CALCIUM CO-AMENDMENTS MODIFY EXTRACTABLE ORTHOPHOSPHATE LEVELS IN FRESH AND COMPOSTED CATTLE MANURE

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Abstract. Historically, manure has been recognized as an excellent soil amendment that can improve soil quality and provide nutrients for crop production. In areas of high animal density, however, repeated high applications of manure can lead to phosphorus (P) accumulations in soils, and migration of excess manure P from soils to ground and surface waters has been linked to eutrophication of aquatic systems. The objectives of this study were to determine whether the calcareous co-amendments CaO, $Ca(OH)_2$, cement kiln dust (CKD), and a cement kiln dust/fly ash mixture (CKD/FA) could lower the amount of extractable orthophosphate (ortho-P) in fresh and composted manure, and to monitor extractable ortho-P concentrations in cropped soils that received fresh and composted manure mixed with calcareous co-amendments. Extractable ortho-P concentrations declined by up to 37% in fresh manure and 49% in composted manure with the application of 200 g co-amendment kg^{-1} manure (dry weight basis), and lower extractable ortho-P levels persisted during a 6 week laboratory incubation. In a greenhouse experiment, fresh manure mixed with CaO and $Ca(OH)_2$ co-amendments inhibited P uptake by barley and decreased extractable ortho-P concentrations in soils. Chemical and biological reactions in plant-soil systems led to the apparent dissolution or desorption of ortho-P from fresh manure mixed with CKD and CKD/FA and composted manure-co-amendment mixtures. Factors controlling the stability of newly formed Ca-P compounds in manure after they are added to plant-soil systems will require further study.

Keywords: barley production, calcium co-amendments, composted beef manure, fresh beef manure, incubation, orthophosphate, phosphorus, soil

1. Introduction

A major concern in areas of high animal density is the potential for water pollution resulting from improper storage or disposal of manure. Manure from commercial feedlots is generally disposed through land application, but most feedlots have a limited land base, and it is often not economical to broaden the land base by hauling manure long distances (Freeze *et al.*, 1999; McKenzie *et al.*, 2000). As a result, the land closest to cattle feedlots may be amended with large quantities of feedlot manure on a continual basis.

It has become apparent in the past two decades that manure applications based on crop N requirements provide P in excess of crop P requirements, and that repeated applications of manure lead to P accumulation in agricultural soils (Chang

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et al., 1991; Paik *et al*., 1996; Sims *et al*., 1998; Whalen and Chang, 2001). The primary concern with P pollution is eutrophication of surface waters, mainly through P transport in surface runoff, although leaching and erosion also contribute to P pollution of surface waters (Sharpley *et al*., 1993; Lennox *et al*., 1997). Intensive confined livestock production operations have the potential to contribute to P pollution in water bodies if the manure from these operations is stored or disposed of improperly.

There is a large body of evidence indicating that the amount of P transported from soils in runoff is related strongly to soil test P concentrations (Mozaffari and Sims, 1994; Sharpley, 1995; Liu *et al*., 1997). These findings have led to the identification of critical levels of soil test P, and there are now regulations in some parts of the U.S. and Europe restricting manure or fertilizer applications to soils with soil test P levels that exceed the critical limits. Soil test P critical levels in the U.S. range from 75 to 200 mg P kg^{-1} in the top 15 cm of soil (Sharpley and Tunney, 2000). Livestock producers in the U.S. are not permitted to apply manure in excess of crop P requirements when critical levels of soil test P are reached. Soil test P measures ortho-P extracted from soil solution and soil particles, and is often used as measure of the P available to plants during a growing season. Since the guidelines are based on extractable ortho-P rather than total P, it seems possible that more manure could be safely applied to cropland if the quantity of extractable ortho-P in manure was lowered.

Ortho-P is readily adsorbed and precipitated by soil minerals, including Ca, Al, and Fe. Calcium phosphates such as insoluble hydroxyapatite ($Ca_{10}(PO_4)_6(OH)_2$) and fluorapatite ($Ca_{10}(PO_4)_6F_2$) are formed with ortho-P reacts with Ca, while aluminum and iron phosphates include variscite ($AlPO_4 \cdot 2\ H_2O$) or strengite ($FePO_4 \cdot 2\ H_2O$) (Parfitt, 1978). A variety of nonhazardous municipal and industrial by-products, such as alum sludge, bauxite red mud, cement kiln dust, caliche and fly ash, have been tested to lower the ortho-P in manure and soils with high extractable ortho-P concentrations (Peters and Basta, 1996; Robertson *et al*., 1997; Dao, 1999). The water-extractable P concentrations in stockpiled and composted cattle manure declined by up to 50 to 90% following additions of 500 g alum sludge, caliche or fly ash per kg of manure (Dao, 1999). Water-extractable P concentrations in sandy-loam and clay-loam soils were lower following application of stockpiled or composted manure treated with co-amendments than untreated manure, indicating stability of P complexes in manure and reactions between co-amendments and soils that decreased soil P concentrations (Dao, 1999). Other co-amendments, including quick lime, slaked lime, ferrous chloride and ferrous sulfate, have proven effective at lowering the water-extractable P concentration in poultry litter (Moore and Miller, 1994; Shreve *et al*., 1995). The co-amendment of manure with materials containing Ca minerals may have the potential to stabilize ortho-P, protecting soil and water quality in sensitive watersheds.

However, most studies have not considered the stability of P from manure mixed with co-amendments in plant-soil systems. Plant roots exert a considerable influence on P cycling in soils by exuding organic acids that solubilize soil minerals and through interactions with microbial populations (Hale and Moore, 1979). Phosphatase enzymes released from plant roots and soil microorganisms catalyze the mineralization of organic P compounds to inorganic P forms available for plant uptake (Speir and Ross, 1978). In addition, many plants form associations with mycorrhizal fungi that can increase P mineralization and facilitate the transfer of P through fungal hyphae to the plant (Harley and Smith, 1983). The effects of plants on P cycling must be investigated to determine whether the ortho-P in manure treated with co-amendments remains in relatively stable forms when applied to cropped agricultural soils.

The objectives of this study were to determine (1) how much of different calcareous co-amendments would lower the amount of extractable ortho-P in fresh and composted manure significantly, and (2) whether insoluble ortho-P compounds in Ca-treated fresh and composted manure can be mobilized in the presence of plants.

2. Materials and Methods

Fresh manure was collected from an open, unpaved commercial cattle feedlot at the Lethbridge Research Centre in July 1999. Fresh manure contained cattle manure and straw bedding, and the material was finely ground (< 2 mm mesh) to ensure sample homogeneity and stored at –20 °C prior to use. Cattle manure from the feedlot was composted aerobically (turned with a windrow turner every 2 weeks) for 3 months and collected in February 2000. Composted manure was coarsely sieved (< 25 mm mesh) prior to use. Some physical and chemical properties of the fresh and composted manure used in the study are given in Table I.

2.1. Incubation experiment

The quantities of manure used for the incubation experiment were about 250 g (oven-dry basis) of fresh manure (550 g of fresh manure, wet weight) and about 500 g (oven-dry basis) of composted manure (700 g of composted manure, wet weight). Fresh and composted manure were placed in separate 4 L plastic containers and calcium co-amendments were mixed thoroughly by hand (stirring and shaking) at rates of 0, 25, 50, 100 and 200 g co-amendment kg^{-1} manure (oven-dry basis). Four replicate containers of each manure type and co-amendment rate were prepared giving a total of 40 containers (2 types of manure $\times$ 5 co-amendment rates $\times$ 4 replicate bins). Two of the calcium co-amendments used in this study were CaO and $Ca(OH)_2$ (supplied by the Exshaw Plant of Continental Lime, Calgary, Alberta, Canada). The other two calcium co-amendments were byproducts of the cement industry, namely cement kiln dust (CKD) and a cement kiln dust-fly ash mixture (CKD/FA) containing about 35% cement kiln dust and 65% fly ash (supplied by Lafarge Canada, Edmonton, Alberta, Canada). No extra water was added to the

TABLE I

Properties of fresh and composted cattle manure used in this study[a]

Parameters	Fresh manure	Composted manure	Method used
Moisture content (kg kg^{-1})	0.45	0.27	Oven-dried, 105 °C for 48 h
pH	6.8	7.1	1:2 manure:water slurry
Electrical conductivity (dS m^{-1})	29.2	21.9	1:2 manure:water slurry
Organic C (g kg^{-1})	249.3	254.1	Carlo-Erba C and N analyzer (Milano, Italy).
Total N (g kg^{-1})	22.8	16.6	Carlo-Erba C and N analyzer (Milano, Italy).
Total P (g kg^{-1})	7.0	5.4	H_2O_2/H_2SO_4 digest, molybdate-ascorbic acid method
$NH_4 + NO_3$(g kg^{-1})	2.9	1.1	2M KCl extract, phenate/Cd reduction methods
Ortho-P (g kg^{-1})	5.2	2.9	$NaHCO_3$-soluble P, molybdate-ascorbic acid method
Available K (g kg^{-1})	21.5	5.8	Saturated paste extract, AAS
Available S (mg kg^{-1})	4.9	0.7	Saturated paste extract, methylthymol blue
Available Ca (cmol kg^{-1})	1.4	0.5	Saturated paste extract, AAS
Available Mg (cmol kg^{-1})	5.8	0.8	Saturated paste extract, AAS

[a] Values are the means of at least ten replicate determinations. Nutrient analyses are expressed on a per kg of manure (dry weight) basis.

mixtures. The plastic containers had no drains to prevent nutrient losses, were sealed to the atmosphere with a snap-on lid and incubated at 20 °C (± 1 °C) in a controlled climate room for 6 wks. The effects of co-amendments on phosphorus availability in fresh and composted cattle manure was determined by taking subsamples from each container within 2 h of adding the co-amendments (week 0) and then 1, 2, 4 and 6 wks after adding the co-amendments.

2.2. GREENHOUSE EXPERIMENT

Barley (*Hordeum vulgare L.* cv. Galt) was seeded in pots (15 cm tall × 10 cm diameter) that were 75 to 80% full of soil. Each pot contained 1.0 kg (oven-dry basis) of sieved (< 2 mm) soil obtained from the A horizon (0- to 15 cm) of a sandy-loam Orthic Brown Chernozemic soil under native rangeland. The soil contained 720 g sand kg^{-1}, 60 g silt kg^{-1} and 220 g clay kg^{-1} with 8 g organic C kg^{-1} and pH 6.9 (in $CaCl_2$). Soils were moistened to 75% of field capacity and fertilized with macronutrients (100 mg N kg^{-1}, 100 mg K kg^{-1}, and 20 mg S kg^{-1}) and micronutrients (2 mg B kg^{-1}, 2 mg Cu kg^{-1}, 8 mg Fe kg^{-1}, 4 mg Mn kg^{-1}, 0.8 mg Mo kg^{-1} and 8 mg Zn kg^{-1}) prior to seeding. The P recommendations for barley production in southern Alberta for the soil used in this study were 40 kg P ha^{-1}, which is equivalent to 25 mg P kg^{-1} (Alberta Agriculture, 1997). Therefore, we incorporated 10 g kg^{-1} (wet weight basis) of the fresh manure mixtures, and 12 g kg^{-1} (wet weight basis) of the composted manure mixtures from the incubation study into the top 10 cm of soil in pots. These quantities were chosen to meet the P fertilizer requirements of barley, based on the total P content of fresh and composted manure. We also prepared eight control pots that received 25 mg P kg^{-1} from inorganic KH_2PO_4 fertilizer. Pots were seeded on March 24, 2000, and ten days later, the barley was thinned to five plants per pot. The barley was grown under full light intensity (about 350 μmoles m^{-2} s^{-1}) with a 12 h photoperiod in a controlled climate growth chamber (25 $\pm$ 1 °C during the day, 18 $\pm$ 1 °C at night). The pots had no drains to prevent nutrient losses, and were watered regularly with distilled water to prevent moisture stress. Additional N fertilizer (105 mg N kg^{-1} soil) was added 30 and 60 d after seeding. Plants were harvested at maturity on June 20, 2000 in pots receiving composted manure and inorganic P fertilizer, and on June 26, 2000 in pots receiving fresh manure.

2.3. Manure, soil and plant analysis

Extractable ortho-P and mineral N concentrations in manure-co-amendment mixtures were determined on sub-samples obtained after 0, 1, 2, 4 and 6 wk of incubation. After barley was harvested, soil was separated from plant roots, sieved (< 2 mm mesh), and analyzed for extractable ortho-P and mineral N. Extractable ortho-P was determined in Kelowna (0.015*M* NH_4F + 0.25*M* CH_3COOH) soil extracts (1:25 manure:extractant, 1:10 soil:extractant). Ortho-phosphate was measured colorimetrically by the ammonium molybdate-ascorbic acid method using a Technicon IV flow-injection autoanalyzer (Technicon Industrial Systems, Tarrytown, New York, U.S.A.). Mineral N (NH_4-N and NO_3-N) was determined in 2*M* KCl extracts (1:10 manure:extractant, 1:5 soil:extractant) and measured colorimetrically using the phenate and cadmium reduction-diatotization methods with a Technicon II flow-injection autoanalyzer (Technicon Industrial Systems, Tarrytown, New York, U.S.A.).

The total C, N and P content of manure-co-amendment mixtures from the incubation experiment was analysed on sub-samples collected after 6 weeks of incubation. Soil, plant grain and plant straw samples collected after barley harvest were also analyzed for total N and P content. Samples were oven-dried (70 °C for 48 h), ground and analyzed for total C and N content on a Carlo-Erba C and N analyser (Milan, Italy). The total P content of oven-dried, ground manure and soil samples was determined using the wet digestion method of Parkinson and Allen (1975). Briefly, one g of the sample was weighed into a digestion tube and digested at 360 °C for 2.5 h with concentrated H_2SO_4, Li_2SO_4, Se powder and H_2O_2. The total P content of oven-dried, ground plant tissue samples was determined by wet digestion at 300 °C with concentrated H_2SO_4 and H_2O_2 (Thomas *et al.*, 1967). Ortho-P in digests was analyzed colorimetrically by the ammonium molybdate-ascorbic acid method using a Technicon IV flow-injection autoanalyzer (Technicon Industrial Systems, Tarrytown, New York, U.S.A.).

The pH of manure-co-amendment mixtures from the incubation experiment was determined in 1:10 manure:distilled water slurries of sub-samples taken after six weeks of incubation. The pH of soils from the greenhouse experiment after barley harvest was determined in 1:2 soil:distilled water slurries.

2.4. Calculations

Extractable ortho-P in manure-co-amendment mixtures from the incubation experiment was fit to a first-order exponential decay rate model to determine the maximum ortho-P concentration in solution (P_{max})

$$P_i = P_{max} + k_P\, e^{-t} \tag{1}$$

P_i is the extractable ortho-P concentration (g P kg^{-1}), k_P is the rate constant (week^{-1}) and t is the incubation time (week). The maximum mineral N concentration in solution (N_{max}, mg N kg^{-1}) was calculated by substituting mineral N for extractable ortho-P in Equation 1.

2.5. Statistical analysis

Data were evaluated statistically by ANOVA in a general linear model (GLM) using SAS software (SAS Institute, 1990). The containers in the incubation experiment were arranged in a completely random design. Extractable ortho-P concentrations were log transformed to equalize variance, and the effects of co-amendment rates (0, 25, 50, 100 and 200 g kg^{-1}), incubation time (0, 1, 2, 4 and 6 wks) and co-amendment (CaO, $Ca(OH)_2$, CKD and CKD/FA) for each type of manure (fresh and composted) were evaluated with ANOVA. The interaction between co-amendment rate and incubation time was not statistically significant ($P > 0.05$), so the data from different incubation times were pooled among amendment rates for analysis. The effects of co-amendment rates and co-amendment source on extractable ortho-P and pH concentrations were then evaluated with a two-way ANOVA for each manure treatment, and compared with an LSD test at the 95% confidence level. Results reported in all tables and figures are untransformed means (± standard errors).

First order rate equations to calculate P_{max} and N_{max} and associated rate constants were fit with a nonlinear least square regression analysis using the PROC NLIN function of SAS (SAS Institute, 1990). Linear regressions were fit using the SAS/INSIGHT function of SAS software (Version 6.12).

Pots for the greenhouse experiment were arranged in a completely random design in controlled climate growth chambers. The effects of fertilizer (fresh manure, composted manure and inorganic fertilizer), co-amendment rates (0, 25, 50, 100 and 200 g kg^{-1}) and co-amendment source (CaO, $Ca(OH)_2$, CKD and CKD/FA) on barley yield, nutrient uptake, soil pH and extractable ortho-P after barley harvest were evaluated with ANOVA. The main effects of fertilizer and co-amendment source affected barley yield and nutrient uptake significantly, and differences between co-amendments for each fertilizer were evaluated with a one-way ANOVA and compared with an LSD test at the 95% confidence level. Differences in soil pH and extractable ortho-P after barley harvest for each fertilizer were evaluated with a two-way ANOVA of the co-amendment rate x co-amendment source interaction, and results were compared statistically with an LSD test at the 95% confidence level.

3. Results and Discussion

3.1. Incubation Experiment

3.1.1. *pH of Manure-Co-Amendment Mixtures*

The pH of fresh and composted manure mixed with co-amendments were measured after the 6 wk incubation, and the pH of untreated fresh and composted manure increased by about 1 unit during the incubation (Table I, Figure 1A, 1B). The pH of fresh manure mixed with co-amendments ranged from 8.3 to 12.0, whereas the pH of composted manure mixed with co-amendments was between 8.2 and 12.0 (Figure 1A, 1B). The pH of fresh manure mixed with CaO, CKD, more than 25 g kg^{-1} of $Ca(OH)_2$ or more than 50 g kg^{-1} of CKD/FA was significantly ($P <$ 0.05, LSD) higher than the pH of untreated fresh manure (Figure 1A). The fresh manure mixtures with the highest pH were those that received 200 g kg^{-1} of CaO and $Ca(OH)_2$ (Figure 1A). Composted manure mixed with CaO, $Ca(OH)_2$, more than 25 g kg^{-1} of CKD or more than 50 g kg^{-1} of CKD/FA had a significantly ($P <$ 0.05, LSD) higher pH than untreated compost (Figure 1B). The composted manure mixtures with the highest pH were those that received 100 and 200 g kg^{-1} of CaO and $Ca(OH)_2$ (Figure 1B).

It is well established that calcareous materials such as CaO and $Ca(OH)_2$ increase the pH of soils and sludges, and calcareous industrial byproducts, such as cement flue dust (Oguntoyinbo *et al.*, 1996) and fly ash (Sims *et al.*, 1995) have been shown to neutralize soil acidity. Calcareous materials also alter the pH of manure. Moore and Miller (1994) found the pH of poultry litter (pH = 7.30) increased to 12.44 when mixed with 150 g CaO kg^{-1} and to 12.33 when mixed with 200 g $Ca(OH)_2$ kg^{-1}. The pH of beef manure amended with 500 g fly ash kg^{-1} increased from 8 to 8.9 in stockpiled manure and from 8.4 to 9.6 in composted manure (Dao, 1999).

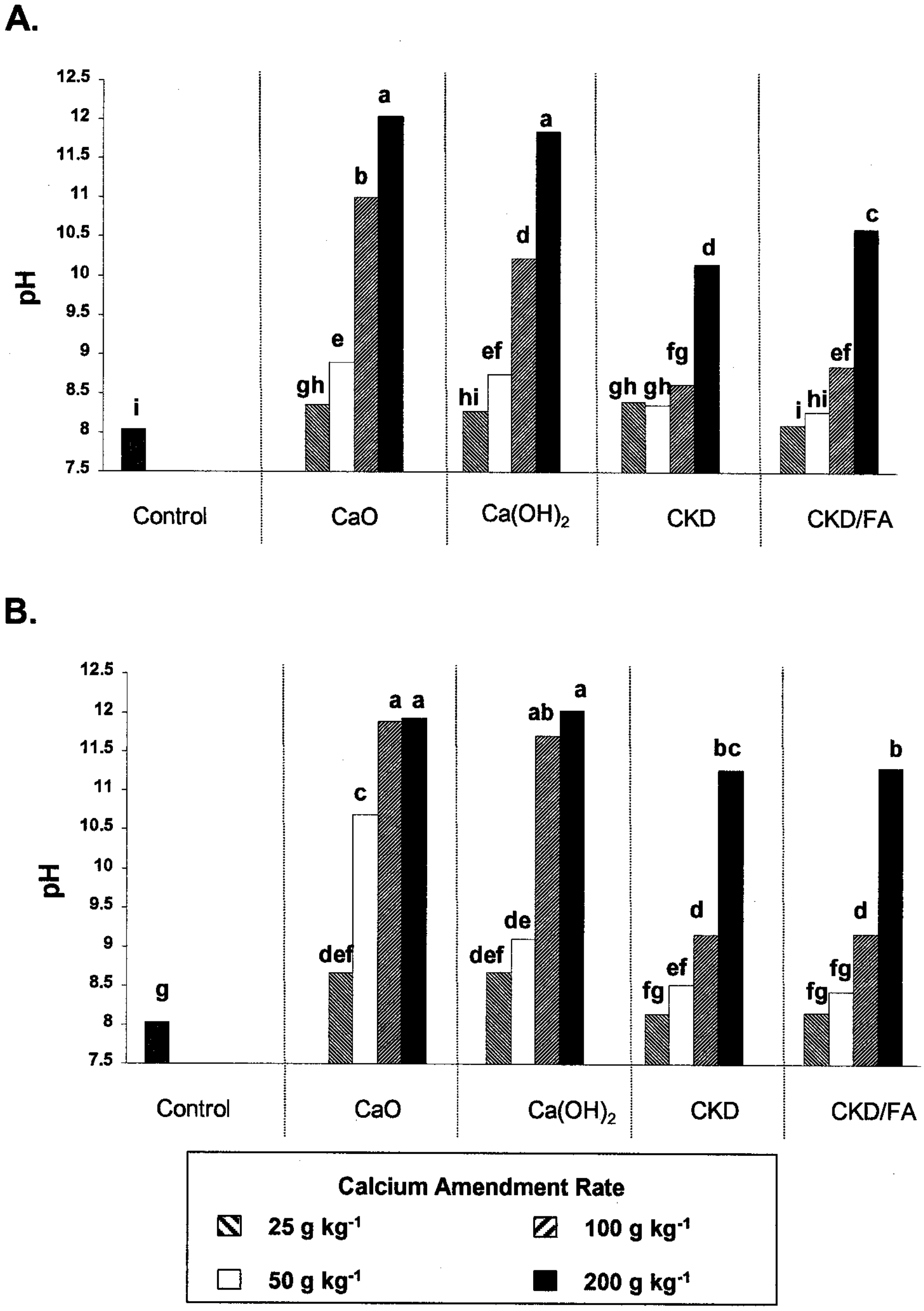

Figure 1. Effect of different rates of co-amendments on the pH of (A) fresh manure and (B) composted manure after a 6 wk laboratory incubation. Means with the same letter are not significantly different at $P < 0.05$ (LSD test).

3.1.2. *Extractable Ortho-P in Manure-Co-Amendment Mixtures*

There was an immediate effect on extractable ortho-P concentrations when fresh and composted manure were mixed with CaO, $Ca(OH)_2$, CKD and CKD/FA co-amendments. The interaction between co-amendment rate and incubation time was not significant ($P < 0.05$), indicating that co-amendments reacted rapidly with ortho-P (within 2 h of mixing with fresh or composted manure), and the reaction products persisted during the 6 wk incubation. Fresh manure mixed with more than 25 g kg^{-1} of CaO and CKD/FA or with 25, 100 or 200 g kg^{-1} of $Ca(OH)_2$ had significantly ($P < 0.05$, LSD) lower extractable ortho-P concentrations than untreated manure (Table II). However, only when fresh manure was mixed with 200 g kg^{-1} of CKD was there significantly ($P < 0.05$, LSD) less extractable ortho-P than untreated manure (Table II). Extractable ortho-P concentrations were 24 to 37% lower when fresh manure was mixed with 200 g kg^{-1} of co-amendments than untreated (Table II). Compost mixed with co-amendments had significantly ($P <$ 0.05, LSD) less extractable ortho-P than untreated compost, and the extractable ortho-P concentrations were 46 to 49% lower in mixtures containing 200 g kg^{-1} of co-amendments than untreated compost (Table II).

The efficacy of co-amendments in lowering manure ortho-P varies with the type of manure, the rate and type of co-amendment added, and the extractant used to evaluate ortho-P concentrations. Other researchers have found that co-amendments could lower the water-extractable ortho-P concentration of manure up to 90% or more (Moore and Miller, 1994; Shreve *et al.*, 1995; Dao, 1999). Extractable ortho-P concentrations in fresh and composted manure were measured in acidic Kelowna extracts, a more rigorous extractant than water used routinely for soil test P analysis in Alberta, Canada. Dao (1999) found three times more ortho-P in manure-co-amendment mixtures when they were measured in acidic Mehlich III extracts than water extracts, and suggested that some of the reaction products (e.g., calcium phosphates) were redissolved by the ammonium fluoride-strong acid extractant. The Kelowna extractant has a similar chemical composition as the Mehlich III extractant, and could have redissolved some of the reaction products in manure mixed with co-amendments. However, the extractable ortho-P concentration was up to 37% lower in co-amended than untreated fresh manure, and up to 49% lower in co-amended than untreated composted manure. These results indicate that CaO, $Ca(OH)_2$, CKD and CKD/FA co-amendments transformed significant quantities of ortho-P in fresh and composted manure into forms that were stabilized against acidic extractants. Dao, (1999) found that the Mehlich III-extractable P concentrations were 62% lower in composted beef manure mixed with 100 g fly ash kg^{-1} than untreated compost, and 87% lower in stockpiled beef manure mixed with 100 g fly ash kg^{-1} than untreated manure after a six week incubation at 35 °C. Caliche co-amendments applied at 100 g co-amendment kg^{-1} did not lower the Mehlich III-extractable P concentrations in composted and stockpiled beef manure below the levels in untreated manure (Dao, 1999). In this study, all four co-amendments

TABLE II

Mean extractable ortho-P concentrations in fresh and composted manure mixed with four rates of CaO, $Ca(OH)_2$, CKD or CKD/FA co-amendments

Treatment	Rate ($g\ kg^{-1}$)[a]	Extractable ortho-P ($g\ kg^{-1}$)	
		Fresh Manure	Composted Manure
None	0	4.11 a	3.07 a
CaO	25	3.74 abc	2.48 bc
CaO	50	3.61 bc	2.37 bc
CaO	100	3.60 bc	2.27 bcd
CaO	200	2.60 e	1.65 e
$Ca(OH)_2$	25	3.57 bc	2.19 bc
$Ca(OH)_2$	50	4.06 ab	2.22 bcd
$Ca(OH)_2$	100	3.39 cd	1.96 de
$Ca(OH)_2$	200	3.13 de	1.59 e
CKD	25	3.75 abc	2.48 bc
CKD	50	3.82 abc	2.59 b
CKD	100	3.94 ab	2.13 cd
CKD	200	3.00 de	1.66 e
CKD/FA	25	3.69 abc	2.55 bc
CKD/FA	50	3.38 cd	2.18 bcd
CKD/FA	100	3.59 bc	2.49 bc
CKD/FA	200	2.90 de	1.57 e

[a] Rates are grams of co-amendment kg^{-1} manure (dry weight basis). Means within a column followed by the same letter are not significantly different ($P < 0.05$, LSD).

tested were equally effective in lowering extractable ortho-P concentrations in fresh and composted manure when applied at a rate of 200 g co-amendment kg^{-1}.

The maximum ortho-P concentration in solution (P_{max}) in fresh and composted manure mixed with co-amendments was linearly related to the pH of manure-co-amendment mixtures (Figures. 2A, 2B). The slopes of the regression lines describing these relationships were negative, indicating a decline in P_{max} with increasing pH in manure-co-amendment mixtures (Figs. 2A, 2B). Soluble forms of P are readily precipitated by Ca in soils at moderate to high pH (pH > 6) into relatively insoluble P compounds (Lindsay, 1979), and, if calcite is present, hydroxy apatites and calcium phosphates are adsorbed on $CaCO_3$ surfaces (Parfitt, 1978). Our findings suggest that the quantities of ortho-P in manure precipitated or absorbed to calcareous materials increased as the pH of manure-co-amendment mixtures increased. Therefore, calcareous materials with a high neutralizing power may be most effective at lowering ortho-P concentrations in fresh and composted manure.

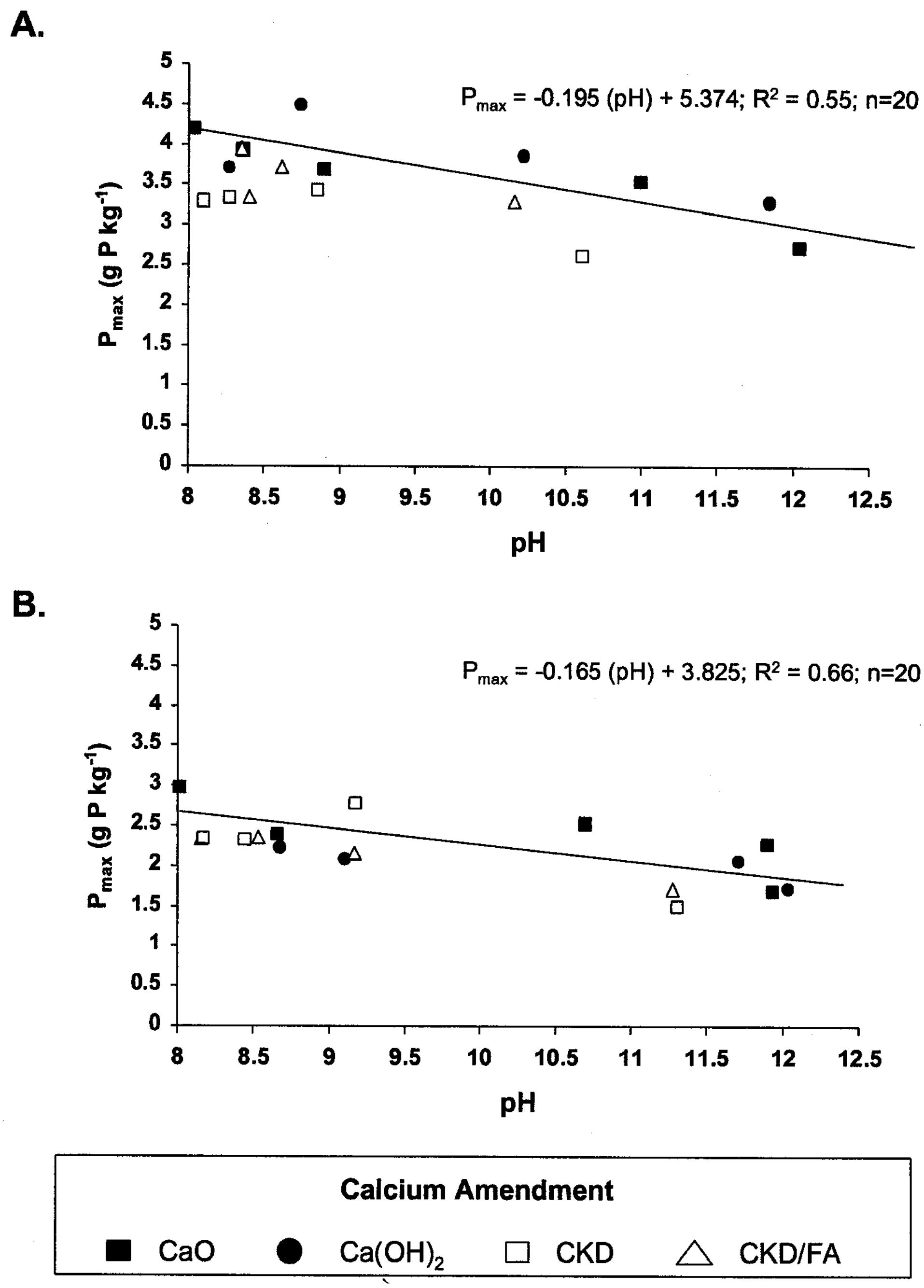

Figure 2. Relationship between pH and the maximum extractable ortho-P concentration (P_{max}) in (A) fresh manure and (B) composted manure.

TABLE III

Mean mineral N (NH_4-N plus NO_3-N) concentrations in fresh and composted manure mixed with four rates of CaO, $Ca(OH)_2$, CKD or CKD/FA co-amendments immediately following mixing (week 0) and after 6 wks of incubation

Treatment	Rate ($g\ kg^{-1}$)[a]	Mineral N ($g\ kg^{-1}$)			
		Fresh Manure		Composted Manure	
		Week 0	Week 6	Week 0	Week 6
None	0	2.25 ± 0.03	0.13 ± 0.01	1.08 ± 0.06	1.02 ± 0.06
CaO	25	0.92 ± 0.09	0.33 ± 0.09	0.45 ± 0.08	0.55 ± 0.13
CaO	50	0.51 ± 0.07	0.44 ± 0.11	0.24 ± 0.03	0.42 ± 0.03
CaO	100	0.48 ± 0.10	0.20 ± 0.03	0.44 ± 0.04	0.40 ± 0.03
CaO	200	0.58 ± 0.05	0.17 ± 0.01	0.40 ± 0.02	0.40 ± 0.01
$Ca(OH)_2$	25	1.51 ± 0.13	0.08 ± 0.01	0.58 ± 0.05	0.54 ± 0.12
$Ca(OH)_2$	50	0.93 ± 0.13	0.90 ± 0.11	0.34 ± 0.06	0.44 ± 0.04
$Ca(OH)_2$	100	0.66 ± 0.06	0.45 ± 0.17	0.44 ± 0.11	0.44 ± 0.05
$Ca(OH)_2$	200	0.53 ± 0.04	0.19 ± 0.02	0.39 ± 0.03	0.48 ± 0.02
CKD	25	2.28 ± 0.14	0.18 ± 0.05	1.01 ± 0.07	1.00 ± 0.05
CKD	50	1.61 ± 0.13	0.33 ± 0.13	0.64 ± 0.07	0.90 ± 0.13
CKD	100	0.98 ± 0.07	1.29 ± 0.03	0.53 ± 0.11	0.69 ± 0.07
CKD	200	0.91 ± 0.06	0.29 ± 0.04	0.48 ± 0.08	0.39 ± 0.04
CKD/FA	25	2.04 ± 0.10	0.12 ± 0.01	0.77 ± 0.05	0.99 ± 0.13
CKD/FA	50	1.18 ± 0.09	0.09 ± 0.01	0.55 ± 0.12	0.54 ± 0.08
CKD/FA	100	0.90 ± 0.15	0.64 ± 0.17	0.50 ± 0.03	0.67 ± 0.10
CKD/FA	200	0.54 ± 0.04	0.24 ± 0.04	0.28 ± 0.04	0.47 ± 0.07

[a] Rates are grams of co-amendment kg^{-1} manure (dry weight basis).

3.1.3. *Mineral N in Manure-Co-Amendment Mixtures*

There was an immediate decline in the mineral N (NH_4-N and NO_3-N) concentrations in fresh and composted manure mixed with co-amendments (Table III). During the 6 week incubation, there was a net decline in mineral N concentrations in fresh manure and most mixtures of fresh manure and co-amendments (Table III). These results suggest transformations of N to forms other than mineral N during the incubation, which could have included N immobilization in microbial biomass or N volatilization/denitrification in NH_3, N_2 and N_2O gases. However, mineral N concentrations in composted manure and mixtures of composted manure and co-amendments generally were unchanged or increased slightly during the incubation, indicating net N mineralization in some mixtures (Table III). Between 19 and 42% of the total N in beef manure may be lost through NH_3 volatilization during composting, and the N remaining in the composted manure is thought to be relatively more stable than N in fresh or stockpiled beef manure (Eghball *et al.*, 1997).

The maximum mineral N concentration (N_{max}) in composted manure mixed with co-amendments, but not in fresh manure mixed with co-amendments, was linearly related to the pH of the manure-co-amendment mixtures (Figures 3A, 3B). The N_{max} in composted manure mixed with calcium co-amendments declined with increasing pH, but the trend was not as clear when fresh manure was mixed with calcium co-amendments (Figures 3A, 3B). One possible explanation is that a portion of the N in manure-co-amendment mixtures was lost via NH_3 volatilization, a reaction that depends on pH (pK_a = 9.3) (Havlin *et al.*, 1999). Yet, Moore *et al.* (1995) found that NH_3 volatilization from poultry manure mixed with 25 or 50 g $Ca(OH)_2$ kg^{-1} was not different from untreated manure, and manure mixed with alum, a water-soluble Al/Fe salt, had lower NH_3 volatilization than untreated manure. Further research is required to determine how calcareous materials alter mineral N concentrations in fresh and composted manure.

3.1.4. *Total C, N and P in Manure-Co-Amendment Mixtures*

There was little difference in the total C, N and P contents of fresh and composted manure analyzed prior to the study and after the 6 wk incubation (data not shown). The total C of fresh and composted manure mixed with co-amendments ranged from 222 to 292 g C kg^{-1}. Total N was between 13.7 and 18.6 g N kg^{-1} in composted manure-co-amendment mixtures and 19.1 to 23.0 g N kg^{-1} in fresh manure-co-amendment mixtures. Composted manure mixed with co-amendments contained 4.2 to 6.0 g P kg^{-1}, and fresh manure mixed with co-amendments contained 7.5 to 8.9 g P kg^{-1}. The C/N ratios of fresh manure mixed with co-amendments were between 10 and 14, whereas the C/N ratios of composted manure mixed with co-amendments were between 15 and 19. The N/P ratios of manure mixed with co-amendments ranged from 2 to 3 in fresh manure and from 3 to 4 in composted manure. Our results are similar to the findings of Dao, (1999), who found stockpiled beef cattle manure mixed with co-amendments had an N/P ratio between 3 and 5, and composted manure mixed with co-amendments had an N/P ratio between 3 and 4.

3.2. Greenhouse experiment

3.2.1. *Barley Yield and Nutrient Uptake*

Barley reached maturity 88 d after seeding under the temperature and light conditions in the growth chamber. Mean barley yields (grain plus straw) ranged from 9.3 to 11.2 g (dry mass) per pot in soils receiving untreated and co-amended manures, and did not differ among treatments (data not shown). The rate of calcium co-amendments mixed with fresh or composted manure did not affect barley grain and straw yields ($P > 0.05$), but barley yields were often influenced by the type of calcium co-amendment mixed with manure (Table IV). Grain yield were significantly ($P < 0.05$, LSD) lower in pots receiving untreated fresh manure or fresh manure mixed with CaO, $Ca(OH)_2$ and CKD/FA than inorganic fertilizer (Table IV). Straw

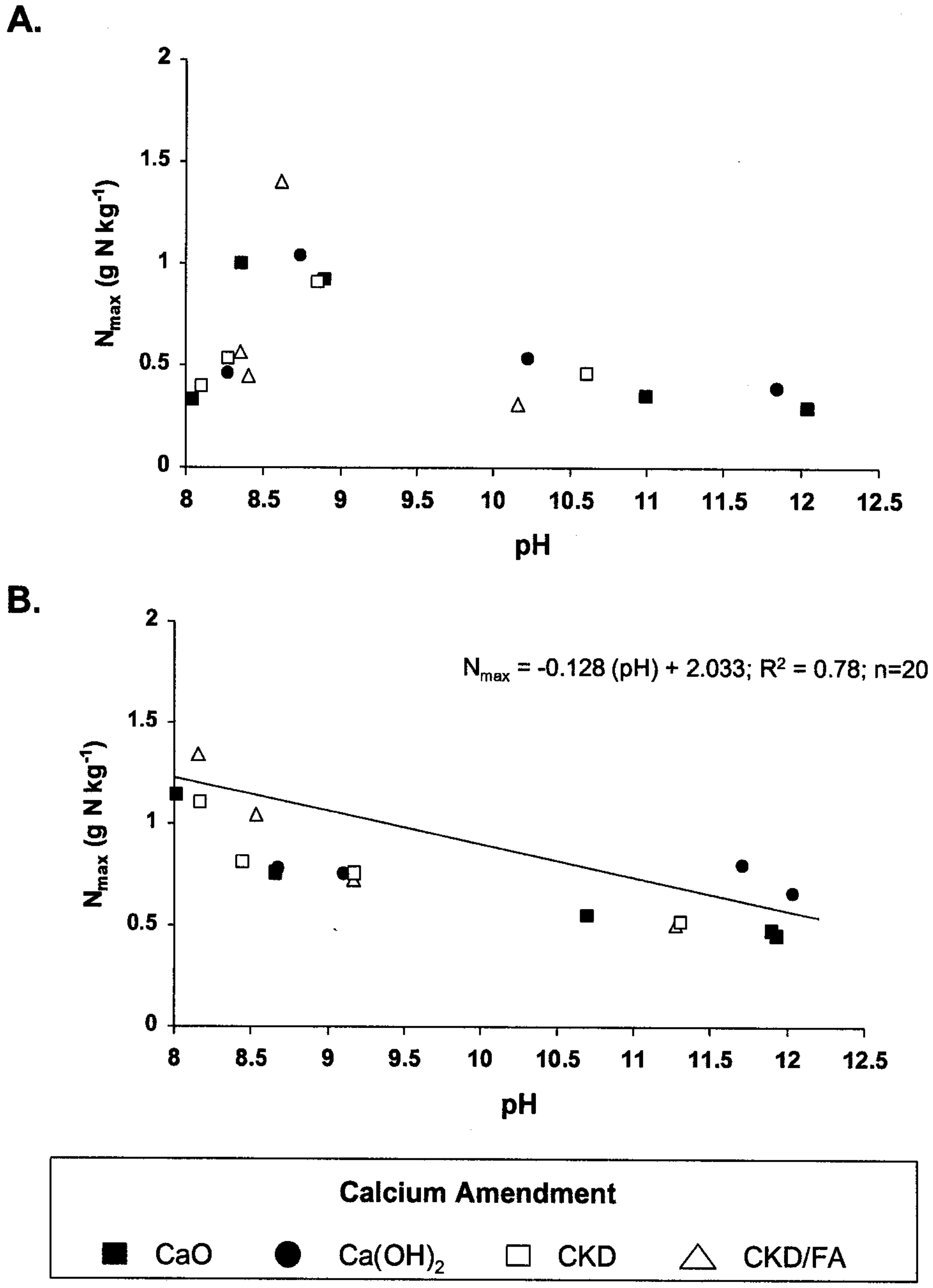

Figure 3. Relationship between pH and the maximum mineral N concentration (N_{max}) in (A) fresh manure and (B) composted manure.

TABLE IV

Barley grain and straw yields, and nutrient uptake in pots amended with inorganic fertilizer, fresh and composted manure. Manure was incubated with calcium co-amendments (CaO, $Ca(OH)_2$, CKD or CKD/FA) for 6 wks before it was added to pots

Treatment	Grain (g pot^{-1})	Straw (g pot^{-1})	Total N (g N pot^{-1})	Total P (g P pot^{-1})
	Inorganic P Fertilizer and Fresh Manure			
Fertilizer	3.0 a	6.1 b	53.8 a	11.6 a
Untreated	1.4 bc	8.9 a	27.3 bc	5.23 bc
CaO	0.9 c	8.7 a	23.5 c	4.36 c
$Ca(OH)_2$	1.2 c	8.1 a	29.4 bc	5.13 c
CKD	2.1 ab	8.5 a	41.8 ab	8.00 ab
CKD/FA	1.8 bc	9.0 a	39.1 abc	6.91 bc
	Inorganic P Fertilizer and Composted Manure			
Fertilizer	3.0 a	6.1 d	53.8 a	11.6 a
Untreated	2.4 a	8.8 a	55.9 a	9.53 a
CaO	3.2 a	6.6 cd	60.1 a	10.5 a
$Ca(OH)_2$	2.9 a	7.4 bc	57.8 a	9.95 a
CKD	3.3 a	7.7 b	61.4 a	10.8 a
CKD/FA	3.3 a	6.8 cd	59.0 a	10.9 a

yield was significantly ($P < 0.05$, LSD) greater in pots receiving fresh manure than inorganic fertilizer, and in pots receiving untreated composted manure, composted manure-$Ca(OH)_2$ and composted manure-CKD mixtures than inorganic fertilizer (Table IV). Fresh manure is well known to depress seedling germination and inhibit root growth (Zucconi *et al.*, 1981), and the lower grain production in pots receiving fresh manure than inorganic fertilizer may have been due to phytoxicity during the early stages of barley growth. This effect might have been overcome had we permitted the barley to grow longer, during which time more of the sugars and proteins in barley straw might have been translocated for grain production.

The amount of N and P removed in barley grain plus straw was greater in pots that received composted than fresh manure (Table IV). Uptake of N and P in barley was not affected by the rate of calcium co-amendments mixed with fresh or composted manure, although the type of calcium co-amendment mixed with fresh manure influenced N and P uptake significantly ($P < 0.05$) (Table IV). There was significantly ($P < 0.05$, LSD) greater N uptake in pots receiving inorganic fertilizer than pots receiving untreated fresh manure, fresh manure-CaO, and fresh manure-$Ca(OH)_2$ mixtures (Table IV). The P uptake by barley was significantly ($P < 0.05$, LSD) higher in inorganically-fertilized pots than pots amended with untreated fresh manure or fresh manure mixed with CaO, $Ca(OH)_2$, and CKD/FA (Table IV).

The N/P ratios of barley ranged from 5 to 6 in grain and straw. These results are consistent with the N/P ratios of 6 to 8 for barley under field conditions reported by Whalen and Chang (2001). An N/P ratio of 5 to 6 in barley indicates that the plant requires 5 or 6 units of N for every unit of P. The N/P ratio of fresh and composted manure was about 3, so for every 6 units of N applied, 2 units of P are applied as well. When the N/P ratio of feedlot manure is lower than the N/P ratio of cereal crops, P accumulation in soils under cereal production would be expected if soils receive repeated manure applications based on crop N requirements (Whalen and Chang, 2001). However, if ortho-P in excess of crop requirements is precipitated or adsorbed in forms not available for plants or susceptible to leaching and surface runoff, the threat to water quality from agricultural P could be minimized.

3.2.2. *Soil pH and Extractable Ortho-P after Barley Harvest*

The pH and extractable ortho-P concentration in soils that received inorganic fertilizer, fresh manure-co-amendment mixtures and composted manure-co-amendment mixtures was determined after barley harvest. Soil pH ranged from 7.0 to 8.1 in pots amended with fresh manure, and from 7.4 to 8.4 in pots amended with composted manure (Table V). The pH was significantly ($P < 0.05$, LSD) higher in pots that received manure mixed with 200 g kg^{-1} of CaO, $Ca(OH)_2$, CKD or CKD/FA than pots that were fertilized with inorganic fertilizers (Table V). The pH of a clay loam soil (initial pH = 5.7) amended with 10 g kg^{-1} of stockpiled beef manure-co-amendment mixtures increased by 0.3 pH units, whereas soils amended with 10 g kg^{-1} of composted beef manure that had been mixed with co-amendments increased by 0.5 pH units (Dao, 1999). However, the pH of a fine sandy loam soil (initial pH = 7.8) was not altered when stockpiled and composted manure mixed with co-amendments were applied (Dao, 1999). Our results show an increase of up to 0.6 and 0.9 pH units in pots receiving fresh and composted manure-co-amendment mixtures compared to inorganically fertilized pots.

The extractable ortho-P concentration in the soil before seeding was 91 mg P kg^{-1} (L. Cramer, personal communication), and after harvest ranged from 72 to 155 mg P kg^{-1}. There was significantly ($P < 0.05$, LSD) more extractable ortho-P in pots receiving untreated fresh manure, fresh manure mixed with 50 to 200 g CKD kg^{-1} and 100 g CKD/FA kg^{-1} than inorganically fertilized pots (Table V). Most pots amended with fresh manure-CaO and fresh manure-$Ca(OH)_2$ mixtures had significantly ($P < 0.05$, LSD) less extractable ortho-P than pots receiving untreated fresh manure, and pots receiving fresh manure mixed with 50 and 100 g CaO kg^{-1} had significantly ($P < 0.05$, LSD) less extractable ortho-P than inorganically fertilized pots (Table V). Only pots receiving composted manure mixed with 200 g $Ca(OH)_2$ kg^{-1} had significantly ($P < 0.05$, LSD) less extractable ortho-P than inorganically fertilized pots (Table V).

TABLE V

Soil pH and extractable ortho-P concentration in pots amended with inorganic fertilizer, fresh and composted manure after barley harvest. Manure was incubated with calcium co-amendments (CaO, $Ca(OH)_2$, CKD or CKD/FA) for 6 wks before it was added to pots

Treatment	Rate ($g\ kg^{-1}$)[a]	Fresh manure		Composted manure	
		pH	Extractable Ortho-P ($mg\ P\ kg^{-1}$)	pH	Extractable Ortho-P ($mg\ P\ kg^{-1}$)
Fertilizer	0	7.5 c	96 de	7.5 ef	96 abc
Untreated	0	7.4 c	118 bc	7.4 f	85 bcd
CaO	25	7.6 bc	101 cde	7.8 cd	92 bc
CaO	50	7.8 ab	72 f	7.9 cd	108 a
CaO	100	7.6 bc	81 f	7.7 de	97 abc
CaO	200	8.1 a	90 def	8.2 ab	103 ab
$Ca(OH)_2$	25	7.0 d	83 ef	7.8 cd	96 abc
$Ca(OH)_2$	50	7.3 cd	93 def	7.9 cd	102 ab
$Ca(OH)_2$	100	7.7 bc	100 cde	8.2 ab	97 ab
$Ca(OH)_2$	200	7.8 ab	86 ef	8.4 a	72 d
CKD	25	7.4 c	103 cde	7.7 de	88 bcd
CKD	50	7.5 c	126 b	7.8 cd	92 abc
CKD	100	7.5 c	125 b	8.0 bc	88 bcd
CKD	200	7.8 ab	155 a	8.3 a	93 abc
CKD/FA	25	7.5 c	114 bcd	7.5 ef	84 cd
CKD/FA	50	7.5 c	113 bcd	7.7 de	93 abc
CKD/FA	100	7.6 bc	122 bc	8.1 bc	103 ab
CKD/FA	200	7.9 ab	112 bcd	8.4 a	100 ab

[a] Rates are grams of co-amendment kg^{-1} manure (dry weight basis). Means within a column followed by the same letter are not significantly different ($P < 0.05$, LSD).

Pots receiving fresh manure mixed with CaO and $Ca(OH)_2$ tended to have the lowest extractable ortho-P concentrations after harvest, and the barley produced in these pots had lower P uptake than inorganically fertilized pots. Although there was more extractable ortho-P in pots receiving untreated fresh manure, fresh manure-CKD and fresh-manure-CKD/FA mixtures than inorganically fertilized pots, barley P uptake was lower in the pots that received untreated fresh manure and fresh manure-CKD/FA mixtures than inorganic fertilizer. Similar findings were observed for barley N uptake, despite regular application of inorganic N fertilizer during the greenhouse study. These findings suggest that fresh manure had an inhibitory, possibly phytotoxic, effect on nutrient uptake by barley. However, fresh manure mixed with CaO and $Ca(OH)_2$ co-amendments tended to inhibit P uptake by barley even more than untreated fresh manure, which was likely due to the formation of Ca-P complexes that were not solubilized in soil. The ortho-P from fresh manure that was apparently precipitated or absorbed to CKD and CKD/FA during the incubation study was more readily released when fresh manure co-amended with these materials was added to a plant-soil system. There was no difference in the P uptake by barley or the extractable ortho-P concentration after harvest when pots received composted manure-co-amendment mixtures. These results suggest that CaO and $Ca(OH)_2$ co-amendments transformed ortho-P from fresh manure, but not composted manure, into forms that were not extractable with a strong acidic extractant after soils were cropped.

Extractable ortho-P levels (measured with an acidic Mehlich III extract) decline when cement kiln dust and other co-amendments were mixed with soil (Peters and Basta, 1996) or when co-amendments were mixed with manure that was then applied to soil (Dao, 1999). There was a 5 to 7% decline in Mehlich III-P concentrations in sandy-loam and clay-loam soils receiving stockpiled beef manure mixed with fly ash compared with soils receiving untreated manure, and a 7 to 20% decline in the Mehlich III-P levels in these soils when they were amended with a composted beef manure-fly ash mixture compared with soils receiving untreated compost (Dao, 1999). However, there was no change in the Mehlich III-P concentrations in these soils when they received stockpiled or composted beef manure mixed with alum and caliche, compared to untreated manure. These results suggest that chemical or biological interactions between soils and manure treated with co-amendments can alter the stability of Ca-P and other compounds formed when co-amendments are mixed with manure (Dao, 1999). Our results are consistent with other reports, and it appeared the soil chemical and biological processes proposed to alter Ca-P stability had a more profound effect on Ca-P compounds formed in composted manure-co-amendments mixtures than fresh manure-co-amendment mixtures. Plant roots and soil microorganisms secrete organic acids and phosphatase enzymes to liberate sufficient P for their metabolic requirements, and these compounds may have dissolved or desorbed some of the P precipitated or adsorbed from fresh and composted manure by co-amendments. The processes controlling the stability of newly formed Ca-P compounds in manure after they are added to plant-soil systems are currently not well understood, and require further study.

4. Conclusions

The following conclusions were drawn from this study:

1. The mixing of calcareous co-amendments with fresh and composted manure had an immediate effect on extractable ortho-P concentrations that persisted during a 6 week incubation. The decline in extractable ortho-P in co-amended fresh and composted manure was related to the effect of calcareous materials on pH.
2. Calcareous co-amendments increased N losses from fresh manure, but not from composted manure.

3. Untreated fresh manure and fresh manure mixed with co-amendments had an inhibitory effect on barley grain production and N and P uptake, compared to inorganically fertilized barley. There was no difference in barley grain production and nutrient uptake in pots that received composted manure mixed with co-amendments and inorganic fertilizer.
4. The CaO and $Ca(OH)_2$ co-amendments transformed ortho-P from fresh manure into forms that were not available to plants or extractable with a strong acidic extract after barley harvest.
5. Further research will be required to determine whether results from laboratory and greenhouse studies can be extrapolated to the scale of cattle feedlots and agricultural crop production.

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