Article

Soil Aggregate-Associated Carbon Fraction Dynamics during the Process of Tea (*Camellia sinensis* L.) Planting in Southern Guangxi, China

Chao Zhang , Xinxin He, Shaoming Ye and Shengqiang Wang *

Forestry College, Guangxi University, Nanning 530004, China; czhang@gxu.edu.cn (C.Z.); xhe@gxu.edu.cn (X.H.); yeshaoming@gxu.edu.cn (S.Y.)

* Correspondence: shengqiang@gxu.edu.cn; Tel.: +86-0771-3236100

Abstract: Revealing the variation in soil aggregate-associated organic carbon (C_{org}) in tea plantations of various planting ages is crucial to shed more light on the accumulation and decomposition of soil C_{org} in the tea-planting period. This study measured the concentrations of soil C_{org} , active carbon (C_{act}), and recalcitrant carbon (C_{rec}) in different-sized aggregates obtained from tea plantations of various planting ages (8, 17, 25, and 43 years old) at the soil depths of 0–20 and 20–40 cm in southern Guangxi, China. According to the wet-sieving approach, soil aggregates were classified as macro- (>0.25 mm) and micro- (<0.25 mm) aggregates, and the former were further divided into coarse (>2 mm), medium (2–1 mm), and fine (1–0.25 mm) fractions. Based on the mean weight diameter (MWD), the stability of soil aggregates was the highest in the 17-year-old tea plantations, and it was closely related to the concentration of soil C_{act} (0–20 cm: $R^2 = 0.9744$, $p < 0.05$; 20–40 cm: $R^2 = 0.8951$, $p < 0.05$), but not C_{org} (0–20 cm: $R^2 = 0.1532$, $p > 0.05$; 20–40 cm: $R^2 = 0.4538$, $p > 0.05$), during the tea-planting process. In the 0–20 and 20–40 cm soil layers, the coarse and medium macro-aggregates had higher concentrations of C_{org} , C_{act} , and C_{rec} , regardless of the tea-planting age; meanwhile, the soil C_{act}/C_{rec} ratio, indicating the C_{org} availability, increased as aggregate size increased, implying that the soil C_{org} was younger and more labile in coarse macro-aggregates relative to finer aggregates. Moreover, the tea-planting age significantly affected the C_{org} , C_{act} , and C_{rec} reserves in both soil layers. To be specific, continuous tea planting facilitated the accumulation of soil C_{org} and C_{rec} , but their reserves' increase rates decreased over time; meanwhile, the soil C_{act} reserve increased during the early (from 8 to 17 years) tea-planting stage and later decreased. Therefore, during the middle (from 17 to 25 years) and late (from 25 to 43 years) tea-planting stages, maintaining the soil as an C_{act} pool plays a vital role in facilitating the formation and stabilization of soil aggregates in southern Guangxi, China.

Citation: Zhang, C.; He, X.; Ye, S.; Wang, S. Soil Aggregate-Associated Carbon Fraction Dynamics during the Process of Tea (*Camellia sinensis* L.) Planting in Southern Guangxi, China. *Forests* **2021**, *12*, 1530. <https://doi.org/10.3390/f12111530>

Academic Editor: Frank S. Gilliam

Received: 13 October 2021

Accepted: 4 November 2021

Published: 6 November 2021

Publisher's Note: MDPI stays neutral with regard to jurisdictional claims in published maps and institutional affiliations.

Copyright: © 2021 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (<https://creativecommons.org/licenses/by/4.0/>).

Keywords: soil aggregate; tea plantation; chronosequence; organic C

1. Introduction

Tea (*Camellia sinensis* L.) [1], which is one of the crucial industrial crops among numerous developing countries, has been extensively grown in China, India, and Sri Lanka. China is the greatest tea producer worldwide, in which the tea plantation area takes up 3.17 million hectares as of 2020 and has been continuously increasing [1]. Guangxi, located in the subtropical zone, is a region with plenty of light, heat, and water resources, and it provides all the favorable conditions for the development of tea plantations. As a result, it is one of the important tea-producing areas in China [2]. According to the statistics, over 80% of tea plantations in Guangxi are located in poverty-stricken counties; meanwhile, tea planting has become the pillar industry for most of these poverty-stricken counties to get rid of poverty [2].

Maintenance of soil organic C (C_{org}) is important for the sustainable use of soil resources in tea plantations due to the multiple effects of C_{org} on soil nutrient status and

structural stability [3]. Moreover, the global concern surrounding climate change has increased interest in promoting C sequestration in soils to mitigate the increasing CO₂ level in the atmosphere [3]. Because most forest soils have experienced some degree of C_{org} depletion, elevating the soil C_{org} pool has been suggested as an important method to sequester CO₂ from the atmosphere [4]. Soil represents a carbon sink or source, which is greatly determined by the balance between the accumulation and decomposition of soil C_{org} [4]. Soil aggregates serve as the basic soil structural units. On the one hand, aggregate composition can regulate the interactions between minerals and organic substances in numerous soil physical, chemical, and biological processes [5]. On the other hand, aggregate stability can be used to evaluate the soil structure dynamic [6]. Thus, it is essential to explore soil C_{org} change in aggregates so as to get more knowledge about the effects of soil management practices on the soil C_{org} accumulation and decomposition.

On the basis of hierarchy concept, soil aggregates are categorized either as macro- (>0.25 mm) or as micro- (<0.25 mm) aggregates [4]. During their formation processes, primary particles are merged to form micro-aggregates in the cementing action of persistent bindings (i.e., polyvalent metal cation complexes and humified organic matters) [7]; meanwhile, macro-aggregates are constituted by micro-aggregates in the aggregating action of temporary bindings (i.e., plant roots, fungal hyphae, plant- and microbial-derived polysaccharides) [8]. It should be noted that the decline of soil structure stability can lead to the disintegration of macro-aggregates and the release of micro-aggregates. Subsequently, micro-aggregates will become the building blocks for the later formation cycles of macro-aggregates [9].

Soil aggregation can provide physical protection for soil C_{org} through isolation from decomposers, thus increasing the residence time of soil C_{org} [10]. According to the residence time (RT), soil C_{org} is classified as active C (C_{act}) (RT < 2 years) and recalcitrant C (C_{rec}) (RT > 2 years) [11]. So far, the distribution of C_{org} in soil aggregates is extensively reported. As suggested in some studies, C_{org} concentration was higher in micro-aggregates compared with macro-aggregates [7,12]. However, in other studies, higher C_{org} concentration was observed in macro-aggregates instead of micro-aggregates [8,9]. In short, soil aggregate-associated C_{org} concentration and availability are dependent on the size of aggregates. However, the composition of diverse sized aggregates is controlled by the human activities, among which land-use conversion exerts a vital part in the change of soil aggregate composition [13].

Over the past century, high population pressure and severe deforestation have resulted in land degradation and reduction in soil productivity. For the sake of restoring the degenerated ecosystem, the Grain for Green Program was put into effect by the Chinese government [14]. Since the 1960s, based on the unique geographical location and the specific climate conditions, the transformation of abandoned lands into tea plantations represents an important practical step in developing sustainable agriculture in southern Guangxi [15], which not only enhances the stability of soil aggregates but also increases the reserve of soil C_{org} [16,17]. However, it remains unclear as to how different chronosequence stages of tea planting impact the dynamics of soil C_{org} and its fractions (including C_{act} and C_{rec}), in particular at the aggregate scale.

As a result, this study focused on examining the short- and long-term effects of land-use change from abandoned lands to tea plantations on (i) the composition and stability of soil aggregates, and (ii) the C_{org}, C_{act}, and C_{rec} concentrations and reserves in soil aggregates. Furthermore, the hypotheses in this study were proposed as follows: (i) soil aggregate stability would be enhanced during the tea-planting process due to the accumulation of soil C_{org} over time and (ii) the change (increase or decrease) rates of soil C_{org}, C_{act}, and C_{rec} reserves would be different among the different stages of tea planting because of the variations in litter quantity and quality during the natural aging process of tea plants.

2. Materials and Methods

2.1. Location of the Experiment

This research was performed at the Hengxian agricultural center of Guangxi University (slope gradient, 13–15°; elevation, 557–563 m) (Figure 1). This region is dominated by the subtropical monsoon climate, with an average annual precipitation and temperature of 1304 mm and 21.6 °C, respectively. The exposed soil layer is mainly generated in the Mesozoic, with the latosol agrotpe [18] and loamy clay texture. As early as the 1960s, large amounts of abandoned lands have been utilized to plant tea, because tea shows a relatively high economic value. In such areas, the “Baimao tea” (*Camellia sinensis* (L.) O. Kuntze var. Nanshanbaimao) is the main tea variety, and the planting ages are different among tea plantations. No-tillage is adopted as the cultivation practice, with a tea-planting density of about 6×10^4 plants ha^{-1} . The annual fertilizer regime since the initial land-use change was presented in our previous study [19], and there was no herbicide applied in the tea-planting period. Yellow viscous boards were covered on the tea plantations to prevent pests, because the yellow color can attract pests, which are later trapped on the boards. In September every year, each tea plant was slightly pruned and the tea residue was returned into soil.

Figure 1. Location of the experimental site. 8 year: the 8-year-old tea plantations; 17 year: the 17-year-old tea plantations; 25 year: the 25-year-old tea plantations; 43 year: the 43-year-old tea plantations.

2.2. Design of the Experiment

This research adopted the space-for-time replacement approach for exploring the impact of tea-planting age on the variations in soil aggregation as well as aggregate-associated C_{org} . In general, soil spatial variation may have potential confounding effects on the results from this study. To maximally reduce such effects, therefore, different-aged (8-, 17-, 25-, and 43-year-old) tea plantations (“Baimao tea”) were selected at alike geomorphologic units that had alike soil parent materials, slope gradient, and slope direction. Since each tea-planting age was replicated in quintuplicate, this research chose 20 tea plantations in total. The mutual distance of these tea plantations was set at 300–1500 m for avoiding pseudo-replication and minimizing spatial auto-correlation. For every tea plantation, we randomly established a plot ($S_{square} = 20 \text{ m} \times 20 \text{ m}$) at $>50 \text{ m}$ from the edge of the tea plantation.

2.3. Sampling for Litter and Soil

For every plot, 5 litter samples were collected on the soil surface from 5 randomly selected sub-plots ($S_{square} = 1 \text{ m} \times 1 \text{ m}$), and then these litter samples were integrated to a

mixed litter sample. All of the 20 mixed litter samples were dried at a temperature of 80 °C until a constant weight was obtained (Table 1). Afterwards, we measured the dry weight of these mixed litter samples as well as the concentrations of C [20] and N [21] (Table 1). To be specific, the quantity of tea litter was 821, 974, 786, and 648 g m⁻² in the 8-, 17-, 25-, and 43-year-old tea plantations, respectively. Meanwhile, the C/N ratio of tea litter was 14.23, 12.68, 17.32, and 21.37, respectively.

Table 1. Litter and soil properties under the tea plantations with different ages. Data represent the average of 5 replicates $\pm$ standard deviations. Different lowercase letters indicate significant differences ($p < 0.05$) among the different tea-planting ages.

Soil Layer (cm)	Tea-Planting Age (Year)	Soil				pH
		Bulk Density (g cm ⁻³)	Sand (2–0.02 mm) (%)	Silt (0.02–0.002 mm) (%)	Clay (<0.002 mm) (%)	
0–20	8	1.29 $\pm$ 0.03 a	30.18 $\pm$ 0.14 a	36.21 $\pm$ 0.28 a	33.61 $\pm$ 0.16 a	4.57 $\pm$ 0.03 a
	17	1.27 $\pm$ 0.04 a	31.24 $\pm$ 0.22 a	36.33 $\pm$ 0.24 a	32.43 $\pm$ 0.17 a	4.51 $\pm$ 0.02 a
	25	1.30 $\pm$ 0.03 a	30.84 $\pm$ 0.16 a	35.89 $\pm$ 0.21 a	33.27 $\pm$ 0.18 a	4.32 $\pm$ 0.01 b
	43	1.31 $\pm$ 0.02 a	29.31 $\pm$ 0.23 a	37.46 $\pm$ 0.11 a	33.23 $\pm$ 0.09 a	4.17 $\pm$ 0.02 c
20–40	8	1.34 $\pm$ 0.04 a	29.00 $\pm$ 0.32 a	38.68 $\pm$ 0.36 a	32.32 $\pm$ 0.25 a	4.62 $\pm$ 0.01 a
	17	1.36 $\pm$ 0.03 a	27.40 $\pm$ 0.48 a	39.42 $\pm$ 0.21 a	33.18 $\pm$ 0.36 a	4.58 $\pm$ 0.03 a
	25	1.41 $\pm$ 0.05 a	30.46 $\pm$ 0.57 a	38.47 $\pm$ 0.19 a	31.07 $\pm$ 0.45 a	4.45 $\pm$ 0.02 b
	43	1.37 $\pm$ 0.02 a	28.89 $\pm$ 0.32 a	38.22 $\pm$ 0.34 a	32.89 $\pm$ 0.27 a	4.26 $\pm$ 0.04 c

For every plot, 5 soil samples were collected from each layer (namely, 0–20 and 20–40 cm) in 5 sub-plots used for litter sampling, and then these soil samples in each layer were integrated to a mixed soil sample. All of the 40 mixed soil samples were carefully fragmented into the natural aggregates, and then these aggregates were filtered using the 5 mm sieve for removing soil fauna, plant roots, and stones. Afterwards, one part was adopted for the measurement of soil texture, pH, C_{org}, C_{act}, and C_{rec} concentrations (Table 1) and the other part was adopted for the separation of soil aggregates. For every plot, 5 additional soil samples were randomly collected by cutting rings ($\varnothing = 50.46$ mm, depth = 50 mm, V = 100 cm⁻³) from each layer for assessing soil bulk density (Table 1).

2.4. Separation of Soil Aggregates

According to the wet-sieving process [22], 250 g of each mixed soil sample was successively filtered with the 2-, 1-, and 0.25-mm sieves. To be specific, we immersed mixed soil samples into distilled water for 15 min, and then we shook them perpendicularly for 15 min at a 1 s⁻¹ oscillation rate and 5 cm amplitude. As a result, macro- (>0.25 mm) and micro- (<0.25 mm) aggregates were obtained, and the former included coarse (>2 mm), medium (2–1 mm), and fine (1–0.25 mm) fractions. All of the soil aggregates were dried and weighed, and then their C_{org}, C_{act}, and C_{rec} concentrations were measured.

2.5. Analyses of Soil Physic-Chemical Properties

The oven was used to dry the soil samples at a temperature of 105 °C until they reached a constant weight for ascertaining the bulk density. Prior to the test of soil texture, pH, C_{org}, C_{act}, and C_{rec} concentrations, soil samples were air-dried at indoor temperature. The hydrometer was used to measure soil texture. A glass electrode was used to measure soil pH. The acid dichromate wet oxidation approach was applied to determine C_{org} concentration [20]. The 18 N H₂SO₄ oxidation approach was applied to determine C_{act} concentration [12]. The C_{rec} concentration was calculated by subtracting C_{act} concentration from C_{org} concentration [23].

2.6. Analyses of Calculations and Statistics

Soil aggregate stability was indicated by the mean weight diameter (MWD, mm) [24]:

$$\text{MWD} = \sum_{i=1}^4 (X_i \times M_i) \quad (1)$$

where X_i denotes the i th size aggregates' average diameter (mm) and M_i denotes the i th size aggregates' proportion (% in weight).

Soil C_{org} reserve (C_{orgR} , g m^{-2}) was calculated by the following formula [25]:

$$C_{\text{orgR}} = \sum_{i=1}^4 (M_i \times C_{\text{org}i}) \times B_d \times H \times 10 \quad (2)$$

where M_i is the i th size aggregates' proportion (% in weight), $C_{\text{org}i}$ is the i th size aggregates' C_{org} concentration (g kg^{-1}), B_d is the soil bulk density (g cm^{-3}), and H is the soil depth (cm). Similarly, soil C_{act} and C_{rec} reserves were also obtained.

Pearson's correlation analysis was adopted to detect the association between the stability of soil aggregates and concentrations of C_{org} , C_{act} , and C_{rec} . One-way ANOVA was used for estimating the impact of tea-planting age on the characteristics of tea litter and bulk soil. A general linear model regarding the ANOVA formulated for the split-plot was used for estimating the impacts of aggregate size, as well as tea-planting age, on the aggregate composition and aggregate-associated C_{org} , C_{act} , and C_{rec} dynamics.

3. Results

3.1. Composition and Stability of Soil Aggregates

In the 0–20 cm soil layer, the coarse macro-aggregates were dominant in soil aggregate composition, which accounted for an average proportion of 42.70% across the four tea plantations differing in age (Table 2). In the 20–40 cm soil layer, however, the primary fractions in soil aggregates were micro-aggregates, which accounted for an average proportion of 52.79%. This showed that the coarse macro-aggregate proportion decreased while the micro-aggregate proportion increased as the soil depth increased. In both soil layers, continuous tea planting significantly influenced the proportion of different-sized aggregates, except for fine and medium macro-aggregates. The coarse macro-aggregate proportion increased significantly up to the 17th year of tea planting and later decreased significantly. Nonetheless, the micro-aggregate proportion displayed an opposite trend during the tea-planting process. In each soil layer, the highest value of soil MWD was observed in the 17-year-old tea plantations (Figure 2).

3.2. Concentrations of C_{org} and Its Fractions in Soil Aggregates

Soil aggregate-associated C_{org} , C_{act} , and C_{rec} concentrations and the $C_{\text{act}}/C_{\text{rec}}$ ratio showed an increasing trend as aggregate size increased (Figure 3). To be specific, soil C_{org} , C_{act} , and C_{rec} concentrations were significantly higher in coarse and medium macro-aggregates relative to finer aggregates. At the same time, the soil $C_{\text{act}}/C_{\text{rec}}$ ratio was the greatest in the coarse macro-aggregates, and the lowest ratio was observed in the micro-aggregates. For all tea plantations, soil aggregate-associated C_{org} , C_{act} , and C_{rec} concentrations and the $C_{\text{act}}/C_{\text{rec}}$ ratio in the 0–20 cm soil layer were higher than those in the 20–40 cm soil layer. In both soil layers, aggregate-associated C_{org} and C_{rec} concentrations elevated significantly during the tea-planting process. Differently, aggregate-associated C_{act} concentration and the $C_{\text{act}}/C_{\text{rec}}$ ratio showed an increasing trend initially and later declined over time, with the highest levels being detected in the 17-year-old tea plantations.

Table 2. Soil aggregate distribution under the tea plantations with different ages. Data represent the average of 5 replicates $\pm$ standard deviations. Different lowercase letters indicate significant differences ($p < 0.05$) among the different tea-planting ages. Different capital letters indicate significant differences ($p < 0.05$) among the different aggregate sizes. T: tea-planting age; A: aggregate size. ** and * indicate significance at $p < 0.01$ and $p < 0.05$, respectively.

Soil Layer (cm)	Tea-Planting Age (Year)	Distribution of Soil Aggregates (%)				Significance		
		>2 mm	2–1 mm	1–0.25 mm	<0.25 mm	T	A	T $\times$ A
0–20	8	42.57 $\pm$ 3.27 b	17.46 $\pm$ 1.38 a	9.56 $\pm$ 0.89 a	30.41 $\pm$ 2.88 b	**	**	*
	17	53.19 $\pm$ 4.03 a	15.91 $\pm$ 0.94 a	8.38 $\pm$ 0.93 a	22.52 $\pm$ 3.04 c			
	25	40.41 $\pm$ 2.26 b	16.94 $\pm$ 1.24 a	9.43 $\pm$ 1.02 a	33.22 $\pm$ 3.53 b			
	43	34.62 $\pm$ 2.13 c A	17.38 $\pm$ 1.16 a C	7.25 $\pm$ 0.78 a D	40.75 $\pm$ 4.27 a B			
20–40	8	27.42 $\pm$ 3.07 b	15.21 $\pm$ 3.05 a	6.65 $\pm$ 0.89 a	50.72 $\pm$ 3.68 b	**	**	*
	17	36.19 $\pm$ 2.85 a	13.17 $\pm$ 2.04 a	7.33 $\pm$ 1.02 a	43.31 $\pm$ 4.02 c			
	25	24.18 $\pm$ 2.63 bc	11.01 $\pm$ 1.17 a	5.46 $\pm$ 0.98 a	59.35 $\pm$ 3.84 a			
	43	21.36 $\pm$ 3.41 c B	12.89 $\pm$ 2.18 a C	7.98 $\pm$ 2.87 a D	57.77 $\pm$ 2.35 a A

Figure 2. Soil aggregate stability (as indicated by the MWD) under the tea plantations with different ages. Data represent average of 5 replicates and error bars represent standard deviations. Different lowercase letters (i.e., a, b, c...) indicate significant differences ($p < 0.05$) among the different tea-planting ages.

3.3. Reserves of C_{org} and Its Fractions in Soil Aggregates

In the tea-planting period, the reserves of soil C_{org} , C_{act} , and C_{rec} were the highest in coarse macro-aggregates at the depth of 0–20 cm and in micro-aggregates at the depth of 20–40 cm (Figure 4). For instance, in the 0–20 cm soil layer, the soil C_{org} reserve across the four tea-planting ages was 1.79–2.53 kg m^{−2} in the coarse macro-aggregates, contributing to 39.65–58.87% of the soil C_{org} reserve (Table 3). In contrast, in the 20–40 cm soil layer, the soil C_{org} reserve was 0.69–1.23 kg m^{−2} in the micro-aggregates, contributing to 37.11–51.01% of the soil C_{org} reserve (Table 3). In both soil layers, continuous tea planting had significant effects on the soil C_{org} , C_{act} , and C_{rec} reserves. Specifically, the soil C_{org} and C_{rec} reserves increased significantly over time; meanwhile, the soil C_{act} reserve was relatively high in the 17-year-old tea plantations compared with other ages.

Figure 3. Soil aggregate-relevant C_{org} , C_{act} , and C_{rec} concentrations under the tea plantations with different ages. Data represent average of 5 replicates and error bars represent standard deviations. Different lowercase letters (i.e., a, b, c...) indicate significant differences ($p < 0.05$) among the different tea-planting ages. Different capital letters (i.e., A, B, C...) indicate significant differences ($p < 0.05$) among the different aggregate sizes. ** and * indicate significance at $p < 0.01$ and $p < 0.05$, respectively.

Figure 4. Soil aggregate-relevant C_{org} , C_{act} , and C_{rec} reserves under the tea plantations with different ages. Data represent average of 5 replicates and error bars represent standard deviations.

Table 3. Percentage contribution of C_{org} , C_{act} , and C_{rec} reserves in aggregates to soil under the tea plantations with different ages. Data represent the average of 5 replicates $\pm$ standard deviations. Different lowercase letters indicate significant differences ($p < 0.05$) among the different tea-planting ages. Different capital letters indicate significant differences ($p < 0.05$) among the different aggregate sizes. T: tea-planting age; A: aggregate size. ** and * indicate significance at $p < 0.01$ and $p < 0.05$, respectively.

Item	Soil Layer (cm)	Tea-Planting Age (Year)	Percentage Contribution (%)				Significance		
			>2 mm	2–1 mm	1–0.25 mm	<0.25 mm	T	A	T $\times$ A
C_{org}	0–20	8	49.97 $\pm$ 2.47 b	19.55 $\pm$ 1.97 a	8.54 $\pm$ 0.47 a	21.94 $\pm$ 1.47 c	**	**	**
		17	58.87 $\pm$ 3.13 a	17.05 $\pm$ 2.01 a	7.04 $\pm$ 0.85 a	17.03 $\pm$ 2.03 d			
		25	47.11 $\pm$ 2.05 b	19.11 $\pm$ 2.34 a	8.26 $\pm$ 0.35 a	25.52 $\pm$ 1.87 b			
		43	39.65 $\pm$ 3.41 c	19.83 $\pm$ 1.03 a	7.13 $\pm$ 0.27 a	33.39 $\pm$ 1.55 a			
	20–40		A	C	D	B			
		8	32.71 $\pm$ 2.03 b	17.58 $\pm$ 1.02 a	6.81 $\pm$ 0.38 b	42.90 $\pm$ 2.67 b	*	**	*
		17	41.20 $\pm$ 3.27 a	15.13 $\pm$ 1.38 ab	6.56 $\pm$ 0.64 b	37.11 $\pm$ 3.57 c			
		25	32.80 $\pm$ 1.98 b	13.25 $\pm$ 1.47 b	5.44 $\pm$ 0.57 b	48.50 $\pm$ 3.41 a			
C_{act}	0–20	43	25.37 $\pm$ 2.67 c	15.37 $\pm$ 2.13 ab	8.25 $\pm$ 0.49 a	51.01 $\pm$ 2.97 a			
			B	C	D	A			
		8	55.11 $\pm$ 3.02 b	21.25 $\pm$ 1.35 a	8.44 $\pm$ 0.51 a	15.19 $\pm$ 2.41 b	**	**	*
		17	64.23 $\pm$ 3.74 a	18.17 $\pm$ 2.03 a	6.78 $\pm$ 0.91 bc	10.83 $\pm$ 2.36 c			
	20–40	25	53.82 $\pm$ 2.48 b	19.50 $\pm$ 2.44 a	7.55 $\pm$ 0.54 b	19.12 $\pm$ 3.06 b			
		43	45.21 $\pm$ 3.28 c	20.74 $\pm$ 1.09 a	5.66 $\pm$ 0.63 c	28.39 $\pm$ 3.36 a			
			A	B	C	B			
		8	38.93 $\pm$ 2.98 b	19.07 $\pm$ 2.58 a	6.93 $\pm$ 0.36 a	35.07 $\pm$ 1.29 c	*	**	*
C_{rec}	0–20	17	48.92 $\pm$ 2.06 a	15.59 $\pm$ 3.03 ab	6.98 $\pm$ 0.51 a	28.51 $\pm$ 3.45 d			
		25	35.91 $\pm$ 3.84 b	13.66 $\pm$ 1.02 b	5.83 $\pm$ 0.84 a	44.60 $\pm$ 2.48 b			
		43	28.03 $\pm$ 2.51 c	15.75 $\pm$ 1.48 ab	7.03 $\pm$ 0.69 a	49.19 $\pm$ 1.03 a			
			A	B	C	A			
	20–40	8	48.65 $\pm$ 2.03 b	19.12 $\pm$ 1.05 a	8.57 $\pm$ 1.03 a	23.67 $\pm$ 2.34 b	**	**	**
		17	57.39 $\pm$ 3.05 a	16.75 $\pm$ 1.47 b	7.12 $\pm$ 0.89 a	18.74 $\pm$ 3.21 c			
		25	45.81 $\pm$ 1.47 b	19.03 $\pm$ 2.31 a	8.40 $\pm$ 1.21 a	26.77 $\pm$ 0.89 b			
		43	38.86 $\pm$ 2.36 c	19.69 $\pm$ 1.98 a	7.34 $\pm$ 0.58 a	34.11 $\pm$ 2.14 a			
C_{rec}	0–20		A	C	D	B			
		8	31.33 $\pm$ 2.47 b	17.26 $\pm$ 2.03 a	6.79 $\pm$ 0.47 b	44.63 $\pm$ 3.48 b	*	**	*
	20–40	17	39.49 $\pm$ 2.03 a	15.03 $\pm$ 1.35 ab	6.47 $\pm$ 0.68 b	39.02 $\pm$ 1.02 c			
		25	32.30 $\pm$ 1.89 b	13.19 $\pm$ 2.36 b	5.38 $\pm$ 0.39 b	49.14 $\pm$ 3.21 b			
		43	25.03 $\pm$ 3.47 c	15.32 $\pm$ 2.49 ab	8.40 $\pm$ 0.88 a	51.24 $\pm$ 2.19 a			
			B	C	D	A			

4. Discussion

4.1. Composition and Stability of Soil Aggregates

Regardless of the tea-planting age, the coarse macro-aggregates and micro-aggregates played predominant roles in the 0–20 and 20–40 cm soil layers, respectively (Table 2), indicating a transformation of soil aggregate composition from coarse macro-aggregate-dominant to micro-aggregate-dominant as the soil depth increased. Similarly, coarse macro-aggregates were dominant in the 0–20 cm soil layer as corroborated by Li et al. [16] from a study on tea plantations in southwestern China. In addition, it was also reported that coarse macro-aggregates were dominant in the surface soil (0–20 cm) of dry tropical forests in India [26]. In this study, coarse macro-aggregates were the dominant fractions in the 0–20 cm soil layer, which might be because of surface accumulation of soil C_{org} (especially C_{act}) (Figure 3) that provided cementing agents to facilitate coarse macro-aggregate formation [27,28]. Moreover, the data reported here—that the proportion of

macro-aggregates decreased as the soil depth increased (Table 2)—conformed to the results obtained from Modak et al. [12]. The decreased macro-aggregate proportion was possibly ascribed to the elevated soil compactness (based on the bulk density) as the soil depth increased (Table 1). Soil densification could prevent plant root growth, thus leading to reduced activity of microbes, like fungi [23]. Reduced activity of fungi could lower polysaccharides and glomalin-related soil protein (GRSP) production from the fungal hyphae, thus resulting in a reduced macro-aggregate proportion [29]. Likewise, according to our prior studies [30,31], soil microbial activity and GRSP concentration were higher at the 0–20 cm depth compared with the 20–40 cm depth, and they played important roles during the formation and stabilization of soil macro-aggregates in the ecosystem of tea plantations.

At the early (from 8 to 17 years) stage, tea planting facilitated coarse macro-aggregate formation in the 0–20 and 20–40 cm soil layers (Table 2). By contrast, at the middle (from 17 to 25 years) and late (from 25 to 43 years) stages, tea planting caused coarse macro-aggregate destruction and micro-aggregate release (Table 2). These results confirm the previous findings that the age of tea planting significantly affects the aggregate composition in soil [32,33]. During the tea-planting process, the changed soil aggregate stability is evidenced by the changed soil MWD [34]. Regardless of the soil layer, the decline in MWD after 17 years of tea planting might be associated with the variation in soil aggregate composition, particularly for the decomposition of coarse macro-aggregates to micro-aggregates. These results indicated that 17-year-old tea plantations had higher soil aggregate stability than the rest of the plantations (Figure 2). According to the soil aggregates' hierarchy concept [4], the quality of plant litter that returns into the soil decides the distribution of litter in various sized aggregates, which eventually affects the composition of soil aggregates. In the early period of tea planting, tea litter showed higher availability (based on the smaller litter C/N ratio, Table 1), implying that the litter was easily combined into the coarse macro-aggregates during its decomposition process at this stage, thus facilitating coarse macro-aggregate formation [10]. On the contrary, in the middle and late periods of tea planting, tea plants experienced a natural aging process and litter was gradually humified, which caused the decomposition of coarse macro-aggregates to micro-aggregates [35].

Soil C_{org} has been extensively recognized as a vital binding agent of soil aggregation [4]. Most reports have revealed the high association between aggregate stability and C_{org} concentration in soil [36–38]. Nonetheless, findings from this study showed no significant association ($p > 0.05$) between MWD and C_{org} concentration during the tea-planting process, irrespective of the soil layer (Figure 5). Besides, our first hypothesis was not sufficiently supported by the findings that soil aggregate stability declined following 17 years of tea planting, but soil C_{org} concentration increased continuously over time. In the period of tea planting, the 17-year-old tea plantations had the highest level of soil aggregate stability (Figure 2), possibly due to the soil C_{act} accumulation in these plantations (Figure 3). Soil C_{act} is important for soil aggregation, and it combines micro-aggregates to form macro-aggregates, thereby promoting macro-aggregate formation and enhancing aggregate stability [4]. Therefore, the correlation between MWD and C_{act} concentration in soil across the four different tea-planting ages was analyzed in this study, which indicated the positive association ($p < 0.05$) between them (Figure 5), further implying that soil aggregate stability was highly associated with the C_{act} concentration rather than C_{org} during the tea-planting process.

Figure 5. Relationships of soil aggregate stability (as indicated by the MWD) with C_{org}, C_{act}, and C_{rec} concentrations across the 4 tea-planting ages. Data represent average of 5 replicates and error bars represent standard deviations.

4.2. Concentrations of C_{org} and Its Fractions in Soil Aggregates

In this study, soil coarse and medium macro-aggregates contained higher C_{org} concentrations in all tea plantations (Figure 3), consistent with the research by Six et al. [4] suggesting that primary particles combine with recalcitrant bindings for micro-aggregate formation; later, micro-aggregates combine with active bindings to form macro-aggregates. In addition, macro-aggregates can physically protect C_{org} , thereby resulting in the higher C_{org} concentration in the macro-aggregates [22]. Similarly, O'Brien and Jastrow [39] as well as Gentile et al. [40] also confirmed that soil aggregates' hierarchy concept resulted in an increase in C_{org} concentration with the increase in aggregate size and soil macro-aggregate stability was vital for soil C_{org} sequestration. Likewise, the distribution patterns of C_{act} and C_{rec} concentrations were similar to those of C_{org} in soil aggregates (Figure 3). Soil C_{act}/C_{rec} ratio, also called the "oxidation stability coefficient", is an important indicator for indicating soil C_{org} availability [12]. As revealed by Blair et al. [41], the reduced soil C_{act}/C_{rec} ratio stood for the decreased soil C_{org} availability within the agroecosystems. In this study, soil C_{act}/C_{rec} ratio showed an increasing trend with the increase in aggregate size, irrespective of the tea-planting age (Figure 3), which implied that soil C_{org} in coarse macro-aggregates was more labile and younger than that in finer aggregates; meanwhile, micro-aggregate-associated C_{org} experienced greater degradation and became more recalcitrant. In short, the present study found that a higher concentration of C_{org} was observed in the macro-aggregates, and the C_{org} in these aggregates was younger with a higher availability.

Continuous tea planting was beneficial for the accumulation of soil C_{org} (Figure 3), which demonstrated the results obtained by articles stating that afforestation was an important way for increasing the concentration of soil C_{org} [13]. In this study, the increase in soil C_{org} concentration over time is associated with the following mechanisms. First, the application of manure is demonstrated to have positive impact on the soil C_{org} accumulation [34]. Likewise, an increase in the concentration of soil C_{org} during the process of tea planting may be because large amount of swine manure is applied every year ($12 \text{ Mg ha}^{-1} \text{ year}^{-1}$) [19]. Second, plant litter is the main C_{org} source in soil [42]. In the period of tea planting, the accumulation of soil C_{org} could occur because the quantity of above-ground tea litter increases through routine trimming of tea leaf and branch (Table 1). Third, no-tillage contributes to protecting the integration of soil aggregates with C_{org} , thus enhancing soil C_{org} sequestration [43].

The stand age-associated trend of soil C_{rec} concentration in tea plantations was consistent with that of soil C_{org} concentration (Figure 3). The recalcitrant organic compounds remain during the decomposition and transformation of plant litter [23] and then accumulate in soil due to the selective preservation [44]. Additionally, there are lignin and tannin within tea litter, which can increase the concentration of soil C_{rec} [45]. Notably, contrary to soil C_{org} and C_{rec} , the C_{act} concentration in soil was the highest in the 17-year-old tea plantations (Figure 3), which was mainly related to the characteristics of tea litter [46]. According to our results, the 17-year-old tea plantations showed the highest levels of litter quantity and quality (based on the smallest C/N ratio of litter), but later the litter quantity and quality declined significantly over time (Table 1) due to the natural aging process of tea plants, which could explain why soil C_{act} concentration was consistent with the litter dynamic in the tea-planting period [47]. Litter decomposed in soil could strongly affect soil C_{org} fractions by changing the contacts between litter decomposition products and soil solid phases, thereby affecting soil C_{org} availability [47]. In the early period of tea planting, the growing rate of C_{act} concentration (0–20 cm layer: 27.60%; 20–40 cm layer: 30.58%) was greater than that of C_{rec} concentration (0–20 cm layer: 15.41%; 20–40 cm layer: 11.92%) in both soil layers (Figure 3); meanwhile, a significant decrease in soil C_{act} concentration in the middle and late periods was accompanied by a significant increase in soil C_{rec} concentration (Figure 3), which led to the highest soil C_{org} availability (based on the highest C_{act}/C_{rec} ratio in soil) in the 17-year-old tea plantations (Figure 3).

The higher aggregate-associated C_{org} concentration in the 0–20 cm soil layer (Figure 3) conformed to findings by Kurmi et al. [23] stating that the surface accumulation of soil C_{org} was associated with increased input of organic matters in the 0–20 cm soil layer. For all tea plantations, the application of swine manure, as well as the input of tea litter and roots, enriched the aggregate-associated C_{org} concentration in the 0–20 cm soil layer [16]. Besides, in the 20–40 cm soil layer, aggregate-associated C_{org} concentration depended mainly on the input of root exudation and debris, as well as on the downward transfer of organic matters by rainfall water and soil fauna [48]. Similarly, soil aggregate-associated C_{act} and C_{rec} concentrations were relatively high in the 0–20 cm soil layer compared with the 20–40 cm (Figure 3). Soil C_{act}/C_{rec} ratio at the 0–20 cm depth was greater than that at the 20–40 cm depth in the period of tea planting (Figure 3), implying that the soil layer of 20–40 cm had a more stable soil C_{org} pool in tea plantations. Moreover, the impact of tea-planting age on the concentration of aggregate-associated C_{org} was more significant at the soil depth of 0–20 cm than at 20–40 cm (Figure 3), because aggregate-associated C_{org} at the soil depth of 0–20 cm was more easily influenced by the tea litter returned to the soil, as well as the fertilizer application.

4.3. Reserves of C_{org} and Its Fractions in Soil Aggregates

In the tea-planting period, the reserve of C_{org} in coarse macro-aggregates made a greater contribution to that of soil in the surface layer (0–20 cm), implying that coarse macro-aggregates were the dominant carriers of C_{org} in the surface soil. Differently, in the deeper soil layer (20–40 cm), the dominant carriers of C_{org} were micro-aggregates (Table 3). Although micro-aggregates had the lowest concentrations of C_{org} in the deeper soil layer (Figure 3), the contribution of C_{org} reserve in micro-aggregates to soil was greater than that of the other aggregates (Table 3), because micro-aggregates occupied the highest proportion in soil of the deeper layer (Table 2). According to the above findings, the C_{org} reserve in soil aggregates was mainly dependent on the proportion of different-sized aggregates in tea plantations.

The variation in the soil C_{org} reserve in a period of tea planting divided by the length of this period is utilized as an index for estimating the increase or decrease rate of the soil C_{org} reserve [49]. Most of the reported soil C_{org} sequestration rates have been within an order of magnitude, with a mean of $33 \text{ g } C_{org} \text{ m}^{-2} \text{ year}^{-1}$ for forestry and agricultural chronosequences [50]. In this study, as the tea-planting age increased from 8 to 43 years (length, 35 years), the increase rate of the soil C_{org} reserve was 40.83 and $23.17 \text{ g } C_{org} \text{ m}^{-2} \text{ year}^{-1}$ in the 0–20 and 20–40 cm layers, respectively (Figure 4), implying that soil C_{org} accumulation was faster in the surface layer than in the deeper layer during the tea-planting process.

Notably, the increase rate of the soil C_{org} reserve was different at various stages of tea planting (Figure 6), which was consistent with our second hypothesis. To be specific, in the early, middle, and late periods of tea planting, the soil C_{org} reserve increased by 78.49, 39.85, and $22.44 \text{ g } C_{org} \text{ m}^{-2} \text{ year}^{-1}$ in the 0–20 cm layer, and increased by 58.54, 12.40, and $10.27 \text{ g } C_{org} \text{ m}^{-2} \text{ year}^{-1}$ in the 20–40 cm layer, respectively, with the soil C_{org} reserve of the 8-year-old tea plantations as a reference point. In addition, the soil C_{rec} reserve showed a similar trend over time in both layers (Figures 4 and 6), indicating that although continuous tea planting facilitated the accumulation of soil C_{org} and C_{rec} , the increase rates of their reserves showed a decreasing trend with the increase in tea-planting age. Different from the soil C_{org} and C_{rec} reserves, the highest level of soil C_{act} reserve was observed in the 17-year-old tea plantations (Figure 4). In the surface layer, the soil C_{act} reserve increased by $21.82 \text{ g } C_{act} \text{ m}^{-2} \text{ year}^{-1}$ in the early period of tea planting, but it decreased by 22.51 and $6.76 \text{ g } C_{act} \text{ m}^{-2} \text{ year}^{-1}$, respectively, in the middle and late periods (Figure 6). A similar trend was also observed in the deeper soil layer (Figure 6). Such findings indicate that soil C_{act} had a risk of reduction in the middle and late periods of tea planting.

Figure 6. Change rates of soil C_{org}, C_{act}, and C_{rec} reserves in the different tea-planting stages. Data represent average of 5 replicates and error bars represent standard deviations. Different lowercase letters (i.e., a, b, c...) indicate significant differences ($p < 0.05$) among the different tea-planting stages. Early stage: from 8 years to 17 years; middle stage: from 17 years to 25 years; later stage: from 25 years to 43 years.

According to the above discussion, measuring the diverse active fractions of soil aggregate-associated C_{org} is effective to characterize the changes of soil C_{org} quantity and quality during the tea-planting process. Nonetheless, for achieving sustainable utilization of soil resources in tea plantations, more efforts should be made to understand the decomposition potential of soil aggregate-associated C_{org} at diverse stages of tea planting. Based on our results, soil C_{org} concentration and availability in aggregates were mainly dependent on the aggregate size. Meanwhile, tea-planting age played an important role in changing the composition of different-sized aggregates in soil, which implied that tea-planting age did not affect soil C_{org} accumulation and decomposition individually; by contrast, the variation in soil aggregate composition was of great importance in the tea-planting period. Therefore, based on the connection (namely, “treatment-aggregate-carbon”), investigation of the soil C_{org} fraction distribution in different-sized aggregates is helpful in acquiring a better understanding of the soil carbon cycle for other tea-planting regions with different soil types (especially for the latosol) in the subtropics.

5. Conclusions

During the process of tea planting, soil aggregate stability was the highest in the 17-year-old tea plantations, and it was closely related to the concentration of soil C_{act} , but not C_{org} . Continuous tea planting facilitated the accumulation of soil C_{org} and C_{rec} , but their reserves' increase rates decreased as the tea-planting age increased. Additionally, the soil C_{act} reserve increased in the early (from 8 to 17 years) period of tea planting and then decreased over time. Therefore, in the middle (from 17 to 25 years) and late (from 25 to 43 years) periods of tea planting, maintaining soil as an C_{act} pool plays a vital role in facilitating the formation and stabilization of soil aggregates in southern Guangxi, China. This study sheds more lights on how tea-planting age affects the dynamic of soil C_{org} . For better understanding soil carbon sink or source effect in the tea plantation ecosystem, further investigations of soil carbon-relevant enzyme activities at the aggregate scale are necessary, because determining the activities of soil carbon-relevant enzymes is important for assessing the potential of soil C_{org} decomposition.

Author Contributions: S.W. and S.Y. designed the experiments; C.Z. and X.H. carried out the experiments; C.Z. and X.H. analyzed the experimental results; C.Z., X.H., S.W. and S.Y. wrote and edited the manuscript. All authors have read and agreed to the published version of the manuscript.

Funding: The National College Students' Innovation and Entrepreneurship Training Program of Guangxi University (202110593049) and the Natural Science Foundation of Guangxi (2019GXNSFBA185012) financed this study.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: The data supporting the discovered information here can be presented by the relevant author based on reasonable requests.

Acknowledgments: The authors would like to express their gratitude to the editors and innominate reviewers for giving conducive advises and optimizing this article.

Conflicts of Interest: The authors declare no conflict of interest.

References

1. Chinese Tea Committee. An area planted with tea in various provinces at the end of each year. *Ann. Bull. Stat.* **2020**, *47*, 20.
2. Jiang, J.; Jiang, W.; Jiang, W. The present situation and countermeasures of the development of tea industry in Guangxi. *Popular Sci. Technol.* **2018**, *20*, 72–73. [[CrossRef](#)]
3. Bleuler, M.; Farina, R.; Francaviglia, R.; Di Bene, C.; Napoli, R.; Marchetti, A. Modelling the impacts of different carbon sources on the soil organic carbon stock and CO₂ emissions in the Foggia province (Southern Italy). *Agric. Syst.* **2017**, *157*, 258–268. [[CrossRef](#)]
4. Six, J.; Bossuyt, H.; Degryze, S.; Denef, K. A history of research on the link between (micro)aggregates, soil biota, and soil organic matter dynamics. *Soil Tillage Res.* **2004**, *79*, 7–31. [[CrossRef](#)]

5. Liu, Y.; Wang, C.; Chen, M. Carbon flow analysis of China's agro-ecosystem from 1980 to 2013: A perspective from substance flow analysis. *J. Environ. Sci.* **2017**, *55*, 20–32. [[CrossRef](#)] [[PubMed](#)]
6. Huang, R.; Tian, D.; Liu, J.; Lv, S.; He, X.; Gao, M. Responses of soil carbon pool and soil aggregates associated organic carbon to straw and straw-derived biochar addition in a dryland cropping mesocosm system. *Agric. Ecosyst. Environ.* **2018**, *265*, 576–586. [[CrossRef](#)]
7. Mizuta, K.; Taguchi, S.; Sato, S. Soil aggregate formation and stability induced by starch and cellulose. *Soil Biol. Biochem.* **2015**, *87*, 90–96. [[CrossRef](#)]
8. Li, C.; Li, Y.; Xie, J.; Wang, Y.; Liu, X. Accumulation of organic carbon and its association with macro-aggregates during 100 years of oasis formation. *CATENA* **2018**, *172*, 770–780. [[CrossRef](#)]
9. Voltolini, M.; Taş, N.; Wang, S.; Brodie, E.; Ajo-Franklin, J.B. Quantitative characterization of soil micro-aggregates: New opportunities from sub-micron resolution synchrotron X-ray microtomography. *Geoderma* **2017**, *305*, 382–393. [[CrossRef](#)]
10. Tisdall, J.M.; Oades, J.M. Organic matter and water-stable aggregates in soils. *Eur. J. Soil Sci.* **1982**, *33*, 141–163. [[CrossRef](#)]
11. Medinski, T.V.; Freese, D.; Böhm, C.; Slazak, A. Soil carbon fractions in short rotation poplar and black locust coppices, Germany. *Agrofor. Syst.* **2014**, *88*, 505–515. [[CrossRef](#)]
12. Modak, K.; Ghosh, A.; Bhattacharyya, R.; Biswas, D.R.; Das, T.K.; Das, S.; Singh, G. Response of oxidative stability of aggregate-associated soil organic carbon and deep soil carbon sequestration to zero-tillage in subtropical India. *Soil Tillage Res.* **2019**, *195*, 104370. [[CrossRef](#)]
13. Li, G.-L.; Pang, X.-M. Effect of land-use conversion on C and N distribution in aggregate fractions of soils in the southern Loess Plateau, China. *Land Use Policy* **2010**, *27*, 706–712. [[CrossRef](#)]
14. Zeng, L.; Li, J.; Zhou, Z.; Yu, Y. Optimizing land use patterns for the grain for Green Project based on the efficiency of ecosystem services under different objectives. *Ecol. Indic.* **2020**, *114*, 106347. [[CrossRef](#)]
15. Zhang, Q.; Gao, W.; Su, S.; Weng, M.; Cai, Z. Biophysical and socioeconomic determinants of tea expansion: Apportioning their relative importance for sustainable land use policy. *Land Use Policy* **2017**, *68*, 438–447. [[CrossRef](#)]
16. Li, W.; Zheng, Z.; Li, T.; Zhang, X.; Wang, Y.; Yu, H.; He, S.; Liu, T. Effect of tea plantation age on the distribution of soil organic carbon fractions within water-stable aggregates in the hilly region of Western Sichuan, China. *CATENA* **2015**, *133*, 198–205. [[CrossRef](#)]
17. Zhu, R.; Zheng, Z.; Li, T.; Zhang, X.; He, S.; Wang, Y.; Liu, T.; Li, W. Dynamics of soil organic carbon mineralization in tea plantations converted from farmland at Western Sichuan, China. *PLoS ONE* **2017**, *12*, e0185271. [[CrossRef](#)]
18. Wang, S.; Tang, L.; Ye, S. Dynamics of soil bacterial community diversity and composition at aggregate scales in a chronosequence of tea gardens. *CATENA* **2021**, *206*, 105486. [[CrossRef](#)]
19. Wang, S.; Ye, S. Dynamics of bacterial function characteristics in soil aggregates during the tea-planting process. *Geoderma* **2020**, *377*, 114609. [[CrossRef](#)]
20. Nelson, D.W.; Sommers, L.E. Total carbon, organic carbon and organic matter. In *Chemical Methods*; Sparks, D.L., Ed.; ASA and SSSA: Madison, WI, USA, 1996; Part 3, No. 5; pp. 961–1010.
21. Bremner, J.M. Nitrogen-total. In *Chemical Methods*; Sparks, D.L., Ed.; ASA and SSSA: Madison, WI, USA, 1996; Part 3, No. 5; pp. 1085–1121.
22. Six, J.; Conant, R.; Paul, E.A.; Paustian, K. Stabilization mechanisms of soil organic matter: Implications for C-saturation of soils. *Plant Soil* **2002**, *241*, 155–176. [[CrossRef](#)]
23. Kurmi, B.; Nath, A.J.; Lal, R.; Das, A.K. Water stable aggregates and the associated active and recalcitrant carbon in soil under rubber plantation. *Sci. Total. Environ.* **2020**, *703*, 135498. [[CrossRef](#)]
24. Nimmo, J.R.; Perkins, K.S. Aggregate stability and size distribution methods of soil analysis. In *Physical Methods*; Dane, J.H., Ed.; ASA and SSSA: Madison, WI, USA, 1996; Part 4, No. 5; pp. 317–328.
25. Eynard, A.; Schumacher, T.; Lindström, M.; Malo, D. Effects of agricultural management systems on soil organic carbon in aggregates of Ustolls and Usterts. *Soil Tillage Res.* **2005**, *81*, 253–263. [[CrossRef](#)]
26. Singh, M.K.; Singh, S.; Ghoshal, N. Impact of land use change on soil aggregate dynamics in the dry tropics. *Restor. Ecol.* **2017**, *25*, 962–971. [[CrossRef](#)]
27. Al-Kaisi, M.M.; Douelle, A.; Kwaw-Mensah, D. Soil microaggregate and macroaggregate decay over time and soil carbon change as influenced by different tillage systems. *J. Soil Water Conserv.* **2014**, *69*, 574–580. [[CrossRef](#)]
28. Ge, Z.; Fang, S.; Chen, H.Y.; Zhu, R.; Peng, S.; Ruan, H. Soil Aggregation and Organic Carbon Dynamics in Poplar Plantations. *Forests* **2018**, *9*, 508. [[CrossRef](#)]
29. Ji, L.; Tan, W.; Chen, X. Arbuscular mycorrhizal mycelial networks and glomalin-related soil protein increase soil aggregation in Calcaric Regosol under well-watered and drought stress conditions. *Soil Tillage Res.* **2018**, *185*, 10. [[CrossRef](#)]
30. Wang, S.; Li, T.; Zheng, Z. Distribution of microbial biomass and activity within soil aggregates as affected by tea plantation age. *CATENA* **2017**, *153*, 29. [[CrossRef](#)]
31. Zhu, R.; Zheng, Z.; Li, T.; He, S.; Zhang, X.; Wang, Y.; Liu, T. Effect of tea plantation age on the distribution of glomalin-related soil protein in soil water-stable aggregates in southwestern China. *Environ. Sci. Pollut. Res.* **2018**, *26*, 1973–1982. [[CrossRef](#)] [[PubMed](#)]
32. He, Y.; Xu, C.; Gu, F.; Wang, Y.; Chen, J. Soil aggregate stability improves greatly in response to soil water dynamics under natural rains in long-term organic fertilization. *Soil Tillage Res.* **2018**, *184*, 281–290. [[CrossRef](#)]

33. Wu, W.; Zheng, Z.; Li, T.; He, S.; Zhang, X.; Wang, Y.; Liu, T. Distribution of inorganic phosphorus fractions in water-stable aggregates of soil from tea plantations converted from farmland in the hilly region of western Sichuan, China. *J. Soils Sediments* **2017**, *18*, 906–916. [[CrossRef](#)]
34. Yin, J.; Zheng, Z.; Li, T.; Zhang, X.; He, S.; Wang, Y.; Yu, H.; Liu, T. Effect of tea plantation age on the distribution of fluoride and its fractions within soil aggregates in the hilly region of Western Sichuan, China. *J. Soils Sediments* **2016**, *16*, 2128–2137. [[CrossRef](#)]
35. Six, J.; Paustian, K. Aggregate-associated soil organic matter as an ecosystem property and a measurement tool. *Soil Biol. Biochem.* **2014**, *68*, A4–A9. [[CrossRef](#)]
36. Luna, L.; Miralles, I.; Andrenelli, M.C.; Gispert, M.; Pellegrini, S.; Vignozzi, N.; Solé-Benet, A. Restoration techniques affect soil organic carbon, glomalin and aggregate stability in degraded soils of a semiarid Mediterranean region. *CATENA* **2016**, *143*, 256–264. [[CrossRef](#)]
37. Li, C.; Cao, Z.; Chang, J.; Zhang, Y.; Zhu, G.; Zong, N.; He, Y.; Zhang, J.; He, N. Elevational gradient affect functional fractions of soil organic carbon and aggregates stability in a Tibetan alpine meadow. *CATENA* **2017**, *156*, 139–148. [[CrossRef](#)]
38. Xue, B.; Huang, L.; Huang, Y.; Zhou, F.; Li, F.; Kubar, K.A.; Li, X.; Lu, J.; Zhu, J. Roles of soil organic carbon and iron oxides on aggregate formation and stability in two paddy soils. *Soil Tillage Res.* **2019**, *187*, 161–171. [[CrossRef](#)]
39. O'Brien, S.L.; Jastrow, J. Physical and chemical protection in hierarchical soil aggregates regulates soil carbon and nitrogen recovery in restored perennial grasslands. *Soil Biol. Biochem.* **2013**, *61*, 31. [[CrossRef](#)]
40. Gentile, R.; Vanlauwe, B.; Six, J. Integrated Soil Fertility Management: Aggregate carbon and nitrogen stabilization in differently textured tropical soils. *Soil Biol. Biochem.* **2013**, *67*, 124–132. [[CrossRef](#)]
41. Blair, G.; Lefroy, R.; Lisle, L. Soil carbon fractions based on their degree of oxidation, and the development of a carbon management index for agricultural systems. *Aust. J. Agric. Res.* **1995**, *46*, 1459–1466. [[CrossRef](#)]
42. Loss, A.; Lourenzi, C.R.; dos Santos, E.; Mergen, C.A.; Benedet, L.; Pereira, M.; Piccolo, M.D.C.; Brunetto, G.; Lovato, P.E.; Comin, J.J. Carbon, nitrogen and natural abundance of ^{13}C and ^{15}N in biogenic and physicogenic aggregates in a soil with 10 years of pig manure application. *Soil Tillage Res.* **2017**, *166*, 52–58. [[CrossRef](#)]
43. Varvel, G.; Wilhelm, W. No-tillage increases soil profile carbon and nitrogen under long-term rainfed cropping systems. *Soil Tillage Res.* **2011**, *114*, 28–36. [[CrossRef](#)]
44. Ostertag, R.; Marin-Spiotta, E.; Silver, W.L.; Schulten, J. Litterfall and Decomposition in Relation to Soil Carbon Pools Along a Secondary Forest Chronosequence in Puerto Rico. *Ecosystems* **2008**, *11*, 701–714. [[CrossRef](#)]
45. Kraus, T.E.C.; Dahlgren, R.; Zasoski, R.J. Tannins in nutrient dynamics of forest ecosystems—A review. *Plant Soil* **2003**, *256*, 41–66. [[CrossRef](#)]
46. Castellano, M.J.; Mueller, K.; Olk, D.C.; Sawyer, J.E.; Six, J. Integrating plant litter quality, soil organic matter stabilization, and the carbon saturation concept. *Glob. Chang. Biol.* **2015**, *21*, 3200–3209. [[CrossRef](#)]
47. Cotrufo, M.F.; Wallenstein, M.D.; Boot, C.M.; Deneff, K.; Paul, E. The Microbial Efficiency-Matrix Stabilization (MEMS) framework integrates plant litter decomposition with soil organic matter stabilization: Do labile plant inputs form stable soil organic matter? *Glob. Chang. Biol.* **2012**, *19*, 988–995. [[CrossRef](#)] [[PubMed](#)]
48. Fierer, N.; Schimel, J.P.; A Holden, P. Variations in microbial community composition through two soil depth profiles. *Soil Biol. Biochem.* **2003**, *35*, 167–176. [[CrossRef](#)]
49. Su, Y.Z.; Liu, W.J.; Yang, R.; Chang, X.X. Changes in Soil Aggregate, Carbon, and Nitrogen Storages Following the Conversion of Cropland to Alfalfa Forage Land in the Marginal Oasis of Northwest China. *Environ. Manag.* **2009**, *43*, 1061–1070. [[CrossRef](#)] [[PubMed](#)]
50. Post, W.M.; Kwon, K.C. Soil carbon sequestration and land-use change: Processes and potential. *Glob. Chang. Biol.* **2000**, *6*, 317–327. [[CrossRef](#)]