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To the Graduate Council:

I am submitting herewith a dissertation written by James David. O'Dell entitled "Phosphorus speciation, mineralization, and uptake by corn in a soil amended with liquid swine manure." I have examined the final electronic copy of this dissertation for form and content and recommend that it be accepted in partial fulfillment of the requirements for the degree of Doctor of Philosophy, with a major in Plant, Soil and Environmental Sciences.

Michael E. Essington, Major Professor

We have read this dissertation and recommend its acceptance:

Donald D. Howard, John T. Ammons, Donald D. Tyler, Milton H. Lietzke

Accepted for the Council:

Carolyn R. Hodges

Vice Provost and Dean of the Graduate School

(Original signatures are on file with official student records.)

To the Graduate Council:

I am submitting herewith a dissertation written by James David O'Dell entitled "Phosphorus Speciation, Mineralization, and Uptake by Corn in a Soil Amended with Liquid Swine Manure." I have examined the final copy of this dissertation for form and content and recommend that it be accepted in partial fulfillment of the requirements for the degree of Doctor of Philosophy, with a major in Plant and Soil Science.

Michael E. Essington, Major Professor

We have read this dissertation
and recommend its acceptance

John T. Ammons
Donald D. Howard
Donald D. Tyler
Milton H. Lietzke

Accepted for the Council:

CWMinkel
Associate Vice Chancellor
and Dean of the Graduate School

PHOSPHORUS SPECIATION, MINERALIZATION, AND UPTAKE BY CORN IN A SOIL AMENDED WITH LIQUID SWINE MANURE

A Dissertation
Presented for the
Doctor of Philosophy
Degree
The University of Tennessee, Knoxville

James David O'Dell
May, 1995

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ABSTRACT

The soil application of liquid swine (*Sus scrofu domesticus*) manure as a source of P for crop production provides a means for the utilization of waste material. Manure rates have usually been based upon manure N concentrations, but this has sometimes resulted in excess P loadings due to the high N requirement of some crops, as well as the lower availability of N in manures relative to that of P. Applying higher than recommended P rates may result in greater runoff losses, which could lead to eutrophication of surface waters. As a result, future regulations may require that manure applications be based on P rather than N content, which would increase the amount of land needed for application.

A two-year field study was conducted to examine the variability in nutrient content of soil-applied liquid swine manure, to determine the effects of liquid swine manure application on the distribution of soil P fractions, and to assess the availability of manure P to corn (*Zea mays* L.). Liquid swine manure was surface-applied to plots in an attempt to provide 15, 30, and 60 kg P ha^{-1}. Control plots received 60 kg P ha^{-1} as triple superphosphate and 170 kg N ha^{-1} as ammonium nitrate. Manure plots also received 170 kg N ha^{-1} by assuming 40 percent availability of manure N and using ammonium nitrate to supplement the deficit. Plots were planted with 'DeKalb 689' and 'Pioneer 3343' varieties of corn. Prior to and after manure application and when corn was at four leaf, ten leaf, and silking growth stages, depth incremented soil samples were collected for P analysis. Plant samples were also collected at each growth stage.

Soil samples were analyzed to determine total and organic P, and a sequential dissolution procedure was used to fractionate the inorganic P into operationally defined aluminum (Al-P), calcium (Ca-P), iron (Fe-P), and reductant-soluble (Red-P) phosphate chemical pools. Available P was estimated using Olsen and Mehlich 3 extractants. Plant samples were analyzed for P content, and yields were calculated from grain harvested from the plot center rows.

Nutrient concentrations in liquid swine manure were highly variable, and resulted in lower than expected P application rates. In 1992, manure P rates were 17, 32, and 49 kg ha^{-1}, while 1993 rates were 5, 11, and 22 kg ha^{-1}. Effects of P rate on soil P concentrations were observed only in samples taken from the surface 10 cm. Of the inorganic P fractions measured, Al-P was most responsive to P application. Mehlich 3 and Olsen extractable P were also consistently affected by manure and fertilizer application. Manure P was generally less effective than triple superphosphate in maintaining or increasing soil P levels.

Corn yields were not affected by manure or TSP addition in either year. Ear leaf P content increased with P rated in 1992 but was unaffected in 1993. Correlation of P uptake with soil P fractions suggested that Al-P and Mehlich 3 P were the most accurate predictors of P availability, but correlation coefficients were low and not consistent between years. In order to assess P fertility status in this manure-amended soil, methodologies are needed which are more sensitive to changes in the pool of available P.

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CHAPTER 1

INTRODUCTION

Tennessee ranks 18th in the nation in both corn (*Zea mays* L.) and swine (*Sus scrofu domesticus*) production, with Obion and Weakley counties leading the state in production of the two commodities (Tenn. Dep. Agric., 1994). In 1993, 470,000 hogs and pigs were produced and more than 220,000 ha of corn were harvested for grain throughout the state. Swine manure is excreted at the rate of 32 kg per kg live weight per year and contains approximately 1.3 g P kg^{-1} (Ensminger and Olentine, 1978). Swine manure is usually a liquid consisting of fecal matter, urine, and fresh water used to flush the animal houses. This liquid manure is often stored in anerobic lagoons, although aeration systems have been used to reduce odors and provide some control over the proportions of organic, ammonium, and nitrate N while possibly reducing pathogen survival (Baines, 1976). The waste is commonly disposed of by pumping onto land without regard to the chemical content or fertilizer value of the manure. Since swine farms are in close proximity to much of Tennessee's corn production, land application for corn production may be a practical alternative to disposal.

The application of animal manure as a source of P for crop production provides a means for the utilization of this waste material. In general, manure application rates are base on N content. However, this may result in excess P fertilization due to reduced N concentration from storage in the lagoon, limited manure N availability (approximately 50% during the growing season) and the high P content of manure.

Since most crops are fertilized with higher N than P rates, manure application to apply the recommended N rate would supply P at levels much greater than those normally required for crop production. Applying higher than recommended P rates may result in more P loss in runoff, which may lead to eutrophication of streams. Such practices may lead to regulations stating that future manure applications be based on manure P content, resulting in more land needed for application.

Information regarding the plant availability of swine manure P is limited since most manure application rates, and thus manure fertility research, have traditionally been based on N content. In order to achieve efficient utilization of manure P, knowledge of P mineralization rates and solid-phase speciation in manure-amended soils, as well as their relationship to plant P uptake, is needed. Intensive characterization of soil P speciation and the determination of P forms available for plant uptake throughout the growing season would contribute to a better understanding of P fertility in manure-amended soils. Although plants do not directly utilize organic P forms, the organic P content of a soil may provide information regarding long-term P fertility status as mineralization to inorganic forms occurs. Since inorganic P compounds have relatively low solubilities, a number of P solid phases may be present in a soil. In older soils with well-developed profiles, P may become coated with Fe oxides and made relatively unavailable for plant uptake. When the soil solution is acidic, P is precipitated with Al and Fe, while phosphates of Ca form under neutral to alkaline conditions. It is these solid phases which control short-term P fertility.

One problem in manure research is the difficulty of providing uniform manure P application rates. Collecting a representative sample from a lagoon or any other storage facility is difficult. A lagoon has both solid and liquid phases, resulting in spatial variablity within the lagoon as the solids settle. Temporal variability in manure P content also occurs as a result of several factors. These include the number of animals and their diet, the amount of dilution by rainfall collected in the lagoon, and the solids content of the manure. The solid phase of the manure contains a large percentage of the total P, so variability in solids content from poor lagoon agitation can make uniform P loadings difficult to obtain.

A second requirement for efficient manure utilization is an understanding of the fate of soil-applied manure P. Monitoring both the organic and inorganic P fractions in the soil after manure application and throughout the growing season improves this understanding. The amounts of Al-P, Ca-P, and Fe-P present in the soil will affect the extractability of soil P by extractants that are commonly used to assess P availability. It is therefore advantageous to examine the relationship of the inorganic P fractions measured by sequential dissolution to the P which is measured by soil test extractants used to determine P fertilization rates.

Although yield measurements are the ultimate method of evaluating fertilizer P rates, research indicates that the yield response to fertilizer P on similar soil was small, even when extractable P levels were low (Parks et al., 1991). Examining the plant P uptake based on applied P rates may be a more sensitive measure of P availability.

More efficient P fertilization rates may be derived by correlating plant P uptake, rather than yield, with extractable soil P.

Efficient manure utilization for crop production requires accurate measurement of manure nutrient concentrations, knowledge of manure nutrient availability, and soil tests which reflect the nutrient availability in manure-amended soils. The objectives of this study were to examine the nutrient variability in land-applied manure, quantify P fractions with time and depth in a manure-amended soil, and to correlate these fractions with P uptake by corn. This information will be used to assess P availability from manure as compared to inorganic P from commercial fertilizer.

CHAPTER 2

SURFACE APPLICATION OF LIQUID SWINE MANURE: CHEMICAL VARIABILITY AND APPLICATOR DESIGN

Introduction

The application of swine manure to soils used for crop production provides a means of utilizing waste while potentially reducing inputs of commercial fertilizers. However, efficient utilization of manures requires a means of providing controlled, uniform application to land, as well as a knowledge of the chemical composition of the manure at the time of application.

Estimating the nutrient content of liquid manures is complicated by several factors. The collection of representative samples is difficult due to the lagoon having both a liquid and a solid phase. The relative proportions of these phases may vary greatly with time and degree of agitation. Since rapid analysis of manure samples is not always possible, the nutrient content of the applied manure may be very different from the manure which was sampled to determine application rates.

The temporal variability in liquid swine manure nutrient concentrations is the result of several factors. These include the number of animals and their diet, the amount of dilution by rainfall collected in the lagoon, volatilization of N, and the solids content of the manure. The solid phase of the manure may contain a large percentage of the total nutrients, so variability in solids content can make uniform nutrient loadings difficult to obtain. Thorough agitation of the lagoon prior to and during collection, as well as in the spreader tank, is needed in order to obtain constant solids concentration of the manure which is being applied.

The solids content of manure is a problem not only in the sampling and analysis, but also during application. In order to prevent the solids from settling in the manure spreader and not reaching the field, an agitation system in the spreader tank is needed which will keep the solids suspended as the liquid is pumped from the tank. However, the application system must be designed so that these solids do not obstruct the flow of manure from the tank to the nozzles. This requires that all plumbing be of sufficient size to handle any solids that may be collected from the lagoon.

Although several authors have used surface application of liquid manure (Warman, 1986; Culley et al., 1981), little information has been provided regarding the specific method of application or the uniformity and accuracy of nutrient loadings applied to plots. Descriptions of manure application range from "sprinkled" (Kuo and Baker, 1982) to "uniformly and accurately" applied using a pump and hose (Sutton et al., 1986). Patni and Culley (1989) used commercial tankers to surface apply liquid manure, and reported means and standard deviations for several nutrients in 40 manure samples collected during a three year period. However, investigators generally only report mean nutrient loading values, without accounting for variations in the chemical properties of the different manure tankloads.

The objectives of this study were to examine the concentrations of N, P, and K in liquid swine manure loads obtained for soil amendment and describe a system of providing uniform surface application of liquid swine manure to plots.

Materials and Methods

The manure spreader is constructed from a 2300-L stainless steel milk tank (DeLaval Bulk Cooler Model DRB 600) mounted on a trailer. A gasoline powered generator supplies electricity to operate a submersible pump which is mounted near the bottom center of the tank. This pump supplies flow to a circulation system in the bottom of the tank (Figure 2.1) as well as to the flow meter and nozzles. The circulation tube consists of 3.8 cm i.d. PVC pipe with several 1.3 cm diameter perforations. Flow to both the circulation tube and nozzles can be turned on or off using the two 1/4-turn ball valves at the pump outlet. Accurate flow measurements were obtained using a turbine flow meter and analyzer (Halliburton Services, Duncan, OK). Four F210 SS flood nozzles were positioned 55 cm above the soil surface at spacings to provide uniform flow across a 3.1 m plot (Figure 2.2). Accuracy of the flow meter was verified by measuring water volume delivered through the nozzles with time.

Approximately two weeks prior to manure application in 1992 and four weeks prior to application in 1993, an anaerobic lagoon was agitated for 24 h with a tractor-powered propellor-type mixer. Following agitation, four grab samples were collected from different locations in the lagoon and subjected to chemical analysis. Total N was determined conductimetrically (Carlson, 1978) after digestion with H_2SO_4/H_2O_2. Phosphorous and potassium were determined by inductively coupled plasma atomic emission spectroscopy after ashing overnight at 773 K and dissolving the residue in 2

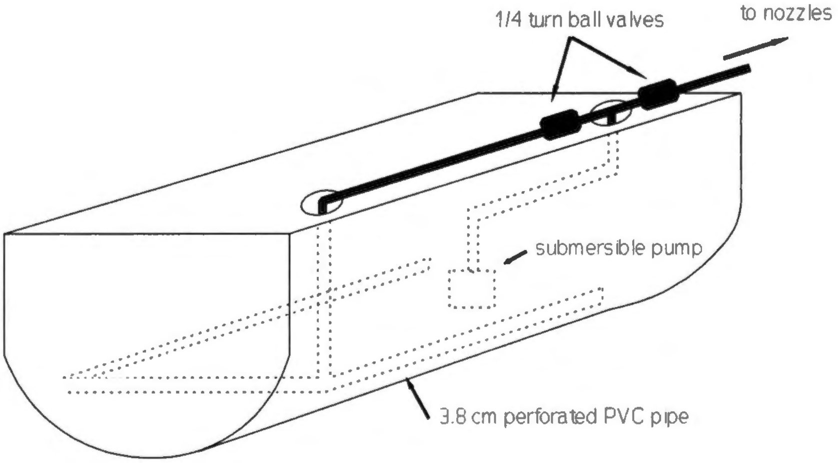

Figure 2.1. Diagram of manure tank and circulation system.

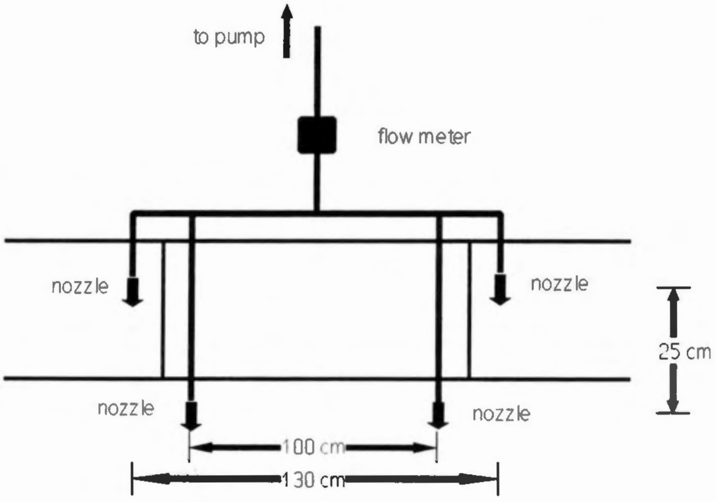

Figure 2.2. Top view of spray boom showing nozzle spacings.

N HCl. The P content of the grab samples was used to calculate manure application rates of 15, 30, and 60 kg P ha^{-1} for corn production.

Lagoon agitation was initiated 24 h prior to loading. Manure from the lagoon was transferred to a 7500-L tank for transport to the plots. Manure from the larger tank was transferred to the applicator tank by gravity flow, with each load from the larger tank supplying manure for two applicator tankloads. In general, the manure residence time in the larger tank was approximately 15 min, limiting the settling of solids. Manure application required several tankloads, with higher application rates achieved by increasing the number of passes over the experimental plots made by the applicator. Manure was applied early in the spring before planting and disked into the soil. Each tankload of manure was sampled and analyzed for total N, P, and K to determine actual application rates. The concentrations of N, P, and K in solution were also determined on manure samples which had been filtered through a 0.45 μm membrane. Solids content of each manure sample was determined by weighing the residue remaining after oven drying at 348 K.

Results and Discussion

Results of initial manure analyses for 1992 and 1993 are shown in Table 2.1. Although N concentration was about the same each year, P and K concentrations were approximately one-half as much in 1993 as in 1992. Variability among concentrations within years was also lower in 1993.

Table 2.1. Nutrient concentrations in 1992 and 1993 swine manure samples collected from the lagoon prior to soil application.

Year	Nutrient	$\bar{x}$	s
		mg L^{-1}	
1992	N	915	140
	P	775	391
	K	746	49.6
1993	N	920	47.6
	P	346	27.0
	K	437	18.8

In 1992, solution N content of manure ranged from about 600 mg L^{-1} during the first half of the spreader loads to a low of 400 mg L^{-1} during the second half of the application process (Figure 2.3). The total N content was much more variable and closely followed the concentration of solids in the manure. Solution P concentrations were nearly constant at about 50 mg L^{-1}, with highly variable total P concentrations which also closely followed the solids content (Figure 2.4). Solution K concentrations were relatively constant throughout the application process (Figure 2.5), and the total K in manure appeared to reside in the solution phase. This resulted in lower total K than solution K due to the solids occupying volume in the sample which was measured for totals. The decrease in actual solution volume made the apparent total concentration appear lower.

Although total N in 1993 samples varied with the solids content of the manure, solution N was relatively constant at about 600 mg L^{-1} as was observed in 1992 (Figure 2.6). Solution P concentrations were also a constant 50 mg L^{-1} (Figure 2.7), with total P again closely following the solids concentration in the manure. As in the previous year, 1993 solution K concentrations were relatively constant with total K only slightly following the solids concentration (Figure 2.8).

Large variations in manure nutrient content were observed by Patni and Culley (1989), who found relative standard deviations (RSD) of 22% for N, 26% for P, and 15% for K in liquid dairy manure. Similarly, Culley et al. (1981) observed RSD values of 11% for N, 17% for P, and 33% for K in liquid manure samples. During the 1992 application, RSD values of 27% for N, 43% for P, and 17% for K were

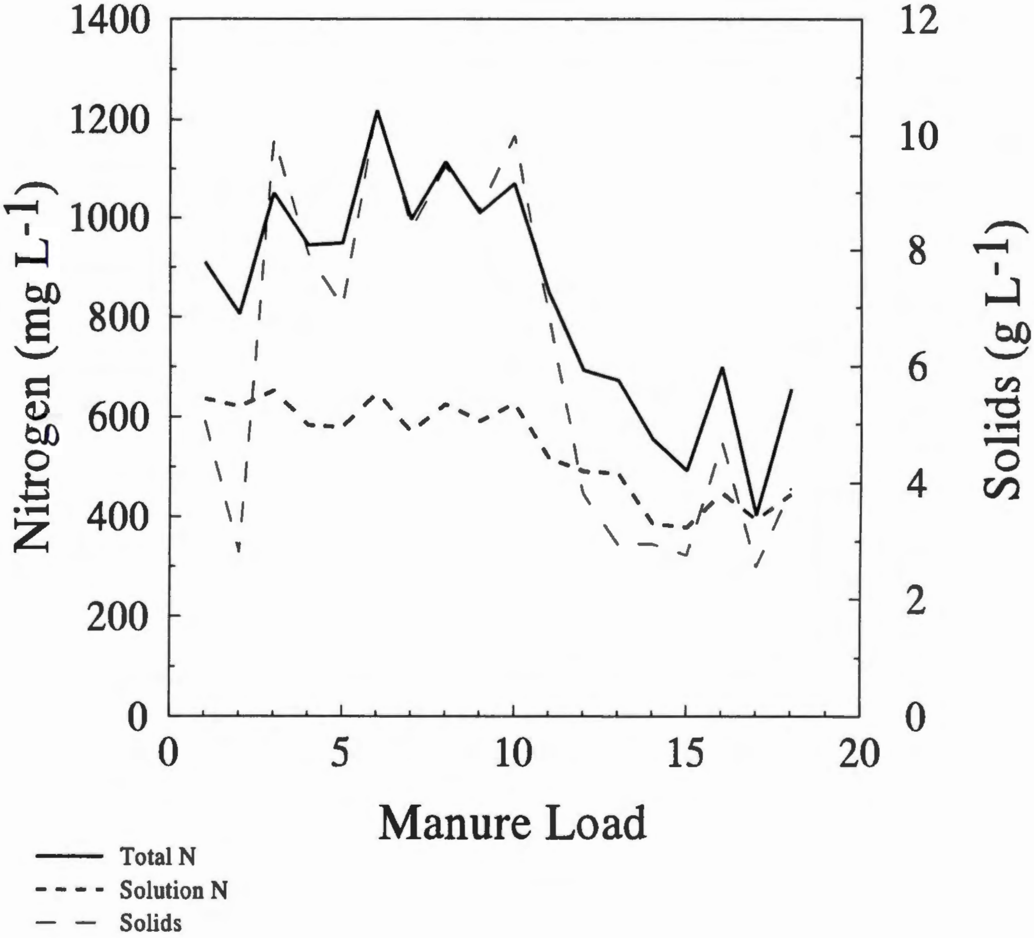

Figure 2.3. Total and solution N content and solids content of liquid swine manure applied to corn plots in 1992.

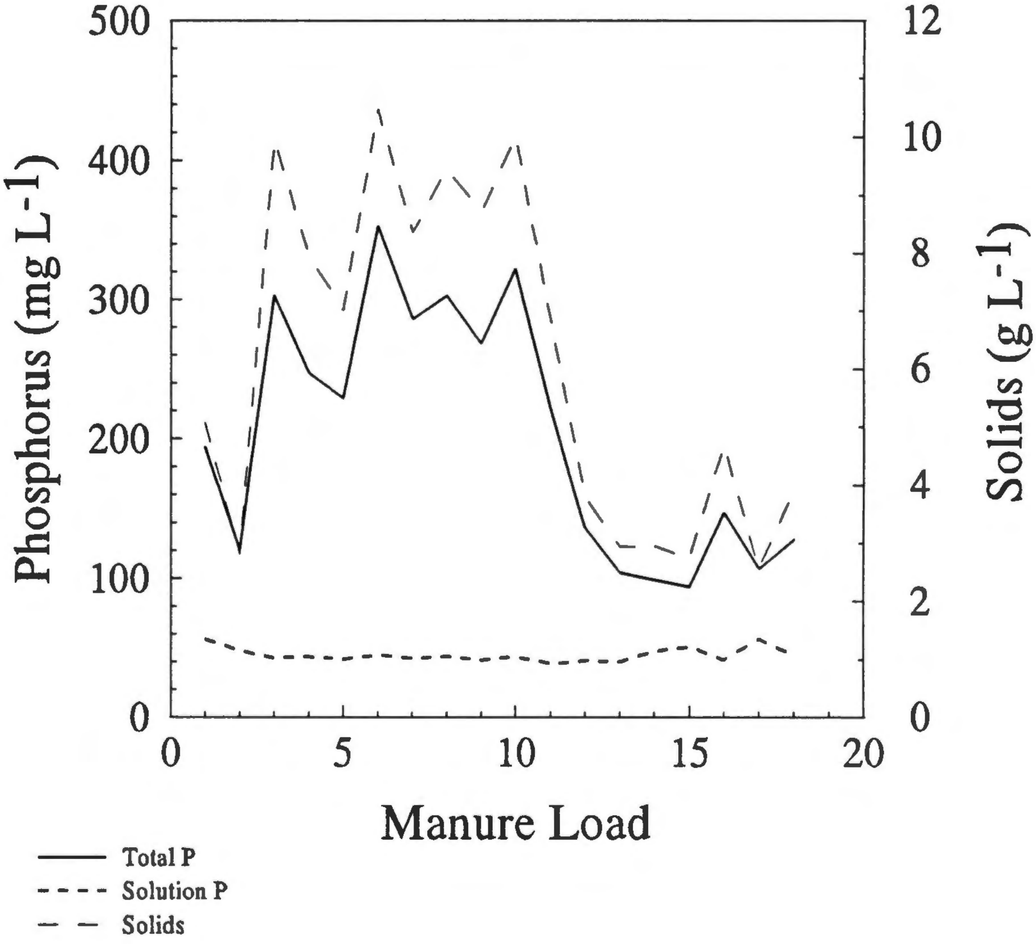

Figure 2.4. Total and solution P content and solids content of liquid swine manure applied to corn plots in 1992.

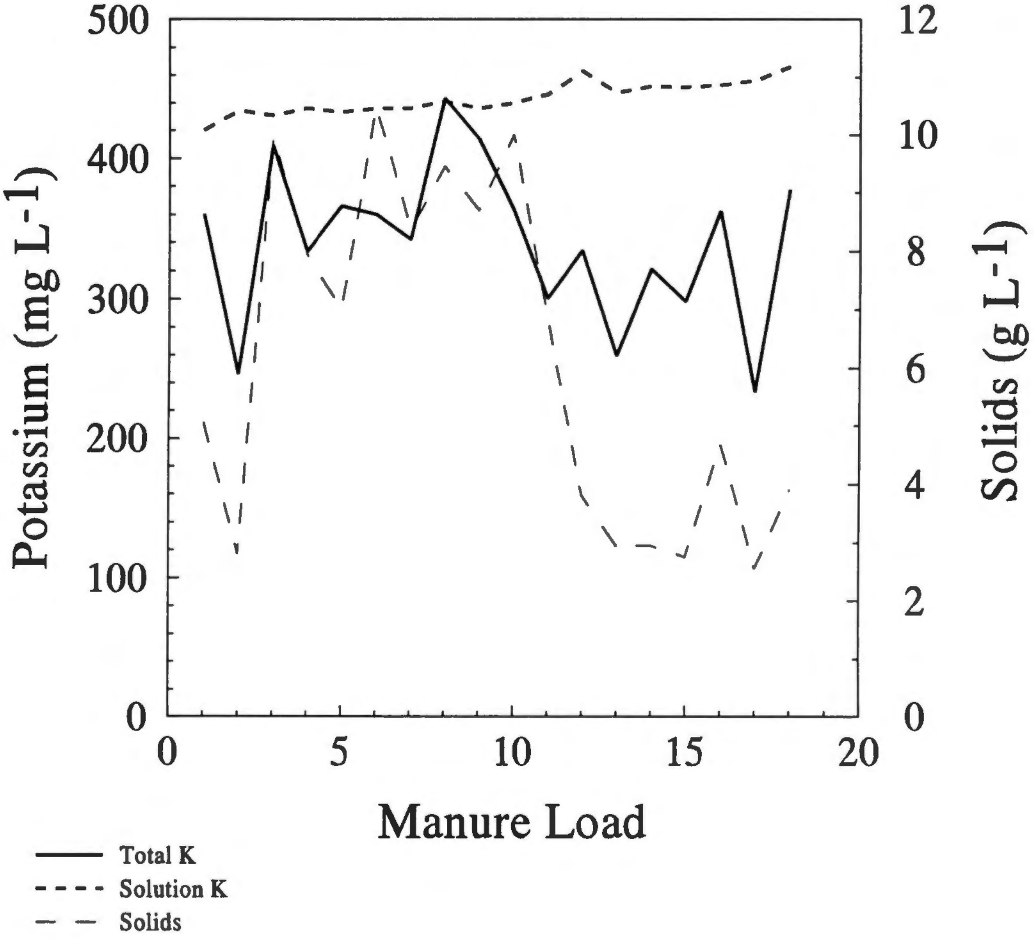

Figure 2.5. Total and solution K content and solids content of liquid swine manure applied to corn plots in 1992.

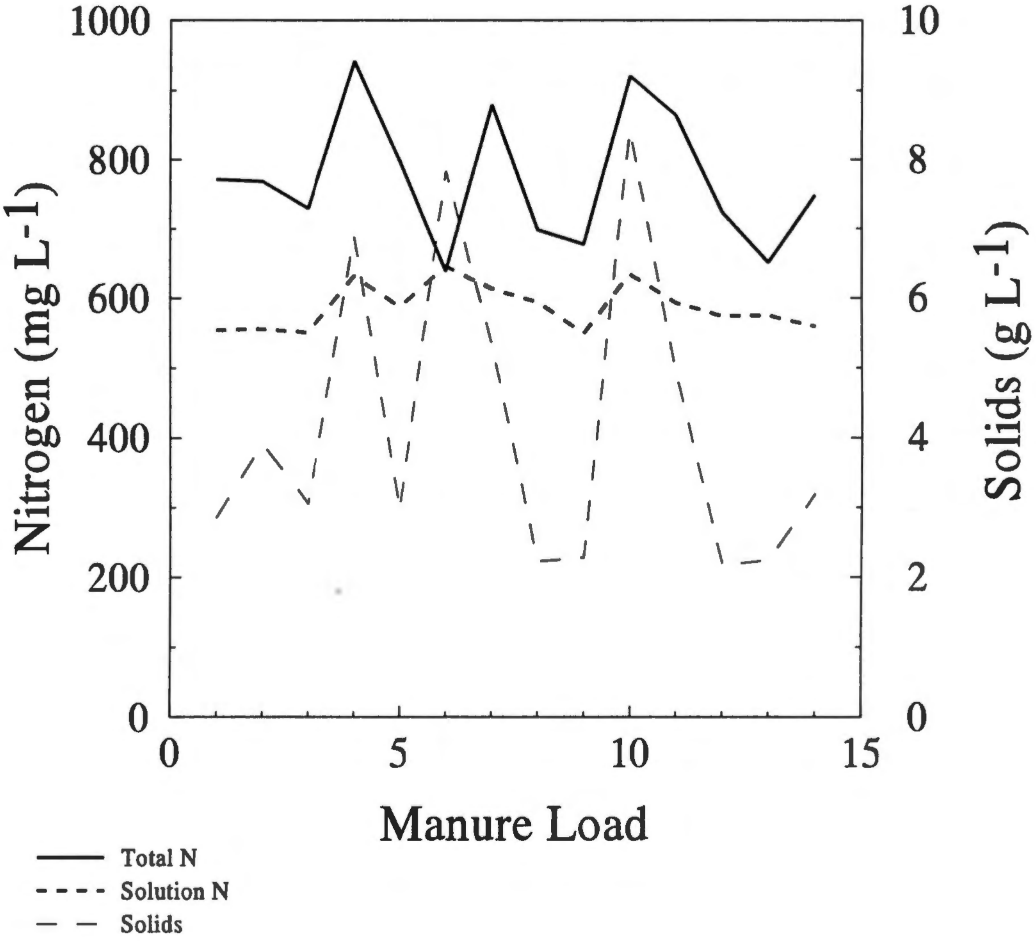

Figure 2.6. Total and solution N content and solids content of liquid swine manure applied to corn plots in 1993.

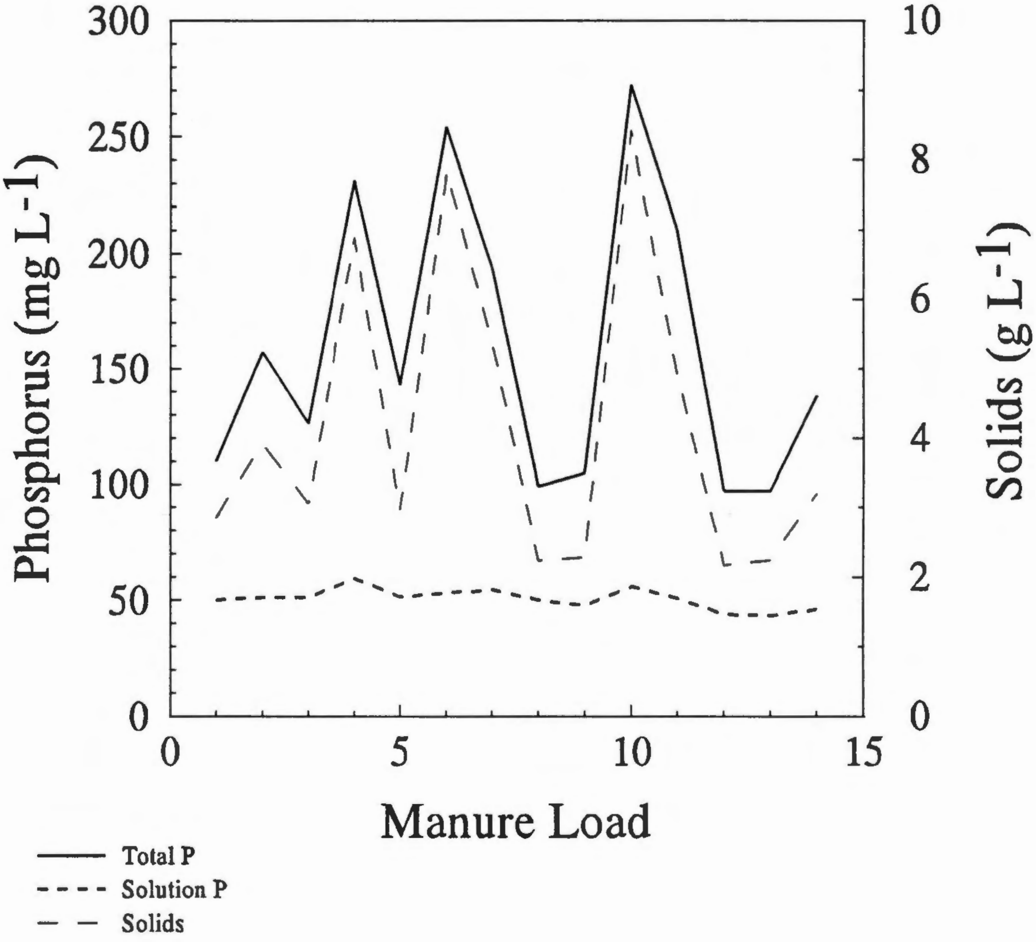

Figure 2.7. Total and solution P content and solids content of liquid swine manure applied to corn plots in 1993.

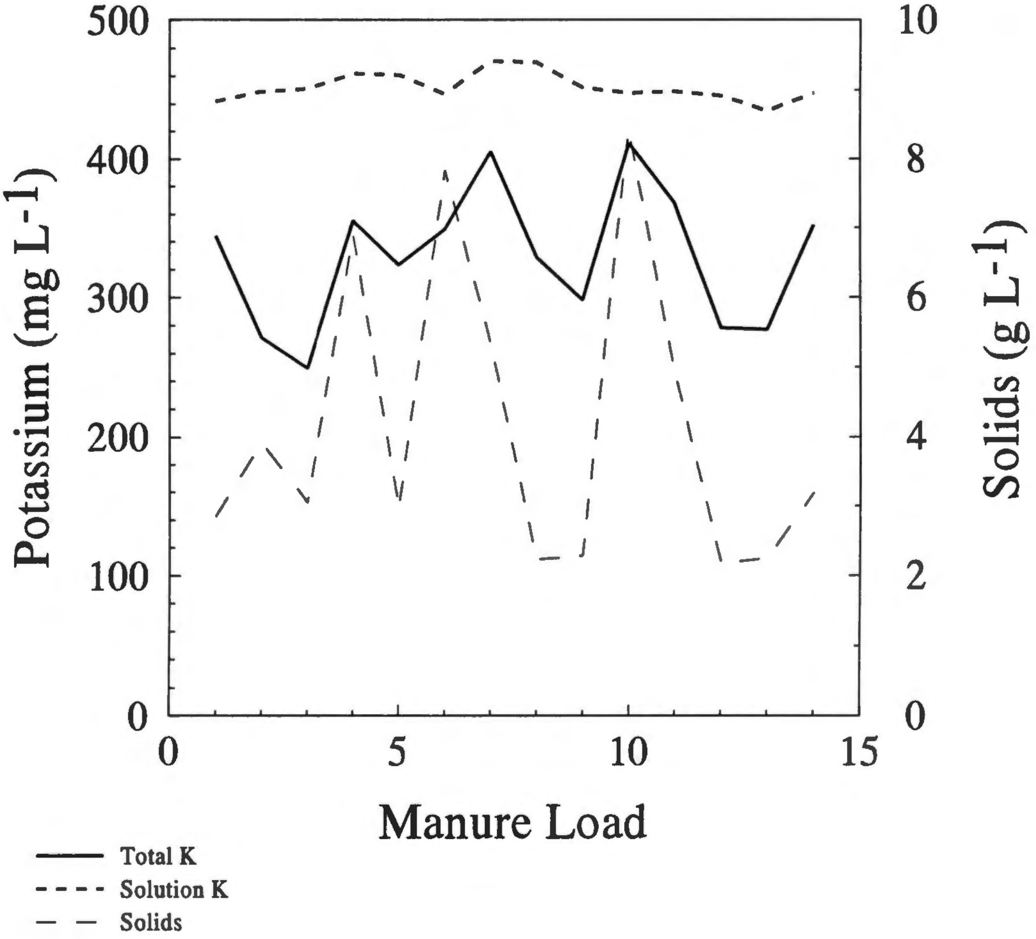

Figure 2.8. Total and solution K content and solids content of liquid swine manure applied to corn plots in 1993.

observed in the liquid swine manure tankloads. In 1993, RSD values of 12% for N, 38% for P, and 15% for K were observed.

A major difficulty associated with the utilization of liquid manures is the determination of application rates. Manure was applied to corn plots in an attempt to provide P rates of 15 (low), 30 (med), and 60 (high) kg ha^{-1}. In 1992 the mean P rates for the low, medium, and high treatments were 17, 32, and 49 kg ha^{-1}, respectively. The lower actual rate for the high rate was the result of decreasing manure P concentrations toward the end of the application process, when additional passes were made over the plots to apply the high treatment. Similar results were reported by Motavalli et al. (1989) with liquid dairy manure, in which highest rates were sometimes lower than expected. Wolt et al. (1984) obtained N loadings of 170-730 kg ha^{-1} from liquid swine manure application. In 1993, the combination of lower manure application rates and lower than anticipated manure P concentrations resulted in treatments of 5, 11, and 22 kg P ha^{-1} (Table 2.2). The treatments did follow a 1x-2x-4x pattern more closely than in 1992 due to the more regular increases and decreases in manure P in the 1993 tank loads, whereas in 1992 P concentrations were higher in the first loads and lower in the last tank loads. However, the actual 1993 P rates received by the corn plots did not meet target levels.

Summary

A spreader was designed which is capable of uniformly delivering controlled rates of liquid manure. However, the accurate determination of manure rates to apply

Table 2.2. Phosphorus rates applied to plots in 1992 and 1993.†

Year	Rep	Low 1	Low 2	Med 1	Med 2	High 1	High 2
		-----kg P ha^{-1}-----					
1992	1	14	14	30	30	46	53
	2	14	19	33	33	50	50
	3	16	16	31	31	45	44
	4	19	19	35	35	50	50
	5	20	20	33	33	51	51
		$\bar{x}=17$	$s=2.6$	$\bar{x}=32$	$s=1.8$	$\bar{x}=49$	$s=2.9$
1993	1	3.3	3.5	10	12	24	21
	2	3.5	5.4	12	11	24	23
	3	4.8	4.8	13	13	25	26
	4	5.2	4.9	11	11	24	20
	5	7.4	7.4	8.0	8.8	18	19
		$\bar{x}=5.0$	$s=1.5$	$\bar{x}=11$	$s=1.7$	$\bar{x}=22$	$s=2.7$

†Target rates were 15 kg P ha^{-1} (low), 30 kg P ha^{-1} (medium), and 60 kg P ha^{-1} (high).

specific nutrient loadings requires a representative sample of the manure. This is complicated by large amounts of N and P residing in the solid phase of the manure. Although attempts were made to ensure that manure was mixed both before and during loading and application, nutrient concentrations were highly variable. Thorough agitation of the lagoon prior to sampling as well as during the spreader loading is needed to help limit variablilty in solids content, thus providing more constant nutrient concentrations.

CHAPTER 3

PHOSPHORUS FRACTIONATION IN A LIQUID SWINE MANURE-AMENDED SOIL

Introduction

The soil application of liquid swine manure for crop production provides a means of utilizing a waste material. Since crops generally require more N than P, computing the rate of manure application based on manure N content and availability (approximately 50%) can supply P at levels far greater than those required for crop production. Excess P from manure amendments can be mobilized through surface runoff to potentially affect surface waters, enhancing eutrophication (Schindler, 1977; Webber, 1981). As a result, it may be mandated that future manure application rates be based on manure P content, resulting in more land needed for application.

Soil P Characterization

Methods of evaluating soil P and its potential availability are many and varied. Although a knowledge of the total P content of a soil provides little information on availability, it is beneficial as a general indicator of soil P supplying capacity. Similarly, organic P is not readily available but is slowly mineralized into available inorganic forms. Inorganic P compounds are sparingly soluble, resulting in the formation of a number of P solid phases. These include phosphates of Al, Fe, and Ca, as well as P coated with Fe oxides. Under a given set of conditions, the distribution of P among Al, Fe, and Ca phosphates is determined by the pH of the soil solution. Acidic conditions favor the formation of Fe and Al phosphates, while Ca phosphates form when the pH is neutral to basic. Soil test extractants are used to measure a portion of these inorganic P fractions, and correlation of extractable P with plant P uptake is used to evaluate the ability of an extractant to measure short-term soil fertility.

Total Soil P

Analysis of total soil P is usually facilitated by either fusion with Na_2CO_3 or digestion with HNO_3 and $HClO_4$ (Olsen and Sommers, 1982). These methods are time consuming and not well suited to large numbers of samples. Bowman (1988) proposed a sequential extraction method using H_2SO_4 and H_2O_2 to oxidize organic matter and HF to dissolve soil minerals. When the method was tested on 11 soils, the mean recovery was 98 percent of that determined by the fusion method while the $HClO_4$ digestion had an average 92 percent recovery. The Bowman (1988) extraction method was relatively rapid, requiring approximately 30 minutes for the entire extraction.

Soil Organic P

The organic P content of soil is highly variable and comprises from 20-80 % of the total P content in surface soils (Dalal, 1977). Since no direct methods exist for the measurement of organic P in soils, it must be estimated by comparing total P to inorganic P. Mehta et al. (1954) devised an extraction with hot HCl followed by hot

NaOH, after which the extracts were combined and analyzed for inorganic and total phosphate. Another common method has been the ignition method of Saunders and Williams (1955), in which P is extracted from soil that has been ignited to oxidize organic matter. This is then compared to P extracted from an unignited sample, and the difference is attributed to organic P. The ignition method is relatively rapid and simple, but tends to yield higher organic P values than the extraction method (Hance and Anderson, 1962; Enwezor and Moore, 1966) due to increased solubility of Al- and Fe-bound phosphates upon ignition (Williams and Walker, 1967).

Several extraction methods have been devised in an effort to save both time and labor in the estimation of organic P. MacLean (1965) proposed the use of 0.5 *M* $NaHCO_3$ at pH 10 and 363 K, and reported 98.8 percent recovery of organic P as compared to the extraction method of Mehta et al. (1954). Sonification of soil in NaOH has been used by Soltanpour et al. (1987) with a mean recovery of 89 percent of organic P by the Mehta et al. (1954) extraction. Steward and Oades (1972) also proposed a sonification method with NaOH which yielded organic P values slightly higher than those obtained by the Mehta et al. (1954) extraction.

An extraction method developed by Bowman (1989) using concentrated H_2SO_4 followed by 0.5 *M* NaOH allowed for relatively rapid extraction of organic P while yielding organic P concentrations similar to those obtained by the Mehta et al. (1954) extraction. The methods of Mehta et al. (1954), Saunders and Williams (1955), and Bowman (1989) were compared by Condron et al. (1990), who analyzed 23 soils containing a wide range of total P and organic P contents. The mean organic P content of the soils was 78 and 88 mg P kg^{-1} for the Mehta et al. (1954) and Bowman (1989) methods, respectively, while the mean organic P content as determined by the ignition method of Saunders and Williams (1955) was 208 mg P kg^{-1}.

Fractionation of Inorganic P

Although soil organic P content may be used to evaluate long-term fertility status, it is the concentration and distribution of inorganic P which affects short-term P availability. Selective sequential extractions are typically used to fractionate inorganic P, but the fractions analyzed are operationally defined and do not necessarily represent the targeted solid phases. However, employment of these techniques is useful for comparative purposes and as an aid in evaluating P supplying capacity.

Early experiments with fractionation of inorganic P in soil were performed by Dean (1938) using 0.2 *N* Na_2CO_3 or 0.25 *N* NaOH overnight at 368 K. Ability of the extractants to release P from Ca minerals was highly variable, especially when excess Ca was added. Both extractants were effective (88-99 percent) in dissolving P from Fe minerals. Sodium hydroxide released 100 percent of the P in $AlPO_4$, while Na_2CO_3 extraction yielded only 48 percent. The sequential extraction of soils with 0.25 *N* NaOH and 0.5 *N* H_2SO_4 in different orders (base-acid-acid, base-base-acid, and acid-base-acid) was also evaluated. The order of extraction had little effect upon the concentrations of P measured, with a mean recovery of 43.2 percent of the total P extracted from 34 soils.

Dickman and Bray (1941) suggested the use of neutral NH_4F to remove adsorbed phosphate from kaolinite, leading to its proposed use as a measure of P availability (Bray and Kurtz, 1945). Chang and Jackson (1957) deduced that this adsorbed P must be primarily Al phosphate and that after removal of soluble P and exchangeable Ca by 1 *N* NH_4Cl, neutral 0.5 *N* NH_4F could be used to extract Al-P if a correction for the solubility of Fe-P was applied. They thus proposed extraction of Fe-P with 0.1 *N* NaOH and that 10 percent of the P in this extract be subtracted from the value obtained for Al-P. After observing that apatite was completely dissolved in 0.5 *N* H_2SO_4, this was selected as the extractant for Ca-P provided it was used after removal of Al- and Fe-P due to their relatively high solubilities in acid solution. Based upon the observation of Bauwin and Tyner (1954), Chang and Jackson (1957) then proposed extraction of reductant soluble Fe-P with sodium citrate and dithionite followed by neutral NH_4F or 0.1 *N* NaOH to remove occluded Al-P or AlFe-P, respectively.

Rather than mathematically correct for solubility of Fe-P in neutral NH_4F, Fife (1959a) varied the pH of NH_4F and observed little solubility of Fe at $pH > 8$. Similar extractions were performed on rockbridgeite $[Fe_5(PO_4)_3(OH)_5]$, and minimum P solubility was observed at pH 8.5. It was then shown that Fe-P in soils exhibited behavior similar to that of rockbridgite, with Fe-P resistant to hydrolysis in alkaline NH_4F (Fife, 1959b).

Petersen and Corey (1966) modified the original Chang and Jackson (1957) procedure by using NH_4F at pH 8.2 to reduce the solubility of Fe-P. They also changed the extraction order, with the extraction of reductant soluble P preceding Ca-P in the selective dissolution procedure.

Recognizing the lack of selectivity of the dissolution procedures for specific solid phases, Williams et al. (1967) modified the procedure of Chang and Jackson (1957) by changing some of the extractants as well as nomenclature of the fractions. Rather than neutral NH_4F, they used NH_4F at pH 8.2 and called this fraction "NH_4F P." The authors added 1 *M* NaCl to the 0.1 *N* NaOH extractant which Chang and Jackson used for Fe-P and called this fraction "first NaOH P." Bicarbonate was added to the citrate-dithionite reagent for extracting reductant soluble P, with the alkaline buffer minimizing apatite solubility. Acid-extractable Ca-P was removed with 0.5 *N* HCl, with a second extraction using 1 *N* HCl performed if the first extract was high in P. Residual organic P was determined by ignition at 823 K followed by extraction with 1 *N* HCl, then residual inorganic P determined by fusion with Na_2CO_3.

In addition to the many modifications of the Chang and Jackson selective sequential P dissolution procedure, other fractionation schemes exist which are not based on the intentions of extracting specific P forms. The method of Hedley et al. (1982) uses increasingly harsher treatments to extract P fractions which may be increasingly less available for plant uptake. These treatments include anion exchange resin, $NaHCO_3$, NaOH, NaOH + sonication, HCl, and digestion with concentrated H_2SO_4. It has been recently pointed out (Barbanti et al., 1994) that all P fractionation procedures have limitations, especially that of readsorption of P during extraction and hydrolysis of organic P prior to analysis. It must be taken into consideration that the P fractions are operationally defined and do not represent actual P solid phases.

Available P

Although the inorganic P fractionation procedures yield useful information on soil P status, their use is not well suited for the routine analysis of large numbers of samples, such as would be encountered in a soil test laboratory. For this reason, extractants have been developed which facilitate rapid P determination for the purpose of predicting crop response to added P fertilizers.

Attempts have been made to quantify available P from soil solution P concentrations (Martin and Mikkelsen, 1960; Olsen and Wanatabe, 1963; Jones and Benson, 1975), but solution P concentrations are usually too low to provide adequate sensitivity for assessing P fertility. For this reason, plant available P is usually estimated by chemical extraction. The amount of P extracted is correlated with plant uptake of P from the soil, then the soil test is calibrated to determine the P levels at which response to added P is likely. Several extractants have been developed for use on a wide range of soils and crops, but most work by one or more of four mechanisms (Kamprath and Watson, 1980): Solvent action of acids, anion replacement, complexing of cations binding P, and hydrolysis of cations binding P. Dilute concentrations of strong acids are effective in dissolving Ca-P, while also solubilizing smaller quantities of Al- and Fe-P (Thomas and Peaslee, 1973). When a complexing ion such as fluoride is added to the acid solution, Al-P is released due to the formation of Al-F complexes

(Chang and Jackson, 1957). It has been postulated that these solutions are also somewhat effective at extracting Ca-P due to the precipitation of CaF_2 (Syers et al., 1972; Thomas and Peaslee, 1973). Dilute concentrations of weak acids (citric, lactic, acetic) form complexes with cations binding P, while also replacing adsorbed P (Dean and Rubins, 1947). Hydrolysis of Al and Fe can be accomplished with buffered alkaline solutions (Tyner and Davide, 1962).

The Mehlich 1 extractant (0.025 N H_2SO_4 + 0.05 N HCl) is currently used by the University of Tennessee Soil Testing Laboratory to estimate available P. However, it has been observed that loess-derived soils which test low in Mehlich 1 P may show little response to P fertilization (Hardin et al., 1989; Parks et al., 1991). Jittanoonta (1980) found Mehlich 1 P was correlated best with Ca-P in acid soils ($r=0.839$), while biological measures such as P uptake and yield were better correlated with Al-P. Based on experiments with 103 Mississippi soils, Jittanoonta (1980) concluded that a good soil test should extract primarily Al-P from both acid and alkaline soils. Although Mehlich 1 P is often correlated to Ca-P, high correlations to Al-P have been observed in soils of Brazil (Cajuste and Kussow, 1974) and North Carolina (Shelton and Coleman, 1968).

The Olsen soil test extractant (0.5 M $NaHCO_3$, pH 8.5) extracts Al-P by hydrolysis of Al in the alkaline solution and by replacement of adsorbed P by the bicarbonate ion. It is also effective in forcing the dissolution of Ca-P by the precipitation of calcite. The Olsen extractant is used by several soil testing laboratories and is often used to evaluate P availability in alkaline and manure-amended soils. Datta and Khera (1969) found Olsen P was significantly correlated with Al-P (r=0.65), Fe-P (r=0.531), and saloid- (NH_4Cl-extractable) P (r=0.406) in 22 neutral to slightly alkaline soils. Olsen P was correlated to Fe-P (r=0.65) and Al-P (r=0.527) in 20 soils studied by Debnath and Mandal(1982). However, Singh et al. (1968) found significant correlation of Olsen P with only Ca-P (r=0.86) in 11 soils having relatively low concentrations of Al-P. In a study of seven calcareous soils ranging from young to well developed, Deo and Ruhal (1970) found no correlation between Olsen P and inorganic P fractions.

The Mehlich 3 extractant (0.2 N HOAc + 0.25 N NH_4NO_3 + 0.015 N NH_4F + 0.13 N HNO_3 + 0.001 M EDTA) (Mehlich, 1984) has been shown to correlate well with Mehlich 1 and Olsen P, although Mehlich 3 extracts two to three times more P than the others (Wolf and Baker, 1985). The Mehlich 3 extractant may be well suited for soils in which several chemical forms control phosphate availability. The HNO_3 would extract Ca-P due to its solvent action, while acetate could replace phosphate adsorbed on the surfaces of Fe and Al oxides. The solution also extracts Al-P through complexation of Al by F, and the EDTA forms complexes with Fe. Jittanoonta (1977) found Mehlich 3 P was well correlated with Al-P (r=0.78) and Olsen P (r=0.76) in 136 Mississippi soils. In a study of 95 soils, Piha (1993) found millet fresh weight to be well correlated (r=0.785) with Mehlich 3 P.

Effect of Manure on Soil P

Manure application has often resulted in soil P accumulation, even when applied at rates consistent with those recommended for P fertilization. Since manure P tends to be more mobile than inorganic P fertilizer forms, increases in subsoil P have been observed when excessive manure P was applied. In a soil which initially had 20 mg kg^{-1} $NaHCO_3$-extractable P, Pratt and Laag (1981) found 79, 136, and 257 mg P kg^{-1} in the surface 30 cm of soil after applying 918, 1836, and 3672 kg P ha^{-1}, respectively, as solid dairy manure over a four-year period. Subsoil P increased, although not as dramatically, from 12 to 29, 7 to 17, and 4.7 to 8.3 mg kg^{-1} in the 30-60, 60-90, and 90-120 cm depth increments, respectively.

Enrichment of soil P may last for many years after manure application, as was shown by Meek et al. (1982). After applying 1366 kg P ha^{-1} over a four-year period, the concentration of $NaHCO_3$-extractable P in the top 30 cm was 98 mg kg^{-1}, compared to 9 mg kg^{-1} in the untreated control plots. Five years later, the soil contained 50 mg P kg^{-1} compared to 17 mg kg^{-1} in the control soil. Sutton et al. (1986), however, found that applying manure P at levels equivalent to recommended P fertilization rates (56 kg P ha^{-1}) resulted in Bray P_1 concentrations equal to those of the fertilizer P check plots after five years of application. Even when manure P was applied at three times the recommended rate, soil P rarely increased during the five years studied.

In studies with both fresh and composted dairy manure, Elias-Azar et al. (1980) found that $NaHCO_3$-extractable P varied as a function of soil type. After a 30-day incubation, soil treated with manure had extractable P at 46-100 percent of soil treated with KH_2PO_4 at 40 mg P kg^{-1}. Bernal et al. (1993) observed that manure application increased $NaHCO_3$-extractable P until the concentration was about 25 mg kg^{-1}, after which increasing the manure rate decreased available P.

In calcareous soils with a high P fixation capacity, inorganic P fertilization increased $NaHCO_3$-extractable P but did not increase P uptake by plants. Addition of sewage sludge to these soils increased $NaHCO_3$-extractable P while also increasing plant P concentrations (O'Connor et al., 1980; Sanchez, 1982). After five years of sewage sludge application, Chang et al. (1983) found Al- and Fe-P were >60% of the total P in two calcareous soils, while Ca-P was 50-60% of the total P in soils which received no sludge.

Efficient utilization of manure as a source of P for crop production requires a thorough understanding of P mineralization and speciation in manure-amended soils. Soil test extractants used to estimate available soil P may not accurately reflect the P status when manure is the only source of added P. Therefore, information is needed regarding the extractability of P from manure-amended soils. The objectives of this study were to quantitatively characterize soil P fractions with time and depth in a soil amended with liquid swine manure as the sole source of added P for corn production.

Materials and Methods

Plots were established in 1992 at the Ames Plantation, Grand Junction, Tennessee, on a Lexington silt loam (fine-silty, mixed, thermic Typic Paleudalf) which had been under undisturbed fescue (*Festuca arundinacea* Shreb.) for several years.

Plots were 3 x 15 m, with four P rates, two corn varieties, and five replications. After initial cultivation, soil samples were collected from the 0-10, 10-20, 20-30, 30-45, 45-60, 60-75, and 75-90 cm depths to determine pH (1:2 soil:water) and background P concentrations.

Approximately one month prior to manure application, an anerobic swine manure lagoon was agitated for 24 h with a tractor-powered propeller-type mixer. Following agitation, four grab samples of swine manure were collected from different locations in the lagoon and subjected to chemical analysis. Total N was determined conductimetrically (Carlson, 1973) after digestion with H_2SO_4/H_2O_2. Total P and K were determined by inductively coupled plasma atomic emission spectroscopy after ashing overnight at 773 K and dissolving the residue in 2 *M* HCl. Manure P concentration was used to calculate manure application rates to provide 15, 30, and 60 kg P ha^{-1} for corn production. Control plots received 60 kg P ha^{-1} as triple superphosphate and 170 kg N ha^{-1} as ammonium nitrate. Manure plots also received 170 kg N ha^{-1} by assuming 40% availability of manure N and using ammonium nitrate to supplement the deficit.

Manure was surface-applied to the corn plots from a stainless steel tank mounted on a trailer as described in Chapter 2. Several tankloads of manure were required for complete application to the plots, with multiple passes with the spreader used to achieve the higher manure rates. Each tankload was sampled and analyzed to determine the specific P loading for each plot. Although the goal was to apply manure to provide 15, 30, and 60 kg P ha^{-1}, 1992 rates were 17, 32, and 49 kg P ha^{-1}, while 1993 rates were 5, 11, and 22 kg P ha^{-1}.

After manure application, soil samples were collected from the plots at depths of 0-10, 10-20, and 20-30 cm. Approximately 10 cores were taken from each plot with a 2.5-cm diameter push probe. Plots were then planted with 'DeKalb 689' and 'Pioneer 3343' varieties of corn. Soil samples were collected in 10 cm increments to 30 cm and in 15 cm increments to 60 cm when corn was at four leaf, 10 leaf, and silking. Four 5-cm diameter cores were collected from each plot at each sampling using a tractor-mounted hydraulic sampler. In 1992, soil samples were collected from the 60-75 and 75-90 cm depths when corn was at 10 leaf and silking.

Soil Analysis

Soil samples were air dried and passed through a 2 mm sieve. A subsample was ground to 0.4 mm and total P determined by digestion with $H_2SO_4/H_2O_2/HF$ (Bowman, 1988). A 0.5 mL aliquot was diluted with 0.5 mL of 5 *M* NaOH and 4.5 mL deionized H_2O, followed by addition of 1 mL ammonium molybdate-potassium antimony tartarate solution (Wanatabe and Olsen, 1965). Phosphorous in solution was determined by absorbance at 880 μm. Organic P was determined by extraction with concentrated H_2SO_4 and 0.5 *M* NaOH (Bowman, 1989), after which the acid and base extracts were combined at a 1:2 ratio for analysis. Organic P was calculated as the difference between total P measured by inductively coupled plasma atomic emission spectrometry (ICP-AES) and inorganic P determined colorimetrically. Inorganic P was determined by combining 1 mL of acid extract, 2 mL of base extract, 5 mL of deionized H_2O, and 1 mL of ammonium molybdate-potassium antimony tartarate solution, and measuring the absorbance at 880 μm (Wanatabe and Olsen, 1965).

A modified Chang and Jackson procedure (Petersen and Corey, 1966) was used to fractionate the inorganic soil P into NH_4Cl-extractable (soluble P), NH_4F-extractable (Al-P), NaOH-extractable (Fe-P), and citrate-dithionite-extractable (Red-P). Soil was initially extracted with 1 *M* NH_4Cl to remove exchangeable Ca and easily soluble P. After separating the soil and extractant by centrifugation, Al-P was extracted with 0.5 *M* NH_4F, pH 8.2. The suspension was centrifuged and separated, after which the soil was washed twice with saturated NaCl. Fe-P was extracted with 0.1 *M* NaOH, and after two NaCl washes, reductant soluble P was extracted with 0.3 *M* sodium citrate and 1 g solid sodium dithionite at 353 K. After two washes with saturated NaCl solution, Ca-P was extracted with 0.25 *N* H_2SO_4. Phosphorous concentrations were determined colorimetrically except in the reductant extract, in which P was determined by ICP-AES. Phosphorus in the NH_4Cl fraction was below detection limits and not measured after the initial extractions. Phosphorus in the Al-P and Ca-P fractions was analyzed by adding 3 mL of chloromolybdic boric acid solution and 100 μL of chlorostannous reductant (Petersen and Corey, 1966) to a 3 mL aliquot of extract, with the absorbance measured at 660 μm. Solutions containing Fe-P were analyzed by adding 5 mL of deionized H_2O, 3 mL of chloromolybdic boric acid solution, and 100 uL of chlorostannous reductant to 1 mL of the extract, and measuring the absorbance at 660 μm.

Available P was determined by Mehlich 3 (0.2 *N* HOAc + 0.25 *N* NH_4NO_3 + 0.015 *N* NH_4F + 0.13 *N* HNO_3 + 0.001 *M* EDTA) and Olsen (0.5 *M* $NaHCO_3$, pH 8.5) extractions using 1 g soil and 20 mL of solution. Samples were shaken for 5 and 30 minutes for Mehlich 3 and Olsen P, respectively. Phosphorus in solution was analyzed by diluting 1 mL of extract with 9 mL of deionized H_2O, followed by addition of 1 mL of ammonium molybdate-potassium antimony tartarate solution (Wanatabe and Olsen, 1965). Absorbance was measured at 880 μm.

Statistical Analysis

Data were analyzed using the GLM procedure of the Statistical Analysis Systems (SAS), with mean separations conducted using the Duncan's multiple range test. Data were initially analyzed by individual depths and sampling times to determine the effect of P rate and source on soil P. Depth and sampling time were then added to the model, and when this interaction was not significant at the 0.05 level of probability, error terms were pooled to determine the effect of depth and sampling time on soil P.

Simple correlation analysis was performed using the CORR procedure of SAS to determine correlation coefficients (r) and to determine their significance. Correlation coefficients which were not significant at the 0.05 level of probability were not reported.

Results and Discussion

Background P concentrations and pH values in samples collected in 1992 prior to manure application are presented in Table 3.1. Total P was higher at depths greater than 20 cm, with lower total P concentrations in the top 20 cm probably due to removal by plant uptake. Organic P decreased with increasing depth as a result of inorganic P cycling by plant uptake and the subsequent decay of plant residues at the soil surface.

Of the P forms measured in the sequential extraction of inorganic P, reductant-soluble P (Red-P) was present in the highest concentrations. This would be expected in a well developed soil profile, in which Fe oxides form with P as an impurity. Once coated with Fe oxides, this P would be relatively unavailable for plant uptake, although small amounts may be solublized by the release of reductants from plant roots (Chaney et al., 1972; Brown and Jones, 1975). The second most prevalent inorganic P form was Fe-P, which increased with depth to 30 cm after which the concentration remained relatively constant. The solubility lines in Figure 3.1 suggest that in a soil with pH approximately 6.2, the potential for control of phosphate solubility is nearly equal among variscite, strengite, and hydroxyapaptie. At pH>7, Ca-P minerals are likely to form, while Al-P and Fe-P minerals would predominate at lower pH. Since this surface soil has a pH near 7, it is difficult to predict the mineralogical fate of added P. However, since the predominant form in background soil samples is Fe-P, one would predict added P to eventually be found in the Fe-P pool. It should be noted that several assumptions were made in contructing Figure 3.1. The activities of Al^{3+} and Fe^{3+} were assumed to be controlled by gibbsite and goethite, respectively. The activity of Ca^{2+}

Table 3.1. Soil pH and P distribution prior to manure treatments in 1992.

Depth	pH	Total	Organic	Al-P	Ca-P	Fe-P	Red-P	Mehlich 3	Olsen
cm		--mg kg^{-1}--							
0-10	6.9	410	68	7.00	16.8	55.8	81.0	6.09	4.78
10-20	6.8	440	45	12.8	21.5	80.5	109	8.26	6.52
20-30	6.7	470	21	18.8	28.0	93.8	123	11.7	9.14
30-45	6.2	480	27	19.5	21.2	96.2	126	13.5	8.70
45-60	5.7	450	21	22.0	19.0	90.0	134	14.8	13.0
60-75	5.5	460	17	21.5	19.8	95.5	92.6	15.2	10.4
75-90	5.6	450	15	23.5	19.0	96.5	151	17.4	14.8

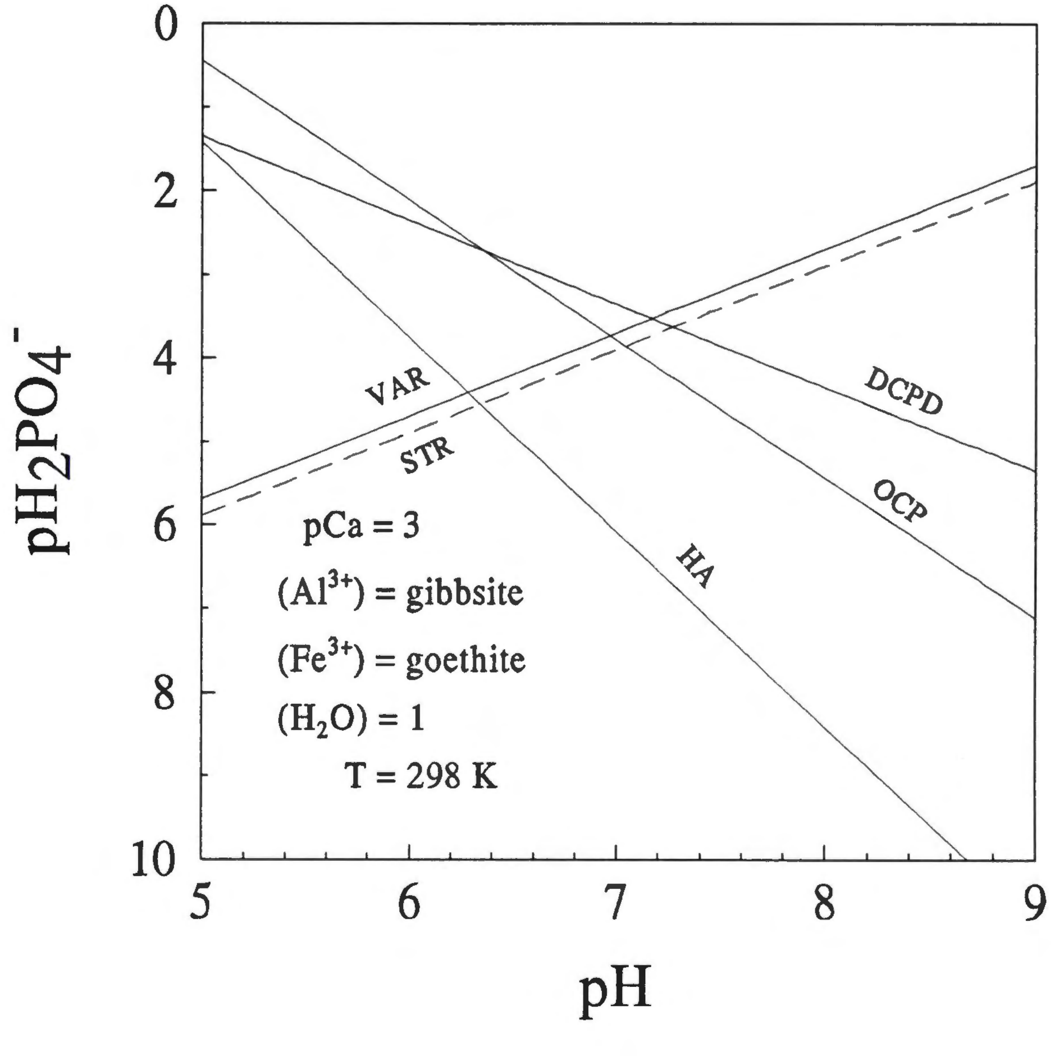

Figure 3.1. Solubility of variscite (VAR), strengite (STR), hydroxyapatite (HA), octacalcium phosphate (OCP), and dicalcium phosphate dihydrate (DCPD) as a function of pH.

was also fixed at 0.001, and increasing the Ca^{2+} activity would cause the solubility lines of the calcium minerals to shift downward. There is also considerable error associated with the strengite solubility line, as Lindsay and Moreno (1960) used an average of several values in determining the solubility product of strengite.

Total and Organic P

Phosphorus application did not affect total soil P in 1992 since P rates were low in comparison to soil P levels. Toward the end of the 1993 growing season, however, plots which received manure P had lower total P in the surface 10 cm than those which received TSP (Figure 3.2). As P removal by plant uptake increased throughout the growing season, manure P rates were not enough to sustain or elevate P concentration in the surface soil.

The concentration of total soil P increased with depth to 45 cm, below which a slight decrease was observed (Table 3.2). In 1992 when samples were taken to 90 cm, total P continued to decrease in the 75-90 cm samples. Total P was also affected by sampling time, with the highest concentrations occurring when corn was at silking. The lowest total P concentrations were observed when corn was at the ten-leaf stage, while intermediate levels were found after P application and when corn was at the four-leaf stage (Table 3.3). These variations in total P content were most likely due to sampling error cause by spatial variability in total P concentrations.

Manure application did not affect organic P in either year due to the small quantity of P applied (5-60 kg ha^{-1}) relative to the amounts of organic P present in the

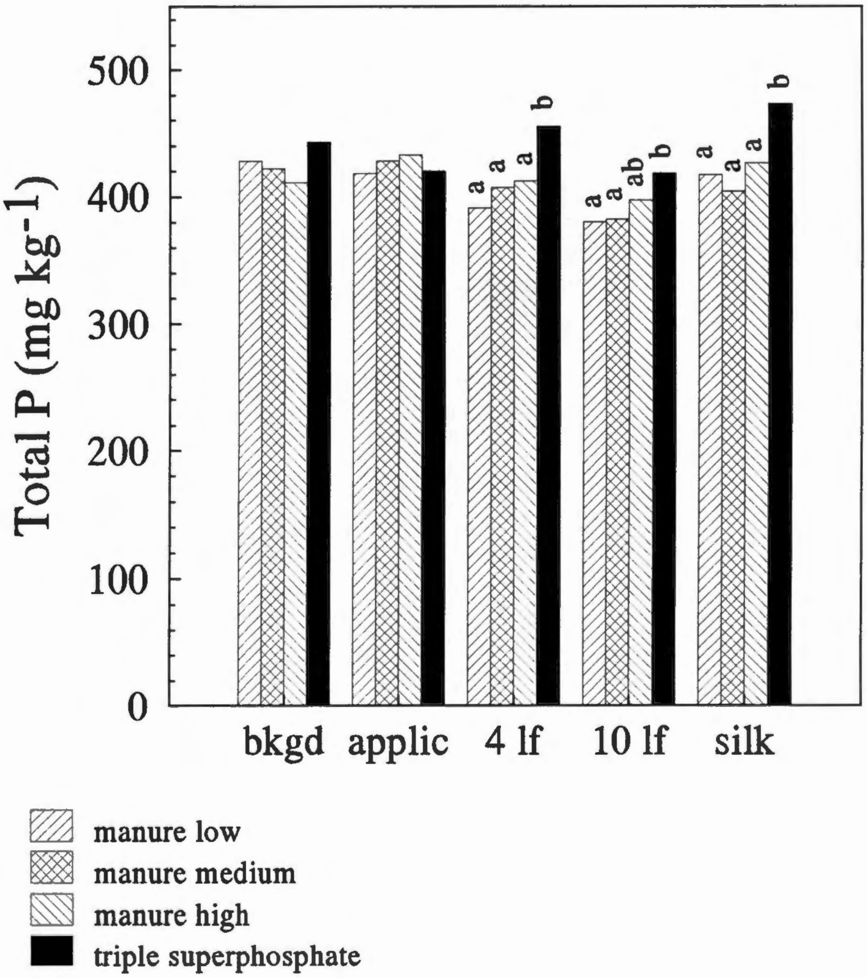

Figure 3.2. Effect of manure and triple superphosphate on total P in the surface 10 cm in 1993. Means within a sampling time followed by the same letter are not significantly different at the 0.05 probability level.

Table 3.2. Effect of depth on total P in 1992 and 1993.

Depth	1992	1993
cm	---------------mg kg^{-1}---------------	
0-10	429 a†	419 a
10-20	433 a	410 a
20-30	482 b	459 bc
30-45	492 b	466 b
45-60	464 c	448 c
60-75	450 c	ND‡
75-90	421 a	ND

† Means in the same column followed by the same letter are not significantly different at the 0.05 probability level.
‡ Not determined.

Table 3.3. Effect of sampling time on total P in 1992 and 1993.

Sampling time	1992	1993
	---------------mg kg^{-1}---------------	
background	NR†	456 a‡
application	458 a	436 b
4 leaf	450 a	430 b
10 leaf	429 b	415 c
silking	475 c	455 a

† Not reported; see Table 3.1.
‡ Means in the same column followed by the same letter are not significantly different at the 0.05 probability level.

surface soil (68 mg kg^{-1}). Sampling time and soil depth interacted to affect organic P concentration. In 1992, organic P decreased with depth at all sampling times (Table 3.4). Since the plots had been in permanent pasture prior to establishment, large amounts of organic P accumulated in the surface soil due to the decomposition of plant residues. In 1993, however, depth effects were observed only at application and when corn was at the 10-leaf stage. During this second year of the study, organic P became more constant with depth due to movement of organic P to lower depths after the soil was disturbed by cultivation. Although cultivated soils tend to have lower organic P concentrations than soils in permanent pasture (Condron et al., 1990; Hedley et al., 1982), addition of fertilizer has also been shown to increase organic P concentrations (Dormaar, 1972; Sharpley, 1985). Rolston et al. (1975) found that $NaHCO_3$-extractable P increased to a depth of 12 cm after surface application of organic P and wetting to 20 cm, while application of KH_2PO_4 increased extractable P only to a depth of 4 cm after wetting to 20 cm. The more even distribution of organic P with depth in 1993 may have been the result of organic P leaching to the lower soil depths, with organic P being replenished in the surface soil by decomposition of plant residues and immobilization of added P. Although organic P inputs were lower in 1993, background organic P levels were sufficiently high to facilitate movement of organic P to lower soil depths. Higher organic P values in 1993 may also have been the result of greater decomposition of plant material after cultivation. When large amounts of biomass are present in soil, extraction methods may not adequately measure organic P in the undecomposed plant material due to incomplete oxidation of organic matter.

Table 3.4. Effect of depth and sampling time on organic P in 1992 and 1993.

		Sampling time				
Year	Depth	Background	Application	4 leaf	10 leaf	silking
	cm	--------------------------------mg P kg^{-1}--------------------------------				
1992	10	NR†	73.5 aA‡	93.4 aB	88.5 aB	91.4 aB
	20	NR	60.8 bA	67.4 bA	80.4 aB	82.1 bB
	30	NR	33.3 cA	47.2 cB	28.5 bA	64.8 cB
	45	NR	ND§	39.8 cA	21.3 bcB	62.3 cC
	60	NR	ND	39.8 cA	15.3 cB	48.8 dA
	75	NR	ND	ND	20.3 bcA	49.0 dB
	90	NR	ND	ND	17.9 bcA	41.0 dB
1993	10	102	105 a	106	100 ab	99.4
	20	81.0	85.5 b	99.6	101 ab	92.2
	30	93.8	113 a	89.2	114 a	97.0
	45	104	ND	84.4	109 a	106
	60	93.2 A	ND	82.0 A	81.7 bA	115 B
	75	ND	ND	ND	ND	ND
	90	ND	ND	ND	ND	ND

† Not reported; see Table 3.1.

‡ For each year, means in the same column followed by the same lower case letter and in the same row followed by the same upper case letter, are not significantly different at the 0.05 probability level.

§ Not determined.

Organic P was affected by sampling time at all depths in 1992, but only at the 45-60 depth in 1993 (Table 3.4). As was observed by Dormaar (1972), organic P values were higher in the second year of the experiment. The trend was for organic P to decrease with time as corn reached the 10 leaf stage, then increase in soil samples taken when corn was at silking. Dormaar et al. (1972) found that organic P was highest in the spring and declined in the summer, followed by an increase in the fall as alfalfa became dormant. The earlier increase observed with corn may be due to the earlier time at which corn reaches maturity, whereas alfalfa is normally cut as bloom is initiated. Sharpley (1985) also reported large monthly fluctuations in organic P content, although no real patterns were observed.

Phosphorus by Selective Sequential Extraction

Manure and TSP application affected Al-P only in the surface 10 cm of soil. In 1992, Al-P increased with P rate after application and when corn was at silking. TSP application resulted in higher Al-P concentrations than manure application (Figure 3.3). Background soil samples collected in 1993 showed no difference in Al-P concentration among P treatments (Figure 3.4). Immediately after application, however, the high manure-P rate had higher Al-P than the other manure rates and the TSP treatment. Throughout the rest of the 1993 growing season, plots which received low and medium manure-P had relatively constant Al-P concentrations, while the high manure-P and TSP treatments resulted in relatively large Al-P concentrations. Plots

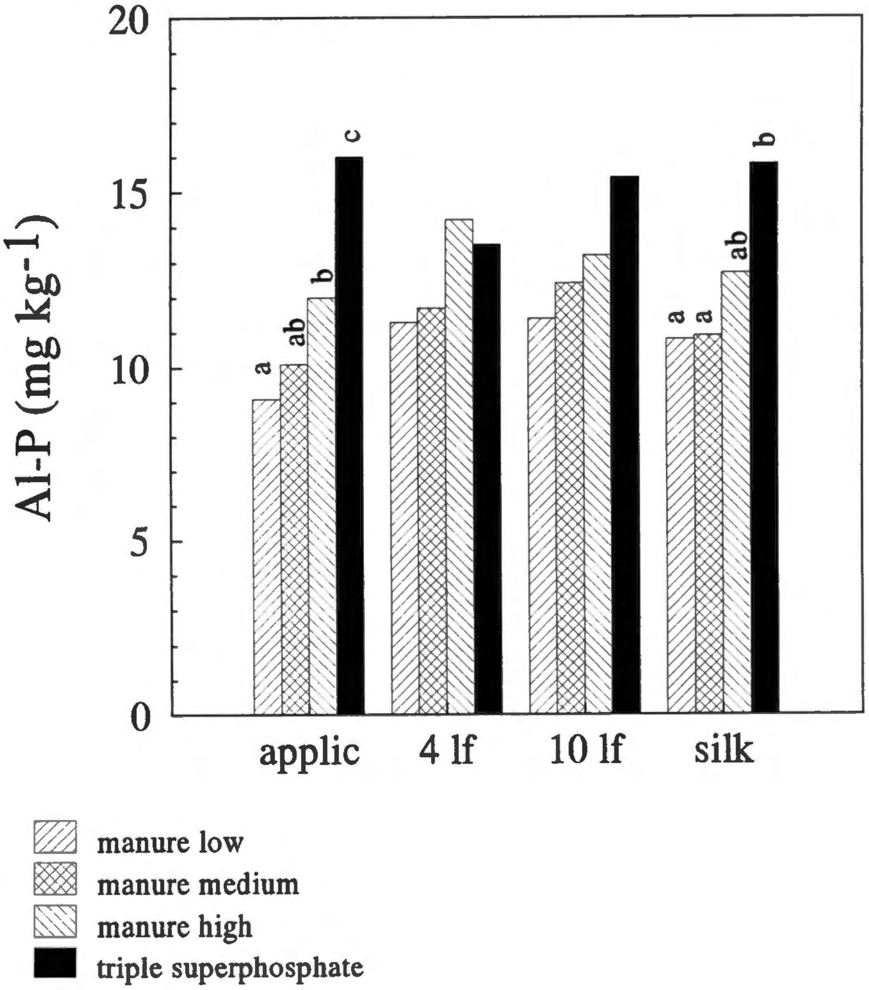

Figure 3.3. Effect of manure and triple superphosphate on Al-P in the surface 10 cm in 1992. Means within a sampling time followed by the same letter are not significantly different at the 0.05 probability level.

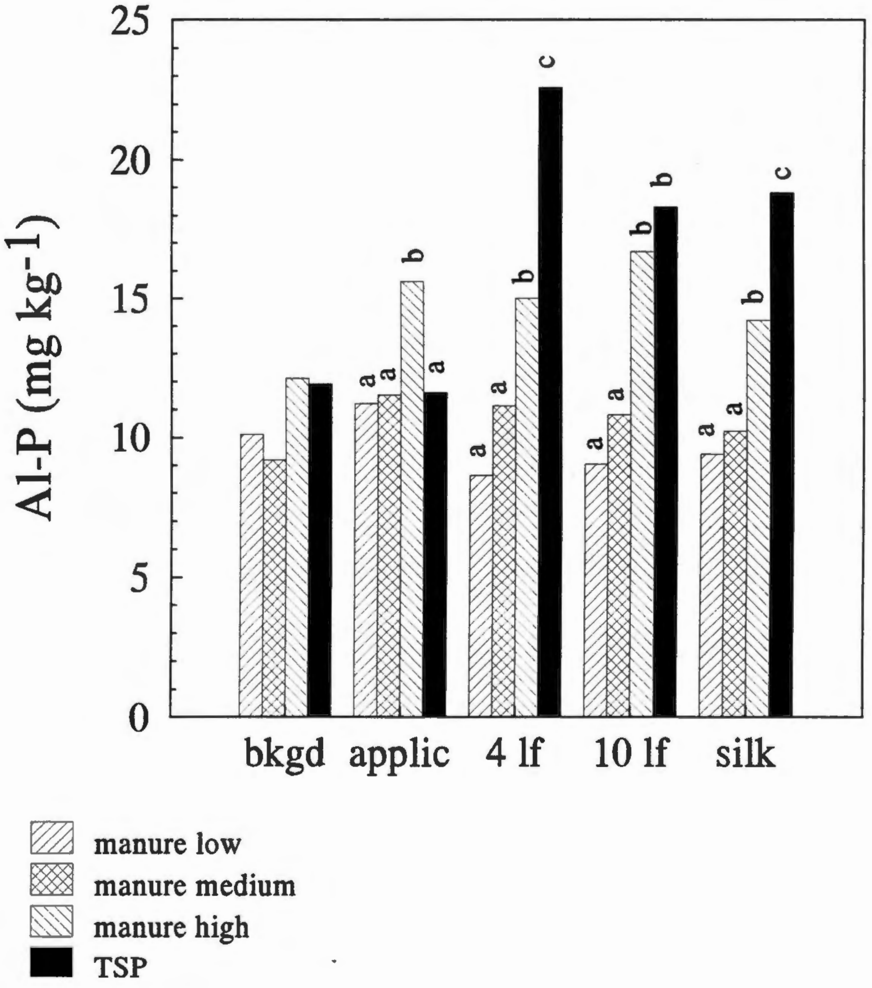

Figure 3.4. Effect of manure and triple superphosphate on Al-P in the surface 10 cm in 1993. Means within a sampling time followed by the same letter are not significantly different at the 0.05 probability level.

which received TSP had approximately twice as much Al-P compared with plots which received low and medium rates of manure P (Figure 3.4).

Sampling time and soil depth interacted to affect Al-P in 1992. In general, Al-P increased with time and depth (Table 3.5), although the time effect was not significant at all depths. There was no time by depth interaction in 1993, but depth and time effects were significant. Aluminum P decreased from background concentrations after application, then returned to background levels when corn was at four leaf and reached its highest concentration when corn was at ten leaf. By the time corn was silking, however, Al-P had returned to background levels (Table 3.6). The concentration of Al-P decreased from 13 mg kg^{-1} in the top 10 cm to 9.61 mg kg^{-1} in the 10-20 cm depth, then increased in the 20-30 and 30-45 cm depths (Table 3.7).

Manure and fertilizer did not affect Ca-P in 1992, but there was a significant effect in the top 10 cm at the last two sampling times in 1993. Although Ca-P was relatively constant in samples taken before application, after application, and when corn was at four-leaf, Ca-P increased by about 5 mg kg^{-1} when corn was at ten-leaf and silking in plots treated with TSP (Figure 3.5). This increase may have been due to conversion of Al-P to Ca-P, since there was a concurrent decrease in Al-P of about 5 mg kg^{-1} in TSP plots (Figure 3.4).

In 1992, sampling time affected Ca-P only in the 10-20 and 20-30 cm depths, with concentrations generally higher in the middle of the growing season (Table 3.5). At all sampling times in 1992, Ca-P increased with depth to 30 cm, after which there was a slight decrease with depth throughout the rest of the profile sampled (Table 3.6).

Table 3.5. Effect of depth and sampling time on inorganic P fractions in 1992.

		Sampling time			
Fraction	Depth	Application	4 leaf	10 leaf	silking
	cm	----------mg P kg^{-1}----------			
Al-P	0-10	11.8 a†	12.7 a	13.1 a	12.5 a
	10-20	9.41 bA	10.9 bB	12.5 aC	11.0 aB
	20-30	16.1 c	15.2 c	15.6 b	17.2 b
	30-45	ND‡	19.1 dA	20.3 cdA	22.6 cB
	45-60	ND	20.4 dA	20.8 cdA	24.0 cB
	60-75	ND	ND	21.6 d	23.3 c
	75-90	ND	ND	18.7 cA	23.7 cB
Ca-P	0-10	14.4 a	14.1 a	15.6 ab	14.0 ad
	10-20	16.1 bAB	17.4 bB	15.5 abA	14.3 aA
	20-30	18.7 cA	21.5 cAB	24.9 cB	19.6 bA
	30-45	ND	19.9 cd	20.9 d	17.9 bc
	45-60	ND	18.1 bd	16.4 a	16.7 ac
	60-75	ND	ND	15.1 ab	14.1 cd
	75-90	ND	ND	13.2 b	11.5 d
Fe-P	0-10	72.3 aA	63.0 aB	93.4 aC	91.5 aC
	10-20	89.7 bA	82.1 bA	97.1 aB	102 aB
	20-30	143 cA	123 cB	144 bA	156 bC
	30-45	ND	138 dA	165 cdB	188 cC
	45-60	ND	137 dA	168 cdB	185 cC
	60-75	ND	ND	176 c	180 c
	75-90	ND	ND	158 d	167 b
Red-P	0-10	83.1 aA	109 aB	108 B	89.6 aA
	10-20	103 bA	133 bB	102 A	97.8 abA
	20-30	144 cA	163 cB	111 C	119 cdC
	30-45	ND	159 cdA	111 B	127 cB
	45-60	ND	152 dA	120 B	129 cB
	60-75	ND	ND	116	120 cd
	75-90	ND	ND	118	107 bd

† For each fraction, means in the same column followed by the same lower case letter and in the same row followed by the same upper case letter, are not significantly different at the 0.05 probability level.

‡ Not determined.

Table 3.6. Effect of sampling time on inorganic P fractions in 1993.

Sampling time	Al-P	Ca-P	Fe-P	Red-P
		---mg kg^{-1}---		
background	14.6 a†	19.2 a	131 a	132 a
application	12.9 b	18.6 ab	124 b	123 b
4 leaf	14.8 a	18.0 b	109 c	134 a
10 leaf	17.0 c	20.6 c	117 d	139 c
silking	14.3 a	17.9 b	153 e	126 b

† Means in the same column followed by the same letter are not significantly different at the 0.05 probability level.

Table 3.7. Effect of depth on inorganic P fractions in 1993.

Depth	Al-P	Ca-P	Fe-P	Red-P
		---mg kg^{-1}---		
0-10	13.0 a†	15.7 a	102 a	107 a
10-20	9.61 b	17.0 b	102 a	119 b
20-30	14.9 c	22.2 c	139 b	150 c
30-45	18.9 d	21.7 c	152 c	149 c
45-60	19.9 d	18.4 d	152 c	138 d

† Means in the same column followed by the same letter are not significantly different at the 0.05 probability level.

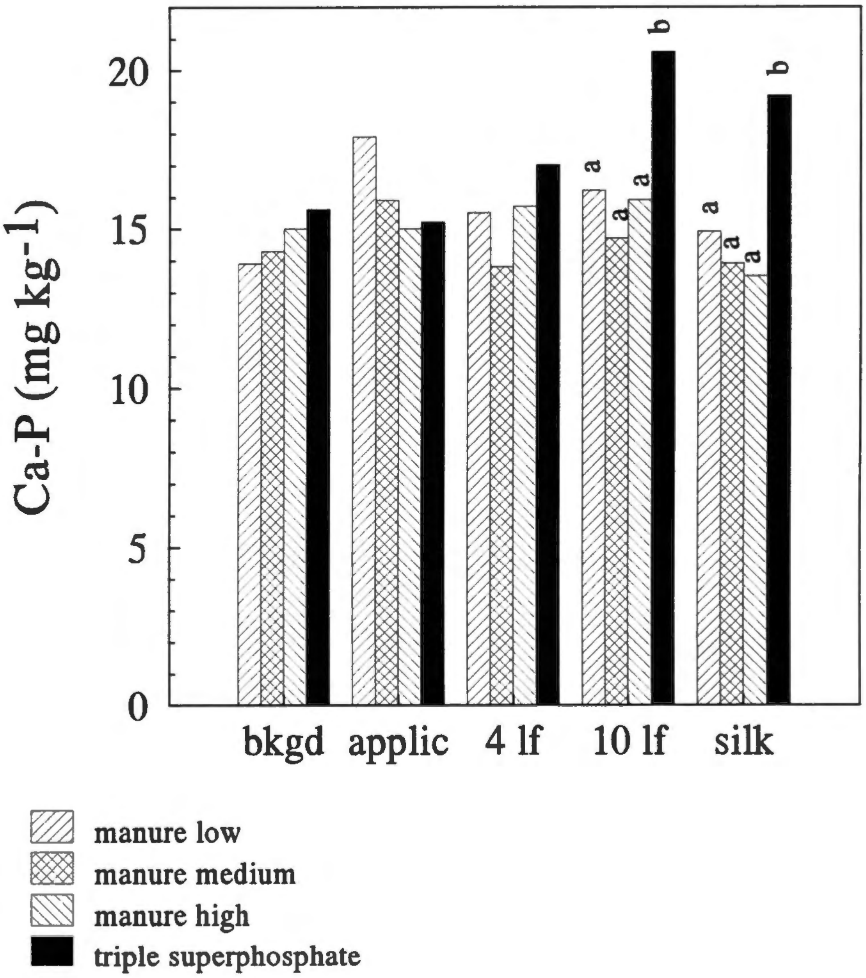

Figure 3.5. Effect of manure and triple superphosphate on Ca-P in the surface 10 cm in 1993. Means within a sampling time followed by the same letter are not significantly different at the 0.05 probability level.

In 1993, Ca-P increased with depth to 30 cm, remained constant at 45 cm, then decreased slightly at 60 cm (Table 3.7). In 1993 Ca-P increased with sampling time until corn was at 10-leaf, after which a slight decrease was observed at silking (Table 3.6).

After manure and fertilizer application in 1992, plots which received TSP had higher Fe-P in the top 10 cm than those which received manure (Figure 3.6), but this affect was not observed in samples collected after corn had emerged. This affect was also observed in the top 10 cm in 1993, but only when corn was at four leaf and silking (Figure 3.7).

Except at depths greater than 60 cm, sampling time affected Fe-P in 1992 (Table 3.5). In general, Fe-P decreased between application at four-leaf, then gradually increased throughout the rest of the growing season. At all sampling times in 1992, Fe-P increased with depth, with the concentration of Fe-P approximately twice as much at 30 cm as at 10 cm (Table 3.5). These trends were similar in 1993, with no depth by time interaction. Iron-P decreased between application and four-leaf, then increased with time throughout the rest of the growing season (Table 3.6). As was observed in 1992, Fe-P increased with depth in 1993, although differences were not as great due to higher levels of Fe-P in the surface soil in 1993 (Table 3.7).

Reductant-soluble P was not affected by manure or TSP application at any of the soil depths or sampling times, but depth and time interacted to influence Red-P in 1992. In general, Red-P increased between application and four-leaf, then decreased at later sampling times (Table 3.5). Except at the 10-leaf sampling time in 1992, Red-P

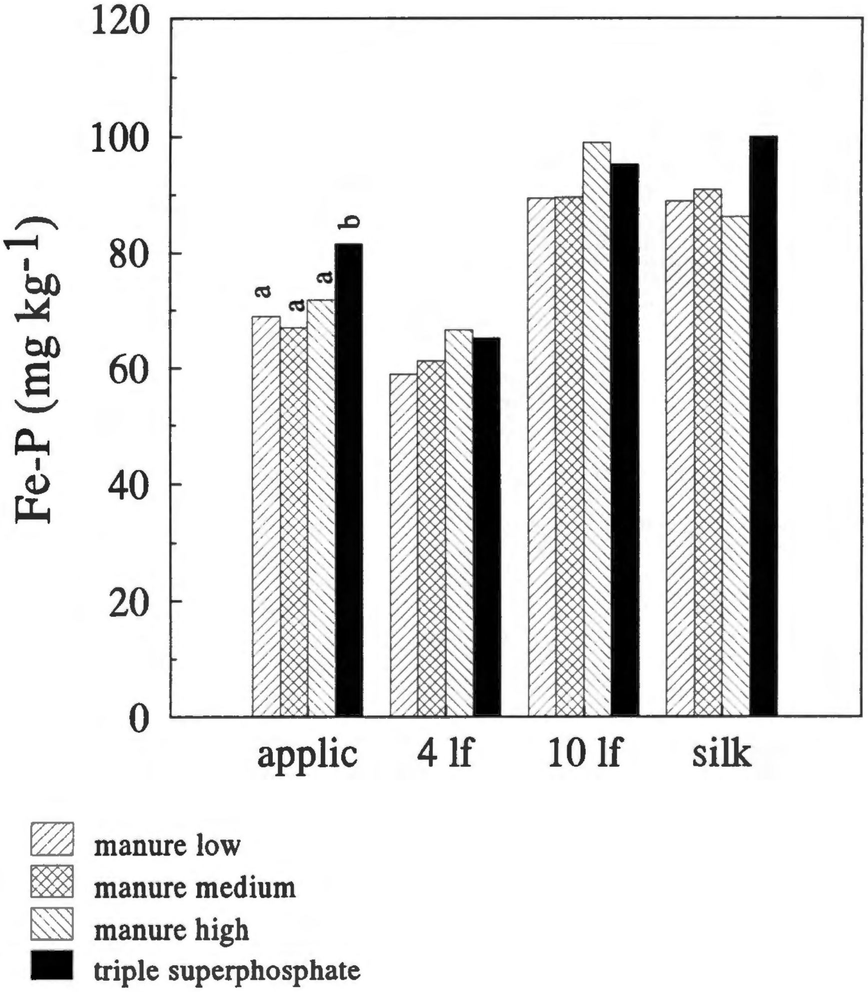

Figure 3.6. Effect of manure and triple superphosphate on Fe-P in the surface 10 cm in 1992. Means within a sampling time followed by the same letter are not significantly different at the 0.05 probability level.

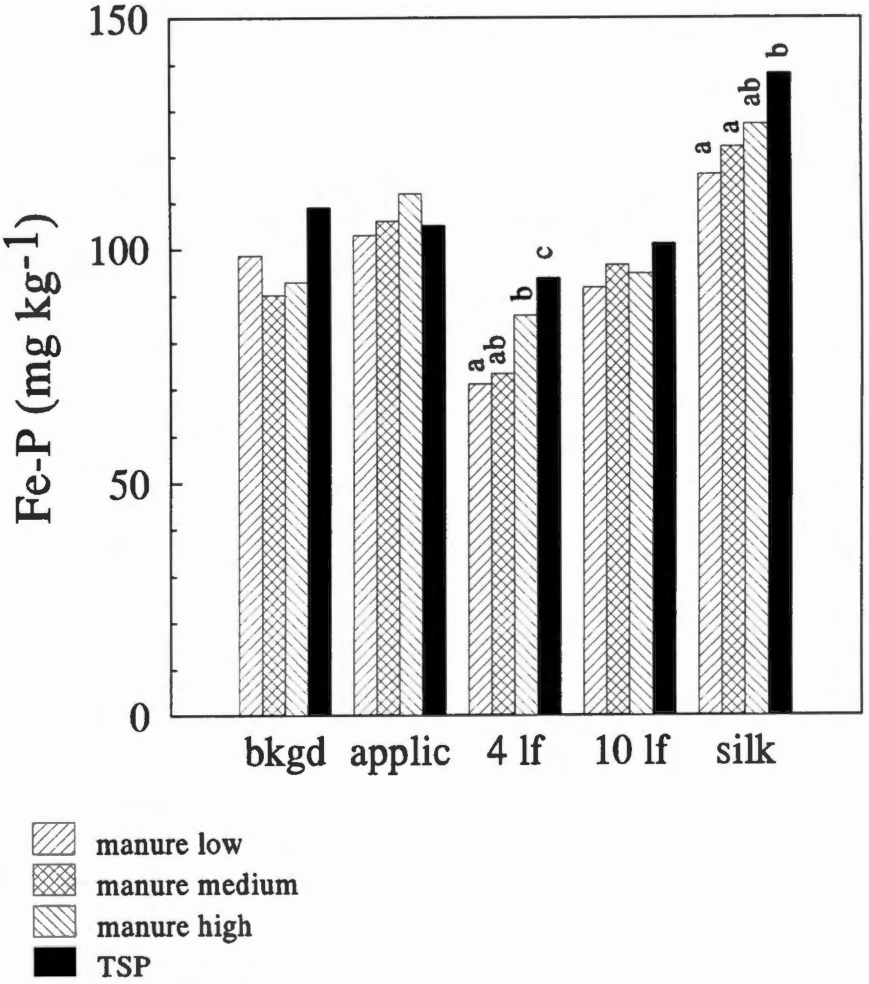

Figure 3.7. Effect of manure and triple superphosphate on Fe-P in the surface 10 cm in 1993. Means within a sampling time followed by the same letter are not significantly different at the 0.05 probability level.

increased with depth to 30 cm, below which it remained constant or decreased slightly with depth (Table 3.5). In 1993 there was no depth by time interaction, but the trends were similar to those observed in 1992. Reductant soluble P was affected by sampling time, although the differences were small (Table 3.6). As was observed in 1992, Red-P increased with depth to 30 cm, below which there was a slight decrease with depth (Table 3.7).

Phosphorus Availability Indices

Mehlich 3 extractable P was affected by P rate only in the surface 10 cm both years. In samples taken after manure and TSP application in 1992, concentrations of Mehlich 3 extractable P increased with P rate (Figure 3.8). The medium and high manure-P plots had higher Mehlich 3 P than the low manure-P plots, and the highest concentrations were observed in the plots which received TSP. Although this effect was not observed in samples taken when corn was at the four leaf stage, it was again observed at 10-leaf and silking that TSP application resulted in higher Mehlich 3 P. In 1993 background samples taken before treatments were applied, plots which had received the low manure-P rate had somewhat less Mehlich 3 P than the other treatments, but in samples taken after application there were no differences among treatments. When corn was at four-leaf, however, the low and medium manure-P treatments had less Mehlich 3 P than those which received high manure-P rates, and TSP application resulted in the highest concentrations. At 10-leaf the manure P plots were equal and TSP plots had greater Mehlich 3 P than the low and medium manure-P

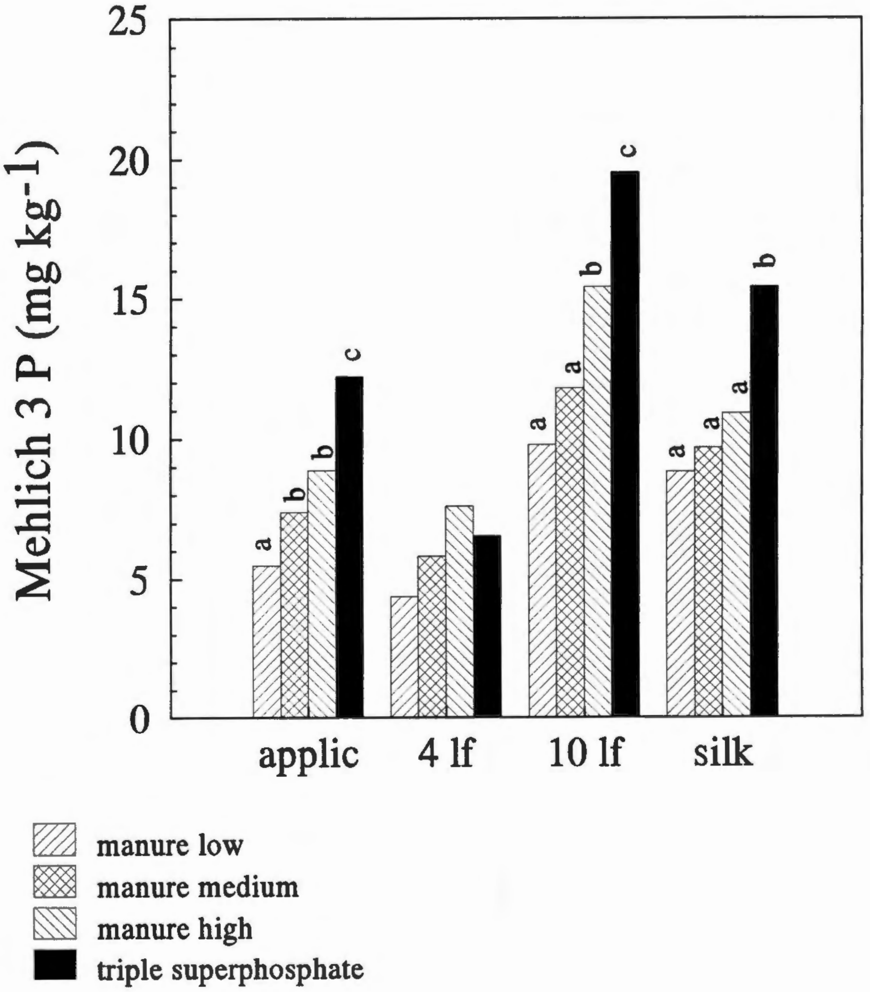

Figure 3.8. Effect of manure and triple superphosphate on Mehlich 3 P in the surface 10 cm in 1992. Means within a sampling time followed by the same letter are not significantly different at the 0.05 probability level.

plots, while at silking the TSP plots had approximately twice as much Mehlich 3 extractable P than manure plots (Figure 3.9).

Sampling time and depth interacted to affect Mehlich 3 P in 1992 and 1993. Mehlich 3 P usually increased with depth and tended to be higher at later sampling times (Table 3.8). The depth effect was probably related to increasing Fe-P at lower depths, while the time effect may have been the result of delayed availability of manure-P.

Manure and TSP application affected Olsen P only in the top 10 cm, with results similar to those observed with Mehlich 3 P. After application in 1992, TSP plots had higher Olsen P than manure plots, but no differences were observed in samples taken when corn was at four-leaf. In samples taken at 10-leaf, TSP plots had higher Olsen P than plots which received low and medium manure-P rates, while at silking there were no significant differences (Figure 3.10). In 1993 no differences were observed in background samples or those taken after application, but throughout the rest of the season TSP produced higher concentrations of Olsen P than did manure (Figure 3.11).

Olsen P was affected by depth (Table 3.9) and sampling time (Table 3.10) in 1992. The concentration of Olsen P in the 60-90 cm depths was approximately twice that of the surface soil, while a four-fold increase was observed from samples taken after application to those taken when corn was at silking. Sampling time had little effect in 1993, but the increase with depth was observed at all sampling times in 1993 (Table 3.11).

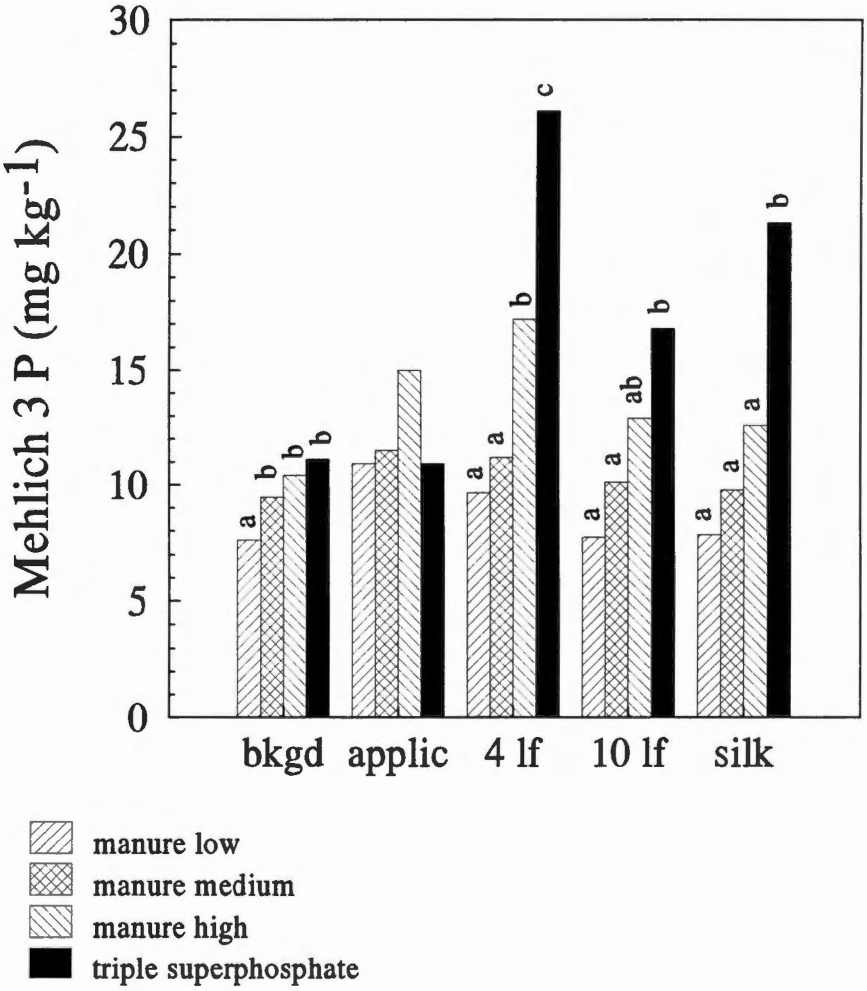

Figure 3.9. Effect of manure and triple superphosphate on Mehlich 3 P in the surface 10 cm in 1993. Means within a sampling time followed by the same letter are not significantly different at the 0.05 probability level.

Table 3.8. Effect of depth and sampling time on Mehlich 3 P in 1992 and 1993.

		Sampling time				
Year	Depth	Background	Application	4 leaf	10 leaf	Silking
	cm	mg P kg^{-1}				
1992	0-10	NR†	8.48 aA‡	6.09 aB	14.2 aC	11.2 aD
	10-20	NR	3.95 bA	3.19 bcA	10.6 bB	9.23 bC
	20-30	NR	3.72 bA	2.96 bA	10.7 bB	11.3 aB
	30-45	NR	ND§	3.76 cA	14.7 aB	15.1 cB
	45-60	NR	ND	5.16 dA	16.0 acB	16.7 cB
	60-75	ND	ND	ND	17.3 c	17.7 cd
	75-90	ND	ND	ND	17.8 c	18.4 d
1993	0-10	9.65 aA	12.1 aA	16.4 aB	12.0 aA	13.0 aAB
	10-20	7.83 bA	8.02 bA	8.21 bA	6.69 bB	7.29 bAB
	20-30	9.90 aA	9.35 bAB	10.7 bcA	8.13 bB	8.35 bB
	30-45	14.4 cA	ND	12.7 cB	11.3 aB	12.6 aB
	45-60	16.2 dA	ND	15.6 aAB	14.5 cB	14.3 aB
	60-75	ND	ND	ND	ND	ND
	75-90	ND	ND	ND	ND	ND

† Not reported; see Table 3.1

‡ For each year, means in the same column followed by the same lower case letter and in the same year followed by the same upper case letter, are not significantly different at the 0.05 probability level.

§ Not determined.

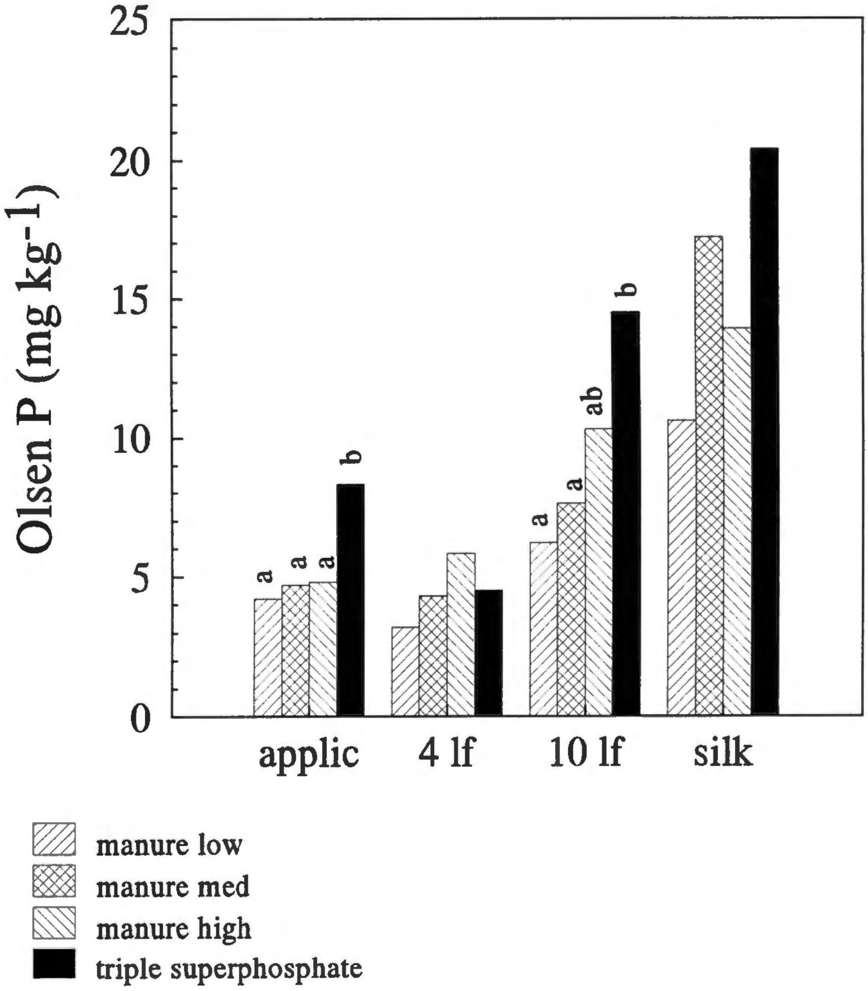

Figure 3.10. Effect of manure and triple superphosphate on Olsen P in the surface 10 cm in 1992. Means within a sampling time followed by the same letter are not significantly different at the 0.05 probability level.

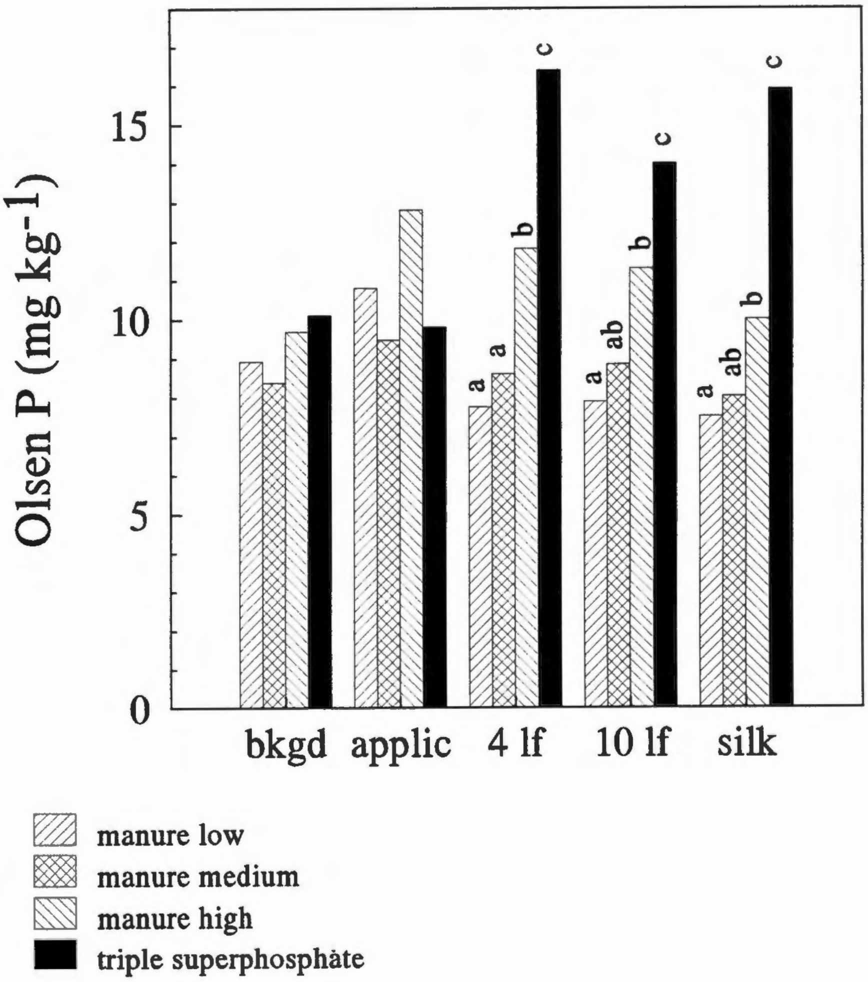

Figure 3.11. Effect of manure and triple superphosphate on Olsen P in the surface 10 cm in 1993. Means within a sampling time followed by the same letter are not significantly different at the 0.05 probability level.

Table 3.9. Effect of depth on Olsen P in 1992.

Depth	Olsen P
cm	-----mg kg^{-1}-----
0-10	8.80 a†
10-20	8.44 a
20-30	9.03 a
30-45	13.9 b
45-60	12.8 b
60-75	18.4 c
75-90	18.0 c

† Means followed by the same letter are not significantly different at the 0.05 probability level.

Table 3.10. Effect of sampling time on Olsen P in 1992.

Sampling time	Olsen P
	-----mg kg^{-1}-----
application	4.78 a†
4 leaf	5.41 a
10 leaf	12.1 b
silking	18.8 c

† Means followed by the same letter are not significantly different at the 0.05 probability level.

Table 3.11. Effect of depth and sampling time on Olsen P in 1993.

	Sampling time				
Depth	Background	Application	4 leaf	10 leaf	Silking
cm	-----------------------------------mg P kg-1-----------------------------------				
0-10	9.27 a†	10.7 a	11.3 a	10.8 a	10.5 a
10-20	8.62 aA	7.86 bB	6.61 bC	6.91 bC	6.50 bC
20-30	10.3 b	9.92 a	8.80 c	9.57 c	9.04 c
30-45	12.5 c	ND‡	11.4 a	12.3 d	12.2 d
45-60	13.8 d	ND	13.0 d	14.0 e	13.4 d

† Means in the same column followed by the same lower case letter and in the same row followed by the same upper case letter, are not significantly different at the 0.05 probability level.

‡ Not determined.

Correlation of available P with soil P fractions

Although a knowledge of the distribution of inorganic P among the fractions described above provides information on the potential P fertility status of a soil, the procedures are not suited to routine analysis of large numbers of samples. If P fractionation data are to be used in soil testing, it is necessary to relate the inorganic P fractions to methods which are better adapted to rapid analysis.

Although significant correlations were observed between Mehlich 3 P and all soil P fractions in 1992, only Al-P and Fe-P had correlation coefficients (r) greater than 0.5 (Table 3.12). Olsen P in 1992 was also correlated best with Al-P and Fe-P, although their correlation coefficients were somewhat lower (r=0.42 and 0.47, respectively). In 1993, Olsen P was significantly correlated with all soil P fractions (Table 3.13). As observed in 1992, Al-P and Fe-P had the highest correlation coefficients (r=0.70 and 0.50, respectively). Mehlich 3 P was correlated with Al-P (r=0.63) and Fe-P (r=0.36) in 1993. Similar correlations were observed by Datta and Khere (1969), with Olsen P correlated with Al-P (r=0.65) and Fe-P (r=0.53). Debnath and Mandal (1982) found Fe-P more highly correlated than Al-P with Olsen P (r=0.65 and 0.53, respectively). Singh et al. (1968) found Olsen P to be correlated with Ca-P, but soils were neutral and Ca-P was the predominant P fraction. No significant correlations were observed by Deo and Ruhal (1970) between Olsen P and inorganic P fractions in experiments with seven calcareous soils. Mehlich 3 P was correlated to Al-P (r=0.78), Fe-P (r=0.31), and Ca-P (r=0.25) in 136 soils studied by Jittanoonta (1977).

Table 3.12. Correlation of Mehlich 3 and Olsen P with soil P fractions in 1992.

Soil P fraction	Mehlich 3	Olsen
	----------correlation coefficient r----------	
Total	0.11 *	0.19 ***
Organic	-0.17 ***	NS
Al-P	0.62 ***	0.42 ***
Ca-P	-0.18 ***	-0.16 **
Fe-P	0.59 ***	0.47 ***
Red-P	-0.24 ***	NS

*,**,*** Significant at the 0.05, 0.01, and 0.001 probability levels, respectively.

Table 3.13. Correlation of Mehlich 3 and Olsen P with soil P fractions in 1993.

Soil P fraction	Mehlich 3	Olsen
	----------correlation coefficient r----------	
Total	0.42 ***	0.45 ***
Organic	0.24 ***	0.20 ***
Al-P	0.63 ***	0.70 ***
Ca-P	NS	0.23 ***
Fe-P	0.36 ***	0.50 ***
Red-P	0.12 **	0.29 ***

*,**,*** Significant at the 0.05, 0.01, and 0.001 probability levels, respectively.

Summary

The addition of manure and TSP affected soil P only in the surface 10 cm during the two years studied. Organic P decreased with depth, while inorganic P fractions generally increased with depth. Reductant-soluble and Fe-P were the most prevalent inorganic P forms measured, while Al-P and Ca-P occurred at much lower concentrations. Available P as measured by the Mehlich 3 and Olsen extractants increased with depth, but was very low in initial samples taken before manure and fertilizer appliation.

In 1992, manure and TSP application had no effect upon total or organic P. This was consistent with expectations, since P rates were low in comparison to the total P initially present. Initial organic P concentrations were also high since the soil had not been cultivated in many years. Manure and TSP application did effect Al-, Fe-, Mehlich 3, and Olsen P in the first year, although not at all sampling times. Application of TSP generally resulted in higher concentrations of these fractions than did maure application.

Soil organic P content was higher and more evenly distributed with depth in 1993. This was likely the result of organic P mobility from organic matter which accumulated at the soil surface, as well as from the added manure. Toward the end of the 1993 growing season, TSP application resulted in higher concentrations of Ca-P in the surface 10 cm of soil as compared to plots which received manure P. This may have been due to the gradual formation of the more stable solid phases.

Available P as measured by the Mehlich 3 extractant was higher in TSP plots than manure plots at nearly all sampling times. Although somewhat responsive to manure P application rate, Mehlich 3 P increased most consistently with TSP application. Similarly, available P as measured by the Olsen extractant was often higher in TSP plots than in manure plots, especially toward the end of the 1993 growing season. Available P by both extractants was much higher than initial levels after two years of fertilization, but would still be considered low in available P.

Correlations of available P with soil P fractions were variable between years. Available P was most highly correlated with Al-P and Fe-P, while inconsistently and less highly correlated with the other soil P fractions. In general, correlation coefficients were higher and more significant in the second year of the experiment.

Application of liquid swine manure results in increased levels of Al-, Ca-, and Fe-P, as well as increased available P as measured by Mehlich 3 and Olsen extractants. However, manure P was not as effective as TSP in increasing these soil P concentrations. Assessment of P availabilty from both sources is complicated by temporal variability in soil P fractions, especially with the Fe-P fraction. Distribution of soil P throughout the growing season must be considered when studying such a dynamic system, and many years of research are needed to evaluate the long-term affect of continuous additions of manure-P at relatively low rates.

CHAPTER 4

CORN YIELD AND PHOSPHORUS UPTAKE AS AFFECTED BY LIQUID SWINE MANURE APPLICATION

Introduction

The application of swine manure as a source of P for corn production provides a means for the utilization of waste material. Efficient utilization of manure P requires knowledge of P availability from manure, as well as soil tests which reflect the availability of P from manure-amended soils. Information regarding the availability of manure P is limited since manure rates have often been based on N content.

In a three-year field study, Sutton et al. (1982) found that manure P applied at 133, 200, and 266 kg ha^{-1} usually produced higher corn yields than control plots receiving no fertilizer, but not higher than plots fertilized with 56 kg P ha^{-1} as triple superphosphate. Ear leaf P concentrations were greater in fertilized than in unfertilized plots, but manure had no effect on ear leaf P content. Bernal et al. (1993) reported higher $NaHCO_3$-extractable P as manure P was increased from 60 to 240 kg ha^{-1}, but P uptake by green pepper (*Capsicum annuum*) was not increased by applying rates greater than 120 kg P ha^{-1}.

Motavalli et al. (1989) found that corn yields were increased by injected dairy manure relative to unfertilized plots, but yields did not increase with increasing manure rate. Silage yields were usually increased by high manure rates relative to unfertilized control plots, while lower manure rates produced dry matter yields equivalent to unfertilized plots. When comparing nutrient uptake from manure-amended and commercially fertilzed soils, the authors found an average fertilizer equivalence of 49 percent for manure P. Availability was calculated to be as low as 12 and as high as 89 percent, which approximates the 80 percent availability estimated by Goan and Jared (1992) for poultry manure.

The ability of a soil test extractant to predict crop response to fertilizer P can be evaluated by correlating extractable P to plant responses such as dry matter yield, grain yield, plant P concentration, and plant P uptake. In a study of 22 soils, Datta and Khera (1969) observed that relative yield of corn was correlated to Al-P (r=0.85), Fe-P (r=0.76), and Olsen P (r=0.79). Hardin et al. (1989) evaluated P uptake by sorghum-sudangrass (*Sorghum bicolor* x *S. sudanese*) from a loess-derived soil and correlated uptake with P measured by Bray 1, Bray 2, Mehlich 1, Mehlich 3, and Mississippi soil test extractants. Regression coefficients (R^2) ranged from 0.35 to 0.74, with no one extractant appearing to be superior as an indicator of P availability.

In order for a soil test to efficiently predict plant response to soil P, an understanding of the relationship between soil P fractions and plant uptake is needed. The objectives of this study were to determine the effects of manure P on corn yield and to correlate yield and P uptake to soil P fractions.

Materials and Methods

Plots were established in 1992 at the Ames Plantation, Grand Junction, Tennessee, on a Lexington silt loam (fine-silty, mixed, thermic Typic Paleudalf). Plots were 3 x 15 m, with four P rates, two corn varieties, and five replications. Control plots were fertilized with 60 kg P ha^{-1} as triple superphosphate and 170 kg N ha^{-1} as ammonium nitrate. Manure plots also received 170 kg N ha^{-1} by assuming 40 percent availability of manure N and using ammonium nitrate to supplement any deficit. Manure was surface-applied to the plots from a stainless steel tank mounted on a trailer, with manure flow to four F210 SS flood nozzles supplied by a submersible pump suspended in the tank. Several tankloads of manure were required for complete application to the plots, with multiple passes with the spreader used to achieve the higher manure rates. Each tankload was sampled and analyzed to determine the specific P loading for each plot. Although the goal was to apply manure to provide 15, 30, and 60 kg P ha^{-1}, 1992 rates were 17, 32, and 49 kg ha^{-1}, while 1993 rates were 5, 11, and 22 kg ha^{-1}. After manure application, plots were sampled to a depth of 30 cm. Plots were planted with 'DeKalb 689' and 'Pioneer 3343' varieties of corn, and soil samples collected to a depth of 60 cm when corn was at four leaf, 10 leaf, and silking.

Soil samples were air dried and passed through a 2 mm sieve. Available P was determined by Olsen (0.5 *M* $NaHCO_3$, pH 8.5) and Mehlich 3 (0.2 *M* HOAc + 0.25 *M* NH_4NO_3 + 0.015 *M* NH_4F + 0.013 *M* HNO_3 + 0.001 *M* EDTA) using a 1:20 soil:solution ratio with shaking times of 5 and 30 minutes, respectively. After grinding the soil to 0.4 mm, total P was determined by digestion with $H_2SO_4/H_2O_2/HF$ (Bowman, 1988). Organic P was determined by extraction with concentrated H_2SO_4 and 0.5 *M* NaOH (Bowman, 1989), after which the acid and base extracts were combined at a 1:2 ratio for analysis. Organic P was calculated as the difference between total P measured by ICP-AES and inorganic P measured colorimetrically. A modified Chang and Jackson procedure (Petersen and Corey, 1966) was used to fractionate the inorganic soil P into aluminum, iron, calcium, and reductant soluble phosphates.

When plants were at four leaf and 10 leaf growth stages, whole plants were harvested from a 1 m section of row. When plants were at silking, ear leaves were harvested from a 1 m row section. After drying at 333 K, plants were finely ground for analysis. A 0.2 g sample was weighed into a 16 x 100 mm test tube and ashed overnight at 773 K, and the residue dissolved in 10 mL of 2 *M* HCl. Phosphorus was determined by ICP-AES. Plot center rows were hand harvested to determine grain yields adjusted to 15.5 percent moisture.

Data analysis was performed using the GLM and CORR procedures of the Statistical Analysis Systems (SAS), and means were separated using Duncan's multiple range test. After determining that soil sampled from depths greater than 10 cm was not affected by treatment and did not improve correlations of soil P to plant response, data analysis was performed using only soil samples from the top 10 cm.

Results and Discussion

Effect of P and variety on grain yield and ear leaf P content

Corn grain yield was not affected by P application in either year (Table 4.1). This is consistent with results reported by Parks (1991) in which P applications rarely increased yield. The lack of a yield difference between the TSP treatments and the low P rates applied with manure in 1993 further substantiates the low response to added P. There was no effect of variety in 1992, but in 1993 'DeKalb 689' yielded higher than 'Pioneer 3343'.

Ear leaf P concentration was affected by P application rate in 1992. Ear leaf P was lower when the low manure rate was applied and highest for TSP, with the medium and high manure rates intermediate between the two (Table 4.1). There was no effect of variety on ear leaf P in either year.

Correlation of ear leaf P concentration with soil P

Correlation of ear leaf P concentration to soil P varied with year and variety. In 1992, ear leaf P content of 'DeKalb 689' correlated with organic P, Al-P, and Mehlich 3 P in soil samples taken at silking. Ear leaf P content of 'Pioneer 3343' was correlated to reductant-soluble P, Fe-P, and Mehlich 3 extractable P in 1992 soil samples taken at silking (Table 4.2). In 1993, ear leaf P content of 'Pioneer 3343' correlated with all P fractions except total and reductant soluble P in soil samples taken at silking, while ear leaf P content of 'DeKalb 689' did not correlate with soil P fractions.

When correlating ear leaf P with soil P in samples collected before planting, different results were obtained (Table 4.3). In 1992, ear leaf P content of 'DeKalb 689' correlated with all soil P fractions except organic and Ca-P. Ear leaf P content of 'Pioneer 3343' correlated with Al-P, Olsen P, and Mehlich 3 extractable P in 1992.

Table 4.1. Effect of P rate and corn variety on yield and ear leaf P content.

	Grain yield (Mg ha^{-1})		Ear leaf P (%)	
Phosphorus applied	1992	1993	1992	1993
Manure low	7.52	9.34	0.204 a†	0.236
Manure medium	9.00	9.54	0.222 b	0.257
Manure high	8.87	9.74	0.233 b	0.251
Triple Superphosphate	7.86	9.68	0.248 c	0.266
Variety				
'DeKalb 689'	8.73	10.1 a	0.226	0.246
'Pioneer 3343'	7.93	9.14 b	0.228	0.258

† Means within a column followed by the same letter are not significantly different at the 0.05 probability level.

Table 4.2. Correlation of ear leaf P concentration with soil P fractions at silking.

	1992		1993	
	'DeKalb 689'	'Pioneer 3343'	'DeKalb 689'	'Pioneer 3343'
	--------------------correlation coefficient r--------------------			
Total P	NS	NS	NS	NS
Organic P	0.54*	NS	NS	0.63**
Al-P	0.45*	NS	NS	0.61**
Ca-P	NS	NS	NS	0.62**
Red-P	NS	0.61**	NS	NS
Fe-P	NS	0.51*	NS	0.73***
Olsen	NS	NS	NS	0.63**
Mehlich 3	0.56**	0.45*	NS	0.68***

*,**,*** Significant at the 0.05, 0.01, or 0.001 probability level or not significant (NS).

Table 4.3. Correlation of ear leaf P concentration with soil P in samples collected after manure and fertilizer application.

	1992		1993	
	'DeKalb 689'	'Pioneer 3343'	'DeKalb 689'	'Pioneer 3343'
	--------------------correlation coefficient r--------------------			
Total P	0.60**	NS	NS	NS
Organic P	NS	NS	NS	NS
Al-P	0.71***	0.57**	NS	NS
Ca-P	NS	NS	NS	NS
Red-P	0.47*	NS	NS	NS
Fe-P	0.54**	NS	NS	NS
Olsen	0.60**	0.57**	NS	NS
Mehlich 3	0.73**	0.61**	NS	NS

*,**,*** Significant at the 0.05, 0.01, or 0.001 probability level or not significant (NS).

In 1993, ear leaf P content was not correlated with soil P in samples taken after manure and fertilizer application.

In general, grain yields were not significantly correlated with soil P indices in samples taken throughout the growing season. Significant correlations were observed between 1992 'DeKalb 689' yield and soil P at silking. Grain yield was correlated with organic P (r=0.62**), Al-P (r=0.47*), Ca-P (r=0.54*), Olsen P (r=0.56*), and Mehlich 3 P (r=0.60*) in soil samples collected when corn was at silking.

Of the inorganic P fractions measured, Al-P was the fraction most consistently correlated with ear leaf P content. Mehlich 3 extractable P was significantly correlated with plant P more often than was Olsen extractable P, which was probably due to the ability of the Mehlich 3 extractant to measure Al-P. These fractions were most often affected by manure and fertilizer application (Table 4.4, Table 4.5), but each was present in small concentrations in the soil. These low concentrations reduce the ability of a soil extractant to predict biological responses to P fertilization due to the difficulty of detecting differences in small values. At the other extreme, Fe-P concentrations were sufficiently large that manure and fertilizer application had little effect on Fe-P. Seasonal variability affected this fraction more than the others.

Although Fe-P was not as highly correlated with P uptake as some of the other soil P fractions, its contribution to the P fertility of this soil should not be overlooked. The pools of Al-P and Ca-P are very small and are probably not the sole forms of plant-available P. It is suggested that Fe-P contributes to the pool of available P in this soil either directly or by conversion to other P forms. The relatively high

Table 4.4. Effect of manure and triple superphosphate on 1992 inorganic P in the top 10 cm.

Time	Treatment	Al-P	Ca-P	Red-P	Fe-P	Olsen	M 3
		------------------------mg kg^{-1}------------------------					
applic	manure low	9.08 a†	14.1	77.5	69.0 a	4.20 a	5.49 a
	manure med	10.1 ab	14.2	81.3	67.0 a	4.70 a	7.40 b
	manure high	12.0 b	13.5	87.0	71.8 a	4.79 a	8.87 b
	TSP	16.0 c	15.7	86.8	81.5 b	8.32 b	12.2 c
4 lf	manure low	11.3	11.8	110	58.9	3.19	4.37
	manure med	11.7	13.6	104	61.2	4.30	5.83
	manure high	14.2	16.6	112	66.6	5.83	7.62
	TSP	13.5	14.4	109	65.2	4.50	6.54
10 lf	manure low	11.4	15.4	108	89.4	6.20 a	9.78 a
	manure med	12.4	15.1	112	89.6	7.62 a	11.8 a
	manure high	13.2	16.4	108	98.9	10.3 ab	15.4 b
	TSP	15.4	15.4	104	95.2	14.5 b	19.5 c
silk	manure low	10.8 a	14.1	87.5	88.9	10.6	8.81 a
	manure med	10.9 a	12.0	92.0	90.9	17.2	9.67 a
	manure high	12.7 ab	13.6	79.4	86.2	13.9	10.9 a
	TSP	15.8 b	16.2	99.7	100	20.4	15.4 b

† For each sampling time, means within a column followed by the same letter are not significantly different at the 0.05 probability level.

Table 4.5. Effect of manure and triple superphosphate on 1993 inorganic P in the top 10 cm.

Time	Treatment	Al-P	Ca-P	Red-P	Fe-P	Olsen	M 3
		------------------------mg kg^{-1}------------------------					
applic	manure low	11.2 a†	17.9	106	103	10.8	10.9
	manure med	11.5 a	15.9	102	106	9.46	11.5
	manure high	15.5 b	15.0	104	112	12.8	15.0
	TSP	11.6 a	15.2	106	105	9.79	10.9
4 lf	manure low	8.62 a	15.5	113	71.0 a	7.75 a	9.65 a
	manure med	11.1 a	13.8	105	73.2 ab	8.59 a	11.2 a
	manure high	15.0 b	15.7	105	85.7 ab	11.8 b	17.2 b
	TSP	22.6 c	17.0	112	93.7 c	16.4 c	26.1 c
10 lf	manure low	9.03 a	16.2 a	108	91.6	7.88 a	7.72 a
	manure med	10.8 a	14.7 a	103	96.5	8.84 a	10.1 a
	manure high	16.7 b	15.9 a	110	94.6	11.3 b	12.9 ab
	TSP	18.3 b	20.6 b	117	101	14.0 c	16.8 b
silk	manure low	9.37 a	14.9 a	103	116 a	7.50 a	7.83 a
	manure med	10.2 a	13.9 a	101	122 a	8.02 ab	9.79 a
	manure high	14.2 b	13.5 a	101	127 ab	10.0 a	12.6 a
	TSP	18.8 c	19.2 b	104	138 b	15.9 c	21.3 b

† For each sampling time, means within a column followed by the same letter are not significantly different at the 0.05 probability level.

concentrations of Fe-P in the soil, as well as the degree of variability in these concentrations, would likey mask the correlation of Fe-P with plant uptake.

Summary

Corn yield was not affected by P rate in either of the two years studied, but ear leaf P concentration increased with P rate in 1992. Correlation of ear leaf P content with soil P fractions was inconsistent, with Mehlich 3 generally providing the highest correlations. Prediction of corn yield and P uptake was complicated by variability in plant response as well as by low sensitivity of the soil extractants to detect changes in P availability. Further work is needed to identify the P fractions which control plant responses and to develop extractants which can accurately quantify these P forms.

CHAPTER 5

SUMMARY

A liquid swine manure spreader was designed to uniformly deliver controlled rates of liquid manure to soil. The spreader was constructed using a 2300-L stainless steel milk tank mounted on a trailer. A submersible pump mounted in a tank supplies flow to an in-tank circulation system and to the flow meter and nozzles. Accurate flow measurements were obtained using a turbine flow meter and analyzer. The accuracy of the flow meter was verified by measuring water volume delivered through the nozzles with time. Nozzles were positioned to provide uniform flow to the soil surface.

The manure P concentrations were used to compute application rates to provide 15, 30, and 60 kg P ha^{-1} for corn production. Actual P application rates were determined from the P content of each manure load and the amount of manure applied to each plot. During 1992, manure applications provided 17, 32, and 49 kg P ha^{-1}. During 1993, manure applications provided 5, 11, and 22 kg P ha^{-1}. The disparity between 1992 and 1993 manure P rates and between actual rates and target rates accentuates the difficulty associated with liquid swine manure applications. Manure application rates were based on the P content of grab samples collected directly from the agitated lagoon, two to four weeks prior to soil application. Manure N and P were highly variable and directly related to the solids content. The solids content of the manure in the lagoon varies with time and is influenced by the degree of agitation, rainfall amounts, and the pumping frequency of the lagoon. Although efforts were made to minimize variation in solids content through agitation of the lagoon, considerable variation in manure nutrient content between tank loads was observed. The nutrient content of each tank load can not be instantaneously determined, limiting control over nutrient application rates.

To assess the ability of manure to provide sufficient P for corn production, a study was initiated to compare manure P to inorganic P from triple superphosphate (TSP). Control plots received 60 kg P ha^{-1} as TSP and 170 kg N ha^{-1} as ammonium nitrate. Manure plots also received 170 kg N ha^{-1} by assuming 40% availability of manure N and using ammonium nitrate to supplement any deficit. Plots were planted with 'DeKalb 689' and 'Pioneer 3343' varieties of corn.

After manure application and when corn plants were at four leaf, ten leaf, and silking, soil samples were collected from the plots for P fractionation. Soil samples were analyzed to determine total and organic P, and the inorganic P was fractionated into Al-P, Ca-P, Fe-P, and reductant-soluble P. Extractable P was determined by the Mehlich 3 and Olsen procedures.

Total P concentrations were between 400 and 500 mg kg^{-1} throughout the study, with organic P accounting for about 20% of the total P in the surface soils. Of the inorganic P fractions, Fe-P and reductant-soluble P had the highest concentrations (75-200 mg kg^{-1}), while Al-P and Ca-P were present in much smaller quantities (10-20 mg kg^{-1}). Both Mehlich 3 and Olsen extractants suggested low P availability, ranging from 5 to 20 mg kg^{-1}.

Effects of manure and fertilizer application were observed only in the top 10 cm of soil. Total P was not affected in 1992, but in 1993 total P was higher in the plots which received triple superphosphate. Organic P was not affected by these treatments in either year.

Of the inorganic P fractions examined, Al-P was most often affected by manure and fertilizer application, with TSP usually resulting in higher Al-P concentrations. Manure plots had lower concentrations of Ca-P, but only toward the end of the 1993 growing season. More seasonal variability was observed for Fe-P, which was occasionally higher in the plots which received triple superphosphate. Reductant-soluble P was not affected by P rate or source.

Available P as estimated by the Mehlich 3 and Olsen extractants generally increased with P application rate and was usually higher in the TSP plots than in the manure plots. Both extractants were correlated ($p=0.001$) with Al-P and Fe-P each year, with Al-P having the highest correlation coefficients.

Corn grain yield was not affected by P rate either year. Variety did not affect yield in 1992, but in 1993 'DeKalb 689' yielded higher than 'Pioneer 3343'. Ear leaf P concentration was affected by P rate only in 1992. Application of TSP resulted in slightly higher ear leaf P concentrations than the medium and high manure P rates, while lower ear leaf P was observed in plots which received the low manure rate.

Correlations of grain yield and ear leaf P concentration with soil P were highly variable between year and variety. Of the inorganic P fractions, Al-P was most often and most highly correlated with ear leaf P content. Of the extractants used to estimate available P, Mehlich 3 was only slightly better than Olsen in predicting P uptake.

Although manure P was found to produce corn yields which equaled those of plots receiving TSP, manure was not as effective as TSP in maintaining or increasing soil P. Many years of research at different locations are needed to adequately evaluate the effectiveness of manure as the sole source of added P for corn production.

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APPENDIX

3	3	3	3	3	3	3	3	Rep
3	3	4	4	2	2	1	1	P Rate
2	1	1	2	1	2	2	1	Corn Variety
301	302	303	304	305	306	307	308	Plot Number

2	2	2	2	2	2	2	2	5	5	5	5	5	5	5	5
1	1	2	2	3	3	4	4	1	1	2	2	3	3	4	4
2	1	1	2	1	2	2	1	1	2	1	2	2	1	2	1
201	202	203	204	205	206	207	208	209	210	211	212	213	214	215	216

1	1	1	1	1	1	1	1	4	4	4	4	4	4	4	4
2	2	1	1	4	4	3	3	4	4	3	3	1	1	2	2
2	1	2	1	1	2	2	1	2	1	1	2	1	2	2	1
101	102	103	104	105	106	107	108	109	110	111	112	113	114	115	116

P Rates:

1=Triple Superphosphate
2=Manure Low
3=Manure Medium
4=Manure High

Corn Varieties:

1='DeKalb 689'
2='Pioneer 3343'

Figure A.1. Plot plan showing field layout of fertilizer and variety treatments.

VITA

James David O'Dell was born August 21, 1965 and is the son of James R. and Mary (Craig) O'Dell of Spencer, West Virginia. He graduated from Spencer High School in 1983.

In September 1983 he entered Berea College, where he received a Bachelor of Arts degree in agriculture in May 1987. The following summer was spent working at the USDA Forest Service Northeast Forest Experiment Station in Berea, where he performed soil and water analysis.

In September 1987 he entered the Graduate School of The University of Tennessee at Knoxville. He received the Master of Science degree in Plant and Soil Science in August 1989, and began taking classes toward a doctorate immediately thereafter. In January 1990 he accepted a full-time position as a research associate in the Department of Plant and Soil Science, working on subsurface solute transport at the Oak Ridge National Laboratory. This continued until the summer of 1992, when he began work on the project which led to this dissertation.