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Title

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Permalink

Journal

ISSN

Authors

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Publication Date

DOI

Peer reviewed

Seasonal Phosphorus Dynamics in a Volcanic Soil of Northern California

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Phosphorus dynamics and management strategies were investigated in a strongly weathered rhyolitic soil in the northern Coast Ranges of California. Recent conversion of upland, native oak (*Quercus* spp.) woodland ecosystems to winegrape production has led to a P deficiency in these soils. We investigated the growing season behavior of Hedley phosphorus fractions, the P sorption index (PSI), microbial biomass C (MBC), and dissolved organic C (DOC) in response to a single P application from three sources: composted steer manure (CSM), triple superphosphate (TSP), and mixed compost and triple superphosphate (MIX). Phosphorous sources, equal in P application rate (39 kg ha^{-1}), were applied in the winter of 2012, and soils were sampled in spring, summer, and fall. Labile inorganic P (Pi) (resin Pi + NaHCO$_3$ Pi), Fe/Al-Pi (NaOH Pi), and total P were significantly ($p < 0.01$) increased by P application, regardless of source. Calcium-Pi (HCl Pi), DOC, MBC, and pH were increased, and PSI was reduced, by compost addition. Net extractable Fe/Al-Pi increased during the study period. Moderately labile organic P (Po) (NaOH Po) declined, independent of fertilization, reflecting background soil P cycling. Calcium-Pi totals were low (mean 9 mg kg^{-1} for untreated plots), and other indicators of chemical weathering were high, suggesting an advanced stage of pedogenesis with respect to Walker–Syers P availability. In spite of advanced pedogenesis, labile Pi increased significantly from fertilization and remained elevated throughout the growing season. Results of this study inform P management in highly weathered, P-fixing soils experiencing P deficiencies.

Abbreviations: CSM, composted steer manure; DOC, dissolved organic C; Fe$_d$, pedogenic Fe oxides; Fe$_t$, total Fe; G–G, Greenhouse–Geisser; MBC, microbial biomass C; MIX, mixed compost and triple superphosphate; Pi, inorganic P; Po, organic P; PSI, P sorption index; SOC, soil organic C; TSP, triple superphosphate.

Core Ideas

- **Phosphorous fertility was evaluated in weathered volcanic soil in winegrape production.**
- **Seasonal Hedley P fractionations, P sorption, microbial biomass carbon, and DOC were investigated.**
- **Application of compost, alone or with TSP, reduced sorption and increased MBC.**
- **Labile Pi responded significantly to fertilization despite advanced pedogenesis.**
- **Significant labile Pi response in weathered soil may be due to low-Fe lithology.**

Phosphorus availability in soils is regulated by interactions between plant, microbial, and geochemical pools (Tiessen et al., 1984; Frossard et al., 2000, 2011; Oberson and Joner, 2005). As soils undergo pedogenesis, primary minerals deplete (including apatite) and Fe and Al (hydr)oxides increase, resulting in low P availability in highly weathered soils (Cross and Schlesinger, 1995; Frossard et al., 2000). Similarly, P is a common limiting nutrient in many volcanic soils due to retention by active Fe and Al compounds (Dahlgren et al., 2004; Simpson et al., 2011). In these P-fixing soils, P application requirements can be as much as five times plant demand to overcome the strong sorption properties (Simpson et al., 2011). Phosphorus deficiency in arable soils is overcome principally by the application of organic or inorganic fertilizers. Given the limited future availability of P fertilizer from global rock phosphate deposits (Cordell et al., 2009; Van Vuuren et al., 2010), understanding P cycling in diverse soils and strategies to improve P availability in P-fixing soils are critical research needs.

This study investigates growing season P dynamics and P management strategies in a strongly weathered volcanic soil in the Coast Ranges of northern

California's winegrape growing region. Winegrape acreage is rapidly expanding in this area, and P deficiency has been identified as the primary nutrient management challenge. In previous studies, in highly weathered or volcanic vineyard soils in California, P deficiency was corrected by saturating sorption sites with high rates of P fertilizer (monoammonium phosphate [MAP] and/or TSP) (Cook et al., 1983; Skinner et al., 1988). An alternative method to increase plant availability of added P in highly weathered or volcanic soils, while conserving P resources, is through the application of decomposed organic materials, such as green manures or composts, alone or in concert with Pi. The action of humic substances from organic amendments can chelate active Fe and Al, increase pH through ligand exchange, and/or competitively inhibit P sorption, all of which reduce sorption and increase P availability to plants (Hue, 1991; Iyamuremye et al., 1996a; Nziguheba et al., 1998; Hunt et al., 2007). Application of organic acids, composted manure, and decomposed organic materials was shown to reduce P sorption by highly weathered soils and Fe and Al (hydr)oxides (Hue, 1991; Iyamuremye et al., 1996a; Nziguheba et al., 1998; Hunt et al., 2007). Further, compost additions increase soil organic C (SOC) and MBC, improving several indicators of soil health (D'Hose et al., 2014). We hypothesize that application of composts, alone or in concert with Pi, will increase P availability and reduce the PSI through the action of added organic matter (Hunt et al., 2007).

Phosphorus fractionation schemes have been widely utilized to assess the fate of both applied P from fertilization and native P during pedogenesis (Walker and Syers, 1976; Hedley et al., 1982; Sharpley and Smith, 1985; Cross and Schlesinger, 1995; Negassa and Leinweber, 2009). In Headley fractionations of arable temperate soils, added Pi was recovered in operationally defined fractions: labile Pi (resin and $NaHCO_3$–extractable Pi), Fe/Al-Pi (NaOH extractable Pi), and Ca-Pi fractions (1 M HCl-extractable Pi) (Wagar et al., 1986; Blake et al., 2003; Negassa and Leinweber, 2009). In highly weathered tropical and subtropical soils, added Pi was primarily recovered in Fe/Al-Pi and occluded pools (concentrated HCl Pi or concentrated H_2SO_4–H_2O_2 Pi), with labile Pi accounting for a smaller portion of total Pi (Linquist et al., 1997; Buehler et al., 2002; Sims and Pierzynski, 2005; Negassa and Leinweber, 2009).

Fewer studies have investigated the fate and distribution of P fractions in weathered soils formed in Mediterranean type (xeric) climates of global importance for winegrape production (Jones et al., 2005). Iyamuremye et al. (1996b) added organic amendments to a xeric Haplohumult and recovered P in labile and Fe/Al-Pi fractions. A recent study of Hedley fractions in California soils (Xerorthents and Haploxeralfs) recovered significant P in Ca-Pi fractions in unfertilized soils, with long-term compost applications in excess of crop P demand significantly increasing P in all Hedley fractions (Maltais-Landry et al., 2015). In other soils within the xeric soil moisture regime, distribution of P fractions was dependent on leaching depth, activity of Ca^{2+}, degree of weathering, amounts and forms of Fe and Al (hydr) oxides, and parent material composition (Meixner and Singer, 1985; Smeck et al., 1994; Saavedra and Delgado, 2005).

Seasonal variability in P fractions will influence both plant availability and potential losses of P to the environment; understanding this variability will allow for the better timing of P applications. Many previous studies have investigated the effect of time on P fractions from long-term fertilization or land-use trials (Hedley et al., 1982; Sharpley, 1985; Sharpley and Smith, 1985; Blake et al., 2003; Shi et al., 2013; Maltais-Landry et al., 2015) or from short-term lab incubations and greenhouse studies (Hedley et al., 1982; Wagar et al., 1986; Iyamuremye et al., 1996b; Guo et al., 2000; Ochwoh et al., 2005). Fewer studies have investigated growing season changes in P fractions under field conditions. For example, He et al. (1997) attributed seasonal variability in labile and Fe/Al-Pi fractions to variations in soil moisture independent of biological processes, while Schwartz et al. (2011) attributed seasonal fluctuations in Pi to fluxes from microbial biomass. Nziguheba et al. (1998) applied organic and inorganic sources of P and fractionated soils after 2 and 16 wk and found diverse P sources behaved differently with respect to P fractions. Magid and Nielsen (1992) attributed changes in Pi to both microbial and Pi cycling as influenced by wet and dry cycles.

This study investigates the effect of P source, and the influence of time, on the distribution of P fractions in a strongly weathered volcanic soil in a xeric moisture regime. The primary objective of this study was to assess the fate of applied P in three forms (compost, TSP, and a compost/TSP mix) and the influence of time on P fractions, P sorption, and soil biogeochemical factors that may influence P dynamics (DOC, MBC, and pH) throughout the growing season. A strong impetus for this study was to clarify P cycling on highly weathered volcanic soils and inform efficient P fertilization.

MATERIALS AND METHODS
Site and Soil Characteristics

This study was conducted during the 2012 growing season in the Clear Lake Volcanic Field of Lake County, California. The Clear Lake Volcanic Field is an active, late Pleistocene, volcanic center in the northern California Coast Range, where regional deformation associated with the San Andreas Fault system activates mafic to felsic eruptions (Hearn et al., 1995). The study site is within the Red Hills American Viticultural Area (AVA), a delimited area of winegrape growing with distinguishing features including volcanic soils. The climate is Mediterranean with an average annual precipitation of 640 mm, occurring primarily from November to March, and a mean annual air temperature of 14°C. The site is cropped to Cabernet Sauvignon winegrapes. The soil investigated was a fine, halloysitic, mesic Ultic Palexeralf, formed in rhyolitic obsidian residuum, with evidence of pyroclastic materials in the upper 30 cm. Clay mineralogy is dominated by halloysite, kaolinite, and hematite with minor gibbsite (Wilson 2015). Soil characterization and selective dissolution data were determined as part of a separate pedologic study (Wilson 2015). Nonsequential selective dissolution in acid

Table 1. Selected physical and chemical characteristics of the dominant rooting zone soil horizons.†

Depth	Sand	Silt	Clay	C	N	Ex-K	Ex-Mg	Ex-Ca	Ex-Na	BS	CEC	pH‡
cm		%					mg kg^{-1}			%	cmol$_c$ kg^{-1}	
0–10	39	32	29	61.6	3.4	489	189	1528	10	95.5	11	4.9
10–30	37	36	28	17.9	0.7	335	286	1408	14	87.9	11.7	4.9

† BS, base saturation; CEC, cation exchange capacity; Ex-K, exchangeable K; Ex-Mg, exchangeable Mg; Ex-Ca, exchangeable Ca; Ex-Na, exchangeable Na.

‡ pH in 1:2 KCl measured in this study.

ammonium-oxalate and citrate-dithionite was used to characterize Fe, Al, and Si in pedogenic pools. Total elemental analysis (<2 mm) was performed on air-dried soils pulverized with a ball mill to a fine powder (<125 µm). Samples were analyzed for Fe, Al, Si, Ca, Mn, and Ti using a Thermo Scientific Xlt 600 XRF and the ex situ analysis procedure. Relevant characterization data for the dominant rooting zone are summarized in Table 1 and Table 2.

Phosphorus Sources, Experimental Design, and Sampling

Three phosphorus sources consisting of CSM, TSP, and an equivalent P content mix of TSP and CSM (MIX) were surface banded under the vine row, with the width of the band equivalent to the width of the vine row or 106 cm wide. Standard grower practice is to surface band TSP using a broadcast spreader with a shroud that concentrates application within the vine row. Application was in January 2012 at a rate of 39 kg total P ha^{-1}. Carbon inputs ranged from zero in the control and TSP treatments to 653.5 kg C ha^{-1} in the MIX treatment and 1307 kg C ha^{-1} in the CSM treatments. Table 3 provides a brief characterization of the CSM amendment. Treatments and control were replicated four times ($n = 16$) and plots were arranged in a randomized complete block design. Soil samples were taken in each plot to a depth of 15 cm at a location approximately 15 cm from drip irrigation emitters; three subsamples (7.5-cm cores) were composited from each plot to form each replicate. Soil samples were taken in spring (April), summer (August), and fall (November) to correspond to phenologically important times for winegrape nutrient management (bud break, veraison, and postharvest, respectively). Soil was stored field moist at 4°C prior to analysis. Field moist soils were gently ground and passed through a 2-mm sieve. Gravimetric soil moisture was determined by drying samples at 105°C for 24 h. All data are presented on an oven-dry weight basis.

Phosphorus Fractionation

Soil phosphorus was fractionated via the Hedley method (Hedley et al., 1982) as modified by Tiessen and Moir (1993). In brief, duplicate 0.5-g field-moist soil samples were sequentially extracted with anion-exchange resin strips (Western Ag Innovations, Inc., Saskatoon, SK, Canada), 0.5 M NaHCO$_3$, 0.1 M NaOH, and 1 M HCl, and digested with 90°C 12 M HCl. Extracts were centrifuged at 3000 rpm, filtered (0.45 µm polycarbonate mem-

brane filter; Millipore), and neutralized before analysis for reactive Pi using the ammonium molybdate-ascorbic acid method (Murphy and Riley, 1962). Total P (Pt) in NaHCO$_3$ and NaOH extracts was determined following digestion in 0.9 M H$_2$SO$_4$ and (NH$_4$)$_2$S$_2$O$_8$, and Po was determined by the difference between digested and undigested samples. A total of six operationally defined fractions were determined: labile Pi (resin Pi and NaHCO$_3$ Pi), labile Po (NaHCO$_3$ Po), moderately labile Po (NaOH Po), Fe/Al-Pi (NaOH Pi), Ca-Pi (1 M HCl Pi), and occluded Pi (90°C 12 M HCl) (Linquist et al., 1997; Blake et al., 2003). Total extractable P was calculated as the sum of all fractions.

Microbial Biomass Carbon and Dissolved Organic Carbon

Microbial biomass C was determined via chloroform fumigation and direct extraction within 24 h of collection (Vance et al., 1987). Duplicate, fumigated and nonfumigated soil samples were incubated at 4°C for 72 h and then extracted with 0.5 M K$_2$SO$_4$ for determination of DOC using a Phoenix 8000 UV-Persulfate total organic C (TOC) Analyzer (Teledyne-Tekmar, Mason, OH). Microbial biomass C was determined as the difference between fumigated and nonfumigated samples. Differences for in situ DOC concentrations among samples were based on the DOC concentration of nonfumigated samples.

Single Point Phosphorus Sorption and pH

A single-point 75-mg PO$_4$–P kg^{-1} sorption index (Sims, 2000) was used to characterize the P sorption potential of soils from all sampling times and treatments. Briefly, 20 mL of 75 mg PO$_4$–P kg^{-1} solution (as KH$_2$PO$_4$) was added to duplicate 1-g soil samples in 50-mL polypropylene tubes. Samples were shaken for 18 h and the extract was centrifuged, filtered (0.45-µm polycarbonate membrane filter; Millipore), and analyzed for reactive Pi using the ammonium molybdate-ascorbic acid method (Murphy and Riley, 1962). Sorbed P was expressed as milligrams P sorbed per kilogram soil. Soil pH was determined in 1:2 soil/1 M KCl following a 15-min equilibration period.

Table 2. Selective dissolution and total Fe (Fe$_t$) of the dominant rooting zone soil horizons.

Depth	Fe$_d$†	Al$_d$†	Fe$_o$‡	Al$_o$‡	Si$_o$‡	Al$_o$ + 1/2 Fe$_o$	Fe$_t$§	Fe$_o$/Fe$_d$	Fe$_d$/Fe$_t$
cm				g kg^{-1}				Ratio	
0–10	14.3	1.9	1.3	1.6	0.3	2.3	40	0.09	0.36
10–30	15.6	2	1.3	1.6	0.2	2.2	43	0.08	0.36

† Citrate dithionite extraction.

‡ Acid ammonium oxalate extraction,

§ Total Fe by X-ray florescence.

Table 3. Compost characterization: total values on an oven-dry weight basis.

Ash	C	N	P	K	Na	Ca	Mg	Fe	Cu	Mn	Zn	S	Cl
		g kg^{-1}							mg kg^{-1}				
589	228	18.2	6.8	25.6	7.9	32.6	10	9455	55.9	377	181	0.87	1.4

Statistical Analyses

All statistical analyses were performed in SAS version 9.3 (SAS Institute, 2009). General linear model ANOVA (PROC GLM) was used to evaluate P source effects on soil factors: phosphorus fractions, pH, MBC, DOC, and P sorption. If no significant time × treatment interactions were found, main effects (e.g., P source effects) were analyzed for each response variable by repeated measures (three sampling times) of the same experimental unit. In the case of significant time × treatment interactions, simple effects were analyzed for response variables at each sampling time. Mean separations for the main effect of P source were performed on least squares means using Tukey's honest significant difference (HSD) at a $p \leq 0.05$ significance level. To satisfy the conditions for the ANOVA, moderately labile Po data were log transformed and P sorption data were power transformed. For repeated measurements of soil factors with time (e.g., time effects), the SAS repeated statement was used, with time and soil measurements as factors. Mean separation for the effect of time on measured soil factors was performed using single df contrasts, and means reported are least squares means. For time effects, the more conservative Greenhouse–Geisser (G–G) adjusted p values were used, with significance indicated by G–G ≤ 0.05 for the effect of time on soil factors. A Pearson's correlation analysis was used to examine correlations among soil factors and P fractions.

RESULTS
Source and Time Effects on Phosphorus Fractions

Application of fertilizer P, regardless of source, significantly increased labile Pi, Fe/Al-Pi, and total extractable P, compared to unamended plots (Table 4). Compost treatment significantly increased the Ca-Pi pool ($p < 0.019$). Fertilization did not significantly influence occluded Pi, labile Po, or moderately labile Po fractions.

Repeated measures analysis (Table 4) revealed significant ($p < 0.01$) time effects on moderately labile Po and barely significant time effects on Ca-Pi ($p = 0.049$) but no time effects for any other P fraction. Amounts of moderately labile Po declined sharply from spring to summer and remained significantly lower through fall (Fig. 1a). This was independent of source effects, as indicated by a lack of significant time × source interactions for moderately labile Po. However, labile Po did demonstrate a significant ($p < 0.05$) time by source interaction (Table 4), indicating that different P sources behaved differently with respect to time for this fraction. Analysis of simple time effects for each treatment (data not shown), revealed a significant ($p < 0.05$) decline in labile Po from spring to fall in control plots, suggesting seasonal utilization of labile Po in the absence of fertilization. In contrast, TSP treatments showed a significant ($p < 0.05$) increase in labile Po from spring to fall, indicating a shift from Pi to Po in TSP-treated plots. Both compost containing treatments had no significant changes in labile Po from spring to fall.

The significant labile Pi and Fe/Al-Pi response in amended plots versus control plots during the growing season suggests a significant buildup in both labile Pi and Fe/Al-Pi from winter to summer following winter fertilizer application. To isolate the behavior of fertilizer P, P extracted in control plots was subtracted from P extracted in treated plots to control for antecedent soil P cycling (Schwartz et al., 2011). This revealed a significant buildup of Fe/Al-Pi from spring to fall in fertilized plots (Fig. 1b). Additionally, the distribution of applied P, expressed as the percentage of P extracted as a function of total net extractable P, showed a significant

Table 4. Summary of main effects and repeated measures analysis showing means of all P fractions for each P source and summary of *F* tests for source effects, time effects, and time × source interactions. Time effects refer to the repeated sampling dates during the growing season.

		Labile Pi†	Labile Po†	Fe/Al-Pi†	Moderately labile Po†	Ca-Pi†	Occluded Pi†	Total P$_{sum}$‡
Least Squares Means§				mg kg^{-1}				
	Control	48[b]	8[a]	96[b]	63[a]	9[a]	167[a]	392[b]
	CSM	172[a]	13[a]	155[a]	69[a]	24[b]	181[a]	615[a]
	MIX	186[a]	11[a]	147[a]	60[a]	15[a]	184[a]	602[a]
	TSP	158[a]	10[a]	165[a]	65[a]	11[a]	165[a]	573[a]
Main Effects					*P > F*			
	Source	<0.001	0.158	0.002	0.395	0.019	0.054	< 0.001
Time Effects¶								
	Time	0.208	0.187	0.414	0.004	0.049	0.116	0.566
Interactions								
	Time × source	0.082	0.045	0.239	0.113	0.183	0.153	0.145

† P fractions indicate Hedley P fractions: labile inorganic P (resin Pi + NaHCO$_3$ Pi), labile organic P (NaHCO$_3$ Po), Fe and Al inorganic P (NaOH Pi), moderately labile organic P (NaOH Po), Ca-Pi (1M HCl extractable inorganic P), and occluded Pi (12M HCl extractable Pi). Pi, inorganic P; Po, organic P.

‡P$_{sum}$, total P by sum of Hedley fractions.

§Means with different letters indicate significant differences by Tukey's honest significant difference (HSD). CSM, composted steer manure; MIX, mixed compost and triple superphosphate; TSP, triple superphosphate.

¶Repeated measures analysis of time effects and time × source interactions use Greenhouse–Geisser-adjusted p values.

($p < 0.01$; G–G < 0.05) seasonal increase in the proportion of applied P extracted as Fe/Al-Pi. This suggests a shift of fertilizer P to less available Fe/Al-Pi forms during the growing season (Fig. 1c).

Source and Time Effects on Biogeochemical Factors

Phosphorus source had a significant effect on all biogeochemical factors measured except soil moisture (Table 5). Compost application increased microbial biomass by more than 30% over both control and TSP applications ($p = 0.01$) (Table 5). The MIX treatment resulted in an apparent 18% increase in MBC, but it was not significantly different from the control, TSP, or CSM treatments. Dissolved organic C was significantly affected by P source, nearly doubling from control plots to compost treated plots ($p < 0.001$) (Table 5)

Compost-containing treatments (CSM and MIX) significantly reduced single-point P sorption (PSI) compared to the control (Table 5), and the MIX treatment significantly reduced PSI when compared to the TSP treatment and control. Triple superphosphate alone did not significantly reduce sorption, suggesting that the traditional application of TSP alone to saturate sorption sites will not significantly reduce sorption. Both compost and mixed-source treatments significantly increased pH above the TSP treatment and control ($p < 0.001$).

Significant time effects were observed for all measured biogeochemical factors, except soil moisture. There were significant increases in spring DOC levels in both treatments receiving compost (CSM and MIX) (Fig. 2). By summer, only the CSM treatment maintained elevated DOC levels, with DOC levels similar among all treatments by fall. Dissolved organic C increased with the proportion of compost applied. Microbial biomass C decreased from summer to fall across all sources ($p < 0.001$) (Table 5, Fig. 2). Both PSI ($p < 0.028$) and pH ($p < 0.001$) exhibited a significant time effect (results not shown).

Correlation among Phosphorus Fractions and Biogeochemical Factors

Correlations among P fractions and measured biogeochemical factors for all treatments and time points are presented in Table 6. Phosphorus sorption index showed a strong negative correlation with labile Pi and was negatively
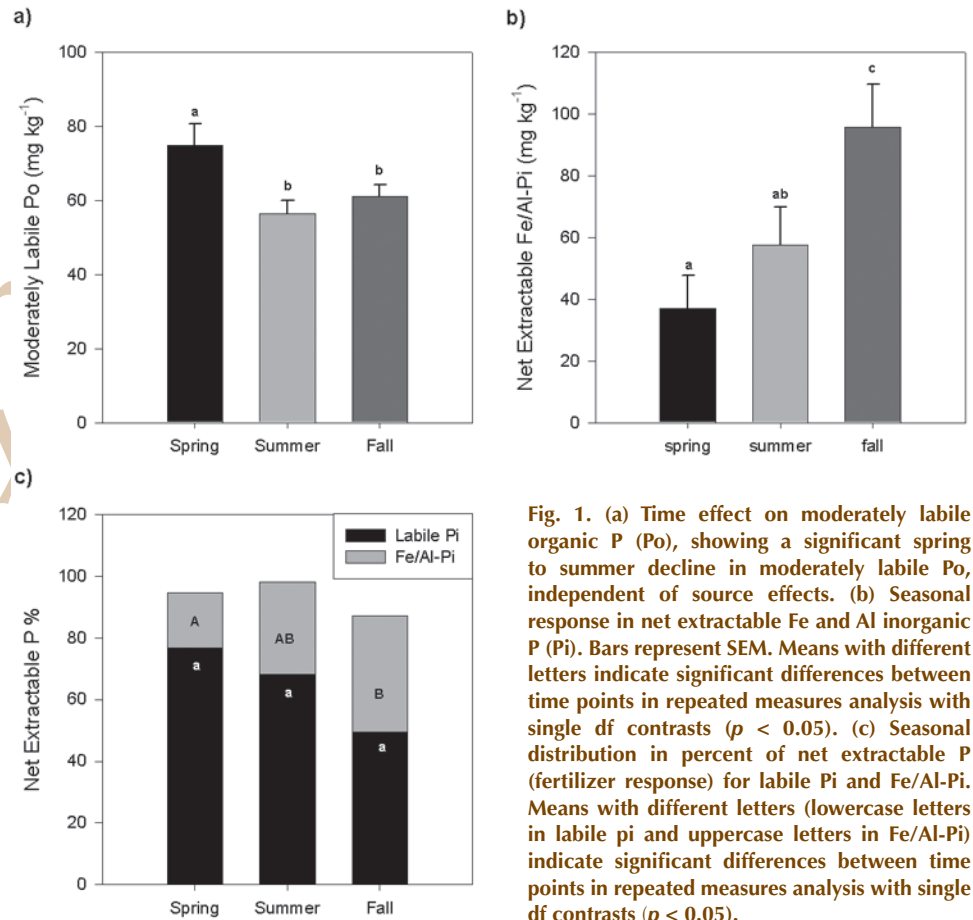
Fig. 1. (a) Time effect on moderately labile organic P (Po), showing a significant spring to summer decline in moderately labile Po, independent of source effects. (b) Seasonal response in net extractable Fe and Al inorganic P (Pi). Bars represent SEM. Means with different letters indicate significant differences between time points in repeated measures analysis with single df contrasts ($p < 0.05$). (c) Seasonal distribution in percent of net extractable P (fertilizer response) for labile Pi and Fe/Al-Pi. Means with different letters (lowercase letters in labile pi and uppercase letters in Fe/Al-Pi) indicate significant differences between time points in repeated measures analysis with single df contrasts ($p < 0.05$).

correlated with labile Po, Fe/Al-Pi, moderately labile Po, and Ca-Pi. Dissolved organic C was strongly correlated with pH and labile Pi. Microbial biomass C was weakly correlated with labile Pi, labile Po, and Ca-Pi. Phosphorus fractions were auto-

Table 5. Mean values for biogeochemical factors and summary of *F* tests for time, source, and time × source interactions.

	MBC†	DOC‡	PSI§	pH (KCl)¶	Soil Moisture#
Least squares means††			—mg kg⁻¹—		%
Control	77[b]	60[c]	333[a]	4.9[c]	13.5[a]
CSM	101[a]	110[a]	267[bc]	5.6[a]	14.2[a]
MIX	91[ab]	93[ab]	216[c]	5.3[b]	14.2[a]
TSP	67[b]	75[bc]	320[ab]	4.8[c]	13.3[a]
Main effects			—$P > F$—		
Source	0.010	0.000	0.002	<0.001	0.240
Time effects‡‡					
Time	<0.001	<0.001	0.028	0.001	0.038
Interactions					
Time × source	0.367	0.001	0.117	0.047	0.136

†Microbial biomass C by chloroform fumigation direct extraction.

‡DOC, dissolved organic C.

§Phosphorus sorption index by single-point 75 mg/L PO_4–P sorption.

¶ pH in 1:1 in 1 M KCl.

#Gravimetric soil moisture.

††Means with different letters indicate significant differences by Tukey's honest significant difference (HSD) at $p < 0.05$. CSM, composted steer manure; MIX, mixed compost and triple superphosphate; TSP, triple superphosphate.

‡‡Repeated measures analysis of time effects and time × source interactions use Greenhouse–Geisser adjusted p values.

correlated with each other, with strong correlations between Fe/Al-Pi and labile Pi, but there was no correlation ($p > 0.05$) between Fe/Al-Pi and moderately labile Po. To gain an understanding of antecedent soil P cycling factors, correlation analysis was performed exclusively on the unamended (control) plots (Table 7). For unamended plots, a highly significant ($r = 0.89$; $p < 0.001$) correlation was found between MBC and labile Po as well as significant correlations between MBC and Fe/Al-Pi and Ca-Pi fractions. Neither DOC nor pH was significantly correlated with any P fraction in unamended plots.

DISCUSSION

Phosphorus fertilization sources, applied in equivalent amounts of total P, behaved similarly with respect to labile and Fe/Al-Pi fractions, with application shifting the balance of P fractions from unamended plots dominated by occluded Pi and moderately labile Po forms to amended plots dominated by labile and Fe/Al-Pi forms. Recovery of applied P in labile and Fe/Al-Pi
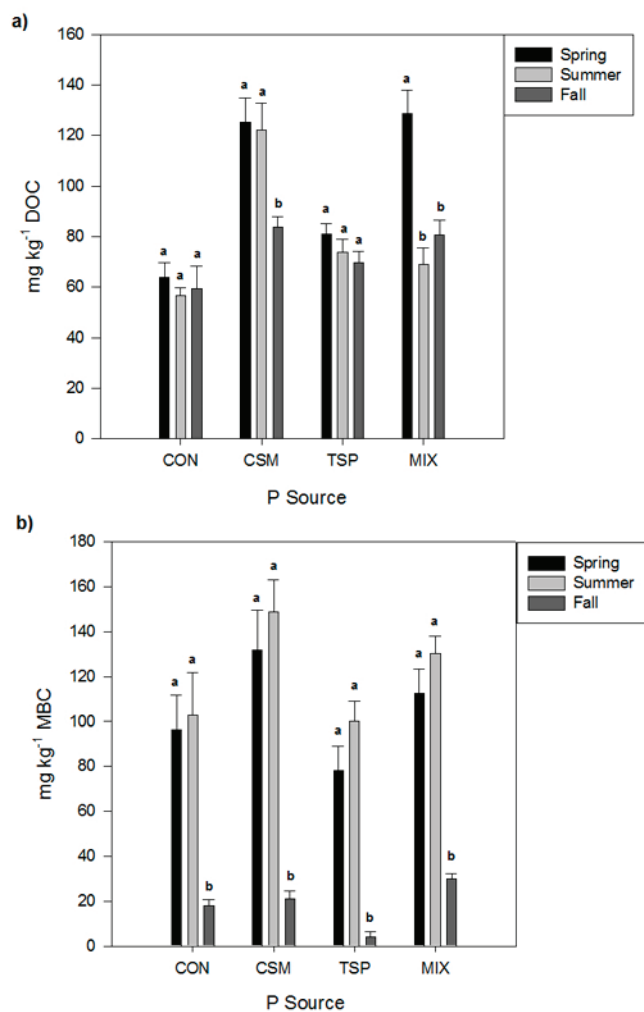

Fig. 2. (a) Simple effects showing seasonal variability of microbial biomass C (MBC) by treatment. (b) Simple effects showing seasonal variability of dissolved organic C (DOC) by treatment. Bars represent SEM. Within each treatment, means with different letters indicate a significant difference between time points in repeated measures analysis according to single df contrasts ($p < 0.05$). CON, control; CSM, composted steer manure; TSP, triple superphosphate; MIX, mixed compost and triple superphosphate.

is characteristic of fertilizer response (Hedley et al., 2005). The significant recovery of Pi in the labile fraction suggests that all sources, compost included, resulted in high Pi availability. This is in good agreement with Blake et al. (2003) who found that labile Pi (resin Pi plus $NaHCO_3$–Pi) accounted on average for 40% of total P values in soils amended with superphosphate or farmyard manure. Our findings demonstrated that the labile Pi fraction accounted for about 30% of total extracted P in fertilized soils.

The P fractionation as a whole extracted very little Ca-Pi (i.e., apatite); this suggests a strongly weathered soil as described by the Walker–Syers model (Walker and Syers, 1976). In their review of P fractionation in arable temperate soils, Negassa and Leinweber (2009) found between 87 and 990 mg kg^{-1} of Ca-Pi. Here, only 9 mg kg^{-1} of Ca-Pi was extracted in unamended soils, one to two orders of magnitude less than that found in the review of P fractionations for temperate soils by Negassa and Leinweber (2009). Additionally, in a recent P fractionation investigation of weakly weathered California soils (Xerorthents/Hapoxeralfs), Maltais-Landry et al. (2015) recovered similar amounts of total P but an order of magnitude more Ca-Pi (243–273 vs. 9–24 mg kg^{-1}). Further, fractionation of the highly weathered soils in this study (Palexeralf) extracted more than twice the amounts of both Fe/Al-Pi (96–165 vs. 40–79 mg kg^{-1}) and Po (71–82 vs. 31–37 mg kg^{-1}). The noted differences in Fe/Al-Pi, Ca-Pi, and Po compared to other temperate soils suggest a more advanced stage of pedogenic development with respect to P availability and Walker and Syers P evolution (Walker and Syers, 1976; Cross and Schlesinger, 1995). This was highlighted by the unamended soils where the largest P pool (42%) was the occluded fraction, with additional P recovered as Fe/Al-Pi and moderately labile Po.

Selective dissolution revealed a moderate to advanced degree of weathering, with about 36% of the total Fe (Fe$_t$) released to pedogenic Fe oxides (Fe$_d$) (Table 2). These Fe oxides were dominantly crystalline, with only 9% poorly crystalline Fe, as measured by the ratio of oxalate extractable Fe (Fe$_o$) to Fe$_d$ (Fe$_o$/Fe$_d$ = 0.09) (Table 2). These indicators of pedogenesis suggest a soil of moderate to high sorption potential; however, significant increases in the labile Pi fractions following fertilization contrasted with our hypothesis. This may be due, in part, to a lower potential to generate Fe oxides in the Fe-poor rhyolitic parent material despite the advanced stage of weathering. The influence of parent material on P fractions was suggested by Walker and Syers (1976) and was more recently addressed by Dieter et al. (2010) and Porder and Ramachandran (2013), who noted that parent material composition, especially the initial concentration of Fe in the parent material, can influence expected outcomes of P fractionation schemes. Iron concentrations in the rhyolitic soil investigated in this study were nearly three times lower in Fe$_t$ (40–43 g kg^{-1} Fe$_t$) compared to a soil formed from basalt in the same volcanic field (112 g kg^{-1} Fe$_t$) (Wilson 2015). In addition, the high degree of crystallization associated with the Fe oxides (e.g., dominated by hematite) results in lower P retention than that measured for poorly crystalline Fe-hydr(oxide) forms (Parfitt, 1978; Parfitt, 1989). Therefore, we attribute the

Table 6. Correlation coefficients (*r*) between P fractions and measured biogeochemical factors for all plots.

	P fractions†							
	Labile Pi	Labile Po	Fe/Al-Pi	Moderately labile Po	Ca-Pi	Occluded Pi	PSI‡	pH (KCl)§
PSI	−0.60***	−0.45**	−0.49***	−0.37*	−0.41**	−0.50***	1	−0.37*
DOC¶	0.54***	0.16	0.18	0.11	0.33*	−0.03	−0.14	0.76***
pH§	0.37*	0.16	0.03	0.17	0.40**	0.13	−0.37*	1
Soil moisture#	−0.1	−0.05	−0.27	0.14	−0.16	−0.07	−0.15	0.33*
MBC††	0.32*	0.34*	0.12	0.25	0.36*	0.19	0.02	0.29*

*Indicates correlation is significant at $p < 0.05$.

**Indicates correlation is significant at $p < 0.01$.

***Indicates correlation is significant at $p < 0.001$.

† P fractions indicate Hedley P fractions: labile inorganic P (resin Pi + $NaHCO_3$ Pi), labile organic P ($NaHCO_3$ Po), Fe and Al inorganic P (NaOH Pi), moderately labile organic P (NaOH Po), Ca-Pi (1 M HCl extractable inorganic P), and occluded Pi (12M HCl extractable Pi). Pi, inorganic P; Po, organic P.

‡ Phosphorus sorption index by single-point 75 mg/L PO_4–P sorption.

§ pH in 1:1 soil/1 M KCl.

¶ DOC, dissolved organic C.

Gravimetric soil moisture.

†† Microbial biomass C by chloroform fumigation direct extraction.

significant response in labile Pi from fertilization, in part, to lower Fe (hydr)oxide generation and more crystalline Fe (hydr) oxides with fewer sites available for sorption and occlusion of phosphate. This suggests that the distribution of P fractions, and the response to applied P, is dependent not only on the degree of weathering, and the evolution of P forms from Ca-Pi to occluded P as suggested by the Walker–Syers model, but also on the absolute amount of P, Ca, and Fe in the parent material and the crystallinity of Fe and Al (hydr)oxides (Walker and Syers, 1976; Anderson, 1988; Porder and Ramachandran, 2013).

In agreement with soils of a similar stage of weathering, applied P did shift to less labile forms (labile Pi to Fe/Al-Pi pools) over the 11-mo study period (Ochwoh et al., 2005). A significant increase in net extractable Fe/Al-Pi was observed during the duration of the study, with the amount of Fe/Al-Pi increasing with time, which is in agreement with an Alfisol and Ultisol in South Africa (Ochwoh et al., 2005). However, Ochwoh et al. (2005) observed substantial increases in Ca-Pi, in contrast with our findings, wherein Ca-Pi formation was not a significant sink for applied P, again suggesting that initial parent material composition, in this case low Ca^{2+}, influences extracted P forms. Increases in Ca-Pi in soils receiving long-term manure applications are associated with shifts from Ca phosphates from apatite-type minerals to tricalcium and octacalcium phosphates (Sharpley et al., 2004). Further, P in composted cattle manures is dominantly in labile Pi and Ca-Pi forms (Takahashi, 2013). Thus, in a soil derived from parent material lacking significant apatite-type minerals (Maher and Thorrold, 1989; Porder and Ramachandran, 2013), observed increases in Ca-Pi from CSM application were likely due to inputs of calcium phosphates derived from the compost.

Observed declines in moderately labile Po from spring to summer were likely due to mineralization of Po. Since there were no significant source effects on Po fractions, this is likely a result of background C and P cycling processes independent of current season fertilizer application effects (Sharpley, 1985). Magid and Nielsen (1992) observed that moderately labile Po declines from spring to late summer/early fall, which is consistent with these findings.

Table 7. Correlation coefficients (*r*) between P fractions and measured biogeochemical factors for unamended plots.

	P fractions†							
	Labile Pi	Labile Po	Fe/Al-Pi	Moderately labile Po	Ca-Pi	Occluded Pi	PSI‡	pH (KCl)§
PSI	−0.655*	−0.506	−0.659*	−0.842*	−0.769**	−0.347	1	−0.271
DOC¶	−0.23	−0.432	−0.311	−0.453	−0.538	−0.539	0.289	−0.094
pH§	−0.267	0.108	−0.237	0.411	0.089	−0.056	−0.271	1
Soil moisture#	0.092	0.135	0.052	0.403	0.122	0.137	−0.288	0.356
MBC††	0.662	0.892***	0.776**	0.54	0.683*	0.601	−0.25	−0.236

*Indicates correlation is significant at $p < 0.05$.

**Indicates correlation is significant at $p < 0.01$.

***Indicates correlation is significant at $p < 0.001$.

†P fractions indicate Hedley P fractions: labile inorganic P (resin Pi + $NaHCO_3$ Pi), labile organic P ($NaHCO_3$ Po), Fe and Al inorganic P (NaOH Pi), moderately labile organic P (NaOH Po), Ca-Pi (1 M HCl extractable inorganic P), and occluded Pi (12 M HCl extractable Pi). Pi, inorganic P; Po, organic P.

‡Phosphorus sorption index by single-point 75 mg/L PO_4–P sorption.

§ pH in 1:1 in 1 M KCl.

¶DOC, dissolved organic C.

#Gravimetric soil moisture.

††Microbial biomass C by chloroform fumigation direct extraction.

Phosphorus Source Effects on Biogeochemical Factors

The most significant treatment effects were observed in biogeochemical factors, rather than P fractions, driven by the addition of organic C in composts. Compost application increased pH, which is commonly observed (Hue, 1992; Iyamuremye et al., 1996a; Sharpley et al., 2004). Compost containing treatments reduced PSI and increased MBC and DOC. A combination of anion sorption site saturation with added phosphate, competitive inhibition of anion sorption sites by organic acid anions, reduction of Al^{3+} activity through chelation, and reductions in exchangeable acidity via exchange of surface hydrogen by Ca^{2+} likely contributed to reduce sorption in soils receiving compost (Iyamuremye et al., 1996a; Nziguheba et al., 1998; Hunt et al., 2007; Jiao et al., 2007). Labile Pi showed a strong correlation with PSI, as anion sorption sites occupied with labile Pi inhibited additional sorption. However, if saturation of P sorption sites with Pi was the only mechanism for reduced P sorption, we would expect that the three treatments, all equivalent in amount of P applied, would also be equivalent in terms of the mass of P sorbed. However, TSP treatments were not different than controls in mass ($mg\ kg^{-1}$) of P sorbed, despite having substantially greater labile Pi, which is in contrast with compost-containing treatments that also increased labile Pi but significantly reduced P sorption. Thus, the combined effects of increases in pH, chelation of active-Al^{3+}, and competitive inhibition of anion sorption sites by organic acid anions, all factors attributable to added organic C, potentially contributed to the observed reduction of single-point P sorption in soils receiving composts.

As expected, MBC was stimulated by compost addition. The lack of correlation between MBC and labile Pi suggests that in the presence of added Pi, microbial C cycling is decoupled from Pi cycling in the presence of added Pi from fertilization. However, when looking only at control plots, correlations between MBC and P fractions were much more significant. Strong correlation between MBC and labile Po, infers interdependence between MBC and labile Po in unamended plots (Stewart and Tiessen, 1987). Therefore, we speculate that, within the time frame of this study (11 mo) and at these application rates, MBC plays a proportionally less important role in P reactions in fertilized plots, but in unfertilized plots, MBC may strongly influence P dynamics.

CONCLUSIONS

Equal amounts of total P were applied in winter to a strongly weathered rhyolitic obsidian soil in the form of compost, TSP, or a combination of the two, and the soil was sampled in the spring, summer, and fall. Compost application increased MBC, DOC, and pH and reduced phosphorous sorption (PSI), confirming that applying compost alone or in concert with TSP will favorably increase the microbial biomass and DOC while decreasing P sorption. Application of a mix of compost and Pi favorably decreases P sorption and increases microbial biomass and is thus a recommended management strategy to increase P

availability in strong P-fixing soils. We hypothesized that added Pi would be recovered in Fe/Al-Pi and occluded fractions, owing to the advanced pedogenesis of the soil (Palexeralf). Little Ca-Pi was recovered during fractionation, supporting advanced pedogenesis with respect to Walker–Syers P availability. However, added Pi was recovered predominantly in labile Pi fractions, with additional recovery in Fe/Al-Pi fractions, with no fertilization-induced change to Po or occluded P fractions. Increases from fertilization were greatest in the labile Pi fraction, and applied Pi was not rapidly transformed to occluded forms, contrasting with our hypothesis and common results in weathered tropical soils. This suggests that in soils derived from low-Fe parent materials, such as rhyolite, sorption and occlusion of applied P will be low and P availability will remain high following fertilization despite the advanced stage of pedogenesis. Therefore, both the expected outcome of P fractionations (Ca-Pi vs. Fe/Al-Pi forms) and the response to fertilization may be more significantly influenced by initial parent material composition (especially Fe and Ca content) in highly weathered soils than commonly attributed.

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