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SoilChip-XPS integrated technique to study formation of soil biogeochemical interfaces

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ABSTRACT

Many soil functions are modulated by processes at soil biogeochemical interfaces (BGIs). However, characterizing the elemental dynamics at BGIs is hampered by the heterogeneity of soil microenvironments. In order to investigate the processes of BGI formation in an upland soil (Mollisol) and a paddy soil (Oxisol), we developed a SoilChip method by assembling dispersed soil particles onto homogeneous 800-μm-diameter microarray chips and then submerging them in a solution that contained dissolved organic matter (OM) extracted from one of the two soils. The chips with Mollisol particles were incubated at 95–100% humidity, whereas the chips with Oxisol particles were incubated at 100% humidity. Dynamics of individual elements at the soils' BGIs were quantitatively determined using X-ray photoelectron spectroscopy (XPS). Distinct differences in the soil-microbe complexes and elemental dynamics between the Mollisol and Oxisol BGIs suggested that the formation of specific BGIs resulted from the complex interaction of physical, chemical, and microbial processes. By integrating the SoilChip and XPS, it was possible to elucidate the dynamic formation of the two different soil BGIs under standardized conditions. Therefore, the SoilChip method is a promising tool for investigating micro-ecological processes in soil.

1. Introduction

Soil biogeochemical interfaces (BGI) within the three-dimensional soil structure are considered unique and represent complex hotspots of microbe-soil interactions (McClain et al., 2003; Chorover et al., 2007; Totsche et al., 2010). The formation of BGIs can alter the physical-chemical properties of soil, such as its wettability, for which changes can result in the formation of preferential water flow, thus affecting the transport and fate of dissolved compounds, clay minerals, and microorganisms (Goebel et al., 2011). Furthermore, in addition to the association of microbes with the BGIs of primary and secondary particles, microbes also transform and decompose organic matter (OM) and secrete extracellular polymeric substances (EPS) to modify their environment (Gleixner, 2013; Kleber et al., 2015).

The chemical extraction methods that have traditionally been used to investigate the biogeochemical behavior of elements obscure the details of microbe-soil interactions (Lehmann and Kleber, 2015). Furthermore, soil fractionation techniques, which are based on the operational separation procedures for bulk soil samples, will also homogenized the microenvironment of the

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microbial habitat, and are not suited to investigate the linkages between habitat architecture and biological functioning (Six et al., 2004; Young and Crawford, 2004). Recently, advances in non-destructive techniques for soil microaggregate analysis have enriched our understanding of elemental cycling traits in soil at the microscale (Wan et al., 2007; Remusat et al., 2012). For example, the use of nanoscale secondary ion mass spectrometry (NanoSIMS) revealed that fresh OM preferentially attaches to partially rough surfaces (Vogel et al., 2014). Lehmann et al. (2008) used near-edge X-ray fine structure spectroscopy (NEXAFS) to illustrate that the nanoscale carbon species distribution of OM exhibited significant heterogeneity at specific mineral surfaces. However, most soil surface characteristics are investigated using one-time snapshot measurements, which are of limited use for assessing the dynamic mechanisms of multi-element cycling and microbe-mediated transformations at the microsite scale (Lützow et al., 2006; Lehmann and Kleber, 2015). Thus, mineral-OM-microbe interactions at the soil BGI are still poorly understood (Huang et al., 2005; Chorover et al., 2007; Totsche et al., 2010; Mueller et al., 2013; Sparks, 2015). There is growing concern regarding the importance of microscale and dynamic processes at the soil BGI, which could unravel the linkage between physical-chemical and biological processes within the continuum of soil-microbe interactions (O'Donnell et al., 2007; Kuzyakov et al., 2009; Remusat et al., 2012).

The development of “lab on a chip” techniques, such as microfluidics and microfabrication, has facilitated the microscale confinement of small objects, such as bacterial populations and cells, and has provided powerful tools for exploring microscale and dynamic processes (Weibel et al., 2007; Wessel et al., 2013). For example, Kim et al. (2008) used microfluidics to co-culture microbes and clearly demonstrated that a proper spatial distribution stabilized multi-species bacterial communities. In addition, Valiei et al. (2012) used microfluidic porous media mimics, in which an array of polydimethylsiloxane (PDMS) microposts were embedded in a microchannel, to monitor the behavior of microbe transportation in a soil pore system and found that hydrodynamics govern the formation, morphology, and distribution of biofilm streamers. Moreover, novel devices such as the RootChip and PlantChip, which take advantage of miniaturization for handling small volumes of liquids, have been developed for plant cell analysis, thus facilitating large-scale investigations of root metabolism and signaling (Grossmann et al., 2011). These applications suggest that “lab on a chip” techniques could also be useful for mimicking soil processes.

Inspired by the complex and adaptive nature of soils, in which mineral particles, OM, and microbes self-organize into complex aggregates, and the structural dynamic evolves with changing microenvironments (e.g., available C, nutrients, or water; Oades and Waters, 1991; Young and Crawford, 2004; Jozefaciuk and Czachor, 2014), we applied a controllable microfluidic method to reconstruct a soil suspension, in which all the bulk soil components were included, onto homogeneous microarray chips, which were then submerged in dissolved organic matter (DOM) to initiate soil BGI processes. For the tracking of multi-element dynamics at the soil surface, the SoilChip was coupled with X-ray photoelectron spectroscopy (XPS), a sensitive surface detecting method with a penetrating depth of <5–10 nm, which is perfectly suited for investigating mineral surfaces (Amelung et al., 2002; Woche et al., 2017). We tracked temporal dynamics of the BGI formation and modification through the different sampling time during the 21-d incubation and followed by XPS measurements.

Our objective was to develop a SoilChip method to mimic a soil microenvironment that could be used to assess the heterogeneous and temporally dynamic properties of microinterfaces. By comparing two different soils, a Mollisol and an Oxisol, we aimed to elucidate the effects of different interface and solute compositions on BGI formation.

2. Materials and methods

2.1. Soil sample characterization

Two contrasting soil samples were used for our experiments: a Mollisol from a long-term fertilization trial at Gongzhuling, Jilin Province, China, and an Oxisol from a continuous (>30 years) rice plantation at the Taoyuan Station of Agro-ecology Research in Hunan. The top layer (0–20 cm depth) of both soils was sampled, air dried in the laboratory for one week, gently crushed with a wood rolling pin, and then passed through a 2-mm sieve. Visible organic residues were then removed from the samples using forceps, after which the physicochemical properties of the soil samples were determined using standard procedures (Li et al., 2008; Chen et al., 2012; Table S1).

2.2. Soil suspension preparation and DOM extraction

The two soils were pre-incubated in the dark for one week at 45% water holding capacity and a constant temperature of 25 °C, in order to allow the microorganisms to recover to their normal activity levels. After pre-incubation, each soil was divided into three parts, for 1) assembly of soil microaggregates on the SoilChip, 2) DOM extraction, and 3) micro interface analysis of the soil aggregate. Fresh field soil (1 g d.w. equivalent) was vortexed (800 r min^{-1}) in 3 ml distilled water for 2 min, sonicated at 60 W min^{-1} for 3 min (Biosafer1000; Biosafer, Nanjing, China) to disperse soil macroaggregates, and passed through a <0.25-mm sieve to remove particulate OM. The particle size distribution of the suspensions was determined using a Mastersizer 3000 (Malvern Instruments, Worcestershire, UK). To strengthen the structure of the microaggregates assembled on the SoilChip, polyvinyl alcohol (PVA) was added to the soil suspensions (500:1 mass ratio of PVA and soil) to act as an organic cement (Cai et al., 2013).

To construct the soil microenvironment, DOM solutions were extracted from the two incubated soils and used to cover the soil microarrays. The solutions were prepared by mixing fresh soil (1 g d.w. equivalent) with double distilled H_2O at a soil:water ratio of 1:2 (volume:mass), sonicating the samples at 120 W min^{-1} for 3 min, obtaining supernatants by centrifugation at 8800 × g for 4 min, and pressure filtering the supernatants through 0.2-μm polysulfone membranes (Whatman, Inc., Springfield Mill, UK) to remove microbial bio- and necromass and clay particles. The organic carbon (OC) concentrations of the DOM solutions were measured using a TOC-5050A total organic C analyzer (Shimadzu, Kyoto, Japan).

2.3. SoilChip construction

To obtain a controllable and uniform soil interface, microfluidic devices with hydrophilic microarrays for depositing the soil particles were produced using oxygen plasma modification (Li et al., 2016). Briefly, a stamp of PDMS with 800 μm-diameter cylindrical wells was produced using standard soft lithography methods (Fig. 1a; Weibel et al., 2007). Then, the PDMS stamp and a clean glass slide were modified into super hydrophilic interfaces using low-oxygen plasma exposure treatment for 1 min (PDC-GC-M; Weike Spectrum Instrumental Technology Development Co., Ltd., Chengdu, China). Thereafter, the PMDS was quickly sealed to the glass and torn off. Areas of the glass that were coated with PDMS film became hydrophobic, whereas those without PDMS remained