

Total Phosphorus, Zinc, Copper, and Manganese Concentrations in Cecil Soil Through 10 Years of Poultry Litter Application

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Abstract: Poultry litter (PL) is an inexpensive and effective source of plant nutrients. However, overapplication could result in phosphorus (P) and heavy metal accumulation in soils. A field experiment evaluating PL application to a Cecil soil used for cotton and corn production has been maintained for 10 years. At the end of the cotton phase (i.e., the first 5 years), PL annually applied at 4.5 Mg ha⁻¹ did not increase concentrations of total soil P, zinc (Zn), Cu, or manganese. During the corn phase (i.e. the second 5 years), PL application rates were increased from two to four times that used for cotton partly because of corn's greater N demand. With this change, the average total P in the surface 15-cm soil nearly doubled to about 560 mg kg⁻¹ of dry soil in both conventional till and no-till fields at the end of the corn phase. During the same time, Cu increased from 7 to 22 mg kg⁻¹ and Zn increased from 17 to 32 mg kg⁻¹ of dry soil. Levels of manganese were basically unchanged. Total P and Cu also increased in the 15- to 30-cm depth, with concentrations in the 0 to 15 cm being 1.8 to two times that in the 15 to 30 cm for P and approximately two times for Cu. Relationships between extractable versus total P and Zn changed at a threshold point beyond which extractable P and Zn increased at more than double the initial rate. It seems that once accumulation of P and Zn exceeded the soil buffer capacity, nutrient availability was significantly altered. Therefore, close monitoring of soil nutrients especially P is essential to avoid over application of PL that may potentially pose environmental risks for water pollution.

Key words: No-till, conservation tillage, environmental risk.

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The southern states of Alabama, Arkansas, Georgia, Mississippi, and North Carolina account for more than 60% of the 8.6 billion broilers (*Gallus gallus domesticus*) raised in the United States and consequently produce nearly 9.3 million Mg of poultry litter (PL) a mixture of bedding material and manure) (National Agricultural Statistics Service, 2007). Poultry litter is a readily available source of N, phosphorus (P), potassium (K), and other nutrients used on crops and pastures. Application of PL to meet crop N requirements can over time result in accumulation of P and other elements. Kingery et al. (1994) reported elevated extractable levels of P, K, calcium (Ca),

magnesium (Mg), Cu, and zinc (Zn) in soils receiving PL during an extended period (15–28 years) in the Sand Mountain region of northern Alabama. Mitchell and Tu (2006) found that application of PL during a 10-year period increased Mehlich-1-extractable Ca, Mg, P, K, boron, Zn, and Ca in a Decatur silt loam soil in the Tennessee Valley of Alabama. Gascho and Hubbard (2006) found a buildup of extractable P, Cu, and Zn with long-term application of PL to a Tifton soil in the Southern Coastal Plain. Adeli et al. (2007) found significant buildup of total Cu, Zn, and As in cotton soils in Mississippi after 3 years of PL application under either conventional tillage (CT) or no-till (NT) management. Sistani et al. (2008) reported a Mehlich-3 P increase of 8.7 times (from 18 to 156 mg kg⁻¹) and 14.3 times (from 18 to 257 mg kg⁻¹) with, respectively, 11 and 22 Mg ha⁻¹ year⁻¹ PL application for 4 years. Although Cu and Zn levels also increased to some extent, Sistani et al. (2008) did not consider the Cu and Zn increases environmentally threatening. In contrast, He et al. (2008, 2009b) observed that repeated application of PL during a 20-year period did not profoundly affect P, Ca, and Mg, but did increase K levels in Alabama pasture soils. These reports suggest that PL use must be managed carefully to avoid negative environmental effects.

Schomberg et al. (2009) found for a Cecil soil in the Southern Piedmont that Mehlich-1-extractable nutrients (to a depth of 60 cm) after a 10-year PL application were predominantly in the 0- to 15-cm depth. They found that Mehlich-1-extractable P and Zn increased more than 200%. Accumulation of Mehlich-1-extractable Ca, K, P, and Zn at lower depths was also observed. They concluded, however, that concentrations of Mehlich-1-extractable soil nutrients (P, K, Ca, Mg, manganese [Mn], and Zn) remained below levels of environmental concern. Mehlich-1-extractable nutrients represent only the acid-soluble portion of P and metals in the soil. Environmental risk assessments, on the other hand, are generally based on total rather than extractable concentrations (Franklin et al., 2006; USEPA 1994, 1999) because total concentrations of an element are easier to determine than bioavailable forms that are often related to extractable concentrations. A more accurate assessment of environmental risk would be reflected from the bioavailable form, but more information is needed to relate nutrient pools determined from these different methods.

The change in total P, Zn, Cu, and Mn was evaluated in the same Cecil soil used by Schomberg et al. (2009) from a study of 5 years of cotton followed by 5 years of corn under different combinations of tillage (CT and NT) and fertilizer source (PL and conventional inorganic fertilizer). The objectives were to see how soil total nutrient concentrations were impacted by inputs of nutrients from PL and conventional inorganic fertilizer under the contrasting tillage managements, and if there were definable relationships between extractable and total nutrients useful for identifying critical levels of nutrient accumulation associated with long-term PL use, highlighting potential soil and water environmental risks.

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MATERIALS AND METHODS

Experimental Site and Management

Research conducted from 1995 to 2005 on the water quality facility at the USDA-ARS, J. Phil Campbell, Sr Natural Resource Conservation Center, Watkinsville, GA (83°24'W and 33°54'N), evaluated the effects of tillage and fertilizer source on cotton and corn productivity (Endale et al., 2002; 2008). The research facility is described in detail by Endale et al. (2002; 2008) and Schomberg et al. (2009). Briefly, the facility consists of 12 (10 m × 30 m) tile-drained plots, located on nearly level (<2% slope) Cecil sandy loam soil (fine, kaolinitic, thermic Typic Kanhapludults). Main plot treatments are CT and NT, whereas subplot treatments are conventional fertilizer ([CF] either NH_4NO_3 or $[\text{NH}_4]_2\text{SO}_4$ for N and triple super phosphate for P based on soil test results) and PL. This arrangement results in a factorial combination of treatments: CT-CF, CT-PL, NT-CF, and NT-PL. The experimental design is a randomized complete split-plot with three replications.

From fall 1994 to fall 2000 (cotton phase), the facility was cropped to cotton (cv. Stoneville 474) and a cereal rye (*Secale cereale* L.; cv. Hy Gainer) cover crop. Cotton was fertilized with 67 kg N ha⁻¹ as NH_4NO_3 in the CF treatment, whereas in the PL treatment, starting in spring 1995 (Year 0), an equivalent amount of N was added by applying 4.5 Mg PL ha⁻¹ (fresh weight basis) on the assumption that mineralization of N in PL was 50% during the main cropping season (Ritz and Merka, 2004). In both the CT and NT treatments, the cover crop was chemically killed by applying glyphosate (2.3 L ha⁻¹) 2 to 3 weeks before planting cotton. Beginning in spring 2001 (corn phase), corn (cv. Pioneer 3223) was planted as the summer crop, whereas rye continued to be used as the winter cover crop. Nitrogen fertilization was increased to 168 kg ha⁻¹ based on recommendations for corn and was applied as $(\text{NH}_4)_2\text{SO}_4$ in the CF treatment. The PL treatment received 11.2 Mg PL ha⁻¹, providing an equivalent amount of plant-available N. The N application rate to corn was doubled (to 336 kg N ha⁻¹ in CF and 22.4 Mg PL ha⁻¹ in PL) in the spring of 2003 to investigate hormone concentrations in soil, runoff, and drainage from PL use. Corn N fertilization was reduced to 168 kg ha⁻¹ in 2004 and 2005. The rye cover crop in the PL treatment was fertilized with 3.0, 1.8, and 1.8 Mg PL ha⁻¹ during 2001, 2002, and 2003 and with $(\text{NH}_4)_2\text{SO}_4$ (67 kg N ha⁻¹) in 2004 and 2005 of the corn cropping phase in contrast to the use of commercial fertilizer during the entire cotton phase. Rye in the CF treatment was fertilized with $(\text{NH}_4)_2\text{SO}_4$ (67 kg N ha⁻¹) from 2001 through 2005. Corn and rye residues were shredded with a rotary mower in both the CT and NT treatments but were only incorporated in the CT treatment. No additional PL was added to these fields after the spring of 2005 (Year 10), but CF continued to be used for fall and spring N fertilization as needed.

Sampling and Analysis

Soils samples (0–60 cm) were collected in 1997 (Year 2), 2000 (Year 5), 2006 (Year 11) (where Year X refers to years after PL application that started in spring 1995). In 2005 (Year 10), only 0- to 15-cm depth samples were collected. The 0- to 60-cm samples were collected in the fall with a tractor-mounted hydraulic soil coring device from three locations in each plot. Soil cores (2.5 cm in diameter) were partitioned into 0- to 15-, 15- to 30-, and 30- to 60-cm depth increments and composited. In Year 11, we divided the 0- to 15-cm depth into 0- to 2.5-, 2.5- to 5-, and 5- to 15-cm increments. We used a weighted sum of the shallower increments to compare the Year 11 0- to 15-cm data to that from other years. All samples were dried at 55 °C to

60 °C for 3 to 5 days and kept at room temperature until analyzed. For analysis of total nutrient concentrations, soil samples were digested in concentrated HNO_3 in a CEM MDS-2100 microwave system (Matthews, NC). The resulting solution was analyzed using a TJA Model IRIS 1000 dual-view inductively coupled plasma emission spectrometer for total P and other metals (USEPA, 1986). Soils from 30- to 60-cm depth were not analyzed in this work. Extractable nutrients (Ca, K, Mg, Mn, P, and Zn) were determined in double acid (0.05 M HCl + 0.025 M H_2SO_4) extracts (Mehlich-1) by the Soil, Plant and Water Analysis Laboratory at the University of Georgia, Athens, using an inductively coupled plasma emission spectrograph (UGA, 2002) and reported previously (Schomberg et al., 2009).

Poultry litter used throughout the study originated from the same poultry houses and was brought to the research site and kept under cover for no more than 2 weeks before application. The poultry litter consisted of manure and pine (*Pinus palustris* P. Miller and *Pinus elliottii* Englem.) wood shavings. Variation in numbers of birds between clean outs, moisture content, and diet during the length of the study resulted in variability in nutrient concentrations in the PL. Each batch was analyzed at the Soil, Plant and Water Analysis Laboratory at the University of Georgia, Athens. Total N was determined by the Dumas method (Bremner and Mulvaney, 1982). All other elements were determined using an inductively coupled plasma spectrograph after digestion with nitric and hydrochloric acid (UGA, 2002). Complete details of nutrient inputs from the two fertilizer sources are given in Schomberg et al. (2009).

Statistical Analysis

Data were analyzed as a randomized complete block split-plot experiment using the MIXED procedure of SAS (Little et al., 2000; SAS, 2003). Fixed main effects were taken as tillage, fertilizer, year, depth, and their interactions. Block, block × tillage, and block × tillage × fertilizer were considered random effects (error terms). Year was treated as a repeated factor in the analysis. Means were estimated as least square means and were compared using contrast statements. Plots of extractable P and Zn versus total P and Zn exhibited patterns of abrupt slope change that was evaluated using piecewise regression with the PROC NLIN procedure in SAS to estimate a break point and regression parameters for the two line segments. Unless otherwise indicated, all significant differences are expressed at the level of $P \leq 0.05$. Regression analysis of the change in soil total P levels with soil depth was conducted using Microsoft Excel Data Analysis Tools.

RESULTS AND DISCUSSION

Depth and depth interactions with tillage or fertilizer source effects on total P, Cu, Mn, and Zn were in general significant; therefore, the results are presented by depth. This was also true for the data collected in Year 11 (in the fall of the year after the final PL application) where we analyzed soils at finer depth increments in the top 15 cm.

Total P

0 to 15 cm

During the cotton cropping phase in the first 5 years, total P did not increase with either CF or PL (Fig. 1, a-1). Indeed, the total P levels in CT fields were slightly lower in the Year 5 soil samples than those in the Year 2 samples. During the cotton phase, mean total P concentration ranged from 213 to 310 mg kg⁻¹ (CT-CF to NT-PL). During the corn phase, inputs of P increased from 70 to 292 kg ha⁻¹ year⁻¹ in the PL treatments,

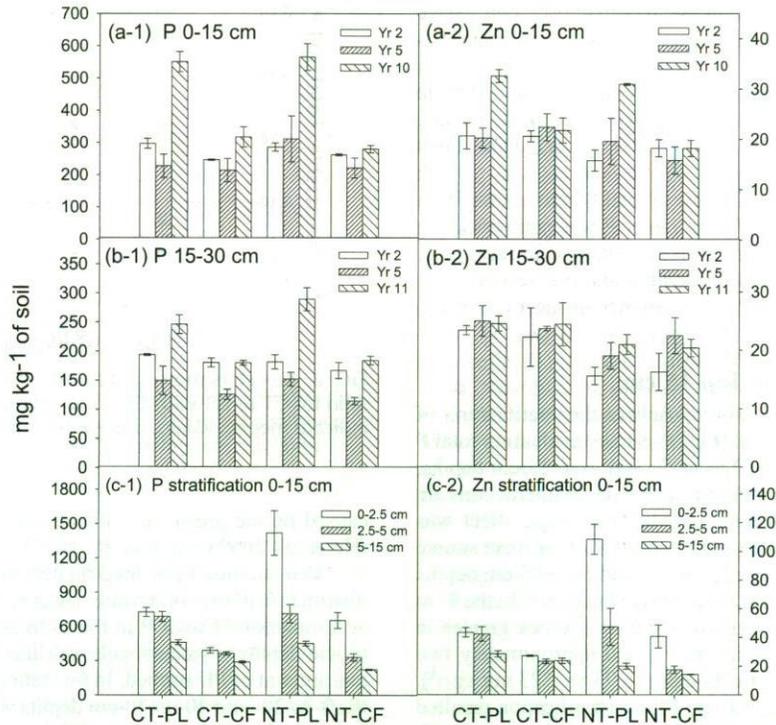


FIG. 1. Total P and Zn concentration in the 0- to 15-cm depth for Years 2, 5, and 10 (a-1 and a-2) and in the 15- to 30-cm depth for Years 2, 5, and 11 (b-1 and b-2) and in the 0- to 2.5-, 2.5- to 5-, and 5- to 15-cm depths for Year 11 (c-1 and c-2) in CT and NT fields with PL or CF application. Data are the average of three field triplicates. Error bars represent the SE.

with a small reduction in the CF treatments (24 and 22 kg ha⁻¹ year⁻¹). With longer repeated fertilizer applications, total P accumulated in the surface soil as shown in Fig. 1, a-1. In the CF fields, total P increased from around 250 mg kg⁻¹ in Year 2 to around 300 mg kg⁻¹ in Year 10 (Fig. 1, a-1), although a lower annual CF application rate was used during the corn phase (Table 1). In the PL treatment during the same period, total P increased from nearly 300 mg kg⁻¹ to more than 550 mg kg⁻¹. Total soil P concentration remained essentially the same in Year 11, 1 year after the last PL application in Year 10. Total P concentration was 1.8 to two times more in NT-PL than in CT-CF in Year 10 and Year 11. There was no change in total P concentration from Year 2 to Year 10 in CT-CF. Tillage management did not impact total P concentration in the top 0- to 15-cm depth. Incorporation of P into the soil through CT was expected to enhance plant P uptake (access to roots) and possibly leaching loss compared with no incorporation as in NT. As it turned out, tillage and the tillage × year interaction effects were not significant ($P > 0.18$), and the three-way interaction among tillage, fertilizer, and year was not significant ($P = 0.1$).

15 to 30 cm

As there were no subsurface soil samples collected at the end of the corn phase (Year 10), the soils collected the following year (Year 11) were used to compare long-term P and metal concentrations in the 15- to 30-cm subsurface soil (Fig. 1, b-1). The response of total P to treatment effects in the 15- to 30-cm depth was similar to that in the 0- to 15-cm depth. Year, fertilizer, and the fertilizer × year interaction significantly influenced total P concentration ($P < 0.001$). Total P concentrations in Year 5 were lower than in Year 2 in all treatments (Fig. 1, b-1). The decrease was more apparent in the two fields with the CF

application. Two years after the start of litter application, total P concentration in the 15- to 30-cm depth varied from 177 to 190 mg kg⁻¹ but decreased slightly to 120 to 151 mg kg⁻¹ in Year 5 (Fig. 1, b-1). The reason for the decrease is not clear, but during this period, there was severe drought and the limited rainfall probably reduced P solubility and translocation from the surface

TABLE 1. Cumulative Amounts of P, Zn, Mn, and Cu Added to the Experimental Fields

Treatment	P	Zn	Mn	Cu
	kg ha ⁻¹			
Year [†] 2				
CT-PL	200	4.1	5.6	3.7
CT-CF	74	0.0	0.0	0.0
NT-PL	200	4.1	5.6	3.7
NT-CF	74	0.0	0.0	0.0
Year 5				
CT-PL	424	7.7	11.5	9.5
CT-CF	148	0.0	0.0	0.0
NT-PL	424	7.7	11.5	9.5
NT-CF	148	0.0	0.0	0.0
Year 10/11				
CT-PL	1722	38.1	47.3	60.8
CT-CF	246	0.0	0.0	0.0
NT-PL	1722	38.1	47.3	60.8
NT-CF	246	0.0	0.0	0.0

[†]Year since start of PL application in spring 1995.

to deeper in the profile. This, along with crop uptake, could have reduced total P in this zone. In Year 11, total P concentrations were again similar to those of Year 2 in the CT-CF treatment but were approximately 1.6 times greater than the Year 2 level for the NT-PL treatment (289 vs. 182 mg kg⁻¹) (Fig. 1, b-1). Tillage, tillage × fertilizer, and tillage × fertilizer × year interactions were not significant for total P ($P > 0.17$).

There were differential changes in total P between the 0- to 15- and 15- to 30-depths. In Year 2, total P was approximately 1.4 times greater in the 0- to 15-cm depth than in the 15- to 30-cm depth in both PL and CF treatments. By Year 11, this had increased to 2.2 and 1.7 times greater in the PL and CF treatments, respectively.

Distribution Within the Top 15 cm

In Year 11, we looked more closely at the stratification of total P in the top 15 cm of soil (Fig. 1, c-1) by evaluating total P distribution in the 0- to 2.5-, 2.5- to 5-, and 5- to 15-cm depths. Significant tillage × depth and fertilizer × depth interactions are apparent from the data in Fig. 1, c-1. The tillage effect was limited to the 0- to 2.5-cm depth, whereas that of fertilizer source was apparent in the 0- to 2.5-, 2.5- to 5-, and 5- to 15-cm depths (Fig. 1, c-1). There was less soil disturbance with NT. In the 0- to 2.5-cm depth, total P was approximately 1.8 times greater in NT than CT (1037 vs. 559 mg kg⁻¹) and approximately two times greater in PL than CF treatments (1,073 vs. 523 mg kg⁻¹). The combined influence of NT and PL on stratification resulted in total P being 3.6, 2.0, and 1.5 times greater compared with CT-CF in the 0- to 2.5-, 2.5- to 5-, and 5- to 15-cm depths, respectively.

Linear and nonlinear regression was performed to compare linear, exponential, logarithmic, and power models as they describe the change of total P with depth (Table 2). Depth was taken as the distance from the soil surface to the center of the 0- to 2.5-, 2.5- to 5-, 5- to 15-, and 15- to 30-cm depth from which the total P data were determined. A power model best described change in total P concentration in NT soil, whereas both linear and exponential models fit the CT data equally. Regardless of which model is best, total P concentration distribution was different in CT compared with NT. For the effects of fertilizer type, the distribution of P indicated slower or less movement from CF than from PL based on the smaller coefficients or constant of X in these equations. For example, the constant was 23.1 and 9.9 for the linear equations of CT-PL and CT-CF, respectively (Table 2). The difference in response between the two nutrient sources could be caused by inorganic P in CF being more susceptible to immobilization by soil components and also

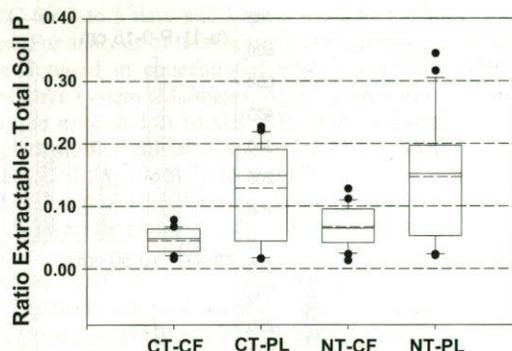


FIG. 2. Box plots showing distribution of extractable to total soil P ratio for CT or NT with CF or PL. Inside boxes and solid lines indicate median values, and dashes indicate mean values.

caused by the greater organic P content and mobility with PL (He et al., 2009a; Hunt et al., 2007).

Few studies have investigated the relationship of total P distribution in the soil profile. He et al. (2009b) reported that the accumulation of total P in the 0- to 20-, 20- to 40-, and 40- to 60-cm depths of pasture soils was linearly related to the cumulative amount of PL applied. In their study, total P accumulation in the 0- to 20- and 20- to 40-cm depths was 545 and 300 mg kg⁻¹, respectively, giving a ratio of 1.8. Using the power model equation determined for NT-PL in Table 2, we estimated that the accumulation of total P from PL in the soil at the 10- and 30-cm soil depths (median depths of the 0- to 20- and 20- to 40-cm layers) was 447 and 247 mg kg⁻¹ soil, respectively. This also gives a ratio of 1.8, similar to the ratio reported by He et al. (2009b), suggesting that our power regression models could be used to predict total P distribution in soils with similar properties such as Cecil after long-term PL application.

In previous work, Schomberg et al. (2009) evaluated the effects of fertilizer source and tillage on extractable nutrients for the same soils and periods used in the current study. Evaluating the relationship between extractable and total nutrients could provide additional insight into management influences on availability of nutrients from the soil reserve. Because of the significant accumulation of P and Zn in these soils, there was special interest in determining how the relationship between the two fractions changed over time. The ratio of extractable to total soil P for the four treatment combinations is presented in Fig. 2. The overall mean ratio was 0.10 ± 0.01 . The effects of fertilizer

TABLE 2. Equations and Coefficients of Determination R^2 for Linear and Nonlinear Regression Between Soil Total P (y; mg kg⁻¹) and Soil Depth (x; cm)[†]

Treatment	Equation and Coefficient of Determination R^2							
	Linear	R^2	Exponential	R^2	Logarithmic	R^2	Power	R^2
CT-PL	$P = 748 - 23.1x$	0.97*	$P = 799e^{-0.052X}$	0.99**	$P = 826 - 169\ln(x)$	0.90*	$P = 925x^{-0.37}$	0.84
CT-CF	$P = 399 - 9.921x$	0.99**	$P = 415e^{-0.037X}$	0.99***	$P = 431 - 71.7\ln(x)$	0.89	$P = 469x^{-0.26}$	0.84
NT-PL	$P = 1116 - 42.4x$	0.65	$P = 1115e^{-0.066X}$	0.84	$P = 1388 - 385\ln(x)$	0.93*	$P = 1551x^{-0.54}$	0.99**
NT-CF	$P = 509 - 17.1x$	0.58	$P = 490e^{-0.05X}$	0.71	$P = 628 - 161\ln(x)$	0.89	$P = 661x^{-0.44}$	0.96*

[†]Soil depth was taken as the distance from the surface to the center of the 0- to 2.5-, 2.5- to 5-, 5- to 15-, and 15- to 30-cm depths.

*, **, and *** represent the statistical significance of R^2 values at $\alpha \leq 0.05$, 0.01, and 0.001, respectively.

P: phosphorous.

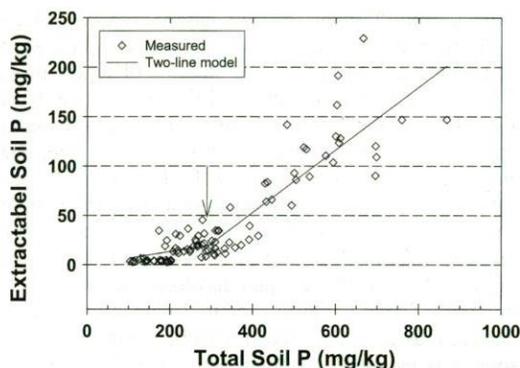


FIG. 3. Measured data and a two-line linear model for extractable soil P versus total P based on data pooled across all treatments. Arrow indicates slope break point. Parameters are shown in Table 3 (Treatment All).

source are apparent, with greater mean values in the PL treatments compared with the CF treatments; however, greater variability of P concentrations in the PL treatments is also apparent from Fig. 2. The greater ratios of extractable to total P in the PL treatments can explain the greater movement of P to the subsoil compared with the CF treatment.

Plots of extractable P versus total P concentration exhibited patterns of abrupt slope change, as illustrated in Fig. 3. Piecewise regression was used to estimate a break point and regression parameters for the two line segments of the various treatment combinations (Table 3). Where piecewise regression estimated a break point, 86% to 97% of the variation in extractable soil P could be explained by variation in total P (Table 3). For data pooled across treatments, the break point was at approximately 295 mg kg⁻¹. Because piecewise regression solutions could not be determined for CF and CT-CF, the potential to compare break points and slopes among various treatments was limited. Some speculative observations can be made: (i) the break point tended to be greater with PL compared

with CF, (ii) break points were smaller with NT compared with CT, and (iii) the rate of change in extractable P was greater with PL than with CF (which would be expected with the greater P inputs). The slope after the break point was approximately seven to eight times that before the break point for PL associated with either CT or NT. He et al. (2009b) found that contents of total P, Mehlich-3-extracted P, and total N were more related to cumulative amounts of PL applied than the years of application or annual application rates alone. Our results indicate that the available P fraction response to cumulative amount applied may be confounded because once a saturation point is reached, availability changes dramatically.

The smaller break point for NT compared with CT probably reflects greater stratification of nutrients in the NT treatment. Stratification could result in saturation of exchange sites in the surface soil and increase extractable P concentration at a lower total P concentration. As the exchange sites (Fe and Al oxides that would tend to sorb P and reduce its availability) became saturated in the surface NT soil, increasing exchangeable P levels became more apparent at lower total P concentrations. Because the CT soil is mixed two times a year, there is a replenishment of sites for sorbing P. In the NT plots, the amount of P needed to saturate the available sorption sites is smaller because there is no soil mixing.

Zn

Concentration of total Zn in the 0- to 15-cm depth was a function of specific combinations of tillage, fertilizer, and year (three-way interaction, *P* = 0.04). The annual application rate of PL and CF during the cotton phase did not lead to a buildup of total Zn (Fig. 1, a-2). This observation agrees with the previous observation that Mehlich-1-extractable nutrients changed very little during the 5 years of cotton production (Schomberg et al., 2009). Much of the increase in total Zn occurred for the PL treatments during the corn phase when inputs of Zn increased from approximately 1.3 kg ha⁻¹ year⁻¹ to 6.8 kg ha⁻¹ year⁻¹ (Table 1; Fig. 1, a-2). Total Zn concentration remained in the narrow range of 21 to 23 mg kg⁻¹ in CT-CF for the 10 year period, whereas in the NT-PL, it increased 1.4 times this amount by Year 10 (31 vs. 21 mg kg⁻¹ for PL and CF treatments, respectively). The Year 11 data indicate that the residual effect of PL continued to be observed for 1 year after the last PL application. Changes in total Zn in the 15- to 30-cm depth were much less dramatic than in the 0- to 15-cm depth (Fig. 1, b-2). There were significant changes over time (*P* = 0.01), but changes caused by fertilizer or tillage (and their interactions) were too

TABLE 3. Parameters and Statistics for One- and Two-Line Regression Between Extractable P (y) and Total P (x)[†]

Treatment	n	Regression Parameter [‡]			Statistics		
		Intercept		c	Adj. R ²	P	
		mg kg ⁻¹	b1	b2			
All	96	0	295.2	0.067	0.317	0.893	<0.001
CT	48	0	307.9	0.055	0.332	0.950	<0.001
NT	48	0	233.7	0.055	0.291	0.863	<0.001
CF	48	-4.92		0.078		0.447	<0.001
PL	48	0	187.8	0.054	0.263	0.912	<0.001
CT-CF	24	-3.37		0.059		0.527	<0.001
CT-PL	24	0	173.4	0.030	0.257	0.976	<0.001
NT-CF	24	0	126.5	0.033	0.114	0.902	<0.001
NT-PL	24	0	151.0	0.035	0.260	0.866	<0.001

[†]Based on extractable and total P data for 0- to 15-cm (Years 2, 5, 10, and 11) and 15- to 30-cm (Years 2, 5, and 10) depths, and for 0- to 2.5-, 2.5- to 5.0-, and 5- to 15-cm depths in Year 11.

[‡]c breakpoint where slope changes from b1 to b2; b1 slope of regression line for x ≤ c; and b2 slope of regression line for x > c. Parameters for CF and CT-CF are for one-line regression only.

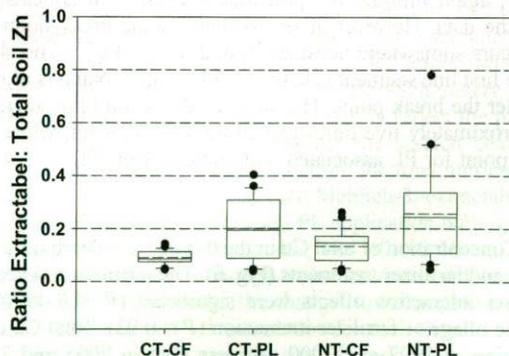


FIG. 4. Box plots showing distribution of extractable to total soil Zn ratio for CT or NT with CF or PL. Inside boxes and solid lines indicate median values, and dashes indicate mean values.

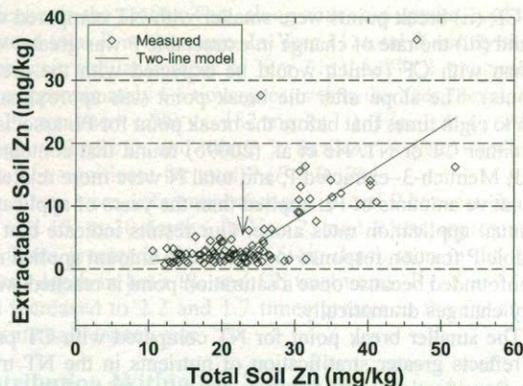


FIG. 5. Measured data and a two-line linear model for extractable soil Zn versus total Zn based on data pooled across all treatments. Arrow indicates slope break point. Parameters are shown in Table 4 (Treatment All).

small to be significant. Total Zn concentrations in CT-CF and CT-PL were essentially similar in the 15- to 30-cm depth as they were in NT-CF and NT-PL.

Distribution of total Zn in the top 15 cm in Year 11 was influenced by specific combinations of tillage and fertilizer ($P < 0.007$; Fig. 1, c-2). The tillage effect was most obvious in the 0- to 2.5-cm depth, whereas fertilizer source effects were obvious in the 0- to 2.5- and 2.5- to 5-cm depths (Fig. 1, c-2). In the 0- to 2.5-cm depth, total Zn was approximately two times more in NT-PL than in the corresponding treatments. Compared with CT-CF, total Zn in NT-PL was approximately four and two times more in the 0- to 2.5- and 2.5- to 5-cm depths, respectively. Total Zn concentrations were similar between CT-CF and NT-PL in the 5- to 15-cm depth.

Ratio of extractable to total soil Zn for the four treatment combinations is presented in Fig. 4. The overall mean ratio was 0.17 ± 0.01 . The ratio was greater in PL treatments than CF treatments. Variability was also greater with PL compared with CF.

As in the case of P, plots of extractable Zn versus total Zn concentration (milligrams per kilogram) exhibited patterns of abrupt slope change (Fig. 5). Piecewise regression was used to define the break point for the slope and establish the parameters for the two lines (Table 4). Where piecewise regression estimated a break point, 73% to 91% of the variation in extractable Zn could be explained by variation in total Zn. Piecewise regression solutions could not be determined for CF, CT-CF, and NT-CF, again limiting the potential to draw firm conclusions from the data. However, it seems that a slope break point for Zn occurs somewhere between 20 and 22 mg kg^{-1} . The slope for the first line segment is between 0.11 and 0.16 and is around 0.7 after the break point. The slope of the second line segment is approximately five times that of the line segment before the break point for PL associated with either CT or NT.

Cu

Concentration of total Cu in the 0- to 15-cm depth reflected tillage and fertilizer treatments (Fig. 6). The fertilizer and year \times fertilizer interaction effects were significant ($P < 0.0001$) as was the tillage \times fertilizer interaction ($P = 0.03$). Total Cu concentration in 1997 and 2000 was less than in 2005 and 2006 ($P < 0.0001$). Total Cu concentration remained in the 5- to 8- mg kg^{-1} range during the cotton phase in CT and NT but rose to approximately 14 mg kg^{-1} at the end of Year 10 (Fig. 6, a-1).

Inputs of Cu from PL increased from approximately 1.6 kg ha^{-1} year $^{-1}$ during the cotton phase to approximately 11.5 kg ha^{-1} year $^{-1}$ during the corn phase. The absence of inputs of Cu in the CF treatment is reflected in the difference in total Cu concentration between the PL and CF treatments (22 vs. 6 mg kg^{-1} , respectively). The concentration of Cu was approximately 3.4 times greater in NT-PL than in CT-CF at Year 10. In Year 11, total Cu continued to remain high in the 0- to 15-cm depth of the PL treatment (similar to 2005). In the 15- to 30-cm depth, the concentration of total Cu showed a significant response only to year ($P = 0.002$). Tillage and fertilizer did not affect the concentration of total Cu at the 15- to 30-cm depth (no significant interactions or main effects). At this depth, total Cu concentration was essentially similar between CT-CF and CT-PL throughout the study as it was between NT-CF and NT-PL (Fig. 6, b-1).

When evaluated more closely in Year 11, there was significant stratification of Cu in the 0- to 15-cm depth. Differences in concentrations at the three depths were a function of fertilizer and tillage ($P = 0.05$). Tillage differences were most apparent in the 0- to 2.5-cm depth, with total Cu concentration being approximately two times greater in the NT than CT treatment (Fig. 6, c-1). Fertilizer source effects were apparent in both the 0- to 2.5- and 2.5- to 5-cm depths, with PL having approximately four times more total Cu than CF in both depths. Compared with CT-CF, total Cu in NT-PL was approximately 8.5, 3.3, and 1.8 times more in the 0- to 2.5-, 2.5- to 5-, and 5- to 15-cm depths, respectively. The pattern of slope change for extractable versus total Cu concentration was not evaluated because extractable Cu data were not available.

Mn

In the 0- to 15-cm depth, the concentration of Mn was only influenced by fertilizer source and time (fertilizer \times year, $P = 0.01$). Variability was high within and among treatments. Total Mn concentration ranged from 150 to 160 mg kg^{-1} for CT and 100 to 120 mg kg^{-1} in NT (Fig. 6, a-2). For CF and PL

TABLE 4. Parameters and Statistics for One- and Two-Line Regression Between Extractable Zn (y) and Total Zn (x)[†]

Treatment	n	Regression parameter [‡]			Statistics		
		Intercept	c		Adj. R ²	P	
		mg kg ⁻¹	b1	b2			
All	96	0	23.2	0.123	0.738	0.731	<0.001
CT	48	0	23.7	0.096	0.611	0.877	<0.001
NT	48	0	20.9	0.150	0.860	0.735	<0.001
CF	48	2.0		0.008		-0.020	0.791
PL	48	0	21.3	0.149	0.669	0.758	<0.001
CT-CF	24	1.3		0.033		0.002	0.316
CT-PL	24	0	22.2	0.113	0.569	0.910	<0.001
NT-CF	24	1.7		0.033		-0.032	0.602
NT-PL	24	0	18.3	0.162	0.784	0.752	<0.001

[†]Based on extractable and total P data for 0- to 15-cm (Years 2, 5, 10, and 11) and 15- to 30-cm (Years 2, 5, and 10) depths, and for 0- to 2.5-, 2.5- to 5.0-, and 5- to 15-cm depths in Year 11.

[‡]c breakpoint where slope changes from b1 to b2; b1 slope of regression line for $x \leq c$; and b2 slope of regression line for $x > c$. Parameters for CF and CT-CF and NT-CF are for one-line regression only.

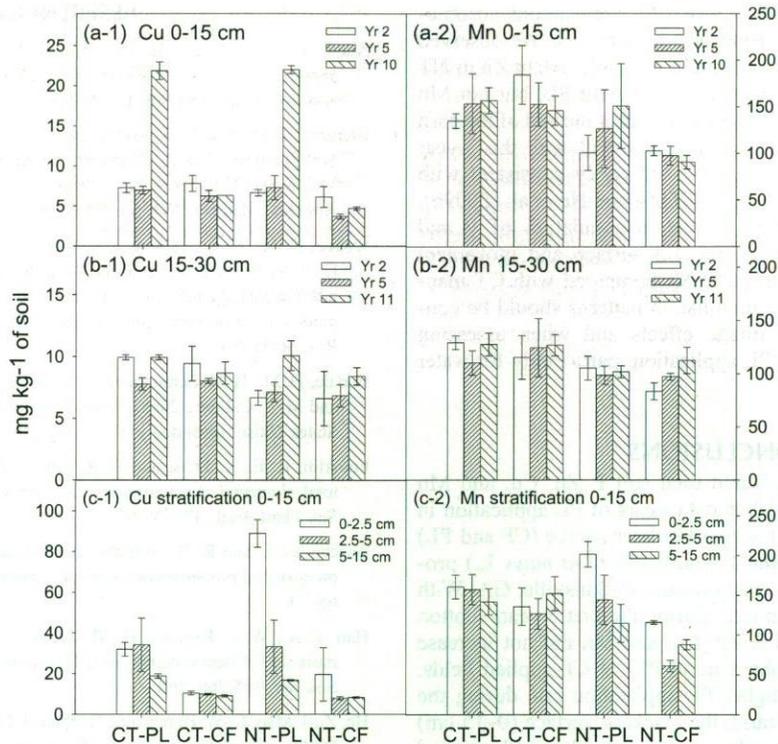


FIG. 6. Total Cu and Mn concentration in the 0- to 15-cm depth for Years 2, 5, and 10 (a-1 and a-2) and in the 15- to 30-cm depth for Years 2, 5, and 11 (b-1 and b-2), and in the 0- to 2.5-, 2.5- to 5-, and 5- to 15-cm depths for Year 11 (c-1 and c-2) in CT and NT fields with PL or CF application. Data are the average of three field triplicates. Error bars represent the SE.

treatments, concentrations were generally in the range 115 to 155 mg kg⁻¹, with no difference between treatments. No residual effect caused by PL was observed in 2006. As was the case for P, Zn, and Cu, input of Mn from PL was greater during the corn phase (8.0 kg ha⁻¹ year⁻¹) than during the cotton phase (1.9 kg ha⁻¹ year⁻¹), but because of the high degree of variability, accumulation of Mn in the soil was not detected. At the 15- to 30-cm depth, none of the treatments or their interactions had any significant effect on total Mn concentration (Fig. 6, b-2). Concentrations ranged from 115 to 130 mg kg⁻¹ for CT and 90 to 110 mg kg⁻¹ in NT, and 95 to 120 mg kg⁻¹ across CF and PL treatments. Data from the 0- to 2.5-, 2.5- to 5-, and 5- to 15-cm depths in 2006 indicated that tillage and fertilizer interactions significantly influenced Mn distribution within the top 15 cm (0.001 < *P* < 0.03, Fig. 6, c-2). The nutrient source effect was most apparent in the 0- to 2.5- and 2.5- to 5-cm depths, in which total Mn was 1.4 to 1.6 times greater in the PL than CF treatments. Compared with CT-CF, total Mn in NT-PL was approximately 1.5 times greater in the 0- to 2.5-cm depth, was not different in the 2.5- to 5-cm depth, and differences were reversed in the 5- to 15-cm depth (Fig. 6, c-2). Plots of extractable versus total Mn concentration did not exhibit patterns of abrupt slope change as was the case for P and Zn.

Accumulations of phosphorus and heavy metals in soils from application of animal manures are of environmental concern for soil and water quality (Kingery et al., 1994; USEPA, 2002; Sharpley et al., 2003; Jackson et al., 2003). Once the limited P fixing capacity of a soil becomes saturated, there is an increased potential for P transfer to surface waters in storm runoff and/or drainage (Sharpley et al., 2003). In our study of nutrient

accumulation after 2, 5, 10, and 11 years of PL application, there was a clear point of saturation that was reached with some nutrients. With low PL and CF application rates during the first 5 years (cotton phase), the levels of total P, Zn, Cu, and Mn showed little indication of excess application either in the PL or CF treatments. With two to four times higher PL application during the corn phase (the last 5 years), the levels of surface (0-15 cm) soil P, Cu, and Zn, but not Mn, rose markedly. Further analysis of the relationship between extractable versus total P and Zn revealed a threshold value beyond which extractable P and Zn increased at more than double the initial rate. In other words, once accumulation of P and Zn exceeded the soil buffer capacity, nutrient availability was significantly altered. A combination of application rate and long-term application were apparently key factors influencing the response to PL in this soil.

Other authors have reported significant accumulation of extractable nutrients from high rates of PL application (e.g., Kingery et al., 1994; Sistani et al., 2008; Schomberg et al., 2009). Our results for total nutrients closely reflect the patterns observed for extractable nutrients, but the sudden change in available nutrients was not obvious from the total nutrient data. He et al. (2009b) reported that both Mehlich-3-extractable and total P from repeated (5-20 years) PL application built up in all three layers (0- to 20-, 20- to 40-, and 40- to 60-cm) of an Alabama pasture soil, although contents decreased with soil depth. Similar to our results, the changes in extractable nutrients were not reflected in a 1:1 association for their data.

Adeli et al. (2007) reported that PL applications increased total trace metals in Mississippi soils after 3 years, but downward movement of Cu and Zn was limited to the top 15 cm of soil. On the other hand, Han et al. (2000) found that a portion of

total Cu and Zn supplied by 23-year PL applications accumulated in the 20- to 40-cm depth of a pasture soil. We observed that from PL application, total P, and to a little extent Zn in NT with PL, and even less extent Cu in NT with PL, but not Mn moved downward to the 15- to 30-cm layer at the end of the corn phase (Year 10), which was partly consistent with the 3-year observation by Adeli et al. (2007) and partly consistent with longer observations by Han et al. (2000) and He et al. (2009b). Regression analysis showed greater accumulation of P and some heavy metals close to the soil surface and movement deeper into the soil profile under NT compared with CT management. These different accumulation patterns should be considered when evaluating tillage effects and when assessing environmental impacts of PL application, particularly for water quality considerations.

CONCLUSIONS

In this work, we measured total soil P, Zn, Cu, and Mn concentrations after 2, 5, 10, and 11 years of PL application in a two tillage (CT and NT) × two fertilizer source (CF and PL) cotton (*Gossypium hirsutum* L.) and corn (*Zea mays* L.) production experiment on a Cecil soil near Watkinsville, GA. With low PL and CF application rates during the first 5 years (cotton phase), the levels of total P, Zn, Cu, and Mn did not increase at the end of the cotton phase in all PL- or CF-applied fields. With two to four times higher PL application rate during the cotton phase (the last 5 years), the levels of surface (0–15 cm) soil P, Cu, and Zn, but not Mn, rose markedly in PL-applied fields at the end of the corn phase. These results indicated that application rate is a key factor influencing the buildup of residual P and heavy metals from PL in soils. The P and Cu from repeated PL application moved downward to deeper soil layers. Regression analysis indicated that the change in total P by depth followed a linear or exponential relationship in CT, whereas it followed a power relationship in NT. This model indicates greater accumulation of P close to the soil surface and movement deeper into the soil profile under NT compared with CT management. These different accumulation patterns should be considered when evaluating tillage effects and when assessing environmental impacts of PL application particularly for water quality considerations.

Further analysis of the relationship between extractable versus total P and Zn revealed a threshold value beyond which extractable P and Zn increased at more than double the initial rate. In other words, once accumulation of P and Zn exceeded the soil buffer capacity, nutrient availability was significantly altered. The data for Mn did not show such a relationship, and there were no extractable Cu data to evaluate the relationship for Cu. Therefore, close monitoring of soil nutrients is essential when using PL to avoid environmental risks. Therefore, close monitoring of soil nutrients is essential when using PL to avoid possible environmental risks.

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