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Soil Organic Carbon, Total Nitrogen, and Soil pH, in a Long-Term Continuous Winter Wheat (*Triticum Aestivum* L.) Experiment

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ABSTRACT

Continuous use of organic and inorganic fertilizers influences soil physical and chemical properties. Stored samples from the last 24 years of the Magruder Plots were analyzed to document changes in soil organic carbon (SOC), total nitrogen (TN) and soil pH. Since 1947, the same six treatments have been evaluated. Treatments included the use of cattle manure, inorganic nitrogen (N), phosphorus (P), potassium (K), and lime (L). Each year, a composite surface soil sample (0–15 cm) was taken in each plot, air-dried at ambient temperature, ground to pass a 2 mm sieve, and stored at room temperature, 25°C. Averaged over 24 years' the manure plots resulted in the highest SOC and TN. Manure application maintained SOC at 0.92 and adequate soil pH (>6.0). The use of commercial fertilizers lowered soil pH over time but had higher yields compared to the manure plot.

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Introduction

Long-term experiments are essential to understand the effects of cropping systems, tillage, manuring, and fertilization practices on soil physical and chemical properties. These studies showed that long-term crop production could be sustained and improved when paying attention to recognized soil fertility practices that may employ legumes and/or the use of manures (Mitchell et al. 1991). Long-term experiments also reveal the effects of soil and weather processes on crop production and health of the ecosystem (Girma et al. 2007). Furthermore, these experiments are required for observing changes such as soil organic matter (SOM) (Girma et al. 2007; Ladha et al. 2003) and total soil nitrogen (TN) (Bhandari et al. 2002) which may only be seen over long periods. Reeves (1997) reported values for soil organic carbon (SOC) because of its impact on physical, chemical and biological factors, and associated impact on soil quality.

Soil organic matter is an important factor for soil quality and productivity (Cannell and Hawes 1994; Larson and Pierce 1991). The quantity and quality of SOC affects soil nitrogen (N) (Hart et al. 1994) and N retention in ecosystems (Aber et al., 1998). Adequate supply of nutrients is necessary to achieve optimum productivity for any cropping system (Havlin et al. 2014). Plants are excellent drains, and in order to guarantee good crop development, fertilizers need to be applied to the soil. However, fertilizer application is susceptible to loss because soils are a complex system with physical, mechanical, chemical, and biological reactions occurring at the same time (Colbert 2004). As such, with global nitrogen use efficiency (NUE) of 33% (Raun and Johnson 1999) and phosphorus use efficiency (PUE) of 16% (Dhillon et al. 2017) in cereal crops it is necessary to promote efficient use, understand the impact of fertilizers on soils and avoid environmental contamination by losses (Finck 1982).

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Khan et al. (2007) and Mulvaney, Khan, and Ellsworth (2009) observed that high rates of N fertilizers decreased the C:N ratio promoting decomposition of crop residues and SOM. This further leads to releasing carbon dioxide (CO_2) to the atmosphere, and decreasing the storage capacity of nutrients and water. As a result, more fertilizer was required to increase yields due to a decline in productivity. However, Powlson et al. (2010) argued that synthetic fertilizer is the reason for decreased carbon (C) content. They claimed previous application of manure as the cause of soil oxidation and C loss. Furthermore, they observed a decline in C concentration in the soil even after 100-year cessation of manure application. Havlin et al. (1990) affirmed that a decrease in SOM could be overcome by fertilizer application due to increased biomass that was returned to the soil.

Haynes and Naidu (1998) stated that manure application resulted in increased SOM. Johnston (1986) noted an exponential SOC increase with annual application of 35 tons ha^{-1} of farmyard manure (FYM) at Rothamsted, England since 1843. The manure plot had three times the amount of SOC compared to the unfertilized plot. In addition, the type of manure influences the decomposition rate. In a review Haynes and Naidu (1998), they stated that composted manure promoted higher amounts of SOC compared to fresh manure. While fresh materials still lose C throughout decomposition, composted materials were more stable and had lower C losses. In three continuous winter wheat system, Aula et al. (2016) observed increased SOC when annual N rates were greater than 90 kg ha^{-1}.

Most of the synthetic fertilizers are soluble and susceptible to N loss resulting in lower N content in the soil (Chen and Avnimelech 1986). Furthermore, N fertilizers decrease the C:N ratio leading to SOM decomposition (Khan et al. 2007; Mulvaney, Khan, and Ellsworth 2009). Additionally, animal manure is a great reservoir of essential nutrients such as C, N, sulfur (S), and phosphorus (P). However, the availability of these elements vary widely (Bohn, McNeal, and O'Connor 2001). Havlin et al. (2014), noted 1–6% of N in manure where 50–75% was organic N, and the remaining part (20–50%) was ammonium. In addition, Sylvia et al. (1998) stated that a narrow C:N ratio of 18 in cattle manure resulted in faster decay. Brady and Weil (2008) reported 35% of the N in manure was utilized in the first year, 15% in second year, 6% during the third year, and 2% during the fourth year. In another study Chae and Tabatabai (1986) noted 13–67% of animal manure was mineralized in 26 weeks, showing that mineralization was followed by a lag period, rapid rate of increase, and finally a slow rate of N release. Furthermore, Beauchamp, Kidd, and Thurtell (1982) stated that 24–33% of the N in manure was lost by volatilization in the first 6 days after application because ammonium can be converted into ammonia under these conditions. However, the prolonged residual N was obtained by large amounts of manure applied for several years (Power and Papendick 1985). Furthermore, lime application increased microbial biomass resulting in increased mineralization of organic N (Haynes and Naidu 1998).

Havlin et al. (2014) stated that the potential acidification of urea and ammonium nitrate fertilizers is equivalent. Both release one mole of hydrogen when they react in the soil. Thus decreasing soil pH at the same level. Schroder et al. (2011) concluded that the decrease in soil pH was not related to N sources, but it was due to high rates of N fertilizer application to the soil for long periods. Any source of organic material reduces soil pH. With increasing N fertilization, soil pH decreased (Aula et al., 2016). High levels of N promote ammonium oxidation, resulting in increased nitrate and hydrogen in the soil (Havlin et al. 2014; Sylvia et al. 1998). In addition, depending on the soil pH, organic acids and carbonic acids (weak acids) can dissociate releasing more hydrogen to soil solution. Thus, both the mineralization and the dissociation of weak acids have the capability to acidify soils (Schaetzl and Thompson 2015). However, Eghball (1999) mentioned that beef cattle manure contained a significant amount of calcium carbonate, which neutralizes H, especially coming from N-based recommendations. Furthermore, to increase soil pH, an alkaline material is required with carbonates, hydroxides, or silicates, which can react with hydrogen (Brady and Weil 2008).

The objective of this study was to document the relationship between SOC, TN, and soil pH over 24 years in a historic long-term experiment.

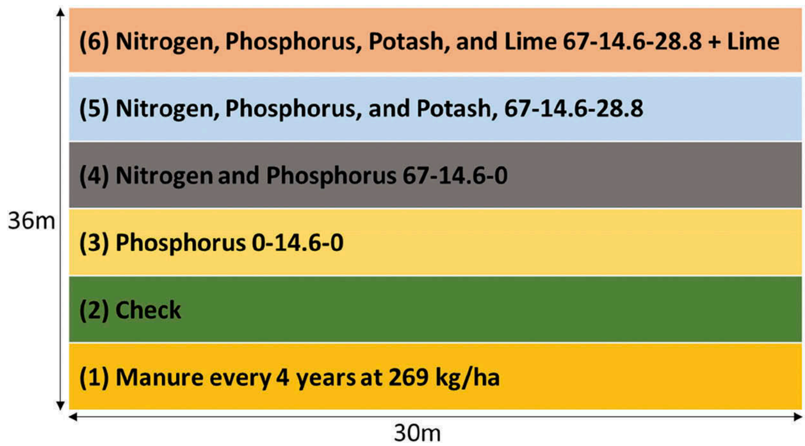

Figure 1. Treatment structure in the Magruder plots, 1947-present.

Material and methods

The Magruder plots used in this study are further described by Girma et al. (2007) and Boman et al. (1996). Stored samples from the last 24 years of the Magruder Plots were analyzed for SOC, TN and soil pH. Currently, this experiment is composed of six-unreplicated plots. It contains four plots receiving synthetic sources of fertilizers, one receiving manure, and one unfertilized check (Figure 1). In the first plot, cattle manure was applied every four years. Manure application was determined based on nitrogen content. Thus, 269 kg N ha^{-1} was applied in 1991, 1995, 1999, 2003, 2007, 2011, and 2015. The second plot is the check plot. It has been unfertilized since the start of the study in 1892. The third plot is the P plot. This plot has received 14.6 kg P ha^{-1} each year as triple superphosphate. The fourth plot is the NP plot. This plot has received 67 kg N ha^{-1} and 14.6 kg P ha^{-1} each year. In 1990, the source of N was ammonium nitrate, and then in 2003, the source was changed to urea. The fifth plot is the NPK plot. This plot has received 67 kg N ha^{-1}, 14.6 kg P ha^{-1}, and 28.8 kg K ha^{-1} per year. The source of P was triple superphosphate and the source of K was muriate of potash (MOP). The sixth plot is the NPKL plot. This plot is similar to the NPK plot; however, lime was applied when soil pH was found to be below 5.5. From 1990 to 2015, 4.9 Mg ha^{-1} of limestone was applied and this took place on September 30, 2009.

The Magruder Plots are located at the Agronomy Research Station EFAW (latitude: 36, 7.1844, N Longitude: 97, 5.3190, W) on a Kirkland silt loam 0 to 2 percent slope (fine, mixed, thermic Udertic Paleustolls). All plots were managed under dryland and have been conventionally tilled (chisel) since 1947. Winter wheat has been planted in the fall with a row spacing of 19.1cm and 67 kg seed ha^{-1}. Plot size for all six treatments is 6 by 30 meters. For each of the 24 years, a composite surface soil sample (0 to 15 cm) was taken from each plot, air-dried at ambient temperature, ground to pass a 2 mm sieve, and stored at room temperature. Soil organic carbon and TN were determined using the dry combustion procedure described by Schepers, Francis, and Thompson (1989). Analysis of variance (ANOVA) was performed using years as replications and Duncan's test was used to compare means over years using SAS 9.4 (SAS Institute, Cary, NC). In addition, a linear regression was performed as a function of time in order to understand dependent variable trends.

Results and discussion

In order to better understand the variables TN, OC, C:N ratio, soil pH, and grain yield, measurements over years were monitored. Thus, a set of tables were generated containing the regression coefficient, mean test (Duncan test), and analysis of variance (ANOVA) for each treatment (Manure, check, P, NP, NPK, NPKL plot) as a function of time. Furthermore, a table containing regression coefficients and means of each variable were generated to examine the effects of lime application in the soil (Tables 1–6). Variability was encountered in all plots, partly due to a continuously changing/ unpredictable environment from year to year that was documented in long-term trials throughout the Midwest (Dhital and Raun 2016).

Manure plot

According to Bohn, McNeal, and O'Connor (2001), animally applied manure is a source of C and N. Since manure was applied to the soil every four years, it was also expected to have a four-year-cycle of increases and decreases in TN and OC after manure application. Total N in the soil ranged from 0.79 g/kg to 1.3 g/kg with a mean of 1.09 g/kg (Table 2). The mean test showed that the manure plot had the highest TN mean among all the other treatments (Table 2). Total N followed a four-year-cycle where the highest peaks were around 1.2 g/kg after one year of manure application and the lowest peaks; four years after manure had been applied.

Table 1. Treatment structure with source of fertilizer and rate, the Magruder Plots, OK 1991–2015.

K Rate	K Rate	K Rate	K Rate kg ha^{-1}	K Rate	K Rate kg ha^{-1}	K Rate	K Rate kg ha^{-1}
1991–2003	1	Manure‡	269	-	0	-	0
	2	-	0	-	0	-	0
	3	-	0	TSP (46-0-0)	14.6	Potash (60-0-0)	0
	4	NH_4NO_3 (33.5-0-0)	67	TSP (46-0-0)	14.6	Potash (60-0-0)	0
	5	NH_4NO_3 (33.5-0-0)	67	TSP (46-0-0)	14.6	Potash (60-0-0)	28.8
	6†	NH_4NO_3 (33.5-0-0)	67	TSP (46-0-0)	14.6	Potash (60-0-0)	28.8
2003–2016	1	Manure‡	269	-	0	-	0
	2	-	0	-	0	-	0
	3	-	0	TSP (46-0-0)	14.6	Potash (60-0-0)	0
	4	Urea (46-0-0)	67	TSP (46-0-0)	14.6	Potash (60-0-0)	0
	5	Urea (46-0-0)	67	TSP (46-0-0)	14.6	Potash (60-0-0)	28.8
	6†	Urea (46-0-0)	67	TSP (46-0-0)	14.6	Potash (60-0-0)	28.8

‡ Manure applied in 1991, 1995, 1999, 2003, 2007, and 2015.
† Lime was applied in 2009.

Table 2. Linear regression analysis between treatments for Total Nitrogen (TN), testing the intercept = 0 and slope = 1 (PR>t, 0.05) and ANOVA evaluating treatments using years as replications, Duncans mean separation procedure (PR>F, 0.05) 1990–2015, Stillwater, OK.

Standard deviation	Standard deviation	Standard deviation	Standard deviation	Standard deviation	Standard deviation	Standard deviation	Standard deviation
Manure Plot	14	Intercept = 0 Slope = 1	y = 0.0008x- 0.4949	0.0031	0.79–1.30	1.0964a	0.1240
Check Plot	15	Intercept = 0 Slope = 1	y = −0.0056x +12.1107	0.2925	0.77–1.08	0.904c	0.0943
P Plot	15	Intercept = 0 Slope = 1	y = −0.0009x +2.7598	0.0102	0.79–1.11	0.9273c	0.0827
NP Plot	15	Intercept = 0 Slope = 1	y = 0.0031x −5.2590	0.0774	0.91–1.23	1.04b	0.1039
NPK Plot	15	Intercept = 0 Slope = 1	y = 0.0003x +0.3886	0.0009	0.88–1.20	1.0386b	0.0972
NPKL Plot	14	Intercept = 0 Slope = 1	y = 0.0017x −2.4270	0.0182	0.92–1.33	1.0542ab	0.1200
Source	DF	Type III SS	Mean square			F Value	PR>|F|
Treatment	5	0.4594	0.0918			21.4	<.0001
Year	14	0.6016	0.0429			10.0	<.0001

† Means followed by the same letter are not significantly different.

Table 3. Linear regression analysis between treatments for Soil Organic Carbon (SOC), testing the intercept = 0 and slope = 1 (PR > t,0.05) and ANOVA evaluating treatments using years as replications, Duncans mean separation procedure (PR > F,0.05) 1990–2015, Stillwater, OK.

Standard deviation	Standard deviation	Standard deviation	Standard deviation	Standard deviation	Standard deviation	Standard deviation	Standard deviation
Manure Plot	14	Intercept = 0 Slope = 1	y = 0.0060x −2.7442	0.0036	7.40–10.65	9.3128a	0.8765
Check Plot	15	Intercept = 0 Slope = 1	y = −0.0556x +118.1658	0.477	6.11–8.84	6.7506d	0.7320
P Plot	15	Intercept = 0 Slope = 1	y = −0.0494x +106.1994	0.3214	6.25–9.40	7.1806c	0.7924
NP Plot	15	Intercept = 0 Slope = 1	y = 0.0086x −8.9663	0.0153	7.44–9.60	8.3013b	0.6320
NPK Plot	15	Intercept = 0 Slope = 1	y = −0.0293x +67.2050	0.2029	7.57–10.20	8.5433b	0.5892
NPKL Plot	14	Intercept = 0 Slope = 1	y = −0.0187x +46.6313	0.0406	7.75–10.55	9.3123a	0.8661
Source	DF	Type III SS	Mean square			F Value	PR>|F|
Treatment	5	80.9850	16.1970			47.9100	<.0001
Year	14	23.4838	1.6774			4.9600	<.0001

† Means followed by the same letter are not significantly different.

Table 4. Linear regression analysis between treatments for the C:N ratio, testing the intercept = 0 and slope = 1 (PR>t,0.05) and ANOVA evaluating treatments using years as replications, Duncans mean separation procedure (PR>F,0.05) 1990–2015, Stillwater, OK.

Treatment	n	Test variable	Estimate	r^2	Range	Mean†	Standard deviation
Manure Plot	14	Intercept = 0 Slope = 1	y = −0.0031x+14.8129	0.0015	7.64–10.13	8.5364ab	0.7045
Check Plot	15	Intercept = 0 Slope = 1	y = −0.0139x+35.3739	0.041	6.62–8.81	7.4960e	0.6253
P Plot	15	Intercept = 0 Slope = 1	y = −0.0456x+99.1241	0.1849	6.74–10.05	7.782de	0.9640
NP Plot	15	Intercept = 0 Slope = 1	y = −0.0176x+43.2727	0.0995	7.37–9.04	8.0193cd	0.5077
NPK Plot	15	Intercept = 0 Slope = 1	y = −0.0311x+70.6830	0.1638	7.37–9.74	8.2673bc	0.7004
NPKL Plot	14	Intercept = 0 Slope = 1	y = −0.0314x+71.7109	0.0406	7.09–10.15	8.7043a	0.8534
Source	DF	Type III SS	Mean Square			F Value	PR>|F|
Treatment	5	14.5255	2.9051			13.1600	<.0001
Year	14	29.9025	2.1358			9.6800	<.0001

† Means followed by the same letter are not significantly different.

Soil organic carbon did not always follow the four-year pattern. Soil organic C fluctuated between 7.40 g/kg and 10.65 g/kg with a mean of 9.31 g/kg (Table 3). In the manure plot, 2.03% OM was maintained by 269 kg/ha. Haynes and Naidu (1998); Johnston (1986); Bohn, McNeal, and O'Connor (2001) stated that manure application can increase SOC; however, as mentioned by Haynes and Naidu (1998), fresh manure is more susceptible to C loss than composted materials. Also, the decomposition rate increases in proportion to manure application. Therefore, even though the application of manure can increase SOC, it is difficult to achieve the same high levels before cultivation. However, manure application and the NPKL plot had the highest SOC mean among all four treatments (Table 3).

The C:N ratio also followed a four-year-cycle; however, it presented the lowest peaks after one year of application while the highest peaks were four years after manure had been applied. The C:N ratio mean was 8.54, and ranged from 7.64 to 10.13 (Table 4). This is consistent with work by Havlin et al. (2014) because this ratio is an indication of N mineralization. Thus, after organic fertilizer (manure) was applied, N became available in the soil and it decreased the C:N ratio promoting N mineralization. During the mineralization process, N was depleted, thus increasing the C:N ratio.

Table 5. Linear regression analysis between treatments for soil pH, testing the intercept = 0 and slope = 1 (PR>t,0.05) and ANOVA evaluating treatments using years as replications, Duncans mean separation procedure (PR>F,0.05) 1990–2015, Stillwater, OK.

Treatment	n	Test variable	Estimate	r^2	Range	Mean†	Standard deviation
Manure Plot	14	Intercept = 0 Slope = 1	y = −0.0181x+42.6790	0.1537	5.34–7.20	6.4892a	0.4003
Check Plot	15	Intercept = 0 Slope = 1	y = −0.0170x+39.8887	0.2664	5.52–6.47	5.822b	0.2995
P Plot	15	Intercept = 0 Slope = 1	y = −0.0173x+40.2083	0.4434	5.34–6.18	5.5846c	0.236
NP Plot	15	Intercept = 0 Slope = 1	y = −0.0173x+39.8705	0.4259	4.80–5.52	5.1326d	0.2416
NPK Plot	15	Intercept = 0 Slope = 1	y = −0.0297x+64.6254	0.6326	4.63–5.78	5.058d	0.3399
NPKL Plot	14	Intercept = 0 Slope = 1	y = −0.0238x+53.2535	0.1919	4.74–6.58	5.4864c	0.5073
Source	DF	Type III SS	Mean Square			F value	PR>|F|
Treatment	5	20.3067	4.0613			58.1600	<.0001
Year	14	5.1531	0.3680			5.2700	<.0001

† Means followed by the same letter are not significantly different.

Table 6. Linear regression analysis between treatments for grain yield, testing the intercept = 0 and slope = 1 (PR > t,0.05) and ANOVA evaluating treatments using years as replications, Duncans mean separation procedure (PR > F,0.05) 1990–2015, Stillwater, OK.

Treatment	n	Test variable	Estimate	r^2	Range Mg ha^{-1}	Mean†	Standard deviation
Manure Plot	14	Intercept = 0 Slope = 1	y = 0.0116x−21.059	0.0208	0.17–3.12	2.126c	0.7296
Check Plot	15	Intercept = 0 Slope = 1	y = −0.0025x+6.1479	0.0015	0.33–1.82	1.0766d	0.3770
P Plot	15	Intercept = 0 Slope = 1	y = 0.0066x−12.036	0.0232	0.29–1.75	1.2046d	0.3948
NP Plot	15	Intercept = 0 Slope = 1	y = 0.0393x−76.392	0.1445	0.18–4.10	2.2606bc	0.9385
NPK Plot	15	Intercept = 0 Slope = 1	y = 0.0132x−24.08	0.0233	0.56–3.87	2.4586ab	0.7892
NPKL Plot	14	Intercept = 0 Slope = 1	y = 0.0135x−24.564	0.0209	0.36–4.05	2.5813a	0.6119
Source	DF	Type III SS	Mean Square			F Value	PR>|F|
Treatment	5	31.5455	6.3091			54.7200	<.0001
Year	14	34.7608	2.4829			21.5300	<.0001

† Means followed by the same letter are not significantly different.

Soil pH over time, had a non-significant slope component ($p < 0.05$) and an average pH of 6.48. It was the highest soil pH among the four treatments (Table 5). This level of soil pH is consistent with findings from Girma et al. (2007) who also reported a soil pH of 6.20 in the same plot. Even though manure application can contribute to soil acidification due to ammonium mineralization or additions to organic acids (Havlin et al. 2014; Sylvia et al. 1998), manure further has a significant concentration of calcium carbonate (Eghball 1999) which reacts with H^+ and increases soil pH. . Sharpley and Smith (1995) stated that manure application increases inorganic P fractions, especially, bicarbonate inorganic P which is the major contributor to Ca-bound in the soil, favoring the formation of hydroxyapatite.

Grain yield did not have a significant positive or negative trend ($p < 0.05$); however, the average mean was 2.12 Mg/ha (Table 6). Manure application was calculated based on N to achieve the maximum yield (90 kg/ha) but it is not calibrated to remedy other limiting elements such as P or K. Furthermore, the actual content of manure varies widely, and this may negatively affect the grain yield when compared to NP, NPK, and NPKL plots that had statistically higher grain yield than the manure plot (Table 6).

Check plot

Total N fluctuated over the years, ranging from 0.77 g/kg to 1.08 g/kg with a significant decrease over time ($p < 0.05$) (Table 2). This result is not consistent with Rice, Smith, and Blevins (1986) who stated that after five years of cultivation in a conventional tillage system, TN stabilized at 1.5 mg/g. The TN mean in the check plot was statistically similar to the P plot and they had the lowest level of TN among all treatments (Table 2).

According to Schaetzl and Thompson (2015), cultivation accelerates soil oxidation promoting C loss as CO_2 due to physical disturbance of the aggregates and oxygen supply to microorganisms. Hence, the depletion of SOC decreases the capability of the soil to store nutrients as mentioned by Sylvia et al. (1998) where 20–80% of the Cation exchange capacity (CEC) is dependent on SOM. Nevertheless, after long periods of cultivation, SOM tends to stabilize where inputs in SOM balances out with losses as decomposition proceeds. However, SOC has not achieved the stability, and has been decreasing with time ($p < 0.05$). Soil organic C started at 6.80 g/kg in 1990 and was 6.30 g/kg in 2015, with a range of 6.11 to 8.84 g/kg (Table 3). The average content of SOC was 6.75 g/kg (Table 3), which was the lowest level of SOC among all the treatments.

The ratio between C and N was variable, similar to TN, but inversely proportional after 2009. This suggests that when TN decreases, the C:N ratio increases.

Soil pH decreased with time at rate of 0.017 per year ($p < 0.05$). Soil pH had a range of 5.52 to 6.47 (Table 5). Initially, soil pH started at 6.10 in 1990 and decreased to 5.59 by 2015. This result is consistent with the literature where SOM oxidation contributes to increased soil acidity by forming CO_2, which reacts with water and produces a weak acid. This acid dissociates and releases H+ to the soil solution due to the acid coefficient constant equaling 6.35 (Brady and Weil 2008). On average, soil pH was 5.8 that was high compared to other treatments and the only one below that observed in the manure plot.

Grain yield averaged 1.01 Mg/ha (Table 6). This result is consistent with Girma et al. (2007) who noted that grain yield in the check plot was 1 Mg/ha without any fertilizer application for more than 114 years of continuous wheat production under conventional tillage. The P plot also had a statistically similar grain yield mean, at 1.20 Mg/ha (Table 6). Alternatively, those plots receiving inorganic N (NP, NPK, NPKL) had grain yields above 2.2 Mg/ha (Table 6).

P plot

Total N had a non-significant regression coefficient ($p < 0.05$) and ranged from 0.79 g/kg to 1.11 g/kg with a mean of 0.92 g/kg (Table 2). This mean is statistically similar to the check plot. Because a decrease in SOC would be associated with mineralized N, a decrease in the C:N ratio was expected. Nonetheless, the ratio between C and N showed a non-significant regression coefficient ($p < 0.05$), ranging from 6.74 to 10.05 and a mean of 7.78 (Table 4).

Schaetzl and Thompson (2015) explained that calcium is an effective agent to stabilize organic matter. Thus the application of triple superphosphate, which contains calcium, can promote resistance of SOM to microbial decomposition and prevent C loss. However, SOC has been decreasing by 0.04 g/kg per year ($p < 0.05$), presenting minimum values of 6.24 g/kg and maximum values of 9.40 g/kg (Table 3). In addition, the average mean of SOC in the P plot was 7.1 g/kg as shown in Table 3. This suggests that SOM had not achieved stability because the P plot was derived from a larger check plot, which received manure application from 1892 to 1929 (Davis et al. 2003).

Soil pH decreased 0.01 per year over 25 years ($p < 0.05$). In 1990, soil pH was 6.18 and dropped to 5.42 in 2015. Similar to work by Aula et al. (2016), P application had little or no effect on soil pH. Therefore, other factors contributed to decreased soil pH such as SOM oxidation, mineralization of N, and nutrient uptake (Brady and Weil 2008). On average, soil pH in the P plot was 5.58 (Table 5).

The average grain yield in the P plot was similar to the check plot at 1.2 Mg/ha (Table 6) and had one of the lowest grain yields in comparison to the other treatments. Even though P was the first most limiting nutrient in 1930–1957, crop response changed once N was depleted from SOM and as was mentioned by Girma et al. (2007).

NP plot

According to Khan et al. (2007) and Mulvaney, Khan, and Ellsworth (2009), application of N decreases the C:N ratio, favoring the decomposition of SOM. Total N over time had a non-significant regression coefficient ($p < 0.05$) but fluctuated between 0.91 g/kg and 1.23g/kg (Table 2). Nitrogen fertilizer is extremely susceptible to loss by leaching, volatilization, denitrification, and plant uptake (Brady and Weil 2008). Thus, total N might not reflect the inorganic additions from N fertilizer but rather the organic portion of SOM. It maintained a TN average of 1.04 g/kg, which was statistically similar to NPK and NPKL plots but lower than the manure plot (Table 2).

Soil organic C had a non-significant regression coefficient ($p < 0.05$), ranging between 7.44 g/kg to 9.03 g/kg and a mean of 8.30 g/kg (Table 3). This mean was statistically similar to the NPK plot, but higher than the check plot and the P plot, yet lower than the manure plot (Table 3). The NP plot was also split from the original check plot that had received manure between 1892 and 1929. However, in contrast to the P plot, SOC has become stable. Nitrogen stimulates plant growth and development of the root system (Brady and Weil 2008). Roots produce organic substances that promote an increase in the microbial population, which excretes polysaccharides that act like a binding agent, stabilizing SOM (Haynes and Naidu, 1997).

The C:N ratio over time had a non-significant regression coefficient ($p < 0.05$) ranging from 7.36 to 9.03 with a mean of 8.54 (Table 4). Nitrogen fertilizer can increase soil acidity due to nitrification of ammonium (Brady and Weil 2008). In Table 5, the slope coefficient shows that soil pH decreased by 0.01 per year ($p < 0.05$). In 1990, soil pH was 5.39 and 4.94 by 2015. The average soil pH in the NP plot was statistically similar to the NPK plot (Table 5). In addition, the change of fertilizer was not noticeable as observed by Schoroder et al. (2011) who concluded that different sources of N did not present a significant acidity difference in the soil. At low pH values, plants tend to accumulate ammonium because nitrifying bacteria are sensitive to low pH (<5.5) as Bohn, McNeal, and O'Connor (2001) suggested, but De Boer and Kowalchuk (2001) concluded that autotrophic bacteria are the main nitrifying agents in acidic soils which can supply nitrate to the soil. At a low pH (<4.7), Al^{+3} can be a predominant ion in the soil solution and that can be toxic to plants. It hydrolyzes water and releases H^+, which decreases soil pH (Bohn, McNeal, and O'Connor 2001). As a result, the availability of nutrients decreases. In addition, Sylvia et al. (1998) stated that mineralization decreases when soil pH is below 4.5. However, these conditions did not affect grain yield which fluctuated between 0.18 Mg/ha to 4.10 Mg/ha and achieved a mean of 2.2 Mg/ha (Table 6). This mean was statistically similar to NPK and the manure plot (Table 6). Nitrogen fertilization stimulates microbial activity by supplying N to the microbial community and then slowly releases N to the crop. This phenomenon is known as the priming effect (Westerman and Kurtz 1973), and it happens when a low to moderate rate of N fertilizer (<67kg/ha) is applied in the soil (Raun et al. 1998).

NPK plot

A similar trend was found in the NPK plot, where TN, OC, and C:N ratio had non-significant regression coefficients ($p < 0.05$). Total N ranged from 0.88 g/kg to 1.12 g/kg with a mean of 1.03 g/kg (Table 2). This mean was statistically similar to the NP and NPKL plots but lower than the manure plot (Table 2). Organic C ranged between 7.5 g/kg to 10.2 g/kg and had a mean of 8.5 g/kg (Table 3). Again, it was similar to the NP plot but lower than the manure plot (Table 3). The C:N ratio ranged between 7.3 and 9.7 with a mean of 8.26 (Table 4).

Furthermore, soil pH decreased by 0.02 units per year ($p < 0.05$). In 1990, soil pH started at 5.5 and decreased to 4.86 by 2015. Average soil pH in the NPK and NP plot were the lowest compared to the other treatments, at 5.0 and 5.13 respectively (Table 5). This could be due to N mineralization.

The average grain yield was 2.4 Mg/ha and it was the highest grain yield among all treatments, but statistically similar to the NPKL plot (Table 6). The reason for this is that most of the liming factors were corrected.

NPKL plot

Lime was applied when soil pH was below 5.5; however, re-analyzing the soil samples revealed that in some years the soil samples had pH values below 5.5. This discrepancy among the pH readings might be related to equipment operation as Summer (1994) mentioned. Despite this issue, soil pH increased from 4.48 to 5.64 after 5 years of lime application. Combining all years, the NPKL plot did not illustrate a significant trend over time for any of the variables. However, the C:N ratio did increase when the analysis focused on years 2010 to 2015. This result suggests that mineralization was occurring. However, it did not affect TN or OC. They fluctuated between 0.92 g/kg to 1.33 g/kg for TN (Table 2) and 7.75 g/kg and 10.55 g/kg for OC (Table 3). Buerkert et al. (2012) stated that in the first year after application, lime favors mineralization and the release of N and C. Furthermore, the long-term effect of lime application stimulates root growth by lowering aluminum in soil solution and that increases OC (Haynes and Naidu 1998).

The NPKL plot grain yield had the highest grain yield among all treatments (Table 6) at 2.5 Mg/ha as a result of lime application which increased the nutrient availability to the crop and decreased aluminum activity (Brady and Weil 2008).

Conclusions

Manure application every four years at a rate of 269 kg N/ha resulted in the highest mean for all dependent variables excluding grain yield, which was below NPK and NPKL plots. Furthermore, it maintained SOC at 0.92% and adequate soil pH.

Soil test parameters in the check plot continue to decrease, evidenced in the TN and OC data reported. It also had the lowest mean among all treatments for the variables evaluated. The P plot showed that even though P was the most limiting factor in 1930–1957, this deficiency is much less pronounced as time has led to N being the most limiting nutrient. All plots receiving inorganic N showed a decrease in soil pH over time, but that resulted in increased yields when compared to the manure plot. Liming the soil when soil pH was below 5.5, helped to alleviate acidity and maintain good yields.

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