# Reduction of Excessive Bioavailable Phosphorus in Soils by Using Municipal and Industrial Wastes

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## ABSTRACT

**Poultry and swine production has created both economic growth in Oklahoma and concern over the effect of excessive land application of animal manure on water quality. The objectives of this study were to evaluate the ability of two drinking water treatment alum hydrosolids (HS1, HS2), cement kiln dust (CKD), and treated bauxite red mud (RM) to reduce excessive amounts of bioavailable P in soil and to determine potential environmental impacts from these treatments. Two soils that contained 553 and 296 mg kg$^{-1}$ Mehlich III-extractable P, as a result of prior treatment with poultry litter or dairy manure, were mixed with amendments at the rate of 30 and 100 g kg$^{-1}$ soil and incubated at 25°C for 9 wk. Reductions in Mehlich III-extractable P from 553 mg kg$^{-1}$ to 250 mg kg$^{-1}$ followed the trend HS2, CKD $\geq$ HS1 $\geq$ RM in the slightly acidic Dickson soil (fine-silty, siliceous, Thermic Glossic Fragiudult). Reductions in Mehlich III-extractable P from 296 mg kg$^{-1}$ to 110 mg kg$^{-1}$ followed the trend HS2 > HS1 > RM > CKD in the calcareous Keokuk soil (coarse-silty, mixed, Thermic Fluventic Haplustoll). Reduction of soluble P followed similar trends. Most treatments did not result in excessive soil pH or increases in soil salinity, in extractable Al, or in heavy metals in soils. Application of alum hydrosolids to soils with excessive amounts of bioavailable P in sensitive watersheds may improve drinking water quality and provide financial savings for municipalities.**

Growing poultry and swine production has contributed to an increase in economic growth for Oklahoma agriculture (Sharpley et al., 1991). Along with economic benefits, producers are faced with disposal of large amounts of animal manure generated from poultry and swine production. Land application of animal manure increases soil-available P and has raised concerns about P runoff from agricultural land (Field et al., 1985; Reddy et al., 1980; Sharpley et al., 1991; Singh and Jones, 1976).

Recent benchmark Conservation Practice Standard and Waste Utilization guidelines set by the Oklahoma Natural Resource Conservation Service (NRCS) limit animal manure applications to soils with excessive amounts of Mehlich III (M3) P (NRCS, 1994). These guidelines were designed to determine the application rate of animal manure beneficial to soils in sensitive watersheds. Application rate is based on soil test P determination by M3 extraction, field slope, soil depth, soil erodibility, flood plain, and other factors that minimize nonpoint source (NPS) P pollution.

Recently, USEPA Region VI promulgated Concentrated Animal Feeding Operation (CAFO) regulations and the Oklahoma Feed Yard Act have utilized Oklahoma NRCS guidelines that limit application of animal manures to P-sensitive watersheds. These guidelines limit animal manure applications to land with excessive amounts of available P. Reduction of bioavailable P in soils that exceed CAFO levels would reduce the NPS threat to sensitive watersheds. Land application of nonhazardous waste materials that reduce P solubility may be a feasible approach to reduce bioavailable P in soils.

Soluble forms of P are readily adsorbed and precipitated by soil or sediment components that contain Al, Fe, and Ca (Hsu, 1964, 1976). Iron and Al oxides (hydrous oxides) strongly adsorb and precipitate P from solution in natural water and soil systems (Stumm and Morgan, 1981; Tisdale et al., 1985). Calcium reacts with soluble P to form insoluble P compounds (Lindsay, 1979).

Alum sludge, or alum hydrosolid, is a waste byproduct generated from drinking water pretreatment. Alum hydrosolids contain aluminum oxides capable of adsorbing and precipitating soluble P. Alum hydrosolids were investigated as a soil amendment to improve the physical properties of potting media and plant growth by Bugbee and Frink (1985). Alum hydrosolids improved water-holding capacity and served as a liming material, but higher application rates of alum hydrosolids caused severe P deficiency and decreased lettuce (*Lactuca sativa* L.) yield (Bugbee and Frink, 1985). Alum hydrosolid additions to soil improved soil structure and plant growth, but high application rates (>2 MT ha$^{-1}$) induced P deficiencies and reduced corn (*Zea mays* L.) yields (Rengasamy et al., 1980). Land application of alum hydrosolids have induced similar P deficiencies in other studies (Heil and Barbarick, 1989). The ability of commercial alum to reduce P solubility in poultry litter (Moore and Miller, 1994) and reduce soluble P in field runoff water (Shreve at al., 1995) has recently been reported. Acidity derived from alum is neutralized by ammonia from poultry litter resulting in production of amorphous Al oxides in alum-treated poultry litter (Moore et al., 1995). Similarly, Al in alum hydrosolids from water treatment plants exists as insoluble aluminum oxides. Untreated alum (aluminum sulfate) is a very soluble salt that releases toxic Al and produces acidity when dissolved in water. Land application of alum may result in undesirable soil acidification and phytotoxic levels of $Al^{3+}$. However, alum-treated litter or alum hydrosolids have neutral or alkaline pH and Al exists as insoluble Al oxides, which should not release toxic Al or produce acidity in soil or aqueous systems.

Bauxite RM, which contains large amounts of Al and Fe oxides and Ca, is a waste product of the Al industry (Shiao and Akashi, 1977). Shannon and Verghese (1976)

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**Abbreviations:** HS, hydrosolid; CKD, cement kiln dust; RM, red mud; M3, Mehlich III; NRCS, Natural Resource Conservation Service; CAFO, concentrated animal feeding operations; NPS, nonpoint source; TCLP, toxicity characterization leaching procedure; ICP, inductively coupled plasma atomic emission spectroscopy.

suggested that bauxite RM could be economically used as a P removal amendment by precipitation. After treatment with acids, bauxite RM is an effective adsorbent for the removal of P (Barrow, 1982; Shiao and Akashi, 1977; Weaver and Ritchie, 1987). However, bauxite RM has undesirable properties. Bauxite RM contains lye and has a high pH (9–12), large electrical conductivity (60–350 dS $m^{-1}$), and large amounts of soluble Na (9 meq 100 $g^{-1}$) and aluminate. Bauxite RM is corrosive and is classified as a hazardous waste (Thompson, 1987). Land application of bauxite RM results in saline and alkaline conditions and poor soil physical structure (Wong and Ho, 1991). These undesirable properties have prevented use of bauxite RM in natural water and soil systems (Vachon et al., 1994). Most studies have focused on reclamation of soils rendered infertile by bauxite RM (Thompson, 1987). In a study by Vlahos et al. (1989) on the effects of P reduction on sandy soils, bauxite RM was found to be an extremely effective material in removal of total P in leachate after application of superphosphate fertilizer. Water retention, pH, Ca, and total soluble salt increased after application of the bauxite RM amendment to soil.

The ability of Ca to *fix* P into relatively insoluble forms is well known (Ford, 1933). Calcium reacts with soluble P to form insoluble Ca phosphates in soils at moderate to high pH (pH >6). Reagent-grade CaO and $Ca(OH)_2$ were the best amendments in reducing soluble P in chicken litter (Moore and Miller, 1994). Cement kiln dust (CKD) is a waste product generated during production of cement. Cement kiln dust is rich in Ca and K oxides.

Land application of nonhazardous waste materials has the potential to reduce excessive amounts of bioavailable P in soil, but more information is needed. Waste materials that contain hydrous oxides (e.g., alum sludge, bauxite RM) or Ca (cement kiln dust) are readily available. Additional information is needed to assess the ability of waste amendments to reduce bioavailable P and not cause any potential adverse environmental impacts. The objectives of this study were (i) to evaluate the ability of two drinking water treatment alum hydrosolids, cement kiln dust, and treated bauxite RM to reduce bioavailable P in soils; and (ii) to determine potential environmental impacts including excessive pH, salinity, available Al, and heavy metal availability associated with application of these waste materials to soil.

## MATERIALS AND METHODS

### Amendments

The effect of soil amendments to reduce bioavailable P was evaluated in a laboratory incubation study. Four amendments used in this study were two alum hydrosolids (HS1, HS2) collected from two water treatment facilities, cement kiln dust, and treated bauxite RM. Both alum hydrosolid materials were alum sludges. Untreated bauxite RM (21 kg) was treated with gypsum (4.1 kg) and leached with deionized water to remove excess lye and Na before analysis and use in this incubation study.

All amendments were analyzed for pH, salinity, Ca carbonate equivalent (CCE), total metal content, and extractable heavy metals by USEPA Toxicity Characteristic Leaching Procedure (TCLP; USEPA, 1990).

Amendment pH was analyzed in 1:2 amendment/0.01 *M* $CaCl_2$ solution using a glass electrode (McLean, 1982). Electrical conductivity (EC) of each amendment was analyzed in 1:2 amendment/deionized water solution (Rhoades, 1982). Calcium carbonate equivalent (CCE) was determined by reaction with HCl and backtitration as described by Rund (1984).

Total metal content of amendments was determined by wet digestion with $HNO_3$ and $HClO_4$ (Burau, 1982) and subsequent determination of Al, Ca, Cd, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, Pb, and Zn by inductively coupled plasma atomic emission spectroscopy (ICP). Metals were extracted from amendments according to the USEPA TCLP method (USEPA, 1990). The samples were filtered using 0.45-μm membrane filters and analyzed by ICP instrumentation for Ba, Cd, Cu, Mo, Ni, Pb, and Zn.

### Soils

Two soils with a history of animal manure application and containing large amounts of M3 P were selected for the incubation study (Table 1). Surface (<10 cm) soil samples were collected, air dried, and sieved (<2 mm). Mehlich III–extractable P levels were 553 and 296 mg P $kg^{-1}$ soil and soil pH were 5.3 and 8.2 for Dickson silt loam (fine-silty, siliceous, Thermic Glossic Fragiudult) and Keokuk very fine sandy loam (coarse-silty, mixed, Thermic Fluventic Haplustoll), respectively. The Dickson soil received >10 yr of poultry litter and the Keokuk soil received >10 yr of dairy manure.

Cation exchange capacity of the soils was determined by using $BaCl_2$ as described by Rhoades (1982). The citrate-bicarbonate–dithionite procedure described by Olsen and Ellis (1982) was used to measure free Fe oxides. A modified Mebius method was used to determine the soil organic C content of each soil (Yeomans and Bremner, 1988).

Soils were incubated with amendments in a growth chamber for a 9-wk period. Incubation temperature was 26°C during the day (16 h) and 24°C during the night (8 h). The incubator was maintained at approximately 60% humidity. Soil (250 g) was mixed with amendment rates of 30 and 100 g $kg^{-1}$ in plastic pots. The experimental design was a completely randomized block with three replications and controls (no amendment) for each soil. Soils were maintained at field capacity (−0.3 bar) moisture content. Triumph 64 wheat (*Triticum* spp.) was planted on the amended soils as a qualitative indicator of potential impact of amendments on crop growth.

The ability of amendments to reduce plant-available P was determined by measuring P extracted by the M3 procedure from amended soils at 3, 5, and 9 wk of incubation (Mehlich, 1984). Soils were sampled by thoroughly mixing the entire volume of treated soil and removing a subsample (10 g). Twenty milliliters of M3 extractant was added to 2 g of soil from each sample and placed on a rotary shaker for 5 min. The samples were then filtered using Whatman no. 2 filter

**Table 1. Soil chemical properties and characteristics.**

| Parameter | Soil: Dickson | Soil: Keokuk |
|---|---|---|
| Soil texture | silt loam | sandy loam |
| Mehlich III P, mg P $kg^{-1}$ | 553 | 296 |
| Soluble P, mg P $kg^{-1}$ | 13.5 | 4.1 |
| pH | 5.3 | 8.0 |
| Electrical conductivity, dS $m^{-1}$ | 0.18 | 0.37 |
| Cation exchange capacity, $cmol_c$ $kg^{-1}$ | 12.9 | 13.7 |
| Free Fe oxides, g Fe $kg^{-1}$ | 0.15 | 0.01 |
| Soil organic C, g $kg^{-1}$ | 17.2 | 13.1 |

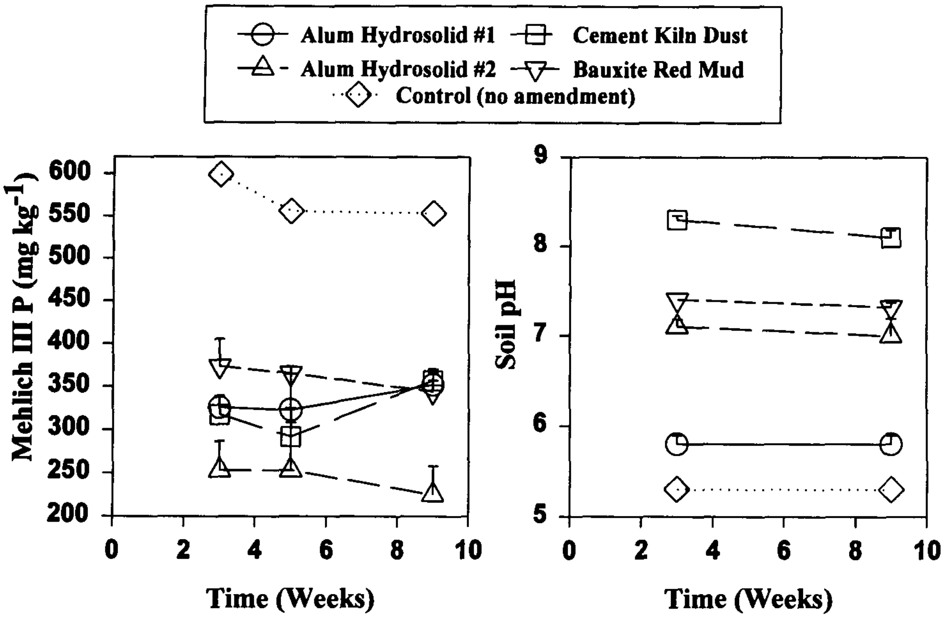


**Fig. 1. Effect of amendments on Mehlich III-extractable P and soil pH treatment means with time in the Dickson soil.**

paper. Phosphorus extracted by the M3 P procedure was measured by the Modified Murphy-Riley method (Murphy and Riley, 1962). Soluble P in amended soils was determined after 9 wk of incubation. Ten milliliters of 0.01 *M* $CaCl_2$ was added to soil (5 g) and placed on a shaker for 15 h. The Modified Murphy-Riley method was used to analyze soluble P in filtered soil solution. Extractable Al was determined using potassium chloride (KCl) extraction (Barnhisel and Bertsch, 1982). Aluminum in KCl extracts was measured by ICP.

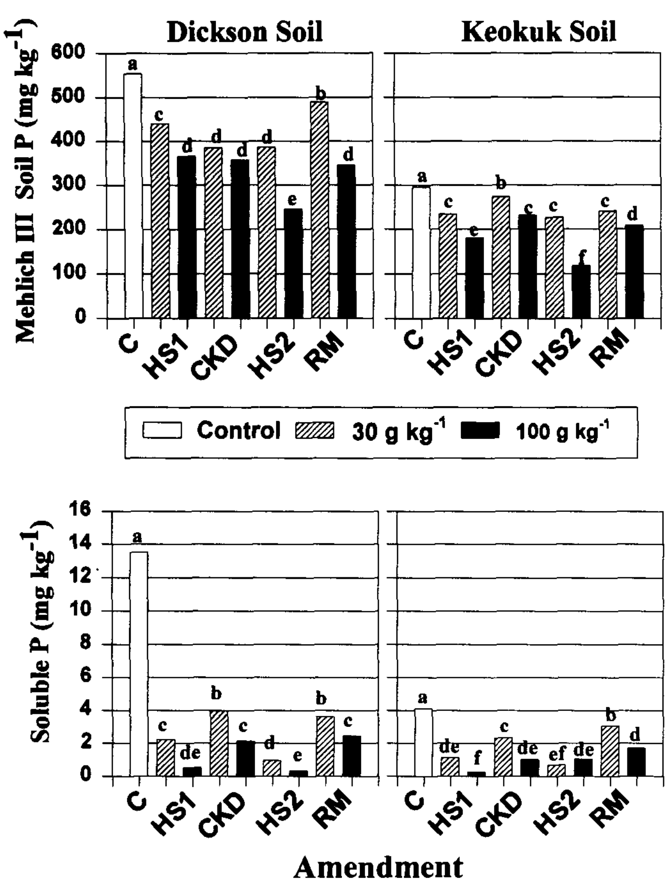


**Fig. 2. Mehlich III-extractable P and 0.01 *M* $CaCl_2$-soluble P in amended soils after 9 wk of incubation. Columns with the same letter are not different at $P < 0.05$ within each soil. *C* = control; HS1 = alum hydrosolid no. 1; CKD = cement kiln dust; HS2 = alum hydrosolid no. 2; RM = bauxite red mud.**

Treatment effects on measured parameters were evaluated by using multiple comparison of means by Duncan's multiple range test (Steele and Torrie, 1980). Statistical analysis of data was performed using appropriate procedures given by the SAS Institute (SAS Inst., 1988).

## RESULTS AND DISCUSSION

### Reduction of Bioavailable Phosphorus

The greatest changes in M3 P and pH occurred in the first 3 wk of incubation for the Dickson (Fig. 1) and Keokuk soils (data not shown). Chemical reactions between the soils and amendments were assumed to be essentially complete after 9 wk of incubation. Therefore, only results after 9 wk of incubation will be presented.

Both soils showed significant ($P < 0.05$) decreases in M3 P for all amendment treatments after 9 wk of incubation (Fig. 2). Reduction of M3 P followed the trend HS2, CKD ≥ HS1 ≥ RM for the slightly acidic Dickson soil. A different trend for the calcareous Keokuk soil was found: HS2 > HS1 > RM > CKD. The alum hydrosolids followed the same general trend in both the slightly acidic and the calcareous soils: HS2 ≥ HS1. Greater reduction of M3 P by alum hydrosolids than CKD or RM is likely due to highly reactive amorphous Al oxides (Elliott et al., 1988; Young et al., 1988). Because drinking water treatment may involve adjustment of water pH with liming materials, some alum hydrosolids may contain significant amounts of Ca. The larger total Ca in HS2 compared with HS1 (Table 2) suggests larger reductions in M3 P may be partly due to formation of Ca precipitates. The ability of CKD to

**Table 2. Chemical properties and total metal content of amendments.**

| Property | Amendments† HS1 | HS2 | CKD | RM | |
|---|---|---|---|---|---|
| pH | 7.0 | 7.6 | 12.6 | 8.1 | |
| EC‡ | 0.31 | 0.58 | 17.8 | 2.63 | |
| CCE§ | 1.87 | 14.8 | 87.5 | 24.2 | |
| Total metal | g kg$^{-1}$ | | | | Normal metal content in soil¶ |
| Al | 141 | 147 | 17.6 | 111 | 11-79 |
| Ca | 2.1 | 21.9 | 205 | 65.0 | 1-18 |
| Fe | 35.8 | 29.6 | – | 209 | 0.7-56 |
| K | 0.79 | 1.2 | – | – | 0.8-33 |
| Mg | 3.26 | 7.2 | 7.2 | 0.79 | 0.6-12 |
| Mn | 11.0 | 0.82 | 0.13 | 0.03 | 0-4 |
| Na | 0.09 | 0.38 | 1.65 | 9.25 | 0.7-22 |

† HS1 = hydrosolid 1; HS2 = hydrosolid 2; CKD = cement kiln dust; RM = bauxite red mud.
‡ Electrical conductivity (dS m$^{-1}$).
§ Calcium carbonate equivalent expressed in percent.
¶ From Isaac and Kerber, 1971.

reduce P in the calcareous soil is significantly less than P reductions in slightly acidic soil. Although CKD does not contain large amounts of Al oxides, it does contain significant amounts of Ca (Table 2). Large amounts of Ca in CKD may reduce P bioavailability in soil by forming Ca–P precipitates. The ability of CKD to reduce bioavailable P in the calcareous soil was significantly less than in the slightly acidic soil. These differences may be related to relative contribution of CKD to changes in soil pH of the treated soils (Table 2). Treatment of soil with CKD may have resulted in formation of greater amounts of Ca–P precipitates in the Dickson soil than in the Keokuk soil. In general, increasing rates of all amendments from 30 to 100 g kg$^{-1}$ decreased soil M3 P.

None of the amendments reduced the M3 P to <200 mg kg$^{-1}$, the severe nonpoint source (NPS) P pollution level guideline of the Oklahoma Natural Resource Conservation Service (NRCS, 1994), in the Dickson soil. However, the HS2 100 g kg$^{-1}$ rate lowered the M3 P from 553 to 250 mg kg$^{-1}$ in the Dickson soil. In the Keokuk soil, the HS1, HS2, and RM 100 g kg$^{-1}$ rate lowered the M3 P from 296 to <200 mg kg$^{-1}$. The addition of these amendments should decrease the NPS runoff threat of bioavailable P in soil to sensitive surface waters.

The M3 P procedure extracts both readily soluble and insoluble P minerals that may not be immediately bioavailable in aquatic environments (Fixen and Grove, 1990). Although M3 P is related to potential P availability and Oklahoma NRCS guidelines are based on M3 P, soluble P may be a better environmental indicator of bioavailability and impact on aquatic life. All amendments reduced soluble P in soils (Fig. 2). Reduction of soluble P in Dickson and Keokuk soils followed the trend HS2 ≥ HS1 > CKD, RM. Soluble P reduction was similar to M3 P reduction results. A greater reduction in soluble P was found in the slightly acidic Dickson soil than in the calcareous Keokuk soil. Similar to M3 P reduction, increasing the amendment rates from 30 to 100 g kg$^{-1}$ reduced soluble P in all treatments except for HS2 in the Keokuk soil.

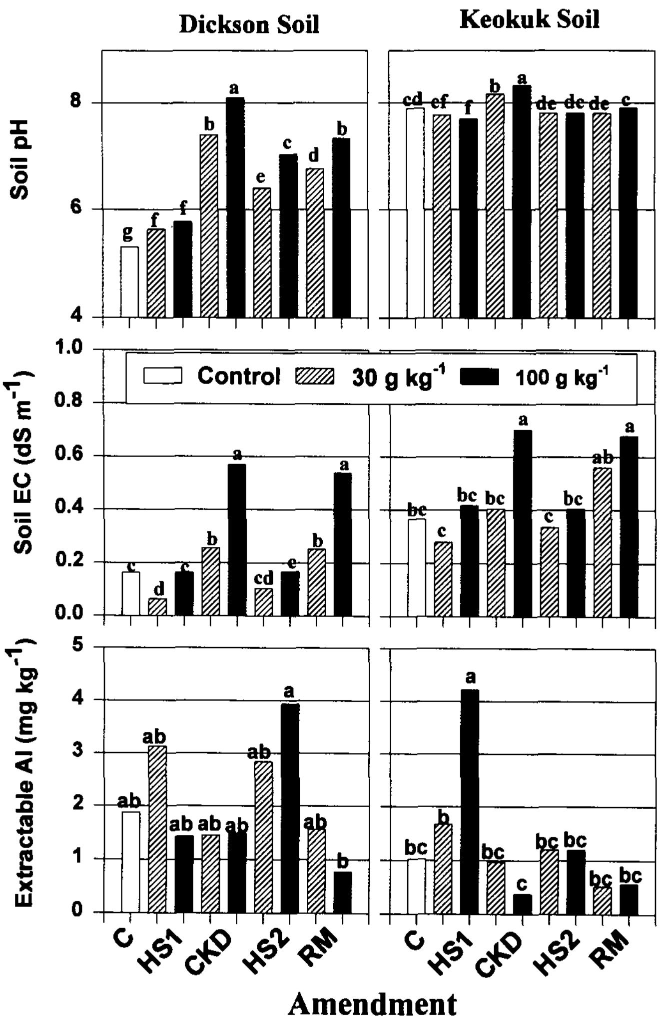


**Fig. 3. Effects of amendments on soil pH, soil EC, and KCl-extractable Al in amended soils after 9 wk of incubation. Columns with the same letter are not different at $P < 0.05$ within each soil. *C* = control; HS1 = alum hydrosolid no. 1; CKD = cement kiln dust; HS2 = alum hydrosolid no. 2; RM = bauxite red mud.**

## Potential Environmental Impact

Soil pH, salinity (EC), extractable Al, and heavy metal content, and extractability were determined in amendments and soils to ensure the addition of amendments to soil did not result in undesirable potential environmental impacts.

The Triumph 64 wheat that was planted in the treated soils as a qualitative indicator showed no indications of nutrient problems unlike other studies that found P deficiencies in sorghum–sudangrass [*Sorghum bicolor* (L.) Moench 'NB2805'-5-*Sudanese* (Piper) Stapf] (Heil and Barbarick, 1989), in lettuce (Bugbee and Frink, 1985), and in tomato (*Lycopersicon esculentum* L.) (Elliott and Singer, 1988; Elliott et al., 1990; Young et al., 1988) induced by alum sludge. Plant-available P in the Dickson and Keokuk soils treated with alum hydrosolids were well above P requirements for wheat production (Allen and Johnson, 1993).

The slightly acidic Dickson soil showed significant increases in pH from 5.7 to 8.0 after the addition of CKD at the 100 g kg$^{-1}$ rate (Fig. 3). Cement kiln dust, a known liming material, can easily increase soil pH > 7.0 (Gelderman et al., 1992). In general, increasing the amendment rate from 30 to 100 g $kg^{-1}$ significantly increased pH for all amendments in the Dickson soil. Amendments had little effect on soil pH in the Keokuk soil. Final pH < 8.3 of all treated soils is not considered excessive and is not typically associated with potential environmental hazards.

Several amendments (CKD, RM) have significant amounts of soluble salts (Table 1) and might increase soil salinity. The alum hydrosolids did not increase soil salinity (Fig. 3). However, 100 g $kg^{-1}$ rates of CKD and RM resulted in small but significant ($P < 0.05$) changes in EC that may affect salt-sensitive plants (Rhoades and Miyamito, 1990).

The effect of alum hydrosolids and other amendments on extractable Al in soil is shown in Fig. 3. None of the amendments significantly increased extractable Al in the Dickson soil. Only the 100 g $kg^{-1}$ rate of HS1 showed a significant increase in extractable Al in the Keokuk soil. Amendments did not increase extractable Al in incubated soils >5 mg Al $kg^{-1}$. Adverse effects are associated with much higher levels of extractable Al (>60 mg Al $kg^{-1}$) for wheat (Sloan et al., 1995). Therefore, slight increases in available Al from application of amendments should not have adverse effects on soils or plants.

Total metal and TCLP-extractable metal of the amendments are presented in Table 3. With the exception of total Cd in CKD and total Cd and Pb in RM, all amendment total metal contents were within the range of typical soil total metal contents. Alum hydrosolid heavy metal contents were similar to those reported in other water treatment sludges (Elliott et al., 1990). Alum hydrosolids used in this study do not contain elevated levels of Ni, which have been reported for ferric coagulant water-treatment sludges from steel pickling or bauxite extraction wastes (Elliott et al., 1990; Heil and Barbarick, 1989). Therefore, land application of alum hydrosolids should not increase heavy metal concentration in soil. Heavy metals extracted by TCLP were below USEPA regulatory levels and showed the amendments are not hazardous wastes. Comparison of TCLP and total metal values show most heavy metal is not in bioavailable forms (Table 3). Amendment TCLP levels were similar to heavy metals determined by TCLP in typical baseline soils (Scott, 1994). Therefore, land application of alum hydrosolids should not increase heavy metal availability in soil. Similarly, Elliott et al. (1990) found most heavy metals in alum hydrosolids were strongly bound by Al and Fe oxides in forms that do not have potential adverse environmental impacts.

**Table 3. Toxicity characteristic leaching procedure (TCLP) extractable and total heavy metal content of amendments.**

| Total metal | Amendments† HS1 | HS2 | CKD | RM | Normal metal content in soil‡ |
|---|---|---|---|---|---|
| | mg $kg^{-1}$ | | | | |
| Cd | 0.57 | 0.93 | 2.98 | 6.61 | 0.01-1.3 |
| Cu | 24.8 | 37.9 | 7.48 | 27.3 | 1.4-216 |
| Mo | 0.12 | 0.25 | 0.70 | 0.53 | 0-40 |
| Ni | 26.6 | 28.5 | 14.4 | 10.0 | 2.2-154 |
| Pb | 14.0 | 15.9 | 29.7 | 56.4 | 3.0-36 |
| Zn | 86.1 | 80.7 | 36.8 | 56.1 | 3.2-170 |
| TCLP | | | | | USEPA regulatory limit§ |
| | mg $L^{-1}$ | | | | |
| Ba | 1.17 | 0.83 | 0.20 | 0.02 | 100 |
| Cd | 0.03 | 0.03 | 0.03 | 0.03 | 1.0 |
| Cu | 0.03 | 0.02 | 0.03 | 0.03 | – |
| Mo | 0.06 | 0.05 | 0.08 | 0.03 | – |
| Ni | 0.06 | 0.03 | 0.08 | 0.40 | – |
| Pb | 0.08 | 0.08 | 0.16 | 0.16 | 5.0 |
| Zn | 0.07 | 0.08 | 0.13 | 0.40 | – |

† HS1 = hydrosolid 1; HS2 = hydrosolid 2; CKD = cement kiln dust; RM = bauxite red mud.
‡ Cd, Cu, Ni, Pb, and Zn from Holgrem et al., 1993 (1st to 99th percentile); Mo from Alloway, 1990.
§ Regulatory limit specified by USEPA SW-846 Method 1311.

## SUMMARY

The addition of alum hydrosolids, cement kiln dust, and treated bauxite RM reduce excessive amounts of bioavailable P in soil. Increasing the rate of amendment will, in most cases, decrease the amount of bioavailable P. Adverse potential environmental impacts from salinity, pH, Al, and total and extractable metals on application of these municipal and industrial amendments should be insignificant. Most soil treatments did not result in excessive soil pH or increased soil salinity. However, high rates of cement kiln dust and bauxite RM may increase soil salinity in the amended soil, which may affect salt-sensitive crops. Alum hydrosolid applications had little or no effect on extractable Al in soil. Land application of alum hydrosolids used in this study should not increase content or availability of heavy metals in soils. Alum hydrosolid wastes are currently being landfilled at great expense to municipalities. Also, several municipal water-treatment plants producing alum hydrosolids in Oklahoma may have source water degraded by nonpoint-source P pollution. Alum hydrosolid application to soils in sensitive watersheds that have soils with excessive amounts of bioavailable P has the potential to improve drinking water quality and provide financial savings for municipalities.

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