into river systems. Both of these disposal mechanisms are expensive and can increase the costs of purifying drinking water. Instead of viewing WTRs as an expensive waste product, they could be marketed as a soil amendment to tie up excess soil P concentrations. This concept is particularly germane to livestock producers that apply manure to fields that are overloaded with soil P concentrations. If WTR additions to soils can increase their P sorption potential substantially, off-site P transport via runoff and leaching may be reduced, thereby potentially lowering P inputs to nutrient-impaired surface water systems. Before endorsing WTRs as a soil amendment to mitigate excess soil P concentrations, however, it is important that P sorption by alum-treated WTR material be quantified. Our objective is to determine the extent to which soil incorporation of alum-based WTRs would increase the P sorption capacity of sandy Coastal Plain soils.

MATERIALS AND METHODS

Collection of Soils and WTRs

Soils were collected from two locations in the middle Coastal Plain physiographic region of North Carolina (NC) and South Carolina (SC). The upland topography in this physiographic region is nearly level to gently sloping, but it also includes some side slopes along dissected, shallow stream valleys. The two soil series used were an Autryville soil from Duplin Co., NC, and a Norfolk soil from Florence Co., SC. Both of these soil

series are common to the middle Coastal Plain region and are used for row, vegetable crop, and pasture production. Surface horizons are dominated by sands, whereas subsurface horizons have a clay-rich argillic horizon. The parent materials for the Autryville and Norfolk soil series are marine and fluvial sediments (Daniels et al., 1999b). Both soil pedons are well drained, and both are located in forested-upland landscape positions.

At sampling time, the partially decomposed forest litter material (O horizon) was removed from the soil surface before the collection of the topsoil samples (A horizon, 0- to 15-cm deep). The soils were mixed, air-dried, and ground to pass a 2-mm sieve.

Based on significant variations between WTR P_{max} values as reported by Gallimore et al. (1999), two WTR batches were collected on different occasions from the same NC water treatment facility. Water treatment residuals were collected in June 2001 (G1) and in April 2002 (G2). This facility treats raw water from the Nuese River. In June 2001 and in April 2002, from 16.3 to 27.9 $\times$ 10^3 m^3 d^{-1} of raw water was treated. The raw water collected during June 2001 and April 2002 had a near neutral pH (between 6.4 to 7.3), was organic carbon enriched (6 to 10 mg C L^{-1}), and had turbidity values between 9 and 32 NTU (Nephelometric Turbidity Units). Because of the near neutral pH, no carbonates were used during the purification process. Turbidity levels in the raw water were decreased using activated carbon, potassium permanganate, and liquid alum. Alum concentrations typically between 120 and 176 mg L^{-1} were used during purification to meet acceptable organic carbon (2 to 4 mg L^{-1}) and turbidity (1 to 5 NTU) drinking water standards. After alum treatment, the sediments were allowed to flocculate for 1 to 2 weeks. Water treatment residuals were collected from a settling tank, air-dried, and ground to pass a 2-mm sieve.

Chemical Characterization of Soils and WTRs

The pH values of the soils and WTRs were measured using a 1:2 (v/v) solid to deionized H_2O ratio. The Clemson University Agricultural Service Laboratory performed cation exchange capacity (CEC) and exchangeable acidity (Ex. acidity) measurements using analytical methods (available at: http://www.clemson.edu/agsrlb/procedures2/interest.htm). Total organic carbon (TOC) in all samples was measured using a LECO CN2000 analyzer (St. Joseph, MI)[a]. Total P (TP) in all samples was extracted using the $CuSO_4$ + H_2SO_4 digestion method of Gallaher et al. (1976) and was quantified using the ascorbic acid method (Greenberg et al., 1992) with a Technicon Auto analyzer (Tarrytown, NY).

Because noncrystalline and poorly crystalline Al-, and Fe-oxides and hydroxides are important P binding agents, oxalate-extractable Al_{ox} and Fe_{ox} in the soils were determined using the acid ammonium oxalate (pH = 3) method of McKeague and Day (1993). The WTRs Al_{ox} and Fe_{ox} concentrations were determined using a modified method of McKeague and Day (1993) where 25 mL of reagent was used instead of 10 mL. Soils and WTRs were extracted after 4 h of shaking in the dark, and the extracts were centrifuged and filtered using a 0.45-μm nylon syringe filter. The concentrations of Al_{ox} and Fe_{ox} in the extracts were determined by a Thermo Jarrell Ash Enviro-2 inductively coupled plasma atomic emission spectrometer (Thermo-Optek Corp., Franklin, MA).

P Sorption by Soils, WTRs, and Mixtures

The inorganic-P sorption characteristics of the soils, WTRs, and soil + WTR mixtures were determined using batch equilibration techniques (Nair et al., 1984). Briefly, in triplicate 25-mL glass centrifuge tubes, 1 g of WTR was equilibrated with 10 mL of P solutions that ranged in concentrations from 5,000 to 30,000 mg P L^{-1}. The inorganic P solutions were made from KH_2PO_4 dissolved in 0.01 *M* $CaCl_2$. For the soil + WTR mixtures, 0, 2.5, 5.0, 7.5, and 10.0% (w/w) blends of WTR added to soil were prepared and equilibrated with P solutions containing concentrations that ranged from 50 to 2000 mg P L^{-1}. The tubes in all sorption experiments were shaken for 18 h, centrifuged, and the supernatant filtered through a 0.45-μm nylon filter. Phosphorus in the supernatant (equilibrium P conc.) was quantified using the colorimetric method of Murphy and Riley (1962). The P sorption isotherms were plotted using the mean quantity of P sorbed (Q, mg g^{-1}) and the mean P equilibrium concentration (C, mg L^{-1}). The sorption data were also plotted using the linear version of the Langmuir equation:

$$C/Q = (1/P_{max})(C) + 1/(k)(P_{max}) \quad (1)$$

where, P_{max} (mg g^{-1}) is the P sorption maxima or capacity, and k (L mg^{-1}) is a sorption constant relative to P binding energy (Olsen and Watanabe, 1957). A linear regression analysis was performed between C and C Q^{-1}. Previously sorbed

P (as TP) was subtracted from all isotherm values before plotting to obtain corrected P_{max} values.

Simple linear regression analysis was used on laboratory sorption data to extrapolate, at the field scale, relationships between the quantities of WTR applied and the average soil P_{max} values for the Autryville and Norfolk soils. An average P_{max} value was determined for each soil series by summing the P_{max} values obtained after additions of WTR G1 and G2. Next, the percent soil + WTR mixtures values used in the laboratory experiments were converted to metric tons of WTR applied per ha (t ha^{-1}) assuming a 0 to 15-cm soil depth having a bulk density value of 1.5 g cm^{-3}. These regressions were determined using SigmaStat version 3.0 software (SSPS Corp., Chicago, IL).

RESULTS AND DISCUSSION

Characterization of Soils and WTRs

The chemical characteristics of the soils and WTRs alone are presented in Table 1. Both soils are very strongly acidic, and have low CEC, TOC, and TP values. A high percentage of CEC sites in the Autryville and Norfolk soils are occupied by exchangeable acidic cations (H^+ and Al^{+3}) (92 and 75%, respectively). These chemical characteristics are common for forested, sandy, Coastal Plain topsoils that have not been tilled, limed, or used for manure disposal for an extended period of time (Novak and Bertsch, 1991; Novak et al., 2000). On the other hand, both WTRs have medium acidic pH values, and they also have higher CEC and Ex. acidity contents compared with the soils. The higher Ex. acidity values in the WTRs are caused by Al^{+3} dissociation from alum [$Al_2(SO_4)_3$] and by the electrostatic attraction of Al^{+3} to negatively charged exchange sites. Both WTRs also have higher TOC contents than the soils. This is caused by activated carbon additions during the water purification process. The activated carbon compounds likely sorbed to the WTRs surface through cation bridging with functional groups and van der Wal attractive forces (Stevenson, 1994). The high TP contents in both WTRs are caused by sorption of P forms from the raw Nuese River water during purification. The Nuese River, near Kingston, NC, is known to be enriched in both TP and *o*-P concentrations (annual means of 0.020 to 0.190 and 0.070 to 0.110 mg L^{-1}, respectively; U.S. Geological Survey, 1995).

The magnitude of Al_{ox} and Fe_{ox} concentrations between the WTRs and soils is quite different (Table 1). The WTRs have much higher Al_{ox} and Fe_{ox} concentrations than the sandy soils, with differences between the WTRs and soils greater than two orders of magnitude. These WTR samples contain much higher Al_{ox} and Fe_{ox} concentrations when compared with WTRs isolated from several Oklahoma water treatment facilities (Dayton et al., 2003). Dayton et al. (2003) reported a concentration range of 1.33 to 48.7 g kg^{-1} and 0.23 to 7.44 g kg^{-1} of Al_{ox} and Fe_{ox}, respectively, in the 21 WTRs from the Oklahoma facilities. The WTRs in this study have elevated Al_{ox} concentrations because of the high alum concentrations employed by the NC water treatment facility to purify turbid Nuese River water. We speculate that the WTRs have high Fe_{ox} contents because the Nuese River collected sediments that eroded from Piedmont soils known to be high in Fe-containing minerals and oxides (Daniels et al., 1999b).

P Sorption by Soils, WTRs, and Soil + WTR Mixtures

The Autryville and Norfolk soils without WTR addition had similar P sorption characteristics when P equilibrium concentrations were less than 700 mg P L^{-1} (C in Fig. 1). Differences in their P sorption characteristics were apparent when C values were greater than 700 mg P L^{-1}. Regression results from the linear form of the Langmuir equation (Fig. 1) had r^2 values of 0.97 and 0.84 for the Autryville and Norfolk soils, respectively. The slopes from these equations were

TABLE 1

Chemical properties of soils and water treatment residuals G1 and G2

Materials	pH	CEC	Ex. Acidity	TOC	TP	Al_{ox}	Fe_{ox}
		cmol kg^{-1}		g kg^{-1}			
Autryville	4.3	4.8	4.4	17	0.3	0.84	0.28
Norfolk	4.7	3.7	2.8	18	0.3	0.64	0.29
G1	5.9	9.7	7.6	182	1.9	113	21
G2	5.8	9.8	8.8	112	9.3	145	26

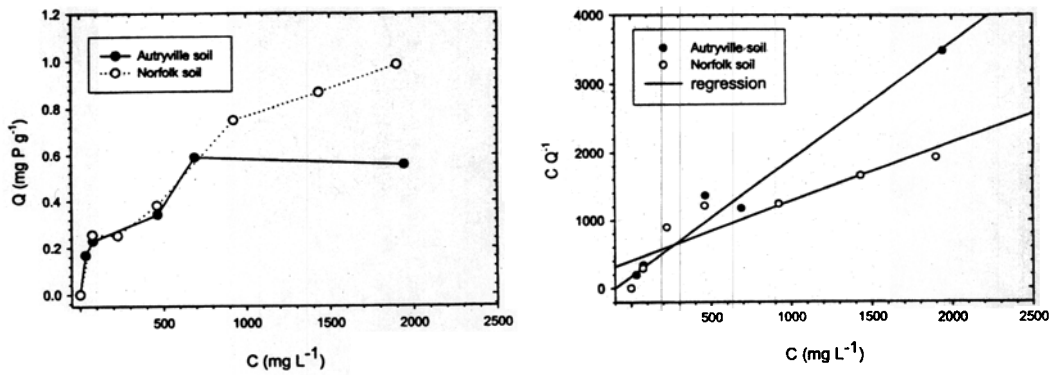

Fig. 1. Phosphorus sorption isotherms for Autryville and Norfolk soils without water treatment residual additions.

1.71 and 0.874 for Autryville and Norfolk soils, respectively. Using these slope values, the Autryville and Norfolk soils without WTR addition (Table 2 0% soil + WTR mixture) had P_{max} values of 0.60 and 0.80 mg P g^{-1}, respectively. Both of the soils P_{max} values are similar to values from three sandy Delaware soils (0.45 to 0.56 mg P g^{-1}, Mozaffari and Sims, 1994).

The alum-based WTRs have vastly different P sorption characteristics when compared with unamended soils. Although not plotted in Fig. 2, both WTRs sorbed 100% of the initial P in solution at concentrations between 5,000 and 10,000 mg P L^{-1}. Isotherms were conducted using solutions containing higher initial P concentrations (20,000 to 30,000 mg P L^{-1}) before suitable isotherm points were obtained for plotting (Fig. 2). Both WTRs were at near-P sorption equilibrium, although near-equilibrium conditions occurred at different C concentrations (Fig. 2, 2000 vs 7500 mg P L^{-1}). This is an important WTR characteristic to note because it foreshadows differences in their P sorption capacity. The linear form of the Langmuir equation fits the data sets very well (both $r^2 = 0.99, P < 0.001$). The slopes of the regression equations for the G1 and G2 were 0.0057 and 0.0118. These slopes correspond to P_{max} values of 175 and 85 mg P g^{-1}, respectively. The WTRs P_{max} values in this study are several times higher than P_{max} values reported for alum-based WTR produced in Oklahoma (0.30 to 5.14 g P kg^{-1}, Dayton, et al., 2003) and in Florida (5 g P kg^{-1}, O'Connor et al., 2002). The much higher P_{max} values for WTRs in this study were explained by their elevated Al_{ox} contents because Dayton et al. (2003) reported that WTRs P_{max} values were statistically correlated to their Al_{ox} content. The huge difference in P_{max} values between WTRs and unamended Coastal Plain soils exemplifies the P sorption potential of WTRs.

Both WTRs had significantly different P sorption isotherms (Fig. 2) and P_{max} values (Table 2), i.e., there is a 2-fold difference in P_{max} values between the WTRs. Heterogeneity in residuals P_{max} values is not uncommon considering that water treatment facilities will vary concentrations of alum or other flocculating agents (e.g., Ca or Fe cationic salts), depending on the quality of the raw water (Gallimore et al., 1999). The P_{max} variation between WTRs is also related to previously sorbed TP (Table 1 1.9 vs 9.25 g P kg^{-1}). Water

TABLE 2

Phosphorus sorption maxima (P_{max}) values for soils alone and soil mixed with water treatment residuals.

Soil	Residual	Percent soil + WTR mixture†				
		0	2.5	5.0	7.5	10.0
		P_{max} (mg P g^{-1})				
Autryville	G1	0.60	2.1	5.1	6.5	8.5
Autryville	G2	0.60	1.7	2.6	5.4	6.9
Norfolk	G1	0.80	2.5	5.0	7.3	8.3
Norfolk	G2	0.80	2.1	4.1	5.0	5.8

†A weight/weight mixture

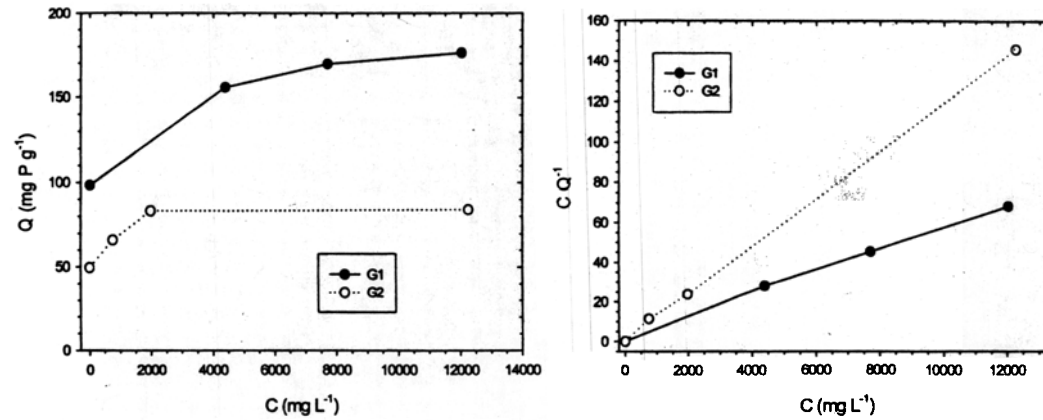

Fig. 2. Phosphorus sorption isotherm of water treatment residuals G1 and G2.

treatment residual G2 had much higher TP concentrations than G1, implying that there were fewer sites available for additional P binding.

Both WTRs were added to the Autryville and Norfolk soils to produce 2.5, 5.0, 7.5, and 10.0% (w/w) mixtures. The P sorption isotherms at these mixture ratios using G1 and Autryville and Norfolk soils are shown in Fig. 3. Mixing WTRs into these two soils resulted in higher amounts of P sorbed relative to unamended soils.

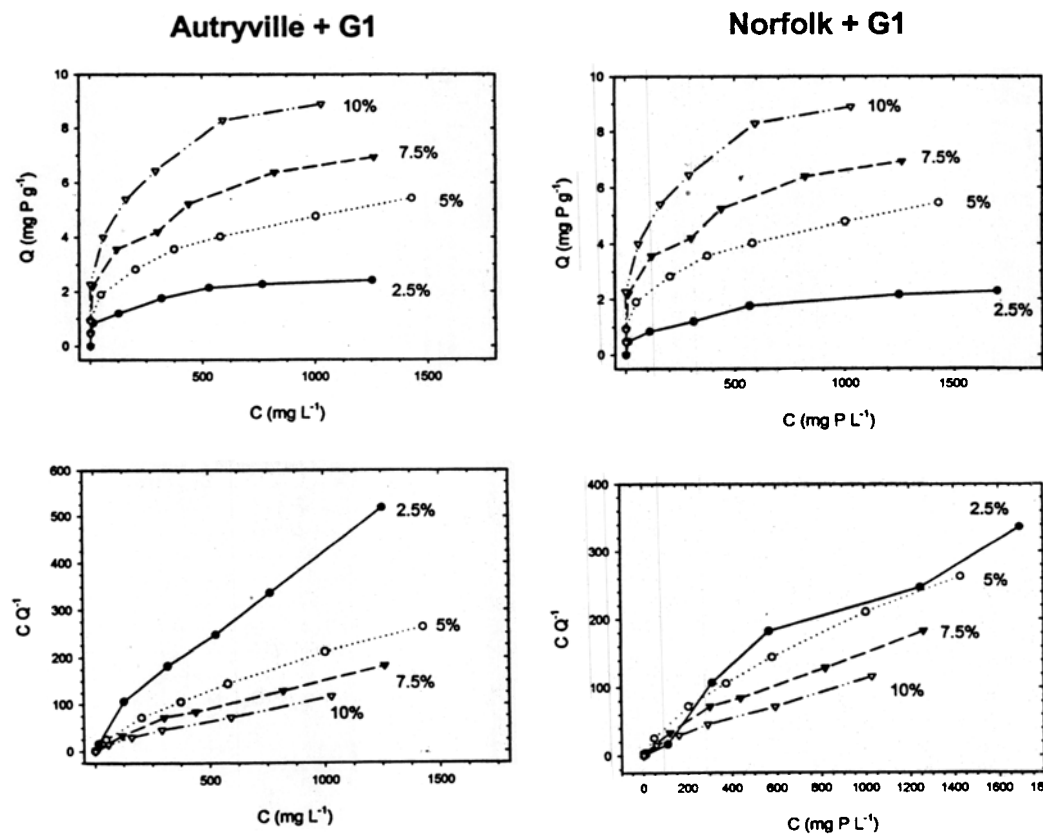

Fig. 3. Phosphorus sorption isotherms of G1 mixed into Autryville and Norfolk soils.

Regression of the linear form of the Langmuir equation for both of the soil + WTR mixtures fit the data very well (both r^2 values > 0.9; $P < 0.05$). The slopes of the regression equations were used to calculate the P_{max} values for the unamended soils (0% soil + WTR mixture) and the soil + WTR mixtures (Table 2). In all cases, adding WTRs to the Autryville and Norfolk soils increased their P_{max} values severalfold. For instance, unamended soils sorbed less than 1 mg P g^{-1}, whereas adding 10.0% WTRs to both soils increased P_{max} to between 5.8 and 8.3 mg P g^{-1}.

There were distinct dissimilarities between the soils P_{max} values after mixing in the two WTRs (Table 2). For instance, additions of G1 to both soils increased their P_{max} values to a slightly higher value than G2 (Table 2). This finding supports the conclusions of Gallimore et al., (1999) and Dayton et al. (2003) that the ability of WTRs to sorb P can vary among residual. Because of this P_{max} variation among WTRs, they probably should be screened using standard P sorption isotherms to determine their P binding potential.

The laboratory results show that augmenting WTRs into the Autryville and Norfolk soils caused a several-fold increase in their P_{max} values. To extrapolate the laboratory data to a field scale, regression analyses was used to determine relationships between amounts of WTRs incorporated (as metric tons WTR applied per ha (t ha^{-1}) and the average P_{max} values for both soil series (Fig. 4). The slopes from the regression equations for the Autryville and Norfolk soils were similar (0.0335 and 0.0316, respectively) and fit the data points very well (r^2 values between 0.98 and 0.99; $P < 0.05$). The regression data show that to increase soil P sorption from <1 to approximately 2 mg P g^{-1}, about 50 t ha^{-1} of WTRs applied to the soils will be required. To

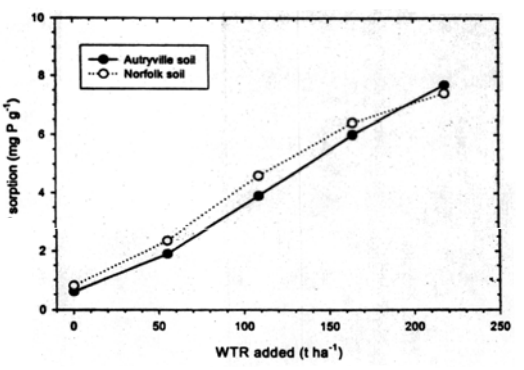

Fig. 4. Relationship between WTRs application and P sorption increase for the Autryville and Norfolk soils.

increase soil P sorption to higher values (>2 mg P g^{-1}), larger WTRs application rates will be required (between 60 and 240 t ha^{-1}). These large WTR application rates to soils may not be economically feasible because of high transportation and application costs. In addition, application of a few hundreds tons of WTRs could potentially reduce crop yields by binding up too much P, creating micronutrient imbalances, and/or increasing heavy metal concentrations that may be phytotoxic to sensitive crops. Prudent judgment must be used when extrapolating laboratory data to field situations. The laboratory P sorption data were collected under controlled conditions and did not account for some field environmental variables (alternating soil moisture conditions, rainfall, etc.) that can influence WTRs reactions with soil P. Additional field trials applying WTRs to soils containing excess P concentrations are needed to evaluate their potential for reducing off-site P transport fully. Nonetheless, the regression data imply that applying approximately 50 t ha^{-1} of WTR G2 to two sandy, Coastal Plain top soils can result in a doubling of the soils P sorption potential.

CONCLUSIONS

Although the ability of WTRs to reduce extracted soil P concentrations has been investigated previously, (Peters and Basta, 1996; Codling et al., 2002), this study evaluated the ability of WTRs to increase the soil's P_{max} values. Laboratory batch P sorption experiments showed that unamended Coastal Plain soils had P_{max} values of <1 mg P g^{-1}, and alum-based WTRs had P_{max} values between 85 and 175 mg P g^{-1}. Augmenting soils with WTRs resulted in a substantial increase in their P_{max} values to between 1.7 and 8.5 mg P g^{-1}. Based on laboratory extrapolation, approximately 50 t ha^{-1} of WTR applied to the Autryville and Norfolk soil series would result in an increase in their P_{max} value. The magnitude of the increase in a soils P_{max} value, however, depended on the P sorption effectiveness of the WTRs per se.

The data in this study suggest that application of alum-based WTRs to manure-treated soils could serve as a new chemical-based BMP. Applying alum-based WTRs to soils can increase their P sorption values, thereby, potentially reducing off-site P movement from fields via runoff and leaching. However, the magnitude of the soil P sorption increase will probably be dependent on the P binding effectiveness of the WTRs. Reducing off-site P transport may lower

P loads into nutrient sensitive surface water systems, thereby minimizing the occurrence of eutrophication.

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