Organic Phosphorus Source Effects on Calcareous Soil Phosphorus and Organic Carbon

C. W. Robbins,* L. L. Freeborn, and D. T. Westermann

ABSTRACT

The loading, solubility, mobility, and plant availability of P is a growing environmental concern to regulators and planners of nutrient management plans, confined animal feeding operations, and wastewater land application permit sites. Insufficient information is available on how P reacts from different organic sources when applied to calcareous soils. A field study was conducted to determine the interactions among P application rate, source, extractability, and soil organic carbon (OC) concentration. A Portneuf silt loam (Coarse-silty, mixed, superactive, mesic Durinodic Xeric Haplocalcids) topsoil and freshly exposed subsoil were fertilized with monocalcium phosphate (MCP), cheese whey, and dairy manure. Organic matter added with the whey did not influence soil OC concentrations whereas organic matter added with the manure doubled the subsoil OC and increased the topsoil OC concentrations. Bicarbonate and saturation paste extractable ortho- and organic-P concentrations were linearly related to soil OC concentrations but were not related to the amount of ortho- or organic-P added. All forms of P increased more per unit of added P in the order manure $>$ whey $>$ MCP and were correlated with the soil OC concentrations. These results suggest organic waste applications should be managed from soil P test data rather than on P application rates.

M to increase soil fertility on the basis of crop land
to increase soil fertility on the basis of crop nitro-ANURE traditionally has been applied to crop land gen requirements. More recently, field application of manure from large confined animal feeding operations is driven by waste disposal rather than by soil fertility. Because NO_3-N is mobile with the soil solution and P has been considered relatively immobile, N disposal rates have been used to determine manure application rates, even though annual crop P requirements may be exceeded two- to threefold (Parry, 1998). The USEPA reported that runoff from agricultural lands is a major source of nonpoint-source pollution in lakes and streams and prevents reaching the water quality goals of the Clean Water Act (Parry, 1998). Each state has been mandated by the USEPA to develop Total Maximum Daily Loads (TMDL) for surface waters (Gburek and Sharpley, 1998). Phosphorus is one of the most common constituents exceeding the suggested limits. In order to develop reasonable P management strategies, it is necessary to develop sampling and characterization methods that identify potential P sources in the landscape and agronomic practices that contribute to P movement into surface and ground waters. To do this it is necessary to identify the interactions among application rate, source, solubility, and mobility of P. One of the requirements for attaining TMDL goals is the ability to identify the relationships between threshold soil P

concentrations and potential P enrichment in runoff and the threshold soil concentrations in relation to economic agricultural production (Daniel et al., 1998).

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Most of the available data for these relationships comes from rain fed landscapes with acid to neutral soils. Phosphorus equilibrium and plant availability in calcareous surface soils are controlled by different mineral reactions than in lower pH soils (Lindsay, 1979). Consequently, different measurement procedures and limits are required to define solubility, mobility, and plant availability of P (Olsen et al., 1954). Inorganic P fixation in calcareous soils is relatively rapid whereas the rate and extent of fixation may be much slower or less extensive when P is added with organic matter or in an organic form (Meek et al., 1979).

Phosphorus movement into calcareous soils used for waste disposal depends on loading rate, clay content, and waste material. Phosphorus leaching in southern Idaho potato *(Solanum tuberosum* L.) processing plant waste sites was dependant on loading rate and clay content (Robbins and Smith, 1977). In three of the six sites monitored, water extracted through ceramic cups at 0.9 and 1.5 m contained 1.7 to 5.6 mg and 1.2 to 2.1 mg total P L^{-1} respectively. These sites received 122 to 654 kg total P ha⁻¹ yr⁻¹ for 2 yr prior to sampling. Utah calcareous soils that had received turkey and beef manures for two to four decades showed increased bicarbonate extractable ortho- and total-P (STP and STPt) concentrations to a depth of 2.1 m in most cases (James et al., 1996). A calcareous California soil that received 1336 kg P ha^{-1} with applied feedlot manure still contained 53 mg STP kg^{-1} in the surface 0.30 m after 6 yr (Meek et al., 1979). Higher manure rates increased surface soil STP concentrations to 237 mg kg^{-1} . Phosphorus moved to deeper depths when 4000 kg P ha⁻¹ was applied. Comparable amounts of inorganic P were not applied as a comparison in the above studies.

In contrast to those findings, when acid cheese wheys were added to a sodic and two calcareous soils in a lysimeter study and three field studies, STP and bicarbonate extractable organic-P (STPo) did not appear to be as mobile as the P in the potato waste or the manures (Jones et al., 1993; Robbins et al., 1997a). A single application of cottage cheese whey to a sodic soil at total P rates up to 1050 kg ha⁻¹ caused no STP increases deeper than the 0.30 m tillage depth. A creamed cheesemozzarella cheese whey mix applied to an Idaho calcareous soil during the winter at rates up to 752 kg total P ha^{-1} over a 2-yr period did not increase STP below 0.6 m

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Abbreviations: OC, organic carbon; MCP, monocalcium phosphate; STP, bicarbonate extractable ortho-phosphorus; STPt, bicarbonate extractable total-phosphorus; STPo, bicarbonate extractable organicphosphorus; SPP, saturation paste extractable (water soluble) orthophosphorus; SPPo, saturation paste extractable organic-phosphorus.

I- **Applied after the spring 1991 and fall 1991 soil samples were taken.**

Applied after the fall 1994 soil samples were taken.

§ Manure was applied to the Low-whey plots after the F94 soil samples were taken.

or STPo below the 0.3 m tillage depth. The saturation paste extract (water soluble) ortho-P (SPP) did not increase below 0.3 m and the saturation paste extractable organic-P (SPPo) did not increase below 0.6 m. For whey applied during the growing season at the same site, the STP and SPP results were the same as the winter applications, while the STPo and SPPo concentrations did not increase below 0.30 m. When a Swiss cheese-mozzarella cheese whey mix was added to a northern Utah, highly calcareous soil at rates up to 675 kg total P ha^{-1} over a 1.5 yr period, no increases in STP, STPo, or SPPo were measured below 0.3 m. There was an increase from 0.11 to 0.35 mg SPP kg⁻¹ in the 0.3 to 0.6 m depth increment by the highest (675 kg total P ha^{-1} treatment rate.

Over time, the P added from most sources tends to become less soluble and less available to plants (Sample et al., 1980). On a seasonal basis, however, P is cycled through plant tissue and there is a decrease in soil P solubility during the growing season, often followed by an increase in P extractability and solubility during the noncropping season in calcareous soils. (Robbins et al., 1999).

In 1991, a long term study was initiated to develop a method to restore yields to severely eroded calcareous Southern Idaho silt loam soils (Robbins et al., 1997b). Topsoil and freshly exposed subsoil plots were treated with MCP, acid cheese whey, and fresh dairy manure. The soil STP concentrations in that study varied depending on the P source rather than on the P application rate or crop rotation. Ortho-P solubilities were later compared with hydroxyapatite, beta tricalcium phosphate, and octacalcium phosphate solubilities over eight years on the same site (Robbins et al., 1999). Here again, the soil ortho-P solubilities were not related to the P application rates or crop rotations across all treatments, but appeared to be related to soil OC concentration changes due to the various P sources. These plots provided the opportunity to look at soil P across crop rotations, P sources and rates, and soil OC concentrations.

The object of this paper is to report and discuss the interactions among P application rates, P sources, soil OC, and P extractability in STP and SPP extracts.

Applied after the S91 and F91 soil samples were taken.

I- Applied after the F94 soil samples were taken.

§ **Manure was applied to the Low-whey plots after the F94 soil samples were taken.**

Least significant difference (LSD) across years for each treatment is calculated at *P <* **0.05.**

S, **spring; F, fall, followed by year.**

Sample date	S91#	F91#	S92	F92	S93	F93	S94	F94	F95	S96	S97	S98	LSD¶
	$mg P kg^{-1}$												
Treatment													
Subsoil MCP+		12	31	25	36	29	32	21	26	32	30	22	
Subsoil High Whey†‡		48	71	57	74	49	48	35	77	82	64	57	13
Subsoil Low Whey†§		27	51	38	55	37	31	25					
Subsoil Manure94‡§									118	137	142	126	20
Subsoil Manure91+		70	114	124	134	122	125	88	85	118	111	99	33
Topsoil	25		25	24	28	24	22	19	22	26	22	17	
Topsoil Manure94‡	23		21	22	27	26	19	18	105	121	95	93	
Topsoil Whev94‡	22		22	21	25	22	19	17	47	42	40	34	21

Table 3. Bicarbonate extractable ortho-P (STP) concentrations in the surface 0.30 m depth by treatment and year (from Robbins et al., 1999).

t Applied after the S91 and F91 soil samples were taken.

Applied after the F94 soil samples were taken.

§ Manure was applied to the Low-whey plots after the F94 soil samples were taken.

I Least significant difference (LSD) across years for each treatment is calculated at *P <* **0.05.**

S, spring; F, fall, followed by year.

MATERIALS AND METHODS

In 1991, a long-term study was initiated to find an economical method(s) for restoring crop yields of freshly exposed subsoil to that of the associated topsoil (Robbins et al., 1997b). The soil was a Portneuf silt loam. The topsoil contained 80 g calcium carbonate equivalent and the subsoil contained 240 g calcium carbonate equivalent kg^{-1} soil. The surface soil saturation paste pH was 7.9 and the subsoil pH was 8.0. The surface 0.3 m (plow depth) of topsoil was removed in long narrow strips from the top to bottom of the field to create the subsoil plots. Topsoil strips were left for plots between the subsoil strips. This arrangement was necessary because the plots were furrow irrigated and the study was to last for at least 8 yr. Two crop rotations were planted along the topsoil strips and three crop rotations were grown along the subsoil strips in each of four replications. Four fertilizer treatments were applied in strips across each replication for a total of twenty, 9 by 21 m plots (Robbins et al., 1997b).

Dairy manure, acid cottage cheese whey, and MCP were applied to the subsoil plots in the spring and fall of 1991 (Robbins et al., 1997b). Dairy manure and sweet-whey (from cultured cheese) were applied to the subsoil and topsoil plots in the fall of 1994 after the crops were harvested (Robbins et al., 1999) (Table 1). The total- and ortho-P and total OC added with each treatment is also shown in Table 1. The treatments and crop residues were incorporated by plowing the plot area to 0.3 m each year.

Three different crop rotations were used. Rotation 1 was barley *(Hordeum vulgare* L.), alfalfa *(Medicago sativa* L.), wheat *(Triticum aestivum* L.), beans *(Phaseolus vulgaris* L.), alfalfa, wheat, beans. Rotation 2 was sorghum *[Sorghum bi-* *color* (L.) Moench]-sudangrass *[Sorghum drummondii* (Nees ex Steud.) Millsp. & Chase] hybrid, alfalfa, sorghumsudangrass hybrid, beans, sweet corn *(Zea mays* L.), alfalfa, sweet corn, beans. Rotation 3 was barley, beans, wheat, beans, wheat, beans, wheat, beans. Rotations 1 and 2 were grown on subsoil and topsoil plots, while Rotation 3 was grown on subsoil plots only. Crop residue was removed from the alfalfa plots and was incorporated into all other plots after harvest. The sorghum-sudangrass hybrid was not harvested due to very low yields.

Soil samples for this study were collected each spring from 1991 through 1998. Five soil cores were taken from each plot with a 75 mm diameter bucket auger to 0.30 m, mixed, and air dried for analysis. Soil OC was measured in all soil samples using the Walkley-Black method (Nelson and Sommers, 1982). The STP and STPo and SPP and SPPo were measured on all soil samples (Nelson and Sommers 1982; Olsen et al., 1954; Watanabe and Olsen 1965). Ortho-P concentrations were also measured in 1:20 soil to 0.01 *M* CaCl₂ (CaCl₂P) extracts (Fox and Kamprath, 1970) each spring from 1991 to 1998 except in the spring of 1995 following the second treatment application and in the autumn in 1994 and 1995. Ortho-P solubilities are reported elsewhere (Robbins et al., 1999).

Linear regression was calculated between OC and STP, STPo, SPP, SPPo, and CaCl₂P for all topsoil and subsoil treatments using the spring 1997 soil sample data. Linear regression was also calculated between STP and SPP, SPPo, and CaCl₂P for all treatments, topsoil treatments, and subsoil treatments using data from all eight years. Correlations between soil OC and STP concentrations were calculated using the subsoil and

t Applied after the S91 and F91 soil samples were taken.

t Applied after the F94 soil samples were taken.

§ Manure was applied to the Low-whey plots after the F94 soil samples were taken.

I Least significant difference (LSD) across years for each treatment is calculated at *P <* **0.05.**

S, spring; F, fall, followed by year.

Table 5. **Saturation paste ortho-P (SPP) concentrations in the surface 0.30 m depth** by treatment and year (from Robbins et **al., 1999).**

Sample date	S91#	F91#	S92	F92	S93	F93	S94	F94	F95	S96	S97	S98	LSD1
	$mgP L^{-1}$												
Treatment													
Subsoil MCP+	0.05	0.25	0.59	0.27	0.51	0.34	0.39	0.26	0.21	0.31	0.20	0.24	0.11
Subsoil High Whey†‡	0.05	0.53	1.65	0.76	1.09	0.67	0.56	0.29	1.51	1.71	0.78	0.85	0.41
Subsoil Low Whey†§	0.05	0.23	1.12	0.41	1.04	0.51	0.36	0.19					0.32
Subsoil Manure94‡§									3.04	3.87	3.23	3.32	0.50
Subsoil Manure91 †	0.05	0.47	2.65	1.80	3.24	2.86	2.20	1.68	2.20	3.23	2.35	2.34	0.64
Topsoil	0.78		0.78	0.64	0.69	0.56	0.50	0.44	0.54	0.72	0.48	0.48	0.13
Topsoil Manure94‡	0.58		0.48	0.31	0.46	0.34	0.31	0.26	3.01	3.82	3.24	3.35	0.62
Topsoil Whey94‡	0.53		0.61	0.39	0.65	0.43	0.35	0.30	2.02	1.82	1.10	1.15	0.42

t Applied after the S91 and F91 soil samples were taken.

Applied after the F94 soil samples were taken.

§ Manure was applied to the Low-whey plots after the F94 soil samples were taken.

II Least significant difference (LSD) across years for each treatment is calculated at *P <* 0.05.

The lower ortho-P detection limit for these samples is about 0.03 mg P L^{-1} .

S, spring; F, fall, followed by year.

t Applied after the S91 and F91 soil samples were taken.

Applied after the F94 soil samples were taken.

§ Manure was applied to the Low-whey plots after the F94 soil samples were taken.

¹¹Least significant difference (LSD) across years for each treatment is calculated at *P <* 0.05.

The lower ortho-P **detection limit for these samples** is about 0.03 mg P

S, spring; F, fall, followed by year.

topsoil data from the spring 1999 samples. Least significant difference (LSD) for each treatment across years for OC, STP, STPo, SSP, and SSPo was calculated. All calculations were made using SAS (SAS Institute, 1989).

RESULTS AND DISCUSSION

Prior to treatment applications in 1991, the subsoil contained 4.5 \pm 0.3 g OC kg⁻¹ and the topsoil contained 9.4 \pm 0.3 g OC kg⁻¹ (Table 2). The Subsoil MCP and untreated topsoil OC did not change over the course of the study. The OC concentration in the Subsoil High Whey treatment plots, which received 34 Mg OC ha⁻¹, increased slightly over the eight growing seasons. Adding 137 Mg manure ha⁻¹ (41 Mg OC ha⁻¹) in 1991 nearly tripled the OC in the Manure9l subsoil plots. By the fall 1994 sampling the OC concentrations in these plots had decreased to about that of the topsoil concentration and remained relatively constant for the rest of the study. Adding 74 Mg manure ha⁻¹ (22 Mg OC ha⁻¹) to the Low Whey plots and to the topsoil in 1994 increased the soil OC about 5 g kg^{-1} above the pretreatment concentration. Adding whey to the topsoil had no measurable affect on OC.

Even though nearly as much OC was added to the subsoil with the whey as with the manure over the course of the study (Table 1), the whey had very little effect on subsoil OC, while the manure increased the Subsoil Manure9l plot OC concentrations to that of the Topsoil plots (Table 2). The lack of soil OC persistence in the whey treatments is probably due to rapid decomposition of the whey sugars and proteins, while the materials in the manure are more resistant and decompose more slowly.

Application of 265 kg P ha^{-1} in 1991 as MCP to the subsoil, increased STP to about the same concentration as in the untreated topsoil while the STPo, SPP, and SPPo concentrations were generally lower than in the untreated topsoil over the study period (Tables 3-6).

The addition of 1390 kg total \hat{P} ha⁻¹ in 1991 and an additional 225 kg total \overline{P} ha⁻¹ in 1994 in the Subsoil High Whey plots increased the STP to nearly three times that of the topsoil by the spring of 1992 and then

Table 7. **Relationships among soil organic carbon (OC) and STP, STPo, SPP, SPPo, and** CaCl2P **concentrations in the** S97 soil samples.

Comparison	n†	Regression equation	2ء
OC vs. $STP - topsoil$	32	$STP = -147.0 + 19.5 \times OC$	0.82
OC vs. $STP -$ subsoil	48	$STP = -27.6 + 14.9 \times OC$	0.77
OC vs. STPo - topsoil	32	$STPo = -73.4 + 8.6 \times OC$	0.53
OC vs. $STPo$ – subsoil	48	$STPo = -10.9 + 4.1 \times OC$	0.71
$OC vs.$ SPP $-$ topsoil	32	$SPP = -5.89 + 0.71 \times OC$	0.84
OC vs. $SPP -$ subsoil	48	$SPP = -1.41 + 0.41 \times OC$	0.66
OC vs. SPPo - topsoil	32	$SPPo = -3.78 + 0.54 \times OC$	0.73
OC vs. $SPPo - subsoil$	48	$SPPo = -0.55 + 0.15 \times OC$	0.80
OC vs. $CaCl2P$ – topsoil	32	$CaCl2P = -1.91 + 0.24 \times OC$	0.81
OC vs. $CaCl2P - subsoil$	48	$CaCl2P = -0.40 + 0.12 \times OC$	0.91

t Number of samples used to calculate regression.

Fig. 1. The 1997 bicarbonate extractable ortho-P (STP) concentrations for all subsoil (solid symbols) and all topsoil (open symbols) samples as related to the soil organic carbon (OC) concentrations.

decreased after that. The soil STPo concentrations in the High Whey treatment also increased dramatically after each application (Table 4). The SPP concentrations in that treatment increased to about double the topsoil concentrations the first year and then rapidly decreased to about that of the top soil (Table 5). The SPPo concentrations in the High Whey treatment were usually not different from the untreated topsoil. The STP, STPo, and SPP concentrations in the Low Whey treatment followed the same trend, but at a lower concentration than the High Whey treatment.

Application of 1180 kg ha⁻¹ total P with the 1991 manure treatment increased the STP to more than 100 mg P kg^{-1} for most of the study period and remained at or near 100 mg P kg⁻¹ after seven cropping seasons (Table 3). The STPo remained between 25 and 30 mg kg^{-1} after seven cropping seasons and was usually 10 times that of the untreated topsoil (Table 4). The SPP concentrations were above 2.2 mg P kg⁻¹ for most of the period and were consistently higher than the untreated topsoil (Table 5). The SPPo concentrations in the Manure91 soils varied between 0.6 and 4.2 mg P L^{-1} and were always greater than the untreated topsoil samples (Table 6).

The STPo and SPPo concentrations were affected by treatment but were quite variable from year to year (Tables 4 and 6). The fall 1994 concentrations were unusually low for both methods and this may be due to cropping history or residue management.

Soil OC concentration changes due to treatments were correlated with all extractable P methods and forms measured during the study. The correlations for the spring 1997 data show the treatment effect persistence over time (Table 7). The STP-OC correlations for subsoil and topsoil are shown in Fig. 1. Soil SPP and $CaCl₂P$ concentrations for the eight years were also well correlated with STP concentrations (Table 8). However, correlations between STP and STPo and between STP and SPPo concentrations were not well correlated for the same time period.

Bicarbonate extractable ortho-P does not appear to serve as a warning of when soil organic-P concentrations might be a concern. Also, there was no correlation between the extractability of the various P forms and the amount of ortho- or organic-P applied by the MCP, whey, or manure treatments. The increases in persistent soil OC concentration appear to enhance the solubility and extractability of the added ortho- and organic-P.

Table 8. Bicarbonate extractable ortho-P (STP) as a means of predicting saturation paste ortho- (SPP) and organic-P (SPPo) and 0.01 M CaCl₂ extractable P (CaCl₂P).

Comparison	n†	Regression equation		
STP vs. $SPP - all plots$	928	$SPP = -0.179 + 0.0252 \times STP$	0.77	
STP vs. $SPP -$ topsoil	352	$SPP = -0.191 + 0.0333 \times STP$	0.86	
STP vs. $SPP -$ subsoil	576	$SPP = -0.352 + 0.0255 \times STP$	0.79	
STP vs. $STPo - all plots$	848	$STPo = -3.413 + 0.3001 \times STP$	0.58	
STP vs. STPo - topsoil	320	$STPo = -1.840 + 0.2536 \times STP$	0.46	
STP vs. $STPo$ – subsoil	528	$STPo = -4.066 + 0.3096 \times STP$	0.57	
STP vs. $SPPo - all plots$	640	$SPPo = -0.023 + 0.0204 \times STP$	0.41	
STP vs. $SPPo - topsoil$	256	$SPPo = 0.029 + 0.0285 \times STP$	0.42	
STP vs. $SPPo -$ subsoil	384	$SPPo = -0.275 + 0.0209 \times STP$	0.19	
	640	$CaCl2P = -0.031 + 0.0081 \times STP$	0.74	
	256	CaCl,P = $-0.048 + 0.0128 \times STP$	0.89	
STP vs. $CaCl2P - subsoil$	384	CaCl-P = $-0.128 + 0.0082 \times STP$	0.84	
STP vs. $CaCl2P - all plots$ STP vs. $CaCl2P - topsoil$				

t Number of samples used to calculate regression.

The more resistant OC in the manure probably coats the adsorption and precipitation sites, causing the P to stay in solution longer in the manure treatments. Organic matter added to calcite $(CaCO₃)$ precipitation systems has been shown to increase the apparent solubility product of calcite in natural calcareous systems and was attributed to organic matter coating of the reaction sites (Suarez, 1977). The manure probably was also supplying additional P into solution through continual biological cycling during the slower decomposition process.

The reason the P solubility and extractability in the whey treatments was lower than in the manure treatments, even though comparable OC amounts were added, is probably due to less organic-P in the whey, more rapid whey OC decomposition in the soil, and the acidic nature of whey that dissolves calcium carbonate, increasing the calcium activity and enhancing phosphate precipitation from the soil solution.

The STP, SPP (Tables 3 and 5), and 0.01 M CaCl₂P (data not shown here) extractable ortho-P concentrations and their solubility comparisons with hydroxyapatite, beta-tricalcium phosphate, and octacalcium phosphate solubilities over time are presented by Robbins et al. (1999) for these same samples.

CONCLUSIONS

The relative effects of the P added to this calcareous soil with MCP, manure, and whey on STP, SPP, STPo, $SPPo$, and $CaCl₂P$ concentrations varied greatly over time in this field study. All soil P form concentrations increased more per unit of added P in the order manure $>$ whey $>$ MCP. In contrast, the four measured P concentrations were correlated with soil OC concentrations. This trend was present throughout the study period.

The lack of correlation between the traditional soil fertility test for calcareous soils and the amount of various P forms added and the resulting P solubilities from different P sources presents some real challenges in predicting how much P can be added to waste application sites before P leaching or surface transport becomes an environmental concern. The differences in sustained P solubility will likely require waste site monitoring to determine when a chosen upper limit P concentration is exceeded, rather than regulating P loading rates to achieve a given P solubility on calcareous soils. It appears that STP concentration can be used to predict SPP and CaCl₂P extractability. On the other hand, STPo and SPPo do not appear to be well correlated with the STP test and additional methods will be needed to

determine organic-P solubility and movement. Phosphorus movement may be primarily in these organic forms and therefore would be much harder to predict than the ortho-P movement.

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