Abstract Studies were conducted to investigate the effects of long-term management practices on level and distribution of soil P and enzyme activities involved in P transformations in soil. Treatments included manure, P, NP, NPK, and NPK plus lime. Cattle manure was applied every 4 years at 269 kg N ha\(^{-1}\) for over a century and chemical fertilizers were applied every year at 67 kg N, 14.6 kg P, and 28 kg K ha\(^{-1}\) for over 69 years. Total soil P increase in soils ranged from 4.5 to 10.3 kg P ha\(^{-1}\) year\(^{-1}\) with the highest increase detected in the P-treated and the lowest in the manure-treated plot. Approximately 77–86% of the applied inorganic fertilizer-P in the past 69–71 years was recovered either in the harvested grain or remained in the top 30 cm of soil, while only 32% of the applied manure-P was recovered. Microbial biomass C and activities of alkaline phosphomonesterase, phosphodiesterase, inorganic pyrophosphatase, and dehydrogenase were significantly higher in the soil treated with cattle manure. Acid phosphomonesterase activity, however, was significantly higher in soils treated with chemical fertilizers. Results from this study suggested that manure-P is relatively more mobile than inorganic fertilizer-P. Long-term application of cattle manure promoted microbiological activities and P cycling, but did not result in P accumulation to levels close to those in inorganic P fertilizer-treated soils.

Keywords Animal manure · Soil P · Phosphomonesterases · Phosphodiesterase · Pyrophosphatase dehydrogenase

Introduction

In the efforts to achieve sustainable agricultural production while maintaining and preserving the environment, we have come to face a crucial nutrient for life, phosphorus. Agricultural producers increasingly rely on applications of large quantities of chemical P to maximize yields, which may lead to a potential phosphate crisis (Abelson 1999). Meanwhile, large-scale cattle feedlot operators face an increasing risk of P pollution associated with concentrated animal production, generating large quantities of manure that are applied repeatedly in large amounts to limited land areas in proximity to the manure source (NRC 1993). Approximately 1.85\(\times\)10\(^8\) kg of P enters the environment in the form of animal manure per year in the USA (Wodzinski and Ullah 1996). It has been estimated that P accumulation rates in the USA and several European countries range from 8 to 40 kg P ha\(^{-1}\) year\(^{-1}\) with an average rate of 22 kg P ha\(^{-1}\) year\(^{-1}\) (Carpenter et al. 1998). Application of animal manure has led to soil P reaching up to 1,121 kg P ha\(^{-1}\) in Mehlich-3 extracts (Reed et al. 1998). A one-time application of 164 kg P ha\(^{-1}\) from solid beef manure resulted in consistently higher soil Olson P level and P uptake by corn plants when compared with soils treated with chemical fertilizers but not with manure (Ginting et al. 1998a). Although manure has been reported to improve soil properties (Sommerfeldt and Chang 1985) and crop yield (Ginting et al. 1998b; Sutton et al. 1986), studies by Nair et al. (1998) indicated that the manure-treated surface soils they studied had essentially no sorbing capacity. Thus, repeated heavy applications of manure may saturate the soil’s capacity to retain manure-P and result in P runoff and leaching that could potentially contaminate adjacent water bodies with excessive P (James et al. 1996). Most of the reports, however, were derived from results based on short-term studies (1–2 years). Although James et al. (1996) showed that manure-P accumulated in surface soils after 20–40 years of heavy manure application, their study compared different farm soils with farmers’ recollections of manure applications. It is not clear to what extent manure-P accumulates and which major P forms accumulate in the soil. Even less work has been done to understand the importance of organic P in...
manure and its relationship to biological activities in soil.

Since the 1960s, we have learned that inorganic P is practically immobile in soil (Hannapel et al. 1964a). However, organic P seems to be more mobile and this was noted as early as 1902 by Dyer, according to Hannapel et al. (1964a). Hannapel et al. (1964a, b) demonstrated that organic P increased in solutions passed through soil columns amended with plant residues or other organic materials, but this was reduced by formaldehyde treatment, suggesting that P movement in these columns may be related to microbiological activities. More recently, Chardon et al. (1997) reported that 70–90% of total P from leachates of soil columns and lysimeters were in organic form and dissolved organic P (DOP), as a percentage of total P, increased with increasing soil depth, suggesting that DOP is an important form of P in soil solution and leachate.

One possible explanation for the accumulation of P when manure was applied is that manure-P accumulates excessively in soils in short-term application, but its mobility increases with time due to increasing microbial activities induced by manure application. This would eventually lead to increasing movement of manure-P in soils.

Of the extensive work devoted to studying P behavior and the fate of manure-P in soil, relatively little has been directed toward understanding manure-P behavior in soils under long-term animal manure application. Therefore, the objective of this work was to investigate the effects of long-term animal manure and chemical fertilizer treatments on soil P, soil properties, and activities of enzymes involved in P transformations in soil.

Materials and methods

Soil samples were obtained from a century-long continuous winter wheat (Triticum aestivum L.) experiment located in central Oklahoma, USA. The experiment was initiated on a Kirkland (fine, mixed, thermic Udertic Paleustolls) silt loam with a mean particle-size distribution of 37.5% sand and 22.5% clay. The manure treatment plot was initiated in 1899. The chemical fertilizer treatment plots were initiated in 1929. There are six plots still currently under investigation, including manure, P, NPK, NPK plus lime (NPK) and an untreated control plot. Cattle manure from a feedlot was applied every 4 years at 269 kg N ha⁻¹ and was incorporated into soil immediately following application in order to reduce potential surface runoff. The average ratio of N:P of the applied manure is 3.3:1, which suggests that approximately 89.7 kg P ha⁻¹ was applied every 4 years (22.4 kg P ha⁻¹ year⁻¹). Chemical fertilizer plots received an annual application of 67 kg N, 14.6 kg P, and 28 kg K ha⁻¹ before planting in October.

Long-term experiments could potentially provide considerable insights on the impacts of management practices on soil. Unfortunately, the application of statistics to agricultural experiments was not yet in place at the time that the treatments in this study were initiated. To compensate for the no-replication restriction, the underlying changes in these soils were evaluated based on random, individual samples in combination with sequential samples over a period of time. The plots were divided into three subplots with composite samples taken from each subplot to account for spatial variation in the field. Composite soil samples, including 18 cores per plot at 0–10, 10–20, and 20–30 cm, were taken in October 1998, 1 year before the last manure application. The most recent cattle manure application was conducted in September 1999. Surface soil samples (0–10 cm) were taken 4 months (January 2000) and 1 year (November 2000) following manure application. The summer of 2000 was unusually hot and dry in Oklahoma until mid-October. Thus, the sampling time was postponed for about 2 weeks. The field-moist soil samples were sieved through a 2-mm screen and stored at 4°C. A portion of each sample was air-dried and a portion of air-dried samples was ground to pass an 80-mesh (180 µm) sieve.

Soil pH values were determined using a combination glass electrode (soil:0.01 M CaCl₂ ratio = 1:2.5), and those of the organic C (Corg) and total N by dry combustion using a Carlo-Erba NA 1500 nitrogen/carbon/sulphur analyzer (Schepers et al. 1989). Particle size distribution was determined by a pipette analysis (Kilmer and Alexander 1949). Water-soluble P levels were determined by the procedure described by Olsen and Sommers (1982). Mehlich-3 extracts (Mehlich 1984) were filtered with 0.45 µm filters and P levels in these extracts were determined by Murphy and Riley method (Murphy and Riley 1962) (MRP), and by an inductively coupled plasma spectrometer (ICP-P). The difference between MRP and ICP-P was regarded as being DOP (Chardon et al. 1997). Total P was determined using a modified method described by Jones and Case (1990). Briefly, 5 ml HNO₃ was added to 1 g of soil in a digestion tube and digested at room temperature for 48 h. Samples were then heated at 120°C for 4 h. After standing for 5 min, 10 ml of HClO₄ was added, and samples were digested for an additional 2 h at 180°C. Samples were then cooled to room temperature, filtered through Whatman no. 42 filter paper, and diluted to 50 ml with deionized water. P content was determined using ICP. The microbial biomass C (Cmic) was determined by the chloroform–fumigation–incubation method described by Jenkinson and Ladd (1981) using a kₖ factor of 0.45 with subtraction of the control. Dehydrogenase activity was assayed by the method described by Casida et al. (1964). Activity of alkaline phosphomonoesterase (EC 3.1.3.1), acid phosphomonoesterase (EC 3.1.3.2), phosphodiesterase (EC 3.1.4.1), and inorganic pyrophosphatase (EC 3.6.1.1) were assayed with methods described by Eivazi and Tabatabai (1977), Tabatabai and Brenner (1969), Brownian and Tabatabai (1978), and Dick and Tabatabai (1978, respectively).

Organic C, total N, and total P were determined using samples with particle size less than 180 µm. Soil pH and particle-size distribution were determined with air-dried samples that were <2 mm. The Cmic content, dehydrogenase activity, and phosphatase activity were determined using the <2-mm field-moist samples. All results are expressed on a moisture-free basis. Moisture was determined after drying at 105°C for 48 h. Significant differences among treatments were determined using one-way analysis of variance (ANOVA). Comparison of treatment means was done using the least significant difference (LSD) test. Percentage data were transformed using the arcsin √Percentage transformation before analysis (Gomez and Gomez 1984). All results reported are averages of duplicated assays and analyses.

Results

Soil pH, organic C and total N

The pH values in the 0–10 cm surface soils ranged from 4.2 to 5.7, with that of the untreated control soil around pH 5 (Fig. 1A). Manure application increased soil pH significantly, while chemical fertilizer application resulted in slightly lower soil pH values (Fig. 1A). Little significant difference was detected among the samples from the three different sampling times. On the other hand, considerable variations in Corg and total N were detected among samples from different sampling times (Fig. 1B, C). Excluding data from November 2000 samples, Corg and total...