Recovery of Phosphorus in Soils Amended with Manure for 119 Years

Amber Pasket, Hailin Zhang , William Raun and Shiping Deng *

Department of Plant and Soil Science, Oklahoma State University, Stillwater, OK 74075, USA; apasket@okstate.edu (A.P.); hailin.zhang@okstate.edu (H.Z.); bill.raun@okstate.edu (W.R.)

* Correspondence: shiping.deng@okstate.edu; Tel.: +1-405-744-9591

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Abstract: Manure phosphorus (P) accumulation in soils is of environmental concern. The objectives were to determine P concentrations and fate in soils following 119 years of manure and 89 years of chemical fertilizer application. The recovery and distribution of P were evaluated for five years in soils from the untreated check, and soils amended with manure, fertilizer-P, or NPK. Total P concentrations were significantly higher in fertilizer-treated surface soils, compared to manure application. Treated plots had significantly higher P concentrations than the check. Virtually all of the added P was accounted for, either remaining in the soil or harvested in grains. Over 50% of fertilizer-P and about 38% of manure-P were found in the top 15 cm of the soil. A majority (81–99%) of the added fertilizer-P was found in the top 30 cm, while about 40% of manure-P leached down to the 30–90 cm level of the soil profile. Following 119 years of moderate application, manure-P did not reach deeper than 90 cm, suggesting that leaching to groundwater is not a concern at this site. Preserving P in the lower soil profile could enhance the potential for plant uptake.

Keywords: phosphorus; manure; fertilizer; recover; mobility; pollution

1. Introduction

Large-scale livestock feeding operations have resulted in high volumes of concentrated manure applied to nearby land for economically efficient waste disposal [1]. From 2015 to 2017, livestock production in the U.S. increased from 647 million to 677 million heads [2] and generated over 330 million tons of wet-weight manure based on estimation conducted using the method by Zhang and Schroder [3]. Manure has been traditionally applied based on crop nitrogen (N) demand. With an average N:P ratio of 3:1 in manure, compared to 8:1 in crops [4,5], N-based manure application results in P inputs exceeding crop needs and accumulation of P in soils [6,7]. Globally, manure-P applied to soils increased from 15 to 24.3 Tg year⁻¹ from 2000 to 2010 [8,9]. Ten percent of P inputs from either fertilizers or manure to soils may be lost to aquatic systems (~2–3 Tg P year⁻¹) [10,11]. With increasing concerns about harmful algal blooms (HABs) [12], it is crucial to understand the fate of manure-P in the environment.

Following century-long cattle manure and 70 years of chemical phosphorus fertilizer application, Parham et al. [13] recovered 32% of added manure-P and about 80% of added fertilizer-P in the top 30 cm of soil. A lower recovery of manure-P was also reported by van der Bom et al. [14], who found that up to 76% of pig manure-P was unaccounted for at 0–20 cm following application for 20 years. Whalen and Chang [15] reported that after a 16-year study, 1.4 Mg P ha⁻¹ was not accounted for in irrigated plots receiving 180 Mg cattle manure ha⁻¹ year⁻¹. The manure-P unaccounted for was hypothesized to be lost through runoff or leached below 150 cm in the soil profile. Many experiments addressing manure-P issues were short-term studies conducted for <4 years [16,17]. The study by Parham et al. [13] reported on a century-old experiment, but only the top 30 cm of soil was evaluated. Therefore, the century-old

site was revisited with respect to P recovery to a soil depth up to 90 cm. The objectives were to determine P concentrations, and the fate and distribution of added P in these soils.

2. Materials and Methods

The study site is a continuous winter wheat (*Triticum aestivum* L.) experiment located in central Oklahoma, U.S.A. (36°07′03.7″ N 97°05′37.0″ W). According to Oklahoma Climatological Survey [18], the annual average rainfall from 1895 to 2019 was 974.09 mm (38.35 inches) and annual average temperature is 15.3 °C (59.5 °F). The experiment was initiated on a Kirkland (fine, mixed, thermic Udic Paleustolls) silt loam, with the manure treatment (MT) initiated in 1899, and the P fertilizer (PF) and nitrogen-P-potassium (NPK) treatments initiated in 1930. Cattle feedlot manure was applied every four years at 134.5 kg N ha⁻¹ until 1966 then at 269 kg N ha⁻¹ since 1967. The manure was incorporated into soil immediately following application before planting in October. The average N:P ratio of the applied manure was about 3.3:1, which is equivalent to 40.8 kg P ha⁻¹ from 1899 to 1966 and 81.5 kg P ha⁻¹ from 1967 to 2018 that was applied every four years (equivalent to an annual input of 10.19 and 20.38 kg P ha⁻¹ year⁻¹ for 1930–1966 and 1967–2018, respectively). Chemical fertilizers were applied annually at 67 kg N, 14.6 kg P and 28 kg K ha⁻¹. Phosphorus was used in the form of ordinary (single) superphosphate from 1930 to 1967, and triple superphosphate from 1968 to the present. Plots received N as sodium nitrate between 1930 and 1967, ammonium nitrate from 1968 to 2004, and urea from 2005 to the present. Potassium was added as salts of nitrates, sulfates and chlorides. All plant residues were returned to the soil. No other treatments were done (i.e., no irrigation, application of pesticides or herbicide, etc.)

Soil samples were taken prior to planting in five different years (1998, 1999, 2000, 2006, and 2018), at depths of 0–15, 15–30, 30–60, and 60–90 cm from untreated check (CK), MT, PF, and NPK. Unfortunately, the application of statistics to agricultural experiments was not yet in place at the time when this study was initiated. To compensate for the no-replication restriction, evaluations were based on random, individual samples in combination with samples in different years. At each sampling event, each treatment plot (30.4 × 6.5 m) was divided into three subplots (10 × 6.5 m each). A five-core composite sample was taken from each subplot to account for spatial variation in the field. Soils were passed through a 2 mm sieve, air-dried, and kept in sealed containers at 23 °C until analysis.

Soil pH was determined using a combination glass electrode (soil: water ratio = 1:2.5), and the organic carbon (C) and total N by dry combustion using a Carlo-Erba NA 1500 Nitrogen/Carbon/Sulphur Analyzer [19]. Particle size distribution was determined using the hydrometer method [20]. Total P was determined using the wet digestion method described by EPA 3050B [21]. The basic properties of soils sampled in 2018 did not differ significantly from those reported by Parham et al. [13] and are shown in Table 1.

The recovery of P (%) from the added P was calculated as follows:

$$\% \text{ P recovery} = ((\text{P removed in grain} + \text{P in treated soil} - \text{CK soil P}) / \text{P input}) \times 100$$

where P input (kg P year⁻¹) = added manure- or fertilizer-P (kg P year⁻¹) and P removal = P in harvested grain (kg P year⁻¹). Unit conversion of soil P is explained in the footnote of Table 2. Data were divided and analyzed in two periods (1930–1966 and 1967–2018) because of major changes in management practices. Phosphorus removal was estimated based on the average grain yield of different periods and average grain-P content of 12 years (1980–1986, 1991–1995). Statistical analysis was performed using SAS 9.4. Evaluations were conducted by repeated measures over the years. Significant differences between the treatments at a specified depth interval were determined using PROC GLM according to Tukey's HSD test at an alpha = 0.05.

Table 1. Basic properties of soils sampled in 2018 by treatment and depth.

Treatment	Depth	pH _w	TN	OC	Sand	Silt	Clay
	(cm)				(%)		
CK	0–15	5.86B	0.06A	0.60A	42.50	37.93	19.60
	15–30	5.88A	0.05A	0.56A	39.17	38.33	22.50
	30–60	6.65A	0.06A	0.59A	32.50	34.60	32.93
	60–90	7.02A	0.04A	0.35A	31.67	30.43	37.93
MT	0–15	6.91A	0.10C	0.96B	40.00	41.30	18.80
	15–30	6.95B	0.08B	0.81B	38.33	40.03	21.70
	30–60	7.10B	0.07A	0.67A	30.00	34.63	35.47
	60–90	7.39A	0.04A	0.35A	30.00	30.83	39.17
PF	0–15	5.86B	0.07B	0.71A	41.67	40.87	17.53
	15–30	6.11A	0.06AB	0.64AB	40.00	38.77	21.27
	30–60	6.65A	0.06A	0.58A	30.83	37.93	31.27
	60–90	6.96A	0.04A	0.35A	33.33	32.53	34.20
NPK	0–15	4.78C	0.11C	1.11B	40.83	40.00	19.17
	15–30	5.01C	0.08B	0.73AB	40.00	39.60	20.43
	30–60	6.23C	0.07A	0.64A	32.50	37.53	30.03
	60–90	6.80A	0.04A	0.32A	30.83	34.60	34.60

Note: CK = untreated check, MT = manure treatment, PF = phosphorus fertilizer, NPK = nitrogen-phosphorus-potassium. pH was measured in water with soil/water ratio = 1:2.5, TN = total nitrogen, and OC = organic carbon. Different capital letters indicate significantly different means between the treatments and check at $p < 0.05$ according to Tukey's HSD test.

Table 2. Input and output of phosphorus (P) to the system under various fertilizer treatments.

Treatment	P Input (kg P ha ⁻¹ year ⁻¹)		Average Yield (kg ha ⁻¹ year ⁻¹)		Grain-P (mg P kg ⁻¹)	Grain-P Removal (kg P ha ⁻¹ year ⁻¹)		Net P Input (kg P ha ⁻¹ year ⁻¹)	
	1930–1966	1967–2018	1930–1966	1967–2018		1930–1966	1967–2018	1930–1966	1967–2018
CK	0.00	0.00	864.2A	1002.8A	3860.6	3.3	3.9	−3.34	−3.87
MT	10.19	20.38	1328.1B	1916.0B	3860.6	5.1	7.4	1.73	9.11
PF	14.68	14.68	1157.0A	1104.1A	3860.6	4.5	4.3	6.87	6.54
NPK	14.68	14.68	1342.1B	2073.7B	3860.6	5.2	8.0	6.16	3.33

Note: CK = untreated check, MT = manure treatment, PF = phosphorus fertilizer, NPK = nitrogen-phosphorus-potassium. Cattle manure was applied every 4 years at 134.5 kg N ha⁻¹ from 1930 to 1966 and at 269 kg N ha⁻¹ since 1967. The average N:P ratio in manure was 3.3:1. Inorganic P was applied annually at 14.68 kg P ha⁻¹ year⁻¹. Average grain-P in 12 years (1980–1986 and 1991–1995). Unit conversions from ha to kg of soil were based on average bulk density of 1.3 g cm³ for 0–30 cm, 1.25 g cm³ for 30–60 cm, and 1.2 g cm³ for 60–90 cm soil layers. For example, 1 ha = 1.95 × 10⁶ kg for 15 cm of soil when bulk density = 1.3 g cm³. Capital letters represent significant differences between treatment means at ($p < 0.05$) using Tukey's HSD test.

3. Results

Compared with the CK, NPK resulted in significantly lower soil pH, while MT had significantly higher pH. The P treatment had little influence on soil pH in the top 30 cm (Table 1). Treatments had a limited effect on pH in subsurface soils. Organic C and total N in MT and NPK were significantly higher than CK, while little difference was detected between PF and CK. Treatments did not result in significant changes in soil texture.

Due to a change in rate, P input to soil from manure was lower than fertilizer-P from 1930 to 1966, but higher from 1967 to 2018 (Table 2). Grain yields between MT and NPK during the 89 years were not significantly different from each other, but significantly higher than CK and PF (Table 2). Total soil P averaged 193.1, 268.4, 326.4, and 319.6 mg P kg⁻¹ soil in 0–15 cm profile over five years for CK, MT, PF, and NPK, respectively (Table 3). All treatment plots had significantly higher total soil P than CK, and fertilizer plots were higher than MT. Total P at 30–60 and 60–90 cm was generally higher in MT than CK, PF or NPK.

Table 3. Total P concentration in soils at depth, treatment, and year specified.

Year	Depth	Total P (mg P kg ⁻¹ Soil) in Treatment Specified			
		CK	MT	PF	NPK
1998	0–15 cm	189.0A	218.6B	310.4C	266.4D
	15–30 cm	151.3A	191.7AB	223.7B	202.3B
1999	0–15 cm	185.4A	236.0B	307.1C	283.4D
2000	0–15 cm	186.3A	254.5B	298.1C	308.6C
2006	0–15 cm	208.0A	279.5B	321.8B	332.8B
	15–30 cm	186.4A	225.7A	225.3A	210.0A
2018	30–60 cm	152.1A	166.4A	158.3A	154.1A
	60–120 cm	105.1A	127.1A	109.9A	104.6A
	0–15 cm	196.7A	353.3B	396.7BC	406.7C
	15–30 cm	180.0A	250.0B	286.7B	283.3B
	30–60 cm	166.7A	183.3A	170.0A	180.0A
	60–90 cm	126.7A	133.3A	136.7A	126.7A

Note: a CK = untreated check, MT = manure treatment, PF = phosphorus fertilizer, NPK = nitrogen-phosphorus-potassium. Different capital letters following total P concentration values represent significant differences ($p < 0.05$) between treatments for each row according to Tukey's HSD test.

Nearly all of the added P was recovered in all treatments (Table 4). The added P from MT was transported deeper into the soil profile than P added from PF and NPK. Phosphorus recovery in five different years in the top 15 cm was markedly higher in NPK (67.2%) than MT (38.2%) or PF (51.6%) (Table 4). For PF and NPK, recovery was about 100% in 0–60 cm. For MT, the soil sampling depth was extended to 90 cm to recover around 100% of the P added. However, P recoveries from each depth were significantly different among treatments in the surface 15 cm of soils (Figure 1). Within each treatment, P recovery from 0–15 cm was significantly higher than subsurface soils. The P recoveries for MT at 15–30, 30–60, and 60–90 cm were not significantly different, while the recoveries for PF and NPK were significantly higher in 15–30 than 60–90 cm. The slightly greater than 100% recovery for NPK in 2018 reflected a discrepancy regarding estimation based on data from long-term treatments.

Table 4. Recovery (%) of added P calculated based on input and output by sampling year, soil depth, and treatment.

Year	Depth	% P Recovery in Treatment Specified		
		MT	PF	NPK
1998	0–15 cm	17.5	50.5	48.2
1999		27.1	50.5	56.6
2000		44.1	45.8	69.9
2006		45.3	48.1	70.4
2018		56.8	63.4	90.7
	Mean ± SE	(38.2 ± 7.0)	(51.6 ± 3.1)	(67.2 ± 7.2)
1998	0–30 cm	35.9	79.6	79.9
2006		64.4	66.8	91.0
2018		84.7	97.1	126.3
	Mean ± SE	(61.7 ± 14.2)	(81.1 ± 8.8)	(99.1 ± 14.0)
2006	0–90 cm	96.0	91.6	105.2
2018		108.2	110.7	129.3
	Mean ± SE	(102.1 ± 6.1)	(101.2 ± 9.6)	(117.2 ± 12.0)

Note: MT = manure treatment, PF = phosphorus fertilizer, NPK = nitrogen-phosphorus-potassium. % P recovery = % of added P recovered in soil or harvested grain. SE = standard error, calculated based on the three subplots.

Figure 1. Average P recovery (%) at 0–15, 15–30, 30–60, and 60–90 cm for 2006 and 2018. MT = manure treatment, PF = phosphorus fertilizer, NPK = nitrogen-phosphorus-potassium. Error bars represent standard error. Different lowercase letters represent significantly different treatment means within each soil depth, and capital letters represent comparisons of depth means within each treatment determined by Tukey's HSD test ($p < 0.05$).

4. Discussion

Although accumulation of P in soil following manure application has been widely reported [22,23], P accumulation in soil was less in MT than PF and NPK following 119 years of manure application and 89 years of fertilizer treatment. One can argue that the application rates of 10.2 and 20.4 kg P ha⁻¹ year⁻¹ are relatively low compared to the 39.6 kg P ha⁻¹ year⁻¹ application reported by Pizzeghello et al. [22]. However, a similar observation was reported when 220 kg P ha⁻¹ year⁻¹ was applied for six consecutive years [24]. In their study, total soil P concentrations in manure- and fertilizer-treated plots from 0 to 30 cm were 1008 and 1107 mg P kg⁻¹, respectively. Accumulation of manure-P is more a concern than fertilizer-P because leaching of fertilizer-P constitutes less than 1% of the P added (<2 kg P ha⁻¹) [25,26].

Repeated manure overapplication could saturate the soil's capacity to retain manure-P [27]. A survey of 10 long-term experiments (10–50 years) revealed that soils reached 25% P saturation after 10–30 years [28]. Some of the accumulated P could potentially reach adjacent water bodies [29] and cause HABs [12]. Nonpoint sources of P in agricultural runoff contribute a significant portion of freshwater P inputs [6]. However, the leaching of organic P should not be dismissed [30]. About 70 to 90% of total P from leachates of soil columns and lysimeters were in organic form, and the percentage of organic P in total dissolved P increased with increasing soil depth [13,31]. Leaching of P to groundwater would be of environmental concern [15]. However, P's movement to lower in the soil profile could enhance P storage and use efficiency in agricultural production.

About 100% of the added P was recovered from soil samples to a 90 cm depth at long-term manure treatment sites (119 years for the current report and 15 years in the study by Boitt et al. [32]), suggesting a low probability of groundwater contamination at these sites. Shepherd and Withers [33] also found close to 100% recovery of added manure- and fertilizer-P in the 0–35 cm soil profile following a 4-year study. However, other studies reported that not all added P was accounted for, including 25–30% in a two-year study [34] and one third from a 12-year experiment [35]. These two studies tested the top 20 cm of soil and the unaccounted for manure-P was assumed to be lost from leaching or erosion.

It is possible that the unaccounted for manure-P leached deeper into the soil profile. However, in a study based on the evaluation of 0–150 cm of the soil profile in irrigated land that was amended with up to 180 Mg ha^{−1} year^{−1} of wet cattle manure (equivalent to 804 kg P ha^{−1} year^{−1}), 7–15% of the added manure-P was not recovered following 16 years of treatment [15]. This was a concern because the water table at this site ranged from 50 to 250 cm. The application rates reported by Whalen and Chang [15] were considerably higher than other studies discussed earlier.

In this study, a portion of added manure-P transported deeper in the soil profile than fertilizer-P. Excluding P removed by grain, the added fertilizer-P was all accounted for in the top 30 or 60 cm of soil following 89 years of application, while on average 40% of the added manure-P was found in the 30–90 cm range of the soil profile. Although 50% of wheat roots are located in the top 16.8 cm of soil [36], the active rooting of winter wheat can reach up to 120 cm [37]. Manure-P in the deeper soil profile could be taken up by plants, enhancing P use efficiency. Wheat and maize are efficient in using residual P that accumulated in soils [38]. Phosphorus is the second-most limiting nutrient, being deficient in 67% of cropland [39]. The potential to enhance P use efficiency from a mere 16% on average between 1961 and 2013 [40] is of great significance in promoting crop productivity while preserving environmental health.

5. Conclusions

Added P accumulated in soils. Chemical fertilizer P accumulated more in the surface soils, while manure P moved more downward to the lower soil profiles. However, nearly all of the added P was recovered by sampling to 90 cm. Over 50% of fertilizer-P remained in the top 15 cm. Excluding grain-P, the added fertilizer-P was mostly in the top 30 cm, while 40% of added manure-P was in the deeper soil up to 90 cm. Considering that the manure treatment has been in place for 119 years, it is not likely that manure-P at this site would reach ground water and cause concern. On the other hand, the P leached lower into the soil profile and could enhance P storage and use efficiency.

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