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ORIGINAL PAPER

Dynamic of Organic Matter, Nutrient Cycling, and PH in Soil Aggregate Particle Sizes Under Long-Term Cultivation of *Camellia Oleifera*

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Abstract

Camellia oleifera is intensively cultivated in subtropical areas of China, exposing soils to various threats. However, the effect of long-term cultivation of *Camellia oleifera* on soil properties remains unclear and needs to be elucidated to improve soil and *Camelia oleifera* sustainable management. This study collected soil samples from different *Camellia oleifera* planting ages (2, 10, and 40 years) and performed aggregate fractionation and various analyses including physico-chemical, soil organic matter chemical composition analyses and a semi-quantitative analysis of organic carbon functional groups to investigate the effect of long-term cultivation of *Camellia oleifera* on soil macroaggregates (<2 mm), mesoaggregates (2-0.25 mm) and microaggregates (<0.25 mm) formation, soil organic matter (SOM), available nitrogen (AN), available phosphorus (AP), and available potassium (AK). The aggregate particles increased with increasing *Camellia oleifera* planting ages. *Camellia oleifera* long-term cultivation did not affected the soil pH in the aggregates, but significantly ($P<0.05$) increased SOM concentration with the decrease in soil aggregate size. Similarly, Phenolics-C, ketones-C, lignins-C, and alkenes-C increased in soil aggregates with increasing cultivation time and are mainly distributed in <0.25 mm fraction. The aromatics-C, carboxylic-C, aliphatic-C, and polysaccharides-C declined with increasing cultivation time and were mainly distributed in 2-0.25 mm and <2 mm fraction. AN is abundantly distributed in 2-0.25 mm and its abundance increases with *Camellia oleifera* long-term cultivation. All soils were poor in AP and AK. Our study indicated that long-term cultivation of *Camellia oleifera* promoted soil aggregate formation, increased available nitrogen (AN), soil organic matter (SOM) and controlled the change of SOM chemical composition. However, our study recommended providing available phosphorus (AP) and available potassium (AK) in soil with *Camellia oleifera* cultivation for sustainable management.

Keywords Aggregates · *Camellia oleifera* · Long-term cultivation · Soil organic matter · Available nutrients

1 Introduction

Long-term tree crops cultivation can have significant effects on soil properties, contributing to changes in soil composition, structure, nutrient cycling, and overall soil health. Several studies reported some key positive effects of long-term tree crops cultivation on soil properties mainly soil aggregates. For instance long-term cultivation of *Robinia pseudoacacia* on abandoned farmlands on the Loess Plateau in China accelerate organic carbon accumulation in macro aggregates (Zhang et al. 2021). Similarly, cultivation of *Pinus tabulaeformis Carr*. and *Populus alba L*. have induced accumulation of soil organic carbon mainly in the macroaggregates of the upper 10 cm (Jiang et al. 2019). This accumulation of organic carbon in macro aggregates after tree crops cultivation improves the accumulation of organic carbon in soils (Qiu et al. 2015). and can be explained by

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a complex process involving plant inputs, microbial activity, and the physical properties of the soil (Bai et al. 2020; Papadopoulos 2011). Additionally, the long-term tree crops cultivation accelerates aggregate formation in soils. Recent study revealed that in the topsoil of degraded karst area, Chinese pine cultivation significantly increased the proportion of large macroaggregate and decreased the quantities of small macroaggregate, microaggregate, and silt + clay (Lan et al. 2022). trees cultivation increased macroaggregate amount, mean weight diameter, and mean geometric diameter but decreased the amount of microaggregate and silt + clay-sized fractions (Wei et al. 2013). Long-term trees cultivation affects not only aggregate formation and stability but also the aggregate-associated carbon (Bai et al. 2020; Wei et al. 2013). The mechanisms controlling the effect of trees cultivation in soils were reported. For instance a study revealed that the mechanism that drives OC sequestration with trees plantation age could be linked to the temporal dynamics of the input and decomposition of litter (Clark et al. 2012). Indeed, SOC accumulation was related to the rapid increase in new OC and the slow loss of old OC in bulk soil and aggregates (Lan et al. 2022). However, few studies have reported the effect of long-term trees cultivation in the nutrients cycling in soil aggregates while soil nutrients cycling in the aggregate can improve soil fertility management.

Camellia oleifera is a plant species belonging to the tea family and native to China, where it is mainly cultivated for its edible oil, extracted from its seeds (Wang et al. 2021). The oils from *Camellia oleifera* trees have an important nutritive properties (high-oleic, medium-linoleic, and low-linolenic acid contents), hold near to 90% of unsaturated fatty acids, have a great number of vitamins E, and some other substances (squalene, and flavonoid) which can not only improve the immunity system but also prevent diseases such as cardiovascular and hypertension diseases (Chen and Tzuang 2001; Haiyan et al. 2007). Owing to these significant benefits, China is becoming more and more interested in boosting the production of *Camellia oleifera*, which was projected to increase from 3.67 million hectares in 2016 to 4.67 million hectares in 2020 (Liu et al. 2017).

The studies revealing the effect of long-term cultivation of *Camellia oleifera* on soil properties indicated that *Camellia oleifera* cultivation in long-term induced higher abundance of soil organic matter (SOM), available nitrogen (AN), and available phosphorus (AP) contents (Lu et al. 2022). Moreover, *Camellia oleifera* in the intercropping system, can have benefit effect on soil properties. A study demonstrated that an intercropping *Camellia oleifera* with peanut induced improvement of bulk density, soil nutrient elements such as Nitrogen (N), Calcium (Ca), Magnesium (Mg) and SOM (Liu 2018). However, the distributions of these soil properties within soil aggregate particle sizes are very limited in soil under *Camellia oleifera* cultivation. The recent studies on the effect of long-term cultivation of *Camellia oleifera* on soil aggregates indicated that cultivation of *Camellia oleifera* affects aggregate stability depending on the land management practices and plantation ages (Zheng et al. 2023). Furthermore, the length of *Camellia oleifera* plantation and aggregate size plays an important role in the regulation of bacterial community composition (Lu et al. 2022). Still, the change in soil properties including soil pH, nutrients cycling, soil organic carbon and their chemical composition in soil aggregates particle sizes under long-term *Camellia oleifera* cultivation remains unclear and need to be elucidated. This missing information limit the understanding of soil aggregates formation and stability and indirectly the soil fertility management under *Camellia oleifera* cultivation. Therefore, the aims of this study were to assess the effect of long-term *Camellia oleifera* cultivation on the distribution of SOM, their chemical composition as well as soil nutrients in the soil aggregate particles. Our fundamental hypothesis was that long-term trees cultivation will improve soil chemical properties including SOM (amount and chemical composition) and available nutrients within aggregate particles.

2 Materials and Methods

2.1 Study Site

The samples were collected at Youxian County, Hunan Province (27°03’45"N, 113°24′31"E), China. The map indicating the sampling points was reported by Lu et al. (2022). Landforms in the experimental area was low hills. The experimental area was located at the foot slope, with an altitude of less than 300 m and the angle of the slope less than 15 degrees.

less than 300 m and average slope of less than 15 degrees. The climate was humid subtropical monsoon, with the annual rainfall of 1410 mm, mainly concentrated in June to September, and the annual temperature of 17.8 °C. The vegetation of the study area was dominated by *Camellia oleifera*. The soil type was Oxisol (Soil Survey Staff 2014), and the sample was collected at the topsoil (0–20 cm).

2.2 Experimental Design

In the study area, three kinds of *Camellia oleifera* cultivation ages were considered. The cultivation periods of *Camellia oleifera* was 2 years periods (Chi-Per), 10 years (You - Per) and 40 years (Old - Per) (Table 1). The Chi- Per treatment was selected to represent the initial soil environment of

Camellia oleifera cultivation. The You-Per treatment presented the high soil nutrient metabolism and plant yield. The degeneration periods (Old-Per) *Camellia oleifera* presented relative low yield. Three 20 × 20 m sample plots with the same growth stages were distributed in different locations of Liantang Ao Village, with the same elevation, slope, and aspect. Five representative plants were selected randomly from each plot as sampling plants. The soils were sampled in Nov. 2019 after harvest and were used for chemical properties analysis after air dried.

2.3 Soil Fractionation and Analysis

Prior to soil analysis, the following wet-sieving method of Elliot (1986) were used to collect soil aggregates: 100 g of soil samples was added to the 5 mm sieve and immersed in 1 cm of deionized water (DDW) for 10 min to allow agitation to occur. After agitation, the soil was sieved for another 10 min to remove the sieve up and down using an aggregate structure analyzer. Fluctuating litter was separated for recovery analysis. The following aggregates > 2 mm, 2-0.25 mm and < 0.25 mm were separated. All separated aggregates were oven-dried at 50 °C, and weighed. The weight of oven-dried soil for each size class was expressed as a percentage of the total weight.

In order to calculate the soil organic matter (SOM), soil organic carbon (SOC) was analyzed using the dichromate wet combustion method and a visible spectrophotometer (Van Gaans et al. 1995) and thus, the SOM content was therefore calculated by multiplying SOC by the conventional "Van Bemmelen factor" of 1.724, which assumes that soil organic matter contains 58% C(Allison 1965). This approach was successively used previously (Shamrikova et al. 2022). After SOM calculation, the SOM/N ratio was calculated.

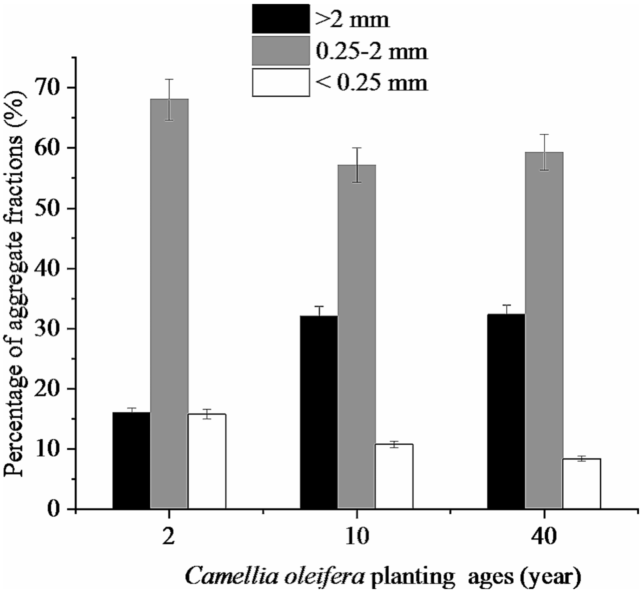

Fig. 1 Percentage of aggregate fractions (%) in the soils from the *camellia oleifera* planting ages. **2**: Chi-Per, soil from the *camellia oleifera* 2 years planting ages; **10**: You-Per, soil from the *camellia oleifera* 10 years planting ages and **40**: Old-per, soil from the *camellia oleifera* 40 years planting ages

The available nitrogen (AN) was analyzed using Kjeldahl method (Kirk 1950). The available phosphorus (AP) in the soil was determined by the digestion method, the soil pH was determined at 1: 2.5 soil water suspensions (Jackson 1973) and available potassium (AK) were determined by the Flame Photometer (Jackson 1973).

2.4 Ftir Analysis

The Fourier-transform infrared spectra of bulk soil and aggregate fractions were recorded as an average of 32 scans, with a wavelength resolution of 4 cm^{-1} in the range of 400–4000 cm^{-1} using a VERTEX70 FTIR spectrometer (Bruker, Hamburg, Germany). Prior to measurement, 1 mg sample of each soil sample was mixed with 100 mg^{-1} KBr and ground in an agate mortar. The mixture was oven-dried at 105 °C for 24 h. The assignment of the main FTIR absorption bands of the bulk soil and aggregate fractions are recorded in Table 2. A semi-quantitative analysis of organic carbon functional groups was determinate according to the method developed previously (Xue et al. 2019).

2.5 Statistical Analysis

Two-way analysis of variance (ANOVA) was used to examine the effect of forest stand age, aggregates size and forest stand age × aggregate sizes on the soil organic matter and available nutrients. Statistical significance was evaluated at the $P < 0.05$ level. Data analysis was carried out using SPSS 20.0 (IBM). Figures were plotted using Origin 2018.

3 Results

3.1 Aggregates Particles and Particles Size Distribution

The distribution of aggregates fractions in function of *Camellia oleifera* cultivation ages was presented in Fig. 1, and the particles size distributions are reported in Table 3. The percentage of aggregates changed with *Camellia oleifera* planting age. In the soil of Chi-per, the percentage of macroaggregates size (> 2 mm) was near to 16%. In soil of You-per, this percentage of macroaggregates increased (32%) to become 2 times higher than the percentage in Chi-per. This percentage (32%) did not change in soil of Old-per.

The percentage of mesoaggregates (0.25-2 mm) varied with *Camellia oleifera* planting age. For the soil of Chi-per, the percentage of mesoaggregates was close to 68%. In You-per, this percentage shifted to near 57%, and did not significantly change in soil of Old-per (59%). The percentage of microaggregates (< 0.25 mm) was close to 16% in soil of Chi-per. In the soil of You-per, the percentage of microaggregates slightly decreased to 11%. This percentage continued to decrease (8%), mostly in soil of Old-per.

The distribution of particle sizes in bulk soil and macroaggregates of Chi-per sample showed that the silt fraction represents near to 55% of the total particle fractions, followed by sand (23%) and clay (23%). With the decrease in aggregates size to < 0.25 mm, the percentage of sand fraction declined to 16% while that of silt and clay increased to 58% and 24%, respectively (Table 3). In the sample of You-per, the percentage of particle size in the bulk and aggregates fractions was not significantly affected. However, in the sample of Old- Per, the sand and clay fractions significantly declined between 6 and 10%, and 37%-26%, respectively while the silt fraction gradually increased in the range of 57-63%. The increasing of *Camellia oleifera* planting age promoted the silt and clay formation in bulk and aggregate size.

3.2 Distribution of Soil Organic Matter in the Bulk Soil and Aggregates Particles

The distribution of SOM was significantly ($P < 0.05$) affected by *Camellia oleifera* planting age (Fig. 2). The lowest content of SOM was observed in soil of Chi-per, with values varying between 12.66 and 14.30 mg/kg. Compared to Chi-per, the content of SOM significantly ($P < 0.05$) increased between 25.41 and 29.20 mg/kg in You-Per. The highest content of SOM was reported in Old-Per, with values changing between 34.36 and 41.53 mg/kg.

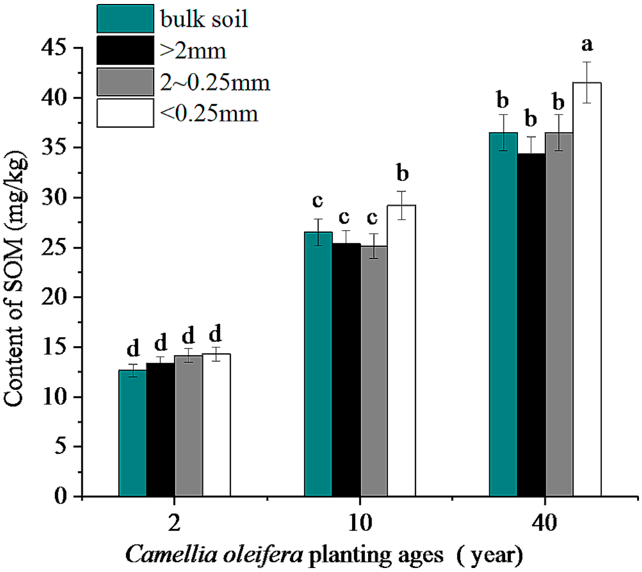

Fig. 2 The soil organic matter (SOM) content in the bulk soil and aggregate fractions for the *camellia oleifera* treatments. **2**: Chi-Per, soil from the camellia oleifera 2 years planting ages; **10**: You-Per, soil from the camellia oleifera 10 years planting ages and **40**: Old-per, soil from the camellia oleifera 40 years planting ages

The distribution of SOM significantly ($P < 0.05$) changed also depending on aggregates size (Table 3). In the bulk soil, the content of SOM was 12.66 mg/kg in Chi-per, then significantly increased ($P < 0.05$) to 26.53 mg/kg in You-per, and to 36.53 mg/kg in Old-per. The distribution of SOM content in macroaggregate showed the content of 13.37 mg/kg in Chi-per. This content of SOM in macroaggregates significantly ($P < 0.05$) increased to 25.41 mg/kg in You-per. The increase of SOM content in macroaggregates remained significant ($P < 0.05$) in Old-per, with the value increasing to 34.36 mg/kg. The distribution of SOM in mesoaggregates showed that the content of SOM which was 14.16 mg/kg in mesoaggregates of Chi-per increased significantly to 25.14 mg/kg in mesoaggregates of You-per and to 36.52 mg/kg in mesoaggregates of Old-per. Similarly, to the distribution of SOM content in mesoaggregates, the content of SOM in microaggregate significantly increase in the order of Chi-per, You-per and Old-per.

3.3 Accumulation of Organic Carbon Functional Group in Aggregates Particles

The FTIR spectra of bulk soil and aggregates were showed in Fig. 3 and revealed the distributions of chemical composition of SOM which were summarized in Table 3. The FTIR spectra of each soil showed adsorption bands in the frequency range of $4000-500$ cm^{-1}, with prominent features. The different bulk soils and aggregates displayed similar spectra relative to each other at the different *Camellia oleifera* cultivation ages. Phenolics-C, ketones-C aromatics-C, carboxylic-C, aliphatic-C, polysaccharides-C, lignins-C, alkene-C were among the major organic carbon chemical composition.

The distribution of soil organic carbon chemical composition changed depending on the aggregates size and *Camellia oleifera* plantation standing age. In the bulk soil of Chi-per, the aromatics-C was dominant. This finding was also observed previously by Xue et al. (2019). This dominance of SOM chemical composition changed to Aliphatic-C group in bulk soil of You-per, and to aromatics-C functional group in bulk soil of Old-per. In macroaggregates, the abundance of aromatics-C in Chi-per changed to alkenes-C in You-per and Old-per. In mesoaggregates of Chi-per, the soil organic matter was dominated by aromatics-C. This dominance of chemical composition of soil organic matter shifted to aliphatic-C and alkenes-C in mesoaggregate of You-per and

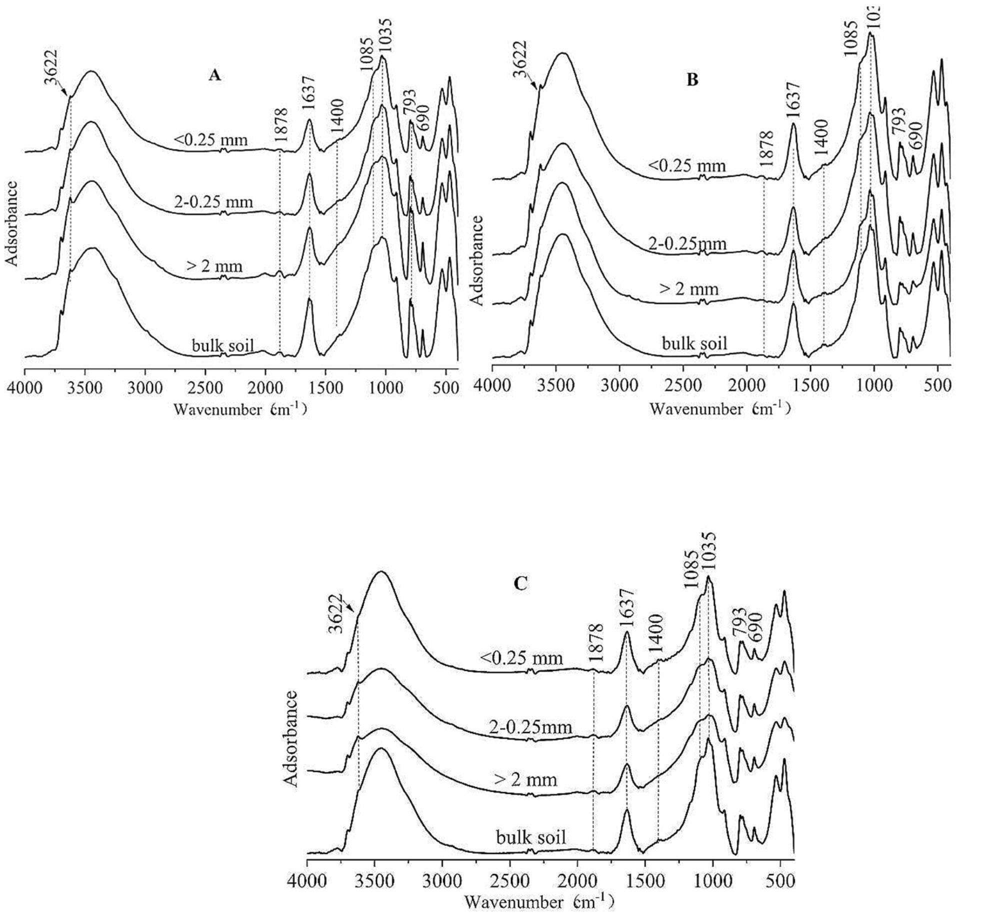

Fig. 3 Fourier-transform infrared spectra of bulk soil and aggregates for the soil with different *camellia oleifera* treatments. **A**: soil from the camellia oleifera 2 years planting ages **(Chi-Per)**; **B**: soil from the camellia oleifera 10 years planting ages **(You-Per)** and **C**: soil from the camellia oleifera 40 years planting ages **(Old-per)**

Old-per, respectively. The alkenes-C and lignin-C were the dominant functional group in microaggregates of all studied soils (Table 4).

3.4 Distribution of ph, Available n, k and p in Bulk and Soil Aggregates Particles

The distribution of pH, available nitrogen (AN), potassium (AK) and phosphorus (AP) in the aggregates were reported in Table 3 and Fig.A (Supplementary data 1). This distribution significantly ($P < 0.05$) changed with aggregate size of different plantation standing age (Table 5).

The soil remained acidic (pH = 4) in bulk soils and in different soil aggregates. Similarly, this pH did not change significantly as the *Camellia oleifera* plantation standing age changed from 2 to 40 years.

In the bulk soil of Chi-per, the percentage of AN was 57.85%. This percentage did not significantly ($P > 0.05$) change in bulk soil of You-per, compared to the bulk soil of Old-per where the percentage of AN became 2 time bigger (120.20%) compared to that in the bulk soil of Chi-per. The distribution of AN in macroaggregates indicated that the percentage of AN was 57.89 in Chi-per. This percentage was not significant ($P > 0.05$) in You-per. Compared to Chi-per, the percentage of AN significantly ($P < 0.05$) increased to 114.32% in Old-per. In the meso and microaggregates size, the percentage of AN followed the similar trend than in macroaggregates size, increased significantly ($P < 0.05$) in the order of Chi-per, You-per and Old-per.

The SOM/N ratio in the aggregates changed significantly ($P < 0.05$) in soils with different *Camellia oleifera* cultivation ages (Table 3). The variation of SOM/N ratio in Chi-per was between 0.22 and 0.23. This ratio significantly increased, with values between 0.49 and 0.62 in You-per. These values significantly decreased in Old-per (0.30–0.34). The distribution in aggregates showed an increasing SOM/N ratio with decreasing aggregates size in all the studied soils.

The AP showed very low percentage content (<2%) in the bulk and aggregates of all the studied soils. These percentage contents declined with the decreasing aggregates size in all soils. As the age of *Camellia oleifera* cultivation increased from Chi-per, You-per and Old-per, the percentage content of AP decreased significantly.

Similar to AP, the percentage of AK was low, with the values varying between 5.22 and 6.20% in soil of Chi-per, 3.02-5.23% in soil of You-per and 2.00-2.18% in soil of

Old-per. This percentage content increased significantly with the decreasing of aggregates size in all the studied soils.

3.5 Discussion

As the *Camellia oleifera* cultivation age increased, the percentage of macroaggregates in soil increased, and that of mesoaggregates and microaggregates slightly decreased. Our study revealed that the long-term *Camellia oleifera* cultivation affected aggregates fractions by increasing and maintaining the aggregates formation. On the other hand, the finding of our study revealed that *Camellia oleifera* has promoted the formation of soil aggregates with size > 2 mm, 0.25-2 mm and < 0.25 mm aggregates, after long-term cultivation. This finding was similar with those reported previously using long-term cultivation of other plant species (Lan et al. 2022; Wei et al. 2013), indicating that long-term trees cultivation improved the aggregates formation.

The formation and stability of soil aggregates are very important for physical protection of soil organic matter (SOM) (Gelaw et al. 2015). Soil aggregation is critical for soil fertility since it reduced soil erosion and mediated soil aeration, water infiltration, and retention (Six et al. 2004; Zhao et al. 2017). Thus, the distribution of soil aggregates was of great significance for the cycling of soil nutrients and for soil carbon sequestration (Xue et al. 2019). Many raisons can explain the increasing of aggregates formation and stability after long-term trees cultivation. Previous studies have supported that increasing soil organic matter provided good planting environment for shade-tolerant plants, and the plentiful vegetation can reduce damage to the soil aggregate structure caused by the direct physical impact of rain water (Bai et al. 2020; Yao et al. 2020). The increasing aggregate formation was also promoted by the soil nutrients abundance. Soil nutrients, particularly those derived from organic amendments, have been found to enhance soil aggregates formation and stability (Csitári et al. 2021). This was done through the production of organic acids, biodiversity improvement, chelates, and increased earthworm population (Bashir et al. 2021).

In our study, the soil organic matter increased with increasing *Camellia oleifera* planting age (Table 3). The analysis of SOM distribution in soil aggregates after long-term *Camellia oleifera* planting age showed that as the *Camellia oleifera* planting age increased from 2, 10 and 40 years, the content of SOM significantly ($P < 0.05$) increased. The increasing of SOM suggested that the long-term cultivation of *Camellia oleifera* promoted the SOM accumulation, highlighting the key role of trees planting age in SOM formation. This was in agreement with previous studies reporting the accumulation of SOM under long-term trees planting age (Ortiz et al. 2022; Zhang et al. 2021). This finding suggested that the soil under trees cultivation may be considered as greater sink for carbon in long-term.

Furthermore, the content of SOM equally increased with decreasing aggregates size from macroaggregates to microaggregate sizes and more accumulated in microaggregates than in macroaggregates and mesoaggregates. This indicated that the storage of SOM with increasing *Camellia oleifera* planting age could also be due to the accumulation of SOM in microaggregates (< 0.25). Previous studies have revelated the accumulation of SOM mostly in macroaggregates (Jiang et al. 2019; Zhang et al. 2021). Our study revealed that while macroaggregates are the primary reservoirs for SOM, the distribution and dynamics of SOM can still vary within different aggregate size classes and thus microaggregates can also be the carbon sink in soil under trees cultivation in long term. The microaggregates can provide the better protection to SOM compared to the macroaggregates where the turnover was supposed to be more rapid (Six and Paustian 2014; Tian et al. 2016). Macroaggregates usually contain enriched labile SOM, which has a faster turnover rate and is easier to be mineralized (Nadal-Romero et al. 2016; Six et al. 2000). In this study, the change in SOM chemical composition revealed that the hardly decomposed carbon including phenolics-C, ketones-C, lignins-C and alkenes-C increased in soil aggregates with trees planting age, and mainly distributed in microaggregates while the decomposed carbon including aromatics-C, aliphatic-C and polysaccharides-C declined, and mainly distributed in meso and macroaggregates. This distribution indicated that organic carbon is more relatively stable in microaggregate compared to macroaggregates in this study. This can explain the abundance of SOM in soil microaggregates in this study compared to macroaggregates. It was reported that organic carbon mineralization in the macroaggregates was controlled by the decomposition of labile organic carbon while the mineralization in the fine fraction is controlled by the decomposition of relatively stable organic carbon (Nadal-Romero et al. 2016; Six et al. 2000).

SOM is not the only parameter affected by aggregate size. Our study revealed that AN, AK and AP were significantly affected by soil aggregates size and *Camellia oleifera* planting age (Table 5). The AN concentration increased and AK and AP decreased with decreasing aggregates size (Table 3). As the *Camellia oleifera* planting age increased, there was a significant augmentation of AN concentration. Our finding is consistent with previous outcome that forest plantation can increase AN concentration over time (Chodak and Niklińska 2010; Parsapour et al. 2018), indicating that *Camellia oleifera* may fixed atmospheric N_2 over time, which may be accumulated in soil (Chodak and Niklińska 2010). Compared to AN, the very low concentrations of AK and AP gradually declined as the *Camellia oleifera* planting age decreased. The lack of AP had been reported previously in *Camellia oleifera* soils in China (Liu 2018). Our study confirmed the low availability of AP and AK which may reduce the *Camellia oleifera* growth. Therefore, good strategies should be employed to improve the content of AP and AK while protecting SOM from decomposition in these soils in order to accelerate the growing of *Camellia oleifera* in this region.

4 Conclusion

This study highlights the effect of *Camellia oleifera* planting age on soil aggregates, SOM, and other available nutrients. Therefore, the main findings of this study revealed that long-term cultivation of *Camellia oleifera* promoted the increasing formation of soil aggregates. The accumulation of soil organic matter (SOM) increased with increasing *Camellia oleifera* planting age. The SOM chemical composition changed depending on the aggregate size and *Camellia oleifera* planting age. The phenolics-C, ketones-C, lignins-C, and alkenes-C increased in soil aggregates with *Camellia oleifera* planting stacking time, and mainly distributed in microaggregates while aromatics-C, carboxylic-C, aliphatic-C and polysaccharides-C declined, and mainly distributed in meso and macroaggregates. The study revealed the increasing abundance of available nitrogen (AN) after 10 years of *Camellia oleifera* cultivation Vs the lowest abundance of available potassium (AK) and available phosphorus (AP) over the time. This information were very helpful for the sustainable management of Camellia oleifera forest soils in China.

Supplementary Information The online version contains supplementary material available at https://doi.org/10.1007/s42729-024-01682-4.

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Data Availability Data sharing not applicable to this article as no datasets were generated or analyzed during the current study.

Declarations

Competing Interests The authors have no competing interests to declare that are relevant to the content of this article.

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