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Combined turnover of carbon and soil aggregates using rare earth oxides and isotopically labelled carbon as tracers

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ABSTRACT

This study used a combined tracer approach of isotopically labelled carbon (C) and rare earth oxides (REO) to determine soil aggregate transfer paths following input of organic matter. A model quantifying aggregate turnover rates over time was verified by a controlled incubation study. Four natural soil aggregate size ranges (<0.053 mm, 0.053–0.25 mm, 0.25–2 mm and 2–5 mm) were labelled with different REO tracers and packed to form a composite soil sample. The organic input was 1 mg $^{13}C\ g^{-1}$ soil of ^{13}C-labelled glucose. There were four treatments: i) soil without REO and ^{13}C as a control, ii) soil labelled with REO, iii) soil without REO but amended with ^{13}C-glucose, and iv) soil labelled with REO and amended with ^{13}C-glucose. Aggregate stability, REO concentrations, soil respiration and ^{13}C were measured after 0, 7, 14 and 28 days incubation. REOs were found to not impact microbial activity ($P > 0.05$). Based on the 84%–106% recovery of REOs after wet sieving of aggregates, and a close 1:1 relationship between measured aggregates and model predictions, REOs were found to be an effective tracer for studies of aggregate dynamics. A greater portion of aggregates transferred between neighbouring size fractions. The turnover rate was faster for macroaggregates than for microaggregates, and slowed down over the incubation time. The new C was accumulated more but decomposed faster in macroaggregates than in microaggregates. A positive relationship was observed between the ^{13}C concentration in aggregates and the aggregate turnover rate ($P < 0.05$). The relative change in each aggregate fraction generally followed an exponential growth over time in the formation direction and an exponential decay in the breakdown direction. We proposed a first order kinetic model for aggregate dynamics which can separate aggregate formation, stabilization and breakdown processes. This study demonstrates that REOs can track aggregate life cycles and provide unique and important information about the relationship between C cycling and aggregate turnover.

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1. Introduction

Soil aggregate dynamics involves aggregate formation, stabilization and breakdown processes (Oades, 1993; Six et al., 2004), largely controlled by biological processes in soil (Tisdall and Oades, 1982) and their interaction with physical processes such as wetting/drying, thawing/freezing, and tillage (Le Bissonnais, 1996; Díaz-Zorita et al., 2002). Although considerable research has explored soil aggregate dynamics, the life cycle of an aggregate and its impact on microbial mediated C cycling remains elusive. This has been addressed to some extent by the use of particle tracers, which have been applied in a few studies of soil aggregate dynamics. Over the past three decades, a number of particle tracers have been employed such a 1–3 mm ceramic spheres (Staricka et al., 1992) or ≈500 μm Dy_2O_3 labelled ceramic prills (Plante et al., 1999; Plante and McGill, 2002a). Due to the size of the tracers, however, these studies were limited to macroaggregate dynamics.

Biological processes driving soil aggregation operate over a much broader range of scales, so smaller tracers are needed. Rare earth oxides (REO) were proposed by Zhang et al. (2001) to trace soil aggregation and erosion, and subsequently employed by De Gryze et al. (2006) to track aggregation dynamics of artificial aggregates. REOs are very small (<5 μm in diameter), easily detected, have strong binding energy with soil mineral surfaces and physicochemical properties similar to soil particles. The aim of De Gryze et al.'s (2006) study was to explore the transfer of soil between

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different aggregate size fractions collected from wet sieving. REO concentrations detected much more rapid macroaggregate turnover than microaggregate turnover, and greater stabilization of soil in microaggregates. They also observed that REOs did not affect soil respiration, so could potentially be employed to investigate aggregate dynamics related to C cycling and inputs.

Modelling soil aggregate dynamics after organic matter input was reported in a few studies. Segoli et al. (2013) used data from De Gryze et al. (2006) to develop the AggModel that simulates macroaggregate and microaggregate dynamics. Another model of aggregate dynamics after organic input was the conceptual CAST model presented by Stamati et al. (2013). An earlier model by Kay et al. (1988) simulated aggregate formation and breakdown using first order kinetics, which they tested with previously published data of aggregate stability. De Gryze et al. (2005) also reported that an exponential equation fitted >2 mm aggregate dynamics data better than other equations. However, these four models did not disentangle the differences in aggregate formation, stabilization and breakdown processes. In contrast to a first order kinetic model, Monnier (1965) proposed a conceptual model that aggregate stability increases immediately after organic amendment and thereafter decreases as the organic matter decomposes. To examine the impact of quality of amended organic substances, Abiven et al. (2008) applied the Monnier's model to simulate changes in aggregate stability from four different organic inputs under controlled laboratory and field conditions. From a review of 48 sets of data published since the 1940s, Abiven et al. (2009) found no clear relationship between aggregate dynamics and organic matter decomposition. They argued that organic materials have different biochemical origins, direct abiotic impacts to aggregation, and varying impacts on aggregate breakdown mechanisms. Such complex processes driving how soil aggregates respond to the decomposition of organic matter and *vice versa* needs much more research.

A useful tool for such research could be REOs as tracers. In this study we develop the approach presented by De Gryze et al. (2006) and improve their model of incubation effect on soil aggregate turnover rate. To track biological transformation of C at the same time as aggregate turnover we employed a double-labelling approach with REOs to determine aggregate dynamics and ^{13}C-labelled glucose to determine new C distribution in aggregates. Our objectives were to propose a new method for labelling REO with natural aggregates and to develop a new model to determine aggregate turnover rate and time after organic input. This study will improve our understanding of the dynamics of soil aggregates and their feedback on C physical protection in soil. With current approaches, the understanding of soil aggregate, C dynamics and physical protection has been assembled from indirect assessments and the insightful interpretation of disparate data. Our new approach could trace pathways directly, providing hitherto unattainable, quantitative data to support a plethora of soil aggregation studies and soil C turnover modelling.

2. Materials and methods

2.1. The soil

The soil in this study was sampled from an arable field planted with peanut (*Arachis hypogaea* L.) at the Red Soil Ecological Experimental Research Station, Chinese Academy of Sciences (28°15′ N, 116° 55′ E), Yingtan, Jiangxi Province. The area has a typical warm and humid subtropical monsoon climate with an annual rainfall of 1795 mm and an annual mean temperature of 17.8 °C. The soil is derived from Quaternary red clay, classified as Acrisols (FAO, 2014), with mineralogy dominated by highly weathered 1:1 clays and iron oxides. Globally these soils are highly significant as they support food production for 40% of China's population, are commonplace in India and found in many other subtropical climates including Africa. The soil samples were collected from the surface 0–20 cm layer, air-dried and broken by hand to pass through a 5 mm sieve. Before imposing experimental treatments, the soil contained 7.50 g kg^{-1} soil organic C, −21.3‰ $\delta^{13}C$, 0.85 g kg^{-1} total N, and 38.9% clay content.

2.2. Rare earth oxides (REO) labelled aggregates

Four REOs (Lanthanum oxide, La_2O_3; Samarium oxide, Sm_2O_3; Neodymium oxide, Nd_2O_3 and Gadolinium oxide, Gd_2O_3) were purchased from Shanghai Heli Rare Earth Material Company, P.R. China. The purity of each oxide is >99.9%. The median diameter of the powder (D_{50}) ranged from 3.2 to 5.2 μm, and the density between 6.5 and 7.6 Mg m^{-3}. The background levels of the four REOs in the investigated soil were 2.59 mg kg^{-1} Gd_2O_3, 26.9 mg kg^{-1} La_2O_3, 5.35 mg kg^{-1} Sm_2O_3, 21.9 mg kg^{-1} Nd_2O_3.

Each REO tracer was added by wet mixing to a separate batch of bulk soil (<5 mm) at a rate of 500 mg kg^{-1}, in addition to a control without a tracer added. This involved first dispersing 3.33 mg ml^{-1} of REO in deionized water by vortex mixing. The soil was continuously mixed and sprayed slowly with the REO suspended in water at a rate of 150 ml kg^{-1} air dry soil. The initial sieving of the soil followed by spraying with an REO water suspension results in the preferential deposition of REOs on the surface of soil aggregates. The wetted soil was then stored at 4 °C for 7 days to allow water equilibration with minimal microbial activity. Afterwards, the soil was oven-dried at 40°C for 48 h, and broken down by hand to pass through a 5 mm sieve. The <5 mm soil was separated into four fractions by Elliott's (1986) method: large macroaggregates (2–5 mm), small macroaggregates (0.25–2 mm), microaggregates (0.053–0.25 mm), and silt and clay sized aggregates (<0.053 mm), indicated by A, B, C and D fractions, respectively. The fractioned aggregates were oven-dried at 40 °C and weighed.

Aggregates from A-D fractions were recombined into a soil in which each aggregate fraction contained a different REO tracer. One batch of bulk soil was mixed thoroughly for use in the REO labelling studies. The investigated soil was composed of A fraction labelled by Gd_2O_3 with 337 ± 8 mg kg^{-1}, B fraction labelled by La_2O_3 with 342 ± 8 mg kg^{-1}, C fraction labelled by Sm_2O_3 with 425 ± 9 mg kg^{-1}, and D fraction labelled by Nd_2O_3 with 577 ± 18 mg kg^{-1}. The protocol for combining four REO labelled aggregates into a soil is illustrated in Fig. 1. The control was subjected to the same procedure except without REO addition.

2.3. Soil incubation

Four treatments were designed as follows i) the soil without REO and ^{13}C as a control (Control treatment), ii) the soil labelled by REO (REO treatment), iii) the soil without REO but with added ^{13}C-glucose (^{13}C treatment), iv) the soil labelled with REO and added with ^{13}C-glucose (^{13}C + REO treatment). The treatments were established by placing 50 g soil in a 100 ml plastic bottle and amending with 7.5 ml ^{13}C-labelled glucose (99 atom% ^{13}C) for the ^{13}C and ^{13}C + REO treatments at an incorporation rate of 1.0 mg ^{13}C g^{-1} soil (equivalent to a glucose concentration in the added solution of 16.7 mg ml^{-1}). For the REO and Control treatments 7.5 ml of deionized water was added to the soil. Soils were then incubated for 28 days at 25°C with moisture kept at 60% water-holding capacity (0.15 g water g^{-1} soil) by regularly adding deionized water to maintain a constant weight. Aggregate dynamics were measured by destructively harvesting batches of soil on 0, 7, 14, and 28 day of

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