Geoderma 279 (2016) 1–10

Contents lists available at ScienceDirect

Geoderma

journal homepage: www.elsevier.com/locate/geoderma

The role of poorly crystalline iron oxides in the stability of soil aggregate-associated organic carbon in a rice–wheat cropping system

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ARTICLE INFO

Article history:
Received 22 January 2016
Received in revised form 18 May 2016
Accepted 19 May 2016
Available online xxxx

Keywords:
Soil organic carbon
Poorly crystalline iron oxides
Aggregates
Mineralization
Carbon sequestration

ABSTRACT

The stability of the soil organic carbon (SOC) in a rice–wheat rotation paddy soil has been of interest because of the global emission of greenhouse gases from the soil and the sequestration of atmospheric carbon dioxide in the soil, as well as in terms of soil fertility in subtropical Asia. The purpose of this study was to explore the relationship between poorly crystalline iron oxides (Feo) and SOC in bulk soil and soil aggregates. Soil samples were collected after a wheat harvest in June 2014 and after a rice harvest in October 2014 and were separated into large macroaggregates, small macroaggregates, microaggregates, and silt and clay by wet-sieving. The long-term (5-year) application of pig manure compost plus chemical fertilizer (NPKM) increased the SOC content more efficiently than other treatments. The specific carbon mineralization rate (SCMR, rate per unit SOC) increased in the following order: microaggregate < macroaggregate < silt and clay, suggesting that SOC in the microaggregates is more stable than in the silt and clay fraction. The Feo concentration was significantly positively correlated with the SOC content in the bulk soil ($P < 0.001$) and the soil aggregates ($P < 0.001$), but negatively correlated with the SCMR ($P < 0.001$). Compared with chemical fertilization alone (NPK), NPKM not only significantly improved soil aggregation but also efficiently activated the iron oxides as indicated by an increase in the aggregate mean-weight diameter (MWD) and the Feo content. Therefore, we suggested that the seasonal variation of wetting and drying drives the redox transformation of iron oxides and the mobility of Feo, and then affects the distribution of Feo in soil aggregates, which may endow paddy soil with a physico-chemical protection of SOC while application of organic fertilizers enhances this protection.

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1. Introduction

Soil organic carbon (SOC) represents a major terrestrial carbon (C) reservoir in the global C cycle and has long been of interest because of its importance in improving the productivity of ecosystems, the sustainability of agro-ecosystems and the fertility of croplands. The SOC content has been widely considered as an important index of soil quality (Reeves, 1997). Moreover, sequestration of atmospheric carbon dioxide into the soil as SOC has long been recognized as a promising measure for soil fertility promotion and global climate change mitigation (Lal, 2004). Fertilization has been considered as an effective nutrient management practice for the improvement of soil fertility. The positive effects of the long-term application of organic manure with or without chemical fertilizer on the SOC content have been well established (Yu et al., 2012b; Zhang et al., 2013).

The formation and stabilization of aggregates are essential for enhancing the SOC, and improving soil fertility and soil quality (Bronick and Lal, 2005; Six et al., 1998, 2000). Previous studies have shown that the turnover of the SOC is more rapid in the macroaggregates compared with the microaggregates and the silt and clay fraction (Ashman et al., 2003; Six et al., 2000) and further attributed this to the physical protection and physico-chemical stabilization of the SOC by the aggregate architecture in the microaggregates and the formation of an organo-mineral complex in the silt and clay fraction. However, Bossuyt et al. (2002) observed that a large amount of labile SOC principally accumulated in the microaggregates using a ^{14}C labeled plant residue. Rabbi et al. (2014) found that the specific C mineralization rate (rate per unit SOC) in macroaggregates was similar to that in microaggregates, while the adsorption of SOC on the silt and clay fraction decreased the specific C mineralization rate in different land uses. Yu et al. (2012a) proposed that the most stable SOC in soil aggregates was associated with the microaggregates rather than the silt and clay fraction. These findings suggest that the stability of SOC in soil aggregates still remains uncertain. Therefore, it is necessary to understand the stability of SOC in the different aggregate fractions and further

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http://dx.doi.org/10.1016/j.geoderma.2016.05.011
0016-7061/© 2016 Published by Elsevier B.V.

explore the underlying mechanisms of SOC stabilization in the soil, especially in paddy soils that experience seasonal waterlogged cultivation.

It has been well established that long-term application of organic fertilizer contributed to the formation and stabilization of soil aggregates and the increase of SOC in the bulk soil and the soil aggregates (Huang et al., 2010; Zhang et al., 2013). For example, Yu et al. (2012b) reported that the long-term (18-year) application of manure in a sandy loam soil improved the proportion of macroaggregates and increased the SOC concentration in all of the aggregate fractions. Six et al. (2000) indicated that no-tillage reduced the turnover of the macroaggregates compared with conventional tillage and thus improved microaggregate formation within the macroaggregates, and an increasing input of fresh organic matter to soils could maintain large proportions of macroaggregates. Huang et al. (2010) emphasized that long-term application of organic manure with or without chemical fertilizer in the red soil of subtropical China significantly improved macroaggregation and the SOC content while chemical fertilizer alone did not cause a significant impact on the SOC content in the aggregates. However, Lugato et al. (2010) and Yu et al. (2012b) reported that chemical fertilization alone also enhanced the SOC concentration, Lugato's group suggested that the sequestration of C in the microaggregates was mainly responsible, and Yu's group primarily implicated the preservation of C in the macroaggregates and the silt and clay fraction. Moreover, many studies have supported the accumulation of SOC in the silt and clay fraction or subfraction for the improvement of macroaggregation, which thus contributed to C sequestration (Liao et al., 2006; Razafimbelo et al., 2008; Yu et al., 2012b). The conflicting results in these reports indicate that much is still to be learned about the accumulation process of SOC in the soil as affected by different fertilizer regimes in a rice–wheat cropping system.

Soil minerals have been shown to play an important role in the preservation of the SOC (Kaiser and Guggenberger, 2003). It has been well established that the dynamics of SOC are closely related to the iron (Fe) and aluminum (Al) oxides in the soil (Torn et al., 1997). The positive role of Fe oxides in the accumulation and stabilization of SOC has long been an important interest to researchers (Wagai and Mayer, 2007; Zhang et al., 2013). Furthermore, Fe and Al oxides and/or carbonates can also act as inorganic binding agents and play important roles in soil aggregate formation (Six et al., 2004). Microbial mineralization of SOC, aggregate stability and soil Fe oxides are sensitive to seasonal variation of wetting and drying, for example, in soils cultivated with wheat and rice or paddy soils that comprise the largest man-made wetlands on earth (Kögel-Knabner et al., 2010). Therefore, understanding the underlying mechanism of SOC sequestration in soil aggregates subjected to a seasonal wetting-drying cycle and different fertilizer regimes may fill the knowledge gap concerning the long-term SOC storage in paddy soil.

The objectives of this study were: (1) to investigate the poorly crystalline Fe oxides (Feo) content by using acid ammonium oxalate selective dissolution procedures as affected by different fertilizer regimes and seasonal variations, (2) to explore the SOC and its stability in the soil aggregates by calculating the C mineralization of the aggregate fractions by means of a laboratory incubation experiment, and (3) to evaluate the effect of Feo on C sequestration and stabilization with a regression analysis.

2. Materials and methods

2.1. Experimental site and design

The field experiment was carried out beginning in 2010 in Jintan, Jiangsu Province, China (31°39′N, 119°28′E, 3 m a.s.l). This site has a typical subtropical monsoon climate and the annual average temperature and rainfall are 15 °C and 1100 mm, respectively. It has long been cultivated with winter wheat (*Triticum aestivum* L.) and summer rice (*Oryza sativa* L.), which represents one of the main cropping systems in China. The soil in this region has a silt loamy texture (5% sand, 70% silt and 25% clay), and is classified as a Gleyic Stagnic Anthrosol (IUSS Working Group WRB, 2015). The X-ray diffraction (XRD) analysis showed that the predominant clay minerals were kaolinite, illite and vermiculite (Fig. S1).

The fertilization experiment included five treatments replicated four times in a randomized complete block design. The treatments were: (1) control without fertilizer (CK); (2) complete chemical fertilizers (NPK), in which nitrogen, phosphorus and potassium fertilizers were applied at rates 240 kg N ha^{-1}, 120 kg P_2O_5 ha^{-1} and 100 kg K_2O ha^{-1} in the wheat season and 300 kg N ha^{-1}, 120 kg P_2O_5 ha^{-1} and 100 kg K_2O ha^{-1} in the rice season; (3) half of the levels of chemical fertilizers in the treatment NPK plus 6000 kg ha^{-1} pig manure compost (NPKM); (4) same levels of chemical fertilizers in the treatment NPK combined with crop straw (NPKS), in which rice straw was returned to soil before planting wheat and wheat straw was returned to soil before planting rice; and (5) one third of the levels of chemical fertilizers in the treatment NPK and 2000 kg ha^{-1} manure organic–inorganic compound fertilizer (NPKMOI). Each plot was 40 m^2 (5 × 8 m) and the straw was removed from fields except for the treatment NPKS. The chemical fertilizers were applied in the form of urea, calcium superphosphate and potassium chloride. Urea was applied both as a basal fertilizer before planting and as a supplementary fertilizer at the tillering and the panicle stage (4:3:3) in each cropping season, while other fertilizers were applied as basal fertilizers before planting summer rice in June (for harvest in late October) and before planting winter wheat in November (for harvest in late May).

2.2. Soil sampling and fractionation

Soil samples were taken from the surface layer (0–20 cm) at eight randomly selected points in each plot using a metal sampler (5 cm in diameter) in June and October 2014 after the wheat and rice harvests. Samples from the same plot were mixed to yield a composite sample, placed in a plastic box and immediately transported to the laboratory.

The field-moist soil samples were gently broken by hand along natural fractures to pass through an 8-mm sieve and then thoroughly homogenized. Stones, plant residues and organic debris visible to the naked eye were carefully identified and removed from the sieved soil with forceps. All of the samples were divided into three parts: one part was used to determine the soil moisture, another was air-dried for a soil properties analysis, and the remaining was used for soil aggregate separation.

Soil aggregate separation was conducted by wet-sieving following the method described by Elliott (1986). Briefly, a 100-g subsample was placed on a 2000-μm sieve and submerged in 1 cm of deionized water for 5 min to allow slaking to occur. After slaking, the soil was sieved by manually moving the sieve up and down 3 cm 50 times in 2 min. Floating litter was separated and set aside for recovery analysis. The material remaining on the sieve was transferred into a pre-weighed drying pan. The water and soil mixture was then passed through 250-μm and 53-μm sieves using the same procedure as described above. All materials that passed through the 53-μm sieve were centrifuged at 12,000 rpm for 15 min at 4 °C using pre-weighed 250-ml centrifuge tubes. The pellets were suspended in 100 ml deionized water with moderate stirring and centrifuged again. This procedure was repeated three times to avoid the transient adsorption of dissolved organic matter by the silt and clay fraction. Thus, four classes of aggregates were obtained: the large macroaggregates, (2000–8000 μm), the small macroaggregates, (250–2000 μm), the microaggregates, (53–250 μm), and, the silt and clay fraction, (<53 μm). After separation, each fraction was divided into three subsamples: one was used to determine the moisture content, another was air-dried for an analysis of the aggregate properties, and the rest was used for laboratory incubation.

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