Influence of Recent Acidification on Available Phosphorus Indices and Sorption in Washington State Soils

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Gradual soil acidification in the Columbia Basin (CB), Washington State, brings into question the validity of the traditional sodium bicarbonate (NaHCO$_3$) extractable P (Olsen phosphorus, OP) method for making fertility recommendations. Our objectives were to determine how soil pH influences the soil P indices after fertilization and sorption capacity of soils with similar levels of available P at varying soil pH levels. Collected soils represented a range of pH (5.2–8.4) and OP (3–40 mg P kg^{-1}). Soils were treated with 0, 44, 88, or 132 kg P ha^{-1} and incubated at 20°C for 6 wk. Subsamples were collected and analyzed for available P with OP, Bray P1 (BP1), modified Morgan (MMP), Mehlich-III (M3P) extractions. Additionally, P sorption isotherms were performed on untreated soils. Analysis of variance indicated that the slope of the change in extractable P was generally not significant ($\alpha = 0.05$) for OP, BP1, MMP, and M3P given a P addition for several soils at different pH but of similar initial available P levels. Grouping soils by pH showed differences in slope with OP (0.27–0.34) yielding the least difference and MMP (0.12–0.33) the greatest related to soil pH. Langmuir parameters of *b*, sorption capacity, and *k*, binding energy, showed variability for soils grouped by initial available P for several pH levels. Grouped by pH there was little difference in P sorption in relation to soil pH. Continued use of OP on acidified soils of the CB appears to be a viable soil testing strategy.

Abbreviations: CB, Columbia Basin; CEC, cation exchange capacity; OP, Olsen phosphorus; MMP, modified Morgan phosphorus; BP1, Bray P1; M3P, Mehlich-III phosphorus; MAP, monoammonium phosphate.

The CB of Washington State is renowned for the production of a variety of crops including potato (*Solanum tuberosum* L.), corn (*Zea mays* L.), wheat (*Triticum aesitvum* L.), and onion (*Allium cepa* L.). Nutrient removal by these crops is significant and addition of fertilizers is necessary to maintain adequate nutrition for optimum crop production. For example, a high yielding potato crop (90 Mg ha^{-1}) may remove more than 250 kg N ha^{-1} (Kleinkopf et al., 1981).

The most commonly deficient nutrient in plant production is N. Nitrogen is usually applied in ammoniacal forms because of availability and low cost. One consequence of using ammonia fertilizers is that as 1 mole of NH$_4^+$ nitrifies, 2 moles of H$^+$ are produced (Helyar, 1976). Continual use of ammonia-based fertilizers accelerates soil acidification beyond natural N-cycle processes (Mahler and Harder, 1984; Rasmussen and Rohde, 1989).

Soils of the CB, before anthropogenic influence, have a pH of 6.1 to 9.0 (Cochran et al., 2006; Soil Survey Staff, 2011). All soil pH data in this work refer to a 1:1 soil/water measurement. However, recently observed soil pH values <5.0

are not uncommon (Stevens et al., 2003). The change in soil pH raises the question of whether the OP extraction (Olsen et al., 1954) can be used reliably on low pH soils because the method was developed for neutral to alkaline soil to determine plant available P, a nutrient that varies greatly in phyto-availability depending on soil pH. The OP method, using $NaHCO_3$ to extract plant available P, is most commonly used for calcareous soils where P complexes with Ca.

Washington State has established P fertilizer recommendations based on the results of the OP method. Soil testing laboratories continue to use the OP extraction on recently acidified soils regardless of soil pH (R. Miller, personal communication, 2011). Sharpley (2000) indicated that the reagents used in the extraction of available P must be suited to the particular soil properties to be successful. Further, Holford (1980) suggests that the P test used should have good correlation with plant yield or P uptake across varying soil chemical conditions. Alternative extraction methods of plant available P for acidic soils are: BP1 (Bray and Kurtz, 1945), M3P (Mehlich, 1984), MMP (McIntosh, 1969), or ion exchange resins. Data for use in CB soils and crops are lacking for these methods; however, OP has shown promise (Kuo, 1990) in extracting plant available P from low pH soils. For this reason the OP method continues to be the preferred method for CB soils regardless of soil pH.

The extraction procedures for P in this study are availability indices. In other words, an amount of P that is assumed to be directly related to plant availability is extracted and is dependent on soil properties and plant requirements. Phosphorus sorption isotherms not only take into account the intensity of extracted P, but also the capacity of the soil to provide P for plant production (Singh et al., 2005). Determination of plant P requirements based on data from sorption isotherms has been demonstrated to be more accurate than using only conventional P soil tests (Hue and Fox, 2010; Klages et al., 1988). The Langmuir equation is often fitted to P sorption isotherm data in environmental applications because the maximum sorption (b), and binding energy (k), can be calculated and analyzed statistically. The Langmuir sorption maximum has been shown to be well correlated with exchangeable Ca (Tunesi et al., 1999) as well as Fe and Al oxides (Zhang et al., 2005), parameters often associated with soil pH.

The objectives of this research are (i) to determine how soil pH influences the results of the soil P indices after fertilization for soils with similar levels of plant available P and (ii) to evaluate the P sorption capacity of soils with similar levels of available P at varying soil pH levels.

MATERIALS AND METHODS

To evaluate plant available P extractions, 10 CB soil samples were collected in bulk from 0 to 30 cm representing three soil pH ranges and three OP levels within each pH range (Table 1). Soil pH was determined on a 1:1 soil/water slurry using a Thermo Orion EA 940 pH meter (Waltham, MA). The three soil pH ranges were: L (<6.0), M (6.2–6.8), and H (>7.0). The three initial P ranges (measured as OP) were: 1 (<10), 2 (15–25), and 3 (>30) mg P kg^{-1}. The OP was used as the basis for comparisons because it is the preferred test method in the region from which the soils were collected. A CB soil with low pH (<6.0) and low OP (<10 mg kg^{-1}) was not located. Soil 1 represented a native (not influenced by agricultural activities) soil. In addition to available P, soils were analyzed for total P (Kuo, 1996), calcium carbonate ($CaCO_3$) (Loeppert and Suarez, 1996), organic matter (OM), exchangeable Al, Ca, K, Mg, and Na, and cation exchange capacity (CEC) by ammonium replacement (Gavlak et al., 2003). Total bases and percent base saturation (sum of Ca, Mg, K, and Na divided by CEC) were calculated.

Soil Incubation

To determine how extraction method results differ across soil pH ranges following fertilization, 250 g of each soil was weighed into 450 mL polystyrene cups, fertilized with 0, 21, 42, 63 mg P kg^{-1} (which represent field application levels of 0, 44, 88, or 132 kg P ha^{-1}) added as monoammonium phosphate, MAP (11–52–0) in four replicates. Because no plants were to be grown, the N was not adjusted to reach the same N application rate across treatments. Soils were mixed thoroughly, moistened to slightly less than saturation, and maintained with deionized water at 20°C for 6 wk. At 2 wk intervals, 50-g subsamples were taken from each cup, air dried, and ground to pass a 2-mm sieve.

Subsamples from 2, 4, and 6 wk time intervals were extracted by OP, BP1, MMP, and M3P (Gavlak et al., 2003). Phosphorus determination was performed colormetrically (Milton Roy Spectronic 401, Ivyland, PA; Watanabe and Olsen, 1965) for OP, BP1, and MMP. The M3P was not analyzed colormetrically because the color development is prohibitively rapid. The M3P analysis was performed with a PerkinElmer DV3300 Optima Inductively Coupled Plasma Atomic Emission Spectrophotometer (ICP–AES, PerkinElmer, Waltham, MA).

Analysis of variance was conducted for available P by incubation time, and available P by soil pH interactions using PC SAS system for Windows version 9.1 (SAS Institute, Cary, NC). Means were calculated with PROC GLM. Data were considered significantly different at $P \leq 0.05$ according to Tukey's method of mean separation within PROC GLM. Multiple contrasts were made using Bonferroni's method.

Phosphorus Sorption Isotherms

Phosphorus sorption isotherms were determined on soils 1 to 10 by adding P solutions of 0, 6.45, 16.13, 32.26, 161.3, and 323 μmol P L^{-1} as KH_2PO_4 in 0.01 M $CaCl_2$ at a 1:25 soil/solution ratio (Nair et al., 1984) into 50 mL plastic centrifuge tubes. After end-over-end shaking for 24 h at 25°C the samples were filtered (0.45 μm) and analyzed colormetrically for P concentration by the same method used for the soil incubation samples. This method has been used by other researchers studying P sorption (Sharpley, 2003; Fang et al., 2002; Young and Ross, 2001; Paulter and Sims, 2000). The difference between P added and the P remaining in solution was calculated as the amount of P sorbed. This analysis was conducted on three replicates of untreated soil

Table 1. Soil characteristics of 10 Columbia Basin bulk samples. Included are the soil site and taxonomic descriptions, pH, total P, and extractable P levels (OP, MMP, BP1, M3P)† and several other chemical parameters. Also given are rankings for pH (H = >7.0, M = 6.2–6.8, and L = <6.0) and initial P (measured as OP) (1 = <10, 2 = 15–25, and 3 = >30 mg P kg⁻¹).

Soil ID	Description	Taxonomy	pH	pH rank	Initial P rank	OP	MMP	BP1	M3P	Total	CaCO₃	OM	Al	Ca	Mg	Na	Total bases	CEC	Base saturation
						—mg P kg⁻¹—					—%—			—mg kg⁻¹—			–cmol⁺ kg⁻¹–		%
1	Native	Winchester sand, mixed, mesic Xeric Torripsamments	7.2	H	1	7	4	12	8.0	1023	1.2	0.6	0.68	1010	194	29.9	7.6	8.4	90
2	Agricultural	Timmerman course sandy loam, sandy, mixed, mesic Xeric Haplocambids	6.2	M	2	20	6	23	73.7	943	1.2	1.2	2.33	1270	249	50.6	8.9	12	74
3	Lightly manured agricultural	Royal loamy fine sand, coarse-loamy, mixed, superactive, mesic Xeric Haplocambids	7.7	H	3	33	42	37	63.4	1100	3.5	1.7	0.68	3300	200	69.0	19.7	14.2	139
4	Agricultural	Warden silt loam, coarse-silty, mixed, superactive, mesic Xeric Haplocambids	5.9	L	2	25	7	40	45.7	647	0.7	1.0	0.82	1420	254	55.2	13.5	11.9	88
5	Garden	Strat Stoney loam, loamy-skeletal, mixed, superactive, mesic Calcidic Haploxerolls	7.9	H	2	20	24	25	40.1	482	1.0	1.7	0.83	1240	327	190.9	11.9	11.6	103
6	Pasture	Outlook very fine sandy loam, coarse-silty, mixed, superactive, calcareous, mesic Aeric Halaquepts	6.4	M	2	15	4	9	17.9	707	1.2	1.4	0.50	960	450	234.6	10.6	10.4	102
7	Pasture	Outlook very fine sandy loam, coarse-silty, mixed, superactive, calcareous, mesic Aeric Halaquepts	8.4	H	1	5	7	1	11.3	661	8.1	2.3	0.18	3680	956	112.7	27.3	15.8	173
8	Agricultural	Warden silt loam, coarse-silty, mixed, superactive, mesic Xeric Haplocambids	5.2	L	3	40	8	49	100.0	816	0.3	0.9	14.33	920	206	34.5	7.2	10.1	71
9	Agricultural	Ephrata gravelly sandy loam, coarse-loamy over sandy or sandy-skeletal, mixed, superactive, mesic Xeric Haplocambids	6.7	M	1	3	1	8	9.5	633	1.2	1.4	1.02	1500	242	59.8	10.0	12.4	81
10	Agricultural	Warden silt loam, coarse-silty, mixed, superactive, mesic Xeric Haplocambids	6.5	M	3	32	21	56	70.2	617	1.8	1.1	1.38	2000	370	20.7	14.1	16.4	86

† OP = Olsen phosphorus (NaHCO₃), BP1 = Bray P-1 (HCl + NH₄F), MMP = Modified Morgan phosphorus (NH₄OAc), M3P = Mehlich-III phosphorus (HOAc + NH₄NO₃ + NH₄F + HNO₃ + EDTA); OM, organic matter; CEC, cation exchange capacity.

sample and means presented. Pooled sorbed P (Y = mg P kg^{-1}) data separated only by soil pH and equilibrium P concentration (X = mg P L^{-1}) were fitted using the Langmuir isotherm equation:

$$Y = \frac{bkX}{1+kX} \qquad [1]$$

where Y is the sorbed concentration (mg kg^{-1}), b is the maximum sorption capacity of the soil (mg kg^{-1}), k is the binding constant of the Langmuir equation (L mg^{-1}) and X is the equilibrium concentration (mg L^{-1}). Best fit lines for the Langmuir equation were obtained by minimizing the sum of errors squared of the actual versus modeled isotherm data using the solver tool in Microsoft Excel (Redmond, WA) to adjust the values of b and k. Means of b and k were compared using Bonferroni's a priori method using the PROC GLM procedure in SAS. Correlation statistics were obtained using PROC REG for the relationship between observed and the modeled Langmuir isotherm.

RESULTS AND DISCUSSION
Soil Incubation

To test the hypothesis that soil pH influences the available P index following fertilizer application, change in tested available P over the 0 kg P ha^{-1} treatment was plotted against P rate for each soil at 2, 4, and 6 wk of incubation time. The slope of the line from the plotted data heretofore references the expected change in soil test available P at a given P fertilizer rate. The change in tested P following fertilizer application was comparable to the findings of other researchers (Yang and Jacobsen, 1990; Rubio et al., 2008). Analysis of variance revealed that line slopes were similar for individual soils within an extraction method and showed no change in available P from Week 2 to Week 6 of incubation (data not shown). Therefore, comparisons were made pooling data from Weeks 2, 4, and 6.

Slopes created from the relationship between the changes in extractable P following fertilizer application were analyzed using Bonferroni a priori contrasts for similar initial P level soils across the pH range represented (Table 2) and suggest that, on average,
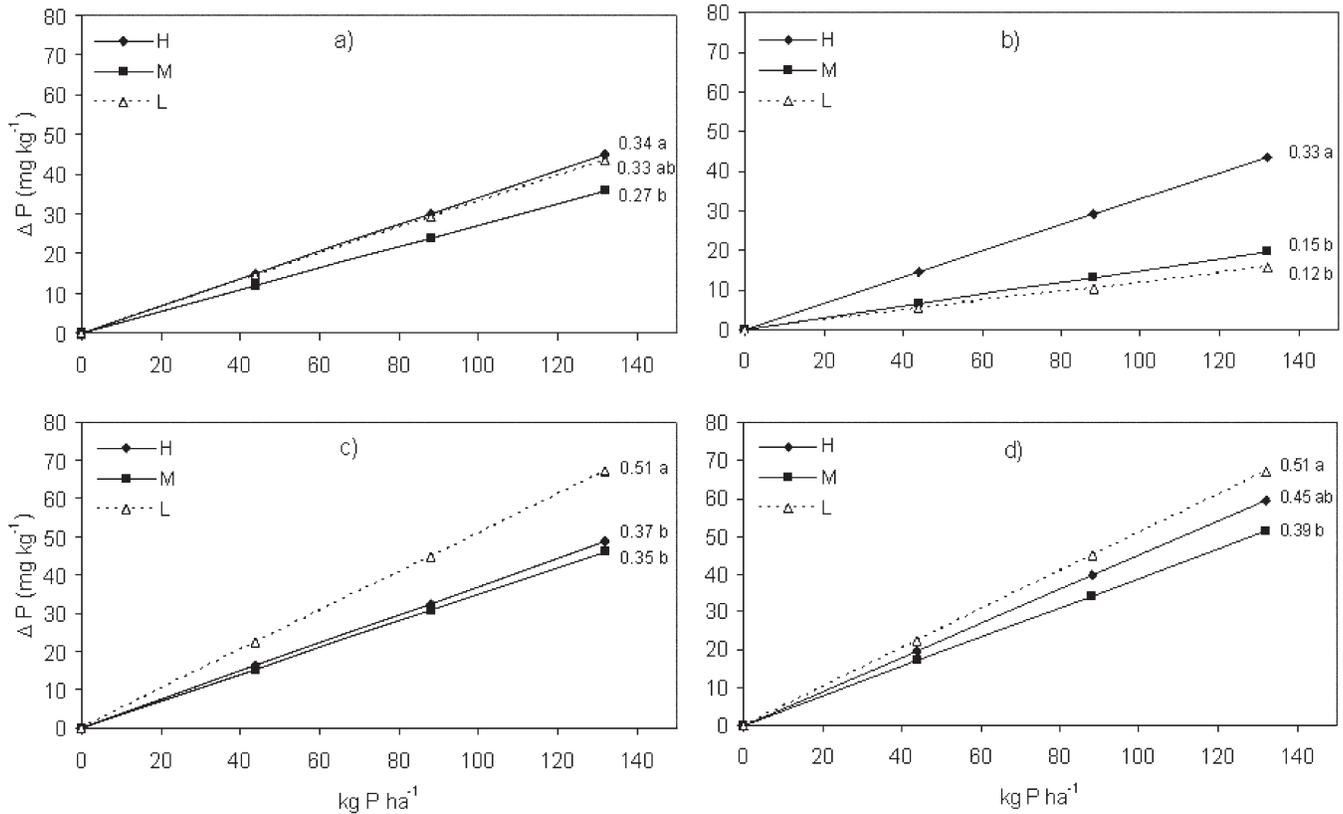
within an extractable P class (low, medium, or high available P), soil P extraction is not affected by pH with any of the tested methods. The single exception was with the MMP extraction of a low P soil where the slope for the higher pH soil was significantly ($P = 0.03$) higher than the medium pH soil.

If the P response slopes are pooled only by soil pH, a trend of higher slopes with higher pH can be observed for OP and MMP, while the opposite (lower soil pH, higher slope) occurs for M3P and BP1 extractions (Fig. 1). The higher slopes with higher pH for OP are the result of the NaHCO$_3$ extractant. As the pH of the soil increases, there is greater tendency to form CaCO$_3$ precipitates, leaving greater amounts of formerly Ca bound P in solution. For M3P and BP1 the likely reason for lower slopes occurring with high pH soils is because Ca (Blanchar and Caldwell, 1964) and CaCO$_3$ (Randall and Grava, 1971) have a neutralizing effect on acid extractants resulting in an incomplete P extraction. Previous research (Nesse et al., 1988) comparing non- and violently effervescent (>7% calcium carbonate equivalent) soils demonstrated that the reliability of acid extractions (M3P and BP1) decreases significantly with increasing effervescence.

Of the four extraction methods tests, OP shows the least amount of slope variability (0.27—0.34) across soil pH while MMP (0.12—0.33) exhibited the greatest differences. Similar consistency in soil test P increases in relation to P fertilization with the OP extraction has been demonstrated on low pH soils (Davenport et al., 2009).

Phosphorus Sorption Isotherms

Phosphorus sorption isotherms were conducted to test whether differences in sorption capacity could be attributed to soil pH. Measured isotherm data was plotted with the best fit lines for the Langmuir isotherm equation for soils 1 to 10 and the results appear in Fig. 2. The observed data for all soils were well correlated ($P < 0.001$) with the modeled Langmuir isotherm (Table 3). Other researchers (Olsen and Wanatabe, 1957; Del

Table 2. Average slope values (n = 12) of the lines for change in soil extractable P vs. P addition rate using four different P extraction techniques (OP, BP1, MMP, and M3P)†. Levels of significance for slope comparison using the Bonferroni a priori contrasts for soils of similar initial P level (<10 = 1, 15–25 = 2, >30 mg P kg^{-1} = 3) across high (>7.0, H), medium (6.2–6.8, M) and low (<6.0, L) pH.

Initial P rank	Soil	pH rank	OP	BP1	MMP	M3P
					Slope	
1	1,7	H	0.34	0.14	0.29	0.46
	9	M	0.22	0.28	0.10	0.31
2		P value	0.25	0.87	0.03	0.82
	5	H	0.45	0.65	0.46	0.64
	2,6	M	0.25	0.31	0.14	0.34
	4	L	0.33	0.50	0.12	0.49
3		P value	0.19	0.44	0.52	0.64
	3	H	0.43	0.40	0.37	0.48
	10	M	0.37	0.63	0.27	0.57
	8	L	0.36	0.52	0.11	0.53
		P value	0.17	0.60	0.75	0.92

† OP = Olsen phosphorus (NaHOC$_3$), BP1 = Bray P-1 (HCl + NH$_4$F), MMP = Modified Morgan phosphorus (NH$_4$OAc), M3P = Mehlich-III phosphorus (HOAc + NH$_4$NO$_3$ + NH$_4$F + HNO$_3$ + EDTA).

Fig. 1. Slope analysis of data pooled for all initial P (as Olsen phosphorus [NaHOC$_3$], OP) levels within soil pH [H = >7.0 (n = 36), M = 6.2–6.8 (n = 36), L = <6.0 (n = 18)] for (a) OP, (b) modified Morgan phosphorus (NH$_4$OAc) (MMP), (c) Bray-P1 (HCl + NH$_4$F) (BP1), and (d) Mehlich-III P (HOAc + NH$_4$NO$_3$ + NH$_4$F + HNO$_3$ + EDTA) (M3P) following 6-wk incubation with fertilizer for Soils 1 to 10. Slopes with different letters are statistically significant at (α = 0.05).

Bubba et al., 2003; Whalen and Chang, 2002) have also found the Langmuir model appropriate for sandy and calcareous soils.

The general shape of the curve was similar for most soils with Soils 6 and 7 being outliers (Fig. 2a, 2b). These two soils draw attention to the fact that despite similarities between many of the soil P sorption capacities, a generalization for all soils in the region is not realistic. Previous research has shown that several factors other than soil pH can account for differences in P sorption capacity of soil: soil surface area (Olsen and Wanatabe, 1957; Del Bubba et al., 2003; Casson et al., 2006); Ca and Mg content of the soil as well as porosity, bulk density, hydraulic conductivity (Del Bubba et al., 2003); and soil organic matter

content (Kang et al., 2009). These soils belong to the Outlook series and are distinguished from the other soils in this project by being fine-textured alluvial soils with a high water table. The fine fraction of the soil texture likely contributes to the large increase in sorption capacity compared to the other soils.

Table 3 gives the mean separation for the binding energy, k, and the sorption maximum, b, for the soils separated by initial available P level. With exception of Soil 5, the lowest values of k were observed in the highest initial P soils. Overall these high P soils also showed the greatest sorption maximum (excluding Soils 6 and 7). This suggests that the soils with low and moderate
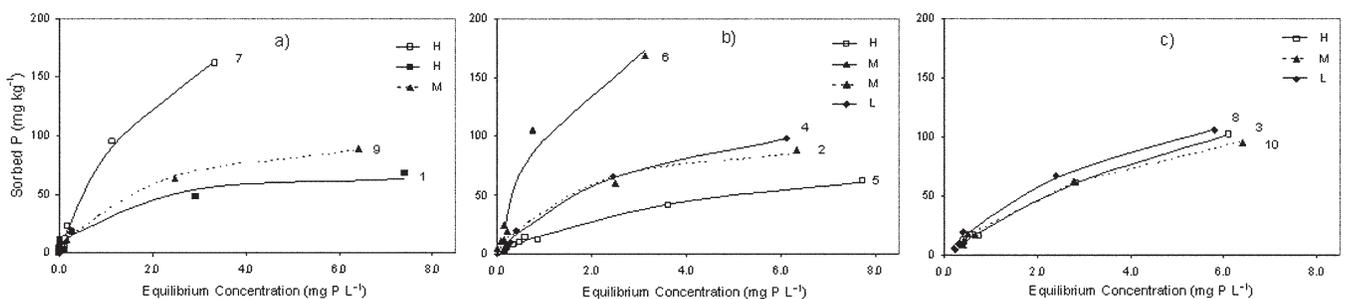
Fig. 2. Best fit lines for Langmuir P sorption isotherms conducted for Soils 1 to 10 (n = 3) grouped by initial P (as Olsen phosphorus, OP) level; (a) <10 mg kg^{-1}, (b) 15 to 25 mg kg^{-1}, and (c) >30 mg kg^{-1}. High (>7.0, H), medium (6.2–6.8, M), and low (<6.0, L) pH soils are shown where applicable. Sorbed P is calculated as the difference between the P added and the P remaining in solution following incubation and 24 h shaking. The points are the collected data.

Table 3. Mean separation for sorption maximum, *b* and binding energy, *k* for the Langmuir isotherm constants performed on Soils 1 to 10 (*n* = 3). Soils are grouped by H (>7.0), M (6.2–6.8), and L (<6.0) pH within an initial P (as OP) level (<10 = 1, 15–25 = 2, > 30 mg P kg⁻¹ = 3). Means with different letters for constants within an initial P group are significant (α = 0.05). Correlation and significance of observed vs. the modeled Langmuir isotherm are also given.
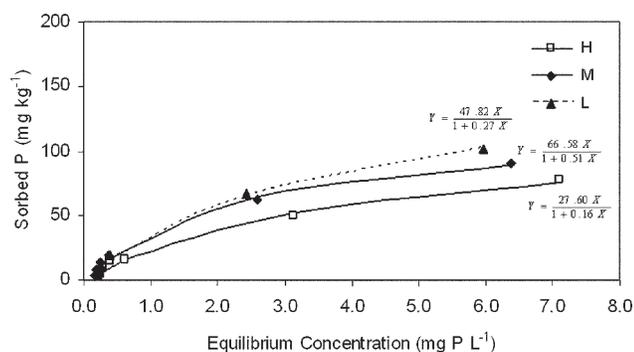
Initial P rank	Soil	pH rank	Langmuir constants		R^2	P value
			k	b		
			mg P L⁻¹	mg P kg⁻¹		
1	1	H	1.17a	64.5c	0.97	<0.001
	7	H	0.42b	282a	0.99	<0.001
	9	M	0.58b	112b	0.98	<0.001
	LSD 0.05		0.28	19.4		
2	5	H	0.20c	101c	0.96	<0.001
	2	M	0.76a	107c	0.98	<0.001
	6	M	0.69ab	255a	0.95	<0.001
	4	L	0.32bc	150b	0.98	<0.001
	LSD 0.05		0.37	27.0		
3	3	H	0.11b	251a	0.98	<0.001
	10	M	0.23a	200ab	0.96	<0.001
	8	L	0.20ab	173b	0.98	<0.001
	LSD 0.05		0.11	59.2		

Table 4. Mean separation for sorption maximum, *b* and binding energy, *k* for the Langmuir isotherm constants performed on Soils 1 to 10 grouped only by pH [H = >7.0 (*n* = 12), M = 6.2–6.8 (*n* = 12), L = <6.0 (*n* = 6)]. Means with different letters for constants within a pH group are significant (α = 0.05). Correlation and significance of observed vs. the modeled Langmuir isotherm are also given.

pH	Langmuir constants		R^2	P value
	k	b		
	mg P L⁻¹	mg P kg⁻¹		
H	0.16b	176a	0.97	<0.001
M	0.51a	131a	0.95	<0.001
L	0.27ab	175a	0.98	<0.001
LSD 0.05	0.25	61.5		

amounts of initial P can sorb less P and the energy required for P removal is greater than other soils with high initial P.

Separating the data only by soil pH and removing Soils 6 and 7, Fig. 3, soil pH was not a factor in P sorption capacity for the 10 CB soils tested. Further analysis (Table 4), shows some separation in *k* for the soils tested, however, sorption maximum for low, medium, and high pH soils is not significant (α = 0.05).

CONCLUSION

The results from this study showed that the increase in several extractable P forms following incubation with fertilizer for soils that vary in initial P status and pH did not significantly differ with soil pH. When the slopes for extractable P concentration with increasing P rate (fertilizer P addition) are compared based solely on soil pH, different slopes were found for each extraction method and was likely related to the chemistry of the extraction reagents. Of the extraction methods tested, OP demonstrated the

least variability in slope across the pH ranges tested and MMP the most. This means that if a P fertilizer addition is made to low or high pH soils, the amount of P extracted by OP will be very similar for both. Further, assessment of plant need for fertilizer P at a given level of OP appears to be valid for several pH levels in the CB and the fertilizer calibrations made on high pH soils are suitable under low pH conditions. There are two possible explanations for OP working well with low pH soil: (i) the soils of the CB, although low in pH in some cases, still exhibit a dominant Ca–P chemistry or (ii) if Fe/Al–P complexes exist, the amount of P extracted is similar to the traditionally assumed Ca–P state.

Isotherm data for Soils 1 to 10 were well correlated with modeled Langmuir values. Analysis of sorption maxima calculated from the Langmuir isotherm show some variability based on soil pH for given initial P ranges. Pooled by soil pH, differences in sorption maximum for low, medium, and high pH soils were not statistically significant.

Fig. 3. Results of P sorption isotherms conducted for Soils 1 to 5 and 8 to 10 grouped by H (>7.0, *n* = 9), M (6.2–6.8, *n* = 9) and L (<6.0, *n* = 6) soil pH. Sorbed P is calculated as the difference between the P added and the P remaining in solution following incubation and 24 h shaking. Best fit lines and equations for the Langmuir isotherm are shown for the data sets.

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