Organic carbon preservation promoted by aromatic compound-iron complexes through manure fertilization in red soil

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Abstract

Purpose This study was aimed at evaluating the effect of manure fertilization on organo-mineral complexes based on unfertilized (CK) and 5-year manure fertilization (M) in red soils (ultisols).

Materials and methods Sequential density fractionation was adopted to obtain particular organic matter (POM, $< 1.8 \text{ g cm}^{-3}$) and mineral-associated organic matter ($1.8\text{--}2.2 \text{ g cm}^{-3}$ (MOM_{1.8–2.2}); $2.2\text{--}2.6$ (MOM_{2.2–2.6}); > 2.6 (MOM_{> 2.6})), which were demineralized by hydrofluoric acid (HF). The HF-dissolved organic carbon (OC) was identified as mineral bound OC, and was characterized by comparing the band area of Fourier transform infrared spectroscopy before and after 10% HF demineralization.

Results and discussion Manure fertilization significantly increased the OC and nitrogen contents of bulk soil, POM, MOM_{1.8–2.2}, and MOM_{2.2–2.6}, but did not affect MOM_{> 2.6}. About 82.9–86.1% soil OC was retained by MOM_{1.8–2.2} and MOM_{2.2–2.6} dominated with iron (Fe) and aluminum oxides and phyllosilicates, in which HF-dissolved OC was significantly positively correlated with poorly crystalline (Fe_o) ($R^2 = 0.61$, $P < 0.01$) and organically complexed (Fe_p) Fe contents ($R^2 = 0.45$, $P < 0.05$). Moreover, the Fe_o and Fe_p contents were higher in M than in CK ($P < 0.05$), and significantly negatively correlated with the reduction of aromatic C (M had more reduction than CK) following HF demineralization in MOM_{1.8–2.2} and MOM_{2.2–2.6} ($P < 0.05$), indicating Fe bound OC was mainly aromatic C. The quartz and feldspar-dominated MOM_{> 2.6} was composed of aromatic C, amide N, and polysaccharides, not being affected by fertilization.

Conclusions Our results suggested that manure fertilization promotes organo-mineral association, particularly for aromatic compound-Fe complexes, contributing to OC preservation in red soils.

Keywords Hydrofluoric acid · Sequential density fractionation · Poorly crystalline mineral oxides · Stabilizing mechanism

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1 Introduction

Red soils widely distributed in the tropical and subtropical regions of South China are characterized by relatively lower productivity and organic carbon (OC), which caused by severe weathering (e.g., abundant rainfall and high temperature) and intensive farming practices (Zhang et al. 2009). Soil organic carbon (SOC) plays a crucial role in cropland productivity, and its content has been included among the parameters in the guidelines for soil quality (Reeves 1997; Tong et al. 2014). Therefore, understanding the mechanisms of SOC preservation and stabilization in the context of manure fertilization of red soils is pivotal to keep the stability of agroecosystem in this region.

SOC pool can be categorized into particulate organic matter (POM) and mineral-associated organic matter (MOM) according to their densities. These MOM are primarily important for long-term SOC preservation in soils because of these SOC absorbed around mineral having less accessible to microorganisms by forming a physical barrier between SOC and microorganisms (Torn et al. 1997; Lavalley et al. 2020). Interactions between minerals and OC are also affected by the soil mineral composition, fertilizer management, and climate condition (Six et al. 2002; Lal 2018). Red soil contains significant amounts of iron (Fe) and aluminum (Al) oxides and phyllosilicate minerals such as kaolinite, which play important roles in SOC preservation through the formation of MOM (Zhang et al. 2013; Bruun et al. 2010). Recent work indicated that the mineral oxides, especially amorphous or non-crystalline types, appear to more strongly interact with SOC based on cation bridges than just phyllosilicates (Mikutta et al. 2007). The capacity of mineral oxides to interact with SOC is controlled by soil physicochemical conditions (e.g., pH and redox potential) and exudates and metabolic compounds secreted from plant roots and microbes, whose activities are also affected by fertilization (Dakora and Phillips 2002; Colombo et al. 2014). Most studies found that manure fertilizer made more organic acids that act as complexing and reduction agents for mobilizing mineral, further promoting the production of non-crystalline types, which is beneficial for the formation of MOM (Yu et al. 2017; Huang et al. 2018). However, the formation of MOM between non-crystalline mineral oxides and specific organic compounds is still unclear. Wan et al. (2019) reported that Fe oxides were selectively associated with polysaccharides and aliphatic compounds in different types of agricultural soil. Zhao et al. (2016) considered that aromatic compounds were the main component of the organo-Fe complexes. Filimonova et al. (2016) also found that the allophane mainly imparts chemical protection to labile organic compounds, e.g., carbohydrates. Therefore, more accurately, evaluation of the contents and compositions of OC bound with different mineral species is needed to understand the dynamics and stabilization mechanisms of SOC following manure fertilization in red soil.

In recent years, many authors adopted the combination of physical and chemical fractionation methods to isolate these MOM effectively (Sleutel et al. 2009). Physical density fractionation can be used to isolate bulk soil into free POM (light fractions) and primary and secondary MOM (heavy fractions) depending on their different densities (Sollins et al. 2009; Wagai et al. 2015). The chemical method, such as the demineralization with hydrofluoric (HF) acid, has also been used to treat MOM for clarifying the binding mechanism between OC and mineral particles

(Helfrich et al. 2007). That is because enormous amounts of OC associated with minerals can be released during the HF demineralization procedure in metal oxide-rich soils, conversely, the non-mineral associated OC is almost unchanged (Rumpel et al. 2008). Sanderman et al. (2017) suggested that the HF demineralization procedure was best suited for characterizing OC that can be absorbed or precipitated with minerals, and the soil physicochemical environment should also be considered. To investigate the structures of MOM, several studies have physically isolated bulk soils into different density fractions, and then being demineralized with HF. Yeasmin et al. (2017a) suggested that HF demineralization was more suitable for analyzing the organic bands in the soil FTIR spectra than dry ashing and sodium hypochlorite oxidation because of the greatly increased organic bands without mineral interference spectra after HF treatment, and then concluded that the HF demineralization in conjunction with density fractions would be beneficial to study the structures of MOM through comparing the organic bonds of Fourier transform infrared (FTIR) spectroscopy before and after HF demineralization (Yeasmin et al. 2017a, b). Although these studies have used a wide range of soil types (i.e., ferralsols, luvisol, vertisol, and solonetz), few have made use of the above procedure to evaluate the manure fertilizer effects on the contents and compositions of MOM.

Considering all the above, this study was aimed at (i) determining the qualitative and quantitative distribution of MOMs, and (ii) investigating the specific organic functional groups bound with organo-mineral complexes following manure fertilization of red soils in southern China based on sequential density fractionation and HF demineralization.

2 Materials and methods

2.1 Sites and soil samples

Soil samples used in the present study were collected in mid-August 2018 from the Static Fertilization at Yueyang City, Hunan Province, in the Southwest China region (E 112° 44' 14", N 28° 57' 11") (Fig. 1), which was initiated in 2013 to study the effect of different amounts of swine manure on soil quality and environment. This site represents a typical agricultural region of subtropical China with red soil developed from quaternary red clay parent material, being classified as ultisols (Soil Survey Staff 2010). This area is affected by a warm and humid subtropical monsoon climate with annual precipitation of 1400 mm and annual average temperature of 17 °C. Prior to experiment, soil properties were pH 6.34, 8.02 g kg⁻¹ SOC, 1.05 g kg⁻¹ total nitrogen (N), 10.32 mg kg⁻¹ available P, and 246.7 mg kg⁻¹ available potassium (K). A full randomized

Fig. 1 Location of the study area

design with two different treatments (three replicates each) was carried out in 6 plots (3 m × 7 m) under a maize-rape system. Maize was planted in late-April (spring) and harvested in mid-August (summer), while rape was planted in mid-October (autumn) and harvested in mid-April (spring) of the next year. Specifically, the two treatments were control (CK) and swine manure treatment (M). The M was annually applied as 15,000 kg hm⁻² swine manure that contained 139.3 g kg⁻¹ OC, 15.3 g kg⁻¹ N, 14.9 g kg⁻¹ P₂O₅, 11.3 g kg⁻¹ K₂O, and 60% H₂O. The maize and rape straws did not return to the field after harvest. In each plot, five soil samples were randomly collected using a core sampler (20 cm high and with a 5-cm diameter) from 0 to 20 cm depth and mixed to form a composite sample. Soil samples were air-dried and passed through a 2-mm sieve to remove some visible plant residues, and the remains of visible plant residues were removed with tweezers, thereby minimizing the influence of plant residues on further chemical analysis.

2.2 Sequential density fractionation

The sequential density fractionation (SDF) method was used to separate bulk soil into four density fractions (Yeasmin et al. 2017b): < 1.8 (POM), 1.8–2.2 (MOM_{1.8–2.2}), 2.2–2.6 (MOM_{2.2–2.6}), and > 2.6 g cm⁻³ (MOM_{> 2.6}). Firstly, 15 g of air-dried soil and 50 mL of 1.8 g cm⁻³ sodium polytungstate (SPT) solution were mixed into a 100-mL centrifuge tube. The mixture was shaken for 3 h and then centrifuged (15 min, 2973 g). The POM (< 1.8 g cm⁻³) was collected on a 0.45-μm membrane filter. These steps were repeated three times to ensure a full recovery. The POM was rinsed with deionized water until salt concentration reached < 50 mS cm⁻¹. The remaining sediment in the centrifuge tube was mixed with 50 mL 2.2 g cm⁻³ SPT solution, following the

same procedure as POM to obtain the MOM_{1.8–2.2}. Similarly, the residual sediment after removing MOM_{1.8–2.2} was mixed with 50 mL 2.6 g cm⁻³ SPT solution to obtain the MOM_{2.2–2.6} (supernatant), and MOM_{> 2.6} (precipitate). All recovered fractions were oven-dried at 40 °C and ground to a fine powder for further analyses.

2.3 Removal of mineral bound OC with HF

Mineral bound OC in the four density fractions was removed by the HF demineralization procedure (Kaiser et al. 2002). About 200–250 mg of sample was weighed into a 25-mL centrifuge tube and 10 mL 10% HF was added into the tube, shaken for 2 h and centrifuged, and the supernatant was decanted. Every sample was washed using 10% HF 4–6 times to remove minerals completely. The residues were rinsed 5 times with deionized water and then oven-dried at 40 °C, weighted, ground, and stored in glass vials for further FTIR and C and N analyses.

2.4 Mineralogy and chemical analyses

Mineralogical compositions of the MOM_{1.8–2.2}, MOM_{2.2–2.6}, and MOM_{> 2.6} fractions were characterized using X-ray diffraction (XRD) with Cu Kα radiation at 40 kV and 40 mA (PANalytical, Netherlands) as previously described (Yeasmin et al. 2017b). These density fractions were ground to fine powder and were scanned from 5 to 45° at a speed of 1° min⁻¹ with a step size of 0.01°.

The quantification of poorly crystalline Fe and Al oxides (Fe_o and Al_o) is achieved using ammonium oxalate (pH = 3) with a soil to solution ratio of 1:50 and shaken for 2 h in the dark. The organically complex Fe and Al oxides (Fe_p and Al_p) were quantified by sodium pyrophosphate (pH 10.0) with a soil to solution ratio of 1:10 and shaken for 16 h (McKeague et al. 1971). Fe and Al in the two extracts were determined using inductively coupled plasma mass spectrometry (ICP-MS) (Agilent 7500CX).

Soil pH was measured with a compound electrode at a soil to water ratio of 1:2.5. Available P was extracted using 0.5 M sodium bicarbonate (pH = 8.5), in which the P content was measured by molybdenum blue colorimetry. Available K was extracted using 1 mol L⁻¹ ammonium acetate (pH = 7) and measured by a flame photometer (Thermo Scientific iCE 3500).

Total OC and N of bulk soil and the untreated and HF-treated density fractions and the natural δ¹³C of untreated density fractions were determined using isotope ratio mass spectrometry (Vario PYRO cube-IsoPrime 100). The proportion and content of HF-dissolved OC or N were calculated as follows (Yeasmin et al. 2017b):

$$\text{the proportion of HF dissolved OC (or N) (\% of initial)} = \left[\frac{(\text{Mass}_{\text{initial}} \times \text{OC}_{\text{initial}}) - (\text{Mass}_{\text{HF}} \times \text{OC}_{\text{HF}})}{(\text{Mass}_{\text{initial}} \times \text{OC}_{\text{initial}})} \right] \times 100$$

$$\text{the content of HF dissolved OC (or N) (g kg}^{-1} \text{ fraction)} = \left[\frac{(\text{Mass}_{\text{initial}} \times \text{OC}_{\text{initial}}) - (\text{Mass}_{\text{HF}} \times \text{OC}_{\text{HF}})}{(\text{Mass}_{\text{initial}} - \text{Mass}_{\text{HF}})} \right]$$

where $\text{Mass}_{\text{initial}}$ = initial weight (kg) of the density fractions, Mass_{HF} = residual weight (kg) of the density fractions after HF demineralization, $\text{OC (or N)}_{\text{initial}}$ = OC (N) content (g kg^{-1}) of the initial the density fractions, and $\text{OC (or N)}_{\text{HF}}$ = OC (N) content (g kg^{-1}) of the density fractions after HF demineralization.

2.5 Diffuse reflectance Fourier transform spectroscopy

The untreated and HF-treated density fractions were used for FTIR analyses. Briefly, 1.0 mg of dried samples was accurately diluted with 200 mg KBr, finely ground, and fully homogenized. The spectra were recorded following 32 scans with a resolution of 4 cm^{-1} in the $400\text{--}4000 \text{ cm}^{-1}$ wavenumber range on an FTIR spectrometer (Bruker Vertex 70 V, Bruker Optics, Germany). KBr background spectra were collected and subtracted from the sample spectra.

All spectra were normalized. The main assignments in FTIR spectra were as follows: OH of phyllosilicates and Fe/Al oxides ($3700\text{--}3440 \text{ cm}^{-1}$), aliphatic C ($3010\text{--}2800 \text{ cm}^{-1}$), aromatic C ($1680\text{--}1580 \text{ cm}^{-1}$), amide N ($1546\text{--}1520 \text{ cm}^{-1}$), and polysaccharide C ($1175\text{--}1148 \text{ cm}^{-1}$), which were integrated to calculate the relative peak areas using the software OPUS version 6.5 (Table S1) (Demyan et al. 2012; Yeasmin et al. 2017b). Relative band area of these four organic functional groups was calculated as follows (Demyan et al. 2012):

$$\text{Relative band area} = \frac{\text{Area of a certain band}}{\text{Sum of the area of all bands}}$$

The changes of the relative band area in these four organic functional groups after HF demineralization were determined by comparing the relative band areas of HF-treated density fractions with the relevant untreated. According to Yeasmin et al. (2017b), the increases and decreases in relative band area indicated organic functional groups were bound with minerals and non-bound respectively. The change of relative band area in the organic functional groups was calculated as follows:

$$\frac{(\text{Relative band area}_{\text{initial}} - \text{Relative band area}_{\text{HF}})}{\text{Relative band area}_{\text{initial}}} \times 100$$

where $\text{relative band area}_{\text{HF}}$ = relative band area of the organic functional groups in the residue density fractions

after HF demineralization and $\text{relative band area}_{\text{initial}}$ = relative band area of the organic functional groups in the initial density fractions.

2.6 Statistical analysis

Statistical analyses were performed with SPSS 19.0. The analysis of variance was used to determine a statistically significant difference ($P < 0.05$) between the CK and M treatments. The correlation between the proportion of HF-dissolved OC and the corrected band area of phyllosilicates or Fe and Al oxide band was obtained with the SigmaPlot (12.5) software.

3 Results

3.1 General characteristics of bulk soils

Manure fertilization (M) significantly increased soil pH (6.65), SOC (9.32 g kg^{-1}), total N (1.38 g kg^{-1}), available P (32.8 mg kg^{-1}), and K (328.3 mg kg^{-1}) contents ($P < 0.05$). In the CK, soil pH value was 6.13, SOC and total N contents were 7.18 and 1.02 g kg^{-1} respectively, and available P and K were 7.8 and 247.2 mg kg^{-1} respectively (Table S2).

3.2 Mineral compositions of MOM

The XRD results for $\text{MOM}_{1.8-2.2}$, $\text{MOM}_{2.2-2.6}$, and $\text{MOM}_{>2.6}$ are presented in Fig. 2. The $\text{MOM}_{1.8-2.2}$ and $\text{MOM}_{2.2-2.6}$ had similar mineral composition, being mainly composed of illite, kaolinite, and iron and aluminum minerals (i.e., gibbsite, goethite, and hematite). The $\text{MOM}_{>2.6}$ was mainly consisted of quartz and feldspars. Meanwhile, the Fe_o and Fe_p contents of the M (3.97 and 4.93 g kg^{-1} for Fe_o ; 0.59 and 0.73 g kg^{-1} for Fe_p in $\text{MOM}_{1.8-2.2}$ and $\text{MOM}_{2.2-2.6}$ respectively) were greater than that of the CK (3.36 and 3.94 g kg^{-1} for Fe_o ; 0.27 and 0.34 g kg^{-1} for Fe_p in $\text{MOM}_{1.8-2.2}$ and $\text{MOM}_{2.2-2.6}$ respectively) in $\text{MOM}_{1.8-2.2}$ and $\text{MOM}_{2.2-2.6}$ ($P < 0.05$), showing a gradually increasing trend from $\text{MOM}_{1.8-2.2}$ to $\text{MOM}_{2.2-2.6}$ ($P < 0.05$). However, the Al_o and Al_p had no consistent patterns in $\text{MOM}_{1.8-2.2}$ and $\text{MOM}_{2.2-2.6}$. The Fe_o , Fe_p , Al_o , and Al_p contents were the lowest in

Fig. 2 X-ray powder diffraction patterns of the density fractions. MOM, mineral-associated organic matter. MOM_{1.8–2.2} (1.8–2.2 g cm^{−3}), MOM_{2.2–2.6} (2.2–2.6 g cm^{−3}), MOM_{> 2.6} (> 2.6 g cm^{−3}). Ill = illite, Kao = kaolinite, Gib = gibbsite, Goe = goethite, Hem = hematite

MOM_{> 2.6} ($P < 0.05$) and had no difference between the M and CK ($P > 0.05$) (Fig. 3).

Fig. 3 Mean values of poorly crystalline Fe and aluminum (Al) oxides and organically complexed Fe and Al oxides in MOM of control (CK) and manure (M) treatments. MOM, mineral-associated organic matter. MOM_{1.8–2.2} (1.8–2.2 g cm^{−3}), MOM_{2.2–2.6} (2.2–2.6 g cm^{−3}), MOM_{> 2.6} (> 2.6 g cm^{−3}). Different uppercase and lowercase letters on the top of

3.3 OC, N, $\delta^{13}\text{C}$, and FTIR in POM and MOM

The Fe and Al oxides and phyllosilicate-dominated MOM_{1.8–2.2} and MOM_{2.2–2.6} had the highest proportion (60%) on a mass basis and retained substantial OC (82.9–86.1%) and N (85.8–87.9%) of bulk soil in both treatments ($P < 0.05$), and the quartz and feldspar-dominated MOM_{> 2.6} with 37.2–38.9% proportion of soil mass had the lowest proportion of OC (4.6–4.7%) and N (7.19–8.68%) ($P < 0.05$) (Table 1). The $\delta^{13}\text{C}$ value showed an increasing trend from POM to MOM_{2.2–2.6}, ranged from -23.3 to -20.5‰ ($P < 0.05$) (Fig. 4a). Total OC (0.82–0.86 g kg^{−1}) and N (0.26–0.28 g kg^{−1}) contents were the lowest in the MOM_{> 2.6} ($P < 0.05$) and then gradually increased with decreasing density ($P < 0.05$). The M showed higher OC and N contents in the POM, MOM_{1.8–2.2}, and MOM_{2.2–2.6} (223.5, 102.6, and 9.9 g kg^{−1} for OC; 16.9, 9.6, and 1.9 g kg^{−1} for N in POM, MOM_{1.8–2.2}, and MOM_{2.2–2.6} respectively) compared with the CK (124.4, 59.6, and 8.8 g kg^{−1} for OC; 8.5, 5.4, and 1.7 g kg^{−1} for N in POM, MOM_{1.8–2.2}, and MOM_{2.2–2.6} respectively) ($P < 0.05$) (Fig. 4b and d). The

the error bars indicate the significant difference among different density fractions (POM vs. MOMs) at the same treatment and between the two treatments (CK vs. M) at the same density fraction, respectively ($P < 0.05$). Error bars represent the standard error ($n = 3$) of a treatment

Table 1 Proportion of soil mass, organic carbon (OC), and nitrogen (N) of their total contents in the four density fractions of control (CK) and manure (M) treatments

Treatments	Density fractions	Proportion of the total in soil (%)		
		Mass	OC	N
CK	POM	0.5 ± 0.03Ca	9.1 ± 0.6Bb	3.4 ± 0.3Cb
	MOM _{1.8–2.2}	1.0 ± 0.11Ca	8.3 ± 0.9Bb	4.3 ± 0.8Cb
	MOM _{2.2–2.6}	59.3 ± 1.63Aa	77.8 ± 1.7Aa	83.6 ± 1.9Aa
	MOM _{> 2.6}	38.9 ± 1.54Ba	4.7 ± 0.3Ca	8.7 ± 0.8Ba
	Total recovery	98.3 ± 0.25	94.1 ± 2.36	106.3 ± 2.7
M	POM	0.6 ± 0.03Ca	13.3 ± 0.7Ca	7.0 ± 0.1Ca
	MOM _{1.8–2.2}	1.3 ± 0.02Ca	15.2 ± 0.1Ba	8.6 ± 0.1Ba
	MOM _{2.2–2.6}	60.6 ± 1.44Aa	67.7 ± 1.9Ab	77.2 ± 0.7Ab
	MOM _{> 2.6}	37.2 ± 1.52Ba	3.8 ± 0.3Db	7.2 ± 0.7Cb
	Total recovery	98.4 ± 0.41	95.1 ± 2.9	103.8 ± 2.1

Density fractions: POM (< 1.8 g cm⁻³), MOM_{1.8–2.2} (1.8–2.2 g cm⁻³), MOM_{2.2–2.6} (2.2–2.6 g cm⁻³), and MOM_{> 2.6} (> 2.6 g cm⁻³). Different uppercase and lowercase letters indicate the significant difference among different density fractions (POM vs. MOM) at the same treatment and between the two treatments (CK vs. M) at the density fractions, respectively ($P < 0.05$). Values are means ($n = 3$) with standard error

POM particular organic matter, MOM mineral-associated organic matter

changing trend of C:N ratio was similar to the OC and N contents ($P < 0.05$) (Fig. 4c).

The FTIR spectra in these four organic functional groups before and after HF demineralization are presented in Fig. S1.

Fig. 4 $\delta^{13}\text{C}$ (a), total OC (b), C:N ratio (c), and total N (d) of the four density fractions following the control (CK) and manure (M) treatments obtained from mass spectroscopy. POM, particular organic matter (< 1.8 g cm⁻³), MOM, mineral-associated organic matter. MOM_{1.8–2.2} (1.8–2.2 g cm⁻³), MOM_{2.2–2.6} (2.2–2.6 g cm⁻³), and MOM_{> 2.6} (> 2.6 g

cm⁻³). Different uppercase and lowercase letters on the top of the error bars indicate the significant difference among different density fractions (POM vs. MOMs) at the same treatment and between the two treatments (CK vs. M) at the same density fraction, respectively ($P < 0.05$). Error bars represent the standard error ($n = 3$) of a treatment

The relative area of aliphatic C (2930 cm^{-1}) had a downward trend with higher density (Fig. 5a). On the contrary, the aromatic C (1620 cm^{-1}) showed an increasing trend from POM to $\text{MOM}_{>2.6}$ (Fig. 5b). These results led to the difference in aromatic C:aliphatic C ratio among POM and MOM, showing an increasing tendency from $\text{MOM}_{1.8-2.2}$ to $\text{MOM}_{>2.6}$, with a higher value for the M in $\text{MOM}_{2.2-2.6}$ and $\text{MOM}_{>2.6}$ (Fig. 5e). Amide N (1530 cm^{-1}) and polysaccharides (1159 cm^{-1}) increased with higher density (Fig. 5c, d).

3.4 HF-dissolved OM in POM and MOM

The significantly positive relationship between the proportion of HF-dissolved OC (%) and the corrected band area of Fe and Al oxides and phyllosilicates ($R^2 = 0.83$, $P < 0.01$) (Fig. 6) confirmed that the HF-dissolved OC was associated with clay minerals. The HF demineralization caused a loss of about 31.2–58.8% OC from $\text{MOM}_{1.8-2.2}$ and $\text{MOM}_{2.2-2.6}$, with the smaller loss occurring in POM (6.3–10.1%) and $\text{MOM}_{>2.6}$ (12.8–17.7%) (Table 2). In $\text{MOM}_{1.8-2.2}$ and $\text{MOM}_{2.2-2.6}$, significantly positive correlations were found between HF-dissolved OC (%) and poorly crystalline ($R^2 = 0.61$, $P < 0.01$) and organically complexed Fe ($R^2 = 0.45$, $P < 0.05$) not Al (Fig. 8a, b). Meanwhile, the contents of HF-dissolved OC and N (g kg^{-1}) in $\text{MOM}_{1.8-2.2}$ and $\text{MOM}_{2.2-2.6}$ were greater in the M (39.01 and 6.45 g kg^{-1} for OC; 4.30 and 1.47 g kg^{-1} for N in $\text{MOM}_{1.8-2.2}$ and $\text{MOM}_{2.2-2.6}$ respectively) compared with that in the CK (21.70 and 4.70 g kg^{-1} for OC; 2.99

and 1.21 g kg^{-1} for N in $\text{MOM}_{1.8-2.2}$ and $\text{MOM}_{2.2-2.6}$ respectively) ($P < 0.05$) (Table 2).

In the MOM of both treatments, the change of the relative band area of aliphatic C (2930 cm^{-1}) showed a gradual increment with increasing density after HF demineralization (Fig. 7a). The reduction in the relative band area of aromatic C (1620 cm^{-1}) after HF demineralization occurred in $\text{MOM}_{1.8-2.2}$, $\text{MOM}_{2.2-2.6}$, and $\text{MOM}_{>2.6}$, and the M had more reduction than the CK (Fig. 7b). Moreover, the significantly negative correlations were observed between the relative band change of aromatic C (%) and the Fe oxide contents ($R^2 = 0.72$, $P < 0.01$ for poorly crystalline Fe; $R^2 = 0.49$, $P < 0.05$ for organically complexed Fe) in $\text{MOM}_{1.8-2.2}$ and $\text{MOM}_{2.2-2.6}$ (Fig. 8c, d). The change of amide N (1530 cm^{-1}) band area increased in $\text{MOM}_{1.8-2.2}$ and $\text{MOM}_{2.2-2.6}$ after HF demineralization and then decreased in $\text{MOM}_{>2.6}$ (Fig. 7c). The polysaccharide band area (1159 cm^{-1}) was depleted in $\text{MOM}_{2.2-2.6}$ and $\text{MOM}_{>2.6}$ after HF demineralization, and the M had more depletion than the CK in $\text{MOM}_{2.2-2.6}$ (Fig. 7d).

4 Discussion

4.1 Organo-mineral complex distribution and composition in MOM

The SDF effectively isolated the bulk soil into POM, Fe, and Al oxides and phyllosilicate-dominated $\text{MOM}_{1.8-2.2}$ and

Fig. 5 Relative band area of organic groups (a–d) and ratio of 1620 cm^{-1} and 2930 cm^{-1} (e) band area in the infrared Fourier transform spectra of the four density fractions of control (CK) and manure (M) treatments. POM, particular organic matter ($< 1.8\text{ g cm}^{-3}$); MOM, mineral-associated organic matter. $\text{MOM}_{1.8-2.2}$ ($1.8\text{--}2.2\text{ g cm}^{-3}$), $\text{MOM}_{2.2-2.6}$ ($2.2\text{--}2.6\text{ g}$

cm^{-3}), and $\text{MOM}_{>2.6}$ ($> 2.6\text{ g cm}^{-3}$). Aliphatic C ($3010\text{--}2800\text{ cm}^{-1}$): 2930 cm^{-1} ; aromatic C ($1680\text{--}1580\text{ cm}^{-1}$): 1620 cm^{-1} ; amide N ($1546\text{--}1520\text{ cm}^{-1}$): 1530 cm^{-1} ; and polysaccharide C ($1175\text{--}1148\text{ cm}^{-1}$): 1159 cm^{-1} . Error bars represent the standard error ($n = 3$) of a treatment

Fig. 6 Relationship between the amount of HF soluble OC and the corrected band area of mineral (phyllosilicates or Fe and Al oxides) in the infrared Fourier transform spectra of the four density fractions of control and manure treatments. Band area integration limit = 3700–3440 cm^{-1}

MOM_{2.2–2.6}, and quartz and feldspar-dominated MOM_{> 2.6} (Fig. 2). The decreased C:N ratio and the increased $\delta^{13}\text{C}$ with higher density (Fig. 4a, c) indicated that OC from microbial processes is more likely to accumulate in higher density fractions (Moni et al. 2012).

The MOM_{1.8–2.2} and MOM_{2.2–2.6} retained about 80% OC and N of bulk soil (Table 1) and had 31.2–58.8% HF-dissolved OC (Table 2), which indicated a greater potential of Fe and Al oxides and phyllosilicates to preserve and store OC. Schimel et al. (1985) also found that SOC content had a strong relationship with clay content in the soil. However, the stronger positive correlation of the HF-dissolved OC (%) with Fe oxides than Al oxides (Fig. 8a) showed that Fe oxides play

a more important role in preserving OC pool by organo-Fe complexes compared with Al oxides in red soil during manure fertilization (Rasmussen et al. 2006; Zhang et al. 2013). The reason may be because of the higher pH and P content in soil caused by manure fertilization (Table S2) going against the organo-Al association (Shen et al. 2018). The substantial reduction of band area just occurred in the aromatic C of the MOM_{1.8–2.2} and MOM_{2.2–2.6} after HF demineralization (Fig. 7b), which could suggest that perhaps OC associated with clay mineral in these fractions was mostly composed of aromatic C. That is because the aromatic compounds have a high affinity for the hydroxyl group (OH[−]) on the surface of minerals by ligand exchange (Sollins et al. 1996). Kramer et al. (2012) also suggested that the binding of carboxyl-rich aromatic compound with short-range order mineral could be invoked to explain the persistence of OC in subsoil. These acidic functional groups (e.g., COO[−]) could be electrostatically associated with hydroxy polyvalent cations of Fe and Al oxides in acid red soil (Mikutta et al. 2007). However, in this study, it was difficult to precisely distinguish the amount and structure of these OC compounds between phyllosilicates and Fe and Al oxides in the MOM_{1.8–2.2} and MOM_{2.2–2.6} based on the existing data and methods. In future studies, X-ray photoelectron spectroscopy (XPS) and scanning transmission X-ray microscopy paired with near-edge X-ray absorption spectroscopy (STXM-NEXAFS) will be effective tools to quantitatively and qualitatively evaluate the organo-mineral complexes among phyllosilicates and Fe and Al oxides (Sowers et al. 2018).

The MOM_{> 2.6} that comprised 37.2–38.9% soil mass merely retained 3.8–4.7% OC and 7.2–8.7% N of bulk soil, and had the lowest OC and N contents (Fig. 3 and Table 1), which attributed to the quartz and feldspar with the low and

Table 2 The proportions of mass, organic carbon (OC), total nitrogen (N), and OC and N contents and C:N ratio of the HF-dissolved organic matter in the four density fractions of control (CK) and manure (M) treatments

Treatments	Density fraction	% of the initial total			Total in dissolved fraction (g kg^{-1})		C:N
		Mass	OC	N	OC	N	
CK	POM	75.4 ± 1.2Ca	10.1 ± 4.5Ca	29.6 ± 5.5 Da	16.53 ± 7.07Ab	3.34 ± 0.60Aa	4.82 ± 1.43Bb
	MOM _{1.8–2.2}	89.8 ± 0.1Ba	31.3 ± 2.9Ba	48.4 ± 5.4Ca	20.75 ± 2.19Ab	2.92 ± 0.49Ab	7.28 ± 1.72Ab
	MOM _{2.2–2.6}	87.2 ± 1.6Ba	46.6 ± 4.1Ab	63.1 ± 4.1Bb	4.70 ± 0.31Bb	1.20 ± 0.06Bb	3.90 ± 0.10Bb
	MOM _{> 2.6}	96.6 ± 0.2Aa	12.8 ± 4.4Ca	84.0 ± 1.5Aa	0.23 ± 0.03Ca	0.24 ± 0.01Ca	0.99 ± 0.17Ca
M	POM	54.4 ± 1.9Db	6.3 ± 0.7 Da	10.3 ± 2.1Cb	25.61 ± 2.12Ba	3.21 ± 0.49Ba	8.06 ± 0.79Ba
	MOM _{1.8–2.2}	82.1 ± 0.6Cb	31.2 ± 3.6Ba	36.7 ± 2.1Bb	39.01 ± 4.24Aa	4.30 ± 0.26Aa	9.06 ± 0.58Aa
	MOM _{2.2–2.6}	90.9 ± 0.3Ba	58.8 ± 4.3Aa	72.1 ± 2.8Aa	6.45 ± 0.41Ca	1.47 ± 0.04Ca	4.39 ± 0.16Ca
	MOM _{> 2.6}	96.5 ± 0.4Aa	17.7 ± 3.2Ca	78.4 ± 0.3Ab	0.22 ± 0.03 Da	0.23 ± 0.01 Da	0.96 ± 0.15 Da

Density fractions: POM (< 1.8 g cm^{-3}), MOM_{1.8–2.2} (1.8–2.2 g cm^{-3}), MOM_{2.2–2.6} (2.2–2.6 g cm^{-3}), and MOM_{> 2.6} (> 2.6 g cm^{-3}). Different uppercase and lowercase letters indicate the significant difference among different density fractions (POM vs. MOM) at the same treatment and between the two treatments (CK vs. M) at the same density fraction, respectively ($P < 0.05$). Values are means ($n = 3$) with standard error

POM particular organic matter, MOM mineral-associated organic matter

Fig. 7 Relative band change (%) of four organic groups in the infrared Fourier transform spectra of the four density fractions of control (CK) and manure (M) treatments after 10% hydrofluoric acid demineralization compared with their relevant untreated sample spectra. POM, particular organic matter ($< 1.8 \text{ g cm}^{-3}$); MOM, mineral-associated organic matter. MOM_{1.8–2.2} ($1.8–2.2 \text{ g cm}^{-3}$), MOM_{2.2–2.6} ($2.2–2.6 \text{ g cm}^{-3}$), MOM_{>2.6} ($> 2.6 \text{ g cm}^{-3}$). Aliphatic C ($3010–2800 \text{ cm}^{-1}$): 2930 cm^{-1} ; aromatic C ($1680–1580 \text{ cm}^{-1}$): 1620 cm^{-1} ; amide N ($1546–1520 \text{ cm}^{-1}$): 1530 cm^{-1} ; and polysaccharide C ($1175–1148 \text{ cm}^{-1}$): 1159 cm^{-1} . Different letters on the top or bottom of the error bars indicate a significant difference between the two treatments (CK vs. M) at the same density fraction ($P < 0.05$). Error bars represent the standard error ($n = 3$) of a treatment

unreactive specific surface areas, low charge, and some unsatisfied siloxane bonds that contribute little to preserve and store the OC pool. On the other hand, quartz and feldspar surfaces are generally coated with Fe and Al oxides or phyllosilicate, which result in a preferential association of OC with the surfaces of Fe and Al oxides or phyllosilicate (Jones and Singh 2014). The substantial reduction in the relative band areas of aromatic C, amide N, and polysaccharides (Fig. 7b, c, d) after HF demineralization suggested that those organic functional groups could be associated with quartz or feldspars. Jindaluang et al. (2013) also reported that $> 2.6 \text{ g cm}^{-3}$ density fraction of coarse-textured soil with the lowest OC content enriched with aromatic C and amide N. Jones and Singh (2014) indicated that the bonding of OC to quartz and feldspar surface consisted of polysaccharides and protonated amide N compounds which might associate with unsatisfied siloxane bonds of quartz and feldspar surface by hydrogen bonding and electrostatic attraction respectively.

4.2 Organo-mineral complex distribution and composition changes following manure fertilization

In the POM, the increased alkyl C and aliphatic C and the decreased aromatic C after manure fertilization indicated that the POM structure tended to become simpler and more aliphatic (Fig. 5a, b, d). This could be explained by the fact that the POM in the M contained more undecomposed or labile swine manure and plant materials (Dou et al. 2008).

Meanwhile, this fraction had few HF-dissolved OC (Table 2) which may be due to the presence of minor encrustations of mineral grains in the POM (Moni et al. 2012; Yeasmin et al. 2017a). After all, organo-mineral association was secondary in the POM.

The dissolved organic matter derived from manure can be adsorbed by Fe oxides forming organo-mineral complexes or it can intercalate between the layers of phyllosilicates (Neff and Asner 2001; Fernández-Ugalde et al. 2017), which caused more HF-dissolved OC (Table 2) in the MOM_{1.8–2.2} and MOM_{2.2–2.6} of the M, contributing to protection and stabilization of manure-derived OC during manure fertilization. The increased mineral bound OC content as a result of manure application affected the OC chemical structure when compared with CK. The polysaccharides associated with MOM_{2.2–2.6} in the M were substantially reduced after the HF demineralization than in the CK (Fig. 7d), indicating that organic fertilization could promote the bonding of polysaccharides with Fe oxides or phyllosilicates. During the degradation of manure, the produced carbohydrate can strongly absorb on Fe oxides or phyllosilicates through cation bridge or hydrogen bonding (von Lützow et al. 2006). Furthermore, manure fertilization could also make the OC associated with MOM_{2.2–2.6} more aromatic (the higher aromatic C:aliphatic C ratio in the M) (Fig. 5e), as further supported by the substantial reduction of aromatic C following HF demineralization in the MOM_{1.8–2.2} and MOM_{2.2–2.6} of the M compared with the CK (Fig. 7b). This may be due to the highly oxidized tannins,

Fig. 8 Relationship of the proportions of HF-dissolved OC (%) with poorly crystalline (a) and organically complexed Fe and Al (g kg^{-1}) (b) and relationship of the relative band change of aromatic C (%) after 10% hydrofluoric acid demineralization with poorly crystalline (c) and

organically complexed Fe and Al (g kg^{-1}) (d) in $\text{MOM}_{1.8-2.2}$ and $\text{MOM}_{2.2-2.6}$ of the control and manure treatments. MOM, mineral-associated organic matter. $\text{MOM}_{1.8-2.2}$ ($1.8-2.2 \text{ g cm}^{-3}$), $\text{MOM}_{2.2-2.6}$ ($2.2-2.6 \text{ g cm}^{-3}$)

polyphenols, and other aromatic compounds derived from decomposed manure bind directly to hydroxylated mineral surface (Kleber et al. 2007). Similarly, aromatic lignin constituents could be specifically stabilized during Fe addition to soil (Hall et al. 2016).

In the present study, the higher contents of poorly crystalline and organically complexed Fe oxides in the $\text{MOM}_{1.8-2.2}$ and $\text{MOM}_{2.2-2.6}$ of the M than CK (Fig. 3a, b) indicated that organic fertilization could increase the mineral availability and promote the formation of organo-Fe complexes. The reason is the fact that organic acid derived from decomposed manure could drive the transformation of Fe oxides from crystalline to short-range order and the formed organo-Fe complexes can stand up to Fe recrystallization (Yu et al. 2017; Huang et al. 2018); another reason is that the near-neutral pH conditions under the M (Table S2) promote the precipitation and stabilization of short-range order Fe minerals (Colombo et al. 2014). These increased Fe oxides were significantly negatively correlated with the relative band area of HF-dissolved aromatic C (%) ($R^2 = 0.72$, $P < 0.01$ for poorly crystalline Fe; $R^2 = 0.49$, $P < 0.05$ for organically complexed Fe) (Fig. 8c, d), suggesting that manure fertilization could increase the formation of

aromatic OC-Fe complexes, which contributed to the SOC preservation in red soil. This is consistent with preferential association between the poorly crystalline Fe oxide mineral and aromatic organic compounds in soil (Kramer et al. 2012; Huang et al. 2018). Adhikari et al. (2017) also investigated the release and transformation of ferrihydrite-bound OC during microbial Fe reduction and found that the transformation of non-crystalline ferrihydrite to crystalline Fe phases was dependent on the OC/Fe ratio, and aromatic OC was preferentially retained within OC-ferrihydrite co-precipitation. In our study, the accumulation of decomposed swine manure rich in aromatic C under manure fertilization facilitated aromatic OC-poorly crystalline Fe oxide association, which promoted the role of organo-Fe complexes in SOC preservation in red soil.

It should be noted that, in this study, we studied the composition of organo-mineral complexes in each density fraction merely depending on some quantity relations between the contents of minerals and the changes of organic functional group areas obtained from FTIR analysis after and before HF demineralization, which could interpret partial information of organo-mineral complexes, but less adequate for elaborating the bonds of organo-mineral. Therefore, further

investigation on organo-mineral complexes under soil incubation with ^{13}C -labeled organic matters, and together with nano-scale secondary ion mass spectroscopy, XPS, and STXM-NEXAFS, may validate how mineral oxides associated with SOC in red soil.

5 Conclusions

The SDF and HF demineralization methods, together with chemical characterization using FTIR spectrometer, can be used to assess the effect of manure fertilization on the quality and quantity of organo-mineral complexes within different density fractions of red soil. Manure fertilization could significantly improve the OC and N contents of POM, $\text{MOM}_{1.8-2.2}$, and $\text{MOM}_{2.2-2.6}$. The Fe and Al oxides and phyllosilicate-dominated $\text{MOM}_{1.8-2.2}$ and $\text{MOM}_{2.2-2.6}$ contributed to 82.9–86.1% of SOC, of which Fe bound OC played the most vital role in SOC stabilization and was susceptible to fertilization. Manure fertilization significantly increased the contents of poorly crystalline and organically complexed Fe oxides within the $\text{MOM}_{1.8-2.2}$ and $\text{MOM}_{2.2-2.6}$ and then facilitated the formation of aromatic OC-Fe complex ($P < 0.05$), which contributed to the OC preservation in red soils. The quartz and feldspar-dominated $\text{MOM}_{>2.6}$, containing aromatic C, amide N, and polysaccharides, merely retained 3.8–4.7% of SOC and had less contribution to the OC preservation, not being affected by fertilization. Although this study contributes some knowledge on potential preservation mechanisms of OC in red soils, one soil type (single red soil) and two treatment (control and manure fertilization) are not enough to draw some general laws and conclusions, especially the interactions between minerals and specific organic compounds. Further studies will focus on turnover times and molecular compositions of organo-mineral complexes under different fertilizer applications (e.g., inorganic fertilizer, different kinds of straws returning) and soil types (e.g., luvisol, mollisol, and vertisol).

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