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Conservation tillage enhances the sequestration and iron-mediated stabilization of aggregate-associated organic carbon in Mollisols

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ABSTRACT

Conservation tillage practices, which involve minimal or no soil disturbance and crop residue retention, are known to help preserve soil organic carbon (SOC). However, the mechanisms underlying the minerals-mediated chemical and physical stabilization of SOC remain unclear. Here, a long-term field experiment was initiated in 2012 to investigate the effects of tillage managements on the contents and chemical composition of SOC density fractions, iron oxides transformation and aggregate stability in Mollisols. We utilized three treatments: conventional tillage (CT) without crop residue, reduced tillage (RT) and no tillage (NT) with straw mulching. Compared to CT, RT and NT significantly increased SOC content by 13.6 % and 17.9 % in the 0–20 cm soil layer due to an increase in the aromatic compound contents. Furthermore, NT and RT increased the mean weight diameter by 17.7 % and 10.7 %, respectively, indicating increased aggregate stability compared to CT. Additionally, the contents of amorphous iron oxides (Feo) and complex iron oxides (Fep) increased under NT and RT by 10.6–14.4 % and 12.7–41.1 %, respectively, in bulk soil and silt + clay fractions within macroaggregates (>0.25 mm). The contents of Feo and Fep were strongly positively correlated with aggregate stability ($p < 0.001$, $r^2 = 0.64$), and promote the physical protection of SOC. Both NT and RT enhanced the aromatic-C content and aromatic-C/aliphatic-C ratio by 13.6 %–24.6 % and 16.5 %–38.9 % in macroaggregates compared with CT. Moreover, the aromatic-C/aliphatic-C ratio increased with increasing Feo plus Fep contents ($p < 0.001$, $r^2 = 0.73$), which led to an increased in the recalcitrant-C proportion, and this shift was benefit for the accumulation of mineral-associated OC. These results indicate that long-term conservation tillage can augment the accessibility of Fe for binding C, possibly by forming organo-Fe complexes, which subsequently improve soil aggregation, and thus promoted the chemical stability and long-term sequestration of SOC in Mollisols of Northeast China.

1. Introduction

Soil is the largest terrestrial carbon (C) pool, holding about three times more C than atmosphere (Wei et al., 2022). Soil organic carbon (SOC), a key indicator of soil quality, is crucially important for crop productivity, soil fertility, and ecological and environmental sustainability (Peixoto et al., 2020). Previous studies have shown that SOC stabilization depends on the comminated effect of physical and chemical protection mechanisms, rather than biological fixation alone (Liu et al., 2022; Plaza et al., 2013). Therefore, sustainable land stewardship will require a holistic understanding of the impact of organo-mineral interactions and soil aggregation in long-term SOC storage.

In contrast to intensive tillage, conservation agricultural practices have been shown to increase SOC sequestration by improving aggregate stability, promoting the biochemical fixation of organic matter, and limiting nutrient loss (Fuentes et al., 2011; Xue et al., 2019a). For example, no tillage (NT) or reduced tillage (RT) practices, when coupled with straw addition, will improve soil aggregation. This is achieved by reducing surface disturbance and preserving cementing agents, including animal and plant residues (Jat et al., 2019). These cementing

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agents combine with primary particles to form macroaggregates, protecting SOC from microbial and enzymatic degradation and oxidation processes (Ma et al., 2019). Moreover, conservation tillage practices was proven to improve the combination of soil minerals with organic matter under changed land microenvironment (Denef et al., 2004; Six et al., 1999). Practices such as NT encourage the development of minerals with a short-range ordered (SRO) structure due to increases in organic matter content, organic carbon (OC) lability, soil moisture, and microbial activity (Inda et al., 2013). Highly reactive SRO minerals preferentially associate with complex substances, which subsequently attached to the aggregate surfaces and improve soil aggregation (Adhikari and Yang, 2015). Moreover, soil minerals promote SOC sequestration by directly enhancing aggregate-associated oOC stability (Plaza et al., 2013). According to the conceptual model by Six et al., (2000), plant litter within macroaggregates undergoes selective decomposition, and leaves the more chemically recalcitrant structural materials to be coated with microbial byproducts and mineral particles, resulting in the formation of stable microaggregates. Conservation tillage reduced the aggregate turnover rate and thereby promotes microaggregates within macroaggregates, in where organo-mineral complexes are stabilized. However, less is known about how different tillage systems affect the mutual transformation of different types of minerals or their effect on SOC stability and sequestration.

As one of the most abundant transition metal oxides, iron (Fe) plays an important role in SOC storage by forming mineral-associated OC through adsorption or co-precipitation (Barberis et al., 1991). Specially, Fe chemically stabilizes SOC and forms strong organo-Fe complex by binding organic molecules via ligand exchange or multivalent cation bridging (Chen et al., 2022a; Qi et al., 2021). In addition, amorphous iron oxides (Feo) can facilitate the formation of stable macroaggregates by acting as inorganic binding agents between primary and secondary particles (Baalousha, 2009). Recently, Huang et al.(2016) stated that straw return increased the contents of Feo and complex iron oxides (Fep), and their associated with small particles to promote soil aggregation. Fe oxides are sensitive to both oxygen and moisture, particularly in altered soil structures under different tillage practices (Moens et al., 2021). Conservation tillage alters soil redox environment due to increased organic matter addition, changed soil pore structure and soil moisture (Inda et al., 2013).Under these conditions, Fe may associate with OC directly through adsorption or indirectly by acting as a flocculant (Xue et al., 2019b). However, the biochemical transformation of Fe in hierarchical aggregates and its contributions to aggregate stability and SOC persistence under different tillage practices requires further study.

The residence time of SOC is affected by its inherent biological stability and chemical composition (Wu et al., 2021). Generally, aromatic compounds and O/N-alkyl-C are considered resistant to biodegradation, while unsaturated aliphatic compounds and alkyl-C are considered easily decomposed by microorganisms (Luan et al., 2019). During the soil aggregation process, partially oxidized aromatic compounds participate in the formation of highly resilient organo-mineral associations (Wu et al., 2021). These hydrophobic substances stabilize aggregates by reducing water infiltration capacity (Urbanek et al., 2007). The formation of organo-mineral complexes may be driven by biochemical interactions between the microorganism and organic material (Kirsten et al., 2021). Fe oxides may affect the action of different OC functional groups. For instance, OC functional groups rich in oxygen (O) and nitrogen (N) functionalities are preferentially targeted by reactive Fe minerals (Lalonde et al., 2012). In addition, amino and carboxylic groups functional groups form strong complexes with Fe oxides through ligand exchange reactions (Chen et al., 2022a). In rice–wheat cropping systems, Fe oxides were found to protect aromatic compounds at the redox interface (Liu et al., 2021). Therefore, the aggregate distribution of various SOC chemical components and their impact on organic-Fe complexes require further study to better understand SOC stability under conservation tillage regimes.

Approximately 40 % of Chinese maize is grown in northeastern Chinese Mollisol soils, which are rich in humus and Fe minerals (Abrar et al., 2020). Soil aggregate stability, SOC storage and Fe oxide levels demonstrate sensitivity to seasonal variations, both dry and wet, particularly in altered soil structures modified by different tillage practices (Jeewani et al., 2021). The soils of this region experience alternating wet-dry conditions and freeze–thaw cycles due to irregular annual rainfall distribution and sub-zero wintertime temperatures. These conditions are expected to intensify due to climate change, resulting in fluctuations in the soil redox environment and the valence state of iron oxides.

Although Fe oxides are well known to promote aggregate stability and SOC preservation, our understanding of the chemical protection mechanisms of organo-mineral complexes under different tillage systems is still limited. The aims of this study were to: (i) evaluate the effect of conservation tillage on SOC sequestration; (ii) explore the role of Fe oxides in the aggregate stability and its association with the chemical composition of aggregate-associated C; and (iii) clarify the contribution of organic-Fe complexes and soil aggregation on the stabilization of SOC in Mollisols. We hypothesized that that reactive Fe oxides and their association with SOC compounds, enhances aggregate stability and further produced recalcitrant compounds within aggregate-associated carbon under conservation tillage. Our result suggest that long-term conservation tillage increases the sequestration and chemical stability of SOC by enhancing organo-Fe associations in Mollisols.

2. Material and methods

2.1. Study site and experimental design

The long-term experiment was begun in 2012 at the Observation Station (44°00′N, 125°24′E) of Northeast Institute of Geography and Agroecology, Chinese Academy of Sciences, in Changchun County, Jilin Province, China. This study site falls within the North Temperate Zone and is characterized by a continental monsoon climate. The region has an average annual temperature of 4.4 °C, and receives a mean annual precipitation is 614 mm, primarily during the June to September period. The experimental site contains clay loam texture, classified as Mollisols (Typic Hapludoll, USDA Soil Taxonomy), with a pH, total carbon, and total nitrogen of 7.1, 19.23 g kg^{-1}, and 1.63 g kg^{-1} at the 0–20 cm soil layer, respectively. The experimental field is flat ($<1\%$ slope) and has sustained agricultural cultivation for over a century, predominantly with continuous maize cropping.

The experiment employed a completely randomized design with four replications for each treatment. The dimensions of each plot were 40 m in length and 8.4 m in width, with rows spaced at intervals of 0.65 m. Three tillage practices were studied: a) CT: conventional tillage without crop residue; b) RT: reduced tillage with straw mulching; and c) NT: no tillage with straw mulching. In the CT experiment, ridge construction occurred in June using a modified lister implement, followed by moldboard plowing (at a soil depth of 20 cm) after the maize harvest each autumn, with subsequent removal of crop residues. In the RT experiment, approximately 30 cm of stubble was intentionally retained, and all unchopped maize stalks were left in the field as mulch after each autumn harvest. The soil remained undisturbed from the autumn harvest until the subsequent spring sowing period, except for sowing and ridge construction in June. In the NT experiment, approximately 30 cm of stubble is intentionally retained, and all crop residues was returned to the field as mulch. No tillage was conducted though the year, and a no-till planter (KINZE-3000NT, Williamsburg, Iowa) was used for seeding in the subsequent spring sowing. Fertilizers were applied at a rate of 150 kg ha^{-1}N (urea), 45.5 kg ha^{-1}P (diammonium phosphate), and 78 kg ha^{-1} K (potassium sulfate). The N was applied as a base and topdressing, while the P and K were only applied during seeding.

2.2. Soil sampling

Soil sampling was conducted during maize harvest stage in September 2020. Using the soil-drilling technique, four soil samples were randomly extracted from each plot and then mixed to accurately represent soil depths of 0–10 cm and 10–20 cm. These samples were collected from the central homogenous portion of each layer and divided into three portions. Two portions were air-dried, ground, and sieved through different screens. Following the methodology outlined by Steffens et al. (2010), one portion of the soil was homogenized and broken into small clumps, passing through an 8 mm sieve for aggregate separation. Another portion was ground to pass through a 0.15 mm sieve for measuring SOC and total nitrogen content. The remaining samples were manually separated, passing through a 5 mm sieve, and stored at 4 °C for subsequent determination of microbial biomass carbon (MBC) and nitrogen (MBN), and soil chemical properties.

2.3. Soil fraction analysis

Total soil carbon (TOC) and nitrogen was determined by an element analyzer (Vario TOC, Elementar, Germany). Given the typically low minimal presence of inorganic carbon in Mollisols (Wissing et al., 2013), the TOC content was deemed equivalent to the SOC content. MBC and MBN were assessed through fumigation-extraction, and their values were calculated utilizing conversion factors of 0.45 for MBC and 0.54 for MBN as outlined by Zheng et al. (2022). The contents of ammonium nitrogen (NH_4^+-N) and nitrate nitrogen (NO_3^--N) were extracted using a 2.0 M KCl solution at a ratio of 1:5 (w/v) and quantified via a continuous flow Autoanalyzer (Autoanalyzer 3, SEAL, Germany) described by Plaza et al. (2013).

The bulk soil refers to the undivided soil before being categorized. Soil aggregates were hierarchically separated using a three-step fractionation initially proposed by Tisdall and OADES (1982), with slightly modified according to requirements (Fig. 1). Briefly, fresh soil (<8 mm) was dispersed in deionized water following sieving into macroaggregates (>0.25 mm), free microaggregates (0.25–0.053 mm), and non-aggregated silt + clay fractions (<0.053 mm). Then, these fractions were dried at 60 °C and weighed for analyses. Next, the macroaggregates were further sonicated by a Sonifier with a 10-mm tip (Ultrasonic pulverizer, SCIENTZ, China), and were wet-sieved again with 0.25-mm and 0.053-mm sieves. Different fractions inside macroaggregates were divided into coarse POM [M(c)POM] at 0.25-mm sieves, microaggregates (mM) at 0.053-mm sieves, and the retained silt + clay (M−silt + clay) fractions (<0.053 mm). Finally, the mM fractions were suspended in NaI solution (1.8 g cm^{-3}), the light fractions of fine POM inside macroaggregates [M(f)POM] was isolated through centrifugation. The remaining heavy fraction passed through a 0.053-mm sieve to divided into POM (>0.053 mm) and silt + clay fractions (<0.053 mm) within mM.

2.4. Mean weight diameter determination

The mean weight diameter (MWD) acts as a gauge of aggregate stability and is calculated using the following equation (Blanco-Canqui et al., 2014).

$$\mathrm{MWD} = \frac{\sum_{i=1}^{n}(W_i \times X_I)}{\sum_{i=1}^{n} W_i}$$

where W_i represents the percentage weight content (%) and X_i (mm) stands for the mean diameter of soil aggregates at different particle sizes.

2.5. Fourier-transform infrared spectroscopy

The OC functional group composition of soil samples was measured using a Fourier transform infrared (FTIR) spectrometer (Nicolet IS10, Thermo Fisher). Prior to analysis, the soil sample was oven-dried (65 °C) for 5 h and ground using a 100-mesh sieve. Subsequently, 1 mg of the

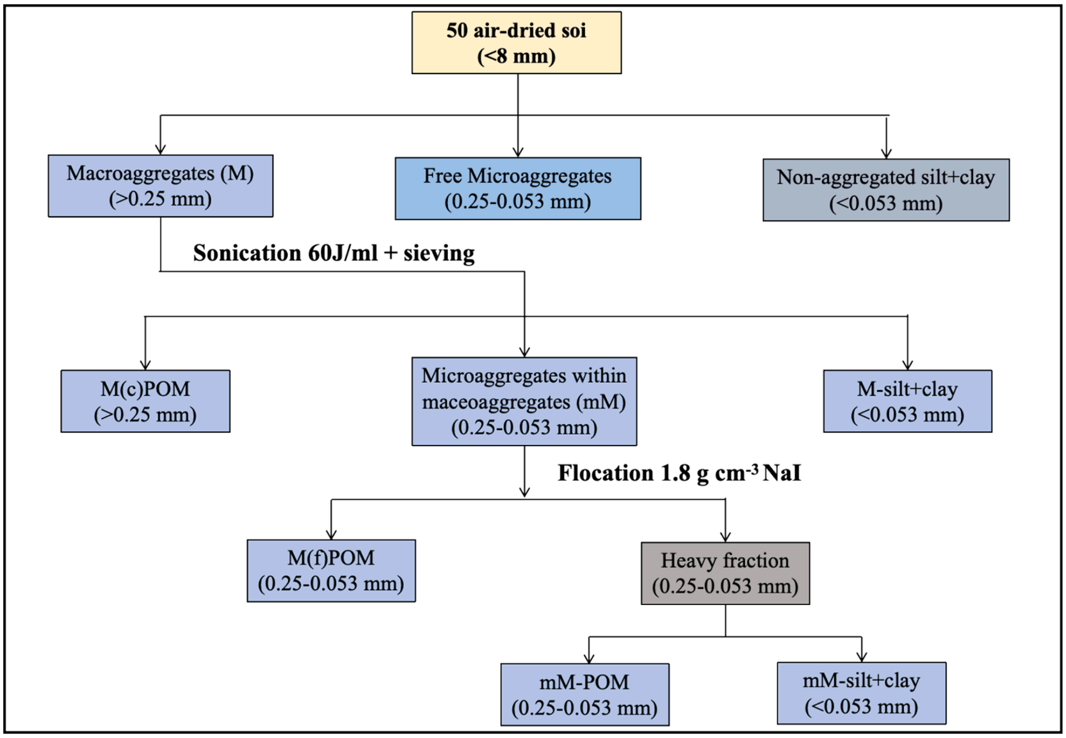

Fig. 1. Three-step fractionation scheme to separate different size classes of soil aggregates. POM: particulate organic matter; M(c)POM: coarse POM in the macroaggregates but outside microaggregates; mM: the microaggregates within macroaggregates; M−silt + clay: silt and clay fractions within macroaggregates; M(f) POM: free POM inside macroaggregates but outside microaggregate; mM-POM: POM inside the mM fractions; mM-silt + clay: silt and clay fractions inside the mM fractions. NaI: sodiumiodide.

ground sample was mixed with 100 mg of potassium bromide particles. Spectral recording was conducted with an average of 32 scans, using a wavelength resolution of 4 cm^{-1} and a range of 200–4000 cm^{-1}. The peak area tool of OMNIC was used to conduct semi-quantitative analyses of the collected spectra and compare the relative intensity of each infrared peak. All FTIR spectra were visualized using Origin 2019.

2.6. Iron oxides and iron-bound organic carbon content analyses

The reactive Fe (Fed) was extracted from bulk soil and different size class of aggregate samples using the dithionite-citrate-bicarbonate method (Inda et al., 2013). The non-crystalline Fe (Feo) fraction was extracted using the ammonium oxalate method (Barberis et al., 1991), while the complexed Fe (Fep) fraction was extracted using sodium pyrophosphate at pH 8.5 (Saidy et al., 2012). Subsequently, all extracted Fe oxides were then diluted, and their concentrations were determined using inductive coupled plasma mass spectrometer (ICP-MS, Model 7700ce, USA). The analysis of Fe-bound OC content was performed using the method by Jia et al. (2022) with minor modifications. Typically, 1 g of dry soil was weighed, mixed with a total of 22.5 ml of buffer solution (pH 7.3), composed of 20 ml of 0.3 M trisodium citrate and 2.5 ml of 1 M sodium bicarbonate, the mixture was introduced into a 50 ml centrifuge tube and heated to 80 °C using a water bath. Subsequently, 0.50 g of sodium dithionite, serving as the reducing agent, was added to the mixture, which was then held at 80 °C for a duration of 15 min. Simultaneously, an equivalent ionic strength control experiment was conducted by dissolving another 0.5 g of dried soil samples using sodium chloride (NaCl). All residues underwent four rinses with 10 ml of 1 M NaCl solution, dried at 60 ℃, and subsequently underwent elemental analysis (Vario TOC, Elementar, Germany).

The concentration and proportion of Fe-bound OC was calculated according to the following equations:

$$\mathrm{Fe-bound\,OC} = \mathrm{OC_{NaCl}} - \mathrm{OC_{sodium\ pyrophosphate}}$$

$$f_{\mathrm{Fe-bound\,OC}}\,(\%) = \mathrm{Fe-bound\,OC/TOC} \times 100$$

Here, $\mathrm{OC_{NaCl}}$ and $\mathrm{OC_{sodium\ pyrophosphate}}$ refer to the SOC within soil residues treated with NaCl and sodium pyrophosphate, respectively.

2.7. Statistical analysis

A one-way analysis of variance (ANOVA) coupled with a least-significant difference (LSD) test was performed to determine statistically significant ($p < 0.05$) differences in OC, Fe oxide, and Fe-bound OC contents in bulk soil and different aggregate size classes using SPSS version 22 (IBM, US). The Shapiro-Wilk assessment was used to evaluate the normality of variance prior to carrying out ANOVA. A Pearson correlation analysis was conducted to investigate the relationships among soil chemical composition, Fe oxides, and Fe-bound OC contents. Furthermore, redundancy analysis (RDA) was performed using Origin 2019 to examine the relationships between soil physical and chemical properties, Fe oxides, and OC functional groups in bulk soil and aggregates. Structural equation modeling (SEM) was performed to reveal the intricate regulatory mechanisms of various factors on aggrege stability under different treatments using the R packages *piecewiseSEM and lme4*.

3. Result

3.1. Soil physicochemical properties

Conservation tillage generally improved the soil physical and chemical properties (Table 1). Specifically, both RT and NT resulted in higher SOC content compared to CT, with NT showing the greatest increase (by 29.6 %) in the 0 − 10 cm layer. At the soil depth of 10 − 20 cm, only RT had significantly higher SOC content. The TN content exhibited a similar trend to SOC across different tillage practices and soil depths. Across both tested soil depths, NT and RT increased the content of microbial biomass C (MBC) by 11.5 %–37.9 % and the content of microbial biomass N (MBN) by 12.6 %–36.9 % compared to CT. Soil ammonium (NH_4^+-N) concentration varied significantly among tillage practices and was enhanced by 16.9 %–38.2 % under NT compared to CT. Furthermore, RT significantly raised soil nitrate (NO_3^--N) concentration relative to CT at both soil depths.

Table 1
Soil physical and chemical properties under three tillage practices.

Soil Depth	Tillage	TOC	TN	MBC	MBN	NO_3^--N	NH_4^+-N
		g kg^{-1}	g kg^{-1}	mg kg^{-1}	mg kg^{-1}	mg kg^{-1}	mg kg^{-1}
0–10 cm	CT	16.5 ± 0.6c	1.38 ± 0.11c	305.1 ± 10.3c	38.5 ± 1.6c	0.74 ± 0.04b	7.64 ± 0.23b
	RT	19.6 ± 0.7b	1.51 ± 0.10b	383.2 ± 7.7b	43.3 ± 1.7b	1.16 ± 0.58 a	8.72 ± 0.34b
	NT	21.4 ± 0.8 a	1.65 ± 0.09 a	420.8 ± 9.3 a	52.7 ± 2.1 a	0.56 ± 0.02b	10.56 ± 0.58 a
10–20 cm	CT	14.7 ± 0.6b	1.29 ± 0.08b	284.9 ± 11.2b	32.5 ± 1.8c	0.54 ± 0.01b	7.27 ± 0.32b
	RT	15.8 ± 0.8 a	1.45 ± 0.11 a	332.7 ± 12.5 a	42.3 ± 2.3 a	0.69 ± 0.03 a	8.05 ± 0.56 ab ababab
	NT	15.4 ± 0.4 ab	1.38 ± 0.09 ab	317.6 ± 15.4 a	37.8 ± 2.5b	0.44 ± 0.02b	8.94 ± 0.61 a

Note: Data represent the mean ± standard error; n = 4. Different lowercase letters indicate statistically significant ($p < 0.05$) differences among the three treatments at the same soil depth. NT: no-tillage. RT: reduced tillage. CT: conventional tillage. TOC: total soil carbon; TN: total soil nitrogen; $NH4^+$-N: ammonium nitrogen; NO_3^--N: nitrate nitrogen; MBC: microbial biomass carbon; MBN: microbial biomass nitrogen.

3.2. Organic carbon distribution in aggregates

Across all three tillage practices, the highest OC content was observed in macroaggregates (13.1–24.9 g kg^{-1}), followed by free microaggregates (12.1–23.2 g kg^{-1}), with the lowest OC content noted in non-aggregated silt + clay fractions (10.7–16.4 g kg^{-1}) (Table S1). In the 0 − 10 cm soil depths, NT resulted in the macroaggregate-associated OC content increased by 10.6 % and 41.2 % in relative to RT and CT, respectively (Fig. 2). Furthermore, both NT and RT significantly increased the OC content within macroaggregates compared with CT (Fig. 2 a). However, a noteworthy reduction in the OC content within the free microaggregates was observed under NT (by 13.1 %–13.9 %) and RT (by 8.5 %–36.0 %) compared to CT at both soil depths (Fig. 2 b). Similarly, the SOC content in non-aggregated silt + clay decreased by 16.5 %–21.7 % under NT and RT compared to CT (Fig. 2 c).

Among the different fractions within macroaggregates, both the NT and RT significantly increased the OC contents in the M(c)POM (by 13.9 %–63.8 %), M−silt + clay (by 28.0 %–55.9 %), and M(f)POM (by 33.2 %–64.2 %) fractions compared to CT at both soil depths (Fig. 2 d-g). Moreover, NT significantly increased the OC content in the mM-silt + clay fractions by 54.6 % compared to CT at the 0–10 cm layer (Fig. 2 i). However, neither tillage practices nor soil depth exerted a significant influence on OC contents in the mM-POM fractions (Fig. 2 h).

3.3. Size distribution and stability of soil aggregates

Soil contained more macroaggregates as tillage intensity was gradually reduced (Fig. 3 A). Compared with CT, both NT and RT increased the proportion of macroaggregates by 11.6 %–13.1 % and 8.4 %–8.6 %, respectively, at both soil depths. On the contrary, the percentage of non-

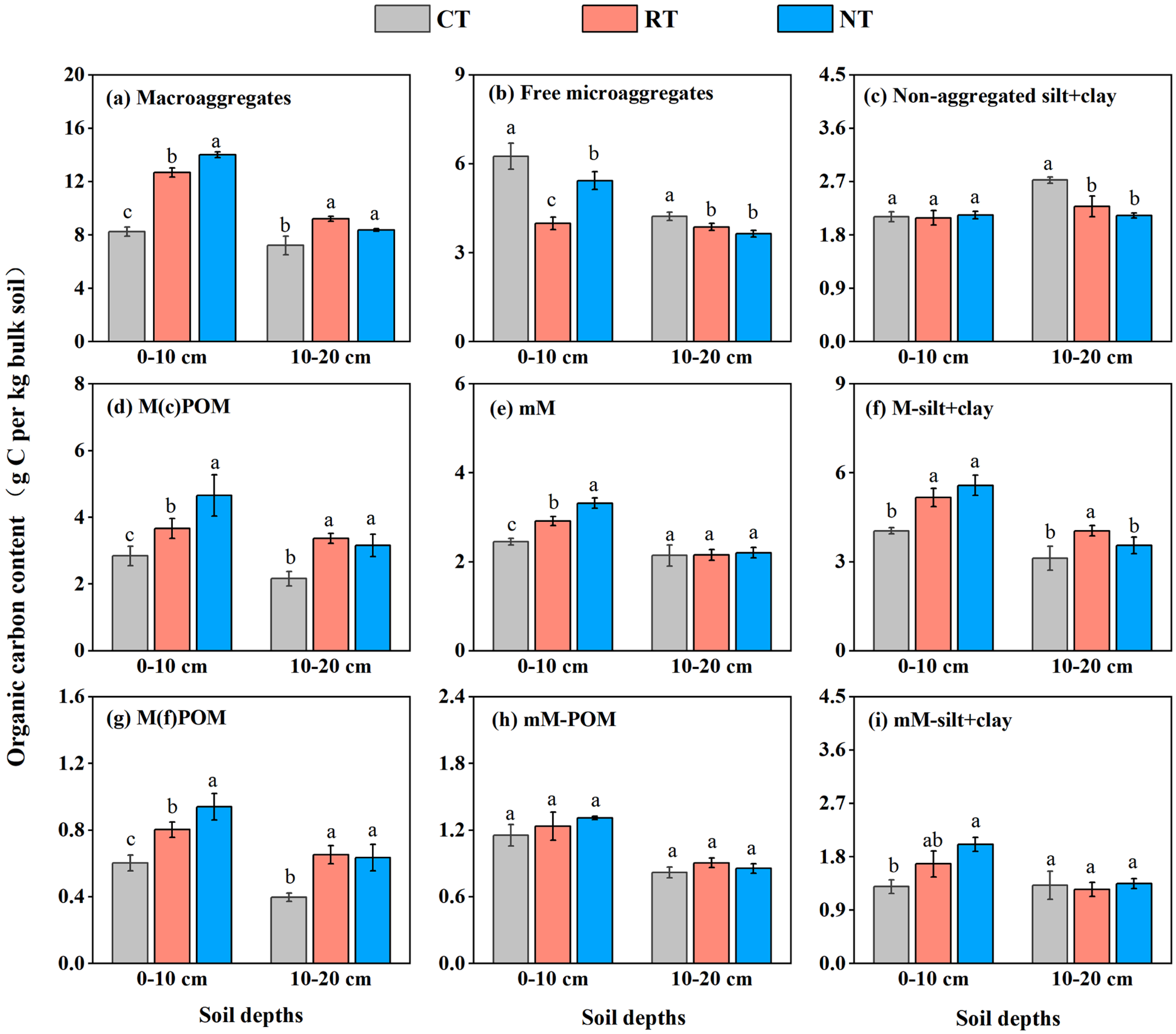

Fig. 2. Effects of different tillage practices on organic carbon content in aggregates of bulk soils from the 0–10 cm and 10–20 cm soil layers. Different lowercase letters indicate statistically significant ($p < 0.05$) differences among the treatments. Bars represent the standard errors (n = 4). POM: particulate organic matter; M(c) POM: coarse POM in the macroaggregates but outside microaggregates; mM: the microaggregates within macroaggregates; M−silt + clay: silt and clay fractions within macroaggregates; M(f)POM: free POM inside macroaggregates but outside microaggregate; mM-POM: POM inside the mM fractions; mM-silt + clay: silt and clay fractions inside the mM fractions. CT: conventional tillage; RT: reduced tillage; NT: no tillage.

aggregated silt + clay under NT and RT was 10.7 %–17.7 % lower than CT. Moreover, the proportion of free microaggregates was reduced under NT by 9.1 %–13.8 % relative to CT.

Within the macroaggregates (Fig. 3 B), the content of M(c)POM and M−silt + clay fractions were increased by 8.4 %–22.3 % under RT and NT compared to CT. Furthermore, both NT and RT increased the proportion of the M(f)POM and mM-silt + clay fractions by 10.9 %–32.8 % in the 0 − 10 cm layer, while the proportion of M(f) POM was significantly increased only under RT in the 10 − 20 cm layer (Fig. 3 C). In addition, the MWD values were 27.1 % and 13.9 % higher under NT and RT that CT, respectively, at the 0 − 10 cm soil depth, whereas only NT resulted in significantly higher MWD compared to CT at the 10–20 cm soil depths (Fig. 3 D).

3.4. *Iron oxides content in bulk soil and different aggregates*

The soil Fed content was significantly influenced by different tillage practices (Fig. 4). In general, CT resulted in the highest Fed content in bulk soil and different size fractions within macroaggregates. In the 0–10 cm soil layer, both RT and NT significantly reduced the Fed content by 7.7 %–16.5 % in bulk soil, M−silt + clay and mM-silt + clay fractions compared to CT (Fig. 4 A and B). Moreover, the Fed content was 7.8 %–20.5 % lower under NT and RT than under CT in macroaggregates, free microaggregates, mM, mM-POM, and mM-silt + clay fractions in the 10–20 cm soil layer (Fig. 4 C and D). In contrast, both RT and NT significantly increased the Fed content by 29.3 %–33.2 % in the free microaggregates at the 0–10 cm soil depth (Fig. 4 A). Furthermore, RT increased the Fed content by 11.3 % in the non-aggregated silt + clay fractions at the 10–20 cm soil depth (Fig. 4 C).

The Feo content was higher in the silt + clay fractions than in the

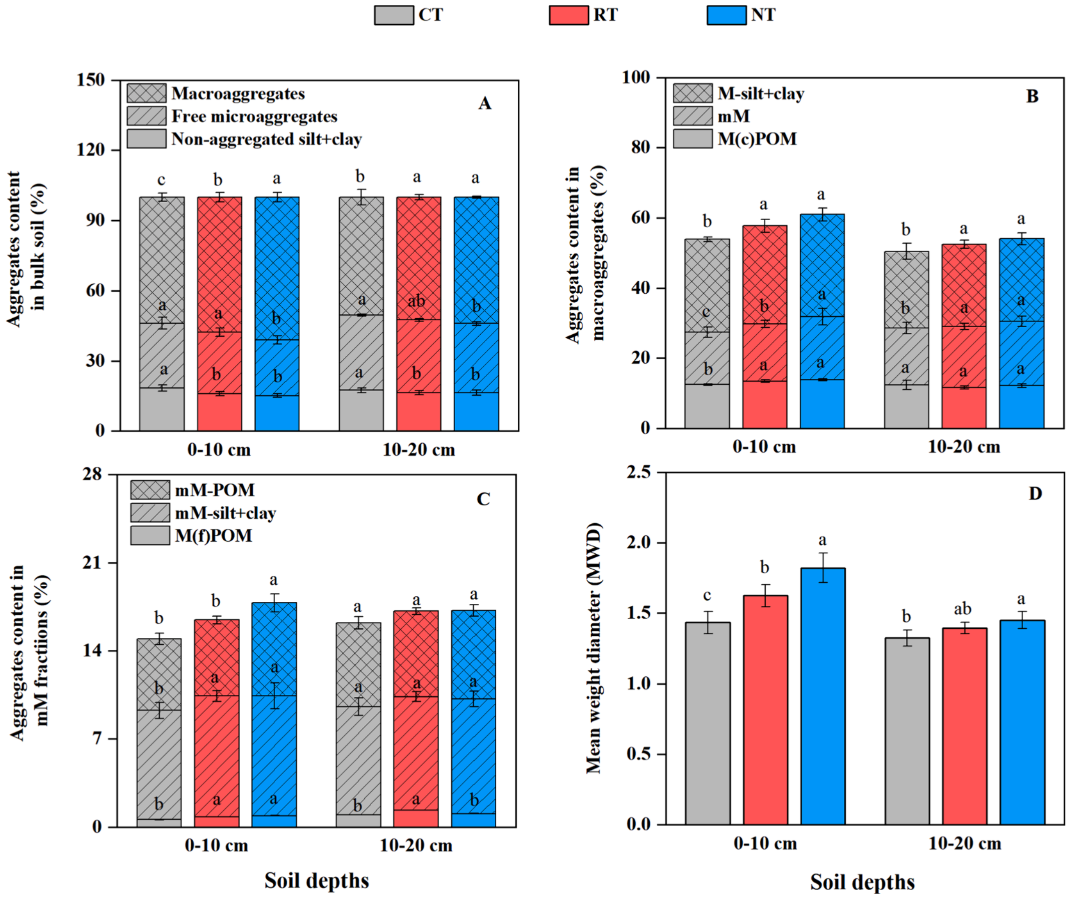

Fig. 3. The aggregate distribution in bulk soil (a), macroaggregates (b), and microaggregates within macroaggregates (c), and the soil aggregate stability (d), under three tillage practices and two soil depths. Different lowercase letters indicate statistically significant differences ($p < 0.05$) among the treatments. Bars represent the standard errors ($n = 4$). POM: particulate organic matter; M(c)POM: coarse POM in the macroaggregates but outside microaggregates; mM: the microaggregates within macroaggregates; M−silt + clay: silt and clay fractions within macroaggregates; M(f)POM: free POM inside macroaggregates but outside microaggregate; mM-POM: POM inside the mM fractions; mM-silt + clay: silt and clay fractions inside the mM fractions. CT: conventional tillage; RT: reduced tillage; NT: no tillage.

POM and microaggregate fractions (Fig. 4 E, F, G and H). At the 0–10 cm soil layer, both RT and NT significantly increased the Feo content by 11.2 %–27.6 % compared to CT in bulk soil, macroaggregates, M−silt + clay, and mM-silt + clay fractions (Fig. 4 E and F). In contrast, the Feo content was 17.1 % higher under CT than under RT in free microaggregates, and it increased by 9.7 %–13.4 % under both RT and NT compared to CT in the non-aggregated silt + clay fractions (Fig. 4 E). At the 10–20 cm soil layer, both NT and RT resulted in significantly higher Feo contents in all aggregates except the mM, mM-POM, and mM-silt + clay fractions (Fig. 4 G and H).

The Fep content exhibited a similar trend to that of SOC and was highest in macroaggregates. At the 0–10 cm soil layer, the Fep content was nearly 50 % lower in free microaggregates than in macroaggregates (Fig. 4 I). Moreover, the Fep content increased with decreasing tillage intensity (NT > RT > CT) in bulk soil, macroaggregates, M(c)POM, M−silt + clay and mM-silt + clay fractions (Fig. 4 I and J). At the 10–20 cm soil layer, both RT and NT significantly increased the Fep content by 9.7 %–21.8 % compared to CT in bulk soil, macroaggregates, M(c)POM, mM, and mM-silt + clay fractions (Fig. 4 K and L). In addition, no statistically significant differences were observed among the three tillage practices in free microaggregates, non-aggregated silt + clay and mM-POM fractions at the 10–20 cm soil layer (Fig. 4 K and L).

3.5. *Iron-bound organic carbon content in bulk soil and different aggregates*

Both the content of Fe-bound OC and $f_{\text{Fe-bound OC}}$ were higher in the silt + clay fractions than in macroaggregates or microaggregates (Fig. 5). Similar trends were observed across two soil layers and three tillage practices. Compared with CT, NT and RT increased the Fe-bound OC content by 16.2 %–37.9 % and 15.4 %–28.5 %, respectively, in bulk soil (Fig. 5 a), macroaggregates (Fig. 5 d), M−silt + clay (Fig. 5 f) and M(c)POM fractions (Fig. 5 g). In the mM and mM-silt + clay fractions, only NT significantly increased the Fe-bound OC content compared with CT (Fig. 5 h and i). The $f_{\text{Fe-bound OC}}$ was 8.3 %-12.7 % higher in M−silt + clay, and mM-silt + clay fractions under NT and RT at the 0–10 cm soil layer compared to CT (Fig. 5 f and i). At the 10–20 cm soil layer, the $f_{\text{Fe-bound OC}}$ was 9.5 %–35.7 % higher in bulk soil, macroaggregates, and M−silt + clay fractions under NT and RT compared with CT. However, the $f_{\text{Fe-bound OC}}$ was decreased by 7.6 %–20.3 % under NT and RT in the non-aggregated silt + clay fractions compared to CT (Fig. 5 c). Meanwhile, CT resulted in the highest Fe-bound OC content in free microaggregates and non-aggregated silt + clay fractions in both soil layers, which was increased by 4.1 %–34.3 % and 12.2 %–42.1 % compared to NT and RT, respectively (Fig. 5 b and c).

3.6. *Relationship between iron oxides and the stability of organic carbon and aggregates*

To explore the relationship between iron oxide content, SOC content, and aggregate stability, we generated a correlation matrix (Fig. 6). Overall, the contents of Feo, Fep, Fe-bound OC, and Feo/Fed were positively correlated with SOC content in bulk soil, macroaggregates, and silt and clay fractions within macroaggregates (M.SC and mM.SC).

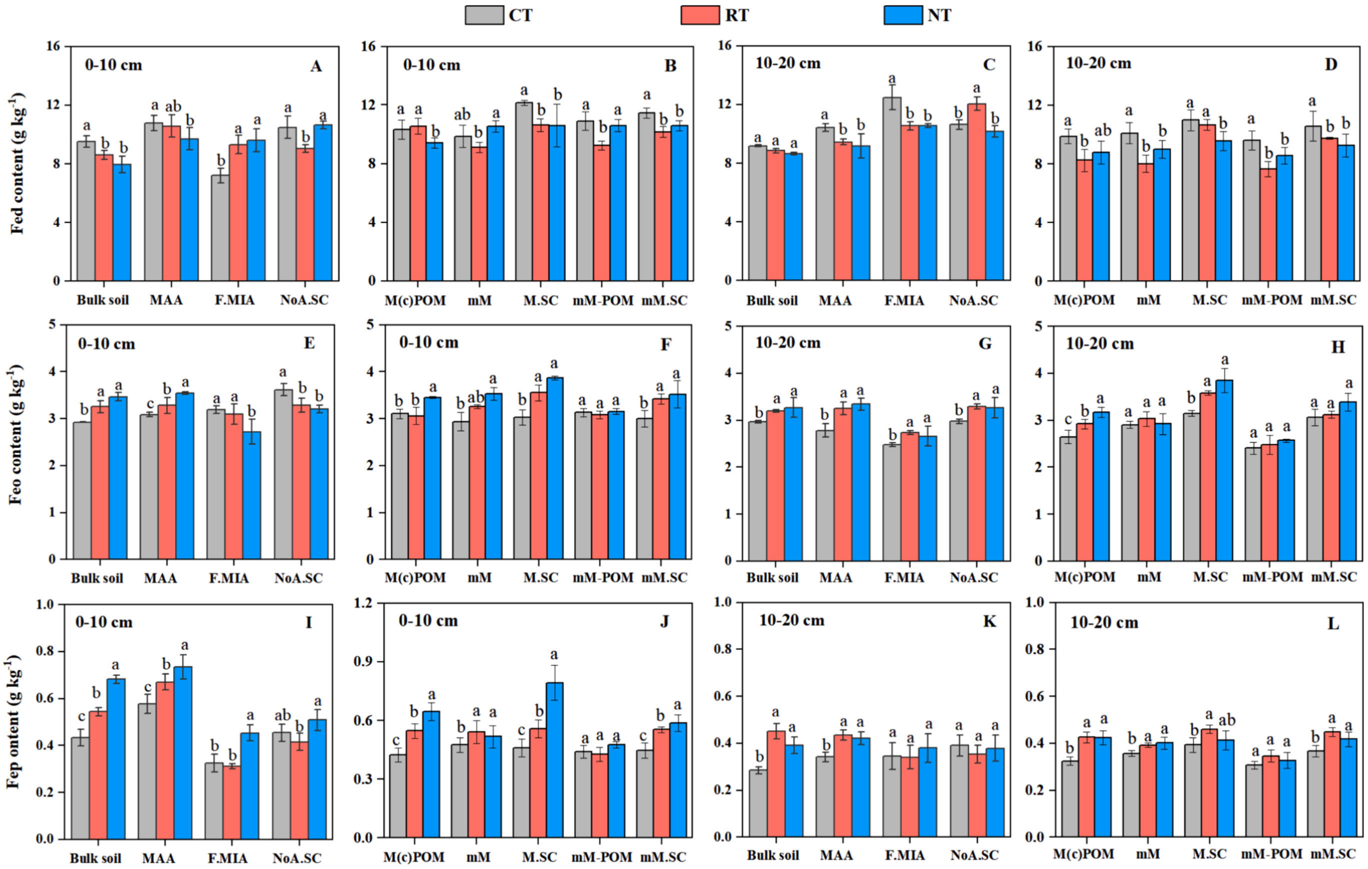

Fig. 4. The iron oxides content in different aggregates under three tillage practices at the 0–10 cm and 10–20 cm soil depths. Different lowercase letters indicate statistically significant ($p < 0.05$) differences among the treatments. Bars represent the standard errors (n = 4). Fe: iron; Fed: reactive Fe content; Feo: non-crystalline Fe content; Fep: complexed Fe content. MAA: macroaggregates; F.MIA: free microaggregates; NoA.SC: non-aggregated silt + clay fractions; POM: particulate organic matter; M(c)POM: coarse POM in the macroaggregates but outside microaggregates; mM: the microaggregates within macroaggregates; M.SC: silt and clay fractions within macroaggregates; M(f)POM: free POM inside macroaggregates but outside microaggregate; mM-POM: POM inside the mM fractions; mM.SC: silt and clay fractions inside the mM fractions. CT: conventional tillage; RT: reduced tillage; NT: no tillage.

In addition, the Fep, Feo/Fed, and Fe-bound OC contents were significantly positively related to the SOC content in the M(c)POM fraction. However, the Fed content exhibited negative relationship with SOC content only in the M.SC fractions. Finally, the contents of Feo, Fep, and Feo/Fed exhibited significant positive relationships with MWD under all three tillage practices at two soil depths.

3.7. *Chemical composition of organic carbon in bulk soil and different aggregates*

We evaluated the OC functional groups present in bulk soil and aggregate fractions (Fig. 7; S Fig. 1). Different SOC fractions exhibited a predominant presence of phenolic-C, aliphatic-C, aromatic-C, and polysaccharides-C as the major OC functional groups (Fig. 7). Across three tillage practices and two soil depths, polysaccharides-C comprised the highest proportion (42.9 %–64.1 %), followed by phenolic-C (30.1 %–49.09 %), aromatic-C (3.55 %–7.06 %), and aliphatic-C (0.64 %–1.11 %) compounds (Table 2; S Table 2).

Within aggregates, the organic C functional groups exhibited similar results across both soil depths (Table 2; S Table 2), and the following description is based on the result of the 0–10 cm soil layer. In general, the percentage of phenolic-C was higher under CT compared to NT and RT in bulk soil and macroaggregates (Table 2). Moreover, the proportion of aromatic-C and the aromatic-C/aliphatic-C ratios were increased under NT and RT relative to CT for bulk soil (0–10 cm) and for macroaggregates, mM, M−silt + clay and mM-silt + clay fractions at both soil depths. On the contrary, the free microaggregates and non-aggregated silt + clay exhibited an opposite pattern in terms of the identical proportion and ratio of OC functional groups described above. More precisely, in comparison to CT, the percentage of the phenolic-C was significantly decreased by 8.2 %–24.9 % under NT and 7.1 %–10.8 % under RT. However, both NT and RT result in an increase in the percentage of aromatic-C by 9.1 %–36.4 % and the aromatic-C/aliphatic-C ratio by 7.4 %–28.6 % at topsoil depths in the bulk soil, macroaggregates, mM, M−silt + clay and mM-silt + clay fractions. Additionally, the percentage of polysaccharide-C was increased in macroaggregates, but decreased in the non-aggregated silt + clay fractions under NT and RT compared to CT. Finally, apart from the non-aggregated silt + clay fractions, there were no statistically significant differences in the proportion of aliphatic-C among the three treatments at both soil depths.

3.8. *Relationship between iron oxides and organic carbon functional groups in bulk soil and aggregates*

The redundancy analysis (RDA) was conducted to investigate the relationship between iron oxides and OC functional groups across three tillage practices. Overall, bulk soil, macroaggregates, free microaggregates, and non-aggregated silt + clay fractions accounted for 71.16 %, 72.20 %, 80.27 %, and 72.21 % of the variation in OC functional groups, respectively (Fig. 8). Furthermore, both SOC and Fe-bound OC significantly affected the distribution of OC functional groups in bulk soil. The variation in OC functional groups was notably impacted by the Feo content and Feo/Fed in macroaggregates, M−silt + clay, and mM-silt + clay fractions, and it was influenced by Fep and Fe-

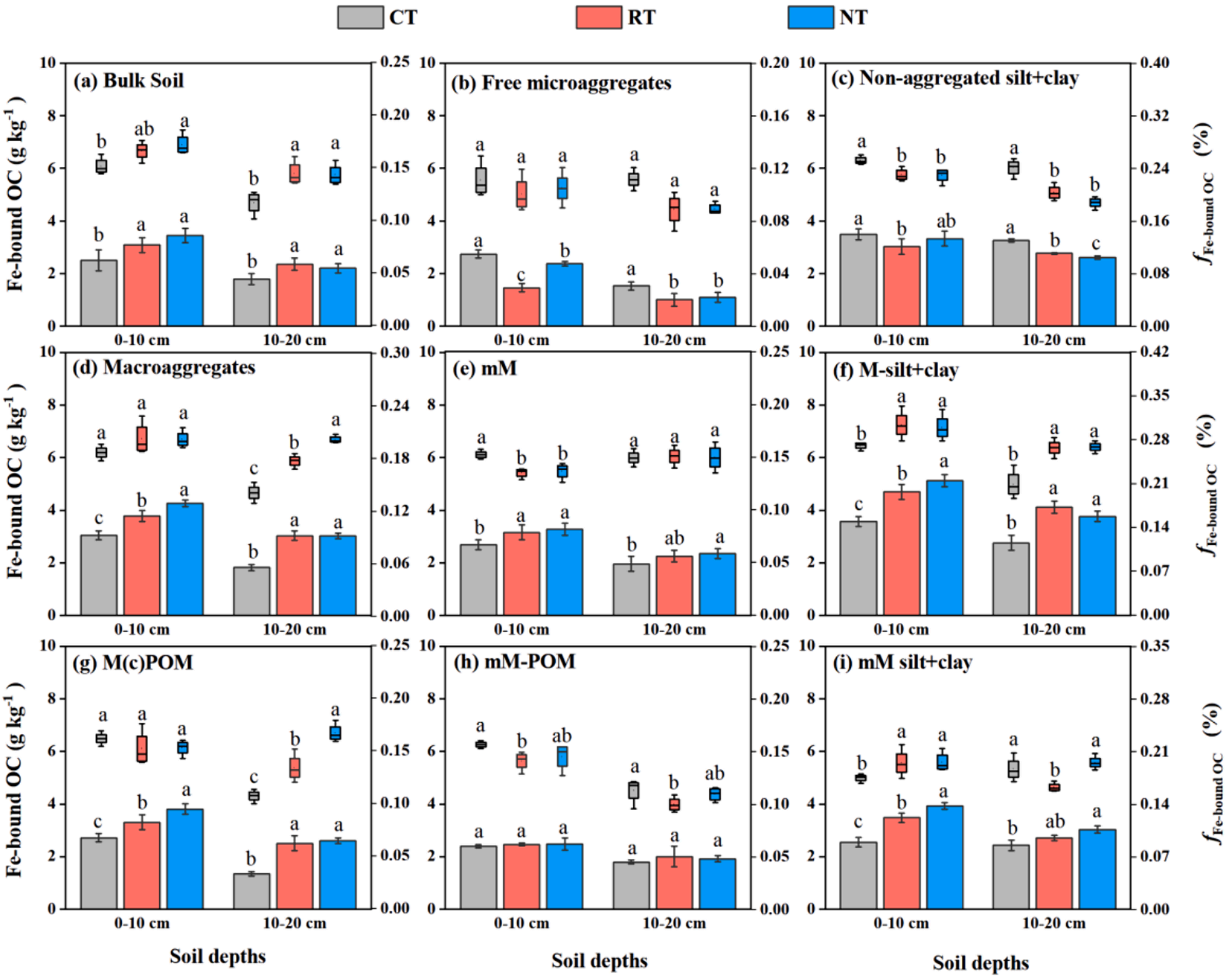

Fig. 5. Variation in Fe-bound organic carbon in bulk soil and aggregate fractions under three tillage practices at the 0–10 cm and 10–20 cm soil depths. The left column coordinate axis represents the Fe-bound OC content. The box diagram on the right represents the proportion of Fe-bound OC to total SOC ($f_{\text{Fe-bound OC}}$). Different letters indicate statistically significant ($p < 0.05$). Bars represent the standard errors (n = 4). POM: particulate organic matter; M(c)POM: coarse POM in the macroaggregates but outside microaggregates; mM: the microaggregates within macroaggregates; M−silt + clay: silt and clay fractions within macroaggregates; M(f) POM: free POM inside macroaggregates but outside microaggregate; mM-POM: POM inside the mM fractions; mM-silt + clay: silt and clay fractions inside the mM fractions. CT: conventional tillage; RT: reduced tillage; NT: no tillage.

bound OC contents in the M(c)POM and mM-POM fractions. These indicators were positively related to aromatic-C and aromatic-C/aliphatic-C in bulk soil and macroaggregates. However, within free microaggregates and the non-aggregated silt + clay fractions, the positive relationship was observed between Fed content and aromatic-C/aliphatic-C. The proportion of aromatic-C and aromatic-C/aliphatic-C showed a strong association with NT, followed by RT, across both soil depths in bulk soil, macroaggregates, M−silt + clay, and mM-silt + clay fractions, while the two indicators were correlated with CT in free microaggregates and the non-aggregated silt + clay fractions.

3.9. Key factors regulating soil organic carbon sequestration under conservation tillage practices

Fe oxides explained 87 % of the total variance in aggregate stability and 54 % of the total variance in chemical stability (Fig. 9 A). The combined contents of Feo and Fep, along with polysaccharide-C, exhibited significant positive correlations with MWD ($p < 0.001$; Fig. 9B). Moreover, the total Feo and Fep content demonstrated the most substantial direct positive effects on both aggregate stability (path coefficient = 0.84, Fig. 8A; $p < 0.01$) and chemical complexity (path coefficient was 0.75, Fig. 9A; $p < 0.01$). Concurrently, the Feo plus Fep contents increased alongside the proportion of aromatic-C and the aromatic-C/aliphatic-C ratio ($p < 0.01$; Fig. 9B). Correlation analysis showed that the content of aromatic-C exhibited the highest R^2 and was more strongly related to SOC content, followed by the combined Feo plus Fep content and the aromatic-C/aliphatic-C ratio ($p < 0.001$; Fig. 9B). Overall, mineral associated OC, aggregate stability and chemical composition explained 95 % of the variation in SOC content (Fig. 9A). Moreover, the chemical resistance (path coefficient = 0.48, Fig. 9A; $p < 0.05$) was a greater contributor than aggregate stability (path coefficient = 0.29, Fig. 9A; $p < 0.05$) to SOC sequestration (Fig. 9A). These results suggest that conservation tillage enhances the availability of Fe for binding C, potentially facilitated through the creation of organo-Fe complexes, which in turn promote soil aggregation and further contribute to SOC chemical stability. Thus, it appears that increased SOC sequestration predominantly results from chemical protection in northeastern Chinese Mollisols.

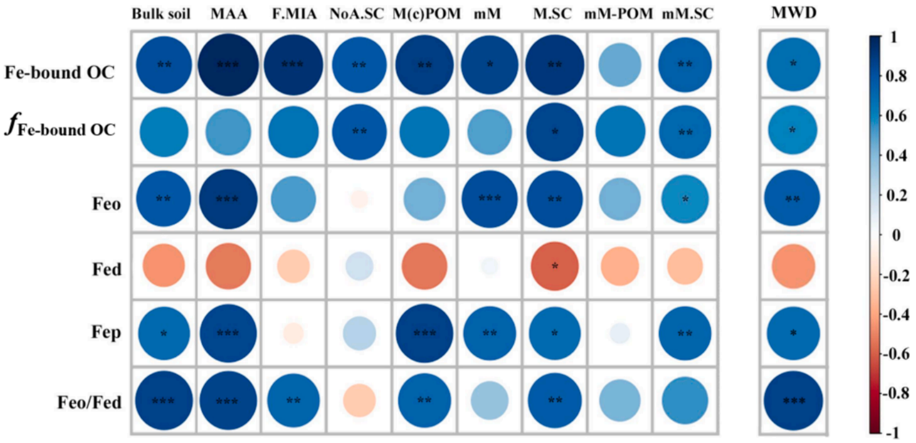

Fig. 6. Pearson correlation analysis of the relationship between iron oxide content (Fe-bound OC, $f_{\text{Fe-bound OC}}$, Fed, Feo, Fep, and Feo/Fed) and organic carbon content and mean weight diameter in bulk soil and different size class of aggregates. MWD: mean weight diameter; MAA: macroaggregates; F.MIA: free microaggregates; NoA.SC: non-aggregated silt + clay fractions; M(c)POM: coarse particulate organic matter (POM) inside macroaggregates; mM: microaggregates within macroaggregates; M.SC: silt and clay fractions inside macroaggregates; mM-POM: POM within mM fractions; mM.SC: silt and clay fractions within mM fractions. The color of each circle corresponds to the magnitude of the Pearson's correlation coefficient. Asterisks indicates statistical significance: *$p < 0.05$, **$p < 0.01$, ***$p < 0.001$, $n = 24$.

4. Discussion

4.1. *Tillage practices influence the soil aggregation and aggregate-associated organic carbon distribution*

Our results showed that conservation tillage and straw return had the higher MWD value and enhanced physical protection to aggregate associated organic carbon (OC) compared to CT (Fig. 9). As integral components of soil structure, soil aggregates serve as crucial repositories for SOC preservation and against mineralization (Liu et al., 2022). In this study, NT and RT significantly increased the macroaggregates content, and decreased the proportion of free microaggregates and non-aggregated silt and clay fractions, thus enhancing aggregate stability (Fig. 1). This phenomenon could be attributed to a confluence of two mechanisms: diminished physical disruption of large aggregates and heightened resistance to oxidation of SOC (e.g., aromatic-C and alkyl-C compounds) under conservation tillage regimes (Mustafa et al., 2021). As mentioned, the crop residues under NT and RT were all returned to the field. Maize straw contains mainly cellulose and hemicellulose, both of which exhibit resistance to decomposition and are challenging to mineralize in anaerobic environments subjected to NT and RT (Kan et al., 2022; Wang et al., 2020). Additionally, the continuous addition of organic material provides a fresh source of OC and triggers the liberation of certain soil minerals, thereby facilitating the generation of stable organo-mineral complexes (Chen et al., 2022a). The mineral-associated OC generates polymers with stronger adhesion to format macroaggregates. Thereby, conservation tillage with straw return enhanced aggregate stability via the physical isolation of decomposers and chemical bounding of minerals particles to organic material.

The OC content was increased in bulk soil and all aggregate fractions under conservation tillage compared to CT (Table S1). The beneficial effects of NT are mainly reflected in the increased OC contribution from macroaggregates (Fig. 2), which act as mainly binders of SOC under conservation tillage (Modak et al., 2020). A higher percentage of M(c)POM and M(f)POM was observed in NT, followed by RT and CT (Fig. 2). The POM pool is a newly generated collection comprised of partially decomposed fragments derived from plant and animal residues (Baalousha, 2009; Witzgall et al., 2021). Maize straw incorporated into the soil typically possesses a relatively high C/N ratio, which is less favorable for microorganisms (Dou et al., 2016). Furthermore, minimized disturbance to soil aggregates under conservation tillage physically protects M(c)POM and M(f)POM against exposure to microbial decomposition (Gao et al., 2017). Here, a reduced proportion of OC in free microaggregates, while a greater content of occluded microaggregate-associated OC, was found under NT and RT compared to CT (Fig. 2). This finding is consistent with the conceptual model proposed by (Six et al., 1999, Six et al., 2004; Six et al., 2000). According to this model, no tillage promotes the form of microaggregate within macroaggregates, leading to the physical protection of free particulate OC due to a lower macroaggregate turnover rate. However, it is the OC associated with silt and clay fractions that appears to better explain the difference in total OC content observed between conservation tillage and CT (Fig. 2). Studies suggest that occluded microaggregates or clay fractions can promote the accumulation of stable organo-mineral complexes through intense ligand exchange and multivalent cation bridging (Liu et al., 2022). Based on these results, we propose that organic materials adsorbed to mineral surfaces and potentially shielded by the occluded microaggregates constitute the most important pool of OC stabilization and sequestration under conservation tillage.

Prior research has additionally indicated that SOC sequestration under NT practices is primarily augmented within the mineral-associated OC fraction (Assuncao et al., 2019; Kan et al., 2022; Wang et al., 2017). For instance, (Denef et al., 2004) reported that more than 67.5 % of the total difference in SOC between NT and CT was explained by fine silt and clay-associated OC. In the current study, we found that a significant contribution of silt and clay fractions to the formation of macroaggregates and microaggregates within macroaggregates, suggesting that mineral-associated OC may act as the primary binding agents for large aggregate formation (Fig. 1). Furthermore, it was observed that the silt and clay fractions exhibited higher SOC content compared to the particulate organic matter (POM) content within macroaggregates (Fig. 2), implying that minerals associated OC mainly contribute SOC sequestration and stabilization. The microscopic techniques of very small microaggregates have revealed remnants of plant cell walls, bacterial constituents, and amorphous OC are surrounded by clay fractions (Bayer et al., 2006; Ding et al., 2015). Conservation tillage practices have been reported to foster a more complex bacterial and fungal network, with higher bacterial necromass observed under NT compared to CT (Zhang et al., 2022; Zheng et al., 2022). These very small silt and clay fractions are believed to more easily associate with microbial material to form stable organo-mineral complexes, and thus constitute major sites of OC stabilization and aggregate cohesion.

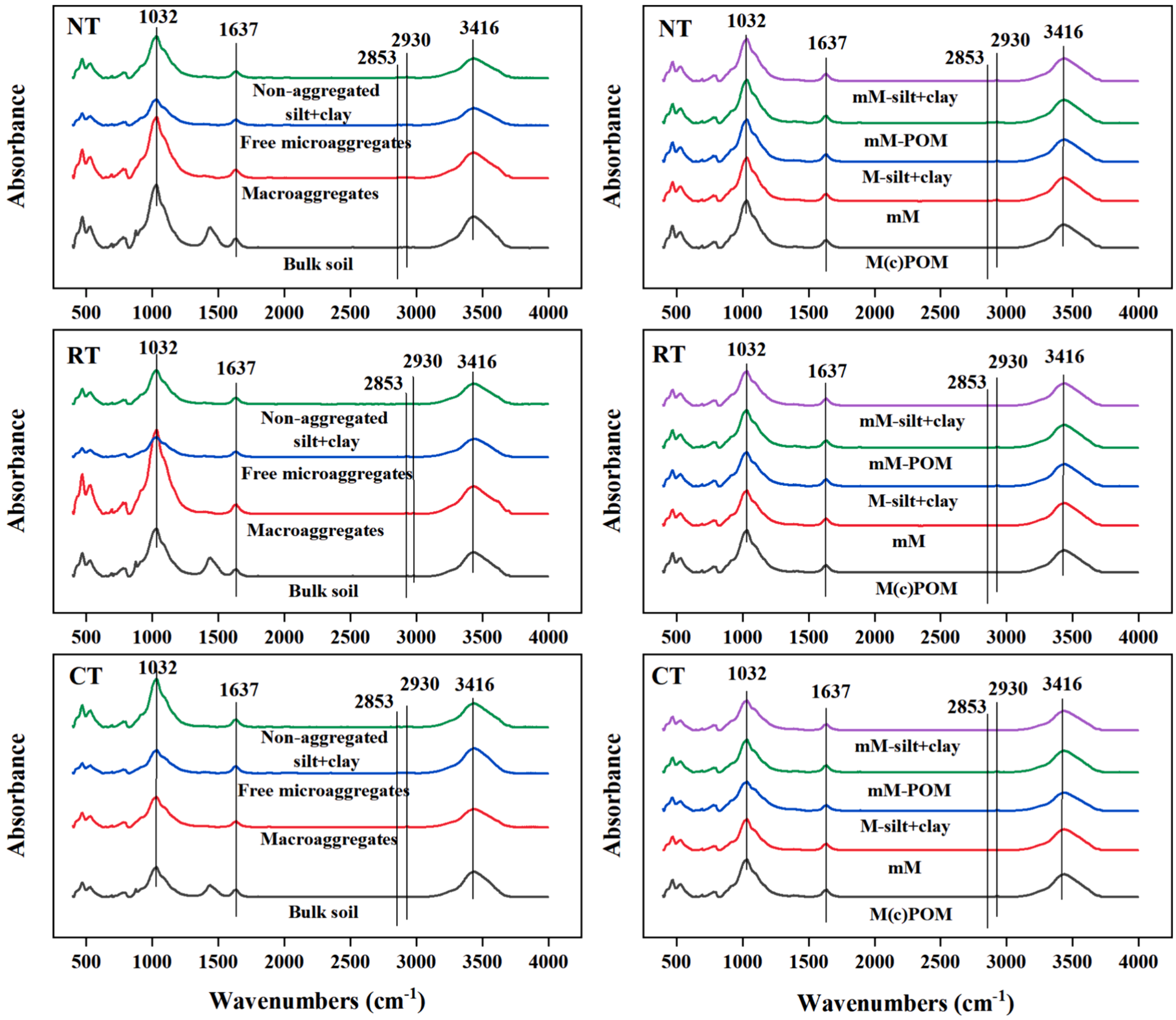

Fig. 7. Fourier transform infrared spectra of the bulk soil and different aggregate fractions for three practices at 0–10 cm soil depths. POM: particulate organic matter; M(c)POM: coarse POM in the macroaggregates but outside microaggregates; mM: the microaggregates within macroaggregates; M−silt + clay: silt and clay fractions within macroaggregates; M(f)POM: free POM inside macroaggregates but outside microaggregate; mM-POM: POM inside the mM fractions; mM-silt + clay: silt and clay fractions inside the mM fractions. CT: conventional tillage; RT: reduced tillage; NT: no tillage.

4.2. Effects of iron oxides and soil organic carbon on aggregate stability

Both Feo and Fep contents were enhanced under NT and RT by 12.7–41.1 % in bulk soil and macroaggregates compared to CT (Fig. 4). These results may be explained by the fact that long-term crop residue addition enriches soil organic material and promotes the formation of short-range-ordered Fe oxides on the aggregate surface (Zhu et al., 2016). Additionally, changes in oxygen availability in the soil environment are tightly coupled with the oxidation–reduction state of Fe elements. Limited soil disruption and organic material addition promote the formation of anaerobic microenvironments, wherein Fe^{3+} is reduced to Fe^{2+} (Inda et al., 2013). Furthermore, NT improves soil structural stability by enhancing soil connectivity in large aggregates and creating aerobic microenvironments where soluble Fe^{2+} undergoes reprecipitation in the form of metastable, poorly crystalline oxides (Guo et al., 2021; Li et al., 2020). Here, we observed that these poorly crystalline oxides (Feo and Fep) held greater significance than Fed (Fig. 6), primarily owing to their elevated hydration levels, expansive surface area, and diverse charge characteristics. These attributes contribute to the creation of more resilient mineral-bound organic carbon (OC) via reactions involving anion and inner-sphere ligand exchanges, and their advantageous geometrical properties may enhance their ability to provide physical safeguarding for SOC (Kirsten et al., 2021). Moreover, MWD exhibited a positive correlation with the concentrations of Feo and Fe-bound OC (Fig. 7), suggesting that complex Fe or aluminum (Al) oxides promote soil aggregation (Huang et al., 2016). Others have also consistently reported that NT not only improves soil aggregation but also activates Fe oxides, evidenced by the rise in Feo and Fep content (Inda et al., 2013; Xue et al., 2019a). Conservation tillage management creates a favorable physicochemical environment for facilitating the formation of amorphous iron oxide, thereby promoting soil aggregation.

In the current study, conservation tillage was observed to increase iron oxides content and the generation of SOC-Fe (oxyhydr) oxide associations within macroaggregates, thus enhancing MWD (Fig. 7 B). This phenomenon may be attributed to interactions between different OC functional groups and iron oxides. Recent research examining the molecular structure of OC across 12 test sites revealed more pronounced connections between Feo and polysaccharides and peptides compared to associations between carboxylic acids and lignin derivatives (Wan et al., 2019). According to the RDA, the polysaccharide-C demonstrated a strong positive correlation with Fep and Fe-bound OC in the POM fractions (Fig. 6). M(c)POM vulnerable to microbial attack and releases adhesive polysaccharides during degradation, which adhere to mineral surfaces or stay within free microaggregates (Dou et al., 2016). The

Table 2

Fourier-transform infrared spectra for the different aggregate fractions under different tillage practices at the 0–10 cm soil depths.

Aggregate size	Tillage	Phenolics-C	Aliphatic-C	Aromatic-C	Polysaccharide-C	Aromatic-C/ Aliphatic-C
		$3416\ cm^{-1}$	$2930–2853\ cm^{-1}$	$1637\ cm^{-1}$	$1085, 1032\ cm^{-1}$	$1637/2930–2853\ cm^{-1}$
Bulk soil	CT	36.9 ± 0.9 a	0.91 ± 0.04 a	5.13 ± 0.16b	57.1 ± 0.9 a	5.67 ± 0.41b
	RT	36.7 ± 2.9 a	0.89 ± 0.05 a	5.83 ± 1.08 a	56.6 ± 3.6 a	6.54 ± 0.78 a
	NT	33.8 ± 1.5 a	1.01 ± 0.04 a	6.14 ± 0.14 a	58.9 ± 1.6 a	6.07 ± 0.16 a
Macroaggregates	CT	42.4 ± 2.8 a	0.91 ± 0.02 a	5.31 ± 0.18b	51.4 ± 2.7b	5.83 ± 0.38b
	RT	35.9 ± 1.5b	0.93 ± 0.06 a	5.70 ± 0.09 a	57.4 ± 1.5 a	6.12 ± 0.61 a
	NT	34.4 ± 2.2b	1.02 ± 0.08 a	6.41 ± 0.53a	58.2 ± 1.9 a	6.28 ± 0.18 a
Free. Microaggregates	CT	30.4 ± 0.8b	0.86 ± 0.01 a	5.53 ± 0.21 a	63.3 ± 1.0 a	6.45 ± 0.19 a
	RT	35.2 ± 0.2 a	0.88 ± 0.01 a	5.20 ± 0.14 a	58.7 ± 0.4b	5.95 ± 0.17b
	NT	31.4 ± 2.6b	0.96 ± 0.03 a	5.52 ± 0.11 a	62.2 ± 2.6 a	5.78 ± 0.31b
Non-aggregated. silt + clay	CT	35.5 ± 0.6b	0.71 ± 0.02b	5.41 ± 0.23 a	58.4 ± 0.7 a	7.65 ± 0.21 a
	RT	39.6 ± 2.7 a	0.96 ± 0.04 a	4.92 ± 0.11 a	54.5 ± 2.8b	5.18 ± 0.43b
	NT	42.6 ± 2.2 a	1.02 ± 0.05 a	5.15 ± 0.14 a	51.2 ± 2.2b	5.07 ± 0.15b
M(c)POM	CT	43.2 ± 3.5 a	0.91 ± 0.01 a	3.55 ± 0.26b	52.3 ± 4.6b	3.92 ± 0.34 a
	RT	44.6 ± 3.1 a	1.16 ± 0.04 a	3.99 ± 0.28 ab	50.3 ± 2.9b	3.45 ± 0.21 a
	NT	32.5 ± 2.3b	1.20 ± 0.07 a	4.34 ± 0.28 a	62.0 ± 4.8 a	3.61 ± 0.31 a
mM	CT	43.0 ± 3.4b	1.06 ± 0.02 a	5.71 ± 0.05b	51.2 ± 3.4b	5.38 ± 0.21b
	RT	46.1 ± 4.3 a	1.03 ± 0.03 a	5.96 ± 0.27b	46.9 ± 3.9b	5.78 ± 0.28 a
	NT	35.1 ± 2.6c	1.06 ± 0.08 a	6.10 ± 0.56 a	57.7 ± 2.3 a	5.75 ± 0.28 a
M−silt + clay	CT	37.9 ± 1.8 a	0.78 ± 0.01 a	4.87 ± 0.35b	56.5 ± 2.2 a	6.24 ± 0.34b
	RT	35.9 ± 0.6 a	0.83 ± 0.01 a	5.51 ± 0.07a	57.7 ± 0.6 a	6.64 ± 0.33 a
	NT	32.8 ± 1.8b	0.84 ± 0.04 a	5.63 ± 0.13 a	60.7 ± 1.7 a	6.70 ± 0.12 a
mM-POM	CT	32.9 ± 1.1b	0.82 ± 0.03 a	3.85 ± 0.20b	61.4 ± 0.8 a	4.70 ± 0.77 a
	RT	36.8 ± 1.8 a	1.01 ± 0.06 a	4.20 ± 0.19 ab	57.9 ± 1.7 a	4.14 ± 0.21 a
	NT	34.8 ± 2.1b	1.00 ± 0.04 a	4.58 ± 0.41 a	59.6 ± 1.7 a	4.56 ± 0.02 a
mM-silt + clay	CT	38.8 ± 1.1 a	0.89 ± 0.02 a	5.12 ± 0.26b	55.2 ± 1.4b	5.75 ± 0.16b
	RT	33.0 ± 1.6b	0.81 ± 0.05 a	5.60 ± 0.05 a	60.6 ± 1.6 a	6.91 ± 0.42 a
	NT	33.5 ± 2.0b	0.80 ± 0.03 a	5.77 ± 0.27 a	59.9 ± 1.8 a	7.21 ± 0.08 a

Note: Data represent the mean ± standard error; n = 4. Different lowercase letters indicate statistically significant ($p < 0.05$) differences among the three treatments at the same soil depth. POM: particulate organic matter; M(c)POM: coarse POM in the macroaggregates but outside microaggregates; mM: the microaggregates within macroaggregates; M−silt + clay: silt and clay fractions within macroaggregates; M(f)POM: free POM inside macroaggregates but outside microaggregate; mM-POM: POM inside the mM fractions; mM-silt + clay: silt and clay fractions inside the mM fractions. CT: conventional tillage; RT: reduced tillage; NT: no tillage.

hierarchical model of soil aggregates shows that microbial products easily associate with clay fractions and bind together to form macroaggregates (Tisdall and OADES, 1982). Moreover, the microbial reduction of crystalline oxides to poorly crystalline ferrihydrite results in an expanded specific surface area, enabling the adsorption of a greater quantity of aromatic compounds (Kirsten et al., 2021). In this study, we observed that conservation tillage increased the proportion of polysaccharide-C and aromatic-C, and positive relationship was revealed among aromatic-C, MWD and Feo + Fep content (Fig. 7 B). It is reasonably speculated that Fe oxides have the potential for selectively adsorbing aromatic compounds, thereby promoting the formation of organo-Fe complexes, and playing vital roles in macro-aggregation and aggregate stability.

4.3. Soil organic carbon chemistry and soil aggregation contribute to the stabilization of SOC under conservation tillage

The aromatic-C/aliphatic-C ratio can be used as an indicator for assessing changes in the quality and quantity of SOC over time. A higher aromatic-C/aliphatic-C ratio typically represents more recalcitrant OC fractions (e.g., lignin, suberin, and humic substances), which are resistant to microbial decomposition (Almagro et al., 2021). In this study, we found that both NT and RT increased the proportion of aromatic-C and the aromatic-C/aliphatic-C ratio compared to CT in bulk soils at both soil depths (Table 2). These results suggest that conservation tillage promotes a more complicated OC molecular structure and improves C sequestration. Contrary to our findings, other researchers have reported that retaining straw elevates aliphatic-C content while diminishing aromatic-C content by reducing semiquinone free radicals (Zhou et al., 2022). The composition of different OC functional groups depends not only on the inherent properties of the residue, but also on soil type and climatic conditions (Wu et al., 2021). Mollisols are prevalent in the regions of northeastern China characterized by seasonally frozen soils, where the mean annual temperature ranges from −2 ℃ to 6 ℃. Extremely low temperatures limit the rate of microbial enzymatic digestion of crop residues into simpler molecules for their growth and activity (Li et al., 2022; Piazza et al., 2020). Conversely, the frequent disturbance associated with intensive tillage disrupts soil structure and stimulates microbial respiration, accelerating the decomposition of exposed organic matter (Li et al., 2020). As a result, conservation tillage increases SOC stability and mitigates mineralization in Mollisols.

The increased proportion of aromatic-C in the soil was mainly attributed to the protection of aromatic-C within macroaggregates under NT and RT (Table 2). It has been previously reported that minimal tillage results in improved retention of macroaggregates and SOC preservation (Wacker et al., 2022; João de Andrade Bonetti et al., 2017). Previous studies have reported a relationship between macroaggregates and aromatic-C, as microaggregates are transiently bound into macroaggregates (Chen et al., 2022b). In addition, the content of aromatic-C decreases with increasing soil aggregate size, and highly recalcitrant C accumulates selectively in smaller aggregates (Steffens et al., 2010). Here, we found that CT disrupts macroaggregates and renders fresh plant litter and labile fractions more susceptible to microbial attack. In contrast, RT and NT weaken these processes, resulting in a higher aromatic-C/aliphatic-C ratio and more stable SOC in large aggregates and mM-silt + clay fractions within macroaggregates (Table 2). This was confirmed by the presence of more polysaccharide-C in M(c)POM under CT. Ding et al. (2006) also indicated that the macroaggregate fraction had the highest concentrations of total amino sugars, while the silt and clay fractions displayed the lowest levels. These results suggest that conservation tillage results in more stable OC by reducing macroaggregate turnover rate and increasing the proportion of microaggregates bound to macroaggregates.

In addition, the aromatic-C/aliphatic-C ratio was significantly positively correlated with macroaggregates and silt + clay fractions within macroaggregate-associated OC (Fig. 6), indicating that mineral-

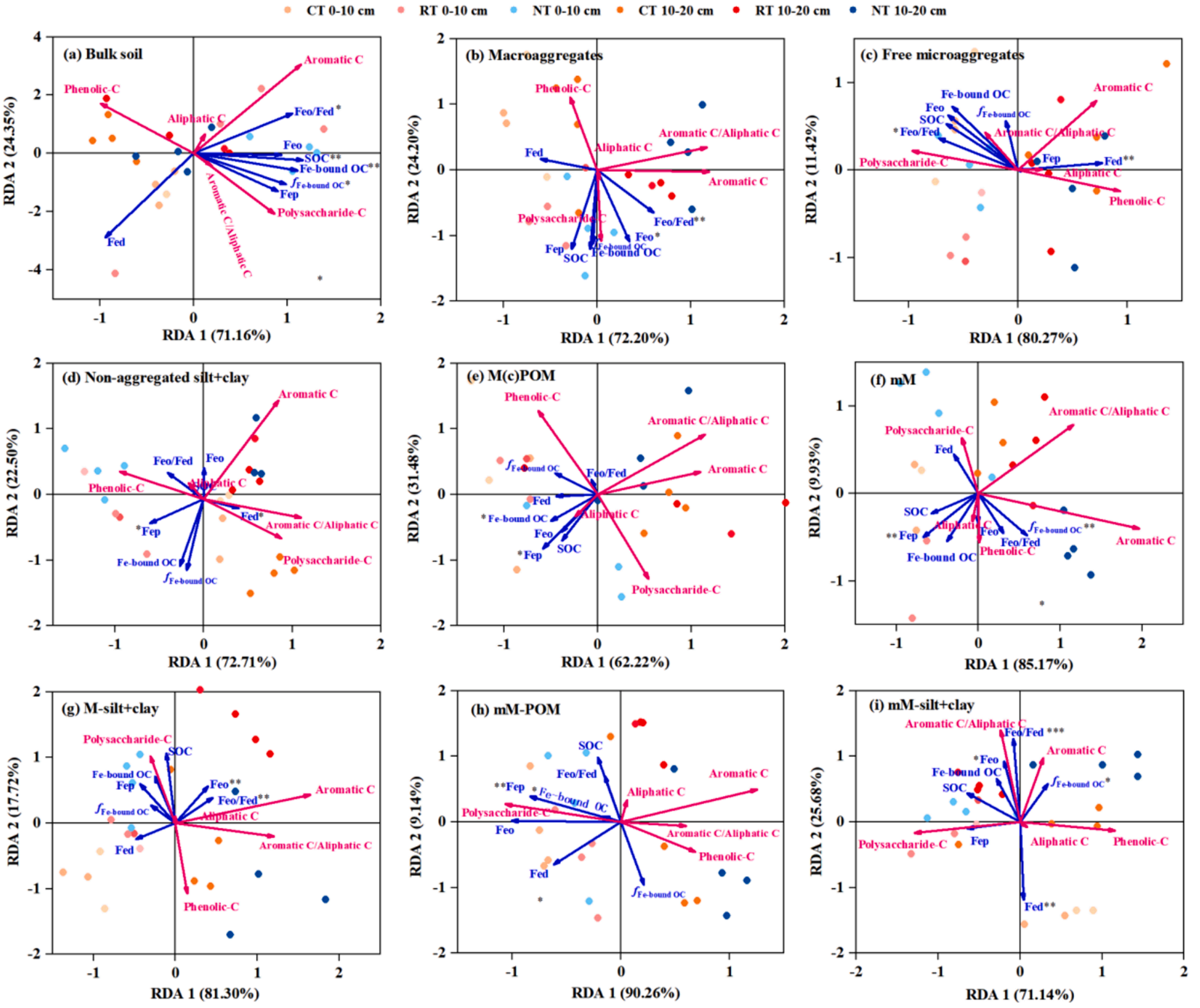

Fig. 8. Redundancy analysis (RDA) between soil physicochemical traits (SOC, Fe-bound OC, $f_{\text{Fe-bound OC}}$, Fed, Feo, Fep, and Feo/Fed) and OC functional groups in bulk soil and aggregate fractions. Asterisks indicate statistical significance: $^*p < 0.05$, $^{**}p < 0.01$, $^{***}p < 0.001$, $n = 24$. POM: particulate organic matter; M(c)POM: coarse POM in the macroaggregates but outside microaggregates; mM: the microaggregates within macroaggregates; M−silt + clay: silt and clay fractions within macroaggregates; M(f)POM: free POM inside macroaggregates but outside microaggregate; mM-POM: POM inside the mM fractions; mM-silt + clay: silt and clay fractions inside the mM fractions. CT: conventional tillage; RT: reduced tillage; NT: no tillage.

associated SOC may play an important role. Here, we found that conservation tillage increased the concentrations of soil Fe oxides and the formation of SOC-Fe (oxyhydr) oxide associations in macroaggregates (Fig. 4), thus strongly promoting the structural complexity of aggregate-associated OC (Fig. 7 B). As described above, microbial reduction of ferrihydrite increases its specific surface area, resulting in the formation of aromatic-Fe complexes that can selectively adsorb a greater amount of aromatic molecules at the aggregate interface (Chen et al., 2022a). In the RDA analysis, the ratio of aromatic-C/aliphatic-C was strongly positively correlated with the Fe-bound OC content in microaggregates and silt and clay fractions (Fig. 6), suggesting that Fe oxides may play a role in shaping the structural complexity of aggregate-associated OC (Zhu et al., 2016). Large amounts of aromatic-C have the potential to increase aggregate stability through mechanisms such as reduced water infiltration capacity or by affecting the presence of free lipids and other hydrophobic compounds (Adhikari and Yang, 2015; Mustafa et al., 2022). This, in turn, may lead to the development of a hydrophobic coating around soil organic matter that acts as a nucleus for aggregate formation and resists microbial degradation (Abrar et al., 2020). Our findings indicate that conservation tillage not only enhanced the influx of exogenous C but also heightened the accessibility of Fe for C binding when compared to CT (Figs. 3, 4). Consequently, this enhancement facilitated the formation of organo-Fe complexes more effectively (Kan et al., 2022). It is therefore plausible to infer that the increase in mineral-associated OC under conservation tillage was primarily due to the development of organo-Fe complexes.

Based on our results, chemical resistance (path coefficient = 0.48, Fig. 7A; $p < 0.05$) was a greater contributor to SOC sequestration than aggregate stability (path coefficient = 0.29, Fig. 7A; $p < 0.05$). We suggest that the formation of stable organo-mineral complexes plays a central role in stabilizing SOC under conservation tillage compared to CT. As time progresses, labile compounds may diminish, and microbes may either perish or enter a dormant state, resulting in their physical separation from electron acceptors, energy sources, and predators (Plaza et al., 2013). The presence of plant residues or microbial by-products adhering to short-range-ordered iron (SRO-Fe) surfaces or protected

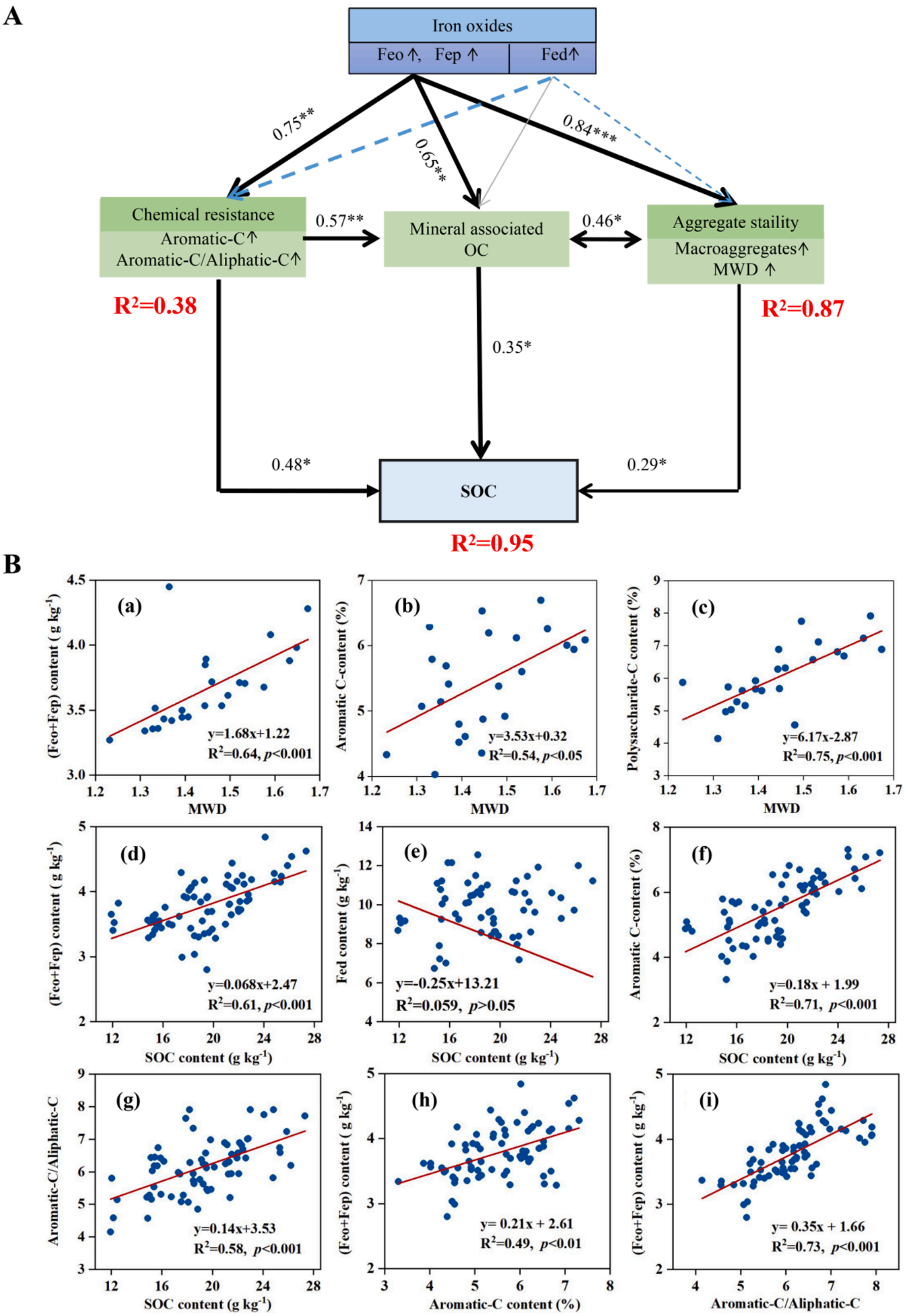

Fig. 9. Structural equation model (SEM) of the effect of mineral-associated OC, OC chemical resistance, and iron oxides on aggregate stability (mean weight diameter; MWD) under long-term conservation tillage ($\chi2/df = 3.72$, RMSEA = 0.105, GFI = 0.962) (A). Correlation analysis (B) between between Feo + Fep and MWD(a), aromatic-C and MWD (b), polysaccharide-C and MWD (c), Feo + Fep and SOC(d), Fed and SOC(e), aromatic-C and SOC(f), aromatic-C/aliphatic-C and SOC (g), Feo + Fep and aromatic-C(h), and Feo + Fep and aromatic-C/aliphatic-C(i) in bulk soil and aggregates. Mineral associated OC is obtained by adding the OC content of all silt + clay fractions; chemical resistance includes aromatic-C content and the aromatic-C/aliphatic-C ratio; iron oxides include Fed, Feo, and Fep content. Single-headed and double-headed arrows in the panel indicate causal links and covariations, respectively. Solid and dotted black arrows indicate positive and negative causal links, respectively. Solid grey arrows indicate insignificant relationships. The values adjacent to the column represent the standardized coefficients in SEM. Asterisks indicate statistical significance: *$p < 0.05$; **$p < 0.01$; ***$p < 0.001$. For each variable, the percentage of the variance explained by the model is indicated next to the box corresponding to the variable.

by encapsulation within minute microaggregates is emerging as a significant mechanism for the stabilization and retention of soil organic carbon (SOC) under conservation tillage practices. Selective adsorption to some specific OC functional groups by iron oxides and physically protected by entrapment within occluded microaggregates, appear to be an important pool for OC stabilization and C sequestration in soils under NT and RT. This mineral-associated OC pool has likely been underestimated in comparison to crop-derived OC physically protected within macroaggregates. The stabilization mechanism of soil organic carbon described here is probably valid not only for conservation tillage systems but for any agricultural practice or vegetation restoration able to reduce soil disturbance and aggregate breakup or increase organic material inputs into the soil. Especially in some wetlands that rich in Fe, the combination mechanisms of mineral-organic association and physical protection are crucial to SOC sequestration in farmland, meadows or forests. It will be important for the further studies distinguishing the relative amounts of stable necromass and labile cell- or plant-derived substances adsorbed to mineral surfaces. This will be crucial for determining whether conservation tillage alters biochemistry stability and uplift potential of SOC in agriculture systems. In addition, future research efforts should prioritize examining the entire soil profile, rather than solely concentrating on the surface layer in this study, attain a more holistic comprehension of SOC storage and to better evaluate soil carbon management strategies.

5. Conclusions

Compared to CT, conservation tillage involving straw incorporation (RT and NT) were beneficial to enhance SOC content and stability. Through the comprehensive physical fractionation, we provide evidence that organo-mineral complexes constitute the most significant pool for long-term SOC persistence under conservation tillage systems in the studied Mollisols. NT and RT not only exhibited significantly higher contents of aromatic-C and polysaccharide-C but also promoted the formation of amorphous iron oxides (Fig. 7), possibly facilitating the formation of very stable organo-Fe associations. Moreover, the result emphasized the importance of physical protection within occluded microaggregates of light and heavy carbon fractions adsorbed on mineral surfaces for SOC preservation under NT and RT. Although the higher aromatic-C/Aliphatic-C ratio of SOC improved its inherent biochemical recalcitrance, the generation of organo-Fe complexes under conservation tillage systems also promote soil aggregation and physically stabilized SOC. Therefore, we believe that conservation tillage contributes to SOC sequestration in Mollisols by improving the Fe-mediated stabilization of aggregate-associated OC (via forming organo-minerals complex), and thus promise as a feasible approach to alleviate carbon loss in Northeast China for sustainable agricultural development.

CRediT authorship contribution statement

Zixuan Han: Conceptualization, Methodology, Formal analysis, Writing – original draft, Writing – review & editing. **Xueping Wu:** Project administration. **Aizhen Liang:** Resources. **Shengping Li:** Conceptualization. **Huizhou Gao:** Methodology. **Xiaojun Song:** Software. **Xiaotong Liu:** Software. **Angyuan Jia:** Methodology. **Aurore Degré:** Supervision, Writing – review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Appendix A. Supplementary material

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