



# Assessment of soil aggregation properties after conversion from rice to greenhouse organic cultivation on SOC controlling mechanism

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## Abstract

**Purpose** Organic manure is beneficial for macro-aggregate formation and soil organic carbon (SOC), but how SOC change in aggregate fractions in time-series is still uncertain. Moreover, greenhouse systems converted from cereal fields quickly faced soil degradation. Thus, the role of organic manure here should be discussed. The main objectives of this study were to determine the change of SOC fractions in bulk soil and aggregation level affected by long-term organic manure application.

**Materials and methods** Using <sup>13</sup>C solid-state nuclear magnetic resonance (NMR) spectroscopy, we investigated the SOC and its fraction changes within bulk soil and aggregate fractions under 1-year, 9-year, and 14-year organic greenhouse vegetable cultivation, and we also analysed the soil properties of rice-wheat rotation (RWR) fields as the control. Soil aggregate samples were wet sieved into large macro-aggregates (> 2 mm), small macro-aggregates (2–0.25 mm), micro-aggregates (0.053–0.25 mm), and silt and clay particles (<0.053 mm).

**Results and discussion** The proportion of large macro-aggregates increased significantly ( $P \leq 0.05$ ) from 1.68 to 7.76% during the 14-year organic cultivation period. Similar trends could also be found in SOC and its fractions. Specifically, the O-alkyl C increased fastest by 14.5 g kg<sup>-1</sup> over these years, but its proportion decreased. The change in aggregate associated C was concentrated on large macro-aggregates and micro-aggregates. Pearson's correlation suggested that there was a non-significant relation between SOC and soil aggregation, while the soil aggregate associated C had a significantly positive relationship with SOC and its fraction amounts. However, the proportion of O-alkyl C was less; the aggregation associated C was higher.

**Conclusions** This study has shown the different effects of SOC fractions on soil aggregation and aggregate associated C in organic greenhouse vegetable fields. The stable C fractions might show contribution in soil aggregate associated C than active C fractions. It might improve our knowledge about how organic manure may influence soil aggregation by increasing SOC after a long-term greenhouse vegetable plantation.

**Keywords** Land-use conversion · Organic manure · Soil aggregation · Soil organic carbon · Aggregate associated carbon · <sup>13</sup>C solid-state NMR spectroscopy

## Abbreviations

BD bulk density  
AN available nitrogen

AP available phosphorus  
AK available potassium  
TN total nitrogen

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TP	total phosphorus
TK	total potassium.
RWR	rice-wheat rotation plots
OGV1	one-year-old organic greenhouse vegetable plots
OGV9	nine-year-old organic greenhouse vegetable plots
OGV14	fourteen-year-old organic greenhouse vegetable plots

## 1 Introduction

Soil aggregates, which affects crop yield and quality, are considered as essential bases and units of soil structure (Bashir et al. 2016), and their distribution and stability are closely associated with other soil physical-chemical properties (Dorji et al. 2019). Generally, soil organic carbon (SOC) is one of the major factors involved in the formation of soil aggregates (Wang et al. 2018). The application of organic manure constitutes a typically sustainable agricultural practice to increase SOC contents (Naveed et al. 2013) and improve soil aeration (Wolf & Snyder 2003) in the short term period. However, the effects of organic manure on soil physical properties vary, although total SOC increase (Xu et al. 2018).

Organic manure is always recognized as sustainable agricultural practice and plays a vital role in the decrease of bulk density (BD) (Padbhushan et al. 2016) and macro-aggregate formation (Brar et al. 2013). A 9-year Indian field experiment suggested that the aggregate size distribution (> 0.25 mm) in farmyard manure was higher than the 100% NPK treatment (Padbhushan et al. 2016). Similar results were obtained by Mangalassery et al. (2019), they found that the proportion of macro-aggregates (> 0.25 mm) and mean weight diameter (MWD) increased under after 5-year organic manure application. Moreover, Ghosh et al. (2019b) proved that organic manure in intensive agricultural systems could protect soil from physical degradation after an 11-year study in the European Union. Plaza-Bonilla et al. (2013) reported that organic manure slightly increased water-stable macro-aggregates compared with chemical fertilization but caused no differences in SOC or macro-aggregate C concentrations. However, most of the research focused on the comparison among different fertilizers (Ghosh et al. 2016, Guo et al. 2018, Hernandez et al. 2017, Wang et al. 2017), the effects in different time series were always ignored. Besides, Xu et al. (2018) and Papadopoulos et al. (2009) confirmed that organic manure played a stronger positive effect on soil structure improvement in the earlier period, and the effects might lessen with time. Overall, organic manure could enhance soil physical quality (Mikha et al. 2015, Williams et al. 2017), but the role of organic manure in time scale has still not well realized.

It is essential to analyse the change of SOC to evaluate the effects of organic manure on soil aggregation characteristics (Mi et al. 2018). SOC is the primary source of crop nutrients

and could improve the water holding capacity and aeration of soil (Wolf & Snyder 2003). Organic manure is beneficial to SOC sequestration (Hu et al. 2018, Huang et al. 2010). For example, Cai et al. (2016) indicated that long-term organic manure applications significantly increased the SOC within both bulk soil and particle size fractions. The research of Weyers et al. (2018) showed that organic manure had a positive impact on maintaining SOC over time compared with the chemical fertilization. The consistent conclusion has been obtained by Jiang et al. (2018). Besides, SOC fractions such as labile carbon, hot water-soluble carbon, particulate organic C (Chaudhary et al. 2017, Pritchett et al. 2011), are more sensitive to reflect the soil quality than total SOC, and obtained attention widely by researchers (Plaza-Bonilla et al. 2013). Li et al. (2018b) found that a 26-year organic manure application could significantly increase total SOC amount and labile organic C fractions compared with chemical fertilization. In addition, a 15-year experiment suggested that labile fractions of SOC were early indexes of SOC on a short-term basis and that different organic manures played different roles in long-term C sequestration (Chaudhary et al. 2017).

In recent years, a  $^{13}\text{C}$  solid-state nuclear magnetic resonance (NMR) spectroscopy has widely applied in the soil science research as a non-destructive technique (Ghosh et al. 2019a). Shrestha et al. (2015) found that farmyard manure may contribute to fertility loss by evaluating SOC quantity and quality with the  $^{13}\text{C}$  solid-state NMR spectroscopy under long-term crop and management practices in Norway. A 34-year fertilization experiment suggested organic manure increased the levels of alkyl C and aromatic C but decreased O-alkyl C in Vertisols. However, Li et al. (2018a) believed that the organic manure treatment had a higher aromatic C–O and  $\text{OCH}_3$  and lower alkyl C and OCH abundance in a Calcaric Fluvisol. The different soil types might cause the difference of SOC fractions. Thus, more evidence is required to discuss the change of SOC fractions with organic manure.

Besides, the mechanism between soil structure and SOC has always been referred in organic manure experiments (Abid & Lal 2008, Jensen et al. 2019, Naveed et al. 2014). Generally, SOC could contribute to the stabilization of macro-aggregates (Jiang et al. 2018, Somasundaram et al. 2017, Zheng et al. 2016). However, as a group of complexes, the SOC fractions affecting the soil aggregation should be considered. With the development of technology, the research on SOC fractions combined with soil aggregation through some non-destructive methods has obtained attention widely. Sarker et al. (2018) suggested that the high proportion of aromatic carbons hampered soil aggregation. Besides, Mizuta et al. (2015) found that soil aggregate formation and stability were induced by polysaccharides. Although there has existed an extensive knowledge about the mechanisms of soil aggregation controlled by SOC (Regelink et al. 2015), the relation between soil

structure and SOC dynamics has still not well understood (Blanco-Canqui & Lal 2004).

Therefore, the objectives of this study were: (1) to evaluate the change of soil aggregation distribution; (2) to analyse the SOC and its fractions in both bulk soil and aggregate level; and (3) to preliminarily establish the relation between soil aggregation properties and SOC characteristics. To solve it, we chose the organic greenhouse vegetable fields converted from rice-wheat rotation (RWR) plots for different durations (one year, nine years, and fourteen years). Greenhouse converted from RWR easily faced soil structure degradation (Rodrigo-Comino et al. 2018), so the impacts of organic manure should be distinguished (Xu et al. 2019, Zikeli et al. 2017). It might further improve our knowledge of how SOC fractions affect soil aggregation in response to applications of long-term organic manure, which has a theoretical significance for sustainable agriculture.

## 2 Methods and materials

### 2.1 Site description

The research site is located in the urban area of Nanjing city (latitude 31°52' N, longitude 118°42' E), Jiangsu Province, Southeast China. The annual mean temperature and precipitation are 15.7 °C and 1072.9 mm, respectively. The parent material of the agricultural soil is Quaternary loess, and the main soil type is Anthrosol (Inceptisol) (IUSS-WRB 2014). The soil is a silty loam, and the main crop system before our research was a RWR system. All of these vegetable plots are managed by a local company. The main crops include leaf vegetable species such as cabbage (*Brassica oleracea* var. *capitata*) and spinach (*Spinacia oleracea*) (Chen et al. 2014). The amount of organic manure added to the vegetable fields was approximately 14,000 kg ha<sup>-1</sup> during a long growth period (one or two crops per year) and approximately 7500 kg ha<sup>-1</sup> during a short growth period (more than two crops per year) before vegetable plantations. The total nutrients (N + P<sub>2</sub>O<sub>5</sub> + K<sub>2</sub>O) was up to 10%, and the organic matter was more than 45% in this manure.

There were four treatments in our research, which were one-year-old organic greenhouse vegetable plots (OGV1), nine-year-old organic greenhouse vegetable plots (OGV9), and fourteen-year-old organic greenhouse vegetable plots (OGV14). Besides, we chose the RWR fields near the organic greenhouse fields as the control for this study. All organic greenhouse vegetable plots converted from RWR fields experienced approximately three-year-long fallow periods before the first time of vegetable cultivation.

### 2.2 Soil sampling and pre-processing

Soil samples were collected from the top layer (0–15 cm) in the summer of 2016 after the vegetable and cereal harvest. Three disturbed soil samples were randomly sampled in each treatment. For each treatment, three intact cores approximately 50 mm in diameter and 50 mm in length were extracted, and the cutting ring method was used to determine the BD in the laboratory (Githinji 2014). Each disturbed sample was gently broken apart along natural breakpoints and then passed through a 10 mm sieve. Stones and plant roots were removed, after which the remaining soil was allowed to dry naturally. Each sample was divided into two parts: one part was used for the determination of the aggregate size distribution, and the other part was used for the evaluation of bulk soil. Disturbed soil samples were also taken to measure soil basic properties (e.g., pH, SOC, soil total nutrients, and available nutrients) according to the methods of Lu (2000).

### 2.3 Aggregate separation

The soil aggregates were separated by wet sieving according to previously described methods (Kemper & Rosenau 1986, Plaza-Bonilla et al. 2013); sieving was performed with a series of three sieves that separated the samples into four different soil fractions: large macro-aggregates (> 2 mm), small macro-aggregates (0.25–2 mm), micro-aggregates (0.053–0.25 mm), and silt and clay particles (< 0.053 mm). Soil particles that passed through the sieves were dried (40 °C) and weighed. The MWD, geometric mean diameter (GMD), mean weight-specific surface area (MWSSA), and fractal dimension (D) were determined according to Cui et al. (2019) and Huang et al. (2017). It indicated the stability of soil aggregates. When the MWD and GWD are relatively high, the MWSSA and D are relatively low, and the aggregate structure is strong. Samples were collected to determine the SOC, while the remaining samples were mixed within their respective class and then passed through a 100 mesh sieve for SOC fraction analysis.

### 2.4 <sup>13</sup>C NMR spectroscopy

The soil samples were pre-processed for SOC fraction analysis according to the methods of Skjemstad et al. (1994). A five g soil sample was placed into a 100 ml centrifuge tube to which 50 ml of 2% (v/v) hydrofluoric acid (HF) was added and mixed. The mixture was shaken for 2 h and then centrifuged at 3600 rpm for 10 min, after which the supernatant was discarded. This process was repeated 5 times, and the shaking time was increased to 16 h in two additional replications. The residual soil sample was finally washed with distilled water three times, dried at 40 °C, and passed through a 100 mesh sieve for further analysis.

A  $^{13}\text{C}$  cross-polarization/total sideband suppression (CP/TOSS) and CP/TOSS with dipolar dephasing experiments were performed via a Bruker Ultra Shield Plus 400 MHz wide-bore spectrometer for  $^{13}\text{C}$  with 4 mm sample rotors. The experiments were conducted at a spinning speed of 10 kHz and with both a connection time of 2 s and a recycle delay of 2 s. Following the methods of previous studies (Mao et al. 2008, Mathers et al. 2007), the  $^{13}\text{C}$  NMR signals were categorized as follows: alkyl C ( $\delta = 0\sim 45$  ppm), O-alkyl C ( $\delta = 45\sim 110$  ppm), aromatic C ( $\delta = 110\sim 165$  ppm), and carbonyl C ( $\delta = 165\sim 220$  ppm). All of these proportions were obtained via Mest ReNova 9.0.1 (Mestre-lab Research, 2014). The SOC amount in each fraction was determined by multiplying the proportion of each SOC fraction by the total SOC (Zhang et al. 2017).

## 2.5 Statistical analysis

All statistical analyses were conducted using the Statistical Package for the Social Sciences (SPSS) version 19.0 (SPSS Inc., Chicago, IL) and Excel 2013. The mean values were tested via the least significant difference (LSD) (Duncan's LSD) method at the  $P \leq 0.05$  level of statistical significance. The interactions among soil properties were analysed by Pearson's correlation. The statistical significance of the Pearson's correlation coefficient was determined at the  $p = 0.05$  and  $p = 0.01$  levels.

## 3 Results

### 3.1 Variation of basic soil properties

The trends of basic soil properties differed significantly ( $P \leq 0.05$ ) over the 14-year organic greenhouse plantation converted from RWR fields except for the pH value (Table 1). For example, the BDs in four treatments were as follows: OGV1 ( $1.40 \text{ g cm}^{-3}$ ) > OGV9 ( $1.14 \text{ g cm}^{-3}$ ) > RWR ( $1.09 \text{ g cm}^{-3}$ ) > OGV14 ( $1.02 \text{ g cm}^{-3}$ ). In other words, the BD decreased with the long-term organic manure application. Besides, the BD of RWR furtherly confirmed that long-term organic manure application could restore the soil structure and return to the original background value compared with OGV14. Besides, compared with soil nutrients, there was no significant ( $P > 0.05$ ) difference in available nutrients between RWR and OGV1 plots. While the soil available nutrients increased obviously during 14-year organic greenhouse plantation. These results were similar to the figure for soil total nutrients. Thus, better soil structure and more soil nutrients accumulation were found in long-term organic greenhouse systems.

### 3.2 Aggregate distribution and stability

The distribution patterns in the proportions of soil aggregates were similar among four treatments (Fig. 1), and the percentage of micro-aggregates was the highest among these aggregations. However, there was an obvious change of these aggregates during the 14-year organic greenhouse cultivation. For example, the proportion of large macro-aggregates ( $> 2$  mm) accounted for 51.3%, 16.8%, 28.3%, and 37.9% of the aggregates in RWR, OGV1, OGV9, and OGV14 plots, respectively. A similar trend could be obtained in the small macro-aggregates (0.25–2 mm). However, the trend was different in the micro-aggregates (0.053–0.25 mm) and silt and clay ( $< 0.053$  mm). The data in Table 2 also indicated that the MWD and GWD significantly ( $P \leq 0.05$ ) increased but that the D and MWSSA decreased in response to long-term organic manure. Overall, the aggregation stability improved, and the proportion of macro-aggregation ( $> 0.25$  mm) increased after land-use conversion. It might be caused by the continuous organic manure application.

### 3.3 SOC in bulk soil and aggregates

In our study, the SOC in bulk soils differed significantly ( $P \leq 0.05$ ) during the 14-year organic greenhouse plantation (Fig. 2). For example, the OGV14 plots ( $41.5 \text{ g kg}^{-1}$ ) presented the highest SOC content, with five times greater than the SOC content in the OGV1 plots ( $6.7 \text{ g kg}^{-1}$ ). The difference of SOC in the RWR and OGV1 plots was because of the conversion period, during which the soil in the plough layer was brought to the surface. To sum up, organic manure could help the SOC stock after land-use conversion.

Besides, the SOC at different aggregate fractions showed various tendencies over the 14-year organic cultivation (Fig. 2). The SOC within large macro-aggregates ( $> 2$  mm) increased most rapidly (from  $7.06 \text{ g kg}^{-1}$  to  $43.69 \text{ g kg}^{-1}$ ), followed by that within the micro-aggregates (0.053–0.25 mm) and small macro-aggregates (0.25–2 mm). The SOC within the silt and clay particle ( $< 0.053$  mm) fractions remained stable. As expected, our results indicated a clear difference in SOC within aggregate size fractions in response to organic manure. In conclusion, organic manure played a positive role in SOC, but the effects in different aggregates were different.

### 3.4 SOC fractions in bulk soil and aggregates

The SOC fractions in bulk soil increased significantly ( $P \leq 0.05$ ) with continual organic manure input in the organic greenhouse vegetable plots converted from RWR plots (Fig. 3). For example, the amount of O-alkyl C increased as follows: OGV1 ( $3.5 \text{ g kg}^{-1}$ ) < OGV9 ( $14.6 \text{ g kg}^{-1}$ ) < OGV14 ( $17.9 \text{ g kg}^{-1}$ ); a similar trend was found for alkyl C, aromatic

**Table 1** Indices of soil basic properties in the long-term organic greenhouse fields converted from rice-wheat rotation ( $n = 3$ )

Treatments	RWR	OGV1	OGV9	OGV14
BD ( $\text{g cm}^{-3}$ )	1.09 ± 0.04 a	1.40 ± 0.02 b	1.14 ± 0.02 c	1.02 ± 0.03 d
pH	5.42 ± 0.32 a	6.01 ± 0.11 a	5.65 ± 0.18 a	5.49 ± 0.11 a
AN ( $\text{mg mg}^{-1}$ )	127.40 ± 28.25 a	120.05 ± 9.80 a	286.65 ± 11.23 b	289.10 ± 6.48 b
AP ( $\text{mg mg}^{-1}$ )	24.90 ± 10.60 a	11.67 ± 3.29 a	136.66 ± 11.16 b	194.04 ± 6.82 c
AK ( $\text{mg mg}^{-1}$ )	65.00 ± 4.33 a	107.50 ± 9.46 a	499.17 ± 173.93 b	800.00 ± 76.03 b
TN ( $\text{g kg}^{-1}$ )	1.51 ± 0.21 a	0.97 ± 0.07 b	3.19 ± 0.05 c	3.90 ± 0.02 d
TP ( $\text{g kg}^{-1}$ )	0.79 ± 0.09 a	0.56 ± 0.02 ab	1.16 ± 0.11 b	1.71 ± 0.22 c
TK ( $\text{g kg}^{-1}$ )	12.54 ± 0.29 a	18.39 ± 0.42 b	21.38 ± 0.70 bc	22.71 ± 2.13 c

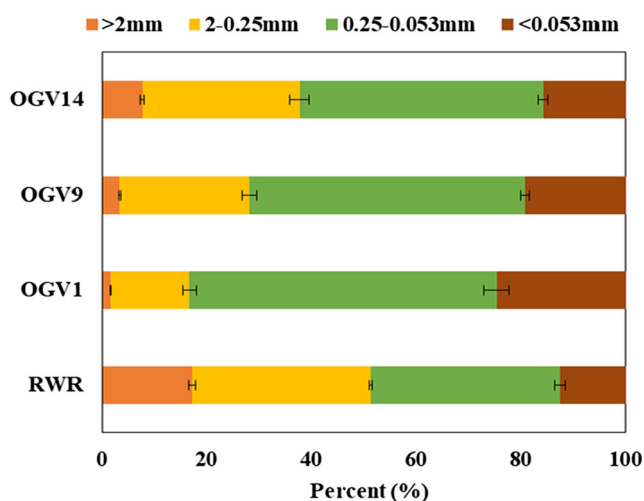
Notes: the different letters indicate significant differences among the four treatments (Duncan's post hoc test at  $p < 0.05$ ). The error bars are the standard errors

C, and carbonyl C. Besides, the increase in SOC fractions under long-term organic greenhouse vegetable plots were concentrated within macro-aggregate ( $> 0.25$  mm) and micro-aggregate (0.053–0.25 mm) fractions (Fig. 4). For example, the O-alkyl C in large macro-aggregate ( $> 2$  mm) obviously increased from 2.21 to 14.15  $\text{g kg}^{-1}$  in the fourteen-year organic plantation. Among that, O-alkyl C was mostly affected by organic manure application in both bulk soil and aggregate level. Even that, the proportion of the O-alkyl C in bulk soil tended to be opposite, decreasing from 52.1 to 43.21% under organic cultivation (Fig. 5). However, the proportions of the aromatic C and carbonyl C in bulk soil increased by 5.77% and 3.44%, respectively (Fig. 5). In addition, the SOC fractions within silt and clay ( $< 0.053$  mm) remained relatively stable throughout the entire study. Overall, organic manure could help to increase all the SOC fractions in both bulk soil and aggregate level; the change in aggregation was concentrated on within macro-aggregates ( $> 0.25$  mm) and micro-

aggregates (0.053–0.25 mm), and O-alkyl C increased most rapidly throughout the whole organic cultivation.

### 3.5 Pearson's correlations

The effects of soil aggregation properties on organic manure are complex, and we analysed the main factors in this study through Pearson's correlation. The results suggested that there was a significantly positive ( $P \leq 0.05$ ) relationship between BD and micro-aggregates (0.25–0.053 mm) opposite with the macro-aggregates (Table 3). Besides, there was no significant relation between the soil aggregate distribution and soil fertility indexes (e.g., soil available nutrients, soil total nutrients, and SOC) except total potassium (TK). However, the soil aggregate associated C had a significant relationship with these indexes, especially the SOC in large macro-aggregates ( $> 2$  mm) and micro-aggregates (0.25–0.053 mm). Similar results were also obtained in Table 4; the soil aggregate-associated C showed a more obvious relationship with SOC fractions compared with soil aggregation. On the one hand, the increase of these SOC fractions could be beneficial to soil aggregate-associated C especially in large macro-aggregates ( $> 2$  mm) and micro-aggregates (0.25–0.053 mm). On the other hand, the proportion of O-alkyl C had a significantly negative ( $P \leq 0.05$ ) effect on soil aggregate-associated C. On the contrary, the proportion of aromatic C and carbonyl C played a positive role on it. Above all, BD had a close relation with soil aggregation, and SOC and its fractions were related to soil aggregate-associated C. The proportion of O-alkyl C were less, the aggregate-associated C increased more.



**Fig. 1** Changes in aggregate size distribution under different treatments ( $n = 3$ ). The error bars illustrate the standard errors of the means ( $\pm$ ). RWR, rice-wheat rotation plots; OGV1, one-year-old organic greenhouse vegetable plots; OGV9, nine-year-old organic greenhouse vegetable plots; OGV14, fourteen-year-old organic greenhouse vegetable plots

## 4 Discussion

### 4.1 Effects of organic manure on soil aggregation and aggregate associated C

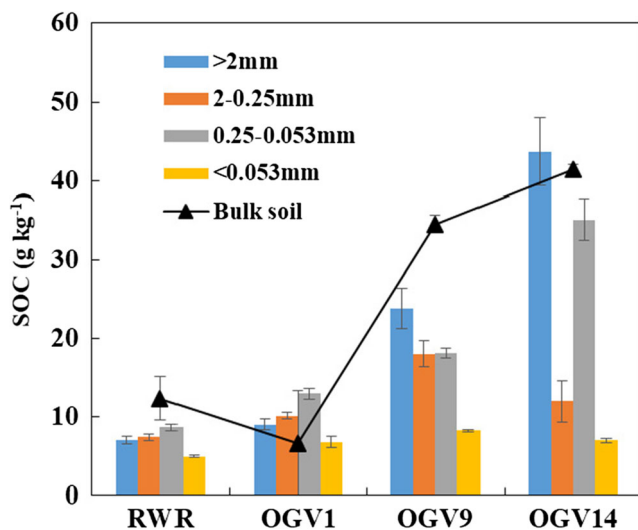
The distribution of soil aggregates reflects the soil physical properties and soil moisture supply (Wang et al. 2015). The

**Table 2** Indices of soil aggregate characteristics in long-term organic greenhouse fields converted from rice-wheat rotation ( $n = 3$ )

Treatments	RWR	OGV1	OGV9	OGV14
MWD (mm)	1.47 ± 0.06 a	0.36 ± 0.03 b	0.56 ± 0.04 c	0.88 ± 0.03 d
GWD (mm)	0.46 ± 0.01 a	0.14 ± 0.01 b	0.20 ± 0.02 b	0.28 ± 0.03 c
D	2.74 ± 0.02 a	2.86 ± 0.03 b	2.82 ± 0.03 ab	2.78 ± 0.03 ab
MWSSA (cm <sup>2</sup> g <sup>-1</sup> )	16.77 ± 0.91 a	30.06 ± 3.44 b	24.71 ± 2.21 b	20.93 ± 2.04 c

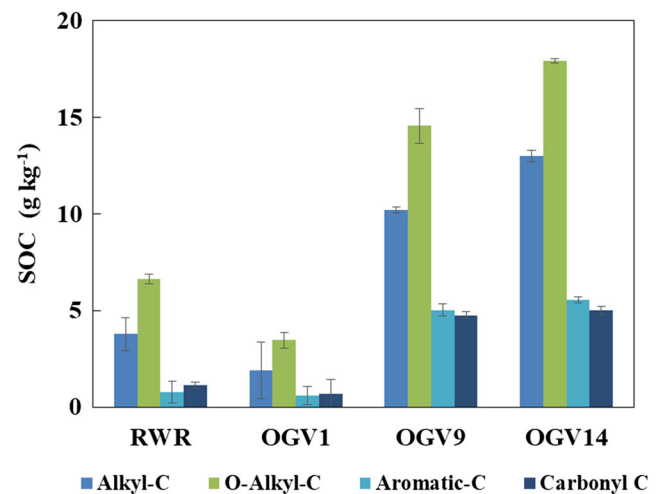
Notes: the different letters indicate significant differences among the four treatments (Duncan’s post hoc test at  $p < 0.05$ ). The error bars are the standard errors

proportion of macro-aggregates is higher; the soil structure is better (Zou et al. 2018). In our research, organic manure could increase the macro-aggregation (>0.25 mm) and improve soil aggregate stability (an increase of MWD and GWD values, decrease of D and MWSSA) (Fig. 1). Consistent conclusions were also obtained by prior studies in the comparisons of organic manure and chemical fertilizer, as well as the change of the different amount of the organic manure (Udom et al. 2016, Zhou et al. 2016). Guo et al. (2019a) also found the soil aggregate stability increased after organic manure application in southern China. Besides, Wang et al. (2014) investigated that vegetable fields converted from paddies over 20 years, and the results showed that 14.01% of water-stable macro-aggregates were broken into the micro-aggregates after the land-use conversion. Haghighi et al. (2010) also found the MWD in the pasture was higher than dryland framing. It showed that intensive land-use conversion might lead to the degradation of soil macro-aggregation. Compared with our results, we could find that the organic manure played a significantly ( $P \leq 0.05$ ) positive role in the soil macro-aggregate formation (Fig. 1).



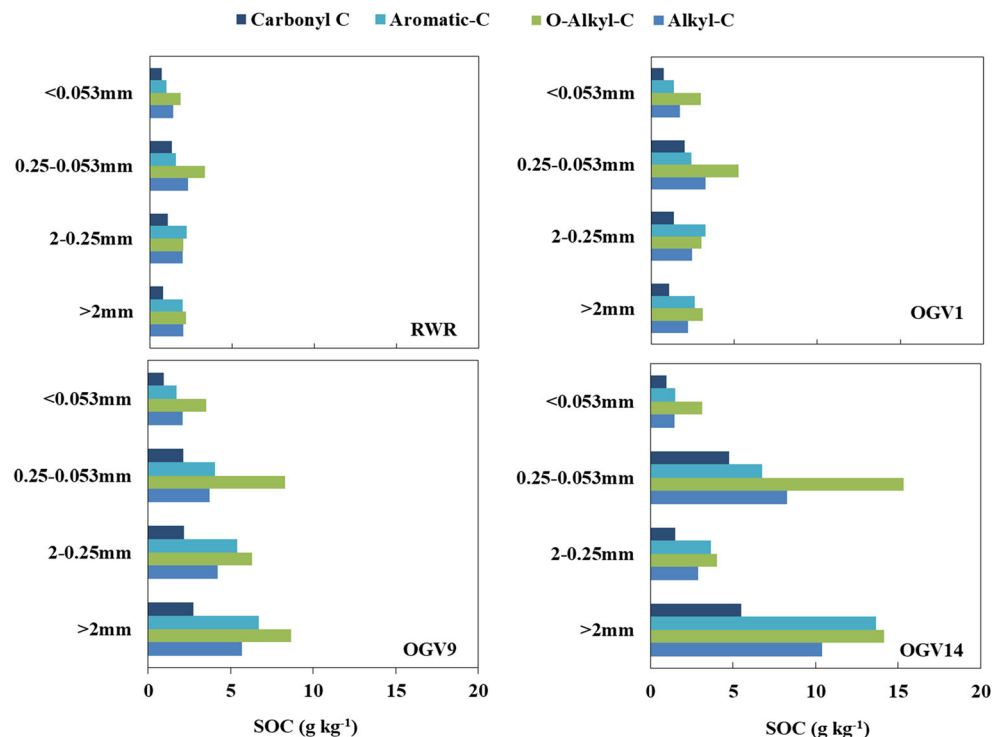
**Fig. 2** Changes in the SOC within bulk soil and water-stable aggregate fractions under different treatments ( $n = 3$ ). The error bars illustrate the standard errors of the means ( $\pm$ ). RWR, rice-wheat rotation plots; OGV1, one-year-old organic greenhouse vegetable plots; OGV9, nine-year-old organic greenhouse vegetable plots; OGV14, fourteen-year-old organic greenhouse vegetable plots

Besides, the soil aggregate associated C presented an obvious change during the 14-year organic plantation. The SOC within both large macro-aggregates (>0.25 mm) and micro-aggregates (0.053–0.25 mm) is strongly affected by organic manure (Fig. 2). SOC was the lowest within silt and clay (< 0.053 mm) size fractions. Similarly, E et al. (2012) noted that the SOC of aggregates was generally affected by organic manure, and that the magnitude of SOC increases was the greatest for large macro-aggregates. Bashir et al. (2016) also reported that the SOC within large macro-aggregates (2–8 mm) and micro-aggregates (0.05–0.25 mm) was the greatest in response to applications of 1% organic manure (the highest level in their study) in dryland under fallow-wheat rotation for two years. While, Xie et al. (2015) investigated the SOC in aggregate fractions in the long-term experiment of loess soil, and the results showed that fertilization did not affect the SOC in all aggregates compared with no nutrient input under dryland farming condition. Mangalassery et al. (2019) found the organic amendments increased SOC in all aggregate fractions in a weathered tropical soil. The difference could be mainly attributed to differences in soil types and climate conditions. Overall, continual organic manure input can increase fresh soil C contents, accelerate SOC turnover, and enhance



**Fig. 3** Mean SOC fractions under different treatments ( $n = 3$ ). The error bars illustrate the standard errors of the means ( $\pm$ ). RWR, rice-wheat rotation plots; OGV1, one-year-old organic greenhouse vegetable plots; OGV9, nine-year-old organic greenhouse vegetable plots; OGV14, fourteen-year-old organic greenhouse vegetable plots

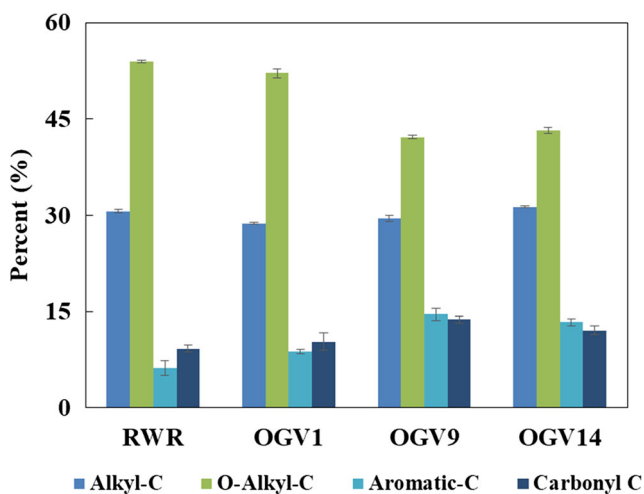
**Fig. 4** SOC fractions within different water-stable aggregates under different treatments. RWR, rice-wheat rotation plots; OGV1, one-year-old organic greenhouse vegetable plots; OGV9, nine-year-old organic greenhouse vegetable plots; OGV14, fourteen-year-old organic greenhouse vegetable plots



macro-aggregate formation (Jiang et al. 2015). It also played a positive role in macro-aggregate-associated C, as noted by Bronick and Lal (2005).

## 4.2 Benefits of organic manure on SOC fractions

The effect of long-term organic manure on SOC fractions is a complex and dynamic process. Among all the



**Fig. 5** Mean percentages of SOC fractions under different treatments ( $n = 3$ ). The error bars illustrate the standard errors of the means ( $\pm$ ). RWR, rice-wheat rotation plots; OGV1, one-year-old organic greenhouse vegetable plots; OGV9, nine-year-old organic greenhouse vegetable plots; OGV14, fourteen-year-old organic greenhouse vegetable plots

fractions, The O-alkyl C increased most rapidly throughout the whole organic cultivation (Fig. 3). O-alkyl C is the most readily available C source for microorganisms and consists of polysaccharides and cellulose, which are preferentially decomposed (Baldock et al. 1992). Plenty of organic materials input increased O-alkyl C following the continuous organic manure application. Meantime, the proportion of O-alkyl C decreased significantly ( $P \leq 0.05$ ) and remained stable after nine years of organic cultivation in contrast to that of alkyl C and aromatic C (Fig. 5). Generally, alkyl C and aromatic C are hydrophobic compounds, which are difficult to decompose (Guo et al. 2019b). In other words, the active SOC fractions increased readily, but compared with that in the original period, the composition of SOC tended to become more stable in response to applications of organic manure, which is consistent with the results of Preston et al. (2009). Moreover, the O-alkyl C within large macro-aggregates ( $> 2$  mm) increased most rapidly during the first nine-year cultivation period; thereafter, it increased within both large macro-aggregates ( $> 2$  mm) and micro-aggregates (0.053–0.25 mm) (Fig. 4). The amount and proportion of aromatic C within large macro-aggregates ( $> 2$  mm) was greater in the 14-year greenhouse plots than in the other plots, suggesting that the accumulated SOC became aromatic and stable over time (Fig. 4).

We speculated that the role of long-term organic manure on SOC fractions could be divided into two parts: one mainly involves the increase of the active C and soil

**Table 3** Pearson’s correlation between soil basic characteristics and soil aggregation parameters ( $n = 12$ )

	BD	AN	AP	AK	TN	TP	TK	SOC
Soil aggregation								
> 2 mm	-0.57	-0.24	-0.18	-0.21	-0.09	-0.03	<b>-0.67*</b>	-0.12
2-0.25 mm	<b>-0.80**</b>	0.22	0.33	0.24	0.35	0.41	-0.20	0.34
0.25–0.053 mm	<b>0.69**</b>	0.06	-0.03	0.09	-0.08	-0.16	0.42	-0.04
< 0.053 mm	0.56	-0.13	-0.20	-0.22	-0.23	-0.25	0.30	-0.24
Soil aggregate associated carbon								
> 2 mm	-0.56	<b>0.81**</b>	<b>0.93**</b>	<b>0.86**</b>	<b>0.90**</b>	<b>0.91**</b>	<b>0.79**</b>	<b>0.88**</b>
2-0.25 mm	-0.13	<b>0.68*</b>	0.50	0.53	0.15	0.15	0.45	0.56
0.25–0.053 mm	-0.46	<b>0.76**</b>	<b>0.87**</b>	<b>0.89**</b>	<b>0.83**</b>	<b>0.83**</b>	<b>0.74**</b>	<b>0.82**</b>
< 0.053 mm	0.06	<b>0.61*</b>	0.52	0.51	0.50	0.35	<b>0.79**</b>	0.52

Notes: bold values represent a significant correlation between the indexes

\*means  $P \leq .05$  with Duncan’s post hoc test

\*\*means  $P \leq .01$  with Duncan’s post hoc test

fertility improvement, and the other includes C sequestration. The effect of organic manure on SOC fractions still has been debated; some research found that farmyard manure applications can increase the proportion of alkyl C and aromatic C but decrease the proportion of O-alkyl C (Guo et al. 2019b), whereas another long-term experiment suggested that compost applications can increase the proportion of alkyl C and O-alkyl C (Lima et al. 2009). The difference might be attributed to the different kinds of organic manure materials. For example, the proportion of O-alkyl C in straw is approximately 70% (Mahieu et al. 1999), but it is nearly 30% within pig manure (Zhang et al. 2013). Overall, the organic manure could help the SOC fraction stock variously depending on the different kinds of the manure.

### 4.3 Interactions among soil aggregation characteristics and SOC fraction parameters

The increase of SOC could promote the macro-aggregate formation (Plaza-Bonilla et al. 2013, Sui et al. 2012, Trakooyingcharoen et al. 2012, Zhao et al. 2018). In our research, the SOC and its fractions did not have a significant ( $P > 0.05$ ) relation with soil aggregation, but it affected soil aggregate associated C (Table 4). In other words, the effects of SOC on soil aggregation might promote the turnover of SOC in the aggregate size fractions to format the soil aggregation. Besides, SOC played a significantly ( $P \leq 0.05$ ) positive role in soil aggregate-C in macro-aggregates (> 2 mm) and micro-aggregates (0.25–0.053 mm), consistent with the results from Bronick and Lal (2005) and Udom et al. (2016). Similarly, the

**Table 4** Pearson’s correlation between SOC fractions and soil aggregation characteristics ( $n = 12$ )

	Alkyl C (P)	O-alkyl C (P)	Aromatic C (P)	Carbonyl C (P)	Alkyl C (A)	O-alkyl C (A)	Aromatic C (A)	Carbonyl C (A)
Soil aggregation								
> 2 mm	0.49	0.43	-0.57	-0.52	-0.10	-0.06	-0.26	-0.21
2–0.25 mm	<b>0.69*</b>	0.00	-0.15	-0.22	0.36	0.41	0.19	0.23
0.25–0.053 mm	-0.52	-0.27	0.41	0.39	-0.06	-0.10	0.09	0.04
< 0.053 mm	<b>-0.59*</b>	-0.03	0.15	0.23	-0.27	-0.31	-0.12	-0.14
Soil aggregate associated carbon								
> 2 mm	0.46	<b>-0.82**</b>	<b>0.75**</b>	<b>0.62*</b>	<b>0.90**</b>	<b>0.86**</b>	<b>0.87**</b>	<b>0.84**</b>
2-0.25 mm	-0.22	<b>-0.71*</b>	<b>0.77**</b>	<b>0.75**</b>	0.52	0.53	<b>0.65*</b>	<b>0.63*</b>
0.25-0.053 mm	0.47	<b>-0.76**</b>	<b>0.70*</b>	0.53	<b>0.85**</b>	<b>0.80**</b>	<b>0.81**</b>	<b>0.79**</b>
< 0.053 mm	-0.19	<b>-0.75**</b>	<b>0.78**</b>	<b>0.81**</b>	0.50	0.46	<b>0.62*</b>	<b>0.61*</b>

Notes: “P” presents the proportion of SOC fractions, “A” represents the amount of SOC fractions. Bold values represent a significant correlation between the indexes

\*means  $P \leq .05$  with Duncan’s post hoc test

\*\*means  $P \leq .01$  with Duncan’s post hoc test



previous research also suggested that SOC was stabilized in both macro-aggregates (> 0.25 mm) and micro-aggregates (0.25–0.063 mm), and the physical-chemical protection of SOC was the key aspect to maintain soil C stocks (Garcia-Franco et al. 2015).

According to the role of SOC fractions, all SOC fractions amount had a significantly ( $P \leq 0.05$ ) positive role on soil aggregate associated C, consistent with the relation between total SOC and soil aggregate associated C. Pulido-Moncada et al. (2018) found that there were significant associations between aggregate stability and SOC (0.79\*\*), light fraction (0.69 \*\*) and heavy fraction (0.70\*\*). It meant that all SOC fractions stock could improve soil aggregate associated C, while, the level of the effects was different. With regard to the proportion of SOC fractions, the low proportion of O-alkyl C could accumulate soil aggregate associated C, opposite to the results of aromatic C and carbonyl C (Table 4). O-alkyl C is always recognized as the active C fractions, which is easily affected by external conditions (Erhagen et al. 2013). In other words, stable C fractions (e.g., aromatic C and carbonyl C) played a more obvious positive role on soil aggregate associated C rather than active C fractions. However, the research of Sarker et al. (2018) suggested O-alkyl C was positively associated with the aggregation index, but the aromatic fractions showed an opposite pattern. The difference might be due to the experiment time. Sarker et al. (2018) were concentrated on the short term observation over the 300 days of incubation, and the effects of aromatic C fractions could not play a role in this period. Overall, there was a close relation between SOC fractions and aggregation associated C, and the stable C fraction might play a more important role during the long-term organic manure application.

## 5 Conclusions

This study analysed the aggregation and SOC characteristics in response to long-term organic manure applications. The results suggested that both macro-aggregates and SOC and its fractions increased under fourteen years of organic cultivation. There was the most rapid increase in O-alkyl C during the organic greenhouse vegetable plantation, but its proportion decreased and then kept stable over time opposite to alkyl C and aromatic C. The change of SOC in aggregate sizes was mostly concentrated on large macro-aggregates (> 2 mm) and micro-aggregates (0.25–0.053 mm), the change in silt and clay (< 0.053 mm) fractions was little. Besides, the correlation between soil aggregation characteristics and other properties were analysed. It suggested that all soil fertilizer indexes did not have a significant ( $P > 0.05$ ) relation with soil aggregation distribution, but it had a close relation with soil aggregate associated C. All of these soil fractions amounts could increase soil aggregate associated C stock, but the proportion of aromatic C and carbonyl C played a more positive role than

O-alkyl C. It might improve our knowledge on how organic manure may influence soil aggregation by increasing SOC after long-term greenhouse vegetable plantation. Consequently, it has a theoretical significance for sustainable agricultural practices.

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