The appearance of soil organic carbon compositions for short-term applications of biochemical contrasting organic residues using synchrotron-based fourier transform infrared (FTIR) microspectroscopy

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Soil organic carbon (SOC) chemical composition affects SOC stabilization that is one of the key mechanisms of carbon sequestration. The objective of this study was to assess SOC compositions in soils treated with organic residues with difference in biochemical compositions for short-term (0 and 28 days) using synchrotron-based fourier transform infrared (FTIR) microspectroscopy. Four treatments consisting of untreated soil (control), groundnut stover (high nitrogen (N) but low lignin (L) and polyphenols (PP)), tamarind litter (medium N, L and PP) and rice straw (low N, L and PP but high cellulose) were compared. The FTIR spectra of soil treated with organic residues showed similar functional groups of SOC compositions at 0 and 28 days including aliphatic amine C-N stretching and aromatic C-H out of plane bending. Organic compositions of the soils treated for short-term were associated with cellulose content in organic residues. The results indicated that short-term application of biochemical contrasting organic residues unaltered SOC chemical composition. The timing of organic residues application is important for carbon sequestration and change in SOC chemical compositions.

Keywords: groundnut stover, organic residue quality, rice straw, soil organic carbon functional groups, tamarind litter

INTRODUCTION

Soil organic carbon (SOC) is one part in the much larger global carbon cycle that involves the cycling of carbon through the soil, vegetation, ocean and the atmosphere (Lefèvre et al., 2017). The SOC pool stores an estimated 1,500 PgC in the first meter of soil, which is more carbon than is contained in the atmosphere (roughly 800 PgC)

and terrestrial vegetation (500 PgC) combined (FAO and ITPS, 2015). Furthermore, SOC plays an important role in ensuring food security (Lefèvre et al., 2017). This is achieved by enhancing soil productivity and maintaining consistently high yields, particularly by increasing water and nutrient holding capacity and improving soil structure that are suitable for plant growth (Zdruli et al., 2017).

SOC stock is important for not only sustaining yield of agricultural crops in high-input commercial agriculture but also increasing yield for the crops grown in low-input degraded land (Lefèvre et al., 2017). SOC is chosen as the most important indicator of soil quality and agricultural sustainability (Liu et al., 2006). Soil organic matter (SOM) stock is more important in tropical sandy soils, which are commonly highly weathered, low in SOC reserves and characterized by rapid microbial decomposition of added organic residues (Rasche and Cadisch, 2013). The most rapid loss of the SOC pool occurs in the first 5-10 years in the tropics (Lal, 2001). Hence, the manipulation of litter quality has been advocated as a management practice to improve SOC stabilization (Drinkwater et al., 1998; Cadisch and Giller, 2001; Palm et al., 2001; Whitbread et al., 2003).

The importance of applying crop residues as an amendment to enhance the SOC pool has long been recognized (Melsted, 1954; Tisdale and Nelson, 1966). The levels of SOC storage were controlled by the amount and the type of organic residues applied to the soil (FAO and ITPS, 2015), and the quality of residues regulated the accumulation potential of carbon (C) in the SOC pool (Traversa et al., 2008; Clemente et al., 2013). Litter quality including chemical compositions and nutrient quantity strongly controlled the rate of litter decomposition and nutrient release (Heal, 1997). The most important parameters determining litter quality are nitrogen, lignin, and soluble polyphenol contents (Palm and Rowland, 1997; Parton et al., 2007). The organic residues with high nitrogen, low lignin and low phenolic compounds increased soil microbial decompositions better than the organic residues with low nitrogen, high lignin and high phenolic compounds (Hadas et al., 2004; Puttaso et al., 2011).

Biochemical compositions of organic residues were closely related to SOC chemical compositions in sandy soil (Kunlanit et al., 2014). For example, polysaccharides were affected first by decomposition, leading to a decrease in O-alkyl-C (Helfrich et al., 2006), while lignin was resistant to decomposition due to its aromatic rings (Derenne and Large, 2001). Moreover, the duration of application time is an important factor affecting the chemical compositions of SOC. Change in chemical compositions of SOC was firstly affected by fertilization strategies, and further change was influenced by the duration of application time (0, 10 and 20 years), whereas the chemical compositions of SOC were rather similar at long-term application (Li et al., 2015). Regular application of organic

manure for 4 or 21 years decreased aromatic C abundance and alkyl C/O -alkyl C ratio of SOC compared to application of chemical fertilizers (Wang et al., 2012; Zhang et al., 2013). According to Li et al. (2013), long-term intensive forest management practices (0, 5, 10, 20 and 30 years) including annual inorganic fertilizer application, tillage, and removal of understory vegetation significantly changed the organic C chemical compositions and increased alkyl C and carbonyl C contents and the A/O-A ratio in the soil of Moso bamboo forests, whereas O-alkyl C and aromatic C contents and aromaticity were reduced. Applications of balanced and unbalanced chemical fertilizers in paddy soil or upland soil for 24 or 29 years were not significantly different for SOC chemical compositions (Yan et al., 2013). Although bulk information on the effects of long-term application of organic/inorganic amendments on SOC chemical compositions is available in the literature, the information on the effects the application of organic residues with different compositions and duration of application time on SOC chemical compositions is scarce.

Application of biochemical contrasting organic residues for 13 to 22 years increased the number of additional SOC functional groups including labile organic compounds and recalcitrant organic compounds (Somboon et al., 2018). The authors observed that the number of labile organic compounds in the soil treated with organic residues for 22 years was larger than that of recalcitrant organic compounds and the increase in the number of labile organic compounds may be due to decomposition or transformation of recalcitrant organic compounds. Under short-term incubation condition of tropical soil, the decomposition of groundnut stover, tamarind, rice straw and dipterocarp leaf litter was very rapid at four weeks after incubation together with high initial C loss through microbial respiration, reflecting high amounts of available carbon that were readily utilized by decomposers (Puttaso et al., 2011). Moreover, enzyme activities related to decomposition of organic C compounds such as invertase, B-glucosidase, phenoloxidase and peroxidase were highest at the first 4 weeks of decomposition stages (Kamolmanit, 2014). Consequently, the use of different organic residues at this period (4 weeks) may be related to the chemical compositions of SOC.

Currently, farmers are interested in organic farming by application of organic residues to improve soil fertility, and the organic residues are usually applied at one month before planting. The

assumption underlying the research project is that the SOC functional groups may be different if the organic residues with difference in biochemical compositions are applied in short-term. The characterization of SOC chemical composition remains challenging as the information gained through currently available techniques, including chemical and physical fractionation, is still limited relative to the complexity of SOC biochemistry (von Lützow et al., 2007). However, synchrotron radiation has opened new opportunities for the study of C in soils because the high energy provides significant improvement of the spectra as well as the spatial resolution (Lehmann and Solomon, 2008). Recent investigations using synchrotron-based fourier transform infrared (FTIR) microspectroscopy have indicated that these techniques are powerful and noninvasive, and can be used to identify and fingerprint the complex structural characteristics of SOC and dissolved organic C (DOC) as well as to investigate the impact of management on the composition and biogeochemical cycling of organic C at the molecular level in terrestrial ecosystems (Jokic et al., 2003; Kinyangi et al., 2006; Lehmann et al., 2005, 2007; Myneni et al., 1999; Schäfer et al., 2003; Scheinost et al., 2001; Solomon et al., 2005, 2007a,b; Schumacher et al., 2005, 2006).

Synchrotron infrared radiation is more advantageous than conventional method in that it has brightness of 100-1000 times greater than a conventional source (Carr et al. 1995; Miller and Dumas, 2006), resulting in better spectral properties and better signal-to-noise ratios (Lehmann and Solomon, 2008). The information on SOC chemical compositions as the results from the application of biochemical contrasting organic residues is scarce in the literature especially for the comparison of short-term and long-term applications. Therefore, the objective of this study is to assess SOC chemical compositions in soil treated with biochemical contrasting organic residues for short-term (0 and 28 days) using synchrotron-based FTIR microspectroscopy.

MATERIALS AND METHODS

Soil sampling

This study was done in short-term pot incubation experiment (28 days). Four treatments in this study consisting of untreated soil (control; CT), groundnut (*Arachis hypogaea*) stover (GN) with high nitrogen (N) but low lignin (L) and polyphenols (PP) (N 22.8, L 67.6, PP 12.9 and cellulose (CL) 178.0 g kg^{-1}), tamarind (*Tamarindus*

indica) leaf and petiole litter (leaf:petiole ratio = 7:1 on dry weight basis) (TM) with medium N, L and PP (N 13.6, L 87.7, PP 31.5 and CL 143.0 g kg^{-1}) and rice (*Oryza sativa*) straw (RS) with low N, L and PP but high CL (N 4.7, L 28.7, PP 6.5 and CL 507.0 g kg^{-1}) (Puttaso et al., 2011).

The soil used in the experiment is Khorat soil series with sandy loam texture (Oxic Paleustults). The soil was air-dried (under shade) and sieved to pass through a 2 mm screen. The sieved soil of 6 kg was then loaded into each pot. The organic residues at the rate of 10 mg ha^{-1} dry weight were incorporated into the soil samples according to the treatments and soil moisture content in each pot was maintained daily at 70% of water holding capacity. Soil samples were collected for SOC analysis at 0 (before incubation) and 28 days after incubation.

Synchrotron-based FTIR microspectroscopy

Soil samples were air-dried, passed through 2 mm sieve, ball milled into fine powder and dried overnight at 32 °C. The samples characterization were done by synchrotron-based FTIR microspectroscopy. Each soil sample was prepared using a diamond anvil cell which is a high pressure cell permitting to operate and adjust under microscope for transmission studies.

Spectral data were collected at an infrared microspectroscopy beamline (BL4.1 Infrared Spectroscopy and Imaging) at the Synchrotron Light Research Institute (SLRI). Spectra were acquired with a SR-FTIR (Hyperion 2000, Bruker Optics, Ettlingen, Germany) coupled with an Infrared microscope (Hyperion 2000, Bruker) using the 36x objective. The detector of the infrared microscope was a liquid nitrogen cooled mercury cadmium telluride (MCT-A) detector (of 100 μm in size).

The measurements were performed in transmission mode, using an aperture size of 20×20 μm^2 with a spectral resolution of 4 cm^{-1}, with 64 scans co-added. Spectral acquisition and instrument control were performed using OPUS 7.2 software (Bruker Optics Ltd, Ettlingen, Germany). The spectral changes of the functional groups were performed at the integral area of each peak.

Data analysis

Data were analyzed statistically by two-way analysis of variance (ANOVA) using SPSS software program. Treatment means were separated by least significant difference (LSD) at 0.05 probability level. Pearson correlation was used to determine the relationships between FTIR

peak areas and biochemical compositions of organic residues.

RESULTS

The SOC chemical composition of short-term application of biochemically contrasting organic residues

The dominant functional groups in the FTIR spectra of the soils treated with organic residues for 0 and 28 days are displayed in Figure 1a and 1b, respectively, and their band assignments are described in Table 1. The soils treated with different organic residues had similar organic functional groups evaluated at 0 and 28 days after incubations. These groups consisting aliphatic amine C-N stretching (1250-1091 cm^{-1}) and aromatic C-H out of plane bending (860-796 cm^{-1}), which were characteristic of protein and lignin, respectively.

Moreover, the integral area of average spectra for each functional group is described in Table 2. The integration value of aliphatic amine C-N stretching of the soil incubated for 0 day was 5.03, and the values for all treatments were increased after 28 days of incubation. The integration values of aliphatic amine C-N stretching ranged between 6.65 for groundnut to 11.53 for control. The control treatment was significantly higher than groundnut, tamarind and rice straw, whereas groundnut (6.65) tamarind (6.68) and rice straw (7.46) were similar. The integral values of aromatic C-H out of plane bending ranging from 1.67 to 1.89 were not significantly different among treatments.
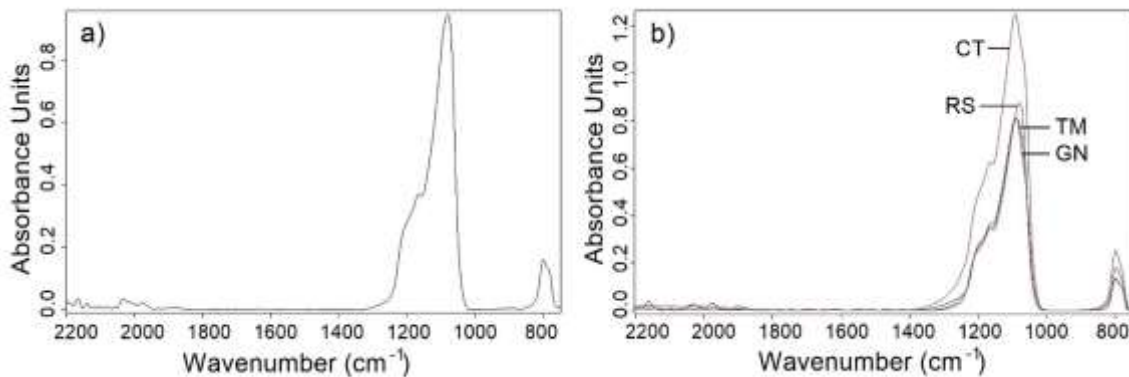

Figure: 1. Average FTIR spectra of soil treated with biochemically contrasting organic residues after 0 day (a) and 28 days (b) (CT: control, GN: groundnut stover, TM: tamarind and RS: rice straw)

Table: 1. SOC structure assignment of short-term and long-term applications of biochemically contrasting organic residues of the soils treated with organic residues for 0 and 28 days

Treatment	Wavenumber (cm^{-1})	Structural assignment	Chemical group	Reference
0 day				
CT, GN, TM, RS	1250-1091	Aliphatic amine C-N stretching[a]	Protein	Yang and Yen (2002)
	860-796	Aromatic C-H out of plane bending[b,c]	Lignin	Popescu et al. (2006)
28 days				
CT, GN, TM, RS	1250-1091	Aliphatic amine C-N stretching[a]	Protein	Yang and Yen (2002)
	860-796	Aromatic C-H out of plane bending[b,c]	Lignin	Popescu et al. (2006)

[a] Aljamali (2015), [b] Baes and Bloom (1989), [c] Senesi et al. (2003), CT: control, GN: groundnut stover, TM: tamarind and RS: rice straw

Table: 2. The integral area of average spectra from the soils treated with biochemically contrasting organic residues for 0 and 28 days

Treatment	$1250\text{-}1091\ cm^{-1}$	$860\text{-}796\ cm^{-1}$
0 day		
Soil prior to incubation	5.03	1.67
28 days		
Control	11.53a	1.89
Groundnut	6.65b	1.72
Tamarind	6.68b	1.81
Rice straw	7.46b	1.69
F-test	*	ns

ns=non-significant, *=significant at $P<0.05$

Table: 3. Correlation (Pearson's correlation coefficient (r)) between FTIR peak areas and organic residue quality of the soils treated with organic residues for 28 days

Structural assignment	Initial biochemical composition of organic residues			
	Nitrogen	Lignin	Polyphenols	Cellulose
28 days				
Aliphatic amine C-N stretching	0.004	-0.174	-0.184	-0.629**
Aromatic C-H out of plane bending	0.021	-0.071	-0.096	-0.099

Significance levels: ** ($P<0.01$), ns ($P>0.05$)

Relationships of FTIR peak areas with the biochemical composition of organic residues

Correlation coefficients between FTIR peak areas of the soils treated for 0 and 28 days and the biochemical compositions of organic residues (i.e. N, L, PP and CL) are presented in Table 3. Negative and significant correlation ($r=-0.629$; $P\leq0.01$) was found between CL and aliphatic amine C-N stretching in the soil treated 28 days, whereas the correlations of aromatic C-H out of plane bending with the biochemical compositions of organic residues were not significant.

DISCUSSION

In this study, the soils treated with organic residues that had different quality for short-term (0-28 days) were evaluated for SOC chemical compositions. Quality of organic residues did not significantly affect the number of SOC functional groups after the treatment of organic residues for short-term. The previous study showed the change in the number of SOC functional groups was affected by the application time as indicated by the present of new functional groups in the soils treated for 13 and 22 years (Somboon et al., 2018). The additional bands representing organic functional groups including carbohydrate overtones of C-OH stretching were observed in the soils treated for 13 years, and they were classified into carbohydrate, aromatic C-H stretching, aromatic COO-stretching and aromatic amine C-N stretching, which were characteristic of lignin. Most organic functional groups found in the soil treated for 13 years were also found in the soil treated for 22 years except for aromatic COO-stretching. The new bands representing organic functional groups including -NH stretching of secondary amine, carboxylic acid O-H stretching and aliphatic C-H stretching were found to be unique to the soil treated for 22 years. -NH stretching of secondary amine was assigned to carbohydrate, carboxylic acid O-H stretching was assigned to cellulose and aliphatic C-H stretching was assigned to protein.

In this study, quality of organic residues did not significantly affect the number of SOC functional groups but it did affect the integral area. The report of Qiao et al. (2018) also supported that long-term monoculture may affect the chemical structures of SOC but the chemical structures did not vary within crop species. Whereas, organic C input from leaf litter materials did not directly contribute to the formation of SOC chemical fractions, whereas soil microbial community could be a main factor influencing the chemical composition of SOC (Wang et al., 2016).

The change in the number of SOC functional groups may be associated with SOC decomposition rates (Guo et al., 2016). The decomposition rates of SOC varied considerably as a function of environmental conditions (Ise and Moorcroft, 2006). Temperature is the most important factor directly affecting the decomposition rates of SOC. The soil organic residues decompose more rapidly in tropical regions with higher temperature than in temperate regions with lower temperature (Shelake et al., 2019) and the soils in the tropical regions have

lower SOC than the soils in temperate regions (Canadell et al., 2007). Additionally, Tian et al. (2016a) reported that the labile carbon pool size was affected by the mean annual temperature. Soil moisture is also an important factor affecting the degradation of soil organic residues. Change in mean annual precipitation altered the carbohydrate degradation potential of a bacterial community that directly affects the SOC sequestration (Martiny et al., 2017). Besides, change in SOC chemical compositions was also influenced by microbial communities in soil (Kallenbach et al., 2016). Similarly, the variations in SOC chemical functional groups were strongly associated with change of soil microbial community taxa and functional genes, which might be affected by the changes of soil characteristics (Deng et al., 2019).

In this study, the new SOC functional groups that were not found in the soil treated for 28 days were found in the soils treated for 13 and 22 years of Somboon and team's report. These functional groups were classified into both labile (carbohydrate, protein, cellulose) and recalcitrant (lignin) organic functional groups, and the soil treated for 22 years had higher number of labile organic compounds than the soil treated for 13 years.

In previous study, change in C fractionation into labile and recalcitrant pools was based on microbial degradability of the SOM (Ahmed, 2018). These two broad pools are further classified into different organic compounds, and many microorganisms are involved in the degradation of these compounds. Moreover, fresh litter and root may also stimulate the microbial activities and lead to rapid decomposition of old C in soil, creating an antagonistic effect on the storage of soil organic C. Similarly, Dou et al. (2016) found that application of long-term fertilization stimulated both new C input and the decay rate of the old C. Furthermore, some compositions of labile C cannot be subjected to microbial attack as they were due protected by clay particle or coated by recalcitrant materials, and, therefore, these compositions of labile C must be chemically degradable and physically accessible to microbes (Baldock and Skjemstad, 2000).

In this study, negative and significant correlation was showed between CL and aliphatic amine C-N stretching (protein) in the soil treated for 28 days. It was possible that the reduction of CL content in soil (which was consequence from CL decomposition) can stimulate the products of CL decomposition (e.g. dissolved organic C and microbial biomass C (Chen et al., 2018)) accordingly increase in the composition of aliphatic amine C-N stretching (protein) in soil. Moreover, the quality of organic residues i.e. N, L, PP were not correlated with the SOC functional groups in the soil treated for 28 days. However, L and PP significantly affected the change in the new SOC functional groups in the soils treated for 13 and 22 years as indicated by the positive and negative correlations of L and PP with the additional SOC functional groups (Somboon et al., 2018).

According to Kunlanit et al. (2014), L and PP components of the organic residues were both positively and negatively correlated with labile organic functional groups of SOC such as carbohydrate overtones of C$-$OH stretching, aliphatic C$-$H stretching and C$-$OH of aliphatic OH, and were positively correlated with stable organic functional groups of SOC such as C$-$O stretching of COOH and ketones, C$=$C of aromatic groups, C$-$H and N$-$H amide II, aliphatic C$-$H deformation, benzoic acids, C$-$O of aryl ethers and C$-$O of phenolic groups. Furthermore, our study showed that SOC functional groups in the soil treated for 13 years were found to be aromatic COO$-$stretching, which was positively correlated with L and PP, but these groups were missing in the soil treated for 22 years. According to Todorciuc et al. (2009), the reactivity of lignin depends on its structure and origin, while the characteristics of the synthesized products result from the modifications induced by the new functional groups. Moreover, Li et al. (2015) revealed reducing production of lignin degrading enzymes by white-rot fungi due to increasing available N levels, which may result in relative accumulation of aromatic C derived from the lignin, and biochemical protection by binding N to the aromatic C rings, leading to the formation of more recalcitrant composites. The white rot fungi generated a great amount of conjugated and unconjugated C=O, which may be due to oxidative mechanism involved in lignin biodegradation (Yang et al., 2010; Sahadevan et al., 2016).

Our results indicated that short-term application of biochemical contrasting organic residues did not change SOC chemical compositions, whereas long-term application did change SOC chemical compositions by increasing both labile and recalcitrant organic functional groups. Moreover, a change in SOC chemical compositions was correlated with the biochemical compositions of organic residues especially for L, PP and CL. Therefore, improvement of C

sequestration requires long-term application of organic residues to release the functional groups from these organic residues to the soil.

CONCLUSION

The short-term application of biochemical contrasting organic residues unaltered SOC chemical composition. Organic compositions of the soils treated for short-term were associated with cellulose content in organic residues.

CONFLICT OF INTEREST

The authors declared that present study was performed in absence of any conflict of interest.

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AUTHOR CONTRIBUTIONS

PL was principal researcher. PL designed and performed the experiments. SS, BK, WN and KT carried out the experiments, PL and SS wrote and reviewed the manuscript. All authors read and approved the final version.

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