

Aluminum-Containing Residuals Influence High-Phosphorus Soils and Runoff Water Quality

G. K. Haustein, T. C. Daniel,* D. M. Miller, P. A. Moore, Jr., and R. W. McNew

ABSTRACT

Phosphorus (P) loading in surface water can degrade water quality. Previous research has shown that soil test P levels are directly correlated to runoff P levels and that aluminum (Al) will bind P in the soil. Both water treatment residuals (WTR) and HiClay Alumina (HCA) are readily available waste materials high in Al. Water treatment residuals and HCA are by-products of the potable water treatment and commercial alum production process, respectively. Our objective was to determine if runoff P from fields excessively high in soil test P could be decreased by land applying these materials. Water treatment residuals and HCA were surface applied at rates of 0, 2.2, 9.0, and 18 Mg ha⁻¹ to plots high in P. We used rainfall simulation to produce runoff 1 d, 1 mo, and 4 mo following application. The P adsorption capacity for the WTR was 20 times higher than HCA because it was predominantly clay (95%) and contained three times as much Al. High rates of WTR increased the total recoverable Al concentrations in the soil, whereas HCA had no effect. High rates of both materials decreased Mehlich III soil test P levels due to the increased levels of soil Al. The two highest rates of WTR decreased runoff P levels significantly below those of the control plots for all dates, whereas the two highest rates of HCA decreased P levels for only the first two dates. Relative to the control, runoff concentrations of either total or dissolved Al were not significantly increased by WTR.

SOILS in areas of confined animal operations often contain excessive amounts of nutrients, particularly P, due to long-term overapplication of animal waste (Sims, 1993). When soil P levels are near the P adsorption capacity, much of the soil P is held only loosely by clay surfaces and is therefore readily available for transport in runoff water (Pote et al., 1996). While loss of P from these soils is of little concern from an economic point of view, P loading of surface waters can accelerate eutrophication, leading to algal blooms and growth of aquatic weeds.

Besides being unsightly and thus rendering surface waters unfit for recreation, these aquatic plants can clog water treatment filters and, upon their death and subsequent decomposition, cause fish kills due to decreased oxygen levels in the water. Some algae produce toxins that can result in death if ingested by animals (Sharpley et al., 1994). Fish kills have also resulted from outbreaks of the bacteria *Pfiesteria piscicida* (Kratch, 1997). These dinoflagellates cause lethal lesions on fish and have been linked to skin rashes, respiratory problems, and memory loss among people who come in contact with water containing these algae. Usually these microbes are not

toxic, but excessive P loadings into water bodies promote the production of the toxic form of *Pfiesteria* (Kratch, 1997). In the fall of 1997, after a particularly bad outbreak of *Pfiesteria* along the east coast, the popular press blamed the poultry producers for accelerated eutrophication of surface waters (Cohen, 1997).

With the importance of controlling runoff P having been established, it is important to understand the relationship between soil P and runoff P. The P fixation capacity of soils is positively correlated with, among other parameters, the Al content (Freese et al., 1992). It is well known that Al and orthophosphate ions interact strongly to form either stable surface complexes or insoluble Al phosphate minerals (Moore and Miller, 1994). In soils high in Al and iron oxides (e.g., oxisols), P deficiencies can occur because of these adsorption and precipitation reactions. Soils with excessive manure applications, however, may have insufficient Al to react with all the P, making it possible for water-soluble P levels to increase.

Pote et al. (1996) demonstrated that a linear relationship exists between soil test P and runoff P concentrations. Because of this, many states are considering establishing threshold soil P values above which no additional P may be added to the soil. In order to control nutrient runoff in high-P soil situations, loads must be decreased and/or the P adsorption capacity of the soil increased. An effective yet economical way to increase the P adsorption capacity of the soil (and therefore decrease runoff P) may be to apply high surface area, Al-bearing solid phase materials (Freese et al., 1992) contained in a waste product. For example, some WTRs and by-product residuals from HCA production contain large amounts of Al. As pointed out by other investigators (Elliott et al., 1990; Ippolito et al., 1999), these by-products are abundant, have limited disposal options (landfill), and appear to be useful as a soil amendment for reducing eutrophic runoff from soils high in P. By applying materials high in Al to soils that are high in P, it is anticipated that Al will increase the P adsorption capacity of the soil resulting in decreased runoff P levels.

Chemical characterization of WTR reveals that it is similar to natural soils, both in the concentration of metals such as Al and also in its trace element content (Fortenberry et al., 1994). It is composed mostly of coagulated aluminum compounds and materials such as sand, silt, clay, bacteria, and color-forming compounds removed from the raw water in the water treatment process. Unless the water source is grossly contaminated, the WTR has little potential for negatively affect-

G.K. Haustein, T.C. Daniel, and D.M. Miller, Crop, Soil, and Environ. Sci. Dep., 115 Plant Science, and R.W. McNew, Agric. Stat. Lab., 101A Agric. Annex, Univ. of Arkansas, Fayetteville, AR 72701. P.A. Moore, Jr., USDA-ARS, 115 Plant Science, Fayetteville, AR 72701. Received 13 Sept. 1999. *Corresponding author (tdaniel@comp.uark.edu).

Abbreviations: DP, dissolved phosphorus; HCA, HiClay Alumina; ICP-AES, inductively coupled plasma-atomic emission spectrometry; WTR, water treatment residuals.

ing the environment (Elliott and Singer, 1988). Since the composition of WTR varies depending on the content of the raw water source, all residuals from water treatment plants need to be analyzed completely before being applied to the land to ensure they do not contain levels of pollutants of environmental concern.

HiClay Alumina, a by-product of commercial alum production, consists of the bauxite impurities that did not react with sulfuric acid (H_2SO_4) used to produce alum. The elemental composition is similar to that of highly weathered soils, and its pH is acidic due to the addition of sulfuric acid (Barnett, 1996). Because Al is more soluble under acidic conditions, elevated soluble Al in the runoff is of concern (Sparks, 1996).

Aquatic organisms are very sensitive to elemental concentrations in water and, due to these concerns, it is necessary to examine how these materials will affect water quality when land applied. Because of the structure of their food chains, bioaccumulation of toxic elements can occur and eventually enter the human food stream. Adding to the importance of protecting runoff water quality is the growing dependence on surface water as a drinking water source (Forstner and Wittmann, 1981). Monitoring runoff concentrations of Al is especially important because both WTR and HCA contain high amounts of this element.

Much of the research dealing with the land application of WTR has centered around its effect on P availability (Cox et al., 1997; Heil and Barbarick, 1989; Ippolito et al., 1999; Lucas et al., 1994; Peters and Basta, 1996). Some of this research also included analysis for metals, but the metals were not found to negatively affect plant growth (Lucas et al., 1994; Peters and Basta, 1996; Rengasamy et al., 1980). We have found no research dealing with the runoff of metals from land receiving applications of WTR and HCA.

Applying these materials to land as a potential beneficial use option looks promising, but more research is needed to evaluate the effect of these materials on water and soil quality. Our research objectives were to characterize WTR and HCA with respect to key parameters, evaluate the effect of land application of WTR and HCA on total and dissolved runoff concentrations of Al and P, and to determine the effect of land application on important chemical properties of the soil.

MATERIALS AND METHODS

Residuals Characterization

We obtained the WTR from the Beaver Water District (Lowell, AR) and the HCA from a General Chemical alum production plant (Ashdown, AR). The Al content of WTR and HCA was determined by USEPA Methods 200.2 (total recoverable metals) and 200.7 (inductively coupled plasma) (USEPA, 1994). The calcium carbonate equivalent of the two wastes was determined as described by Johnson (1990). Percent organic matter was calculated using the modified Walkley-Black procedure (Donohue, 1983a), and pH and electrical conductivity were determined by electrode in a 1:2 soil-water solution (Donohue, 1983b). Percent solids were gravimetrically determined before and after drying at 105°C for 48 h. Qualitative clay mineralogical analysis was performed

using the methods described by Jackson (1956) and particle size analysis was performed according to the procedure described by Day (1956). Adsorption capacity of P was determined by the method described by Nair et al. (1984), using 0.03 M NaCl as opposed to 0.01 M $CaCl_2$.

Field Plots

The plots were located at the University of Arkansas Agricultural Experiment Station (Fayetteville, AR) on a Captina silt loam (fine-silty, siliceous, mesic Typic Fragiudult) soil. The surface soil had a pH of 5.0, approximately 1.3 g cm^{-3} bulk density, 11 g kg^{-1} organic matter content, and a particle-size distribution of 23% sand, 69% silt, and 8% clay (Pote et al., 1996). Individual plots were 1.5 × 3 m with a uniform slope of approximately 3% and installed borders to isolate runoff. On the downslope end of each plot, a trough channeled runoff water to a collection point and a silt plate existed to ensure a good interface between the soil and collection trough (Edwards and Daniel, 1993; Nichols et al., 1994; Pote et al., 1996).

Material Application

Both WTR and HCA were applied separately to plots cropped in fescue (*Festuca arundinacea* Schreb.) at loading rates of 0, 2.2, 9.0, and 18 Mg ha^{-1} (dry weight basis). Because preliminary data indicated that WTR would be more effective than HCA, a fifth rate (1.1 Mg ha^{-1}) of WTR was also applied. At approximately 8% solids (92% water), the WTR was applied "as is" to each 4.5- m^2 plot. The HCA contained 60% solids and required diluting to approximately 22% solids to achieve uniform distribution. Both materials (liquid form) were applied using a garden watering can fitted with a fan spout to aid in distribution. The materials visibly covered leaf surfaces, especially at the highest rate where 100 L of WTR was required to meet the highest application rate.

Runoff Collection and Analyses

Rainfall simulations similar to those described by Edwards et al. (1992) were used to generate runoff and were applied at an intensity of 65 mm h^{-1} . Runoff was generated in 1997 at 1 d (mid-May), 1 mo (mid-June), and 4 mo (mid-September) after WTR and HCA application. Twenty-four hours before each runoff collection, all plots were irrigated (low intensity) to normalize antecedent soil moisture.

After runoff began, rainfall simulations were conducted continuously to produce 30 min of continuous runoff. During runoff and at 5-min intervals, six 1-L samples were collected and used to create a flow-weighted composite sample. The time required to obtain each sample was noted for flow rate and runoff volume calculations. An aliquot of the runoff water was filtered (0.45 μm) in preparation for dissolved phosphorus (DP) and Al analyses. The filtered and unfiltered samples were then acidified in the field with concentrated HCl (one drop acid to 10 mL runoff) to extend sample holding time and stored at 5°C until analyzed. Total concentrations of Al and DP of the filtered and unfiltered runoff samples were determined using a nitric acid digest in combination with inductively coupled plasma-atomic emission spectrometry (ICP-AES) according to the American Public Health Association Method 3120 B (American Public Health Association, 1998; Sauer et al., 1999).

Soil Analyses

Plant-available soil test P levels (0–2.5 cm depth from all plots) fell within the high to excessive range (150 to 300 mg

P kg⁻¹ soil) as determined by Mehlich III extractions (Mehlich, 1984). Prior to each rain simulation, composite soil samples (0–2.5 cm deep) were taken for Al, P, and pH determinations. The composite samples were dried at 60°C for 48 h and passed through a 2-mm mesh screen. The soils were then digested according to USEPA Method 200.2 (USEPA, 1994) and analyzed for total recoverable Al via ICP–AES. Plant-available soil P was determined by Mehlich III extractions (Mehlich, 1984) and soil pH (pH_w, 1:2 slurry) was determined on the respective composite samples by electrode.

Statistical Methods

We used a randomized block with three replicates as the experimental design for this study with type of material applied and application rate as the factors. Data for each date were analyzed separately. The analysis of variance and correlations between parameters were conducted on all data at $p = 0.05$ and $p = 0.10$ (SAS Institute, 1988). All statistics in this paper are discussed at the $p = 0.10$ significance level, with the $p = 0.05$ and $p = 0.10$ LSDs being given in Tables 2 and 3.

RESULTS AND DISCUSSION

HiClay Alumina and Water Treatment Residuals Characterization

Table 1 presents the WTR and HCA characterization results. While both materials contained large amounts of Al, the WTR contained approximately three times more than the HCA, and, combined with the high clay content (98%), resulted in a P adsorption capacity 20 times higher than HCA's. Because HCA had undergone a dewatering process, the solids content between the two residuals also differed considerably; 8 and 59% solids for WTR and HCA, respectively. While the HCA had higher electrical conductivity than the WTR, the levels were well below those considered hazardous to most agronomic crops. The WTR had a higher percent organic matter, pH, and clay content. Both materials contained kaolinitic clay, while WTR also contained smectite, and HCA contained gibbsite. Different production practices between the two residuals can, in part, explain differences in the chemical and physical properties between the two residuals. Production of WTR involves addition of alum (aluminum sulfate) to large volumes of water containing suspended materials, whereas HCA

Table 1. Characterization of water treatment residual (WTR) and HiClay Alumina (HCA).

Parameter	WTR	HCA
Al,† g kg ⁻¹	46.7	15.9
Organic matter, %	3.75	2.54
pH	5.8	3.4
Electrical conductivity, dS m ⁻¹	0.04	0.20
Calcium carbonate equivalent, %	2.9	nd‡
Particle size distribution, %		
Sand	1	25
Silt	1	70
Clay	98	5
Dominant clay minerals	smectite, kaolinite	gibbsite, kaolinite
Solids, %	8	59
Phosphorus adsorption capacity, nmol P kg ⁻¹	1 750	86

† Total recoverable Al.

‡ Not detectable.

is produced from the addition of a harsh acid (sulfuric) to solid material. This explains why WTR would have a higher pH, percent organic matter, calcium carbonate equivalent, and clay content than HCA.

Runoff Concentrations

The sensitivity to various elemental concentrations in water is different for humans and aquatic organisms. Even among aquatic organisms, the acceptable concentrations of elements are highly variable according to species. Complicating the situation even more is the fact that acceptable concentrations of many elements are dependent upon the hardness or softness of the water (USEPA, 1986). Since there are no standards for P and Al in runoff, the principal approach used in evaluating the affect of the residuals on soil and runoff water quality was whether or not the elemental levels were significantly different from the control. Both total and dissolved concentrations were examined because of the importance of knowing whether the element was in solid or soluble form. The 1-d runoff event represents the worst-case scenario and the elemental concentrations given represent edge-of-field runoff loss. The reported runoff concentrations are probably higher than those anticipated to enter the surface water because of the application buffer zone required by most states, and it is assumed that runoff would traverse over land prior to reaching a stream or lake.

Phosphorus

Water Treatment Residuals. Since DP constituted at least 85% of total phosphorus (TP), and TP followed the same trends as DP for both materials, only the DP results are presented. At the first runoff event, the plots receiving the two lower application rates of WTR (1.1 and 2.2 Mg ha⁻¹) did not have runoff DP levels significantly different from the control (Fig. 1). However, the upper two application rates of WTR (9.0 and 18 Mg) significantly decreased DP concentrations relative to the control. After 1 mo, the plots receiving the lower two rates were significantly different from the control but still not significantly different from each other. For the other application rates, as rate increased, DP levels significantly decreased. At the 4-mo runoff event, the lower two rates (1.1 and 2.2 Mg ha⁻¹) were not significantly different than the control. However, the upper two rates (9 and 18 Mg) continued to have significantly lower DP levels than the control.

With the exception of the control, none of the rates had DP levels that changed over time. However, due to inherent field variability, the control plots had significantly (LSD = 0.30) lower DP levels at the 4-mo rain event than they did after 1 d. While not statistically significant, the plots receiving the two highest WTR rates (9.0 and 18 Mg ha⁻¹) had DP levels that decreased between the 1-d and 1-mo rains, but increased again at the 4-mo sampling (Fig. 1). The most probable explanation for the reductions in runoff P levels for the two highest rates of WTR is that the P adsorption capacity of the soil was increased immediately following application of the residual and continued to remain elevated

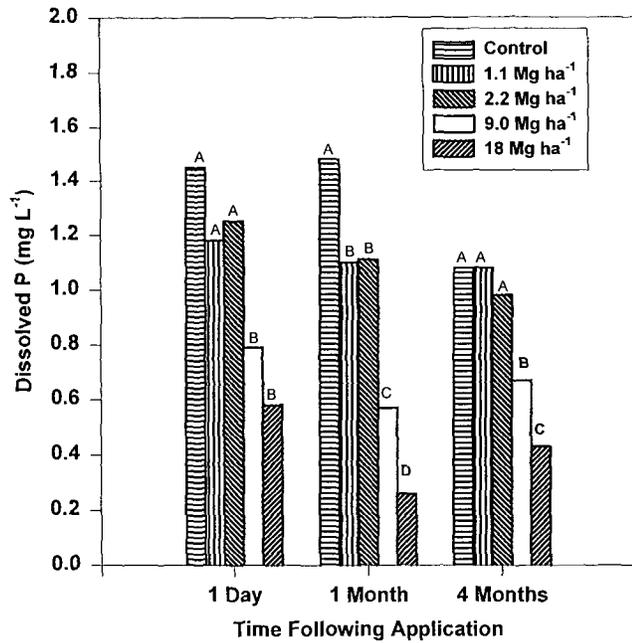


Fig. 1. Runoff dissolved phosphorus (DP) concentrations as a function of water treatment residual (WTR) application rate and time. Within each date, data with the same letter are not significantly different. To compare each rate across dates, use LSD = 0.30.

up to 4 mo following application. These results are consistent with WTR's high P-binding capacity (Table 1).

HiClay Alumina. At 1 d following application, the HCA plots showed a trend very similar to that of WTR: the lowest rate had levels of DP that were not significantly different from the control, but the upper two rates were significantly lower than the control, while not being significantly different from each other (Fig. 2). After 1 mo, the upper two rates were still lower than the control, but both had increased significantly (LSD = 0.30) relative to their respective 1-d runoff levels. The DP levels for the lowest HCA rate and the control did not change between the 1-d and 1-mo runoff events. After 4 mo, the DP levels in all treated plots were not significantly different from the control. It is unclear why the HCA was so effective at reducing DP initially, but then appeared to lose its effectiveness by 1 mo after application. Perhaps as early as 1 mo after application, the HCA's P adsorption capacity was being exceeded and by the fourth month (September), additional P was made available (re-equilibration of adsorbed soil P and mineralization of organic matter) that could be adsorbed by the WTR, but not the HCA (Pote et al., 1999). Also, the HCA may not be as stable as WTR, resulting in quicker breakdown and release over time, because by 4 mo the HCA was totally ineffective at reducing runoff DP levels.

Aluminum

Water Treatment Residuals. For all dates, no statistical differences in total Al were found compared with the control (data not shown). However, for the 1-d runoff event, a very clear trend was evident that application rate affected total Al concentrations. High amounts of total Al were found in the runoff for the two highest

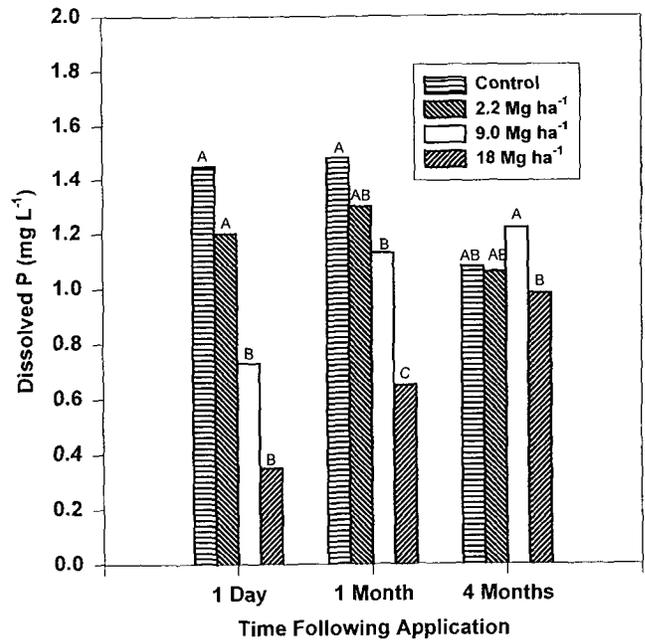


Fig. 2. Runoff dissolved phosphorus (DP) concentrations as a function of HiClay Alumina (HCA) application rate and time. Within each date, data with the same letter are not significantly different. To compare each rate across dates, use LSD = 0.30.

application rates. This is not surprising since WTR was found to contain almost 47 g Al kg⁻¹ (Table 1). But the fact that this effect was only seen at the 1-d event and not at the 1-mo and 4-mo rains suggests that the total Al detected arose from washing of the freshly applied material from the soil or plant surface. Measures to prevent this loss could be taken by applying the WTR to closely mowed or grazed grass when there was little chance of rain, not applying the materials at the higher rates, or incorporating these materials into the soil prior to cropping.

None of the treated plots for any of the sampling dates had dissolved Al runoff levels above that of the control (Table 2), probably because the pH was not low enough for Al to be soluble. Detected Al could easily be explained by erosion of the material, and since there are no water quality regulations set for Al concentrations (USEPA, 1986), runoff Al from land application of WTR is not seen as a potential water quality concern provided precautions are taken to minimize total Al loss during the first runoff event.

HiClay Alumina. Total and dissolved runoff Al concentrations from the HCA-treated plots followed the same trend as the WTR treatments: for all dates and rates, none of the treated plots had Al levels above that of the control (Table 2). Probably due to the erosion of the material during the first runoff event, high total Al concentrations were measured for the higher rates at the 1-d runoff event and a significant decrease in total Al concentrations occurred over time.

Soil Concentrations

Aluminum

Especially at the two highest rates, WTR increased the total recoverable Al in the soil 4 mo after applica-

Table 2. Mean concentrations of dissolved runoff Al from fescue plots following application of water treatment residual (WTR) and HiClay Alumina (HCA). LSD values are provided for comparisons within each row of data.

Parameter	Date	Material and rate applied (Mg ha ⁻¹)									LSD	
		Control	WTR					HCA			$\alpha = 0.10$	$\alpha = 0.05$
		0	1.1	2.2	9	18	2.2	9	18			
mg L ⁻¹												
Al	1 d	0.09	0.08	0.08	0.08	0.09	0.11	0.12	0.13	0.04*	0.05*	
	1 mo	0.20	0.15	0.18	0.14	0.11	0.13	0.15	0.14	0.07*	0.08*	
	4 mo	0.14	0.17	0.11	0.15	0.10	0.11	0.13	0.14	0.07	0.08	

* Significant differences between treatments were found using this LSD value.

tion, but none of the HCA-treated plots had Al concentrations significantly above the control (Table 3). For example, the Al concentration in the 18 Mg ha⁻¹ WTR rate was nearly twice that of the control but still within the range of normal soils. However, P availability could be decreased because of the additional Al, since Al is a known P adsorber (Freese et al., 1992). In our study, there was abundant soil P initially, so the adsorption of P to the extent of produce deficiency symptoms was not an issue. However, in areas already affected by low P levels, the application of these materials at the high rates would not be recommended.

Phosphorus

Since the Mehlich III soil test P levels in the plots fell within a broad range, and the effect of these materials on P was a main focus of this study, the soil test P levels for the 4-mo sampling were subtracted from the initial soil P concentrations for each plot. The difference (initial Mehlich III – Mehlich III 4 mo after application) in soil P levels 4 mo after application gives a better representation of the treatment effect on the soil P status and availability (Table 3).

There was a clear rate effect on soil test P, with the two highest WTR rates decreasing P levels significantly relative to the control (Table 3). HiClay Alumina had the same effect, but only at the highest rate (Table 3). A reduction in available P (Mehlich III) as a result of these high application rates was not surprising since the reduction in soil P closely followed the elevated total recoverable Al level in the soils for each rate. So, WTR at the upper two rates and HCA at the highest rate both significantly decreased the available P (Mehlich

III) levels in the soil. However, even at these high rates, the level of plant-available P remaining in the soil after 4 mo was twice that required for maximum crop production.

pH

After four mo the WTR did not significantly affect soil pH levels as compared with the control (Table 3). However, the HCA, with a pH of 3.4 (Table 1), tended to reduce soil pH below the control.

CONCLUSION

Residuals containing Al can reduce runoff concentrations of DP from high-P soils. However, the extent of the effect depends on the P adsorption properties of the residual as determined by the residual's content of Al and/or P adsorption capacity. Thus, appropriate characterization of the residual in question is the first step in evaluating a residual's potential effect on soil and runoff water quality. Generally, a higher Al content equals a greater P adsorption capacity and a greater reduction in runoff DP concentration. For example, the P adsorption capacity for the WTR was 20 times higher than HCA because it contained more clay and almost three times as much Al. High rates of WTR increased the total recoverable Al concentrations of the soil, while HCA had no effect. High rates of both materials clearly decreased Mehlich III soil test P levels due to the increased levels of soil Al. While both materials effectively reduced DP levels initially, the higher P-adsorption capacity of the WTR allowed the material to be effective longer.

Table 3. Selected properties of composite soil samples (0–2.5-cm depth) from fescue plots 4 mo after application of water treatment residuals (WTR) and HiClay Alumina (HCA). LSD values are provided for comparisons within each row of data.

Parameter	Date	Material and rate applied (Mg ha ⁻¹)									LSD	
		Control	WTR					HCA			$\alpha = 0.10$	$\alpha = 0.05$
		0	1.1	2.2	9	18	2.2	9	18			
mg kg ⁻¹												
Al†	4 mo	4380	4517	4465	5963	8353	4319	4815	5170	987*	1202*	
P‡	4 mo	-44.5‡	-36.9	-51.9	-93.6	-101.0	-66.3	-52.0	-92.4	-36.3*	-44.2*	
		169.7§	167.5	173.7	211.3	203.0	200.3	182.0	216.0			
		125.2¶	130.6	121.8	117.7	102.0	134.0	130.0	123.6			
pH	4 mo	5.53	5.43	5.60	5.67	5.50	5.53	5.37	5.27	0.14*	0.17*	

* Significant differences between treatments were found using this LSD value.

† Total recoverable Al in the composite soil sample.

‡ Reported as the decrease in Mehlich III soil P levels after 4 mo (initial Mehlich III – Mehlich III 4 mo after amendment application).

§ Initial Mehlich III.

¶ Mehlich III 4 mo after amendment application.

The majority (>85%) of the runoff P detected was in the form of DP, and in all cases the DP levels in the treated plots were less than or equal to that of the control. After 4 mo this trend continued for the WTR treated plots, but the HCA plots were statistically the same as the control. While not statistically significant, total Al levels for both materials tended to increase at the 1-d runoff event due to washing of the residual from the plant. Methods for minimizing this phenomenon include mowing the vegetation prior to application, not applying at high rates, and soil incorporation of the materials.

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