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Dynamics of Soil Organic Carbon and Labile Carbon Fractions in Soil Aggregates Affected by Different Tillage Managements

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Abstract: Our study aimed to provide a scientific basis for an appropriate tillage management of wheat-maize rotation system, which is beneficial to the sustainable development of agriculture in the fluvo-aquic soil areas in China. Four tillage treatments were investigated after maize harvest, including rotary tillage with straw returning (RT), deep ploughing with straw returning (DP), sub-soiling with straw returning (SS), and no tillage with straw mulching (NT). We evaluated soil organic carbon (SOC), dissolved organic carbon (DOC), permanganate oxidizable carbon (POXC), microbial biomass carbon (MBC), and particulate organic carbon (POC) in bulk soil and soil aggregates with five particle sizes (>5 mm, 5–2 mm, 2–1 mm, 1–0.25 mm, and <0.25 mm) under different tillage managements. Results showed that compared with RT treatment, NT treatment not only increased soil aggregate stability, but also enhanced SOC, DOC, and POC contents, especially those in large size macroaggregates. DP treatment also showed positive effects on soil aggregate stability and labile carbon fractions (DOC and POXC). Consequently, we suggest that no tillage or deep ploughing, rather than rotary tillage, could be better tillage management considering carbon storage. Meanwhile, we implied that mass fractal dimension ($D_m$) and POXC could be effective indicators of soil quality, as affected by tillage managements.

Keywords: tillage; soil aggregates; soil organic carbon (SOC); labile carbon fractions

1. Introduction

Soil organic carbon (SOC) is one of the most widely used indicators of soil fertility. SOC affects soil physical, chemical, and biological characteristics, regulating soil quality and ecosystem function [1]. In addition, due to its potential for carbon sequestration, SOC plays an essential role in global carbon cycle and climate change [2]. Agricultural soils, as strongly affected by human activities, are important parts of terrestrial SOC pools [3]. Increasing soil organic carbon pools through appropriate agricultural measures is of great significance, not only in agricultural production, but also in the sustainable development of ecological environment. However, SOC stocks are not sensitive to changes in agricultural soil management due to the complexity and stability of its multiple compounds. Consequently, the labile pools of SOC are usually measured to evaluate the rapid change of soil quality [1].

Labile organic carbon is easily mineralized, and it is directly available for microbial activity. It originates from the decomposition of plant litter, root exudates, hydrolysis of soil organic matter, microbes, and their metabolic products [4]. Dissolved organic carbon (DOC) refers to organic carbon in the soil solution, which is directly involved in soil biochemical process [5]. Particulate organic carbon (POC) is determined by particle size. It consists of undecomposed or semi-decomposed microbial biomass, plants, and root residues [6]. Permanganate oxidizable carbon (POXC) and microbial biomass carbon (MBC) are important carbon fractions that regulate key processes such as nutrient cycling and its availability, soil aggregation, and soil carbon accrual [7]. Labile organic carbon fractions...
include various components, characterized by short turnovers; hence, they are considered to be more sensitive indicators of agricultural managements, as compared to SOC [8].

Soil aggregates are basic units of soil structure. Aggregate-protected soil organic matter is an important platform for organic carbon stabilization [9]. It is reported that macroaggregates (>0.25 mm) make a larger contribution to SOC accumulation than microaggregates (<0.25 mm), and are more sensitive to land use change [10]. However, tillage physically disrupts macroaggregates and exposes protected organic carbon to microbial decomposition. Tillage affects the turnover of macroaggregates and soil organic matter, thus, having impacts on soil aggregate stability and carbon sequestration [11].

The fluvo-aquic soil areas in North China Plain are important sources for food production in China. It is of vital importance to conduct a suitable tillage management for improving soil quality and ensuring sustainable development. The typical cropping system in the North China Plain is the rotation of winter wheat and summer maize. The common agricultural managements are rotary tillage after maize harvest. Negative effects, such as slowing soil tilled layers, the decline in soil capacity for water storage, and degradation of soil physical structure and quality, could be induced by such long-term single tillage mode [12]. No tillage is a typical conservation tillage method, which pursues minimum soil disturbance, so that it is beneficial to soil aggregation and soil structure. It has been confirmed that no tillage can enhance soil aggregate stability, protects SOC from mineralization, increases total organic carbon and labile organic carbon fractions in cultivated soil [13]. Deep ploughing and subsoiling are effective managements to break up pans in subsoil, reduce soil bulk density, and improve the tilled layer, therefore, facilitating root proliferation [14]. Furthermore, deep ploughing can translocate the SOC formed near the surface into the subsoil and mix SOC-poor subsoil material into the new topsoil. Some previous studies have reported the improvements in the aspects of soil physical and chemical properties by deep ploughing or subsoiling [15,16].

The content of soil macroaggregates and labile organic carbon have been used to access the impact of different tillage methods on soil quality [17,18] in many studies. However, the distribution of organic carbon and labile organic carbon fractions in different particle sizes of soil aggregates, and the mechanisms by which tillage managements affect them have not been well elucidated in the fluvo-aquic soil areas. In addition, it is not clear which labile fraction can be used to monitor the changes in SOC, as affected by tillage management. The objective of this study was to optimize a suitable tillage management that could promote soil structure and soil carbon sequestration. To this aim, we determined the soil aggregates distribution and stability, and associated organic carbon and labile carbon factions under different tillage practices. Soil aggregates were divided into five particle sizes: >5 mm, 5–2 mm, 2–1 mm, 1–0.25 mm, <0.25 mm. We tested the content of total organic carbon and four labile carbon fractions include DOC, POXC, MBC, and POC in aggregates with these five particle sizes. Moreover, we evaluated the relationship of different labile fractions with soil organic carbon and soil aggregate stability, which are linked to soil fertility and soil structure. We hypothesized that deep ploughing, subsoiling, or no tillage would enhance SOC contents compared with traditional rotary tillage.

2. Materials and Methods

2.1. Experimental Site and Soil Sampling

The tillage experiment was established in the Songfang Farm in Qihe County, Shandong province, China (36°47′ N, 116°46′ E), where the mean annual temperature is 13.4 °C and the mean annual precipitation is 622 mm. The soil type is fluvo-aquic soil. The typical cropping system is the rotation of winter wheat and summer maize.

Our tillage experiment started in 2016. Four plots in the farm with an area of about 10,000 m² were selected randomly for different tillage treatments. In all the treatments, maize was sowed directly with no tillage after wheat harvest. The other managements were the same apart from the tillage managements conducted in October, after maize harvest and straw returning. The specific tillage treatments were as follows, rotary tillage (RT
treatment), deep ploughing (DP treatment), subsoiling (SS treatment), and no tillage (NT treatment). RT treatment involved rotary cultivating twice by a rotary tiller to a depth of 10 cm. It was the local conventional tillage method before we carried out the experiment, which was taken as control in our study. DP treatment consisted of ploughing to a depth of 25 cm then rotary cultivating twice. SS treatment involved tilling to a depth of 30 cm with a subsoiler machine then rotary cultivating twice. NT treatment consisted of no tillage and straw mulching.

Soil samples were collected in September 2019, before the maize harvest. Three plots (10 m × 10 m) regarded as three repetitions were selected randomly in each treatment area. Soils were taken from 0–30 cm layer using an auger with 5 cm internal diameter, which allowed cores to be taken without compaction. Each sample is comprised of 15 soil cores collected in S-shape in each plot. Visible plants residues and stones were removed and then soil samples were gently broken by hand and passed through a 10 mm sieve. Parts of the soils were stored at 4 °C before analysis and the others were air-dried. DOC and MBC were analyzed within one week.

2.2. Soil Aggregates Fraction

Samples were mechanically sieved for 2 min to separate the soil into the following soil aggregate particle sizes: >5 mm, 5–2 mm, 2–1 mm, 1–0.25 mm, <0.25 mm [19]. The data on each aggregate size fraction was expressed as a percent relative to the total dry weight of the sample. Mean weight diameter (MWD), geometric mean diameter (GMD), and mass fractal dimension (D_m) were calculated as Cagna [20] and Tagar [21] to evaluate soil aggregate stability.

2.3. SOC and Labile Fractions Analysis

Soil organic carbon was determined by H2SO4-K2Cr2O7 oxidation followed by measuring with an UV–VIS Spectrophotometer and then calculated by the absorbance. DOC was obtained by water with soil-to-solution ratio of 1:5 and then passed 0.45 µm filter membranes [22]. MBC was extracted using the chloroform fumigation method [23]. DOC and MBC were both measured using a Multi N/C 3000 analyzer. POXC was determined by the KMnO4 (333 mM) oxidation procedure and calculated by loss of KMnO4. POC was obtained by (NaPO3)6 solution and passed through a 0.053 mm sieve, and finally determined as SOC method.

2.4. Statistical Analysis

All statistical calculations were carried out using SPSS 20.0 software (SPSS Inc., Chicago, IL, USA). Significant differences among treatments were analyzed by one-way analysis of variance (ANOVA) with Duncan’s Multiple Range test. Treatment and aggregate effects were considered to be statistically different when p < 0.05. Pearson correlation was used to evaluate the relationship between soil aggregate stability indices and organic carbon fractions.

3. Results

3.1. Soil Aggregate Distribution, MWD, GMD, and D_m

Soil aggregate distribution was influenced by tillage effects (Figure 1). Compared to RT treatment, DP treatment showed a significantly higher proportion of >5 mm macroaggregates with a lower proportion of 5–2 mm and 1–0.25 mm macroaggregates. SS treatment showed higher microaggregate proportion. However, there was no statistical difference in the macroaggregate fractions between SS and RT treatments. Compared with RT, there was an increase in >5–2 mm macroaggregate proportion while a decrease in 1–0.25 mm macroaggregate proportion in NT treatment. Furthermore, the microaggregate proportion of NT treatment was significantly lower than the other treatments. In other words, NT treatment had the highest proportion of macroaggregates.
The MWD enhanced in DP and NT treatments compared to RT, with the value of 18% and 11%, respectively (Table 1). DP and NT treatment showed greater GMD than RT, with the value of 23% and 21%, respectively. There was no significant difference between SS and RT with regard to MWD and GMD. Besides, NT treatment showed a significantly lower $D_m$ compared to RT, indicating that the soil aggregate size distribution was mainly dominated by larger fragments in NT treatment.

Table 1. Effects of tillage on MWD, GMD, and $D_m$.

| Treatment | MWD (mm)     | GMD (mm)     | $D_m$      |
|-----------|--------------|--------------|------------|
| RT        | 2.59 ± 0.14b | 1.77 ± 0.18b | 2.15 ± 0.10a |
| DP        | 3.07 ± 0.16a | 2.18 ± 0.15a | 2.08 ± 0.01ab |
| SS        | 2.60 ± 0.12b | 1.76 ± 0.13b | 2.18 ± 0.06a |
| NT        | 2.88 ± 0.06a | 2.14 ± 0.05a | 1.99 ± 0.09b |

RT: rotary tillage, DP: deep ploughing, SS: subsoiling, NT: no tillage. MWD: mean weight diameter, GMD: geometric mean diameter, $D_m$: mass fractal dimension. Different letters in the same column indicate significant difference of the indices at 0.05 level among treatments.

3.2. Soil Organic Carbon Distribution

In the bulk soil, SOC content significantly increased by 8% in NT treatment compared to RT treatment (Table 2). Yet, the SOC content in DP and SS treatment had no statistical difference with RT.

The distributions of SOC content within soil aggregates are shown in Figure 2. The content of SOC was higher in microaggregates than in macroaggregates under all tillage treatments. Furthermore, there was a trend that SOC content substantially increased with the decrease in aggregate particle sizes in RT, SS, and NT treatments. However, in DP treatment there was no statistical difference in the organic carbon contents among four sizes of macroaggregates. Compared to RT treatment, NT treatment showed significantly higher organic carbon content in >5 mm, 2–1 mm and <0.25 mm aggregates. Organic carbon content in DP treatment was higher than RT in >5 mm aggregates, while it was lower in 1–0.25 mm and <0.25 mm aggregates. Besides, SS treatment also had lower organic carbon content than RT treatment in 1–0.25 mm and <0.25 mm aggregates.

Tillage had impacts on contribution rates of different soil aggregate fractions to SOC content (Table 3). In RT, DP, SS, and NT treatment, the aggregates-associated organic carbon...
were dominated by the 1–0.25 mm, >5 mm, 1–0.25 mm, and 5–2 mm fractions, respectively. The <0.25 mm aggregates, followed by 2–1 mm fractions, contributed the lowest organic carbon to total SOC under all treatments. Compared with RT treatment, DP significantly increased the contribution rates of >5 mm aggregates, while decreased that of 5–2 mm and 1–0.25 mm fractions. In comparison, NT treatment increased the contribution rates of 5–2 mm aggregates and decreased that of 1–0.25 mm and <0.25 mm aggregates.

Table 2. Effects of tillage on contents of SOC and labile fractions in bulk soil.

| Treatment | SOC (g kg⁻¹) | DOC (mg kg⁻¹) | POC (g kg⁻¹) | MBC (mg kg⁻¹) | POXC (g kg⁻¹) |
|-----------|--------------|---------------|--------------|---------------|---------------|
| RT        | 10.78 ± 0.61b | 36.99 ± 1.96b | 2.52 ± 0.17b | 575.08 ± 21.74a | 3.11 ± 0.06b  |
| DP        | 10.36 ± 0.15b | 45.59 ± 0.77a | 2.59 ± 0.08b | 507.75 ± 15.26b | 3.27 ± 0.03a  |
| SS        | 10.23 ± 0.34b | 39.68 ± 2.07b | 2.65 ± 0.10b | 421.15 ± 11.23c | 2.93 ± 0.01d  |
| NT        | 11.70 ± 0.16a | 44.36 ± 0.61a | 2.90 ± 0.14a | 500.12 ± 23.06b | 3.03 ± 0.04c  |

RT: rotary tillage, DP: deep ploughing, SS: subsoiling, NT: no tillage. SOC: soil organic carbon, DOC: dissolved organic carbon, POC: particulate organic carbon, MBC: microbial biomass carbon, POXC: permanganate oxidizable carbon. Different letters in the same column meant significant difference of the content in bulk soil at 0.05 level among treatments.

Table 3. Contribution rates of different particle sizes soil aggregates to SOC content.

| Treatment | >5 mm | 5–2 mm | 2–1 mm | 1–0.25 mm | <0.25 mm |
|-----------|-------|--------|--------|-----------|----------|
| RT        | 20.91 ± 2.25b | 23.55 ± 1.82b | 14.38 ± 1.58a | 29.75 ± 2.75a | 8.94 ± 1.03a |
| DP        | 41.14 ± 2.79a | 20.01 ± 1.64c | 13.00 ± 1.76a | 19.01 ± 3.52b | 8.20 ± 0.54ab |
| SS        | 22.80 ± 0.97b | 24.37 ± 1.52ab | 15.13 ± 1.74a | 27.46 ± 2.14a | 10.89 ± 2.41a |
| NT        | 24.89 ± 4.32b | 27.18 ± 1.36a | 14.39 ± 0.38a | 21.14 ± 1.17b | 5.89 ± 0.89b  |

RT: rotary tillage, DP: deep ploughing, SS: subsoiling, NT: no tillage. The value of the contribution rates are percent (%). Different small letters in the same column meant significant difference of aggregate contribution rates at 0.05 level among treatments.

3.3. Labile Organic Carbon Fractions Distribution

As shown in Table 2, tillage exerted different effects on organic carbon in four labile fractions. Compared to RT, DOC content was significantly higher in DP and NT treatments. Regarding POC content, there was only significant difference between NT and RT treatments. MBC contents showed the highest value in RT treatment, while the lowest MBC
content was found under SS treatment. SS treatment, followed by NT treatment, showed lower POXC content than RT. While POXC content in DP increased by 15% compared with RT treatments.

Figure 3 shows that DOC and POXC content increased with the decrease in aggregate particle sizes in all treatments. Compared with RT treatment, DP and NT treatments showed significantly higher DOC content in large size macroaggregates. However, significant lower DOC values were found in microaggregates in DP treatment. It was 1–0.25 mm macroaggregates that showed the highest POC content in RT, DP, and SS treatments, while it was 5–2 mm in NT treatment. DP, SS, and NT treatments showed lower POC content in small size macroaggregates and microaggregates. Only NT treatment showed significantly higher POC in large macroaggregates. Compared with RT treatment, SS and NT treatments contained lower MBC content in all particle sizes aggregates. In DP treatment, there were higher values of MBC content in 5–2 mm and 2–1 mm aggregates, while lower values of that in 1–0.25 mm and <0.25 mm aggregates than RT. Regarding POXC content, DP treatment showed significantly higher POXC content in all aggregate sizes. While the values in SS treatment were significantly lower. NT treatment showed higher POXC in macroaggregates but lower values in microaggregates than RT.

Figure 3. Effects of tillage on DOC, POC, MBC, and POXC content in aggregates. Shown is the data for: (a) DOC content in aggregates; (b) POC content in aggregates; (c) MBC content in aggregates; (d) POXC content in aggregates. Different filling types refer to different treatments. RT: rotary tillage, DP: deep ploughing, SS: subsoiling, NT: no tillage. Different letters in the same group of bars indicate significant difference of labile organic carbon content in aggregates at 0.05 level among treatments.

3.4. Correlation between Aggregate Stability and Organic Carbon Fractions

As shown in Table 4, there are significant relationships among MWD, GMD, and $D_m$. In particular, we found that $D_m$ was negatively correlated to MWD and GMD. Among the labile carbon fractions, POXC and POC showed the strongest relationship with SOC. Moreover, SOC, DOC, POXC, and POC were strongly negatively correlated with $D_m$, while their statistical relationship with MWD and GMD were not found, with the exception of significantly positive correlation found among MWD, GMD, and DOC.
### Table 4. Correlation between aggregate stability indices and organic carbon fractions.

| Item   | MWD   | GMD   | Dm   | SOC   | DOC   | POXC  | MBC   | POC   |
|--------|-------|-------|------|-------|-------|-------|-------|-------|
| GMD    | 0.971 ** | 1     |      |       |       |       |       |       |
| Dm     | −0.703 * | −0.843 ** | 1    |       |       |       |       |       |
| SOC    | 0.284  | 0.490 | −0.832 ** | 1    |       |       |       |       |
| DOC    | 0.925 ** | 0.924 ** | −0.749 ** | 0.331 | 1    |       |       |       |
| POXC   | 0.324  | 0.056 | −0.771 ** | 0.928 ** | 0.314 | 1    |       |       |
| MBC    | −0.049 | 0.039 | −0.085 | 0.416 | −0.280 | 0.455 | 1    |       |
| POC    | 0.437  | 0.590 * | −0.774 ** | 0.755 ** | 0.577 * | 0.632 * | 0.138 | 1    |

MWD: mean weight diameter, GMD: geometric mean diameter, Dm: mass fractal dimension, SOC: soil organic carbon, DOC: dissolved organic carbon, POXC: particulate organic carbon, MBC: microbial biomass carbon, POX: permanganate oxidizable carbon. *, ** meant significant difference at 0.05, 0.01 probability level, respectively.

### 4. Discussion

#### 4.1. Effects of Tillage on Soil Aggregate Stability

MWD and GMD are general indices to characterize soil aggregate stability. Higher values of MWD and GMD indicate higher stability of soil structure [20]. In recent years, mass fractal dimension (Dm) are widely used and considered to be more suitable than MWD and GMD to evaluate the impact of agricultural management on soil aggregate stability [24]. The value of mass fractal dimension depends on the shape of individual objects within the distribution, and the overall extent of soil aggregate fragmentation [25]. The lower value of Dm is associated with more stable soil aggregates.

In our study, NT treatment had the highest proportion of macroaggregates, higher value of MWD and GMD, and lower value of Dm than RT. This is consistent with one previous study, which found that no tillage had a positive effect on soil aggregation and aggregate stability [26]. No tillage could not only reduce the mechanical disturbance and the fragmentation of macroaggregates, but also decrease the damage to the roots and the network of fungal hyphae, and increase the content of organic binding agents, thereby protecting macroaggregates [27]. Compared with rotary tillage, which mixes the topsoil but does not change the structure of subsoil, deep ploughing turns soil and inverts the compacted subsoil to surface, which may explain the higher mass proportion of >5 mm macroaggregates and higher soil aggregate stability in DP treatment. However, the portion of macroaggregates in DP might finally decrease after the subsoil were broken and mixed into topsoil in the long term of tillage treatment.

#### 4.2. Effects of Tillage on Soil Organic Carbon and Labile Carbon Fractions

Tillage affected SOC by impacting the mass of aggregates and the content of aggregate-associated organic carbon. According to Six et al. [28], organic carbon in macroaggregates is younger and more mineralizable, while organic carbon in microaggregates is mostly highly humified inert components. An important factor of carbon accumulation is that fresh residues gradually decompose and finally enter microaggregates from macroaggregates. Tillage promotes the turnover of macroaggregates and the organic carbon mineralization in macroaggregates, therefore, reduces the stabilization of plants-originated SOC in microaggregates [29]. Our results showed that the increased SOC content in bulk soil of NT treatment was mainly reflected in >5 mm macroaggregates, which indicated that no tillage is beneficial to organic carbon accumulation in macroaggregates. Besides, DP and SS treatments decreased SOC content in microaggregates, indicating their stronger disturbance to soil aggregate turnover.

DOC consists of organic compounds present in soil solution. Due to its high bioavailability, DOC is an important substrate for soil microbial growth, influencing the availability and mobility of soil nutrients in farmland ecosystems [30]. Dong et al. [31] suggested that intensive tillage may result in less immobilization of soil carbon by microorganisms. In our study, different tillage treatments affected DOC content, but did not change the distribution
mode of DOC in soil aggregates, this is, DOC content increases with the decreasing soil aggregate particle sizes. The higher DOC content in DP and NT treatment may originate from the decomposition of rooting system, which involves in less tillage disturbance, and mainly occurred in macroaggregates.

Six [11] suggested that the development of POC is an important mechanism by which organic carbon was stored in no tillage soil. Firstly, soil particles combine plant residues into macroaggregates. Then the POC and organic binding agents are polymerized into microaggregates to be protected. In our study, the greatest POC content was found in NT treatment. One reasonable explanation for this result is that macroaggregates have slower turnover under the no tillage condition. In addition, the highest mass proportion of macroaggregates in NT also contribute to this difference. Further, >5 mm and 5–2 mm macroaggregates in NT contained higher POC than those in the other treatments. This is consistent with the soil aggregate hierarchy model concept by six [31].

In our study, it was large macroaggregates in four treatments that showed greater difference in MBC values. This may indicate that tillage managements exerted larger influence on microbial metabolic activity in large size macroaggregates. Xiao et al. [32] reported that tillage disturbance increases microbial metabolic activity and increased MBC content. This may be caused by the releasing of POC in macroaggregates. It could explain why the greatest MBC content was found under RT treatment in our study. We conjecture that MBC content in RT treatment may decrease with the depletion of newly released POC. According to Zhong et al. [33], however, NT treatment might show higher MBC content after long-term conservation tillage because of its minimum disturbance to microbial habitats.

POXC, which comprises carbon derived from dissolved organic matter and microbial biomass, is influenced by carbon input from retained residues and the distribution of crop roots [34]. In our study, tillage methods did not have influence on POXC distribution in aggregates. The POXC content in bulk soil showed in the following order: DP > RT > NT > SS. The order of POXC in each size of soil aggregates was almost consistent with that in bulk soils. The possible explanation is that deep ploughing buried straw into subsoil, where the relatively high humidity and burying environment facilitated straw decomposition [35]. Singh et al. [36] suggested that long-term no tillage enhanced POXC accumulation and macroaggregation compared to tilled treatments. However, our result showed that NT benefited POXC content in microaggregates, rather than in macroaggregates, yet POXC content might increase along the temporal extension under NT treatment.

4.3. Soil Quality Indicators

Soil structure is the basis for maintaining soil functions. As an important component of soil structure, the size distribution and stability of the aggregates affect the porosity, water holding capacity, permeability, and corrosion resistance of the soil [37]. MWD and GMD are the most widely used indicators for evaluating soil fertility and soil quality. In our study, MWD and GMD were strongly correlated with each other. However, the correlation among these two indices and measured SOC fractions, except for DOC, is not significant. However, \( D_m \) showed significantly negative relationship with MWD, GMD, SOC, DOC, POXC, and POC. It suggested that lower \( D_m \) can characterize not only higher soil aggregate stability, but also better soil fertility and stronger nutrient supply capacity, to a certain extent.

The changes in labile organic carbon fractions have been used as the indicators of soil carbon pool by many researchers. Awale et al. [13] suggested that POXC and DOC were the most sensitive indicators of tillage-induced changes in soil organic matter dynamics. Motta et al. [38] reported that the increase in SOC of topsoil induced by soil management was mainly associated with POC. Guo et al. [39] found that MBC and soil microbial community were affected by short-term conservation management practices, especially in soil aggregates with 2–1 mm sizes.
In our study, DOC, MBC, POXC, and POC had more sensitive responses than SOC. Of all labile carbon fractions, POXC and POC were the two fractions that showed the strongest relationship with SOC. Meanwhile, POXC can be measured relatively cheaply and quickly. In other studies, it was reported that POXC was not only positively correlated with SOC, but also found to be linked with various soil physical, chemical, and biological quality parameters such as soil bulk density, potassium, microbial biomass nitrogen, and so on [6,36]. Consequently, we suggested POXC to be the effective indicator of the early change in SOC pools after short-term tillage treatments.

5. Conclusions

This study focused on a comprehensive evaluation of SOC and its fractions in different particle sizes of aggregates in response to different tillage managements after maize harvest in the fluvo-aquic soil areas. Our result showed that tillage exerted more significant influence on changes of SOC and labile carbon fractions content in large size macroaggregates, while tillage had little impact on SOC and labile carbon fractions distribution modes in soil aggregates. Compared with RT treatment, NT treatment not only increased soil aggregate stability, but also enhanced SOC, DOC, and POC content, especially those in large size macroaggregates. DP treatment also showed positive effects on soil aggregate stability and labile carbon fractions (DOC and POXC). However, SS treatment did not show improvements in soil aggregate properties and organic carbon content in our study. Consequently, we suggest no tillage or deep ploughing, rather than rotary tillage, could be better tillage management considering carbon storage. Meanwhile, we implied that DM and POXC could be effective indicators of soil quality, as affected by tillage managements.

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References

1. Benbi, D.K.; Brar, K.; Toor, A.S.; Sharma, S. Sensitivity of Labile Soil Organic Carbon Pools to Long-Term Fertilizer, Straw and Manure Management in Rice-Wheat System. Pedosphere 2015, 25, 534–545. [CrossRef]
2. Zhang, X.W.; Han, X.Z.; Yu, W.T.; Wang, P.; Cheng, W.X. Priming Effects on Labile and Stable Soil Organic Carbon Decomposition: Pulse Dynamics over Two Years. PLoS ONE 2017, 12, e0184978. [CrossRef] [PubMed]
3. Bhattacharya, S.S.; Kim, K.H.; Das, S.; Uchimiya, M.; Jeon, B.H.; Kwon, E.; Szulejko, J.E. A Review on the Role of Organic Inputs in Maintaining the Soil Carbon Pool of the Terrestrial Ecosystem. J. Environ. Manag. 2016, 167, 214–227. [CrossRef] [PubMed]
4. Liu, M.; Yu, W.T.; Jiang, Z.S.; Ma, Q. A Research Review on Soil Active Organic Carbon. Chin. J. Ecol. 2006, 11, 1412–1417.
5. Jin, X.X.; Gall, A.R.; Saeed, M.F.; Li, S.Y.; Filley, T.; Wang, J.K. Plastic Film Mulching and Nitrogen Fertilization Enhance the Conversion of Newly-Added Maize Straw to Water-Soluble Organic Carbon. Soil Tillage Res. 2020, 197, 104527. [CrossRef]
6. Bongiorno, G.; Bunemann, E.K.; Oguejiofor, C.U.; Meier, J.; Gort, G.; Comans, R.; Mader, P.; Brussaard, L.; de Goede, R. Sensitivity of Labile Carbon Fractions to Tillage and Organic Matter Management and Their Potential as Comprehensive Soil Quality Indicators across Pedoclimatic Conditions in Europe. Ecol. Indic. 2019, 99, 38–50. [CrossRef]
7. Culman, S.W.; Snapp, S.S.; Freeman, M.A.; Schipanski, M.E.; Beniston, J.; Lal, R.; Drinkwater, L.E.; Franzluebbers, A.J.; Glover, J.D.; Grandy, A.S.; et al. Permanganate Oxidizable Carbon Reflects a Processed Soil Fraction That Is Sensitive to Management. Soil Sci. Soc. Am. J. 2012, 76, 494–504. [CrossRef]
8. Nandan, R.; Singh, V.; Singh, S.S.; Kumar, V.; Hazra, K.K.; Nath, C.P.; Poonia, S.; Malik, R.K.; Bhattacharyya, R.; McDonald, A. Impact of Conservation Tillage in Rice-Based Cropping Systems on Soil Aggregation, Carbon Pools and Nutrients. *Geoderma* 2019, 340, 104–114. [CrossRef]

9. Okolo, C.C.; Gebresamuel, G.; Zenebe, A.; Haile, M.; Eze, P.N. Accumulation of Organic Carbon in Various Soil Aggregate Sizes under Different Land Use Systems in a Semi-Arid Environment. *Agric. Ecosyst. Environ.* 2020, 297, 13. [CrossRef]

10. Guo, L.K.; Shen, J.; Li, B.; Li, Q.Q.; Wang, C.Q.; Guan, Y.; D’Acquii, L.P.; Luo, Y.L.; Tao, Q.; Xu, Q.; et al. Impacts of Agricultural Land Use Change on Soil Aggregate Stability and Physical Protection of Organic C. *Sci. Total Environ.* 2020, 707, 136049. [CrossRef]

11. Six, J.; Elliott, E.T.; Paustian, K. Soil Macromonograph Turnover and Microaggregate Formation: A Mechanism for C Sequestration under No-Tillage Agriculture. *Soil Biol. Biochem.* 2000, 32, 2099–2103. [CrossRef]

12. Zhu, C.W.; Long, Q.; Dong, S.G.; Shi, K.; Jiang, G.Y.; Li, X.L.; Zhang, C.Y.; Liu, F.; Shen, F.M.; Liu, S.L. Effects of Rotary and Deep Tillage Modes on Soil Microbial Biomass Carbon and Nitrogen and Enzyme Activities in Fluvo-Aquic Soil under Wheat-Maize Rotation System. *J. Plant Nutr. Fertil.* 2020, 26, 51–63. [CrossRef]

13. Kan, Z.R.; Virk, A.L.; He, C.; Liu, Q.Y.; Qi, J.Y.; Dang, Y.P.; Zhao, X.; Zhang, H.L. Characteristics of Carbon Mineralization and Accumulation under Long-Term Conservation Tillage. *Catena* 2020, 193, 104636. [CrossRef]

14. Awale, R.; Emeson, M.A.; Machado, S. Soil Organic Carbon Pools as Early Indicators for Soil Organic Matter Stock Changes under Different Tillage Practices in Inland Pacific Northwest. *Front. Ecol. Ecol.* 2017, 5, 96. [CrossRef]

15. Wang, Y.X.; Chen, S.P.; Zhang, D.X.; Yang, L.; Cui, T.; Jing, H.R.; Li, Y.H. Effects of Subsoiling Depth, Period Interval and Combined Tillage Practice on Soil Properties and Yield in the Huang-Huai-Hai Plain, China. *J. Integr. Agric.* 2020, 19, 1596–1608. [CrossRef]

16. Wang, H.B.; Bai, W.B.; Han, W.; Song, J.Q.; Lv, G.H. Effect of Subsoiling on Soil Properties and Winter Wheat Grain Yield. *Soil Use Manag.* 2019, 35, 643–652. [CrossRef]

17. Guo, Y.F.; Fan, R.Q.; Zhang, X.P.; Zhang, Y.; Wu, D.H.; McLaughlin, N.; Zhang, S.X.; Chen, X.W.; Jia, S.X.; Liang, A.Z. Tillage-Induced Effects on SOC Through Changes in Aggregate Stability and Soil Pore Structure. *Sci. Total Environ.* 2020, 703, 134617. [CrossRef]

18. Lewis, D.B.; Kaye, J.P.; Jabbour, R.; Barbercheck, M.E. Labile Carbon and Other Soil Quality Indicators in Two Tillage Systems during Transition to Organic Agriculture. *Renew. Agric. Food Syst.* 2011, 26, 342–353. [CrossRef]

19. Burdukovskii, M.; Kiseleva, I.; Perepelkina, P.; Kosheleva, Y. Impact of Different Fallow Durations on Soil Aggregate Structure and Humus Status Parameters. *Soil Sci. Soc. Am. J.* 2019, 83, 1541. [CrossRef]

20. Kan, Z.R.; Virk, A.L.; He, C.; Liu, Q.Y.; Qi, J.Y.; Dang, Y.P.; Zhao, X.; Zhang, H.L. Characteristics of Carbon Mineralization and Accumulation under Long-Term Conservation Tillage. *Catena* 2020, 193, 104636. [CrossRef]

21. Kan, Z.R.; Virk, A.L.; He, C.; Liu, Q.Y.; Qi, J.Y.; Dang, Y.P.; Zhao, X.; Zhang, H.L. Characteristics of Carbon Mineralization and Accumulation under Long-Term Conservation Tillage. *Catena* 2020, 193, 104636. [CrossRef]

22. Lewis, D.B.; Kaye, J.P.; Jabbour, R.; Barbercheck, M.E. Labile Carbon and Other Soil Quality Indicators in Two Tillage Systems during Transition to Organic Agriculture. *Renew. Agric. Food Syst.* 2011, 26, 342–353. [CrossRef]

23. Burdukovskii, M.; Kiseleva, I.; Perepelkina, P.; Kosheleva, Y. Impact of Different Fallow Durations on Soil Aggregate Structure and Humus Status Parameters. *Soil Sci. Soc. Am. J.* 2019, 83, 1541. [CrossRef]

24. Wang, Y.X.; Chen, S.P.; Zhang, D.X.; Yang, L.; Cui, T.; Jing, H.R.; Li, Y.H. Effects of Subsoiling Depth, Period Interval and Combined Tillage Practice on Soil Properties and Yield in the Huang-Huai-Hai Plain, China. *J. Integr. Agric.* 2020, 19, 1596–1608. [CrossRef]

25. Wang, H.B.; Bai, W.B.; Han, W.; Song, J.Q.; Lv, G.H. Effect of Subsoiling on Soil Properties and Winter Wheat Grain Yield. *Soil Use Manag.* 2019, 35, 643–652. [CrossRef]

26. Wang, H.B.; Bai, W.B.; Han, W.; Song, J.Q.; Lv, G.H. Effect of Subsoiling on Soil Properties and Winter Wheat Grain Yield. *Soil Use Manag.* 2019, 35, 643–652. [CrossRef]

27. Guo, Y.F.; Fan, R.Q.; Zhang, X.P.; Zhang, Y.; Wu, D.H.; McLaughlin, N.; Zhang, S.X.; Chen, X.W.; Jia, S.X.; Liang, A.Z. Tillage-Induced Effects on SOC Through Changes in Aggregate Stability and Soil Pore Structure. *Sci. Total Environ.* 2020, 703, 134617. [CrossRef]

28. Lewis, D.B.; Kaye, J.P.; Jabbour, R.; Barbercheck, M.E. Labile Carbon and Other Soil Quality Indicators in Two Tillage Systems during Transition to Organic Agriculture. *Renew. Agric. Food Syst.* 2011, 26, 342–353. [CrossRef]

29. Burdukovskii, M.; Kiseleva, I.; Perepelkina, P.; Kosheleva, Y. Impact of Different Fallow Durations on Soil Aggregate Structure and Humus Status Parameters. *Soil Sci. Soc. Am. J.* 2019, 83, 1541. [CrossRef]

30. Kan, Z.R.; Virk, A.L.; He, C.; Liu, Q.Y.; Qi, J.Y.; Dang, Y.P.; Zhao, X.; Zhang, H.L. Characteristics of Carbon Mineralization and Accumulation under Long-Term Conservation Tillage. *Catena* 2020, 193, 104636. [CrossRef]

31. Kan, Z.R.; Virk, A.L.; He, C.; Liu, Q.Y.; Qi, J.Y.; Dang, Y.P.; Zhao, X.; Zhang, H.L. Characteristics of Carbon Mineralization and Accumulation under Long-Term Conservation Tillage. *Catena* 2020, 193, 104636. [CrossRef]

32. Lewis, D.B.; Kaye, J.P.; Jabbour, R.; Barbercheck, M.E. Labile Carbon and Other Soil Quality Indicators in Two Tillage Systems during Transition to Organic Agriculture. *Renew. Agric. Food Syst.* 2011, 26, 342–353. [CrossRef]

33. Burdukovskii, M.; Kiseleva, I.; Perepelkina, P.; Kosheleva, Y. Impact of Different Fallow Durations on Soil Aggregate Structure and Humus Status Parameters. *Soil Sci. Soc. Am. J.* 2019, 83, 1541. [CrossRef]

34. Wang, Y.X.; Chen, S.P.; Zhang, D.X.; Yang, L.; Cui, T.; Jing, H.R.; Li, Y.H. Effects of Subsoiling Depth, Period Interval and Combined Tillage Practice on Soil Properties and Yield in the Huang-Huai-Hai Plain, China. *J. Integr. Agric.* 2020, 19, 1596–1608. [CrossRef]

35. Wang, H.B.; Bai, W.B.; Han, W.; Song, J.Q.; Lv, G.H. Effect of Subsoiling on Soil Properties and Winter Wheat Grain Yield. *Soil Use Manag.* 2019, 35, 643–652. [CrossRef]

36. Guo, Y.F.; Fan, R.Q.; Zhang, X.P.; Zhang, Y.; Wu, D.H.; McLaughlin, N.; Zhang, S.X.; Chen, X.W.; Jia, S.X.; Liang, A.Z. Tillage-Induced Effects on SOC Through Changes in Aggregate Stability and Soil Pore Structure. *Sci. Total Environ.* 2020, 703, 134617. [CrossRef]

37. Lewis, D.B.; Kaye, J.P.; Jabbour, R.; Barbercheck, M.E. Labile Carbon and Other Soil Quality Indicators in Two Tillage Systems during Transition to Organic Agriculture. *Renew. Agric. Food Syst.* 2011, 26, 342–353. [CrossRef]

38. Burdukovskii, M.; Kiseleva, I.; Perepelkina, P.; Kosheleva, Y. Impact of Different Fallow Durations on Soil Aggregate Structure and Humus Status Parameters. *Soil Sci. Soc. Am. J.* 2019, 83, 1541. [CrossRef]
33. Zhong, S.; Zeng, H.C. Influence of Long-Term Tillage and Residue Management on Soil Biota in Tropical Climate. *J. Biobased Mater. Bioenergy* 2019, 13, 576–584. [CrossRef]

34. Xue, J.F.; Pu, C.; Zhao, X.; Wei, Y.H.; Zhai, Y.L.; Zhang, X.Q.; Lal, R.; Zhang, H.L. Changes in Soil Organic Carbon Fractions in Response to Different Tillage Practices under a Wheat-Maize Double Cropping System. *Land Degrad. Dev.* 2018, 29, 1555–1564. [CrossRef]

35. Guo, Y.F.; Liang, A.Z.; Zhang, Y.; Zhang, S.X.; Chen, X.W.; Jia, S.X.; Zhang, X.P.; Wu, D.H. Evaluating the Contributions of Earthworms to Soil Organic Carbon Decomposition under Different Tillage Practices Combined with Straw Additions. *Ecol. Indic.* 2019, 105, 516–524. [CrossRef]

36. Singh, S.; Nouri, A.; Singh, S.; Anapalli, S.; Lee, J.; Arelli, P.; Jagadamma, S. Soil Organic Carbon and Aggregation in Response to Thirty-Nine Years of Tillage Management in the Southeastern US. *Soil Tillage Res.* 2020, 197, 104523. [CrossRef]

37. Liu, M.; Han, G.L.; Zhang, Q. Effects of Soil Aggregate Stability on Soil Organic Carbon and Nitrogen under Land Use Change in an Erodible Region in Southwest China. *Int. J. Environ. Res. Public Health* 2019, 16, 3809. [CrossRef]

38. Motta, A.C.V.; Reeves, D.W.; Burmester, C.; Feng, Y. Conservation Tillage, Rotations, and Cover Crop Affecting Soil Quality in the Tennessee Valley: Particulate Organic Matter, Organic Matter, and Microbial Biomass. *Commun. Soil Sci. Plant Anal.* 2007, 38, 2831–2847. [CrossRef]

39. Guo, L.J.; Zhang, Z.S.; Wang, D.D.; Li, C.F.; Cao, C.G. Effects of Short-Term Conservation Management Practices on Soil Organic Carbon Fractions and Microbial Community Composition under a Rice-Wheat Rotation System. *Biol. Fertil. Soils* 2015, 51, 65–75. [CrossRef]