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# Geoderma

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

# Long-term fertilization increases the temperature sensitivity of OC mineralization in soil aggregates of a highland agroecosystem



GEODERM

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#### ARTICLE INFO

Article history: Received 14 July 2015 Received in revised form 18 February 2016 Accepted 24 February 2016 Available online 10 March 2016

Keywords: Highland farmland Manure fertilizer Mineral fertilizer OC mineralization Soil water-stable aggregates Temperature sensitivity

# ABSTRACT

The mineralization of aggregate-associated organic carbon (OC) and the effect of long-term fertilization on its temperature sensitivity have not been addressed, but are critical for understanding OC sequestration and dynamics in agricultural soil. We measured the mineralization of OC in total soils and aggregates after 27 years of application of various fertilizers in a highland agroecosystem in central China and calculated the temperature sensitivity of OC mineralization. Our objectives were to understand the effects of long-term fertilization on OC mineralization in aggregates and its temperature sensitivity, and to determine the contribution of each aggregate size class to OC mineralization in total soils. Long-term fertilization decreased cumulative mineralized OC (Cmin) in <0.053 mm size class but increased C<sub>min</sub> in >2, 0.25-2, and 0.053-0.25 mm aggregates. The increases were smaller in treatments with mineral fertilizers than with manure or manure combined with mineral fertilizer. The temperature sensitivity of OC mineralization was decreased in >2 mm aggregate but increased in <2 mm aggregates and total soils by fertilization treatments. The OC mineralization in < 0.053 mm size class accounted for 36% of the OC mineralization in total soils. Manure or manure combined with mineral fertilizer had a higher potential to change the contribution of aggregates to OC mineralization in total soils. Our results suggest that OC mineralization in total soils was determined mainly by mineralization in the <0.053 mm size class of these highland agricultural soils, and long-term fertilization increased the temperature sensitivity of OC mineralization in total soils and <2 mm aggregates.

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# 1. Introduction

Soil organic carbon (OC) plays an important role in maintaining the productivity and yield stability of crops (Lal, 2004; Pan et al., 2009). As the most important and widely used practical approach to improve crop yields, fertilizers, both mineral and organic, have been widely applied throughout the world (Ju et al., 2009). Fertilization has great potential to increase OC content in agricultural soils and thus to influence OC dynamics (Metting et al., 2001; Lal, 2004; Wang et al., 2010). Soil OC has been predicted to increase on a global scale at a rate of 0.4–0.9 Pg C y<sup>-1</sup>, mainly due to the application of fertilizers (Metting et al., 2001; Lal, 2004; Lal, 2004). A synthesis analysis showed that long-term fertilization significantly increased OC content in top soils across mainland China, with a higher rate of increase in paddy soils (0.19 g kg<sup>-1</sup> y<sup>-1</sup>) than highland soils (0.13 g kg<sup>-1</sup> y<sup>-1</sup>), and the increases were larger from organic fertilizers (Wang et al., 2010). These significant increases

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might be due to the relatively lower soil OC content in cropland than natural vegetation.

Mineralization is the most important process of soil OC dynamics, and the effects of fertilization on OC mineralization has been widely studied to understand the stability of OC in fertilized soils (Bidisha et al., 2010; Zheng et al., 2012; Mohanty et al., 2013). For example, Mohanty et al. (2013) found that long-term integrated application of mineral and organic fertilizer significantly increased the potentially mineralizable OC in total soils of a 41-year rice-rice system in India. Similar results were reported in irrigated sandy soil in Germany (Bidisha et al., 2010) and red-earth paddy soil in China (Zheng et al., 2012). Despite a number of valuable studies from this perspective, the mechanism behind the effect of fertilization on OC mineralization has not been well addressed.

Aggregate-associated OC can respond quickly to soil management and can be used as an indicator of changes in soil structure and OC content (Nie et al., 2014; Six and Paustian, 2014; Wang et al., 2014). Longterm application of organic or combined organic/mineral fertilizers can significantly increase the contents and stocks of OC in soil aggregates, mainly due to a higher addition of manure, higher return of crop residues and root biomass (Chirinda et al., 2012; Liu et al., 2013; Ren et al., 2014), and lower loss of either original soil OC or fertilizerderived OC (Ren et al., 2014; Hao et al., 2016). Both fertilizer- and

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plant-derived OC after the application of fertilizer is mainly occluded in soil aggregates and is therefore protected from loss through physical or physicochemical mechanisms (Hassink, 1997; Six et al., 2000; Schöning et al., 2005; Bayer et al., 2006). Macro- and microaggregates generally provide physical protection by excluding decomposers from the micropores within the aggregates where the OC was incorporated with soil particles (Sollins et al., 1996; Hassink, 1997; Six et al., 2000; McCarthy et al., 2008), while fine soil particles provide physicochemical protection through the combination of OC with the fine particles (Hassink, 1997; Six et al., 2000; Schöning et al., 2005; Bayer et al., 2006). The OC associated with aggregates was therefore assumed to be stable. Only a few studies, however, have evaluated the effects of fertilization on OC mineralization in aggregates (e.g. Yu et al., 2012). Yu et al. (2012) showed that the application of compost and mineral fertilizer increased the rate of OC mineralization in aggregates of an intensively cultivated sandy loam soil in central China. The mineralization of OC within the aggregates and its response to long-term fertilization should thus be further addressed, which is critical for understanding the effects of longterm fertilization on OC mineralization in total soils and OC sequestration and dynamics in agricultural soil.

The mineralization of OC is strongly regulated by temperature and usually increases as an exponential function of temperature (Knorr et al., 2005; Froseth and Bleken, 2015). Temperature sensitivity (commonly referred to as  $Q_{10}$ , a metric that describes the proportional increase of mineralization rate with a 10 °C increase in temperature) has often been used to assess the response of OC mineralization rate to temperature changes and in carbon-cycling models at different temporal and spatial scales (Kirschbaum, 1995; Davidson and Janssens, 2006). OC mineralization, and therefore Q<sub>10</sub>, is controlled by the availability and quality of substrate and/or soil organic matter to decomposers (Gershenson et al., 2009; von Lutzow and Kogel-Knabner, 2009). For example, the reduction of OC mineralization has often been attributed to the depletion of readily decomposable substrate (Knorr et al., 2005; Steinweg et al., 2008). The quality of the soil OC also influences the response of OC mineralization to temperature. The temperature sensitivity of labile OC was found to be higher than (Giardina and Ryan, 2000), equivalent to (Fang et al., 2005; Conen et al., 2006), or lower than (Conant et al., 2008; Hartley and Ineson, 2008) that of resistant OC. Changes in the sources, composition, stability, and quality of soil OC due to long-term fertilization will influence substrate availability and quality, and thus affect Q<sub>10</sub>, in either total soils or aggregates. The temperature sensitivity of OC mineralization in aggregates and its response to long-term fertilization has not been well examined. Such information would be key to understanding the temperature sensitivity of soil OC and to determining the selection of parameters for modeling C cycling in agricultural systems.

In this study, we measured OC mineralization in total soils and water-stable aggregate size classes from a 27-year fertilization experiment in a highland agricultural ecosystem in central China. The measurements were conducted at 15 and 25 °C to determine the temperature sensitivity of the mineralization. The proportion of OC mineralization in each aggregate size class was calculated to assess their contributions to OC mineralization in total soils. The objectives were to determine:

- 1. whether OC mineralization and its temperature sensitivity  $(Q_{10})$  varied with aggregate size class in a highland agricultural ecosystem,
- 2. how long-term fertilization affects OC mineralization and  $\rm Q_{10}$  in total soils and aggregates, and
- how much the OC mineralization in aggregates contributes to OC mineralization in total soils.

## 2. Materials and methods

## 2.1. Study site

The field experiment was initiated in September 1984 in Changwu County, Shaanxi Province, China (35°12′N, 107°40′E). The study site has a warm-temperate, subhumid, continental climate. The mean annual temperature was 9.1 °C, the average frost-free period was 171 days, and the mean annual precipitation was 584 mm for 1984–2011. Rain falls mainly between June and September. The soil is a Calcaric Regosol of the FAO/UNESCO classification system, with a clay loam texture. The contents of soil particles >0.02 (sand), 0.02–0.002 (silt), and <0.002 mm (clay) were 38, 38, and 24%, respectively. The OC, N, and P contents of the soils at the start of the experiment were 6.09, 0.8, and 0.7 g kg<sup>-1</sup>, respectively.

#### 2.2. Experimental design and soil sampling

The long-term fertilization experiment in a winter wheat (Triticum aestivum L.) cropping system was conducted in 10.3  $\times$  6.5 m plots with three replicates from 1984. The fertilization treatments were an unfertilized control (CK), nitrogen (N), phosphorus (P), N + P(NP), manure (M), and N + P + M (NPM). Urea and superphosphate were used as sources of N and P, respectively. For all fertilized treatments, the N rate was 120 kg N ha $^{-1}$  y $^{-1}$ , the P rate was 26 kg P<sub>2</sub>O<sub>5</sub> ha $^{-1}$  y $^{-1}$ , and the M rate was 75 t  $ha^{-1}y^{-1}$ . Cattle manure was used, and total-OC, N, and P contents of the manure were 17.68, 1.97, and 0.97 g kg<sup>-1</sup>, respectively. The fertilizers were applied on the soil surface prior to seeding, and the land was plowed with a moldboard plow to a depth of ~20 cm. Winter wheat was sown in mid-September in rows 25 cm apart and harvested at ground level and removed from the plots in late June. The weeds in each plot were removed by hand. Routine practices of crop management for this region were used. Averaged across 27 years, N, P, NP, M, and NPM treatments resulted in -8, 22, 112, 106, and 150% changes in wheat yield, respectively, compared to the CK treatment (Hao et al., 2016).

Five random soil samples were collected in September 2011 from the 0–20 cm layer and combined to a composite sample in each plot with a tube auger 5 cm in diameter for the analysis of soil aggregates and OC and N contents. Visible pieces of organic material were removed, and the moist samples were air-dried in the laboratory.

## 2.3. Laboratory measurement and statistical analyses

Four size classes of aggregates were separated by wet-sieving through 2, 0.25, and 0.053-mm sieves following the procedures described by Cambardella and Elliott (1993). The aggregate samples were dried at 50–60 °C, weighed, and stored at room temperature. A subsample of air-dried, bulk soils from each plot was ground to pass through a 0.25-mm sieve for the measurement of OC content in total soils. OC content in total soils and aggregates was measured by the Walkley–Black method (Page et al., 1982). The efficiency of extraction of soil OC by this method generally varies between 60 and 86%, with a mean recovery of 76%, and a correction factor is commonly used to adjust the data to complete recovery (Wei et al., 2010). We considered the OC data set acceptable, due to the comparative nature of this work and the uniform analysis of all soils, so we did not adjust the data. The OC content in aggregates was reported in Table 1.

Ten grams of total soil or each aggregate size class (>2, 0.25–2, 0.053–0.25, and <0.25 mm) were adjusted to 60% field moisture capacity in 250-ml jars and were pre-incubated at 15 and 25 °C for five days to remove the flush of C mineralization caused by re-wetting, respectively. The samples were then incubated in the dark at corresponding temperatures. A 10-mL glass vial containing 5 mL of 1 M NaOH was placed in each jar to trap released CO<sub>2</sub>. Soil moisture during incubation was adjusted by adding deionized water to maintain the initial weight. Six control jars were prepared with no soil samples. The amount of CO<sub>2</sub> trapped was determined by titration against 0.5 M HCl. CO<sub>2</sub> was measured at 2, 4, 8, 15, 22, 29, 36, 43, 50, and 57 days of incubation. At each sampling, the vials were replaced with another set of vials containing fresh NaOH, and the jars were returned to the incubator. The caps of the jars were opened periodically during sampling to replenish the oxygen supply

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