



# Impact of agricultural fertilization practices on organo-mineral associations in four long-term field experiments: Implications for soil C sequestration

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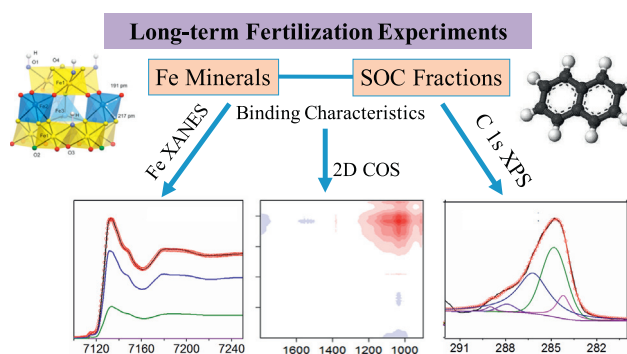
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## HIGHLIGHTS

- Fertilization regimes had contrasting effects on Fe–C associations in soils.
- Combined spectroscopic methods were applied to evaluate the species of Fe and C.
- Poorly crystalline Fe minerals were closely correlated to aromatic C accumulations.
- Long-term organic fertilization improved the capacity of soil organic carbon storage.

## GRAPHICAL ABSTRACT



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## ABSTRACT

Soil organic carbon (SOC) associated with minerals is considered to be one of the most fundamental long-term SOC storage strategies, but little research has integrated the organo-mineral complexes regulated by long-term fertilization. Here, soil samples under three fertilization treatments (Control, no fertilization; NPK, chemical nitrogen, phosphorus and potassium fertilization; NPKM, NPK plus manure) from four 23–34 years long-term field experiment sites across China were examined. Chemical analyses indicated that vigorous iron (Fe) mobilization could be regulated by long-term fertilization regimes. Meanwhile, Fe K-edge X-ray absorption near-edge fine structure (XANES) demonstrated that compared to NPK treated soils, NPKM treated soils contained significantly higher concentration of poorly crystalline ferrihydrite. Results from both the Fourier transform infrared combined with two-dimensional correlation spectroscopy analyses (FTIR-2DCOS) and C 1s X-ray photoelectron spectroscopy (XPS) revealed that aliphatic carbohydrate might play an important role in binding exogenous Fe (III) in all tested four soils. In addition, greater amounts of aromatic C (the most resistant soil C fraction) were under long-term treated NPKM than NPK soils. Furthermore, multiple regression analyses showed a significantly positive relationship between poorly crystalline Fe minerals and SOC or aromatic C. Such relationships indicated that aromatic functional groups had been attached to the poorly crystalline Fe minerals, which could also be

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protected from being transformed to the crystalline counterpart. In conclusion, results from our integrated spectroscopic analyses have evidenced greater improvement of both poorly crystalline Fe minerals and aromatic C in organically fertilized than in chemically fertilized soils.

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## 1. Introduction

Globally about 2344 Pg carbon (C) is stored in soils, which is three-fold greater than those in both the vegetation and atmosphere pools (Davidson and Janssens, 2006). Around 75% of this soil C pool is soil organic carbon (SOC), which plays a key role in the global C budget (Batjes, 1996). Generally, SOC is sensitive to environmental and soil conditions that could reflect the feedback of soil C cycle to climate change. Additionally, SOC is an indicator for soil fertility and quality, playing critical roles in enhancing agricultural productivity. It is of great importance to understand the mechanisms responsible for enhancing the stability and long-term storage of SOC strategies (Schmidt et al., 2011). Studies have showed that soil minerals control the SOC storage by regulating organic inputs and by protecting SOC from microbial attacks (Kleber et al., 2005; Basile-Doelsch et al., 2007). Iron (Fe) is the most abundant transitional metal on the Earth's surface and plays a particularly important role in SOC storage (Weber et al., 2006). Fe (hydr)oxides, which contain abundant of hydroxyl groups at the surfaces, can interactively establish stable organo-mineral associations by interacting with carboxyl groups of SOC (Kleber et al., 2005). Thus SOC is more resistant to degradation when adsorbed to Fe (hydr)oxides. Fe (hydr)oxide sorbent types, such as poorly crystalline ferrihydrite, crystalline goethite and lepidocrocite, play key roles in the SOC preservation because of their variable surface area and reactivity (Cismasu et al., 2016). Meanwhile, up to 21.5% of the organic C in soil is bound to the reactive poorly crystalline Fe minerals (Lalonde et al., 2012).

More than 37% of the Earth's land surface is consisted of agricultural land, where human activities strongly affect its SOC stock (The World Bank, 2015). Fertilization, as an important agronomic practice, has long been used to enhance plant growth, boost SOC stocks, and improve soil structure. Continuous organic manure inputs to soil have led to a higher SOC accumulation when compared to non-fertilization and chemical fertilization (Maillard and Angers, 2014). Such a higher C storage is usually due to a direct C input from the manure and an indirect C input through an increased net primary production (Maillard and Angers, 2014). Chemical fertilizers may also indirectly enhance the SOC storage with the crop residue input to soil (Halvorson et al., 1999; Galantini and Rosell, 2006; Tong et al., 2014). On the other hand, soil minerals could be affected by fertilization practices, as fertilization dramatically changes the soil's physical, chemical and biological properties. For instance, long-term application of organic manure increased soil macro-aggregate content for >54% (Tong et al., 2014). Greater macro-aggregate content in organically fertilized soils increased the potential for more anoxic environments for Fe redox microbes, thus supporting diverse and abundant anaerobic microbial communities (Reeve et al., 2010; Wen et al., 2018). These changes could greatly influence the Fe bioavailability and its interaction with other biologically important nutrients. For instance, soil cation exchange capacity positively related to the accumulation of SOC over a decade of intensive graze (Machmuller et al., 2015). In addition, root exudates had important effects on the formation of reactive minerals (Keiluweit et al., 2015).

Numerous long-term fertilization experiments have been established to address the impact of fertilizer amendments on crop production while providing valuable information to maintain soil fertility by examining changes in soil processes over time. However, information is scarce in depicting the mechanisms that could explain how fertilization regulates the association between soil Fe minerals and SOC accumulation. Besides, the soil water-dispersible colloids act as the linkage between geosphere and hydrosphere, representing the most reactive component in soils.

They typically consist of mixtures of soil minerals and natural organic matter, and are readily affected by fertilization regimes (Schumacher et al., 2005; Van Oost et al., 2007). A better knowledge of the interaction of Fe species with C functional groups in water-dispersible colloids from different fertilizer-treated soils may contribute to our understanding of soil C sequestration.

The Fourier transform infrared spectroscopy (FTIR) is a popular facility to address the organic ligands in various environments, which can differentiate both fluorescent and non-fluorescent substances in comparison with fluorescence excitation-emission matrix (EEM) spectroscopy. However, one-dimensional FTIR usually exhibits a variety of overlapped peaks because of the heterogeneous nature of an examined SOC. Combined with a two-dimensional correlation spectroscopy analyses (2DCOS), it is able to solve such peak overlapping problems by distributing the spectral intensity within a data set along a second dimension. This method could provide valuable information of the complexes of organic ligands with metals in soil dissolved organic matter (DOM) (Yu et al., 2012; Wen et al., 2014a). Recent innovations in the synchrotron-based X-ray absorption near-edge fine structure (XANES) spectroscopy, which is an element-specific technique and sensitive to both the oxidation state and the local structure of the absorber element (Prietzel et al., 2007), make it possible not only identify but also quantify, the mineralogy of Fe presented in soils. Since Fe phases in soils are highly complex, this tool is helpful in comparison with the Mössbauer spectroscopy that might mask magnetically weak phases (Huang et al., 2016). In addition, the X-ray photoelectron spectroscopy (XPS) can provide valuable information on the bonding state of C framework in the surface layers of soil particles, examine the inherently stable structures of SOC, and thus the nature of SOC transformations under different fertilization regimes (Mikutta et al., 2009; Xiao et al., 2015).

This study employed four long-term field sites locating in different regions (Gongzhuling, Shenyang, Urumqi, and Qiyang) of China with four contrasting soil types (black soil, Luvic Phaeozems; brown soil, Haplic Alisol; desert soil, Haplic Calcisol; and red soil, Ferralic Cambisol). All experiments have been performed over 23–34 years. Three soils from each site were examined under three fertilization treatments: (1) no fertilization control; (2) chemical nitrogen, phosphorus and potassium, NPK; and (3) NPK plus manure, NPKM. Several novel techniques including FTIR-2DCOS, XANES and XPS were combined to gain direct insight into the local coordination and characteristics of Fe minerals and their function in preserving SOC. Our objectives were to determine: (1) if the functional composition and speciation of Fe and C in soil colloids could be influenced by long-term fertilization; (2) which functional groups in the soil colloids C were preferentially binding with Fe oxides and (3) how long-term fertilization could affect the interaction between Fe matrix and C fraction in the soil colloids. These results would increase our knowledge of how agricultural fertilization practices could influence SOC by influencing the interaction between SOC and Fe oxides and provide insights into the turnover and preservation of SOC in agricultural soils.

## 2. Material and methods

This study consisted of four long-term (23–34 years) experimental sites on upland soils throughout the major grain-producing areas of China (Fig. 1). The location, climate, cropping regimes, and basic soil properties from these sites at the start of the field experiment were summarized in Table S1, which also identified the major soil types of these sites, based on the soil classification of the Food and Agriculture Organization (FAO) of the United Nations.

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