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Effect of Long-Term Fertilization on Organic Nitrogen Forms in a Calcareous Alluvial Soil on the North China Plain^{*1}

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ABSTRACT

In order to illustrate the change of nitrogen (N) supply capacity after long-term application of manure and chemical fertilizer, as well as to properly manage soil fertility through fertilizer application under the soil-climatic conditions of the North China Plain, organic N forms were quantified in the topsoil with different manure and chemical fertilizer treatments in a 15-year fertilizer experiment in a Chinese calcareous alluvial soil. Soil total N (TN) and various organic N forms were significantly influenced by long-term application of chemical fertilizer and manure. TN, total hydrolysable N, acid-insoluble N, amino acid N and ammonium N in the soil increased significantly ($P < 0.05$) with increasing manure and fertilizer N rates, but were not influenced by increasing P rates. Also, application of manure or N fertilizer or P fertilizer did not significantly influence either the quantity of amino sugar N or its proportion of TN. Application of manure significantly increased ($P < 0.05$) hydrolysable unknown N, but adding N or P did not. In addition, application of manure or N fertilizer or P fertilizer did not significantly influence the proportions of different soil organic N forms.

Key Words: chemical fertilizer, long-term fertilization, manure, North China Plain, soil organic nitrogen forms

INTRODUCTION

In most topsoils organic nitrogen (N) comprises more than 95% of soil total N (TN) (Stevenson, 1982a; Zhu, 1997). It contains old and new N fractions, some of which continually undergo microbially mediated mineralization and immobilization processes. Soil organic N can be divided into different forms according to chemical procedures proposed by Bremner (1965), and the chemical properties of different organic N forms in soil have some relationship with their availability to a crop after mineralizing (Stevenson, 1982a; Wu, 1986; Shen and Shi, 1990; Wang *et al.*, 1991). There have been numerous reports in the literature concerning the effects of fertilization on quantity and composition of organic N forms in the soil (Keeny, 1964; Khan, 1971; Smith and Young, 1975; Stevenson, 1982a, b; Wu, 1986; Wang *et al.*, 1991; Allison, 1997; Zhu, 1997; Liang *et al.*, 2000), but the conclusions were quite contradictory. For instance, some studies showed that different fertilization management could significantly affect the quantity of different organic N forms; however the composition of these N forms was not significantly changed (Smith and Young, 1975; Stevenson, 1982b; Wang *et al.*, 1991); other studies showed that not only the quantity but also the composition of organic N forms could be significantly affected by fertilization (Keeny, 1964; Khan, 1971; Liang *et al.*, 2000).

Studies on soil N forms conducted in China have mostly concentrated on: 1) the composition of organic N forms in different types of soils or different regions (Zhang *et al.*, 1984; Song, 1988; Zhou and Shi, 1992), 2) the relationship between organic N forms and their availability to crops (Shen and Shi, 1990), and 3) the changes of organic N forms after cultivation of virgin soil (Shi *et al.*, 1992). Few studies have investigated the effect of fertilization on organic N forms (Shi and Zhou, 1995; Xu and Wu, 1991; Liang *et al.*, 2000), especially after long-term fertilization.

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The mineralization and immobilization of different organic N forms in soil is a slow process, thus long-term fertilization experiments are needed to analyze their pool changes. In order to illustrate the change of N supply capacity after long-term application of manure and chemical fertilizer as well as to properly manage soil fertility through fertilizer application under the soil-climatic conditions of the North China Plain, this study quantified organic N forms in topsoil with different treatments of manure and chemical fertilizer in a 15-year fertilization trial in a Chinese calcareous alluvial soil.

MATERIALS AND METHODS

A calcareous alluvial soil (FAO classification) was utilized in a 15-year fertilizer experiment at China Agricultural University's Changping Experimental Station. The climate is a warm-temperate, sub-humid, continental monsoon type, with cold winters and hot summers. The average annual temperature is 11–12 °C; the annual precipitation is 600–950 mm; and the groundwater table is 2–3 m. In 1984 at the beginning of the experiment, the soil chemical properties were: pH, 8.1; organic matter, 13.1 g kg⁻¹; total N, 0.71 g kg⁻¹; Olsen-available P, 6.1 mg kg⁻¹; and exchangeable K, 114 mg kg⁻¹.

A manure and chemical fertilizer experiment was conducted with a split-plot design and three replications. The main treatments consisted of two levels of chicken manure: 0 (F) and 7500 kg ha⁻¹ year⁻¹ (M), while the sub-treatments had nine levels of combined chemical N and P fertilizers, whose N fertilizer and P fertilizer levels were 0 (N0), 135 (N1) and 270 (N2) kg N ha⁻¹ crop⁻¹, and 0 (P0), 67.5 (P1) and 135 (P2) kg P₂O₅ ha⁻¹ crop⁻¹, respectively. Thus, the sub-treatments were N0P0, N0P1, N0P2, N1P0, N1P1, N1P2, N2P0, N2P1, and N2P2 with or without chicken manure. Urea (N, 460 g kg⁻¹) and triple calcium superphosphate (P₂O₅, 460 g kg⁻¹) were used as the N and P fertilizers, respectively. Water and nutrient in the chicken manure varied from year to year with averages from 1984 to 1989 being 475 g kg⁻¹ for water, and 134, 12.5, 9.4 and 12.4 g kg⁻¹ for organic matter, total N, total P and total K (air-dry weight basis), respectively.

For 15 years the field experiment had a crop rotation of winter wheat-maize-maize with one winter fallow every two years. Consequently, crops in each plot had been harvested 21 times when the soil was sampled in 1999. Soil samples were collected at 0–20 cm from each plot, air-dried and ground to pass a 0.15-mm sieve.

Soil TN and organic N forms were determined by the procedures described in Stevenson (1996). The effect of long-term fertilization on soil TN, total hydrolysable N and acid-insoluble N; on soil ammonium N and amino acid N; and on soil amino sugar N and hydrolysable unknown N was determined with an analysis of variance (ANOVA) where significance was compared by least significant difference (LSD) at the 5% level using the SAS statistical package (Version 6.12).

RESULTS

Soil total hydrolysable N, acid-insoluble N, and TN

Long-term chemical N or P fertilization combined with manure compared to that without manure significantly increased ($P < 0.05$) soil total hydrolysable N, acid-insoluble N, and TN by 11.4%, 27.3%, and 15.2%, respectively (Table I). Moreover, the average soil TN (0.74 g kg⁻¹) in the plots receiving chemical fertilizer was slightly higher than the initial soil TN (0.71 g kg⁻¹) at the beginning of the experiment. Regardless of organic manure and P fertilizer applications, soil total hydrolysable N, acid-insoluble N, and TN with the highest N rate (N2) was significantly greater than that with N0 and N1. However, the increasing P rate did not significantly increase total hydrolysable N, acid-insoluble N or TN in the soil (Table I).

Total hydrolysable N ranged from 489 to 653 mg kg⁻¹ with an average of 601 mg kg⁻¹ across the different treatments and formed the major part of TN, comprising 69%–80% of TN with an average of 75% according to Fig. 1. Acid-insoluble N ranged from 133 to 284 mg kg⁻¹ with average of 200 mg kg⁻¹, comprising 20%–31% of TN with an average of 25% (Fig. 1). Nevertheless, the percentage of

acid-insoluble N in TN (CV = 15%) varied more widely than that of total hydrolysable N (CV = 5%).

TABLE I

Influence of long-term fertilization on soil total hydrolysable N, acid-insoluble N and total N (mean of different treatments)

Treatments ^{a)}	Total hydrolysable N	Acid-insoluble N	Total N
		mg kg ⁻¹	
F (N0, N1, N2, P0, P1, P2)	568 a ^{b)}	176 a	744 a
M (N0, N1, N2, P0, P1, P2)	633 b	224 b	857 b
N0 (F, M, P0, P1, P2)	574 a	173 a	747 a
N1 (F, M, P0, P1, P2)	605 a	195 a	800 a
N2 (F, M, P0, P1, P2)	624 b	230 b	854 b
P0 (F, M, N0, N1, N2)	602 a	194 a	796 a
P1 (F, M, N0, N1, N2)	606 a	210 a	816 a
P2 (F, M, N0, N1, N2)	594 a	195 a	789 a

^{a)} Chicken manure: 0 (F) and 7500 (M) kg ha⁻¹ year⁻¹; N fertilizer: 0 (N0), 135 (N1), and 270 (N2) kg N ha⁻¹ crop⁻¹; P fertilizer: 0 (P0), 67.5 (P1), and 135 (P2) kg P₂O₅ ha⁻¹ crop⁻¹; F (N0, N1, N2, P0, P1, P2) and M (N0, N1, N2, P0, P1, P2): all treatments with F and M, respectively; N0 (F, M, P0, P1, P2), N1 (F, M, P0, P1, P2), and N2 (F, M, P0, P1, P2): all treatments with N0, N1, and N2, respectively; P0 (F, M, N0, N1, N2), P1 (F, M, N0, N1, N2), and P2 (F, M, N0, N1, N2): all treatments with P0, P1, and P2, respectively.

^{b)} Mean values in a column followed by the same letter are not significantly different ($P = 0.05$) using LSD.

Fig. 1 Effect of long-term fertilization on soil total hydrolysable N and acid-insoluble N ($n = 3$). F and M: chicken manure of 0 and 7500 kg ha⁻¹ year⁻¹, respectively; N0, N1, and N2: N fertilizer of 0, 135, and 270 kg N ha⁻¹ crop⁻¹, respectively; P0, P1, and P2: P fertilizer of 0, 67.5, and 135 kg P₂O₅ ha⁻¹ crop⁻¹, respectively.

Soil amino acid N and ammonium N

Amino acid N ranged from 148–224 mg kg⁻¹ with an average of 182 mg kg⁻¹ or 23% of TN according to Fig. 2. Also application of manure or N fertilizer significantly increased amino acid N (Table II). Ammonium N ranged from 102 to 212 mg kg⁻¹ or 15% to 26% of TN (Fig. 2). The application of manure or N fertilizer significantly increased ammonium N, whereas application of P fertilizer was not significant (Table II).

Soil amino sugar N and hydrolysable unknown N

Amino sugar N ranged from 2.3 to 65 mg kg⁻¹ with an average of 23 mg kg⁻¹ (Fig. 2). Compared with other organic N forms, the amino sugar N of TN was lowest, being 0.3%–9.1% of TN with an average of 3.0%, and showed the largest variation (CV = 84%). With amino sugar N, application of manure, N or P fertilizer did not significantly affect either the quantity (Table II) or the proportion of TN.

Fig. 2 Effect of long-term fertilization on soil organic N forms ($n = 3$). F and M: chicken manure of 0 and 7 500 kg ha⁻¹ year⁻¹, respectively; N0, N1, and N2: N fertilizer of 0, 135, and 270 kg N ha⁻¹ crop⁻¹, respectively; P0, P1, and P2: P fertilizer of 0, 67.5, and 135 kg P₂O₅ ha⁻¹ crop⁻¹, respectively.

TABLE II

Effect of long-term fertilization on soil amino acid N, ammonium N, amino sugar N, and hydrolysable unknown N (mean of different treatments)

Treatments ^{a)}	Amino acid N	Ammonium N	mg kg ⁻¹	
			Amino sugar N	Hydrolysable unknown N
F (N0, N1, N2, P0, P1, P2)	173 a ^{b)}	164 a	34 a	197 a
M (N0, N1, N2, P0, P1, P2)	192 b	184 b	13 a	245 b
N0 (F, M, P0, P1, P2)	174 a	152 a	34 a	213 a
N1 (F, M, P0, P1, P2)	186 b	185 b	13 a	220 a
N2 (F, M, P0, P1, P2)	187 b	186 b	22 a	229 a
P0 (F, M, N0, N1, N2)	188 a	172 a	25 a	216 a
P1 (F, M, N0, N1, N2)	183 a	180 a	17 a	227 a
P2 (F, M, N0, N1, N2)	177 a	170 a	27 a	220 a

^{a)}Chicken manure: 0 (F) and 7 500 (M) kg ha⁻¹ year⁻¹; N fertilizer: 0 (N0), 135 (N1), and 270 (N2) kg N ha⁻¹ crop⁻¹; P fertilizer: 0 (P0), 67.5 (P1), and 135 (P2) kg P₂O₅ ha⁻¹ crop⁻¹; F (N0, N1, N2, P0, P1, P2) and M (N0, N1, N2, P0, P1, P2): all treatments with F and M, respectively; N0 (F, M, P0, P1, P2), N1 (F, M, P0, P1, P2), and N2 (F, M, P0, P1, P2): all treatments with N0, N1, and N2, respectively; P0 (F, M, N0, N1, N2), P1 (F, M, N0, N1, N2), and P2 (F, M, N0, N1, N2): all treatments with P0, P1, and P2, respectively.

^{b)}Mean values in a column followed by the same letter are not significantly different ($P = 0.05$) using LSD.

Hydrolysable unknown N ranged from 147 to 296 mg kg⁻¹ with an average of 221 mg kg⁻¹ (Fig. 2) and contributed 21%–36% of TN with an average of 28%. Application of manure significantly increased hydrolysable unknown N, while N or P fertilization was not significant (Table II).

Composition of different soil organic N forms

The proportions of soil organic N forms expressed as a percentage of soil total N are given in Table III. It could be seen that application of manure, N and P fertilizer did not significantly influence the composition of the different soil organic N forms.

DISCUSSION

The chemical forms of soil organic N are important in that they affect soil N availability to crops (Shen and Shi, 1990). Different fertilizer practices influence the quantity and composition of soil organic N forms. For example, after N application in their ¹⁵N-tracer experiment, Allen *et al.* (1973) found that

TABLE III

Effect of long-term fertilization on proportions of soil organic N forms expressed as a percentage of soil total N (mean of different treatments)

Treatments ^{a)}	Amino acid N	Ammonium N	Amino sugar N	Hydrolysable unknown N	Acid-insoluble N
	%				
F (N0, N1, N2, P0, P1, P2)	23.3 a ^{b)}	22.1 a	4.5 a	26.5 a	23.6 a
M (N0, N1, N2, P0, P1, P2)	22.4 a	21.5 a	1.5 a	28.5 a	26.1 a
N0 (F, M, P0, P1, P2)	23.3 a	20.4 a	4.6 a	28.6 a	23.2 a
N1 (F, M, P0, P1, P2)	23.3 a	23.1 a	1.6 a	27.6 a	24.4 a
N2 (F, M, P0, P1, P2)	21.9 a	21.8 a	2.6 a	26.8 a	27.0 a
P0 (F, M, N0, N1, N2)	23.6 a	21.6 a	3.2 a	27.2 a	24.4 a
P1 (F, M, N0, N1, N2)	22.4 a	22.1 a	2.1 a	27.8 a	25.7 a
P2 (F, M, N0, N1, N2)	22.4 a	21.6 a	3.5 a	27.9 a	24.7 a

^{a)}Chicken manure: 0 (F) and 7 500 kg ha⁻¹ year⁻¹ (M); N fertilizer: 0 (N0), 135 (N1), and 270 (N2) kg N ha⁻¹ crop⁻¹; and P fertilizer: 0 (P0), 67.5 (P1), and 135 (P2) kg P₂O₅ ha⁻¹ crop⁻¹; F (N0, N1, N2, P0, P1, P2) and M (N0, N1, N2, P0, P1, P2): all treatments with F and M, respectively; N0 (F, M, P0, P1, P2), N1 (F, M, P0, P1, P2), and N2 (F, M, P0, P1, P2): all treatments with N0, N1, and N2, respectively; P0 (F, M, N0, N1, N2), P1 (F, M, N0, N1, N2), and P2 (F, M, N0, N1, N2): all treatments with P0, P1, and P2, respectively.

^{b)}Mean values in a column within each treatment followed by the same letters are not significantly different ($P = 0.05$) using LSD.

the proportions of amino acid N and amino sugar N in TN of soil humus increased, while acid-insoluble N and hydrolysable unknown N decreased. Wang *et al.* (1991) also showed that soil residual N from chemical N fertilizer mainly transferred to hydrolysable unknown N and amino acid N, but soil residual N from manure mainly transformed into ammonium N and amino sugar N. Furthermore, studies of Yang and Wang (1991) and Zhang *et al.* (1989) indicated that an application of manure led to an increase in amino acid N in soils; the results of Xu and Wu (1991) showed that application of urea without manure led to a decrease in total hydrolysable N in soil, but application of urea with manure led to an increase in total hydrolysable N.

In the present study, average TN slightly increased over the 15-year period under chemical fertilizer application alone, perhaps reflecting the increase in returned crop residues (about 2.5 t ha⁻¹ year⁻¹). Nevertheless, application of manure or N fertilizer significantly affected TN and organic N forms, while P fertilizer did not. The results also indicated that the combination of manure with chemical fertilizer increased the N supply potential to the crop, due to total hydrolysable N, acid-insoluble N and TN being significantly increased. Additionally, the manure and N fertilizer applications also significantly increased amino acid N, thereby increasing the N supply potential to crops because it was readily mineralizable. The ammonium N in the present study was slightly lower than the values obtained in other studies (Song, 1988; Liang *et al.*, 2000). Additionally, a considerable amount of ammonium N, usually comprising up to 20%–35% of TN, will be produced during soil hydrolysable processes (Song, 1988; Liang *et al.*, 2000).

On the other hand, application of manure, N and P fertilizer had no significant influence on the proportions of different soil organic N forms. This finding was in accord with the results of Liang *et al.* (2000) under similar soil conditions. In conclusion, long-term application of manure and N fertilizer effectively increased the content of soil TN and most organic N forms, thereby potentially increasing the soil N supply to crops.

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