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Application of ^{31}P NMR spectroscopy in determining phosphatase activities and P composition in soil aggregates influenced by tillage and residue management practices

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ABSTRACT

Soil phosphorus (P) composition and phosphatase activities in aggregates are essential for agricultural productivity and remain poorly understood. A field experiment was conducted from 2007 to study the effect of tillage systems (conventional tillage, T and no tillage, NT) and crop residue management (0, 50% and 100% crop residue incorporation/coverage) on P composition determined by ^{31}P nuclear magnetic resonance (NMR) and phosphatase activities in soil aggregates (>2 mm, 0.25–2 mm and 0.053–0.25 mm). The results showed that crop residue input influenced the concentrations of soil phosphate monoesters and diesters, alkaline phosphomonoesterase (AlP), acid phosphomonoesterase (AcP), phosphodiesterase (PD) activities, and soil aggregate stability significantly, and the addition of crop residue was significantly more effective than tillage. The NT had significantly higher soil phosphatase activities than tillage treatment but not more soil P content. The 0.25–2 mm aggregates showed higher total P, organic P, concentrations of monoesters and diesters, and AlP activity. The structure equation model showed that soil aggregate stability could increase concentrations of monoesters and diesters indirectly by its direct effects on soil phosphatases. Our results suggest that NT and crop residue input could increase the P store and sustainable supply in soil aggregates and that the 0.25–2 mm size aggregates may play an important role in soil organic P maintenance and transformation.

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1. Introduction

Soil phosphorus (P) is one of the major nutrients limiting agricultural production and consists of organic P and inorganic P. Organic P accounts for a high proportion of the total P, but most of it cannot be utilized directly by crops (Schachtman et al., 1998). Because tillage significantly influences soil P (Selles et al., 1997; Redel et al., 2007; Zamuner et al., 2008) and long-term tillage practices have reduced soil P content (Franzluebbers and Hons, 1996; Sisti et al., 2004), it is necessary to study how to increase soil P storage and the utilization efficiency of organic P by adopting optimized soil agricultural management. The effect of conservation tillage on soil nutrients has been widely studied (Franzluebbers and Hons, 1996; Al-Kaisi et al., 2005; Chen et al., 2009). It has been confirmed that crop residue inputs and no tillage can enhance soil organic matter content, improve soil structure and increase the accumulation of soil nutrients (Havlin et al., 1990; Malhi and Lemke, 2007; Wang et al., 2011).

Organic P in soil can be hydrolyzed by phosphatases to release inorganic orthophosphate for crop uptake. In brief, soil diesters can be hydrolyzed by phosphodiesterase (PD) to release monoesters, and then inorganic orthophosphate is released through hydrolysis of monoesters by alkaline phosphomonoesterase (AlP) and acid phosphomonoesterase (AcP) (Tabatabai, 1994; Turner and Haygarth, 2005). Traditionally, the determination of soil organic P was hindered by the difficulties of the extraction, separation and detection of recalcitrant compounds. However, the adoption of solution ^{31}P nuclear magnetic resonance (NMR) spectroscopy eliminated these problems (Turner et al., 2003c). Organic P can be extracted by using NaOH-EDTA extraction and solution ^{31}P NMR spectroscopy (Bowman and Moir, 1993), and identified by their chemical shifts in the spectra (Turner et al., 2003b, 2003c; Cade-Menun, 2005). The organic P composition determined by ^{31}P NMR primarily consists of monoesters and diesters in most soils (Rheinheimer et al., 2002; Turner et al., 2003a).

Although several investigators have found that residue input with no tillage can increase soil phosphatase activities (Deng and Tabatabai, 1997; Wang et al., 2011; Wei et al., 2014) and

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monoester and diester concentrations (Condron et al., 1990; Redel et al., 2011; Zhang et al., 2012a), little is known about the relationship between phosphatases and organic P composition of soils under tillage and crop residue management. Moreover, soil phosphatase activities in aggregates affected by tillage have been reported by Gupta and Germida (1988). They found that macroaggregates in both native and cultivated soils had higher phosphatase activities than microaggregates. However, the study of the distribution of P composition and the relationship between phosphatases and P composition in soil aggregates under tillage and residue management is limited except for the study conducted by McDowell et al. (2007) with a pot trail.

The objectives of our study were to investigate the effect of tillage and crop residue management on soil P composition determined by ^{31}P NMR and phosphatases activities and to explore the relationship between phosphatases and organic P composition in soil aggregates.

2. Materials and methods

2.1. Site description

The field experiment was established in September 2007 at the Fengqiu State Key Agro-Ecological Experimental Station (35°01′ N, 114°32′ E), Henan province, China. The study plots were located in the Huang-Huai-Hai Plain with a semi-arid and sub-humid warm temperate monsoon climate. The annual precipitation in this area ranged from 355 mm to 800 mm, of which two thirds took place from June to September. The average annual temperature was 13.9 °C, and the lowest and highest mean monthly values were −1.0 °C in January and 27.2 °C in July, respectively (Ding et al., 2010). The soil type was Ochri-Aquic Cambosol according to the World Reference Base for Soil Resources (FAO/ISRIC/ISSS, 1998) with a profile of sandy loam (about 9% clay, 21.8% silt) in the plough layer (0–20 cm) and 11.13 g kg^{-1} organic matter, total nitrogen 1.39 g kg^{-1}, total phosphorus 0.72 g kg^{-1}, total potassium 14.53 g kg^{-1}, pH (H_2O) 8.24 and bulk density 1.16 g cm^{-2} (Cai and Qin, 2006; Zhu et al., 2009).

2.2. Experimental design

The experiment was set up using a split-plot design with three replicates and was conducted under field controlled condition. Tillage treatment was the main plots, and crop residue management was the subplots. The tillage treatments were conventional tillage (T) and no tillage (NT). Crop residue managements were 0 (no wheat and maize residue incorporation/coverage), 50% and 100% (7.5 t ha^{-1} in the wheat season and 8.12 t ha^{-1} in the maize season). The tillage treatment was plowed with a moldboard to a depth of 23 cm, then, the soil was disked twice, with a disk harrow before seed sowing. The NT treatment was managed similarly to the tillage treatment, except for tillage which continued to be NT with a no-tillage planter for seed sowing. The chopped crop residues were incorporated into the soil for the tillage systems and covered the soil surface for the no tillage systems after the wheat and maize had been harvested every year. The average phosphate content of maize and wheat residues was 21.4 kg hm^{-2} and 6.2 kg hm^{-2}, respectively (Gao et al., 2009). The experimental plots consisted of six sub-plots: T0 (conventional tillage without residue incorporation/coverage), T50 (conventional tillage with 50% residue incorporation), T100 (conventional tillage with 100% residue incorporation), NT0 (no tillage without residue incorporation/coverage), NT50 (no tillage with 50% residue coverage) and NT100 (no tillage with 100% residue coverage). Each sub-plot was 4 m × 100 m and was under a rotation of winter wheat (early October to mid-May) and summer maize (early June to mid-September). Two fertilizations were applied in the wheat and maize seasons for all treatments. One was applied when sowing in October and June in the amount of 150 kg ha^{-1} (N:P:K = 17:9:5), and the other was applied in March in the wheat season and in August in the maize season with urea (120 kg N ha^{-1}).

2.3. Soil sampling

Soil was collected on September 9, 2010, before the maize harvest and has experienced 3 years' management since the field experiment was conducted. Three random, undisturbed soil subsamples were taken from 0 to 20 cm depth of surface soil (≈2000 cm^3) where tillage mixed soil and the fertilizer and crop residues well. Then, soil subsamples were put into hard plastic containers to prevent the disruption of their structure. After being transported to the laboratory, the three undisturbed subsamples were combined into one soil sample. After plant materials and stones were removed, a portion of each soil sample was sieved (2 mm) and stored at 4 °C until it was analyzed. The remaining samples were used to fractionate soil aggregates.

2.4. Soil aggregate distribution

Soil aggregate size fractions were obtained by adopting the methods of Schutter and Dick (2002) and Sainju et al. (2003). Before soil aggregates were fractionated, large soil clods were gently broken apart and laid out on brown paper to dry slowly for several days. This process was conducted at 4 °C to minimize the impact of air drying on microbial communities and activities (Schutter and Dick, 2002) until an 80 g kg^{-1} soil gravimetric water content was reached so that dry sieving method could be implemented effectively.

Because the disruption of the physical habitat of microbial communities was less with dry-sieving than with wet-sieving (Schutter and Dick, 2002), dry-sieving was used to fractionate soil aggregates. Before sieving, air-dried soils were sieved (5 mm). Fractionation was achieved by placing 100 g of sieved soils on nested sieves mounted on a Retsch AS200 Control (Retsch Technology, Düsseldorf, Germany). Sieves were mechanically shaken (amplitude 1.5 mm) for 2 min to separate soil into the following aggregate size fractions: >2 mm (large macroaggregates), 0.25–2 mm (small macroaggregates), 0.053–0.25 mm (microaggregates) and <0.053 mm (silt + clay size fraction).

Fractionated samples were later combined into one composite sample for each aggregate size fraction. Aggregate distribution was obtained by weighing soil from each aggregate size fraction. Bulk soil and all the soil aggregates were stored at 4 °C until analysis.

The mean weight diameter (MWD) and geometric mean diameter (GMD) of soil aggregates were calculated as follows (Kemper and Rosenau, 1986):

$$\mathrm{MWD} = \sum_{i=1}^{n} x_i w_i$$

where, x_i is the mean diameter (mm), and w_i is the weight proportion of each size fraction.

$$\mathrm{GMD} = \exp\left[\frac{\sum_{i=1}^{n} w_i \ln x_i}{\sum_{i=1}^{n} w_i}\right]$$

where, w_i is the weight of aggregates of each size fraction (g), and $\ln x_i$ is the natural logarithm of the mean diameter of each size fraction.

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