

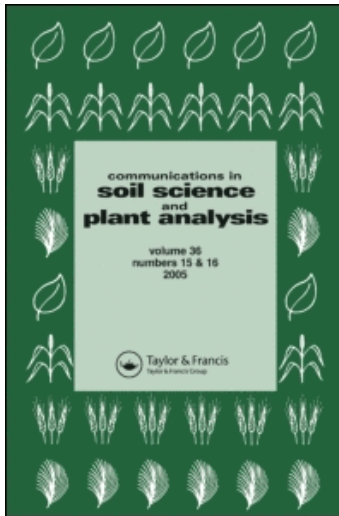
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Phosphorus Extraction Methods for Water Treatment Residual–Amended Soils

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Abstract: Water treatment residuals (WTR) can adsorb tremendous amounts of phosphorus (P). A soil that had biosolids applied eight times over 16 years at a rate of $6.7 \text{ Mg ha}^{-1} \text{ y}^{-1}$ contained 28 mg kg^{-1} ammonium–bicarbonate diethylenetriaminepentaacetic acid (AB-DTPA), 57 mg kg^{-1} Olsen, 95 mg kg^{-1} Bray-1, and 53 mg kg^{-1} Mehlich-III extractable P. To 10 g of soil, WTRs were added at rates of 0, 0.1, 1, 2, 4, 6, 8, and 10 g, then 20 mL of distilled deionized H_2O (DI) were added and the mixtures were shaken for 1 week, filtered, and analyzed for soluble (ortho-P) and total soluble P. The soil–WTR mixtures were dried and P extracted using DI, AB-DTPA, Olsen, Bray-1, and Mehlich-III. Results indicated that all methods except AB-DTPA showed reduced extractable-P concentrations with increasing WTR. The AB-DTPA extractable P increased with increasing WTR rate. The water-extractable method predicted P reduction best, followed by Bray-1 and Mehlich-III, and finally Olsen.

Keywords: Water-treatment residuals (WTR), water extractable, AB-DTPA, Olsen, Mehlich-III, Bray-I, soluble P, total soluble P

INTRODUCTION

Alum [$\text{Al}_2(\text{SO}_4)_3 \cdot 14\text{H}_2\text{O}$] is commonly used in the drinking-water treatment process for particulate flocculation and water clarification. Water treatment residuals (WTRs), a waste product of drinking-water treatment facilities,

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tend to have a mineral form similar to amorphous $\text{Al}(\text{OH})_3$. Because of this amorphous nature, WTRs have a large surface area and are highly reactive. Soils may contain excessive amounts of P because of overapplication of fertilizers, manures, biosolids, or other wastes. Addition of WTRs to these soils can reduce the amount of extractable phosphorus (P) by sorption onto the amorphous $\text{Al}(\text{OH})_3$ present.

Addition of WTR from 2.5 to 10% (w/w) to loamy coastal plain soils increased the soil P sorbing capacity several-fold (Novak and Watts 2004). Water treatment residuals have been shown to sorb all biosolids-borne P at application ratios of 8:1, respectively (Ippolito et al. 1999). Maximum WTRs P sorption has been shown to be $12,500 \text{ mg kg}^{-1}$, with the majority of P associated with WTRs Al-fraction as shown from scanning electron microscopy–energy dispersive spectroscopy analysis (Ippolito et al. 2003).

Application of WTRs to soils with excessive P concentrations will reduce extractable P concentrations and decrease the amount of P available for surface movement to surface water. Determining the most appropriate P extraction method for WTR-amended soils is of paramount importance for determining P losses or predicting P fertilizer recommendations.

WTRs applied at rates of 0, 36, and 52 dry Mg ha^{-1} to plots growing loblolly pine (*Pinus rigida* Mill.) showed no differences in Mehlich-III extractable P 30 months after application (Geertsema et al. 1994). However, a greenhouse project found that increasing WTR application rate from 0 to 17.8 g kg^{-1} decreased Mehlich-I extractable soil P from 70.7 to 61.5 mg kg^{-1} (Cox, Camberato, and Smith 1997). The type of WTR dewatering process and the addition of WTRs to soil caused a significant reduction in Mehlich-III P (Hyde and Morris 2000). WTRs applied to fields at rates equal to 0, 2.2, 9.0, and 18 Mg ha^{-1} resulted in the soils showing decreased Mehlich-III soil test P concentrations with increasing application, and Hausteint et al. (2000) associated this decrease with increasing Al content. Increasing WTRs from 30 to 100 g kg^{-1} reduced Mehlich-III extractable P in soils with a history of manure application but did not lower extractable P concentrations to below a level considered severe (Peters and Basta 1996). The same study found reductions in soluble, or ortho-P, and suggested WTR application in sensitive watersheds may improve water quality by reducing bioavailable P (Peters and Basta 1996). Mehlich-III extractable P was shown to overestimate P plant availability, whereas water extracts were an excellent predictor of ortho-P in WTRs (Basta et al. 2000). Equilibrium studies showed WTRs to be very effective in reducing soluble soil P, and when coapplied with biosolids to sandy soils P, leaching losses were reduced to <1% (Elliott et al. 2002). The addition of WTRs to poultry manure, on a 2 to 1 molar basis of total WTR-Al to total manure P, significantly reduced dissolved reactive phosphorus by 39% as compared to a control (Dao et al. 2001).

The Natural Resource Conservation Service, along with Colorado State University, has developed the Colorado Phosphorus Index Risk Assessment (Sharkoff, Waskom, and Davis 2003). Contained within the assessment

document are four P soil tests recommended for use with different soil types. The Bray-I test is recommended for acidic soils, the Olsen and AB-DTPA tests are used for soils that contain calcium carbonates and have a pH greater than 7, and the Mehlich-III test can be used for both low- and high-pH soils. Objectives were to quantify water-extractable P concentrations in excessive P-containing soils amended with WTRs using the tests described. The goal was to determine and recommend which test best predicts P availability in soils amended with biosolids and subsequently amended with WTRs.

MATERIALS AND METHODS

Aluminum-based WTRs were obtained from the Allen Drinking Water Treatment Facility in Englewood, Colorado. The WTRs were separated into the 0.1- to 0.3-mm size fraction for use in this study. This fraction can sorb 12,500 mg soluble P kg⁻¹ as shown in a previous study (Ippolito et al. 2003).

A soil treated with eight biosolid applications at 6.7 Mg ha⁻¹, an agronomic rate based on the crop nitrogen requirements, applied every other year in a dryland wheat (*Triticum aestivum* L.) fallow crop rotation was collected from the 0–20-cm depth. The soil was classified as a Platner loam, Abruptic Aridic Paleustoll. The land was farmed using conventional tillage practices. A detailed site description can be found in Barbarick et al. (1998).

Elemental composition of WTRs and soil was determined by HClO₄–HNO₃–HF–HCl digestion (Soltanpour et al. 1996) and 4 M HNO₃ digestion (Bradford et al. 1975), respectively, followed by analysis using inductively coupled plasma atomic emission spectrometry (ICP-AES). For both materials, pH and electrical conductivity (EC) were determined using a saturated paste extract (Thomas 1996; Rhoades 1996), NO₃-N and NH₄-N using a 2 M KCl extract (Mulvaney 1996), and total N using a LECO-1000 CHN autoanalyzer (Nelson and Sommers 1996). In addition, both materials were analyzed for extractable P using either AB-DTPA (Barbarick and Workman 1987), Bray-I (Kuo 1996), Mehlich-III (Mehlich 1984), or Olsen (Kuo 1996) extracting solutions. Chemical characteristics for the WTRs and soil are shown in Table 1.

The Colorado Phosphorus Risk Index separates AB-DTPA, Bray-I, Mehlich-III, and Olsen extractable P into four categories of low, medium, high, and very high (Table 2). These extractants were originally intended to be used as fertility indices to advise growers about their probability of a yield response to P fertilizer. Colorado and other state utilize these extractants in P risk indices to predict the potential for P movement.

Although biosolids applications were at agronomic rates, the four extractants suggest that this soil contained medium to high P concentrations (Tables 1 and 2). In addition, AB-DTPA P concentrations greater than 7 mg kg⁻¹ are considered high for dryland winter wheat (Davis et al. 2004). The AB-DTPA

Table 1. Properties of the 0.1- to 0.3-mm size fractions of the Englewood, CO, water treatment residuals (WTRs) and Platner loam- and biosolids-treated soil

Property	WTRs	Platner loam
pH	7.6	6.6
EC ^a (dS m ⁻¹)	6.9	0.8
Total N (%)	0.62	0.13
NH ₄ -N (mg kg ⁻¹)	6.6	3.2
NO ₃ -N (mg kg ⁻¹)	710	22
Al (mg kg ⁻¹)	64100	16500
Ca (mg kg ⁻¹)	41900	7660
P (mg kg ⁻¹)	2340	890
Fe (mg kg ⁻¹)	17600	14700
Mg (mg kg ⁻¹)	3860	4310
Na (mg kg ⁻¹)	450	70
K (mg kg ⁻¹)	3080	2960
Cu (mg kg ⁻¹)	4110	28
Zn (mg kg ⁻¹)	137	110
Ni (mg kg ⁻¹)	12.5	15
AB-DTPA P (mg kg ⁻¹)	7.2	28
Bray-I P (mg kg ⁻¹)	0.3	95
Mehlich-III P (mg kg ⁻¹)	0.5	53
Olsen P (mg kg ⁻¹)	33	57

^aElectrical conductivity.

test is not widely used outside of Colorado but is a test required for use on biosolids-amended Colorado soils.

Fractionated WTRs were added to soil at rates of 0.0 (control), 0.1, 1.0, 2.0, 4.0, 6.0, 8.0, and 10.0 g to triplicate 10.0 g of soil (dry weight) in 50-mL centrifuge tubes. Twenty-mL DI were added, and the mixtures were shaken for 1 week on a reciprocating shaker at 120 oscillations per min.

Table 2. Colorado phosphorus risk index—soil test phosphorus risk^a

Soil extraction test	Soil P risk rating (md kg ⁻¹)			
	Low	Medium	High	Very high
AB-DTPA	<10	10–20	21–40	>40
Bray-I	<30	30–60	61–120	>120
Mehlich-III	<40	40–100	101–200	>200
Olsen	<20	20–40	41–80	>80

^aFrom [Sharkoff et al. (2003)].

The tubes were then centrifuged and the liquid decanted, filtered through Whatman #42 filter paper, and analyzed for water-extractable ortho-P and total water-soluble P using a modified ascorbic-acid method (Rodriguez et al. 1994) and ICP-AES, respectively.

The WTR–soil solids were then air dried, ground, and passed through a 2-mm sieve before extraction using AB-DTPA, Bray-I, Mehlich-III, or Olsen extractants. Extracts were filtered through Whatman #42 filter paper. The AB-DTPA filtrates were analyzed for P by ICP-AES, whereas Bray-I, Mehlich-III, and Olsen filtrates were analyzed for extractable P by the modified ascorbic-acid method. Problems associated with the AB-DTPA extraction method for the WTR–soil mixtures were evident (see Results and Discussion). Therefore, the AB-DTPA extracts were reanalyzed for P, calcium (Ca), and aluminum (Al) content by ICP-AES.

The WTRs were also analyzed alone in triplicate, by weighing either 1.0 g for water-extractable, total water-soluble, Olsen, or Bray-I P, 1.3 g for Mehlich-III P, or 5.0 g for AB-DTPA extractable P. All solutions were passed through Whatman #42 filter paper and analyzed for P using the previous methods. All data was analyzed using regression analysis and analysis of variance at a probability level (p) of 0.05 (Steel and Torrie 1980), and all methods were compared to the Colorado Phosphorus Index Risk Assessment–Soil Test P Risk Index.

RESULTS AND DISCUSSION

Water-extractable P significantly decreased ($p < 0.05$) with increasing addition of WTRs and was negligible after addition of 4.0 g of WTRs (Figure 1). This extraction method was an excellent predictor of soluble P, and followed trends similar to our previous findings (Ippolito et al. 2003). Others have suggested the use of water-extractable P as the criteria to be used for P additions to WTRs (Basta et al. 2000). The addition of WTRs to soils containing excessive P has shown water-soluble ortho-P to be reduced below detection limits of $0.025 \text{ mg P L}^{-1}$ (Hyde and Morris 2000). Total water-soluble P, as determined by ICP-AES, also decreased significantly ($p < 0.05$) and followed a similar trend (Figure 2).

Olsen, Bray-I, and Mehlich-III extractable P all extracted significantly less P ($p < 0.05$) with increasing WTR rate. According to the Olsen method, used for soils with a pH greater than 7 and containing calcium carbonate, WTRs did not extract soil P below its initially “high” risk category at the highest WTR rate (Figure 3). This may be due to the extractant utilized, namely NaHCO_3 , which reacts with and enhances the dissolution of calcium phosphates by precipitating Ca as CaCO_3 . Although previous research suggested the possibility of calcium phosphate precipitation in WTRs, scanning electron microscopy using energy dispersive spectroscopy (SEM-EDS) clearly showed a WTRs Al–P association (Ippolito et al.

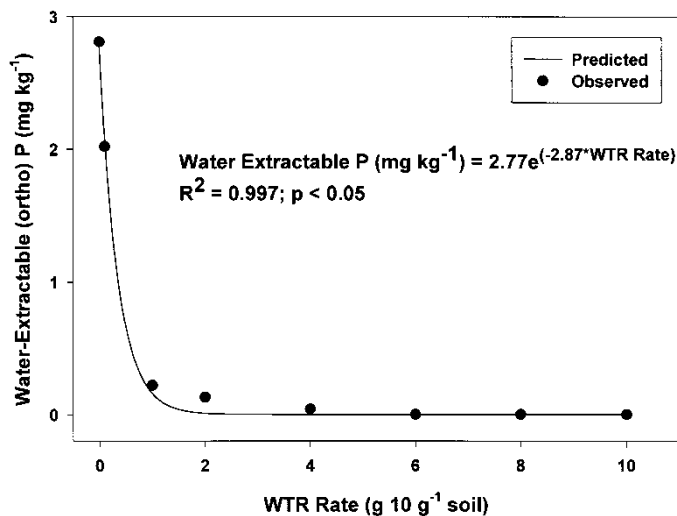


Figure 1. Water-extractable phosphorus (ortho-P) in 10 g of a loam soil amended with 0 to 10 g of WTRs.

2003). However, the potential presence of WTR-borne Ca–P species cannot be excluded because their concentrations may be below SEM-EDS detection limits of approximately 1% by weight, and extracting WTRs alone with the Olsen extractant produced 33 mg P kg⁻¹ (Table 1). The Olsen

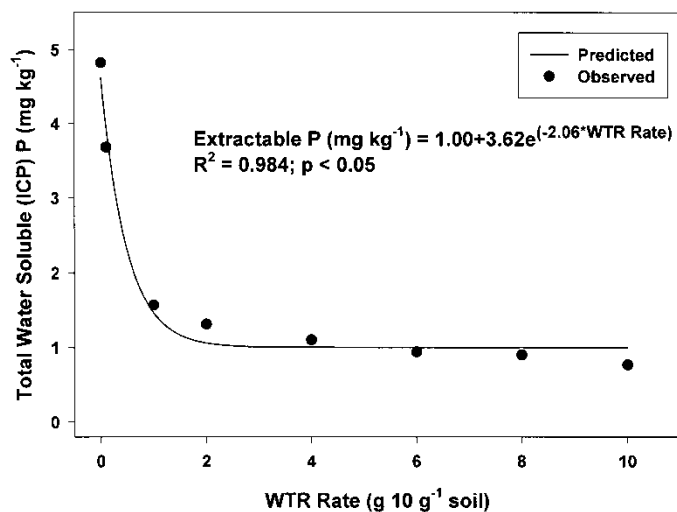


Figure 2. Total water-soluble phosphorus (ICP-P) in 10 g of a loam soil amended with 0 to 10 g of WTRs.

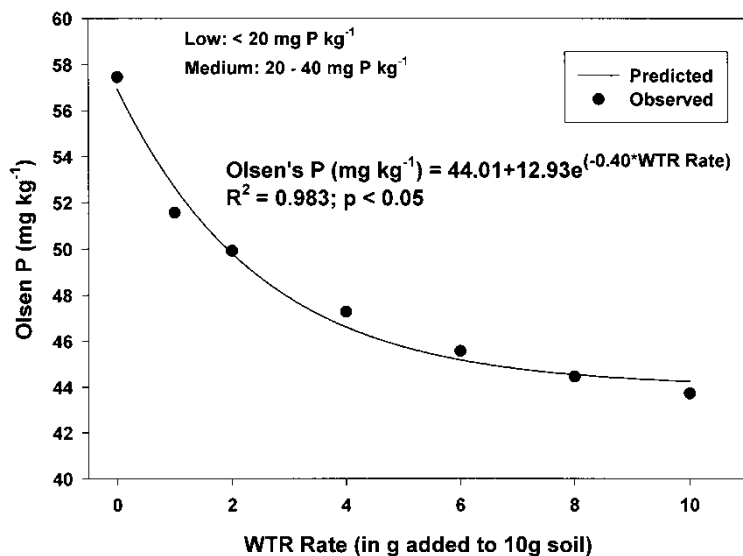


Figure 3. Olsen extractable phosphorus from air-dried WTRs: loam soil mixtures.

test should not dissolve the dominant Al–P phase and subsequently would be a poor predictor of WTR–amended soil P content. This contention is based on previous research showing P to be strongly sorbed to the Al phase of WTRs and not readily releasing P into solution over long periods of time (Ippolito et al. 2003). The Olsen test has been shown by others to have reduced ability to predict P adequately in soils amended with WTRs (Basta et al. 2000).

The Bray-I and Mehlich-III extraction methods both were excellent in predicting soil P reduction with increasing WTR rate (Figures 4 and 5). Increasing the WTR rate significantly reduced Bray-I and Mehlich-III P concentrations ($p < 0.05$) from those considered high and medium, respectively, to low according to the Colorado P Test Risk Index (Table 2). Increasing WTR soil ratio to slightly above 1:2 and 1:5 caused the Bray-I and Mehlich-III tests to predict low P concentration, respectively. Research has shown reductions in Mehlich-III extractable P with WTR addition to slightly acid and basic soils (Hyde and Morris 2000; Haustein et al. 2000; Peters and Basta 1996). Others have shown the Mehlich-III extraction method to overestimate plant available P from WTRs because of the dissolution of WTR sorbed P (Basta et al. 2000).

The AB-DTPA method is used for soils having a pH greater than 7 and containing CaCO_3 , such as many soils of eastern Colorado. However, this extraction method was a poor predictor of extractable P with increasing WTR rates resulting in significantly increased extractable P ($p < 0.05$) (Figure 6). In an effort to quantify problems with this method, the filtrate for P, Ca, and Al was reanalyzed using ICP-AES. Results showed that

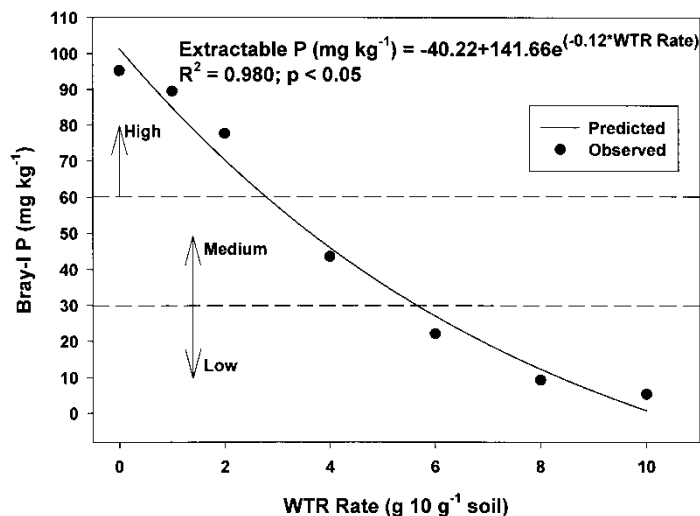


Figure 4. Bray-I extractable phosphorus from air-dried WTRs: loam soil mixtures.

increasing the WTR rate increased extractable-P concentration with a subsequent decrease in Ca and increase in Al content (Table 3).

Similar to the Olsen method, the NH_4HCO_3 could be reacting with and enhancing the dissolution of calcium phosphates by precipitating Ca as CaCO_3 . Previous research suggested the possibility of calcium phosphate

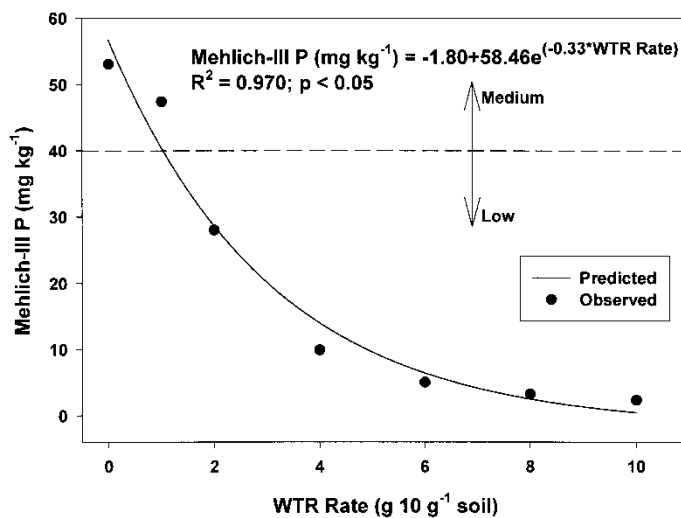


Figure 5. Mehlich-III extractable phosphorus from air-dried WTRs: loam soil mixtures.

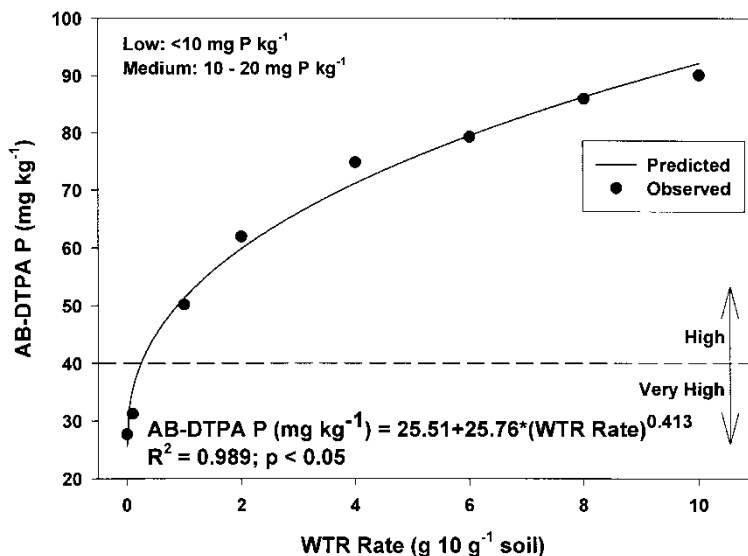


Figure 6. AB-DTPA extractable phosphorus from air-dried WTRs: loam soil mixtures.

precipitation in WTRs, although SEM-EDS clearly showed a WTR Al–P association at (Ippolito et al. 2003). It still may be possible that these WTRs contain Ca–P associates at concentrations lower than SEM-EDS detection limits. A more plausible explanation is the DTPA is chelating Al from amorphous $\text{Al}(\text{OH})_3$ in the WTRs and thus releasing and increasing solution P.

Using MinteqA2 (Allison et al. 1991) and the Lindsay database, we modeled Ca and Al chelation by DTPA using inputs of DTPA concentration of 0.005 M, 1 M NH_4^+ , 1 M CO_3^{2-} , a pH of 7.6 as defined by the AB-DTPA procedure, and $\text{Al}(\text{OH})_3$ (amorphous) and CaCO_3 (calcite) both set as infinite solids, assuming CaCO_3 is present in the WTRs. Results showed 94.8% of DTPA complexed with Al species and 4.9% associated with Ca. The system was remodeled, substituting $\text{Al}(\text{PO}_4) \cdot 2\text{H}_2\text{O}$ (variscite) for $\text{Al}(\text{OH})_3$. Results showed 60% and 39% of DTPA complexed with Al and Ca species, respectively. This suggests the DTPA may be complexing and removing Al from species such as $\text{Al}(\text{PO}_4) \cdot 2\text{H}_2\text{O}$ (amorphous variscite) and thus increasing PO_4 in solution.

CONCLUSIONS

Aluminum-containing WTRs can reduce excessive soil P concentrations. However, choosing the correct P-extraction method is important to accurately predict reductions in P bioavailability. Increasing rates of WTRs were added

Table 3. Increasing water treatment residuals (WTRs) rate to 10 g of soil affects AB-DTPA extractable P, Ca, and AL concentrations

WTRs rate	P (mg kg ⁻¹)	Ca (mg kg ⁻¹)	Al (mg kg ⁻¹)
0 g	28	410	0.8
0.1 g	31	440	0.7
1 g	50	350	0.7
2 g	62	280	0.8
4 g	75	160	1.2
6 g	79	91	1.2
8 g	86	92	1.6
10 g	90	100	1.9
P to Ca correlation	$r = -0.97$		
	$p < 0.05$		
P to Al correlation	$r = 0.86$		
	$p < 0.05$		

to a soil with a history of biosolid application which has resulted in an excessive P concentration. Bray-I, Mehlich-III, and water-extractable P methods demonstrated that with WTRs addition, labile soil P can be lowered from concentrations considered high or medium to low according to the Colorado P Risk Index. The AB-DTPA-extractable P concentrations increased with increasing WTR addition. This may be to DTPA chelating Al from amorphous variscite and thus releasing and increasing P in solution with increased WTR addition. As with other researchers, this study suggests using a water extract to predict P availability in WTR-amended soils.

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