Pol. J. Environ. Stud. Vol. 25, No. 3 (2016), 1215-1221
DOI: 10.15244/pjoes/61628

Original Research

How 23-year Continuous Soybean Cultivation Led to More SOC and Thermal Energy Stored in Mollisol Micro-Aggregates

Yunfa Qiao[1], Shujie Miao[1]*, Shuping Yue[1], Haiyan Wu[2], Xiaozeng Han[3]

[1]Land Carbon Water Cycle and Climate Change Innovation Team,
Nanjing University of Information Sciences and Technology,
Nanjing, 210044, China
[2]Jinlin Academy of Agricultural Sciences
[3]Northeast Institute of Geography and Agroecology, Chinese Academy of Sciences,
Harbin 150081, China

Received: 30 December 2015
Accepted: 30 January 2016

Abstract

Aggregate has been recognized as a key element in the stabilization of soil organic carbon (SOC). Several researchers have done outstanding work on identifying and isolating aggregates and their physiochemical properties. However, thermal stability of SOC in soil aggregates has not yet been adequately explored. The main objective of the study was to clarify the protection of aggregation on SOC from thermal characters, and provide evidence on whether thermal analysis could be a potential rapid method to determine SOC stability in aggregates. We separated 20-cm surface soil into six fractions (>2, 1-2, 0.5-1, 0.25-0.5, 0.053-0.25 and <0.053mm) before and after 23-yr continuous soybean cultivation. The study measured the change of SOC and its thermal characteristics across aggregates using thermogravimetry-differential scanning calorimetry (TG-DSC), which also showed that the thermal stability mechanism of SOC is protected by aggregates. Results showed that 23-yr continuous soybean cultivation led to an SOC increase in 0.053-0.5 mm size aggregates, but a decrease in other large-size aggregates. Energy density in the > 0.5 mm fraction was decreased by 23-yr continuous soybean cultivation, but increased to < 0.5 mm size fraction. The largest energy density was in < 0.053 mm size fractions. In conclusion, long-term continuous soybean cultivation led to more energy transferred to micro-aggregates associated with the protection of micro-aggregates on soil SOC.

Keywords: aggregate, SOC, TG-DSC, energy content, energy density

*e-mail: miaoshujie@126.com

Introduction

Soil aggregates are important agents for SOC retention and protection [1, 2]. It is generally accepted that the stability of SOC in soil aggregates results from the protection of aggregate architecture to microbial decomposition. Using wet sieving procedures in the laboratory, soil aggregates are sub-divided into macro-aggregates (>0.25 mm) and micro-aggregates (<0.25 mm). Dominant SOC stored in the micro-aggregates more than macro-aggregates [2-4] and the SOC in micro-aggregates will be more stable and resistant to degradation [5, 6]. These results indicate that the micro-aggregate fraction has promising potential to explain the soil C turnover in corresponding to crop cultivation. Furthermore, soil aggregation and SOC protection are both long-term processes and less than five years field of experiments could not fully present the real mechanisms included in them. Studies based on long-term experimentation were required to clarify the aggregate mechanism and its role in protecting SOC.

The protection of SOC in soil aggregates is linked with the quantity and quality of SOC [7, 8]. Researchers have to extract SOC from soil aggregates with chemical reagent before studying their mechanisms in them. Though there is a great amount of literature on SOC stocks and sequestration under various management practices based on chemical extraction, a lot of time and manpower have been wasted. Due to the increasing demands for rapid and quantitative assessments of SOC quality, thermal analysis techniques are a unique means to assess soil organic matter quality in reference to decomposability and turnover [9]. Thermal analysis techniques were applied to study the soil science since 1935, particularly in clay mineralogy components [9-11], but thermal properties of soil organic matter have received much less attention. This might be because organic components can influence thermal behavior to a much greater degree than mineral components [12]. Only recently have some studies tested the thermal properties of soil organic matter, and the link between thermal and biological stability of SOC [13-15]. This research indicates that energy density and DSC-T_{50} were strongly corrected with respiration indices derived by incubation, which could be used for forecasting SOM stability [15]. Even though some research focused on SOM thermal stability, to our knowledge there are no reports on thermal characteristics of various soil aggregates.

The objective of this work was to ascertain the protection mechanism of aggregates on SOC from thermal characteristics, and provide evidence of potential rapid analytical tools to determine the turnover and stability of SOM in aggregates. For this aim, a 23-yr continuous soybean cultivation experiment was used. Samples before and after long-term continuous soybean cultivation were submitted for thermal analysis. These results together with the C concentration and changes in aggregates would tell us the thermal stability of SOC in aggregates, and indicated a significant advance in rapid and cost-effective assessments of SOC stability that are indirectly related to chemical composition.

Materials and Methods

Site Description

The long-term continuous soybean cultivation experiment was located at the National Field Research Station of Agro-ecosystem of CAS in Hailun County, Heilongjiang Province, China. The research area is located at 47°26'N and 126°38'E at an altitude of 240 m. The mean annual temperature is 2.2ºC, with the highest monthly temperature in July (35ºC) and lowest in January (-38ºC). The studied soil is Pachic Haploborolls as classified by USDA Taxonomy (Soil Survey Staff 2010), with clay 400 g kg^{-1} and silt 258 g kg^{-1}. Before establishing the soybean/maize rotation and continuous cropping experiment, the soil contained 29.8 g kg^{-1} of SOC, 2.2 g kg^{-1} of total N, 0.74 g kg^{-1} of total P, and 20.8 g kg^{-1} of total K. Soil pH in water (1:2.5) was 6.05. The experiment was established in 1991 to evaluate the ecological effect of maize-soybean-wheat rotation and continuous cropping. Randomized block design with four replicates was applied. The plot area covered around 77 m^2. More detailed information could be found in a previous report by Qiao et al. [16].

Aggregate Preparation

Soil samples were collected using a sampling shovel (20 cm width, 30 cm depth) from three sites in each plot after harvesting one-year soybean cultivation in 1992. Collected soil samples were air-dried and stored in soil samples stored at room temperature (25°C) until analysis began. Soil was sampled and treated with the same method in 1992 as after 23-yr continuous soybean cultivation in 2014. Aggregate fractions were separated with the wet-sieving method using a modified Yoder-type apparatus and the method described by Qiao et al. [16]. A 10 g subsample of dried samples was submerged in water and separated by moving a cascade of sieves with openings of 5, 2, 1, 0.5, 0.25 mm at a frequency of 32 oscillations per min with a stroke length of 3.8 cm for 10 min. Materials floating on the water were dredged up and discarded. The aggregates (<0.25 mm) were then collected and sieved through a 0.053 mm mesh so that 0.053-0.25 mm size aggregates were obtained. All fractions were collected and transferred to pre-weighted 50-ml beakers, oven-dried at 50°C for 48 h, and weighed. The collected dry aggregates were then recombined to generate six aggregate fractions: i) >2 mm, ii) 1-2 mm, iii) 0.5-1 mm, iv) 0.25-0.5 mm, v) 0.053-0.25 mm, and vi) <0.053mm.

Organic Carbon Analysis

Subsamples of differently sized aggregates were used for organic carbon (OC) concentration analysis. The OC concentration was measured by dry combustion using a VarioEL CHN elemental analyzer (Heraeus Elementar Vario EL, Hanau, Germany). Total C is equivalent to total organic carbon because there is no carbonate present in the soil.

Thermal Analysis

Another subsample of differently sized aggregates was analyzed with a Netzsch simultaneous heat flux thermal analyser (STA 409 PC Luxx) equipped with a type-S (Pt/PtRh) TG-DSC sample carrier supporting a PtRh10-Pt thermocouple (Netzsch-Geratebau GmbH, Selb, Germany). Samples were heated from ambient to 700ºC in a furnace atmosphere of laboratory-grade CO_2-free 'zero' air at 10ºC min^{-1} flowing at 50 mL min^{-1}, plus protective gas flowing at 10 mL min^{-1}. During baseline correction, regions <190ºC and >600ºC were defined as baseline supported by Plante et al. [15, 17]. Total exothermic energy content (in J) was determined by integrating the DSC heat flux (in mW) over the exothermic region at 190-600ºC. Mass loss was determined for the same temperature range. Energy density (J mg^{-1} OM) was thus determined by dividing energy content by thermogravimetric mass loss reported by Rovira et al. [18].

Statistical Analysis

Statistically significant differences were identified using analysis of variance (ANOVA) at $p < 0.05$. Changes in aggregation percentage and SOC content across aggregates were conducted with SAS (SAS Institute Inc., 9.1.3 Portable). The standard errors of means were presented in the tables as variability parameter.

Results

Aggregation and SOC

23-yr continuous soybean cultivation had a significant effect on aggregate distribution in soil (Fig. 1). In 2014 the percentage of 0.25-0.5 mm and 0.053-0.25 mm aggregates were increased by 2.94% and 6.21%, respectively, compared to 1992. However, the > 2mm and 0.5-1 mm fractions were decreased by 3.86% and 3.03%, and the 1-2 mm and < 0.053 mm fractions were decreased by 1.05% and 1.21%, respectively. Correspondingly, we calculated the change in C content of aggregate size from 100 g soil after 23-yr continuous soybean cultivation. The C content in 0.25-0.5 mm and 0.053-0.25 mm aggregate size were increased by 171 and 221 mg/100 g soil, while C content in other aggregate sized were decreased with the largest decrease in >2 mm fraction (Fig. 1).

Feature of TG and Mass Loss in Aggregates

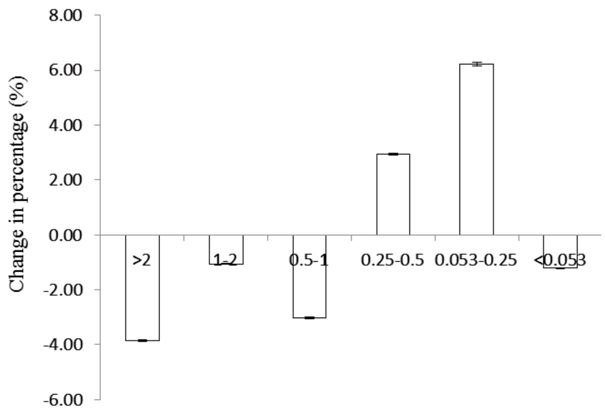
TG curves are shown in Fig. 2, in all cases, which showed a smooth curve without clear bi-modal shape. Almost no weight loss was detected above 600ºC in any cases. Thus, we detected the mass loss at 190-600ºC periods. In 1992 the mass loss across the >0.053 mm fraction was similar and averaged 6.34%, which was significantly larger than that of the <0.053 mm fraction, which averaged 4.22% (Table 1). After 23-year continuous soybean cultivation the lowest mass loss was obtained in the <0.053 mm fraction (4.11%) and the highest was in the >2 mm fraction (7.18%) (Table 1). In contrast, 23-year continuous soybean cultivation led to increased mass loss in the >0.053 mm fraction and increased with aggregate size increase, while there was almost no mass loss in the <0.053 mm fraction (Table 1).

DSC Feature and Peak Position

Typical DSC curves are shown in Fig. 3. In all cases, a bi-modal shape is obtained, with labile and recalcitrant peaks. The labile peak is placed at the 342-363ºC zone (mean value: 352ºC), and the recalcitrant peak at 423-438ºC zone (mean value: 434ºC).

The exact positions of the labile and recalcitrant peaks were quite variable (Table 1). The position of the labile peak shifted to lower temperature after 23-yr soybean continuous cultivation in the > 1 mm fraction, while < 1 mm fractions tended to be a higher temperature zone. Though there was no significant difference among various aggregate sizes for labile peaks, the largest peak was shown

a)



b)

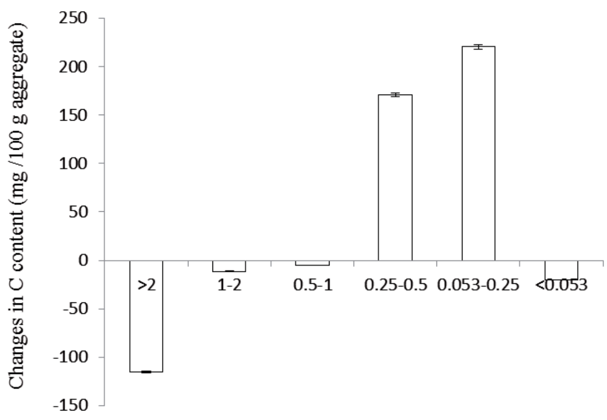


Fig. 1. Changes in: a) various size aggregate percentages to bulk soil and b) organic carbon content (means and standard deviations) for each size aggregate after 23-yr long-term continuous soybean cultivation.

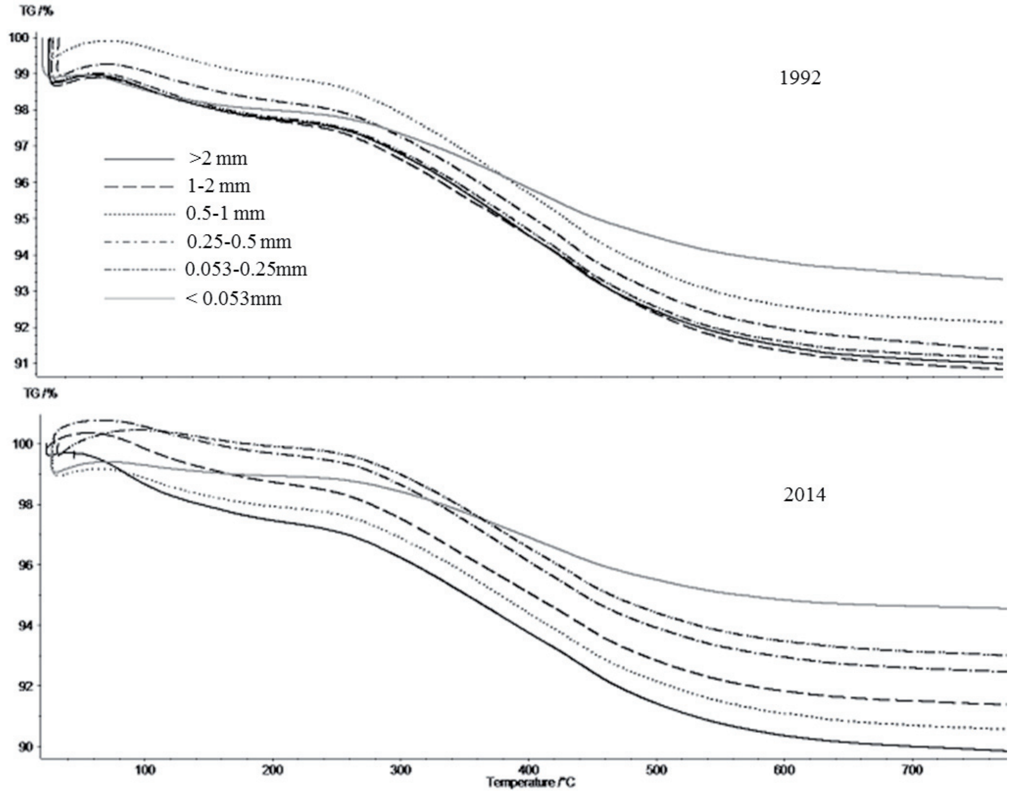


Fig. 2. TG curves of > 2mm, 1-2 mm, 0.5-1 mm, 0.25-0.5 mm, 0.053-0.25 mm, and < 0.053 mm aggregates before (1992) and after (2014) 23-yr continuous soybean cultivation.

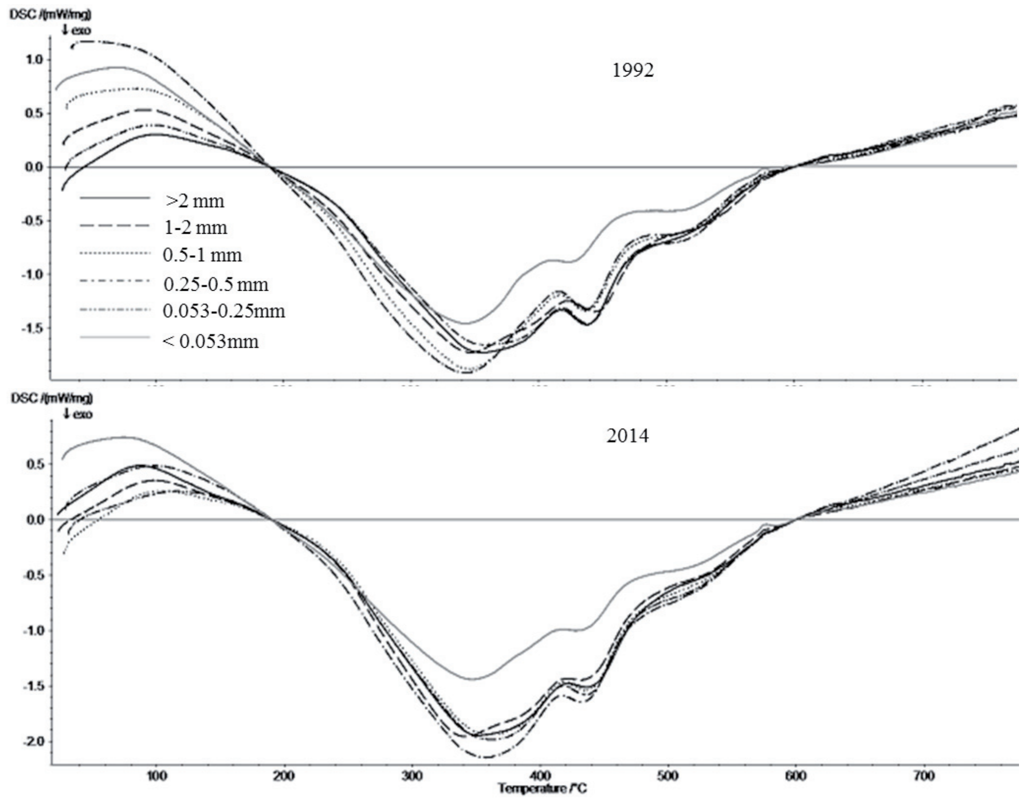


Fig. 3. Differential scanning calorimetry (DSC) curves of > 2mm, 1-2 mm, 0.5-1 mm, 0.25-0.5 mm, 0.053-0.25 mm, and < 0.053 mm aggregates before (1992) and after (2014) 23-yr continuous soybean cultivation.

Table 1. Mass loss (%) in TG analysis and position of the peaks in ºC with DSC analysis. Data are means ± standard deviations.

	>2	1-2	0.5-1	0.25-0.5	0.053-0.25	<0.053
Mass loss						
1992	6.31±0.35	6.43±0.42	6.39±0.41	6.35±0.26	6.24±0.23	4.22±0.12
2014	7.18±0.12	6.97±0.21	6.89±0.11	6.78±0.10	6.47±0.08	4.11±0.12
Labile peak						
1992	354.9±0.3	346.7±0.2	346.1±0.5	344.0±0.2	363.0±0.4	343.7±0.3
2014	353.0±0.2	342.3±0.1	363.1±0.7	357.6±0.3	363.3±0.6	347.9±0.3
Recalcitrant peak						
1992	437.6±0.2	438.0±0.3	437.4±0.4	437.3±0.4	438.2±0.5	423.7±0.1
2014	436.7±0.3	432.4±0.1	436.1±0.3	433.2±0.2	436.6±0.4	427.0±0.1

in the 0.053-0.25 mm fraction. For recalcitrant peaks, the peak temperature shifted to the low-temperature zone after 23-yr continuous soybean cultivation, with the exception of the < 0.053 mm fraction. The lowest peak temperature was obtained in the < 0.053 mm fraction (Table 1).

Energetic State

The integrated energy output calculated from DSC curves depended on aggregate size and soybean cultivation (Table 2). The integrated energy output increased with aggregate size decrease to 0.5-1 mm fraction (largest value of 32.92 J), then decreased to 22.18 J in the < 0.053 mm fraction in 1992. The same trend was obtained in 2014, but the largest value of 36.28 J was shown in the 0.25-0.5 mm fraction. In contrast, the integrated energy output was promoted by 23-yr continuous soybean cultivation in all aggregate sizes.

The < 0.053 mm fraction had the largest energy densities, which were 16.89 J mg^{-1} OM in 1992 and 18.33 J mg^{-1} OM in 2014 (Table 2). For other aggregates, the energy density increased as aggregate size decreased, to the point where the 0.25-0.5 mm fractions showed the second largest value, then decreased again for both years. Overall, the energy densities were generally greater for the > 0.5 mm fraction in 2014 compared to 1992, and lower for the < 0.5 mm fraction in 2014 compared to 1992 (Table 2).

Discussion

Physical Protection of SOC Across Aggregates

23-yr continuous soybean cultivation significantly decreased the percentage of > 0.5 mm fraction aggregate to total aggregates, which is typical of long-term cropping disturbance macro-aggregates [4, 6]. During cultivation, machines integrated with anthropogenic disturbance will significantly affect soil macro-aggregate stability and distribution [19, 20]. Thus, after 23-yr continuous soybean cultivation leading to a larger aggregate breakdown into smaller aggregates, with the percentage of 0.25-0.5 mm and 0.053-0.25 mm fractions increased by 2.94% and 6.21%, respectively. However, the silt+clay-size aggregate

Table 2. Integrated energy output (J) and energy density (J mg^{-1} OM) of before (1992) and after (2014) long-term soybean cultivation. Energy density is the integral of the differential scanning calorimetry (DSC) signal (in J) divided by the thermogravimetric (TG) mass loss of organic matter (in mg) – both determined over the exothermic region 190-600ºC.
Data are means ± standard deviations.

	>2	1-2	0.5-1	0.25-0.5	0.053-0.25	<0.053
Integrated energy output						
1992	29.51±0.6	32.14±0.7	32.92±0.5	32.01±0.7	29.96±0.5	22.18±0.3
2014	32.34±0.8	34.10±0.9	34.11±0.8	36.28±0.9	33.99±0.5	23.44±0.4
Energy density						
1992	14.98±0.4	14.97±0.2	15.41±0.3	15.91±0.4	14.88±0.3	16.89±0.5
2014	14.43±0.2	14.65±0.3	14.80±0.2	16.89±0.6	16.29±0.5	18.33±0.6

fraction (<0.053 mm) did not increase after long-term cultivation, which is not consistent with a continuous maize cropping system [4]. This should be attributed to soybean and its relatively smaller root mass compared to maize, which promotes primary mineral particles into micro-aggregate [21].

To test the physical protection of soil aggregate on SOC, 100 g soil was used to calculate the C content change in various sized aggregates. Higher C content in the micro-aggregates (0.053-0.5 mm fraction) compared to the macro-aggregates (>0.5 mm fraction) in our results were similar to previous studies [2, 4]. All these suggested that a loss of C-rich large-sized aggregate by long-term cultivation, and a gain of C-rich small size aggregate. In the long-term continuous soybean cultivation plots the role of macro-aggregate in protecting SOC showed short-term storage, with much more C stored in the smaller aggregate (0.053-0.5 mm fraction). Several previous studies had found that the SOC turnover is more rapid in macro-aggregates compared with micro-aggregates [22, 23]. This can be explained by macro-aggregates being formed with plant root, oxidation, and dead material, which is easily broken down by cropping practices.

Thermal Properties and SOC Protection Across Years and Aggregates

Thermal analysis showed significant differences in the exothermic reactions in the region 190-600ºC. In 1992 all aggregates were mainly characterized by two peaks of around 350ºC and 435ºC. Corresponding values were around 355ºC and 434ºC in 2014. According to several studies [10, 24], the peak of < 400ºC has typically been attributed to labile SOC, and that of > 400ºC is attributed to more stable SOC. Thus, the present results indicate that 23 years of continuous soybean cultivation led to more stable carbon storage in soil. This could be attributed to special intensive crop cultivation with N application favoring easily available C turnover in soil, which further delayed more complex and recalcitrant organic compound availability [25, 26].

The peak position of thermal labile fraction in 0.25-1 mm aggregate size showed the largest increase after 23-yr continuous soybean cultivation. The transfer to a higher temperature zone indicated a lower thermal stability and easily decomposed SOC stored in the macro-aggregate fractions. This is in line with other studies showing more labile C in macro- than micro-aggregates [27]. However, the recalcitrant peak temperature in the > 0.053 mm fraction tended to lower after 23-yr continuous soybean cultivation, and only the < 0.053 mm size tended to increase. These results could be explained by long-term cultivation management resulting in intensive soil disturbances exposing micro-aggregates and the SOC included in them with more rapid decomposition [2, 19, 22, 28]. In the end, more stable SOC is stored in clay-organic carbon complex [29]. All these indicated that organic-mineral complexes might be the key attributes determining the turnover of SOM.

Energy content is inherently a function of SOC quantity [15]. The present study showed higher integrated energy output after 23 years of continuous soybean cultivation. This told us again that SOC tends to be more stable after long-term cropping. However, the highest energy content appeared in the 0.5-1 mm fraction in 1992 and the 0.25-0.5 mm fraction in 2014, according to Plante et al. [15], who reported that high energy content represents high SOC stability. The present result was inconsistent with the more stable C stored in a < 0.053 mm fraction [15].

In the present study, energy content might be not a good parameter to describe SOC stability across aggregates. The highest energy density value was shown in the < 0.053 mm fraction both in 1992 and 2014. This could be explained by mineral-associated organic matter has higher thermal stability [30], which supports the SOC adsorbed on the < 0.053 mm fraction that had a lower mineralization rate [31]. This is associated with the energy density increasing in a > 0.5 mm fraction and decreasing in a < 0.5 mm fraction after 23 years of cultivation. The present results showed that long-term cropping led to more energy stored in micro-aggregates. All these were consistent with the protection mechanisms in aggregates from physiochemical and biochemical properties [6, 32].

Conclusions

Our findings suggest that micro-aggregates play key roles in protecting SOC based on more recalcitrant SOC stored in micro-aggregates. The turnover mechanisms included in aggregates were associated with the dynamics of thermal reaction, such as mass loss, peak position, and energy density. All these suggested that thermal analysis could be a potential rapid analytical tool to determine the turnover and stability of SOM in aggregates. However, it is necessary to test the thermal characteristics and C turnover across aggregates with ^{13}C labeled technology to fully understand the relationship between thermal stability and biological stability.

Acknowledgements

This work was supported by the National Natural Science Foundation of China (Nos. 41371297 and 41471240) and the Nanjing University of Information Science and Technology Talent Start-up Fund (Nos. 2015r021 and 2015r020). Thermal characteristics were analyzed at the Department of Land, Air, and Water Resources at the University of California–Davis.

References

1. HAILE S.G., NAIR P.K.R., NAIR V.D. Carbon storage of different soil-size fractions in Florida silvopastoral systems. J. Environ. Qual. **37**, 1789, **2008**.

2. SIX J., PAUSTIAN K., ELLIOTT E.T., COMBRINK C. Soil structure and soil organic matter: I. Distribution of aggregate size classes and aggregate associated carbon. Soil Sci. Soc. Am. J. **64**, 681, **2000.**
3. CHRISTENSEN B.T. Physical fractionation of soil and structural and functional complexity in organic matter turnover. Eur. J. Soil Sci. **52**, 345, **2001**.
4. KOU T.J., ZHU P., HUANG S., PENG X.X., SONG Z.W., DENG A.X., GAO H.J., PENG C., ZHANG W.J. Effects of long-term cropping regimes on soil carbon sequestration and aggregate composition in rainfed farmland of Northeast China. Soil Till. Res. **118**, 132, **2012**.
5. FAZLE RABBI S.M., WILSON B.R., LOCKWOOD P.V., DANIEL H., YOUNG I.M. Soil organic carbon mineralization rates in aggregates under contrasting land uses. Geoderma **216**, 10, **2014**.
6. SIX J., PAUSTIAN K. Aggregate-associated soil organic matter as an ecosystem property and a measurement tool. Soil Biolo. Biochem. **68**, A4, **2014**.
7. NOVARA A., GRISTINA L., MANTIA T.L., RUHL J. Carbon dynamics of soil organic matter in bulk soil and aggregate fraction during secondary succession in a Mediterranean environment. Geoderma, **193-194**, 213, **2013**.
8. BOSÁK M., HAJDUOVÁ Z., MAJERNÍK M., ANDREJOVSKY P. Experimental-energy combustion of biomass combined with coal in thermal power plants. Pol. J. Environ. Stud. **24**, 1517, **2015**.
9. PLANTE A.F., FERNANDEZ J.M., LEIFELD J. Application of thermal analysis techniques in soil science. Geoderma **153**, 1, **2009**.
10. PLANTE A.F., PERNES C., CHENU C. Changes in clay-associated organic matter quality in a C depletion sequence as measured by differential thermal analyses. Geoderma **129**, 186, **2005**.
11. ARAB P.B., ARAÚJO T.P., PEJON O.J. Identification of clay minerals in mixtures subjected to differential thermal and thermogravimetry analyses and methylene blue adsorption tests. Appl. Clay Sci. **114**, 133, **2015**.
12. LANGIER-KUZNIAROWA W. Thermal analysis of organo-clay complexes. Yariv, S.; Cross, H., Marcel-Dekker, New York. **2002**.
13. DORODNIKOV M., FANGMEIER A., KUZYAKOV Y. Thermal stability of soil organic matter pools and their $\delta^{13}C$ values after C3-C4 vegetation change. Soil Biol. Biochem. **39**, 1173, **2007**.
14. DUGUY B., ROVIRA P. Differential thermogravimetry and differential scanning calorimetry of soil organic matter in mineral horizons: Effect of wildfires and land use. Org. Geochem. **41**, 742. **2010**.
15. PLANTE A.F., FERNÁNDEZ J.M., HADDIX M.L., MEGAN STEINWEG J., CONANT R.T. Biological, chemical and thermal indices of soil organic matter stability in four grassland soils. Soil Biol. Biochem. **43**, 1051, **2011**.
16. QIAO Y.F., MIAO S.J., LI N., HAN X.X., ZHANG B. Crop species affect soil organic carbon turnover in soil profile and among aggregate sizes in a Mollisol as estimated from natural ^{13}C abundance. Plant Soil **349**, 306, **2015**.
17. MIAO S.J., QIAO Y.F., YOU M.Y., ZHANG F.T. Thermal stability of soil organic matter was affected by 23-yr maize and soybean continuous cultivation in Northeast of China. J. Therm. Anal. Calorim. doi: DOI: 10.1007/s10973-015-4709-7), **2015**.
18. ROVIRA P., KURZ-BESSON C., COUTEAUX M.M., VALLEJO V.R. Changes in litter properties during decomposition: A study by differential thermogravimetry and scanning calorimetry. Soil Biol. Biochem. **40**, 172, **2008**.
19. LICHTER K., GOVAERTS B., SIX J., SAYRE K.D., DECKERS J., DENDOOVEN L. Aggregation and C and N contents of soil organic matter fractions in a permanent raised-bed planting system in the Highlands of Central Mexico. Plant Soil **305**, 237, **2008**.
20. PINHEIRO E.F.M., PEREIRA M.G., ANJOS L.H.C. Aggregate distribution and soil organic matter under different tillage systems for vegetable crops in a Red Latosoil from Brazil. Soil Till. Res. **77**, 79, **2004**.
21. LAGALY G., OGAWA M., DEKANY I. Chapter 10.3-Clay mineral-Organic interactions. Developments in Clay Science. **5**, 435. Handbook of Clay Science, **2013**.
22. QIAO Y.F., MIAO S.J., LI N., HAN X.Z., ZHANG B. Spatial distribution of rhizodeposit carbon of maize (*Zea mays* L.) in soil aggregates assessed by multiple pulse ^{13}C labeling in the field. Plant Soil **375**, 317, **2014**.
23. SIX J., CONANT R.T., PAUL E.A., PAUSTIAN K. Stabilization mechanisms of soil organic matter: implication for C-saturation of soils. Plant Soil **241**, 155, **2002**.
24. MASTROLONARDO G., FRANCIOSO O., DI FOGGIA M., BONORA S., RUMPEL C., CERTINI G. Application of thermal and spectroscopic techniques to assess fire-induced changes to soil organic matter in a Mediterranean forest. J. Geochem. Explor. **143**, 174, **2014**.
25. HAGEDORN F., SPINNLER D., SIEGWOLF R. Increased N deposition retards mineralisation of old soil organic matter. Soil Biol. Biochem. **35**, 1683, **2003**.
26. THIRUKKUMARAN C.M., PARKINSON D. Microbial respiration, biomass, metabolic quotient and litter decomposition in a lodgepole pine forest floor amended with nitrogen and phosphorus fertilizers. Soil Biol. Biochem. **32**, 59, **2000**.
27. BLAUD A., LERCH T.Z., CHEVALLIER T., NUNAN N., CHENU C., BRAUMAN A. Dynamics of bacterial communities in relation to soil aggregate formation during the decomposition of ^{13}C labelled rice straw. Appl. Soil Ecol. **53**, 1, **2012**.
28. GALE W.J., CAMBARDELLA C.A., BAILEY T.B. Root-derived carbon and the formation and stabilization of aggregates. Soil Sci. Soc. Am. J. **64**, 201, **2000**.
29. TRIGALET S., VAN OOST K., ROISIN C., VAN WESEMAEL B. Carbon associated with clay and fine silt as an indicator for SOC decadal evolution under different residue management practices. Agr. Ecosyst. Environ. **196**, 1, **2014**.
30. GREGORICH E.G., GILLESPIE A.W., BEARE M.H., CURTIN D., SANEI H., YANNI S.F. Evaluating biodegradability of soil organic matter by its thermal stability and chemical composition. Soil biol. Biochem. **91**, 182, **2015**.
31. RABBI S.M.F., WILSON B.R., LOCKWOOD P.V., DANIEL H., YOUNG I.M. Soil organic carbon mineralization rates in aggregates under contrasting land uses. Geoderma **216**, 10, **2014**.
32. OSTRPWSKA A., POREBSKA G. Assessment of TOC-SOM and SOM-TOC conversion in forest soil. Pol. J. Environ. Stud. **21**, 1767, **2012**.