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Article

Effects of Tillage Practices on Soil Organo-Mineral Complexes and Organic Carbon Distribution Under Continuous Maize Cropping in the Black Soil Region of Northeast China

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Abstract

Organo-mineral complexes are intimately involved in protecting the stability of soil organic carbon (SOC), as they are influenced by environmental factors such as pH and redox conditions, as well as by the implementation of appropriate management practices. Nevertheless, the mechanism between environmental factors and the fractions of organo-mineral complexes, as well as their response to tillage practices, remain poorly understood. This study investigated the effects of rotary tillage (RT), plow tillage (PT), and no-tillage (NT) on organo-mineral complexes (water-dispersible G0 fraction, sodium-dispersible G1 fraction, grinding-dispersible G2 fraction) and their organic carbon (OC) in the black soil region of Northeast China in 2002 and 2022. Compared to 2002, the content of organo-mineral complexes and their OC in 2022 increased by 5.54% and 3.15%, respectively. Relative to PT, RT and NT increased the organo-mineral complex content by 0.39% and 8.40%, and increased the OC content by 9.41% and 20.56%, respectively. Between 2002 and 2022, tillage measures led to greater contributions of organo-mineral complexes to soil carbon sequestration. RT and PT primarily enhanced the contribution rate of the G0 fraction, whereas NT resulted in enhanced contribution rates for both the G0 and G1 fractions. This suggests NT is most conducive to transforming SOC into stable organo-mineral complexes. Redundancy and correlation analyses identified exchangeable Ca^{2+} in G1, pH, clay, TP, along with iron and aluminium oxides, as key environmental factors influencing the transformation pathways among the complexes fractions.

Keywords: Tillage measures; organo-mineral complexes; organic carbon; iron and aluminium oxides

1. Introduction

The black soil region of Northeast China serves as a major commercial grain production base in China [1,2]. This region produces over 30% of China's total maize yield [3]. Characterized by high organic matter content and abundant humus, black soil is highly fertile and ideal for crop growth [4]. Furthermore, black soil is one of the largest carbon pools in the world and plays a crucial role in climate change mitigation. Given its high productivity and significant potential for yield improvement, black soil is considered as a vital agricultural resource in Northeast China [5], the conservation and effective utilization of this valuable soil resource are of paramount agricultural importance.

However, long-term continuous maize monoculture and conventional tillage practices, primarily based on moldboard plowing over the years, have led to severe degradation of the black soil in Northeast China. This degradation is characterized by a marked soil structure destroying, a depletion of organic matter, and diminished soil fertility [6]. The SOC content in the top 17 cm of uncultivated soil was nearly double that of its cultivated equivalent after 50 years [7], and average thickness of the topsoil has decreased by 13.7 cm [8]. The cation exchange capacity (CEC) of surface

Chinese Mollisol declined with cultivation, from 45.8 cmol·kg⁻¹ in uncultivated soil to 31.5 cmol·kg⁻¹ after 50-cultivation [9]. To fight this degradation, restore soil productivity, and reconstruct a high-yield farmland ecosystem are therefore critical challenges that demand immediate resolution for the region's sustainable agricultural and economic development. Enhancing the protection and rational utilization of black soil is of great importance for safeguarding both global food security and ecological safety.

Soil organic carbon (SOC) long-term persistence is predominantly regulated by mineral protection [10,11]. Soil organic matter binds with minerals to form organo-mineral complexes (as the core of mineral-associated organic matter, MAOM) [12,13], which are relatively active components in the soil and have significant impacts on the stability of soil structure, the retention of soil moisture and nutrients, as well as the transformation of inorganic cementing substance [14,15].

Up to 73% organic matters were protected by organo-mineral complexes [16]. The stability to the protection of SOC by minerals is through forming organo-mineral complexes [17,18]. The formation, composition characteristics and functional roles of soil organo-mineral complexes are extremely complex. They are influenced not only by the material composition of the inherent soil properties, but also by tillage measures and environmental factors. Thus, despite organo-mineral complexes are of great significance in the stability of soil organic matter, fundamental knowledge gaps regarding the nature and behavior of organo-mineral complexes persist.

In the 0–17 cm topsoil layer, physical protection of SOC is greatly diminished by agricultural disturbances like tillage, thereby accelerating decomposition [19]. Studies have shown that different tillage measures (no-tillage, rotary tillage, plow tillage, etc) affected the content of soil organo-mineral complexes [20,21]. The stabilization of organo-mineral complexes was enhanced under no-tillage, as the reduced turnover rate of aggregates facilitated the formation and persistence of micro-aggregates within macro-aggregates [22]. The effects of tillage measures on the protection of organic matter may depend on the type of soil. However, the effects of tillage measures on organo-mineral complexes of low-weathered soils might be greater than that of high-weathered soils [23]. It remains unclear how different tillage measures affect the impact on the stability of organo-mineral complexes and sequestration of SOC.

Globally, exchangeable calcium (Ca) in most soils (from slightly acidic soil to alkaline soil) is positively correlated with SOC. Ca in the soil facilitates the formation of organo-mineral complexes by creating cation bridges between organic colloids and mineral surfaces [24]. This process improves soil permeability and water retention, thereby promoting the accumulation of SOC. Furthermore, soil minerals, particularly iron (Fe) and aluminium (Al) oxides, are regarded as the key controlling factors for the formation of organo-mineral complexes [16,25,26]. The surface of Fe oxides carries a large number of positive charges, enabling them to effectively adsorb negatively charged organic matter and form stable organo-mineral complexes through mechanisms such as ligand exchange, cation bridging, and hydrogen bonding [27,28]. Meanwhile, during continuous redox processes, Fe/Al oxides constantly co-precipitate with organic matter, which can effectively sequester SOC, reduce its accessibility to environmental microorganisms, and thereby slow down SOC decomposition [29,30]. Lower soil pH values promote the adsorption of SOC by Fe/Al oxides by increasing the solubility of Fe/Al ions and the positive charge on the surface of metal oxides [31]. Different tillage measures cause substantial variation in the depth of soil disturbance, resulting in significant differences in soil redox conditions and, consequently, in the protective effects of Fe/Al oxides on organo-mineral complexes [32]. Current, in-depth analysis of the key factors influencing the content of organic–mineral complexes and their bound organic carbon accumulation by tillage measures remains insufficient [33].

The grouping of soil organo-mineral complexes based on A. Φ. Tyulin's dispersion method has long been regarded as the basic classification of complexes [34,35]. MAOM can be studied based on particle size and density. Currently, the more mainstream and functionally significant analytical method is density fractionation. The gel dispersion method is based on the settling velocity of particles. As a type of MAOM analysis method, it has limitations such as "inability to effectively

distinguish the composition of organic matter, interference from dispersants, and potential cross-contamination between fractions". However, this study focuses on exploring the composition of soil particles and their relationship with the soil environment. During the experiment, sodium chloride with lower dispersibility was selected as the dispersant. Therefore, the experimental method is feasible and operable.

According to the gel dispersion classification method, the soil organo-mineral complex can be successively divided into the water dispersion fraction (G0), the sodium dispersion fraction (G1), and the sodium and abrasive dispersion fraction (G2) [36]. The G0 fraction consists of free mineral particles along with microaggregates smaller than 10 μm [34]. Colloids in the G1 fraction are complexes bound by calcium linkages, whereas those in the G2 fraction are complexes bound by iron and aluminum linkages [37]. Recent frameworks separate soil organic matter into distinct pools: particulate organic matter (POM) and MAOM. The G0 fraction is the core component of POM, while the G2 fraction constitutes the core of MAOM. Functionally and in terms of stability, the G1 fraction is intermediate between POM and MAOM. Upon disruption of microaggregates, G1 can be released, potentially transforming into POM or, through further processing, into MAOM. The decomposition of particulate organic matter (POM) provides a key pathway connecting it to the MAOM pool, by releasing dissolved organic compounds that can subsequently associate with soil minerals [13].

Tillage measures strongly affect SOC and their binding agents [38,39]. Tillage practices influence soil acidity and redox conditions, which in turn affect the content of soil Ca^{2+} and forms of iron-aluminum oxides, and ultimately determine the content and fractions of soil organo-mineral complexes. We hypothesized that 1) tillage measures would modify the fractions of organo-mineral complexes and factors of soil circumstance; 2) there would be a strongly positive correlation between different fractions organo-mineral complexes and actors of soil circumstance.

Based on the existing research work, the objectives were (1) to clarify the changes in three fractions of organo-mineral complexes and organic carbon distribution within them under the rotary tillage, plow tillage and no-tillage of long-term continuous corn cropping; (2) to explain the contribution of tillage measures to the fixation of organic carbon in black soil from a mechanistic perspective, and study the interaction between soil organic matter and soil minerals under different tillage measures; (3) further to explore the mechanism of the transformation of organo-mineral complexes in fractions of G0, G1 and G2, with the aim of providing a theoretical basis for the protection, utilization and fertilization improvement of black soil.

2. Materials and Methods

2.1. Experimental Site

The study area was situated in the premium black soil region of Northeast China, widely known as the golden maize belt. The selected experimental sites were characterized by flat topography and with no evidence of water erosion. A latitudinal transect was established, encompassing three zones from north to south, high latitude (4 points), middle latitude (3 points), and low latitude (5 points), as shown in Figure 1. Three tillage measures were applied and compared at every sampling point.

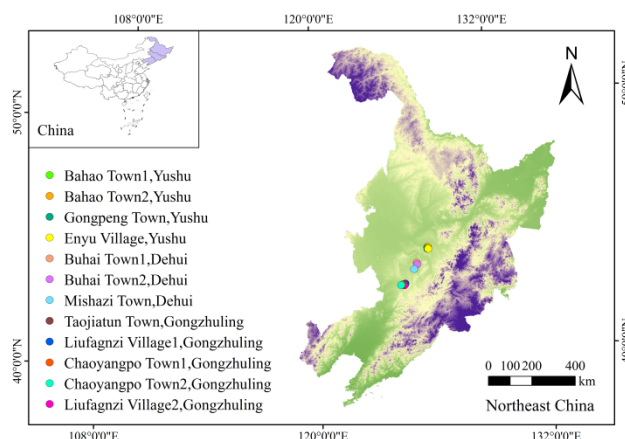


Figure 1. Locations of experimental sites.

The climate of the region is temperate, semi-humid continental monsoon. The soil at the experimental site is classified as Mollisols (Typic Hapludoll, USDA Soil Taxonomy) with a clay loam texture. All 12 sampling points were located in thin-layer black soil regions, characterized by similar soil textures, temperatures, and precipitation levels. The main components of soil clay minerals are montmorillonite, illite, chlorite, quartz and vermiculite. The mean values (0-20 cm) of SOC, pH, TN and TP are 15.93 g kg^{-1} , 6.74, 2.07 g kg^{-1} , and 1.31 g kg^{-1} , respectively at 12 sampling sites after autumn harvesting in 2002. The region has a mean annual temperature of $5.2 \text{ }^{\circ}\text{C}$ and receives approximately 600 mm of precipitation. The frost-free period averages 120 days per year. The experimental field is nearly flat ($<1\%$ slope) and has been under continuous maize monoculture for over two decades.

From 2002 to 2022, all field management practices were consistent across the experimental plots, with the exception of the tillage measures. The basic characteristics of the sampling points are shown in Table 1.

Table 1. Information of sampling sites.

Serial number	Experimental site	Longitude and latitude	Characteristic
1	Bahao Town1	45°02'30.8"N 126°26'20.4"E	Flat and open
2	Bahao Town 2	45°02'29.7"N 126°28'31.5"E	Flat and open
3	Gongpeng Town	44°58'22.4"N 126°26'30.1"E	Flat and open
4	Enyu Village	44°58'21.1"N 126°28'34.3"E	Flat and open
5	Buhai Town 1	44°27'24.7"N 125°43'47.9"E	Flat and open
6	Buhai Town 2	44°22'59.5"N 125°44'11.1"E	Flat and open
7	Mishazi Town	44°12'00.6"N 125°32'47.9"E	Flat and open
8	Taojiatun Town	43°38'49.4"N 124°58'04.5"E	Flat and open
9	Liufagnzi Village 1	43°37'08.6"N 124°57'24.2"E	Flat and open
10	Liufagnzi Village 2	43°34'08.4"N 124°54'11.6"E	Flat and open
11	Chaoyangpo Town 1	43°36'54.7"N 124°47'48.9"E	Flat and open
12	Chaoyangpo Town 2	43°35'45.3"N 124°43'27.6"E	Flat and open

Mishazi Town is part of Kuancheng District, Changchun City, Jilin Province, and is administered by Dehui City.

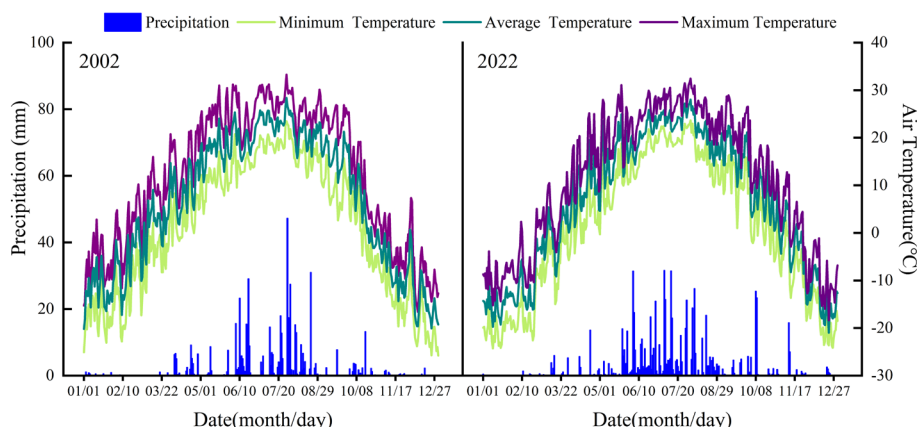


Figure 2. Air temperature and precipitation in the experimental sites of 2002 and 2022.

2.2. Experimental Design

The experimental sites (Table 1) had been under continuous corn cultivation for several years prior to 2002. Before 2002, these experimental sites had undergone at least five years of rotary tillage. Starting from 2002, some plots were complete randomly selected from sites to implement deep plowing and no-tillage methods, and this practice continued until 2022. To facilitate the full mechanization of corn production, this study employed a regional experimental design arranged randomly. Three representative tillage measures were implemented as follows:

(1)RT(Rotary tillage with stubble returning, conventional tillage method). After corn harvest, all above-ground straw was packed and removed. The field was then rotary-tilled twice to a depth of 10-15 cm, with stubble incorporated at 0-12 cm. The soil was leveled and compacted, leaving the surface exposed. Conventional sowing was performed in spring.

(2)PT(Plow tillage with straw returning). After harvest, straw was crushed and spread on the surface, followed by moldboard plowing to a depth of 30-35 cm. Straw was fully buried at 20-30 cm, leaving the surface exposed. Seeds were sown the following spring after heavy harrowing.

(3)NT(No-tillage with straw mulching). About 30 cm of stubble was retained, and crushed straw was evenly spread on the surface (incorporated at 0-3 cm). No tillage was performed throughout the year, and sowing was done with a no-till seeder the following spring.

A systematic sampling design with sufficient sample size and uniform spatial coverage was adopted to account for spatial heterogeneity, thereby ensuring representative parameter estimates and minimizing bias from spatial variation.

Fertilization followed conventional local farming practices, and a locally adapted corn cultivar was used. The chemical fertilizer application amount of N is 220 kg·hm⁻², P is 44 kg·hm⁻², and K is 60 kg·hm⁻². No organic fertilizer is applied.

Sowing and harvesting typically occurred in mid-May and early October, respectively. Soil properties under the three tillage measures across the 12 sampling sites, measured after the autumn harvest in 2002 and 2022, are shown in Table 2.

Table 2. Soil properties of three tillage measures after harvesting in 2002 and 2022.

Year	Tillage methods	pH	SOC (g·kg ⁻¹)	DOC (mg kg ⁻¹)	CEC (cmol·kg ⁻¹)	TN (g·kg ⁻¹)	TP (g·kg ⁻¹)	Clay <0.002 (mm)	Fe(III)/Fe(II)
2002	RT	6.32	17.25	122.53	17.10	2.12	1.33	38.58	12.72
	PT	6.91	13.72	97.41	22.05	1.90	1.20	34.95	10.11
	NT	7.23	16.83	119.55	21.07	2.18	1.41	35.80	12.41
2022	RT	6.08	14.07	99.94	25.07	3.17	0.53	37.79	10.37
	PT	5.42	13.55	96.24	24.48	2.48	0.44	35.66	9.99
	NT	6.13	15.85	112.59	26.36	2.46	0.42	36.11	11.69

SOC, Soil organic carbon; DOC, Dissolved organic carbon; CEC, Cation exchange capacity; TN, Total nitrogen; TP, Total phosphorus. RT, rotary tillage; PT, plow tillage; NT, no-tillage.

2.3. Soil Sampling and Analysis

The sampling was conducted during the maize harvest period in October 2002 and 2022. For each plot, five sampling points were established along an "S"-shaped transect. At each point, approximately 1 kg of soil was collected in the depth of 0-20 cm. All subsamples from a single plot were combined and thoroughly homogenized to form a bulk sample. After air-dried, the soil samples were sieved (0.25 mm mesh) for determination of soil pH, SOC, Ca²⁺ and various forms of Fe/Al oxides. The remaining un-sieved, air-dried soil was reserved for the extraction of organo-mineral complexes.

Soil pH was determined by the potentiometric method (water: soil ratio of 5:1) with a pH meter (PHS-3C, Leici, China). The content of dissolved organic carbon (DOC) was determined by the potassium dichromate external heating method [40]. Kjeldahl method was developed to determine total nitrogen (TN). The determination of the total phosphorus (TP) was carried out using the HClO₄-H₂SO₄ method. The cation exchange capacity (CEC) was determined by the method of ammonium acetate exchange-atomic absorption-flame photometry. After dispersed by the ultrasonic method (at 21.5 kHz, 300 mA, for 30 minutes), the <2 μm clay particles were extracted based on the settling time determined by the Stoke principle [41]. The determination of Fe (II) and Fe (III) was carried out using the phenanthroline colorimetric method [42].

In order not to introduce chemical reagents, organo-mineral complexes were isolated from the soil using a modified physical fractionation procedure according to previous reports [12,43]. Specifically, air-dried soil samples were placed into a 1000 mL glass cylinder and mixed with distilled water to form a suspension at a 4% concentration (w/v), which was then soaked for 24 hours. The suspension was stirred vertically for 1 minute using a perforated-plate stirrer at a rate of 10 up-and-down strokes, and then let stand. According to Stokes' law, the fraction with particle size of < 10 μm was collected by siphoning the upper 10 cm of the suspension after a settling time of 4 minutes and 15 seconds at 25 °C. This siphonation process was repeated until the entire suspension was processed. The extracted fraction was the organo-mineral complex of G0 fraction.

Following the extraction of the G0 fraction, the remaining suspension was repeatedly rinsed with a 1 M sodium chloride solution until the rinsate no longer showed a detectable reaction to calcium reagent. Distilled water was then added to bring the volume back to the mark on the glass cylinder. The organo-mineral complexes corresponding to the G1 fraction were subsequently collected by repeating the same siphonation procedure as described for the G0 fraction.

After the extracting of the G1 fraction, the remaining soil residue was transferred to a 200 mL beaker and subjected to ultrasonic dispersion using a JY92-11 ultrasonic disintegrator operating at 21 kHz and 200 W for 10 minutes. The G2 fraction was then collected by repeating the same siphonation procedure as used for the G0 fraction. The resulting suspensions from the extractions of three fractions of organo-mineral complexes were concentrated by natural sedimentation and subsequent centrifugation. The collected wet samples were air-dried, weighed, and sieved (0.15 mm) for further analysis. The organic carbon content in both the organo-mineral complexes and the bulk soil was determined by the potassium dichromate external heating method [40].

According to the gel dispersion classification method, exchangeable Ca²⁺ is specifically associated with the G1 fraction of organo-mineral complexes, but not with the G0 or G2 fraction. To quantify this, the sodium chloride rinsate collected during the extraction of the G1 fraction was used to determine the content of exchangeable Ca²⁺. Measurements were performed at a wavelength of 422.7 nm using an ultraviolet spectrophotometer (SP-723, Spectrum, China). Exchangeable Ca²⁺ in the bulk soil was measured by the same method.

According to Inda et al. [44] and Chang et al. [16], free iron (Fe_d) and aluminium (Al_d) were extracted from bulk soil samples using dithionite-citrate-bicarbonate method. Briefly, 0.3 g pretreated soil was combined with 0.5 g of sodium dithionite, 20 mL of 0.3 M sodium citrate, and 2.5 mL of 1 M

sodium bicarbonate in a 50 mL centrifuge tube. The mixture was then shaken continuously for 16 h. After centrifugation, the supernatant was collected and transferred into a 200 mL volumetric flask. This extraction procedure was repeated twice, and all supernatants were combined for subsequent analysis.

Amorphous iron (Fe_o) and aluminium (Al_o) were extracted using the acidammonium oxalate method [45]. Briefly, 0.5 g pretreated soil was mixed with 50 mL of 0.2 M ammonium oxalate solution (pH 3.0). The suspension was shaken continuously for 2 h in the dark and subsequently centrifuged at 4000 g for 10 min.

Complexed iron (Fe_p) and aluminium (Al_p) were extracted using sodium pyrophosphate at pH 8.5 [46]. Briefly, 0.5 g of pretreated soil was mixed with 10 mL 0.2 M sodium pyrophosphate. After shaking for 2 h, the mixture was centrifuged at 2000 g for 10 min and filtered. The concentrations of Fe_d and Al_d , Fe_o and Al_o , Fe_p and Al_p were measured at 520 nm by ultraviolet spectrophotometer (SP-723, Spectrum, China).

The organic carbon content in the organo-mineral complex per unit of soil, as well as the crystalline iron (Fe_c) and aluminium (Al_c) oxide contents in the bulk soil, were calculated using the following equations:

$$\text{Organic carbon content per unit soil organo-mineral complex} = \frac{\text{Organic carbon content of the organo-mineral complex} \times \text{Content of the organo-mineral complex in soil}}{\text{Content of the organo-mineral complex in soil}} \quad (1)$$

$$Fe_c = Fe_d - Fe_o \quad (2)$$

$$Al_c = Al_d - Al_o \quad (3)$$

2.4. Statistical Analyses

A one-way analysis of variance (ANOVA) followed by the least-significant difference (LSD) test was conducted using SPSS 17.0 (IBM, US) to evaluate the statistically significant ($p < 0.05$) differences in contents of organo-mineral complex, organic carbon in organo-mineral complex, exchangeable Ca^{2+} in the G1 fraction and in bulk soil, and Fe/Al oxide in bulk soil among treatments. Relationships between the organo-mineral complex, and soil properties under different tillage measures were assessed using Pearson correlation analysis. Furthermore, redundancy analysis (RDA) was performed with Canoco 5 to examine the relationships between p soil properties and the organo-mineral complex under three tillage measures, eliminated the variables with high collinearity. To ensure comparability across variables, both the response and explanatory variables were standardized.

3. Results and Discussion

3.1. The Effect of Tillage Measures on the Content of Each Fraction of Organo-Mineral Complexes

The content of each fraction of organo-mineral complex in soil per unit mass under different tillage measures is presented in Table 3. In 2002 and 2022, the contents of the soil organo-mineral complexes under the RT, PT, and NT treatments all followed the same decreasing order. Compared to 2002, the organo-mineral complex content exhibited a decline in the G1 and G2 fractions, and an increase in the G0 fraction and G0+G1+G2 across all three tillage measures in 2022.

By 2022, compared to the RT treatment, the G0 fraction content increased by 2.16% under PT treatment but decreased by 6.39% under NT treatment. Notably, the NT treatment resulted in significantly lower G0 content than both PT and RT treatments ($p < 0.05$). For the G1 fraction, PT and NT treatments led to increases of 2.05% and 26.72%, respectively, relative to RT treatment. Notably, the G1 content under NT treatment was significantly higher than under PT and RT treatments ($p < 0.05$), while no significant difference was observed between RT and PT treatments. As for the G2 fraction, its content increased by 9.16% under PT treatment but decreased by 18.53% under NT

treatment compared to RT treatment; however, no significant difference between NT treatment and RT treatment. The G0 fraction represents the combination of free mineral particles and micro-aggregates (< 10 μm) [12]. No-tillage with straw mulching favors the formation of larger aggregates over smaller ones [22,23], which may explain the reduced G0 content under NT treatment. In contrast, years of ploughing in PT treatment caused considerable soil disturbance and structural degradation, resulting in a higher G0 content compared to RT and NT treatments.

It has been demonstrated that with increasing straw addition, the content of the non-water-stable G0 fraction decreases, while that of the water-stable G1 fraction increases, indicating a transformation of organo-mineral complexes from non-water-stable to water-stable forms [47,48]. Both G1 and G2 fractions are water-stable complexes. Specifically, the G1 fraction consists of complexes formed by calcium and humus, whereas the G2 fraction is composed of complexes associated with Fe/Al oxides and humus [34]. For the content of G1+G2 organo-mineral complex, the order was NT>PT>RT (in 2022), suggesting that the NT treatment promoted the transformation of organo-mineral complexes from non-water-stable to water-stable. Moreover, compared to RT and PT treatments, the NT treatment was superior in minimizing soil structural disturbance and promoting structural stability.

For all three tillage measures, the content of organo-mineral complexes followed the order G1>G0>G2. The total content of organo-mineral complexes under NT treatment was higher than that under RT and PT treatments. Among three fractions of organo-mineral complexes, the G1 fraction was absolutely dominant, accounting for more than 50% of the total organo-mineral complexes content, followed by the G0 fraction (34.63%), while the G2 fraction constituted only 4.42%. This distribution can be attributed to the pedogenic process of black soil, in which humic acid readily combines with inorganic minerals such as Ca and Fe/Al oxides, leading to the notably high proportion of the G1 fraction. Consistent with this, the G1/G2 ratio exceeded 10 under all three tillage measures in this study.

The formation of soil organo-mineral complexes depends not only on the abundance of ionic binders but also on the stability of the resulting bonds. Although Fe/Al bonds are considerably more stable than Ca bonds, the content of the G2 fraction (associated with Fe/Al) remained notably low across all three tillage measures. This observed distribution may, however, deviate from the actual situation, as the sequential extraction method defined organo-mineral complexes operationally rather than functionally. Potential errors during the separation of organo-mineral complexes with different binding states could also influence the results. Moreover, a substantial portion of Fe/Al oxides tends to precipitate in hydroxylated forms, which may further limit their availability for the formation of the G2 fraction.

Table 3. Effect of tillage measures on the concentration of each organo-mineral complexes in black soil.

Year	Tillage measures	Content of each complex in unit soil ($\text{g}\cdot\text{kg}^{-1}$)					Percentage of each complex (%)			Ratios of content of each complex (%)		
		G0	G1	G2	G1+G2	$\frac{G_0+G_1}{G_2}$	G0	G1	G2	G0/G1	G0/G2	G1/G2
2002	RT	105.15±14.3 2Bb	311.50±12.7 8Aa	21.64±1.52 Aa	333.14	438.29	23.81	71.17	5.02	0.34	5.06	14.99
	PT	89.64±6.30 b	311.74±3.93 Aa	22.56±1.92 Aa	334.30	423.94	21.15	73.55	5.29	0.29	4.68	16.25
	NT	142.95±12.3 1Ba	306.93±4.35 Aa	17.13±2.14 Aa	324.05	467.00	30.67	65.63	3.69	0.47	8.43	18.21
2022	RT	208.38±15.4 6Aa	221.53±7.66 Bb	20.03±1.20 Aab	241.56	449.94	46.31	49.23	4.45	0.94	10.4	11.06
	PT	212.88±8.36 Aa	226.06±5.11 Bb	21.87±1.05 Aa	247.93	460.81	46.2	49.06	4.74	0.94	9.74	10.34
	NT	195.06±7.76 Aa	280.71±3.91 Aa	16.32±1.55 Ab	297.03	492.09	39.64	57.04	3.32	0.40	11.95	17.20

G0, G1 and G2 represent the organo-mineral complex of G0, G1 and G2 fractions respectively. RT, rotary tillage; PT, plow tillage; NT, no-tillage. Different uppercase letters indicated significant

difference between different years for the same tillage measure at 0.05 level according to LSD test. Different lowercase letters indicated significant difference among tillage measures in the same year at 0.05 level according to LSD test.

3.2. The Influence of Tillage Measures on the Content of Organic Carbon in Each Fraction of Organo-Mineral Complexes

The organic carbon content per unit mass of each organo-mineral complex reflects its capacity on carbon sequestration. Among the three fractions, the G2 fraction exhibited the highest organic carbon content (averaging 61.00 g·kg⁻¹), which was approximately twice that of the G0 and G1 fractions (Figure 3a, 3c). The organic carbon contents in both the G0 and G1 fractions were significantly lower than that in the G2 fraction. Additionally, the G1 fraction showed a slightly higher average organic carbon content (25.60 g·kg⁻¹) compared to the G0 fraction (23.43 g·kg⁻¹). These results are consistent with previous studies indicating that the G2 fraction contains the highest organic carbon content among all organo-mineral complex fractions [49].

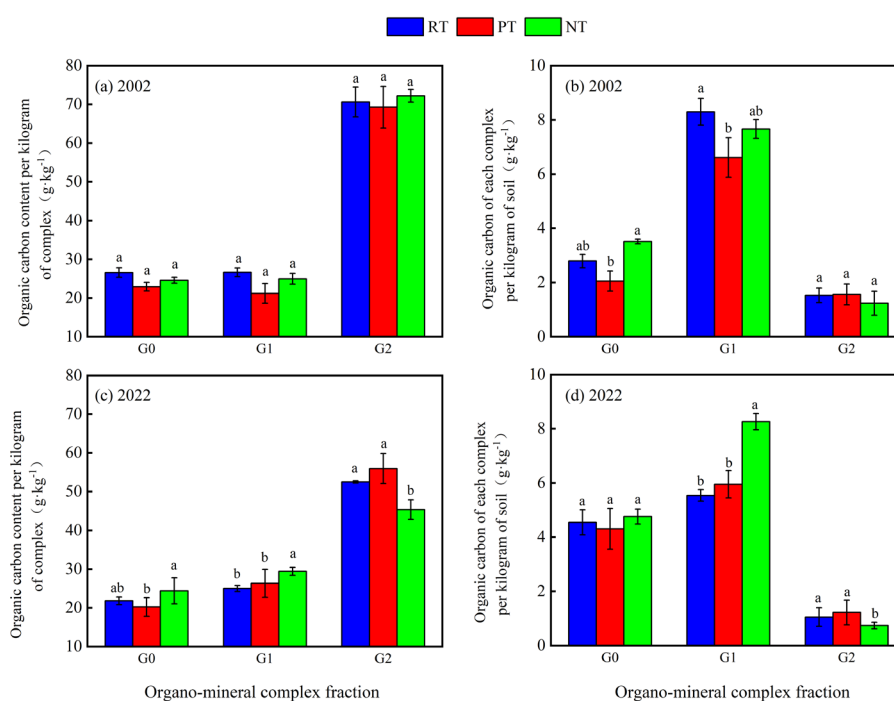


Figure 3. Effect of tillage measures on organic carbon distribution in organo-mineral complex. (a, c) Organic carbon content per kilogram of complex in 2002 and 2022; (b, d) Organic carbon content per kilogram of soil in 2002 and 2022. Different lowercase letters indicated significant difference among tillage measures at 0.05 level according to LSD test. The error bars represent the standard error. G0, G1 and G2 represent the organo-mineral complex of G0, G1 and G2 fractions respectively. RT=Rotary tillage; PT=Plow tillage; NT=No-tillage.

Compared with the RT treatment, the NT treatment increased the organic carbon content in the G0 and G1 fractions by 11.81% and 11.78%, respectively, in 2022 (Figure 3b, 3d). In contrast, the PT treatment led to increases of 5.35% and 6.54% in the G1 and G2 fractions, respectively, relative to RT treatment. However, in 2002, except for the G2 fraction under the NT treatment, the organic carbon content per unit mass of each organo-mineral complex decreased relative to RT treatment.

The organic carbon content in the organo-mineral complex is influenced by both the abundance of inorganic minerals and their surface binding capacity. Calcium (Ca) is known to effectively bind fresh organic matter, whereas aluminum (Al) shows a stronger affinity for humic and fulvic acids, though it exhibits a relatively weak stabilizing effect on fresh organic residues [50,51]. From the perspective of the time and conditions when straw reached complete humification in the soil, in this

study most of the organic matter produced by the decomposition of straw in soil of the NT treatment belonged to fresh organic matter, which made the bonding effect of Ca prominent and increased the content of organic carbon in the G0 and G1 fraction significantly. In terms of the bonding mechanisms between organic matter and soil minerals, the transformation and condensation of humus from G0 to G1 and further to G2 involve progressively stronger interactions and higher stability. Although both G1 and G2 were considered water-stable organo-mineral complexes, they differed in the degree and strength of organic-mineral bonding.

While soil acidification-driven base cation (e.g., Ca^{2+} and Mg^{2+}) leaching compromises MAOM formation and stability, the concurrent solubility of Al^{3+} and Fe^{3+} promotes carbon stabilization via organo-mineral complexes. We propose that under global nitrogen enrichment, a ubiquitous trade-off governs the balance between cation-bridged carbon depletion and the formation of new organo-mineral complexes. This trade-off represents a previously underexploited yet potentially critical mechanism underpinning MAOM pool persistence [52].

3.3. The Contribution of Each Fraction of Organo-Mineral Complexes Under Different Tillage Measures to Soil Organic Carbon Fixation

The organic carbon content associated with each fraction of organo-mineral complex per unit mass of soil reflects its contribution to overall SOC retention. Across the three tillage measures, the G1 fraction contained significantly more organic carbon (avg. $7.06 \text{ g}\cdot\text{kg}^{-1}$) than the G0 and G2 g fractions (Figure 3). This was primarily attributed to its higher mass proportion in the soil relative to the other two fractions of organo-mineral complexes. The G0 fraction also showed a higher organic carbon content (avg. $3.66 \text{ g}\cdot\text{kg}^{-1}$) than the G2 fraction (avg. $1.22 \text{ g}\cdot\text{kg}^{-1}$).

Although the G2 fraction exhibited a higher organic carbon content per unit mass of complex than the G0 and G1 fractions (Figure 3a, 3c), its content per unit mass of soil was considerably lower (Table 3). This explained the reduced overall organic carbon retention associated with the G2 fraction at the soil scale. The effects of tillage measures on the organic carbon content of the organo-mineral complex per unit soil mass varied significantly across organo-mineral complex fractions. In the G0 fraction, no significant differences in soil organic carbon content were observed among tillage practices in 2022. However, in 2002, the PT treatment showed significantly lower organic carbon content compared to the NT treatment. In the G1 fraction during 2022, both NT and PT treatments resulted in higher organic carbon content than the RT treatment -by 7.51% and 49.24%, respectively. In contrast, in 2002, the organic carbon content of NT and PT treatments were lower than RT treatment by 7.64% and 20.33%, respectively. In the G2 fraction, no statistically significant differences were detected among the tillage treatments in either 2002 or 2022 (except NT). The results indicated that although changes in tillage practices may lead to a short-term reduction in the organic carbon content of organo-mineral complexes, their long-term impacts are likely to differ.

In terms of the contribution rate to soil carbon sequestration, the G1 fraction of organo-mineral complexes was the most significant, accounting for 39.35%-52.12%, nearly half of the total (Figure 4). It was followed by the G0 fraction (14.99%-32.32%), while the G2 fraction contributed the least, ranging only from 4.67% to 11.40%. Compared to the RT treatment, both PT and NT treatments reduced the contribution of the G0 fraction but enhanced that of the G1 fraction, with NT treatment showing the most pronounced increase in 2022. The sum organic carbon content of the three complex fractions (G0, G1, and G2) accounted for 73.17% to 86.82% of the total organic carbon in bulk soil, indicating that organo-mineral complexes play a dominant role in SOC sequestration. The remaining portion (approximately one-fifth) of organic carbon was not associated with these complexes, suggesting the presence of other carbon forms or stabilization mechanisms.

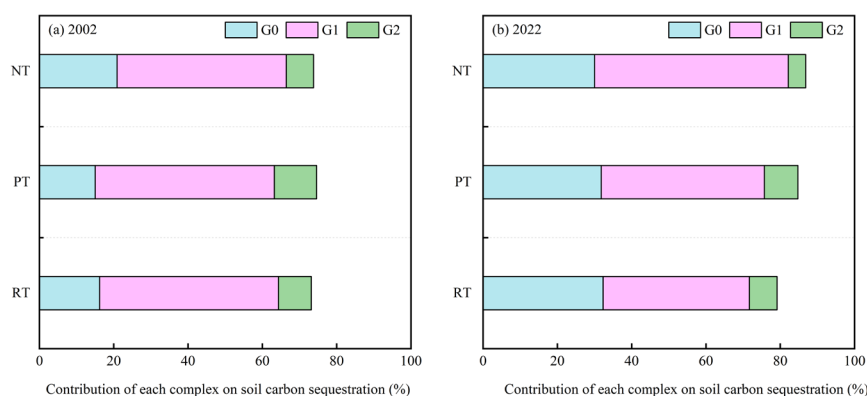


Figure 4. Contribution of each organo-mineral complex to soil carbon sequestration under three tillage measures. G0, G1 and G2 represent the organo-mineral complex of G0, G1 and G2 fractions respectively. RT=Rotary tillage; PT=Plow tillage; NT=No-tillage.

The total mass of the three organo-mineral complexes fractions accounted for 42.39% to 49.21% of the soil mass (Table 3), less than 50%, yet they contained nearly 80% of the total soil organic carbon. This clearly demonstrated the crucial role of organo-mineral complexes in sequestering organic carbon and enhancing soil fertility. In studies on organo-mineral complexes, the concept of “organo-mineral recombination degree” has been proposed, defined as the percentage of organic carbon present in the organo-mineral complex relative to the total soil organic carbon. A higher percentage indicates a greater degree of recombination [53,54]. The results of this study indicated that both NT and PT treatments could effectively increase this recombination degree compared to RT treatment. In particular, the NT treatment was the most conducive to the transformation of soil organic carbon into stable organo-mineral complexes.

3.4. Characteristics of Exchangeable Calcium Ions and Fe/Al Oxides Under Different Tillage Measures

The association of organic carbon with reactive minerals through the formation of organo-mineral complexes represents a key mechanism for long-term soil carbon storage [11]. Cementing agents such as Ca^{2+} , Fe/Al oxides play crucial roles in this process [55,56]. In support of this, Huang et al. [57] reported that over 47.1% of SOC was bound to Fe/Al oxides.

In 2002, no significant difference was observed in the exchangeable Ca^{2+} content across the three tillage practices (Figure 5a). In 2022, the NT treatment showed significantly higher exchangeable Ca^{2+} content than both the RT and PT treatments, by 17.35% and 24.16%, respectively (Figure 5b). No significant difference was detected between the RT and PT treatments in that year. The proportion of exchangeable Ca^{2+} in G1 fraction relative to exchangeable Ca^{2+} in the bulk soil were 36.62%, 37.64% and 39.67% respectively, under RT, PT, and NT treatments. The lower exchangeable Ca^{2+} content in PT and RT treatments may be attributed to frequent soil disturbance, which promoted Ca^{2+} leaching. Furthermore, soil acidification and Ca^{2+} depletion formed a feedback cycle (Table 2). Acidification accelerated the loss of alkaline Ca^{2+} , while the reduction of Ca^{2+} and other alkaline substances further exacerbated soil acidification [58].

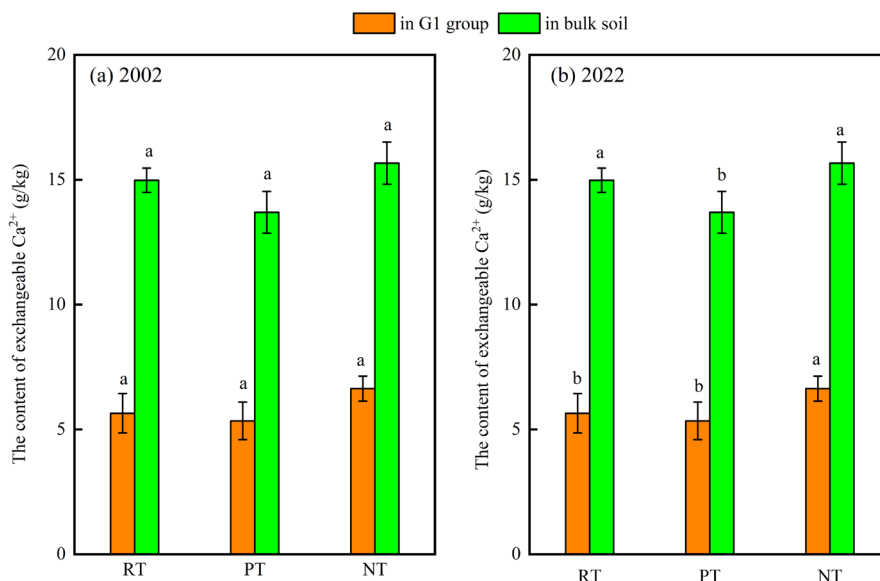


Figure 5. Effects of tillage measures on organo-mineral complexes exchangeable Ca²⁺ in 2002 and 2022. Different lowercase letters indicate significant difference among tillage measures at 0.05 level according to LSD test. The error bars represent the standard error. RT = Rotary tillage; PT = Plow tillage; NT = No-tillage.

Relative to 2002, Fe_d and Fe_c levels in 2022 exhibited a consistent decline across all tillage measures (RT, PT, and NT), while Fe_o and Fe_p showed an overall increasing trend over the same period (Figure 6a, 6c). A similar pattern was observed for aluminum oxides, with Al_a and Al_c, as well as Al_o and Al_p, following analogous trends to their iron oxide counterparts (Figure 6b, 6d). Compared with RT in 2002, PT exhibited an overall increase in the content of both iron and aluminum oxides. By 2022, however, this pattern had shifted: free and crystalline forms of these oxides decreased, whereas amorphous and complexed forms increased under PT treatment. In contrast, the NT treatment displayed a largely consistent trend relative to RT treatment in both years, characterized by an increase in free and crystalline forms and a decrease in amorphous and complexed forms.

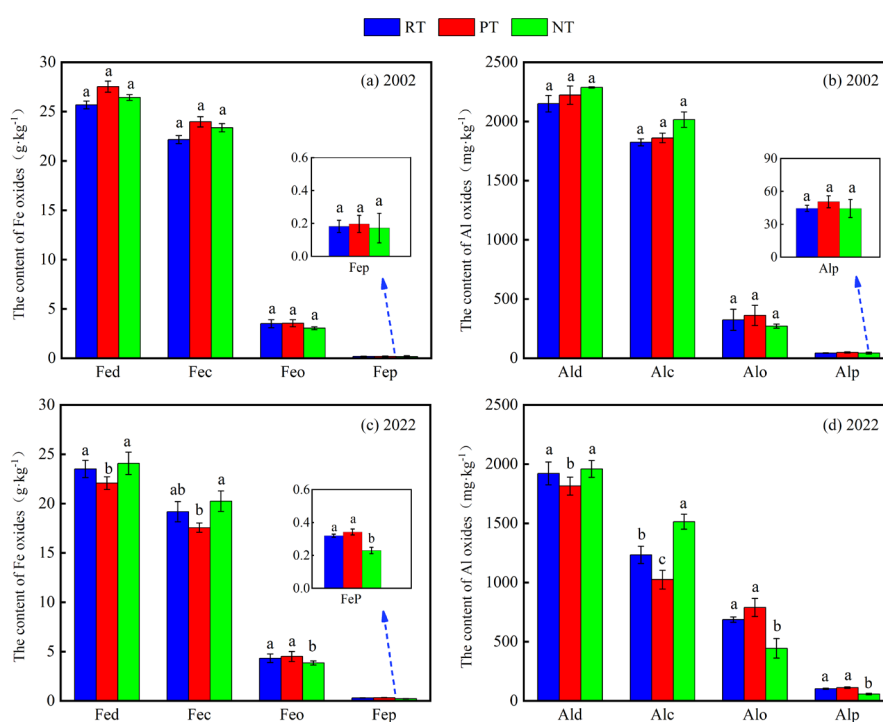


Figure 6. Effects of tillage measures on organo-mineral complexes various forms of iron and aluminum oxides. (a) Iron oxides in 2002; (b) Aluminum oxides in 2002; (c) Iron oxides in 2022; (d) Aluminum oxides in 2022. Different lowercase letters indicate significant difference among tillage measures at 0.05 level according to LSD test. The error bars represent the standard error. RT = Rotary tillage; PT = Plow tillage; NT = No-tillage.

This pattern suggested that tillage practices, particularly repeated soil aeration, may deplete reactive Fe and Al pools. Fe and Al minerals play a crucial role in stabilizing soil organic carbon by adsorbing or co-precipitating with organic matter to form refractory organo-mineral complexes [16,59]. These complexes function as effective “rusty sinks,” enhancing SOC stability through strong chemical bonds and physical protection, thereby representing a key mechanism for SOC storage [60,61].

A decline in soil acidity not only accelerates the loss of exchangeable Ca^{2+} but also influences the content of various Fe/Al oxide forms [16]. Further soil acidification below pH 6-7 increases the solubility of Fe/Al oxides, promoting their activation [60]. No-tillage practices help retain soil moisture and reduce the soil redox potential [62]. No-tillage effectively alleviated the decline in exchangeable Ca^{2+} in the G1 fraction and suppressed the transformation of crystalline Fe/Al oxides into amorphous and complex forms. It also limited the transition from the G1 fraction to the G0 fraction (Table 3). Thus, no-tillage aids in preserving soil structure and improving the stability of SOC.

3.5. Influencing Factors of Organo-Mineral Complexes

Redundancy analysis (RDA) showed that 78.45% of the variation in tillage measures could be explained by the chemical properties and fraction distribution of organo-mineral complexes in the black soil (Figure 7a). The organo-mineral complexes in the G0 and G1 fractions formed acute angles with clay, and an obtuse angle with the G2 fraction, indicating that the contents of organo-mineral complexes were largely dependent of clay. The angles between SOC, DOC, Fe(III)/Fe(II), exchangeable Ca^{2+} in the G1 fraction, and the organo-mineral complexes in fractions of G0 and G2 were close to 90° , indicating that the contents of the G1 and G2 fractions were largely independent of these factors. In contrast, the organo-mineral complexes in the G1 fraction formed acute angles with exchangeable Ca^{2+} , SOC, free and crystalline Fe/Al oxides, pH, and TP, suggesting a strong positive correlation ($p < 0.01$). On the other hand, they exhibited obtuse angles with amorphous and complexed Fe/Al oxides, implying a strong negative correlation ($p < 0.01$). The G0 fraction displayed opposite trends. These results indicated that exchangeable Ca^{2+} in the G1 fraction along with pH, clay, TP, and Fe/Al oxides, were key environmental factors driving the transformation among the G0, G1, and G2 fractions of organo-mineral complexes.

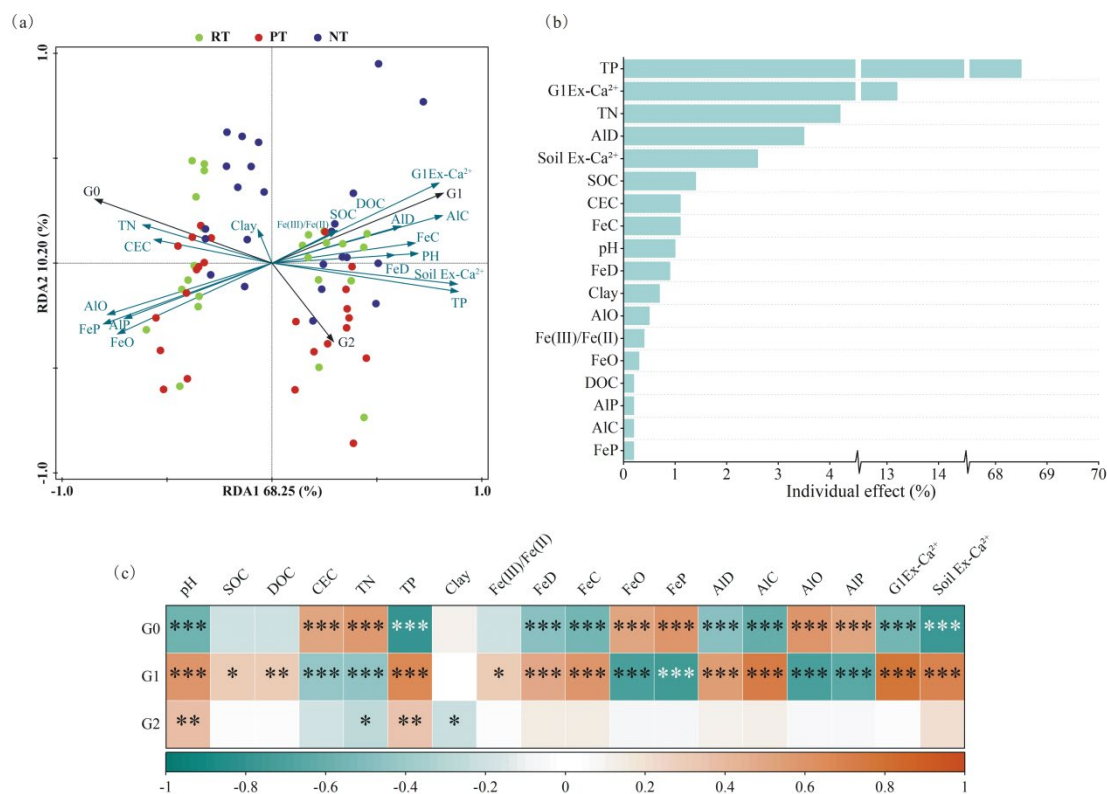


Figure 7. Relationship among tillage measures, organo-mineral complexes fractions, and soil properties. (a) Redundancy analysis and (b) individual effect of chemical properties on organo-mineral complexes fractions. (c) Pairwise Pearson's correlation analysis of organo-mineral complexes fractions, and soil properties. The color of the boxes indicates the strength and sign of the correlation, “*”, “**” and “***” indicate significant correlations at $P < 0.05$, $P < 0.01$ and $P < 0.001$, respectively. G0, G1 and G2 represent the organo-mineral complex of G0, G1 and G2 fractions respectively. SOC, Soil organic carbon; DOC, Dissolved organic carbon; CEC, Cation exchange capacity; TN, Total nitrogen; TP, Total phosphorus. G1Ex-Ca²⁺ represent the exchange Ca²⁺ in G1 fraction. FeD, Feo and FeP represent free, crystalline, amorphous and complex iron oxides respectively. AID, AIC, AIO and AIP represent free, crystalline, amorphous and complex aluminum oxides respectively.

The individual effects of exchangeable Ca²⁺, TN AID, and SOC on each organo-mineral complex fraction (G0, G1, and G2) under tillage measures were relatively high, ranging from 1.1% to 10.4% (Figure 7b). Notably, exchangeable Ca²⁺ in the G1 fraction alone accounted for 53.8% of the explained variance. Pairwise correlation analysis further revealed that Fe/Al oxides and exchangeable Ca²⁺ (both in the G1 fraction and in bulk soil) were positively correlated with the G0 and G1 fractions (Figure 7c), whereas pH, CEC, TN, and TP were negatively correlated with them. The results also suggested that pH, TN, TP and clay were only negatively correlated with the G2 fraction. These relationships can be interpreted in the context of the compositional nature of the organo-mineral complexes. The G0 fraction likely represents a mixture of sols-comprised of Fe/Al oxides coating the surface of individual clay particles or micro-aggregates-without further cementation. In contrast, the formation of water-stable complexes (G2 fraction) involves the transition of these Fe/Al oxide film sols into gels, followed by cementation upon drying and dehydration [63].

Fe/Al can act as a cationic bridge between phosphorus and SOC, thereby maintaining P availability. In addition, nitrogen leaching can lead to soil acidification, which in turn accelerates soil weathering and the solubilization of soil minerals [12]. Nanometer-scale investigation of soil organo-mineral interfaces reveals unambiguous evidence of Al-mediated organic matter association and Fe-bearing surface coatings on silicate clays and primary minerals [64]. Fe precipitates, and Fe-SOC co-precipitates under fluctuating redox conditions (Table 2).

Soil clay particle content is a key determinant of organic carbon content within soil MAOM. Owing to their high specific surface area, clay minerals effectively limit the exposure of SOC to enzymes and oxygen, thereby suppressing oxidative degradation. This physical protection mechanism enhances the storage and stability of organic carbon within organo-mineral complexes [51,65].

Since indicators such as pH, moisture content, and redox potential are difficult to control in field experiments, further research is required—using laboratory incubation experiment—on the transformation of Ca^{2+} , various forms of iron and aluminum oxides, and their sol-gel forms. Based on the characteristics of organo-mineral complexes, exchangeable Ca^{2+} in the G1 fraction, and Fe/Al oxides, along with their sol-gel transformation mechanisms [12,63,66], a preliminary model summarizing the potential transformation pathways among the G0, G1, and G2 fractions was proposed. The transformation process of organo-mineral complexes between G1 and G2 fractions was primarily governed by variations in hydrostable gels of Fe_c , Al_c , Fe_o , Al_o , Fe_p , and Al_p . The transformation process of the organo-mineral complexes between G0 and G2 fractions was affected by the changes in both the sols and hydrostable gels of Fe_o , Al_o , Fe_p , and Al_p . The transformation between the G0 and G1 fractions was influenced by changes in the content of exchangeable Ca^{2+} in the G1 fraction, as well as the hydrostable gels of Fe_c and Al_c and the sols of Fe_o , Al_o , Fe_p , and Al_p . Tillage practices likely intensify the redox cycling of Fe/Al, thereby accelerating the depletion of organo-mineral complexes. This phenomenon is consistent with previously observed lignin degradation under fluctuating redox conditions [67]. In less disturbed no-tillage (NT) soils, Fe/Al may not be significantly involved in organo-mineral complexes degradation.

4. Conclusions

The distribution of organo-mineral complexes and the associated organic carbon in black soil were affected by tillage measures. Compared to plow tillage (PT), both rotary tillage (RT) and no-tillage (NT) increased the content of organo-mineral complexes, the organic carbon stored within them, and overall soil carbon sequestration, with the NT treatment showing the most significant enhancement. This indicated that NT treatment caused the least soil disturbance and most effectively improved soil structure and organic carbon stability. Furthermore, exchangeable Ca^{2+} in the G1 fraction, pH, clay, TP, along with Fe/Al oxides, were identified as key factors governing the transformation among the G0, G1, and G2 fractions of organo-mineral complexes. The NT measure appeared to suppress Fe/Al redox cycling, thereby mitigating the degradation of organo-mineral complexes. These findings elucidate the mechanisms underlying the stabilization of soil structure and organic carbon, as well as the transformation dynamics among different fractions of organo-mineral complexes. This study provides a scientific basis for optimizing tillage practices, enhancing organic carbon retention, and promoting the sustainable use of black soil.

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References

1. Wang, W.J., Wang, W.P., Jia, H.L., Zhuang, J., Wang, Q. Effects of seed furrow liquid spraying device on sowing quality and seedling growth of maize. *Int J Agric Biol Eng.* 2019, 12, 68-74. <https://doi.org/10.25165/ijabe.20191202.3799>
2. Wang, Y.Y., Zhang, Y.G., Liu, Y.Y., Wang, L.B., Dong, Y.L. Effects of different tillage methods on soil properties and maize seedling growth in alternating wide and narrow rows rotation mode in the Songliao Plain of China. *Geoderma.* 2024, 452, 117120. <https://doi.org/10.1016/j.geoderma.2024.117120>
3. Liu, Z.J., Yang, X.G., Chen, F., Wang, E.L. The effects of past climate change on the northern limits of maize planting in Northeast China. *Clim. Change.* 2013, 117, 891-902. <https://doi.org/10.1007/s10584-012-0594-2>.
4. Sun, J.F., Chen, H.M., Wang, Z.M., Ou, Z.W., Yang, Z., Liu, Z., Duan, J.L. Study on plowing performance of EDEM low-resistance animal bionic device based on red soil. *Soil Tillage Res.* 2020, 196, 104336. <https://doi.org/10.1016/j.still.2019.104336>
5. Liu, S.Y., Zhang, X.P., Liang, A.Z., Zhang, J.B., Müller, C., Cai, Z.C. Ridge tillage is likely better than no tillage for 14-year field experiment in black soils: insights from a ¹⁵N-tracing study. *Soil Tillage Res.* 2018, 179, 38-46. <https://doi.org/10.1016/j.still.2018.01.011>.
6. Zhang, S.X., Li, Q., Zhang, X.P., Wei, K., Chen, L.J., Liang, W.J. Effects of conservation tillage on soil aggregation and aggregate binding agents in black soil of Northeast China. *Soil Tillage Res.* 2012,124, 196-202. <https://doi.org/10.1016/j.still.2012.06.007>
7. Liu, X.B., Han, X.Z., Herbert, S.J., Xing, B. Dynamics of soil organic carbon under different agricultural management system in the black soil of China. *Commun Soil Sci Plant Anal*, 2003, 34: 973–984. <https://doi.org/10.1081/CSS-120019103>
8. Zhang, X.Y., Sui, Y.Y., Zhang, X.D., Meng, K., Herbert, S.J. Spatial variability of nutrient properties in black soil of northeast China. *Pedosphere*, 2007, 17: 19–29. [https://doi.org/10.1016/S1002-0160\(07\)60003-4](https://doi.org/10.1016/S1002-0160(07)60003-4)
9. Liu, X.B., Zhang, X.Y., Wang, Y.X., Sui, Y.Y., Zhang, S.L., Herbert, S.J., Ding, G. Soil degradation: A problem threatening the sustainable development of agriculture in Northeast China. *Plant Soil Environ*, 2010, 56(2): 87–97. <https://doi.org/10.17221/155/2009-PSE>
10. Zhou, Z.H., Ren, C.J., Wang, C.K., Delgado-Baquerizo, M., Luo, Y.Q., Luo, Z.K., Du, Z.G., Zhu, B., Yang, Y.H., Jiao, S., Zhao, F.Z., Cai, A.D., Yang, G.H., Wei, G.H. Global turnover of soil mineral-associated and particulate organic carbon. *Nat Commun.* 2024, 15, 5329. <https://doi.org/10.1038/s41467-024-49743-7>
11. Chen, M.M., Zhang, S.R., Liu, L., Chang, B.J., Li, Y.Y., Ding, X.D. Organo-mineral complexes in soil colloids: Implications for carbon storage in saline-alkaline paddy soils from an eight-year field experiment. *Pedosphere.* 2024, 34, 97-109. <https://doi.org/10.1016/j.pedsph.2022.11.007>
12. Zhang N Y, Zhang X Z, Chen Y H, Matelele L A, Zhu P, Liu H F, Zhang X M, Gao H J, Feng G, Peng C, Zhang S X. Mineral-associated organic carbon promoted phosphorus accumulation in long-term fertilized black soil. *Pedosphere.* 2024. <https://doi.org/10.1016/j.pedsph.2024.10.003>
13. Gerrit, A., Kevin, E. M., Michael J. C., Cordula, V., Martin, W., Carsten, W. M. Unlocking complex soil systems as carbon sinks: multi-pool management as the key. *Nat Commun*, 2023,14:2967. <https://doi.org/10.1038/s41467-023-38700-5>
14. Huang, H.L., Zeng, G.M., Tang, L., Yu, H.Y., Xi, X.M., Chen, Z.M., Huang, G.H. Effect of biodelignification of rice straw on humification and humus quality by Phanerochaete chrysosporium and Streptomyces badius. *Int Biodeterior Biodegrad.* 2008, 61,331-336. <https://doi.org/10.1016/j.ibiod.2007.06.014>
15. Arcand, M.M., Knight, J.D., Farrell, R.E. Differentiating between the supply of N to wheat from above and belowground residues of preceding crops of pea and canola. *Biol Fertil Soils.* 2014,50,563-570. <https://doi.org/10.1007/s00374-013-0877-4>
16. Chang, Z.F., Tian, L.P., Li, F.F., Wu, M., Steinberg, C.E.W., Pan, B., Xing, B.S. Organo-mineral complexes protect condensed organic matter as revealed by benzene-polycarboxylic acids. *Environ Pollut.* 2020, 260, 113977. <https://doi.org/10.1016/j.envpol.2020.113977>
17. Li, F.F., Zhang, P.C., Wu, D.P., Xu, Y., Chen, F.Y., Chang, Z.F., Chu, G., Wang, L., Pan, B., Xing, B.S. Acid pretreatment increased lipid biomarker extractability: a case study to reveal soil organic matter input from rubber trees after long-term cultivation. *Eur J Soil Sci.* 2018, 69, 315-324. <https://doi.org/10.1111/ejss.12501>

18. Throckmorton, H.M., Bird, J.A., Monte, N., Doane, T., Firestone, M.K., Horwath, W.R. The soil matrix increases microbial C stabilization in temperate and tropical forest soils. *Biogeochemistry*. 2015, 122, 35-45. <https://doi.org/10.1007/s10533-014-0027-6>
19. Lützow, M.V., Gel-knabner, I.K., Ekschmitt, K., Matzner, E., Guggenberger, G., Marschner, B., Flessa, H. Stabilization of organic matter in temperate soils: mechanisms and their relevance under different soil conditions – a review. *Eur J Soil Sci*, 2006, 57: 426-445. <https://doi.org/10.1111/j.1365-2389.2006.00809.x>
20. Han, Z.X., Wu, X.P., Gao, H.Z., Jia, A.Y., Gao, Q.Q. Long-term conservation tillage increases soil organic carbon stability by modulating microbial nutrient limitations and aggregate protection. *Agronomy*. 2025, 15, 1571. <https://doi.org/10.3390/agronomy15071571>
21. Xu, P.D., Wu, J., Wang, H., Tang, S., Cheng, W.L., Li, M., Bu, R.Y., Han, S., Geng, M.J. Combined application of chemical fertilizer with green manure increased the stabilization of organic carbon in the organo-mineral complexes of paddy soil. *Environ Sci Pollut Res*. 2023, 30, 2676-2684. <https://doi.org/10.1007/s11356-022-22315-2>
22. Han, Z.X., Wu, X.P., Liang, A.Z., Li, S.P., Gao, H.Z., Song, X.J., Liu, X.T., Jia, A.Y., Aurore, D. Conservation tillage enhances the sequestration and iron-mediated stabilization of aggregate-associated organic carbon in Mollisols. *Catena*. 2024, 243, 108197. <https://doi.org/10.1016/j.catena.2024.108197>
23. Bayer, C., Mielniczuk, J., Giasson, E., Martin-Neto, L., Pavinato, A. Tillage effects on particulate and mineral-associated organic matter in two Tropical Brazilian soils. *Commun Soil Sci Plant Anal*. 2006, 37, 389-400. <https://doi.org/10.1080/00103620500446928>
24. Shabtai, I.A., Wilhelm, R.C., Schweizer, S.A., Höschen, C., Buckley, D.H., Lehmann, J. Calcium promotes persistent soil organic matter by altering microbial transformation of plant litter. *Nat Commun*. 2023, 14, 6609. <https://doi.org/10.1038/s41467-023-42291-6>
25. Khomo, L., Trumbore, S., Bern, C.R., Chadwick, O.A. Timescales of carbon turnover in soils with mixed crystalline mineralogies. *Soils*. 2017, 3, 17-30. <https://doi.org/10.5194/soil-3-17-2017>
26. Van De Vreken, P., Gobin, A., Baken, S., Van Holm, L., Verhasselt, A., Smolders, E., Merckx, R. Crop residue management and oxalate-extractable iron and aluminium explain long-term soil organic carbon sequestration and dynamics. *Eur J Soil Sci*. 2016, 67, 332-340. <https://doi.org/10.1111/ejss.12343>
27. Chen, C.M., Hall, S.J., Coward, E., Thompson, A. Iron-mediated organic matter decomposition in humid soils can counteract protection. *Nat Commun*, 2020, 11:2255. <https://doi.org/10.1038/s41467-020-16071-5>
28. Georgiou, K., Jackson, R. B., Vindušková, O., Abramoff, R.Z., Ahlström, A., Harden, J.W., Pellegrini, A.F.A., Wayne Polley, H., Soong, J.L., Riley, W.J., Torn, M.S. Global stocks and capacity of mineral-associated soil organic carbon. *Nat Commun*, 2022, 13: 3797. <https://doi.org/10.1038/s41467-022-31540-9>
29. Riedel, T., Zak, D., Biester, H., Dittmar, T. Iron traps terrestrially derived dissolved organic matter at redox interfaces. *Proc Natl Acad Sci USA*, 2013, 110(25):10101–10105. <https://doi.org/10.1073/pnas.1221487110>
30. Chen, C., Dynes, J.J., Wang, J., Sparks, D.L. Properties of Fe-organic matter associations via coprecipitation versus adsorption. *Environ Sci Technol*, 2014, 48:13751-13759. <https://doi.org/10.1021/es503669u>
31. Porras, R.C., Hicks Pries, C.E., McFarlane, K.J., Hanson, P.J., Torn, M.S. Association with pedogenic iron and aluminum: effects on soil organic carbon storage and stability in four temperate forest soils. *Biogeochemistry*, 2017, 133: 333-345. <https://doi.org/10.1007/s10533-017-0337-6>
32. Jia, N., Li, L., Guo, H., Xie, M.Y. Important role of Fe oxides in global soil carbon stabilization and stocks. *Nat Commun*, 2024, 15: 10318. <https://doi.org/10.1038/s41467-024-54832-8>
33. Zhao, B., Dou, A.M., Zhang, Z.W., Chen, Z.Y., Sun, W.B., Feng, Y.L., Wang, X.J., Wang, Q. Ecosystem-specific patterns and drivers of global reactive iron mineral-associated organic carbon. *Biogeosciences*, 2023, 20(23): 4761-4774. <https://doi.org/10.5194/bg-20-4761-2023>
34. Tyulin, A.T.H. The composition and structure of soil of organo-mineral gels and soil fertility. *Soil Sci*. 1938, 45, 343-358. <https://doi.org/10.1097/00010694-193805000-00010>
35. Hashimoto, H., Harada, T., Hara, M., Yumoto, T., Studies on the organo-mineral colloidal complexes of paddy soil III: G1 colloidal complexes of paddy soil as affected by drainage. *Soil Sci Plant Nutr*. 1959, 5(1):28-35. <https://doi.org/10.1080/00380768.1959.10430891>
36. Yan, C.S. Soil fertility research method. Beijing: Agriculture Publisher. 1988.

37. Edwards, A. P., Bremner, J. M. Microaggregates in soil. *Soil Sci*, 1967, 18 : 64-73. <https://doi.org/10.1111/j.1365-2389.1967.tb01488.x>
38. Bronick, C.J., Lal, R., Soil structure and management: a review. *Geoderma*. 2005,124, 3–22. <https://doi.org/10.1016/j.geoderma.2004.03.005>
39. Curaqueo, G., Miguel Barea, J., Acevedo, E., Rubio, R., Cornejo, P., Borie, F., Effects of different tillage system on arbuscular mycorrhizal fungal propagules and physical properties in a Mediterranean agroecosystem in central Chile. *Soil Tillage Res*. 2011,113, 11–18. <https://doi.org/10.1016/j.still.2011.02.004>
40. Lu, R.K. Analysis methods of soil and agricultural chemistry. Chinese Agricultural Science and Technology Press: Beijing, China. 2000.
41. Holtzapffel, T., Les minéraux argileux: Préparation, analyse diffractométrique et détermination. *Soc Géol Nord Publ*, 12, 1985, 15-43.
42. Li, Y., Yu, S., Strong, J., Wang, H.L. Are the biogeochemical cycles of carbon, nitrogen, sulfur, and phosphorus driven by the “Fe^{III}-Fe^{II} redox wheel” in dynamic redox environments? *J Soils Sediments*, 2012, 12(5): 683-693. <https://doi.org/10.1007/s11368-012-0507-z>
43. Lopez-Sangil, L., Rovira, P. Sequential chemical extractions of the mineral-associated soil organic matter: An integrated approach for the fractionation of organo-mineral complexes. *Soil Biol Biochem*. 2013, 62, 57-67. <https://doi.org/10.1016/j.soilbio.2013.03.004>
44. Inda, A.V., Torrent, J., Barrón, V., Bayer, C., Fink, J.R. Iron oxides dynamics in a subtropical Brazilian Paleudult under long-term no-tillage management. *Scientia Agricola*. 2013, 70, 48-54. <https://doi.org/10.1590/S0103-90162013000100008>
45. Barberis, E., Ajmone, M.F., Boero, V., Arduino, E. Aggregation of soil particles by iron oxides in various size fractions of soil B horizons. *Eur J Soil Sci*. 1991, 42, 535-542. <https://doi.org/10.1111/j.1365-2389.1991.tb00100.x>
46. Saidy, A.R., Smernik, R.J., Baldock, J.A., Kaiser, K., Sanderman, J., Macdonald, L.M. Effects of clay mineralogy and hydrous iron oxides on labile organic carbon stabilisation. *Geoderma*. 2012, 173-174, 104-110. <https://doi.org/10.1016/j.geoderma.2011.12.030>
47. Tisdall, J.M., Oades, J.M. Organic Matter and Water-stable Aggregates in Soils. *Eur J Soil Sci*. 2006, 33, 141-163. <https://doi.org/10.1111/j.1365-2389.1982.tb01755.x>
48. Liu, C., Lu, M., Cui, J., Li, B., Fang, C.M. Effects of straw carbon input on carbon dynamics in agricultural soils: a meta-analysis. *Glob Change Biol*. 2014, 20, 1366-1381. <https://doi.org/10.1111/gcb.12517>
49. Inoue, K., Zhao, L.P., Huang, P.M. Adsorption of humic substances by hydroxylaluminum-and hydroxylaluminosilicate-montmorillonite complexes. *SSSAJ*. 1990, 54, 1166-1172. <https://doi.org/10.2136/sssaj1990.03615995005400040042x>
50. Xiong, Y. Soil colloids (Volume 1). Beijing: Science Press, 1983, 343-344.
51. Kleber, M., Sollins, P., Sutton, R.A. A conceptual model of organo-mineral interactions in soils: self-assembly of organic molecular fragments into zonal structures on mineral surfaces. *Biogeochemistry*. 2007, 85, 9-24. <https://doi.org/10.1007/s10533-007-9103-5>
52. Tang, B., Rocci, K. S., Lehmann, A., Rillig, M. C. Nitrogen increases soil organic carbon accrual and alters its functionality. *Glob Change Biol*, 2023, 29:1971-1983. <https://doi.org/10.1111/gcb.16588>
53. Xu, J.M., Yuan, K.N. Study on organo-mineral complexes in soil: V. Distribution of organo-mineral complexes in zonal soils of China. *Acta Pedol Sin*. 1993, 30, 43-51. (In Chinese, with English Abstract).
54. Six, J., Bossuyt, H., Degryze, S., Denef, K. A history of research on the link between (micro) aggregates, soil biota, and soil organic matter dynamics. *Soil Tillage Res*. 2004, 79, 7-31. <https://doi.org/10.1016/j.still.2004.03.008>
55. Boudot, J.P., Hadj Brahis, A.B., Steiman, R., Seigle-Murandi, F. Biodegradation of synthetic organo-metallic complexes of iron and aluminum with selected metal to carbon ratios. *Soil Biology and Biochemistry*. 1989, 21, 961-966. [https://doi.org/10.1016/0038-0717\(89\)90088-6](https://doi.org/10.1016/0038-0717(89)90088-6)
56. Duchaufour, P. Dynamics of organic matter soils of temperate regions: action pedogenesis. *Geoderma*. 1976,15, 31-40. [https://doi.org/10.1016/0016-7061\(76\)90068-9](https://doi.org/10.1016/0016-7061(76)90068-9)

57. Huang, X.L., Kang, W.J., Guo, J.J., Wang, L., Tang, H.Y., Li, T.L., Yu, G.H., Ran, W., Hong, J.P., Shen, Q.R. Highly reactive nanomineral assembly in soil colloids: Implications for paddy soil carbon storage. *Sci. Total Environ.* 2020, 703, 134728. <https://doi.org/10.1016/j.scitotenv.2019.134728>
58. Rowley, M.C., Nico, P.S., Bone, S.E., Marcus, M.A., Pegoraro, E.F., Cristina, C., Kang, K., Bhattacharyya, A., Torn, M.S., Peña, J. Association between soil organic carbon and calcium in acidic grassland soils from Point Reyes National Seashore, CA. *Biogeochemistry.* 2023, 165, 91-111. <https://doi.org/10.1007/s10533-023-01059-2>
59. Li, Q., Hu, W.F., Li, L.F., Y.C. Interactions between organic matter and Fe oxides at soil micro-interfaces: Quantification, associations, and influencing factors. *Sci. Total Environ.* 2023,855,158710. <http://dx.doi.org/10.1016/j.scitotenv.2022.158710>
60. Lindsay, W.L. Chemical equilibria in soils. New York: John Wiley and Sons Ltd. 1979.
61. Che, M., Gong, Y., Xu, M., Kang, C., Lv, C., He, S., Zheng, J. Effects of elevation and slope aspect on the distribution of the soil organic carbon associated with Al and Fe mineral phases in alpine shrub-meadow soil. *Sci Total Environ.* 2021,753, 141933. <https://doi.org/10.1016/j.scitotenv.2020.141933>
62. Sey, B.K., Whalen, J.K., Gregorich, E.G., Rochette, P., Cue, R.I. Carbon dioxide and nitrous oxide content in soils under corn and soybean. *SSSAJ.* 2008, 72, 931-938. <https://doi.org/10.2136/sssaj2007.0093>
63. Zhang, B., Horn, R. Mechanisms of aggregate stabilization in Ultisols from subtropical China. *Geoderma.* 2001, 99, 123-145. [https://doi.org/10.1016/S0016-7061\(00\)00069-0](https://doi.org/10.1016/S0016-7061(00)00069-0)
64. Possinger, A.R., Zachman, M.J., Enders, A. Levin, B.D. A., Muller, D.A., Kourkoutis, L.F., Lehmann, J. Organo-organic and organo-mineral interfaces in soil at the nanometer scale. *Nat Commun*, 2020, 11, 6103. <https://doi.org/10.1038/s41467-020-19792-9>
65. Kaiser, K., Guggenberger, G. Mineral surfaces and soil organic matter. *Eur J Soil Sci*, 2003, 54: 219-236. <https://doi.org/10.1046/j.1365-2389.2003.00544.x>
66. Underwood, T.R., Bourg, I.C., Rosso, K.M. Mineral-associated organic matter is heterogeneous and structured by hydrophobic, charged, and polar interactions, *Proc. Natl. Acad. Sci. U.S.A.* 2024, 121 (46) :e2413216121. <https://doi.org/10.1073/pnas.2413216121>
67. Hall, S.J., Silver, W.L., Timokhin, V.I., Hammel, K.E. Lignin decomposition is sustained under fluctuating redox conditions in humid tropical forest soils. *Glob Change Biol.* 2015, 21, 2818-2828. <https://doi.org/10.1111/gcb.12908>

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