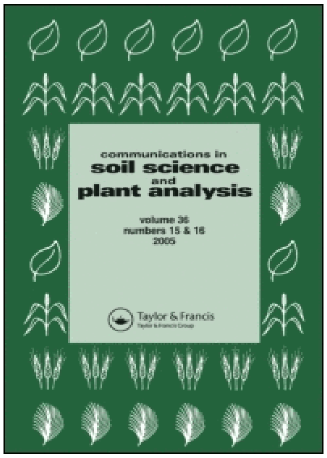

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PHOSPHORUS SORPTION CAPACITIES OF CALCAREOUS SOILS RECEIVING CATTLE MANURE APPLICATIONS FOR 25 YEARS

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ABSTRACT

Manure management is an important issue for cattle producers in areas where many intensive feedlots operate. Accumulation of phosphorus (P) in agricultural soils receiving repeated manure applications and transport of P from manure to ground and surface waters has been documented. This study investigates the P sorption capacities of soils amended with feedlot manure annually for up to 25 years. Non-irrigated plots at the Lethbridge Research Centre, Alberta, Canada, received 0, 30, 60, and 90 Mg manure (wet weight) ha^{-1}, and irrigated plots received 0, 60, 120, and 180 Mg ha^{-1} annually for 25 years. The quantity of P sorbed to surface soils (0 to 15 cm) from solutions containing 0 to 40 μg P mL^{-1} was determined, and sorption isotherm models were fitted to the Freundlich equation. The P sorption capacity of non-irrigated and

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irrigated soils declined with increasing soil available P concentrations, but the P sorption capacity of surface soils that received applications annually for up to 25 years was generally not more than 50% lower than the P sorption capacity of unamended soils. Soil organic matter content, pH, and surface chemistry were altered by manure application, and may explain why the P sorption capacity did not decline more as soil available P concentrations increased. Further investigations will be required to determine the fate of manure P not sorbed in manure-amended calcareous soils to promote manure management practices that are agronomically sound and protect water quality.

INTRODUCTION

Cattle manure has long been used to supply nutrients for crop production. However, strategies to manage and utilize manure are required in areas where many large intensive feedlots operate and land available for manure applications is limited. The number of cattle in Alberta has more than doubled in the last 25 years, and intensive feedlot operations have increased in some areas due to favorable climate, good access to feed and water, and proximity to markets (1). The county of Lethbridge in southern Alberta covers an area of approximately 3,080 km^2 and contains about 522,130 feedlot cattle, 16,650 dairy cattle, 40,950 hogs, and 836,610 poultry (2).

There is a large potential for nutrient losses from manure in feedlots, storage facilities, and after land application through greenhouse gas emissions and transport processes such as leaching, surface runoff, and erosion (3–5). Migration of P from manure to ground and surface waters has been linked to eutrophication of aquatic systems (3,6). Land application has the potential to stabilize manure P in relatively immobile forms because many soils contain minerals with high surface affinity for P. However, land applications rates for manure are generally based on crop N requirements and will supply P in excess of crop uptake when manure N:P ratios are lower than crop N:P ratios. The N:P ratio of cattle manure varies with diet and handling, and generally ranges from 2 to 6 (7–10), while the N:P ratio of barley and corn is between 7 and 8 (11). Over time, repeated manure applications can lead to P accumulation in soils and saturation of P sorption sites (12–14). Evaluation of the P sorption capacity of soils is required to determine suitable land application rates for manure that are agronomically and environmentally sustainable.

The P retention characteristics of soils have often been determined by fitting sorption results to standard isotherm models (15–17). The P sorption capacity is lower in soils that have received high applications of animal manure than unamended soils (18–20). Phosphorus sorption capacities have been reduced by 50% in agricultural soils that have received manure in excess of crop requirements than forest soils (21). Yet, most soils where reduced P sorption capacity with high or repeated manure applications have been documented are acidic and P sorption occurs through reaction with iron (Fe), aluminum (Al), and manganese (Mn) ions. Fewer studies have documented the effect of manure applications on alkaline soils, where P sorption occurs primarily through binding and precipitation with calcium (Ca) ions (22). Scientific evaluation of the P sorption capacity of manure-amended calcareous soils is required to promote manure management practices that are agronomically sound and protect water quality.

A field experiment was initiated in 1973 at Lethbridge, Alberta, Canada, to determine the effects of repeated manure applications, at recommended (before 1992 code practices) as well as higher levels, on both crop production and soil properties. The purpose of this investigation was to evaluate changes in P sorption capacities of calcareous surface (0 to 15 cm) soils that have received feedlot manure applications for up to 25 years.

MATERIALS AND METHODS

Site Description

Soils used in this study were from non-irrigated and irrigated research plots located at the Lethbridge Research Centre in southern Alberta, Canada that have received solid cattle manure applications annually each autumn after harvest since 1973. The soil in the study area was a well-drained calcareous Orthic Dark Brown Chernozemic (Typic Haploboroll) clay loam. Information is provided on initial soil properties (Table 1), cropping history (Table 2), and

Table 1. Soil Physico-chemical Properties in Soil Layers to 150-cm Depth in 1973 Prior to Manure Applications (Values Are the Mean of Seventy-Two Replicate Determinations)

Soil Depth (cm)	Sand ($g\,kg^{-1}$)	Silt ($g\,kg^{-1}$)	Clay ($g\,kg^{-1}$)	pH	Organic Matter ($g\,kg^{-1}$)
0–15	386	220	394	7.75	21.3
15–30	387	213	400	7.78	16.6
30–60	478	225	297	7.90	9.5
60–90	399	257	343	7.89	6.3
90–120	458	247	293	7.86	5.0
120–150	473	227	300	7.82	4.4

Table 2. Cropping History of Non-irrigated and Irrigated Plots at the Study Site

Year	Crop Grown	Cultivar(s)
Non-irrigated plots		
1974–1995	Barley (*Hordeum vulgare* L.)	Galt, Leduc, Virden, Duke[a]
1996	Canola (*Brassica rapa* L.)	Tobin
1997	Barley (*Hordeum vulgare* L.)	Duke
1998	Triticale (*X Triticosecale Wittmack)*	Pronghorn
Irrigated plots		
1974–1995	Barley (*Hordeum vulgare* L.)	Galt, Leduc, Virden, Duke[a]
1996	Canola (*Brassica rapa* L.)	Tobin
1997 to 1998	Corn (*Zea mays* L.)	Pioneer Hybrid 3957

[a] All cultivars were 6-row feed barley.

characteristics of the manure applied from 1973 to 1997 (Table 3). Further details of the research site and the effect of long-term manure amendments on soil chemistry, fertility, and physical properties have been reported (5,11,23–25).

Table 3. Characteristics of Manure Applied to the Study Between 1973 and 1997

Moisture content ($kg\,kg^{-1}$)	32.8 ± 2.2
pH (1:2 manure:water)	7.1 ± 0.1
Electrical conductivity (EC, $dS\,m^{-1}$)	23.4 ± 1.6
Sodium adsorption ratio (SAR)	20.6 ± 1.2
Organic C ($g\,kg^{-1}$)[a]	267 ± 17
Total N ($g\,kg^{-1}$)[a]	15.9 ± 0.9
Total P ($g\,kg^{-1}$)[b]	6.1 ± 0.3
Available NH_4-N ($g\,kg^{-1}$)[c,d]	1.3 ± 0.2
Available NO_3-N ($g\,kg^{-1}$)[c,d]	0.21 ± 0.05
Available P ($g\,kg^{-1}$)[d,e]	2.3 ± 0.2
Available S ($g\,kg^{-1}$)[f,g]	0.53 ± 0.09

Manure was applied each autumn. Nutrient analyses are expressed on a per kg of manure (dry weight) basis. At least 5 manure subsamples were analyzed each year to generate a mean value for manure characteristics.

[a] Analyzed using Carlo-Erba C and N analyzer (Milano, Italy).

[b] H_2O_2/H_2SO_4 digests.

[c] 2 M KCl extracts (1:5 manure:extractant).

[d] Analyzed by autoanalyzer (Technicon Industrial Systems, Tarrytown, NY).

[e] $NaHCO_3$-soluble P (1:10 manure:extractant).

[f] Saturated paste extracts.

[g] Analyzed by AAS (Perkin–Elmer Corporation, Norwalk, CT).

Experimental Design

Solid feedlot manure was applied each fall after harvest beginning in 1973 and incorporated immediately after application by one of three methods: plow, rototiller, or cultivator plus disk. Soil properties and crop production were not affected significantly by tillage (26,27), and since 1986, manure has been incorporated in all subplots with a cultivator. The experiment was initially a split plot design with four manure treatments (7.5 m by 15 m split plots) distributed among three tillage treatments (7.5 m by 60 m main plots). Main and subplot treatments were assigned randomly and the main treatments were replicated three times on 1) non-irrigated research plots and 2) irrigated research plots. Since the tillage treatment has been discontinued, the experiment is now analyzed as a completely randomized block design with 9 replicate manure treatments on the non-irrigated plots and 9 replicate manure treatments on the irrigated plots.

Manure rates (wet weight) were 0, 30, 60, and 90 $Mg\,ha^{-1}\,y^{-1}$ on non-irrigated soils, whereas irrigated soils received 0, 60, 120, and 180 $Mg\,ha^{-1}\,y^{-1}$. At the initiation of the experiment, the recommended annual manure application rates (wet weight) in this area were 30 $Mg\,ha^{-1}$ for non-irrigated soils and 60 $Mg\,ha^{-1}$ for irrigated soils, based on applying manure to supply crop N requirements and permitting some N accumulation in soils (28). Current recommendations are more conservative, and suggest that soil and manure testing should be conducted to better match manure applications to specific crop N requirements to protect water and air quality (29). In 1986, manure applications to 3 replicate manure treatments on the non-irrigated land and 3 replicate manure treatments on the irrigated land ceased completely. These plots do not receive inorganic fertilizer, and are used to assess the residual effects of manure on soil properties and crop production.

Phosphorus Sorption Isotherms

Soil samples were collected annually after harvest and prior to manure application by extracting two cores (to 150-cm depth) from each plot, dividing the cores in 15 to 30-cm increments, and compositing soils from each depth increment. Sodium-bicarbonate soluble P ("available P") was determined shortly after samples were collected (30). Oven-dried (60°C) and ground (<2 mm mesh) soil samples from the top increment (0 to 15-cm depth) were stored in tightly sealed glass jars in the dark, and samples collected in the 10th, 20th, and 25th years of the study were archived for future study. Samples used in this study were collected in 1983 (after 10 manure applications), 1993 (after 20 manure applications or after 13 manure applications and 8 years of no manure) and 1998 (after 25 manure applications or after 13 manure applications and 12 years of no manure). One gram of oven-dried soil was shaken with 40 mL of 0.01 *M* $CaCl_2$ containing eight concentrations (0, 0.625, 1.25, 2.5, 5, 10, 20, or 40 μg P mL^{-1}) of inorganic P (NaH_2PO_4) in 50 mL screw-cap polypropylene centrifuge tubes (17). The tubes were shaken continuously on an end-over-end shaker for 72 h at 20°C, then centrifuged at 27,000 $\times$ *g* for 10 min at 4°C, and aliquots of the supernatant were analyzed for ortho-phosphate colorimetrically at 840 nm with the ammonium molybdate-ascorbic acid method (31).

Sorbed P (mg P kg^{-1} soil) was the difference between added P and P remaining in solution after the 72 h incubation. Temkin, Freundlich, and Langmuir sorption isotherm equations were fitted to the results of sorbed P (Y = mg P kg^{-1} soil) and equilibrium P concentrations (X = mg P L^{-1} soil solution). The slope (B, related to maximum sorption) of the Temkin equation was obtained from Eq. (1).

$$Y = A + B(\log X) \tag{1}$$

The constant (K, related to maximum sorption) of the Freundlich equation was calculated from Eq. (2).

$$Y = KX^{1/n} \tag{2}$$

The constants b (maximum sorption) and K (related to the binding energy) of the Langmuir equation were obtained from Eq. (3).

$$X/Y = Y/b + 1/kb \tag{3}$$

Statistical Analysis

Sorption data for each replicate soil sample were fitted to non-linear forms of the equations using the PROC NLIN procedure of SAS (32). The effect of manure application rates on the constants of Temkin, Freundlich, and Langmuir equations for replicate soil samples within each irrigation treatment (non-irrigated and irrigated) were analyzed statistically by single-factor ANOVA in a general linear model with means separation by LSD at the 95% confidence level. The effect of manure applications on the constant of the Freundlich equation within each irrigation treatment and group of samples with the same manure history were analyzed statistically by single-factor ANOVA in a general linear model with means separation by LSD at the 95% confidence level.

RESULTS AND DISCUSSION

Sorption parameters from Temkin, Freundlich, and Langmuir equations were determined using data from manure-amended soils collected in 1983 after ten annual manure applications (Table 4). Freundlich and Langmuir equations, or modifications of these equations, have been used for more than thirty years to describe the relationship between the quantity of P adsorbed per unit soil weight and the concentration of P in soil solution (Q/I relationships) (15,16). The slope (B) of the Temkin equation, constant (K) of the Freundlich equation, and maximum sorption (b) and binding energy (k) of the Langmuir equation were shown to be highly correlated, suggesting that they all describe relationships between solid and solution P phases and thus can be used as a measure of soil P sorption capacity (33). The mean r value of the asymptotic correlation matrices generated with PROC NLIN for irrigated and non-irrigated soils was 0.558 for the Temkin equation, whereas the r values were 0.949 for the Freundlich equation and 0.880 for the Langmuir equation, suggesting that the Freundlich equation best described the P sorption isotherms of manure-amended soils. The isotherms of non-irrigated soils that received 0 to 90 $Mg\,ha^{-1}$ of manure annually for 10 years are shown in Fig. 1. The slope of the Temkin equation indicates a linear

Table 4. Phosphorus Sorption Parameters of Surface (0 to 15 cm) Soils from Manure-Amended Plots Collected in 1983 (Means Followed by the Same Letter in a Column Within an Irrigation Treatment Are Not Statistically Significantly Different ($P < 0.05$, LSD))

			Langmuir	
Manure Rate ($Mg\,ha^{-1}$)	Temkin [B[a] ($mg\,kg^{-1}$)]	Freundlich [K[b] ($mg\,kg^{-1}$)]	b[c] ($mg\,kg^{-1}$)	k[d] ($L\,mg^{-1}$)
Non-irrigated				
0	154 A	97 A	42 A	0.22 A
30	138 B	63 B	34 B	0.17 B
60	134 B	58 BC	33 B	0.14 B
90	117 B	49 C	34 B	0.10 C
Irrigated				
0	202 A	81 A	42 A	0.37 A
60	144 B	61 B	35 B	0.15 B
120	142 B	31 C	36 B	0.08 B
180	128 B	45 BC	32 B	0.08 B

[a] B = Slope of Temkin equation.
[b] K = Constant of Freundlich equation.
[c] b = Maximum sorption of Langmuir equation.
[d] k = Constant related to binding energy of Langmuir equation.

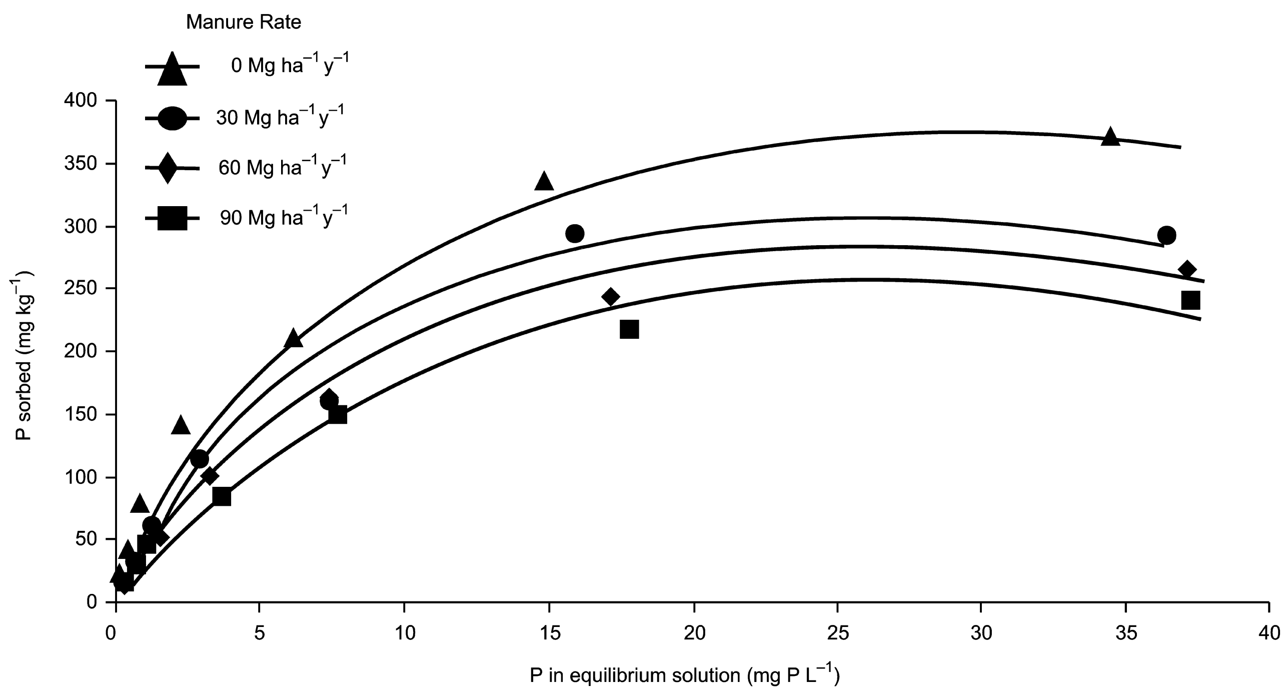

Figure 1. Phosphorus sorption isotherms of surface (0 to 15-cm) soils collected from non-irrigated plots in 1983.

decrease in P sorption, whereas the Freundlich and Langmuir equations suggest a non-linear decrease in P sorption with increasing saturation of soil surfaces (16). Our results indicate P sorption in manure-amended soils declined non-linearly with increasing solution P concentrations, and therefore the Freundlich and Langmuir equations best described the relationship between P sorbed and P in soil solution. The P sorbed in this study may have included P adsorbed to soil surfaces, complexed in organic matter, and precipitated by calcium to form relatively insoluble calcium phosphates. Further investigations, perhaps with radiolabeled ^{32}P, would be needed to quantify the fate of P sorbed from the soil solution.

The constant (K) of the Freundlich equation and the sorption maxima (b) and binding energy (k) of the Langmuir equation were significantly ($P <$ 0.05, LSD) lower in manure-amended than unamended soils from non-irrigated and irrigated plots (Table 4). The constant (K) of the Freundlich equation declined significantly ($P < 0.05$, LSD) as the cumulative amount of manure applied to non-irrigated and irrigated plots increased (Table 4). The constant of the Freundlich equations implies P sorption decreases exponentially with increasing saturation of soil surfaces (16), which is consistent with our findings. The sorption maxima (b) of the Langmuir equation did not change, but the binding energy tended to decrease with increasing manure application rates (Table 4). Results from the Langmuir equation suggest that the sorption capacity was reduced following manure applications, but the main effect of manure applications was a reduction in the quantity of P bound tightly to sorption sites.

The sorption maxima of the Langmuir equation decline as soil available P concentrations increase in soils amended with inorganic P fertilizer, but binding energy was unchanged as soil available P concentrations increased (16). However, the sorption maxima and binding energy values of the Langmuir equation were lower in manure-amended than unamended soils (34). Organic acids produced during manure decomposition may compete for P sorption sites, dissolve precipitated P compounds, alter surface charge, or react with exchangeable Fe, Al, and Ca to form organo-mineral complexes (20,35–38). The decline in P binding energy in manure-amended soils suggests that P may be more easily displaced from sorption sites in manure-amended than unamended soils. There may be a greater potential for P transport from soils that have received manure than inorganic fertilizers, and further work will be required to determine how fertilizer sources affect soil P mobility.

In non-irrigated soils amended with 90 $\mathrm{Mg\,ha^{-1}}$ for 10 years, there was a 55% reduction in P binding energy compared to unamended soils (Table 4). There was nearly an 80% reduction in the P binding energy of irrigated soils that received the highest rate of manure (180 $\mathrm{Mg\,ha^{-1}}$) for 10 years, as compared to unamended soils (Table 4). These results suggest that in soils receiving repeated, higher than recommended, manure applications, P is not bound very tightly to soil surfaces and could be readily desorbed. Significant P accumulation was found throughout the soil profile (to 150-cm depth) of non-irrigated and irrigated soils that received annual manure applications for 16 years, indicating P movement through the soil profile (11). The potential for P transport in the soil profile and possibly to groundwater is likely higher in irrigated than non-irrigated soils. Although there was a balance between applied P in manure and recovered P in crops and soils (to a depth of 150 cm) under non-irrigated conditions, as much as 1.4 Mg P ha^{-1} (180 Mg ha^{-1} manure treatment) was not recovered in the crops and soils of irrigated plots (11). Water table levels under irrigated plots fluctuated from 50 to 250 cm below the soil surface, indicating the P leached below 50 cm could eventually be transported to ground water (11). Similarly, Eghball et al. (39) found that P from beef manure could move through soil layers and could eventually reach ground water, especially in areas with shallow water tables. This study's conclusions from sorption isotherms in the laboratory were consistent with findings from these field studies.

The P sorption constant (K) declined as soil available P concentrations in non-irrigated and irrigated soils increased from manure applications (Fig. 2A and B). P sorption constants were significantly ($P < 0.05$, LSD) lower in soils that received 10, 20, and 25 years of continuous manure applications than unamended soils (Fig. 2A and B). Although P sorption constants declined as soil available P concentrations increased, there was not generally more than a 50% reduction in the P sorption constant of manure-amended soils relative to unamended soils. One possible explanation why we did not see a greater reduction in the P sorption capacity of manure-amended soils from the surface 15 cm is because manure applications altered soil characteristics in ways that partly compensated for the saturation of sorption sites in the soil. The soil organic matter content, particularly in the top 15 cm, increased with repeated manure applications (24). Soil organic matter can be a sink for available P through immobilization reactions and adsorption of P to anion exchange sites in the organic matter (35,38). Eleven annual manure applications at the study site caused a decline in surface soil pH from 7.8 to 7.0 and an increase in the sodium adsorption ratio of surface soils due to loading of Na^{+} from manure and displacement of Ca^{++} and Mg^{++} on the exchange complex (24,27). Adsorption/desorption reactions involving P are influenced by soil pH, and the potential for P adsorption and precipitation is lowest at soil pH 6.5 (40). More P is sorbed onto mineral surfaces saturated with divalent or trivalent cations than monovalent cations, and the displacement of Ca^{+} and Mg^{++} on exchangeable soil surfaces by Na^{+} may have reduced the P sorption capacity of surface soils because these divalent cations were transported deeper in the soil profile. Finally, the P sorption capacity we measured over a 72 h equilibrium period may have been affected by organic and inorganic anions that competed with inorganic P for sorption sites or microbial activity. The P remaining in equilibrium solution after 72 h may have included manure P displaced from

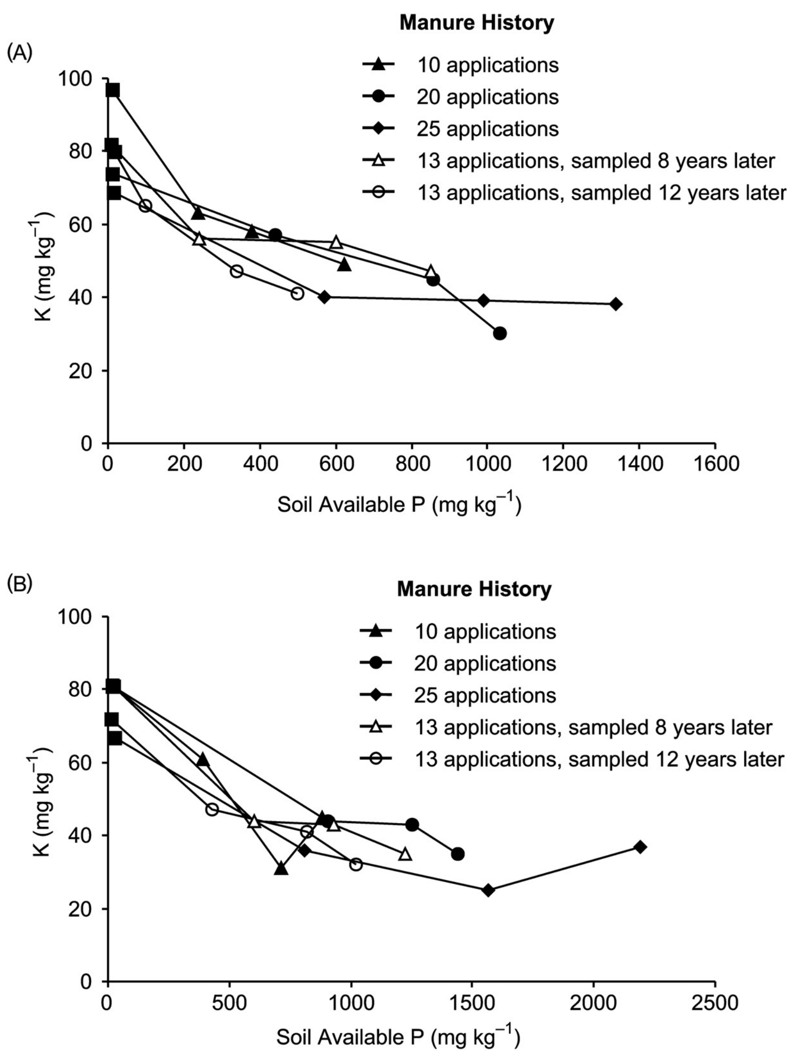

Figure 2. Relationship of the P sorption constant (K) of the Freundlich equation with soil available P concentrations in surface soils (0 to 15 cm) that were (A) non-irrigated or (B) irrigated. Soils unamended with manure (0 Mg ha^{-1} treatment) are indicated with a square (■).

sorption sites, inorganic P we added that was never adsorbed, inorganic P we added that was sorbed and later desorbed, or P immobilized and/or mineralized from microbial biomass. Without using radiolabeled ^{32}P, it is difficult to determine the fate and reactions of P added to soils.

The P sorption constants of non-irrigated and irrigated soils that received annual manure applications from 1973 to 1985 (thirteen manure applications) and were sampled eight years later (in 1993) and twelve years later (in 1998) exhibited similar trends as soils that received manure continuously (Fig. 2A and B). P sorption constants declined as soil available P concentrations increased, and were significantly ($P < 0.05$, LSD) lower in most soils that had received manure than unamended soils (Fig. 2A and B). However, only the P sorption constants of non-irrigated soils that received 30 Mg manure ha^{-1} for 13 years, and then did not receive manure for 12 years, were not different from P sorption constants in unamended soils (Fig. 2A). It appeared that the P sorption capacity increased as soil available P concentrations declined after manure applications were discontinued. However, it took more than a decade for the P sorption capacity of soil with a history of manure applications to reach a level that was not different from soils with no history of manure applications. Part of the reason may be that the crops planted at the study site did not have high P requirements, which would be required to reduce the available P concentration in manure-amended soils. These results suggest that soil P sorption capacities can remain lower in manure-amended than unamended soils for many years, even after manure applications are discontinued. Further investigations will be required to determine the fate of manure P not sorbed in calcareous soils with a history of manure applications. If manure P is not precipitated in Ca–P compounds, sorbed on clay surfaces and in organic-mineral complexes or immobilized in organic matter, it could be transported from soils to surface water bodies.

CONCLUSIONS

Phosphorus sorption by calcareous soils that received cattle feedlot manure annually for up to 25 years was best described by the Freundlich equation, although results from the Langmuir equation suggested that both P sorption maxima and P binding energy were lower in manure-amended than unamended soils. It appeared that P would be more readily displaced from sorption sites in manure-amended than unamended soils. The P sorption capacity of non-irrigated and irrigated soils declined with increasing soil available P concentrations, but the P sorption capacity of surface soils that received manure annually for up to 25 years were generally not more than 50% lower than the P sorption capacity of unamended soils. Soil organic matter content, pH and surface chemistry was altered by manure application, and may explain why the P sorption capacity did not decline more as soil available P concentrations increased. After manure applications were discontinued, the P sorption capacity increased as soil available P concentrations declined, likely through depletion of available P through plant uptake. However, it took more than a decade for the P sorption capacity of soil that received 30 Mg manure (wet weight)

ha^{-1} for thirteen years to reach a level that was not different from soils with no history of manure applications. Since it may take more than 10 years of continuous cropping to reduce soil saturation with P following long-term manure applications based on fertilizer N requirements, there is a risk of P leaching through the soil profile and possibly reaching groundwater in soils that are saturated with P, particularly in irrigated soils. Our results support the adoption of manure application rates based on crop P requirements rather than crop N requirements. Further investigation will be required to determine the fate of manure P not sorbed in manure-amended calcareous soils to promote manure management practices that are agronomically sound and protect water quality.

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