

## Applications of Cavity Ring-Down Spectroscopy to carbon, nitrogen and water cycling in soil

DENNIS, Kate<sup>1\*</sup> ; HUANG, Kuan<sup>1</sup>

<sup>1</sup>Picarro, Inc.

Over the past five years, advances in cavity-enhanced absorption spectroscopy have fundamentally changed the way soil scientists study carbon, nitrogen and water cycling between the subsurface, plants and the atmosphere. With the continuous evolution of performance, including high precision, low drift and part-per-billion resolution, along with compact, field-deployable and easy-to-use instruments, scientists are increasingly able to leave their labs and make measurements directly in the field. Here we describe how Picarro's Cavity Ring-Down Spectroscopy technology has been applied to a number of soil applications, including (i) the determination of soil flux measurements by coupling newly developed real-time software with simultaneous measurements of CO<sub>2</sub>, CH<sub>4</sub>, N<sub>2</sub>O, NH<sub>3</sub> and H<sub>2</sub>O from closed-loop, recirculated soil chambers; (ii) partitioning water loss from evaporation versus transpiration in arid environments using in-situ measurements of ambient atmospheric water vapor measurements, soil water isotopes and the isotopic signature of local transpiration; and (iii) using in-situ measurements of d<sup>15</sup>N and the site preference of <sup>15</sup>N in N<sub>2</sub>O to constrain N<sub>2</sub>O emissions in agricultural settings.

Keywords: CRDS, soil flux, nitrogen isotopes, evapotranspiration

## The effect of the feedback cycle between the soil organic carbon and the soil hydrologic and thermal dynamics

ISE, Takeshi<sup>1\*</sup> ; MORI, Kensuke<sup>2</sup>

<sup>1</sup>Field Science Education and Research Center, Kyoto University, <sup>2</sup>Department of Geomatics Engineering, University of Calgary

Biogeochemical feedback processes between soil organic carbon (SOC) in high-latitude organic soils and climate change is of great concern for projecting future climate. More accurate models of the SOC stock and its dynamics in organic soil are of increasing importance. As a first step toward creating a soil model that accurately represents SOC dynamics, we have created the Physical and Biogeochemical Soil Dynamics Model (PB-SDM) that couples a land surface model with a SOC dynamics model to simulate the feedback cycle of SOC accumulation and thermal hydrological dynamics of high-latitude soils. The model successfully simulated soil temperatures for observed data from a boreal forest near Fairbanks, and 2000 year simulations indicated that the effect of the feedback cycle of SOC accumulation on soil thickness would result in a significant differences in the amount of SOC.

Although it is still under development, the PB-SDM showed the significance of one of the feedback processes in high-latitude organic soil dynamics. Further development of the model has the potential to provide more insights into organic soil feedbacks in response to climate change.

Keywords: biogeochemistry, peat, climate change, soil organic carbon, boreal forest

## Global patterns of soil microbial carbon, nitrogen and phosphorus stoichiometry in terrestrial ecosystems

ASADA, Kei<sup>1\*</sup> ; KANDA, Takashi<sup>2</sup> ; ASANO, Maki<sup>2</sup> ; EGUCHI, Sadao<sup>1</sup>

<sup>1</sup>National Institute for Agro-Environmental Sciences, <sup>2</sup>The Graduate School Life and Environmental Sciences, University of Tsukuba

Incorporating site-specific microbial processes may improve models of carbon (C) and nutrient cycling across terrestrial ecosystems. To better understand the concentrations and stoichiometry of C, nitrogen (N) and phosphorus (P) in soil microbial biomass, we compiled and analyzed the existing published data on microbial C, N and P in soils spanning the global range of land-use types (forest, grassland, paddy, upland and orchard) and soil types based on Harmonized World Soil Database (FAO/IIASA/ISRIC/ISSCAS/JRC, 2012). It was found that the microbial stoichiometry had some flexibility under various environmental conditions. Flooded paddy soil had the highest C:N ratio of soil microbes among the global land-use types while C:P and N:P ratios highlighted the differences between forest and grassland, following upland field, and paddy or orchard fields. Agricultural soils (except paddy) had significantly lower C, N and P concentrations in soil microbial biomass than natural land-use, indicating large anthropogenic effects (e.g., land management). The spatial patterns of microbial-C and nutrient ratios differed considerably among soil types. For global microbial C:N ratio, the geometric mean varied from maximum 12.7 for Phaeozems to minimum 5.8 for Podzols. The microbial N:P ratio for Andosols was significantly higher than other soils. The consistency of this pattern in plant-soil-microbe ecosystems supports that P is often the major limiting element for Andosols. Meanwhile, higher concentrations of microbial-N and P may relate to high soil water contents, i.e., low permeability soils of Gleysols and Fluvisols. In this study, we provide more reliable parameters to determine soil microbial properties especially in agricultural land-use, i.e., Andosols mainly used for upland fields and Fluvisols used for paddy fields, as there are no existing data available.

Keywords: Andosols, Harmonized World Soil Database, Land-use, Global scale analysis, Terrestrial ecosystem, Paddy fields

## Decomposing global soil carbon projection uncertainties in ISI-MIP study

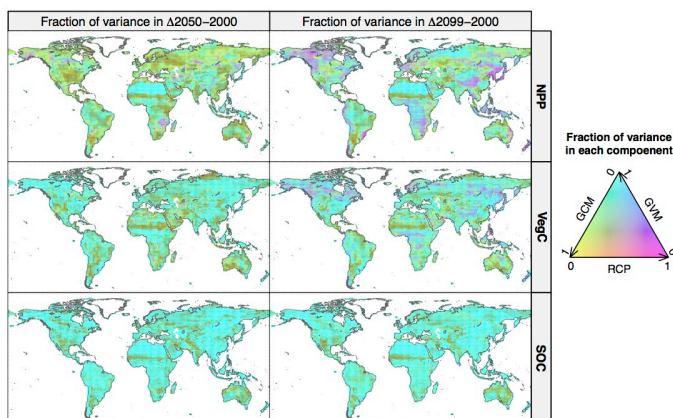
NISHINA, Kazuya<sup>1\*</sup>; ITO, Akihiko<sup>1</sup>

<sup>1</sup>National Institute for Environmental Studies

In earth system processes, because of the vast carbon pool of soil organic carbon (SOC), the behavior of SOC is the key to understanding the feedback of terrestrial ecosystems to atmospheric CO<sub>2</sub> concentration in a warmer world. There seem to be still large uncertainties in SOC projection. Therefore, how the uncertainties in SOC projection matters in future C projection and which uncertainty sources cause SOC projection uncertainty should be clarified to reduce SOC projection uncertainties. In this study, we performed simulations using six global vegetation models (GVM) using climate projections based on five climate models (GCM) forced by four RCP-based atmospheric concentration scenarios, aiming at specifying the relative uncertainty in the projection of global SOC stocks from global and regional perspectives.

At the end of the simulation period (2099), global  $\Delta$ net primary production ranged from -7.0 to 54.3 Pg-C Year<sup>-1</sup>, global  $\Delta$ vegetation biomass C ranged from -27 to 543 Pg-C Year<sup>-1</sup>, and global  $\Delta$ SOC ranged from -195 to 471 Pg-C Year<sup>-1</sup> in the entire simulation set. Thus, SOC projection uncertainty was relatively large compared to above biomass changes. We conducted ANOVA to the changes in NPP, VegC, and SOC as factors to be RCP, GCM, and GVM in global and regional scale, which enable us to know relative importance of these factors to changes in C. For  $\Delta$ NPP, the GCM uncertainty dominated before the year 2020, and the RCP uncertainty increased and dominated after 2040. The GVM uncertainties were approximately 20% in most of the simulation period. GVM dominate uncertainties (60% and 90%, respectively) rather than climate driving scenarios (i.e., GCM and RCP) in the global  $\Delta$ VegC, and  $\Delta$ SOC projections. In addition, we found that the contributions of each uncertainty source were spatio-temporally heterogeneous and differed among the GVM variables (Fig. 1). These results indicated to improve the SOC process in GVM is essential to reduce global C projection uncertainty. In the poster presentation, we will discuss about the difference in SOC processes of GVM in detail.

Figure 1. Relative importance of each uncertainty source to  $\Delta$ NPP,  $\Delta$ VegC, and  $\Delta$ SOC (from Nishina et al., 2014 in ESDD)



## Accumulation processes of organic carbon in peat deposit in a tropical mangrove forest on Pohnpei Island, FSM.

ONO, Kenji<sup>1\*</sup> ; HIRADATE, Syuntaro<sup>2</sup> ; FUJIMOTO, Kiyoshi<sup>3</sup> ; HIRATA, Yasumasa<sup>1</sup> ; HIRAIDE, Masakazu<sup>1</sup> ; TABUCHI, Ryyuichi<sup>1</sup> ; LIHPAI, Saimon<sup>4</sup>

<sup>1</sup>Forestry and Forest Products Research Institute, <sup>2</sup>National Institute for Agro-Environmental Sciences, <sup>3</sup>Nanzan University, <sup>4</sup>Department of Land and Natural Resources, Pohnpei State Government

To quantitatively evaluate organic carbon accumulation processes in peat deposits on a tropical mangrove ecosystems, solid-state <sup>13</sup>C cross-polarization and magic angle spinning nuclear magnetic resonance (CPMAS NMR) signals were monitored to determine the organic carbon composition of humified leaf and root, which account for a large majority of mangrove-produced litters and mangrove peat in a coral reef-type *Rhizophora* forest on Pohnpei Island, Federated States of Micronesia. Radiocarbon dating was also used to estimate the average turnover times of peat deposits at each depth. The mass loss rate of mangrove leaves during humification was much higher than that of roots. Mass loss rates of mangrove leaves and roots are expected to be affected by their varying chemical characteristics and the different aerobic/hydrological conditions present in the two litter types during humification processes. The decomposability of individual organic carbon components also varied markedly between leaf and root litters. Significant increases in aryl-C/O-alkyl-C and aliphatic-C/O-alkyl-C ratios and minor increases in aryl-C/aliphatic-C ratio during the humification of leaves implied that only the O-alkyl-C component was relatively labile compared with aryl- and aliphatic-C, and that the decomposability of aliphatic-C was also slightly higher than that of aryl-C, while the difference was not significant in leaf litters on the forest floor. Regarding roots, a stable aryl-C/O-alkyl-C ratio during humification suggests that the decomposability of aryl- and O-alkyl-C components did not differ greatly in the peat deposit, while the concomitant minor increase in the aliphatic-/O-alkyl-C ratio and the substantial decrease in the aryl-/aliphatic-C ratio with humification imply that aliphatic-C was more recalcitrant than aryl- and O-alkyl-C in the peat. Conversely, the compositional properties of organic carbon and the ages of <sup>14</sup>C of the peat deposit were quite homogenous and relatively modern throughout the peat profile, suggesting that large amount of mangrove roots penetrate up to at least 80-cm depth. These findings provide quantitative and qualitative insights into the potential importance of very high production of mangrove fine roots for organic carbon accumulation in peat in tropical mangrove ecosystems.

Keywords: peat deposit, organic carbon, <sup>14</sup>C dating, coral reef-type mangrove forest, solid-state <sup>13</sup>C CPMAS NMR

## Carbon cycle at different succession stages of agroforestry in Tome-Acu, Para, Brazil

KOBATA, Yuji<sup>1</sup> ; KIMURA, Sonoko dorothea<sup>1\*</sup> ; YAMADA, Masaaki<sup>1</sup> ; IRAE, Amaral g<sup>2</sup> ;  
UMEMURA, Helio makoto<sup>1</sup> ; DOS SANTOS, Dinaldo antonio<sup>3</sup>

<sup>1</sup>Tokyo University of Agriculture and Technology, Graduate School of Agriculture, <sup>2</sup>Sao Paulo State University, Botucatu,  
<sup>3</sup>CAMTA

### Introduction

In Tome-Acu, Para, Brazil, farmers plant crops to establish a series of successive harvests that is called Agroforestry System of Tome-Acu (SAFTA: Subler, 1993; Tanaka, 1997). Its sequence is similar to natural succession and farmers can use the fields for a long period (Subler and Uhl, 1990; Subler, 1993; Yamada and Gholz, 2002). Based on farmers experience, the mechanism of SAFTA has not been well documented. Especially the change during the succession or the effect of the change on nutrients and carbon (C) flow has not been documented. Thus, the objective of this study was to clarify the change in C flow during succession and its mechanism to clarify the advantage of SAFTA.

### Method

Three different succession stages were selected; since 2008 (6 years old; 6YO), since 2002 (12YO) and since 1980 (34YO). The C flow was analyzed by measuring the C abundance in (1) aboveground biomass, (2) soil, (3) litter and (4) yield and (5) the carbon dioxide (CO<sub>2</sub>) emission from soil. In addition to the agroforestry field, abundance of C in soil and litter as well as soil respiration were monitored in a secondary forest. The measurement was conducted from September 2012 to July 2014. The C balance was calculated by the difference of all input (aboveground biomass increase, litter and harvested yield) and output (soil respiration and yield residue).

### Result and Discussion

The increase of aboveground biomass of cocoa from 2013 to 2014 was 3.2 kg tree<sup>-1</sup> in 6YO and significantly larger than other stages (1.5 and 1.9 for 12YO and 34YO, respectively) The yield of cocoa was highest in 12YO and contained 3.3 kg-C tree<sup>-1</sup>. The yield in 6 and 34YO were almost same with 1.5-1.9 kg-C tree<sup>-1</sup>. Litter was highest in secondary forest with 8.2 Mg C ha<sup>-1</sup> year<sup>-1</sup>. The litter of 6YO, 12YO and 34YO increased with the age, and were 4.6, 5.6 and 7.1 Mg C ha<sup>-1</sup> year<sup>-1</sup>, respectively.

Soil carbon stock from 0 to 30 cm depth was 109.8 Mg ha<sup>-1</sup> in secondary forest, those for 6YO, 12YO and 34YO were 57.4, 77.7 and 101.3 Mg ha<sup>-1</sup>, respectively. There was no significant difference among them. But as the agroforestry stages progressed, the amount of litter fall and soil carbon tended to increased.

Soil respiration rate in litter removed chamber (L-) in secondary forest was 129 mg CO<sub>2</sub>-C m<sup>-2</sup> h<sup>-1</sup>, those for 6YO, 12YO and 34YO were 81, 84 and 92 mg CO<sub>2</sub>-C m<sup>-2</sup> h<sup>-1</sup>, respectively. Soil respiration rate in chamber with litter (L+) in secondary forest was 176 mg CO<sub>2</sub>-C m<sup>-2</sup> h<sup>-1</sup>, those for 6YO, 12YO and 34YO were 77, 104 and 113 mg CO<sub>2</sub>-C m<sup>-2</sup> h<sup>-1</sup>, respectively. The soil respiration rate in removed litter chamber was significantly lower than the chamber with litter in each site except 6YO. The respiration is the sum of root respiration and soil organic matter decomposition. The high soil respiration rate in secondary forest can be attributed to the litter fall as well as the high amount of roots.

Soil respiration rates in this study were similar to results reported for tropical forest in Brazil (59-139 mg CO<sub>2</sub>-C m<sup>-2</sup> h<sup>-1</sup> (Fernandes et al. 2002; Davidson et al. 2000), but lower than that of pasture in Brazil (117-317 mg CO<sub>2</sub>-C m<sup>-2</sup> h<sup>-1</sup> (Fernandes et al. 2002)). It was suggested that carbon turnover in agroforestry is similar to forest and harder to decompose compared with herbaceous plant in pasture.

As a consequence, the C balance in 6YO, 12YO, 34YO were 4.1, 4.5, 3.9 ton C ha<sup>-1</sup> year<sup>-1</sup>, respectively.

### Conclusion

Our results showed that the amount of litter fall and soil C increased as the agroforestry stages progressed, indicating a higher internal nutrient cycling according to the Agroforestry age. All agroforestry stages in this study were found to be a C sink. The applied fertilizer at the younger age will be stored in the aboveground plant parts, as well as in the soil.

Keywords: Tropical soil, Soil respiration, Carbon cycle, Agroforestry

## Effects of microorganisms and minerals on size distribution of organic matter adsorbed to soil

HOBARA, Satoru<sup>1\*</sup> ; HASEGAWA, Yuki<sup>2</sup> ; FUJITA, Kyouhei<sup>1</sup> ; SATOU, Takayuki<sup>3</sup> ; IMAI, Akio<sup>3</sup> ;  
OGAWA, Hiroshi<sup>4</sup> ; ITO, Akihide<sup>5</sup> ; AE, Noriharu<sup>1</sup>

<sup>1</sup>Rakuno Gakuen University, <sup>2</sup>Hokkaido University, <sup>3</sup>National Institute for Environmental Sciences, <sup>4</sup>University of Tokyo,  
<sup>5</sup>Azabu University

Sorption of organic matter to soil is a key mechanism for carbon sequestration in terrestrial ecosystems. However, little is understood on how adsorptive organic matter is formed during decomposition process. In this presentation, we show changes in size distribution of adsorptive organic matter in soil during decomposition, and influences of microorganisms and minerals on the changes. We conducted an incubation experiment, in which leaves of three species were separately incubated in low-carbon-content soil, and size distribution of extractable organic matter from the soil was analyzed using HPLC attaching a size-exclusion column (HPSEC). Molecular weight of extractable organic matter distributed mainly in two ranges, about 100kDa-200kDa and about 0.5kDa-20kDa (based on proteins molecular weight) for all plant species. The width of these ranges changed to narrower with decomposition process. This change in molecular weight distribution (MWD) was observed when cycloheximide was added to soil, but not observed when chloramphenicol was added, suggesting bacterial influence on the MWD change during decomposition. In order to clarify influence of minerals on the typical two ranges, metal concentration was measured using ICP/MS for fractionated, eluted solution from HPSEC. The MWD of iron was closely related to that of organic carbon. These results suggest an importance of bacteria and iron for size distribution of adsorptive organic matter in decaying litter.

Keywords: Soil microorganisms, Soil minerals, Soil carbon, Organic matter adsorption, Molecular weight distribution

## Accumulation of sulfur compounds in Japanese volcanic soils

TANIKAWA, Toko<sup>1\*</sup>; HASHIMOTO, Yohey<sup>2</sup>; YAMASHITA, Naoyuki<sup>3</sup>; NOGUCHI, Kyotaro<sup>4</sup>; YOSHINAGA, Shuichiro<sup>1</sup>; TAKAHASHI, Masamichi<sup>4</sup>

<sup>1</sup>Kansai Research Center, Forestry and Forest Products Research Institute, <sup>2</sup>Tokyo University of Agriculture and Technology, <sup>3</sup>Asia Center for Air Pollution Research, <sup>4</sup>Forestry and Forest Products Research Institute

Volcanic soils are found in Asian countries, particularly within the circum-Pacific volcanic belt. Their unique chemical properties are expected to contribute to sulfur (S) retention. Although S is an essential element for plants and microorganisms, there is little information about S dynamics in forest ecosystems of Asian countries. The aim of this study is to characterize S accumulating systems in Japanese volcanic soil from the following four aspects: i) S pool sizes; ii) spatial S distribution in a catchment; iii) organic S transformation processes; iv) upbuilding/topdown pedogenesis.

To achieve the purposes, we investigated i) S pools of forested volcanic soils from 0-2m depth in the Kanto district; ii) the spatial variations of S concentration in a small drainage basin covered by volcanic soil by a geostatistical analysis; iii) organic S transformation rates in incubation experiments; iv) the vertical distribution of S species using K-edge X-ray absorption near-edge structure (XANES) spectroscopy of S species in Melanudands developed above tephra Nt-S, dated at 14-15 cal. ka by upbuilding pedogenesis.

The above four studies showed unique S accumulating systems of volcanic soils in Japanese forests as follows: i) volcanic soils accumulate larger amounts of S than other soil orders previously studied in North America and Europe. Forty percent of organic S was composed of Al-associated forms. Significant correlation between S species and pedogenic minerals suggests that large amounts pedogenic minerals lead to the large S pools. ii) Geostatistical analysis revealed the strong similarity of the spatial patterns of total S concentration and those of the pedogenic minerals throughout the catchment area. More S accumulated in the residual soils (ash soils) on the upper slopes of the catchment than in the colluvial soils of the lower slopes. The most important factor that influenced the distribution of soil S in the catchment was the degree to which volcanic ash was retained in the soils, as reflected by the abundance of the pedogenic minerals derived from volcanic ash deposits. iii) During incubation, the concentration of ester sulfate-S increased in the soils with high concentrations of the pedogenic minerals, whereas the concentration of Carbon (C)-bonded S decreased in all soils. The decrease rate of ester sulfate-S concentrations was negatively correlated with the pedogenic mineral contents. Therefore, when C-bonded S was transformed into ester sulfate-S, complete mineralization to inorganic sulfate might be inhibited, because ester sulfate-S was probably stabilized due to organo-mineral association. iv) The striping distribution patterns of various S fractions were shown in the soil with upbuilding pedogenesis. The predominant S species was highly oxidized S in ester sulfates and inorganic sulfates (+6 oxidation state), nevertheless, proportions of S with reduced and intermediate oxidation states increased episodically. A close correlation was found between ester sulfate-S concentrations and Al-associated organic S concentrations; indeed, the concentrations themselves were roughly similar.

These results suggest that accumulation/transformation of S compounds in Japanese forests is controlled by the pedogenic minerals derived from volcanic ash. Episodic deposits of volcanic ash and eolian dust might trap detritus and humus beneath them during the buildup of the soil surface. The organic S compounds in this material might be decayed as topdown pedogenic processes in the soils. Therefore, the soils with upbuilding pedogenesis have high heterogeneity of S compounds. Volcanic soils might have high ability to retain ester sulfates as Al-associated organic S during transformation processes.

Keywords: Al and Fe hydrous oxides and short-range ordered minerals, forest soils, geostatistical analysis, K-edge X-ray absorption near-edge structure (XANES) spectros, pedogenic minerals, transformation of organic S



## Visualization of organic matter binding on poorly-crystalline mineral phases in soil sub-micron organo-mineral matrix

ASANO, Maki<sup>1\*</sup> ; WAGAI, Rota<sup>2</sup> ; TAKEICHI, Yasuo<sup>3</sup> ; MAEDA, Makoto<sup>4</sup> ; SUGA, Hiroki<sup>5</sup> ;  
YAMAGUCHI, Noriko<sup>2</sup> ; HIRADATE, Syuntaro<sup>2</sup> ; TAKAHASHI, Yoshio<sup>5</sup>

<sup>1</sup>University of Tsukuba, <sup>2</sup>National Institute for Agro-Environmental Sciences, <sup>3</sup>Institute of Materials Structure Science, High-Energy Accelerator Research Organization, <sup>4</sup>Hiroshima University, <sup>5</sup>The University of Tokyo

Soil organic matter (SOM) accounts for a major portion of terrestrial C and is considered to be stabilized against microbial degradation due partly to its interaction with soil minerals. Significant control of poorly-crystalline mineral phases on soil organic matter turnover as well as its storage has been shown based on field correlation studies and incubation experiments. Yet how organic compounds of microbial and plant origins interact with poorly-crystalline and other mineral phases within soil aggregate structure at the spatial scale relevant to microbial extracellular enzymes (tens of nanometers) remain unclear. Here we focused on the sonication-resistant particles that are enriched in organic matter (OM) from an Andisol, the soil order characterized by high contents of OM and poorly-crystalline minerals and high aggregate stability, and tested the hypothesis that submicron-scale OM distribution within a soil particle is controlled by poorly-crystalline minerals using scanning transmission X-ray microscopy (STXM) and near-edge X-ray absorption fine structure (NEXAFS) as well as scanning and transmission electron microscopy (SEM, TEM). We will demonstrate that the combination of synchrotron and other spectroscopic techniques used here is a powerful approach to directly examine soil organo-mineral associations occurring at submicron scale, which can contribute to enhance our mechanistic understanding of SOM stabilization processes.

Keywords: soil organic matter, aggregate, poorly-crystalline minerals, Andisols, scanning transmission X-ray microscopy

## Optimal thermolysis condition for soil C storage upon plant residue burning

KAJIURA, Masako<sup>1\*</sup> ; WAGAI, Rota<sup>1</sup> ; HAYASHI, Kentaro<sup>1</sup>

<sup>1</sup>National Institute for Agro-Environmental Sciences

Crop residues are often burned in the field and mixed into soil. Yet the impact of this agricultural practice on soil carbon (C) sequestration remains unclear due to the heterogeneity in burning condition and the difficulty in monitoring the biodegradation of burned residues that include char in fields. Thus, identifying the quantitative relationship among burning condition, residue chemistry, and biodegradability is a critical first step. The residue burnt at higher temperature reduces greater mass yet the remaining residue becomes more recalcitrant via carbonization. The residue burnt at low temperature, on the other hand, maintain greater mass which experiences faster biodegradation due to its lability. A corollary to the trade-off relationship is the presence of threshold temperature range above and below which the residue carbon remained after experiencing both thermal decomposition and subsequent biodegradation is strongly reduced. To test the idea, we thermolyzed residues (rice straw and husk) at different temperatures (200-600 °C) under two oxygen availability, and measured the changes in thermal C loss and the biodegradability of thermally-altered residues by laboratory aerobic incubation. The empirical model accounting for the both decomposition processes showed the emergence of threshold temperature range (330-400 °C at 10<sup>2</sup>-10<sup>3</sup> year time scale) due to the expected trade-off relationship. This temperature range corresponded to the major loss of O-alkyl-C (cellulose and hemicellulose) and increase in aromatic-C. These findings show that the thermally-altered residues formed by the threshold range contributes the most to the long-term soil C storage in fire-prone ecosystems and may help to develop C sequestration strategy which takes advantage of field biomass burning, a widespread land practice in many parts of the world.

Keywords: black carbon, 13C NMR spectroscopy, rice residues, oxygen concentration, pyrolysis

## Soil organic matter and aggregate hierarchy revisited: a case study using an Andisol

WAGAI, Rota<sup>1\*</sup>; ASANO, Maki<sup>2</sup>; KAJIURA, Masako<sup>1</sup>; INOUE, Yudzuru<sup>3</sup>; HAYAKAWA, Chie<sup>4</sup>; HIRADATE, Syuntaro<sup>1</sup>

<sup>1</sup>National Institute of Agro-Environmental Science, <sup>2</sup>University of Tsukuba, Graduate School of Life & Environmental Sciences, <sup>3</sup>Saitama University, Graduate School of Science and Engineering, <sup>4</sup>The University of Tokyo, Graduate School of Agricultural and Life Sciences

Soil aggregate structure is likely to exert fundamental control on soil biology (e.g., microbial activity and community composition) and soil biogeochemistry including the stabilization and mobilization of soil organic matter (SOM). Aggregate hierarchy concept (Tidall and Oades, 1982) has been verified for most temperate and tropical soil types except for Andisols developed from tephra and volcanic rocks are known for strong aggregate stability and high SOM storage capacity. We recently showed the first evidence of hierarchical structure in Andisol (Asano and Wagai, 2013). After maximum dispersion (achieved only after 5 kJ/mL sonication following sodium saturation), most of macro- and micro-aggregates were broken down to much smaller particles (<0.2 and 0.2-2  $\mu\text{m}$ ) that accounted for dominant proportion of total C, N, and extractable phases of Al, Si, and Fe. These <2 $\mu\text{m}$  particle-size fractions appeared to act as persistent binding agent, contributing to the high physical stability of the aggregates. When the same soil was fractionated by density, the aggregates resistant to mechanical shaking (i.e. higher level in hierarchical structure) showed progressive changes in chemistry along density gradient (Wagai et al. 2014). Among the intermediate density fractions (2.0-2.5 g/cc) where >80% of total C and N and the extractable metals are present, we found general decrease in C:N ratio and  $\delta^{14}\text{C}$  and concurrent enrichment of  $^{13}\text{C}$  and  $^{15}\text{N}$  in accord with previous reports.

The next key question is how the particle-size fractions isolatable after the maximum dispersion are assembled together to form the shaking-resistant aggregates of different density. We attempted to answer this question by examining the organic and inorganic chemistry (C, N,  $^{13}\text{C}$ ,  $^{15}\text{N}$ ,  $\delta^{14}\text{C}$ ,  $^{13}\text{C}$ -NMR, extractable metals) and the surface characteristics (SSA by  $\text{N}_2$ -BET and XPS) of the size and density fractions. We will also discuss the nature of organo-mineral interactions inferred from our results and compare it with the previously-suggested concepts such as aggregate hierarchy (Tidall and Oades, 1982), and "zonal layering" model (Kleber et al. 2007).

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Keywords: soil carbon, soil nitrogen, carbon sequestration, aggregation, organo-mineral interaction, isotope

## Soil moisture variations for landscape trees under humid tropical conditions

JIM, C.y.<sup>1\*</sup>

<sup>1</sup>Department of Geography, University of Hong Kong

Compact urban development in Hong Kong has left little plantable spaces for amenity vegetation. Containers could permit tree growth in cramped sites with masses of buried utilities. This study evaluated the moisture variations in landscape tree containers in urban Hong Kong. Stratified random sampling selected 12 containers of different geometry. Soil moisture was obtained by a Time Domain Reflectometry (TDR) probe. Volumetric soil moisture content was measured at 5 cm depth intervals every two weeks for one year. Topsoil and subsoil samples were taken to analyze key physical and chemical properties. The soil mix, derived from decomposed granite amended with organic material, were highly stony with high sand content and loamy sand texture. The mean bulk density of 1.72 Mg/m<sup>3</sup> suggested degraded structure and compaction to about 33 per cent total porosity. Despite having more organic matter, the topsoil was more compacted and less porous than subsoil mainly due to prolonged rainsplash impact. Inadequate organic matter and excessive sand are unfavorable to soil structure formation and maintenance. The dominant coarse sand could support more air capacity pores to promote infiltration, drainage and aeration, with limited availability of medium pores for storage of plant-available water. Moisture content closely followed the rainfall regime with pronounced variations between dry and wet seasons and episodes. Moisture increased notably with depth in most containers due to less compacted subsoil. Occurrence of lithologic discontinuity in some sites retarded downward water movement and created perched water table. Waterlogging occurred periodically in subsoil due to heavy and prolonged rainfall in the wet season, compounded by blocked drain holes with impeded drainage. Sites shaded from direct sunshine had more water and less temporal fluctuations, indicating the influence of elevated temperature on evapotranspiration. The applications of the findings to container design, soil management for urban trees were explored.