

RESEARCH ARTICLE

The interactions between Al-/Fe-(hydr)oxides and soil organic carbon mediate the aggregation of yellow soils

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Abstract

Soil aggregates are essential for improving soil permeability and reducing soil erosion. Still, in high oxide soils, the roles of soil organic carbon (SOC) and Al-/Fe-(hydr)oxide in soil aggregation are unclear, which limits our ability to predict how soil aggregation responds to soil management and climate change. In this study, soil samples were collected from three depths of a yellow soil profile (0–12 cm, 12–24 cm and 24–36 cm) and fractionated into aggregates by wet sieving. Organic carbon (OC) and Al-/Fe-(hydr)oxide contents of aggregates were determined. We found that the relationships of OC and Al-/Fe-(hydr)oxide to aggregate size were depth-dependent. OC concentrations increased and decreased at 0–12 cm and 24–36 cm depth soils, respectively, as aggregate size decreased. The OC/(Fe + Al) ratios of aggregates suggest that organic carbon will form multi-layer and mono-layer coatings on Al-/Fe-(hydr)oxide in the 0–12 cm and 24–36 cm depth soils, respectively. This leads to steric hindrances of OC and electrostatic repulsion of Al-/Fe-(hydr)oxide at two depths, respectively, and further influenced the aggregation. Based on these findings, we proposed a mechanism in which aggregates are formed by OC and Al-/Fe-(hydr)oxide association with the appropriate OC/(Fe + Al) ratio. This mechanism focuses on the role of the OC/(Fe + Al) ratio mediated in soil aggregation. It can account for the inconsistent responses of aggregation and SOC sequestration to exogenous OC addition in field experiments. Further studies are needed and will be helpful to calculate the dose of organic application for formulating healthy and sustainable soil management.

KEYWORDS

aggregation, Fe/Al-(hydr)oxides, organic carbon, organo-oxide associations, yellow soil

1 | INTRODUCTION

Soil aggregates are fundamental units of soil structure (Udom et al., 2022; Wang et al., 2022) and are recognized to control a series of ecosystem processes (Rillig et al., 2015; Sarker et al., 2018; Xiao et al., 2021). Recent research on soil aggregation has primarily focused on two aspects. One is its role in the stabilization of soil organic carbon (SOC) (Ayuke et al., 2011; Totsche et al., 2018) based on

the urgency of mitigating climate change (Hou, 2021a, 2021b). The other is its role in soil physical structure (Zeng et al., 2018) in the context of land degradation owing to the intensification of agriculture.

Aggregate formation and stabilization depends on the quantity and quality of cementing agents, such as organic matter, Al-/Fe-(hydr)oxides and clays in the soils (Guhra et al., 2019). In general, the role of SOC in soil aggregation is essential, whether in oxide soils or other

soil types (Hou et al., 2020; Sarker et al., 2018; Tisdall & Oades, 1982; Wen et al., 2021). The classical aggregate hierarchy model suggests that SOC concentrations and the C/N ratio of aggregates generally increases with aggregate size because of the contribution of fine roots and hyphae to the macro-aggregate formation (Elliott, 1986). This aggregation model has successfully described Cambisols, Luvisols, Planosols, Gleysols, Vertisols and Chernozems (Totsche et al., 2018). However, the SOC hierarchical relationship disappears in some tropical and oxidic soils, such as Acrisols, Ferralsols and Nitisols (Barthes et al., 2008). The less effect of SOC on soil aggregation may be owing to the high content of Al-/Fe-(hydr)oxides in these soils.

In high oxide soils, either alone (Barthes et al., 2008; Peng et al., 2015) or associated with SOM (Peng et al., 2015; Six et al., 2004), Al-/Fe-(hydr)oxides are key in soil aggregation and their role is strongly influenced by their metal speciation. In disaggregation experiments, Xiao et al. (2018) found a positive correlation between the mean weight diameter (MWD) of aggregates and the amount of dithionite-citrate-bicarbonate (DCB) extractable Fe (Fe_{DCB} ; free Fe oxide). However, Duiker et al. (2003) reported that acid ammonium oxalate extractable Fe (hydr)oxide (Fe_{AO}) played a greater role than Fe_{DCB} in soil aggregate formation. Yin et al. (2016) reported a positive correlation between pyrophosphate-extractable (organically-complexed) Fe (Fe_p) and water stable aggregates (WSAs). The SOC and Fe_{AO} are the most important driving factors for aggregate stability, with Fe_{AO} indirectly improving aggregate stability through its effects on Fe_p (Xue, Huang, Huang, Yin, et al., 2019). Varied findings may be owing to the properties of the soils tested. Xue, Huang, Huang, Zhou, et al. (2019) reported the Fe_{DCB} had a greater effect on the stability of aggregates than Fe_{AO} in high SOC–low iron oxides soil, but both had the same effect in low SOC–high iron oxides soil. Additional, investigations indicate that aggregates formed by Al-/Fe-(hydr)oxide phases may be easily broken by slaking because they exhibit high resistance to mechanical stress but low slaking resistance (Six et al., 2002).

SOC and Al-/Fe-(hydr)oxides coexist rather than exist separately. Thus, in most cases, the association of Al-/Fe-(hydr)oxides and SOC contributes to soil aggregation rather than phase (Zhu et al., 2016). Several studies have assessed the dominant contributions of SOC and Al-/Fe-(hydr)oxides to aggregation and distinguished the role of SOC and Al-/Fe-(hydr)oxides in soil aggregation in high oxide soils. For example, Tisdall and Oades (1982) suggested that SOC is a dispensable aggregating agent, especially in soils with Al-/Fe-(hydr)oxides above 10%. In tropical soils, poor crystalline Fe-(hydr)oxide components (extracted by acid ammonium oxalate) are more critical for soil aggregation than SOC (Barthes et al., 2008; Duiker

et al., 2003). In a study of Ultisols, Peng et al. (2015) found that oxides and SOC are agents of micro- and macro-aggregates, respectively. However, there is no existing mechanistic explanation of why the roles of SOC and Al-/Fe-(hydr)oxides in soil aggregation are distinct in different soils.

The United Nations' Sustainable Development Goals (SDGs) require the adoption of sustainable and healthy soil management strategies. For OC management, positive (Das, Chakraborty, Singh, Aggarwal, Singh, & Dwivedi et al., 2014) and passive (Fungo et al., 2017; Guo et al., 2019; Shirani et al., 2002; Whalen & Chang, 2002) responses of soil aggregation to organic material application in soil management have been reported in the literature (Xie et al., 2015). Baumert et al. (2018), which demonstrates that high levels of exudate materials promote the formation of macroaggregates. However, the effects are much more pronounced in C-poor subsoil than in C-rich topsoil. There are inconsistencies in climate change data as well. The depth-dependent response of macro-aggregation to elevated CO_2 was examined by Keidel et al. (2018) who found macro-aggregation increased in subsoil (15–45 cm depth), but the macro-aggregation of topsoil (0–15 cm depth) was not affected under elevated CO_2 . In general, quantitative predictions of aggregation still cannot be made in the context of soil use and management owing to a lack of sufficient scientific knowledge (Cavicchioli et al., 2019; Hou, 2021b; Jansson & Hofmockel, 2020).

Based on current knowledge, inconsistent responses to soil management and climate changes may be because of the following: (1) a lack of mechanistic understanding to the role of SOC and Al-/Fe-(hydr)oxides in soil aggregation, and, (2) issues related to sampling (sampling depth, removal of humus layer or not) and handling processes (particle size selection, characterization of SOC and Al-/Fe-(hydr)oxides). In this study, we collected samples from three different depths of a yellow soil profile (a type of high oxide soil) and determined the contents of SOC and Al-/Fe-(hydr)oxides, analysed the relationships between SOC as well as Al-/Fe-(hydr)oxides and depth. The main objective of this study was to preliminarily explore the role of SOC and Al-/Fe-(hydr)oxide interactions in soil aggregation; furthermore, to propose a mechanism for yellow soil aggregation that can demonstrate common observations in soil management.

2 | MATERIALS AND METHODS

2.1 | Site description and sampling

The sampling site is located in Guiyang, Guizhou Province, China (26.673°N, 106.684°E). This region is covered in

coniferous forest vegetation and has a subtropical monsoon climate with an annual mean temperature of 15°C and precipitation of 1200 mm. The sample was collected at an altitude of 1310 m. The 0–12 cm depth soil has 69% clay, 13% sand (ASTM D422-63) and a 175 mmol kg⁻¹ cation exchange capacity (CEC; EPA Method 9081).

The main soil type in this region is yellow soil, which is one of the most common zonal soils and is widely distributed in the subtropical regions of China. It is yellow because of the hydration of iron oxide and the formation of goethite (Soil Census Office of Guizhou Province, 1994). The soil is classified as Dystrochrept in accordance with the USA soil taxonomy (Soil Survey Staff, 1999) and yellow soil according to soil generic classification (Soil Census Office of Guizhou Province, 1994). The soils have undergone strong Si loss and Al accumulation, causing a large accumulation of Al-/Fe-(hydr)oxide in soils. The soil is young (<2000 years dated by ¹⁴C), and horizons are relatively homogeneous compared with highly weathered soils. Therefore, depth increments rather than soil horizons were used to sample the soil. The litter on the top of the mineral soil was stripped away. Three soil depths (0–12 cm, 12–24 cm and 24–36 cm) were sampled, with about 30 kg of soil collected from each depth for representation and enough for aggregate-sized fractions. Statistical analyses were limited because of a lack of replication plots. However, we believe that the data obtained from this plot have significant value. Field-moist soil samples were carefully broken up by hand along weak natural planes (diameter <8 mm) and air-dried at room temperature. Fragments, such as roots and stones were removed with a tweezer.

2.2 | Fractionation of the water stable aggregates

Pre-experimental results showed that there were a large number of large aggregates in the soil samples. Therefore, the separation of water stable aggregates (WSAs) was carried out by slaking, which is a relatively strong way of aggregate destruction (Das, Chakraborty, Singh, Aggarwal, Singh, Dwivedi, & Mishra et al., 2014; Xue, Huang, Huang, Zhou, et al., 2019). The method used in this study resembled that used by Cambardella and Elliott (1994) and Dorji et al. (2020), but the separation times, the number of sieves and sample masses were modified. In brief, 25 g of soil were sieved with mesh sizes of 5, 2, 1, 0.5, 0.25 and 0.053 mm with four replications. The nested set of sieves containing the sample was immersed in deionized water for 10 minutes, then raised and lowered mechanically for 2 h at an amplitude of 3 cm and a rate of 40 oscillations per minute. The remaining soil on each sieve was

collected and oven-dried at 50°C. Sample remaining in the deionized water (e.g. <0.053 mm) was left to settle overnight, centrifuged for 10 min (3500 r/min) and oven-dried at 50°C. The dried samples were kept at room temperature for 24 h and weighed. The aggregate separation was repeated several times until sufficient sub-fractions were obtained for analysis. Finally, the soil samples from 0 to 12 cm, 12 to 24 cm and 24 to 36 cm depths were separated into seven WSA sizes, as follows: >5, 2–5, 1–2, 0.5–1, 0.25–0.5, 0.053–0.25 and <0.053 mm.

2.3 | Characterization of soils and water stable aggregates

2.3.1 | Basic properties

An electrode was used to measure the pH of the soil in a slurry with a soil-to-water ratio of 1:5. The total Fe (Fe_T) and Al (Al_T) contents of the soil were determined by a polarized wavelength dispersive X-ray fluorescence spectrometer (Axios PW4400) after the fusion of the dried samples (0.7 g) and Li₂B₄O₇ (7 g) into a glass disc (Wu et al., 2018). The basic properties of soil samples are shown in Table S1.

2.3.2 | Organic carbon and total nitrogen

The extensive free particulate organic matter (POM) and a small amount of Ca in the soil may disturb the relationship expression of SOC and aggregates in this study. For this purpose, POMs and Ca were removed by successively treating the sample with 0.5 mol/L HCl solution and deionized water (Milli-Q water) and freeze-dried. This pre-treatment was performed in triplicate; the three replicates were then combined and collected as one sample. The total organic carbon (TOC) and total nitrogen (N) contents of the samples were determined by a combustion method using an element analyser (Vario El III, Elementar, Germany) with an internal standard (Standard sample no. IVA99994) and a minimum of 10% blind replicates. The C/N ratio was calculated as the atomic ratio of TOC and TN.

2.3.3 | Different forms of Al-/Fe-(hydr)oxides

The Al-/Fe-(hydr)oxides in soil and WSA samples were extracted by sodium dithionite-sodium citrate-sodium bicarbonate (Al_{DCB} and Fe_{DCB} for abbreviation). The extracted Al/Fe phases included, but were not limited to, crystalline Al/Fe-(hydr)oxide phase, short-range-order

Al/Fe-(hydr)oxide phase and organo-Fe/Al complexes (Cornelis et al., 2018). In this study, we followed the protocol proposed by Zhao, Chen, et al. (2017). Briefly, 0.5 g sample was placed in a 50-ml centrifuged tube with the addition of sodium citrate (20 ml, 0.3 mol/L) and sodium bicarbonate (2.5 ml, 1 mol/L, pH 7.3) solutions. The mixture was placed in a water bath at 85°C, and sodium dithionite (0.5 g) was added. The mixture was stirred for 15 minutes, cooled and subjected to centrifugation. The supernatant was transferred into a 250-ml volumetric flask, and the resulting residue was washed with 1 mol/L NaCl twice, which was added to the supernatant.

Ammonium oxalate solution was used to extract Fe (Fe_{AO}) and Al (Al_{AO}) from soil and WSA samples (Cornelis et al., 2018; Zhao, Chen, et al., 2017). The extracted Al/Fe included, but were not limited to, ferrihydrite and organo-Fe/Al complexes (Dahlgren, 1994; Rennert, 2019). Briefly, 0.5 g of soil sample was added to 25 ml of ammonium oxalate (0.2 mol/L). The mixture was shaken for 2 h in dark conditions and centrifuged to separate the supernatant.

OC will interfere with the determination of Fe and Al. Therefore, the supernatant was digested with concentrated sulphuric acid and H_2O_2 . Briefly, 5 ml of supernatant were added into a Teflon digestion tank and heated at 120°C for concentration, and 1 ml of concentrated sulphuric acid was added, and the temperature increased to 340°C until fuming, cooled slightly, and 3 ml of H_2O_2 were added repeatedly until the solution becomes clear. The concentrations of Fe and Al were measured at wavelengths of 370 nm and 600 nm by visible spectrophotometry (Cary 300 UV-VIS spectrophotometer, Agilent Technologies, America) using Ferron colorimetry (Hu et al., 2001; Zhang et al., 2013). Estimations of Fe and Al extracted by DCB and ammonium oxalate solutions were completed in multiple replications (12 for soil sample * 2 for extracted supernatants) for quality assurance.

2.4 | Statistical analysis

The average size of aggregates in soil of each depth was evaluated by mean weight diameter (MWD), and calculated using the following equation (Kemper & Rosenau, 1986):

$$MWD = \sum_{i=1}^n x_i w_i$$

where x_i is the mean diameter of each size of aggregate (mm); w_i is the percentage of each aggregate size to the total weight of the analysed sample (%) and n is the number of different aggregate sizes.

TABLE 1 Aggregate size distribution (% by mass; mean \pm standard deviation) of three horizons in studied soil

| Aggregate size (mm) | Depth increment (cm) | | |
|---------------------|----------------------|----------------|----------------|
| | 0–12 | 12–24 | 24–36 |
| >5 | 59.4 \pm 0.3 | 31.5 \pm 0.9 | 8.7 \pm 0.3 |
| 5–2 | 19.4 \pm 0.5 | 30.3 \pm 0.5 | 17.4 \pm 0.8 |
| 2–1 | 7.2 \pm 0.1 | 15.0 \pm 0.8 | 16.4 \pm 0.9 |
| 1–0.5 | 6.9 \pm 0.3 | 12.0 \pm 0.6 | 22.1 \pm 0.6 |
| 0.5–0.25 | 3.8 \pm 0.2 | 5.6 \pm 0.2 | 16.9 \pm 0.6 |
| 0.25–0.053 | 2.2 \pm 0.1 | 3.5 \pm 0.1 | 11.4 \pm 0.6 |
| <0.053 | 1.0 \pm 0.0 | 2.1 \pm 0.1 | 6.1 \pm 0.3 |
| Recovery (%) | 99.99 | 100.00 | 98.90 |
| MWD (cm) | 3.8 | 3.0 | 1.5 |

Notes: Shown are means \pm standard deviation of 20 independent replicates.

Significant differences of Fe and Al contents in varied sizes of soil aggregates were determined using one-way analysis of variance (ANOVA). The correlations between OC, C/N atomic ratio and Al-/Fe-(hydr)oxide phases in aggregates of different sizes across all depths were determined by the Pearson correlation test ($n = 21$ for 3 depths of soils and the 7 aggregate sizes for each depth). Statistical analyses were performed using IBM SPSS Statistics (version 20).

3 | RESULTS

3.1 | Size distributions of aggregates

Mass recoveries after aggregate-size fractionation were in the range of 98.90%–100% (Table 1). The MWDs of aggregates decreased from 3.8 mm at 0–12 cm depth to 1.5 mm at 24–36 cm depth. The fraction of >5 mm aggregate size sharply reduced with soil depth, from 59.4% at 0–12 cm depth, to 31.5% at 12–24 cm depth, and then to 8.7% at 24–36 cm depth. In contrast, the proportions of <2 mm aggregates increased with soil depth.

3.2 | Concentrations of Al-/Fe-(hydr)oxides

The concentration of various Al-/Fe-(hydr)oxides in WSA size fractions are presented in Figure 1 and Table S2. Fe_{AO} , Al_{DCB} and Al_{AO} concentrations increased at 0–12 cm depth and decreased at 24–36 cm depth, respectively, as WSA size decreased. For example, at 0–12 cm depth, Al_{AO} increased from 2.41 g- Al_{AO} /kg-aggregate in aggregates of >5 mm size to 3.81 g- Al_{AO} /kg-aggregate in <0.053 mm aggregates size. In contrast, at 24–36 cm

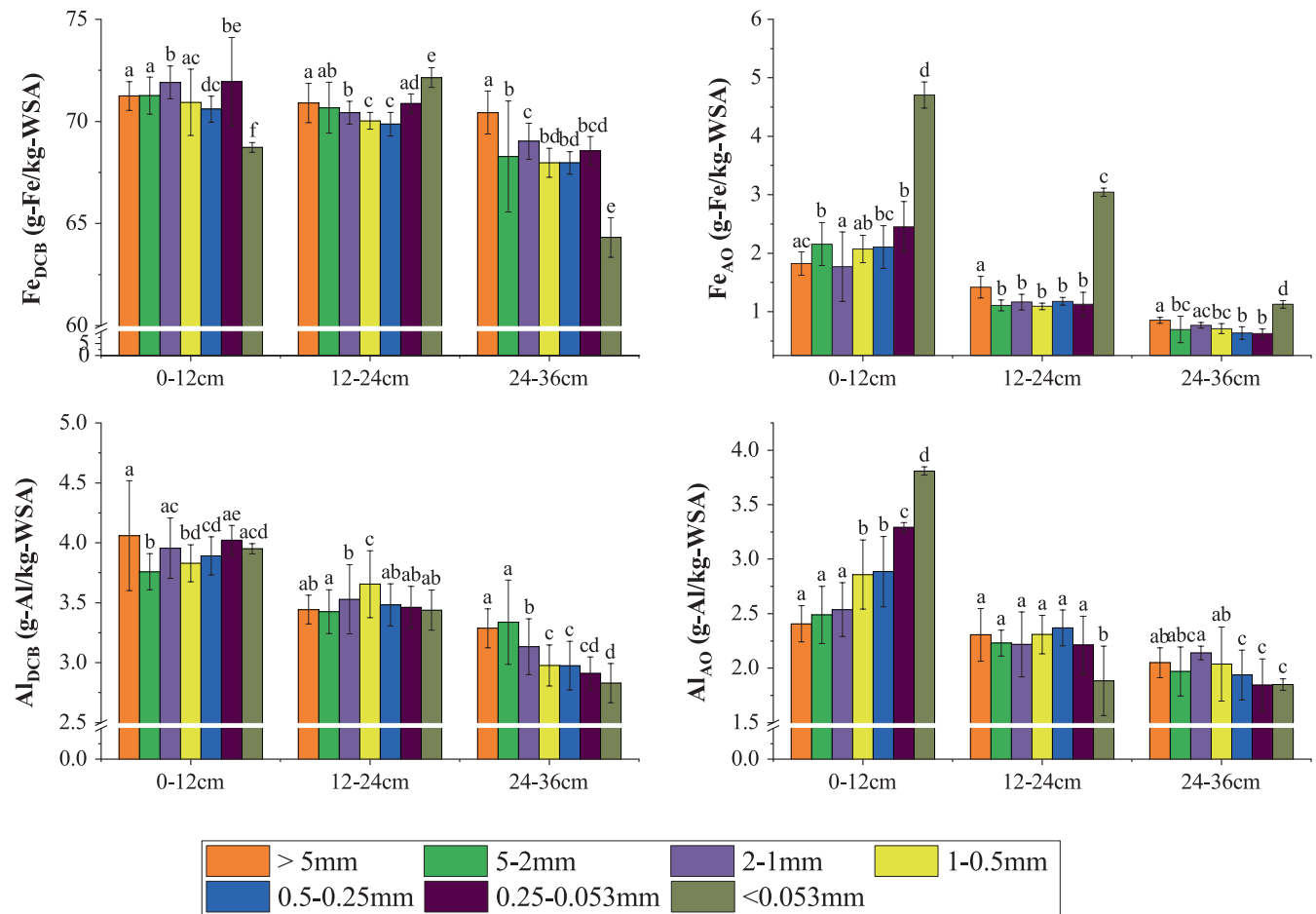


FIGURE 1 Concentrations of the various Al-Fe(hydr)oxides in WSA size fractions (g-Fe[Al]/kg-WSA). Fe_{DCB}/Al_{DCB}: Fe/Al extracted by DCB (dithionite-citrate-bicarbonate); Fe_{AO}/Al_{AO}: Fe/Al extracted by ammonium oxalate. Different lower case letters indicate significant differences of Fe concentrations among aggregate sizes at $p < .05$

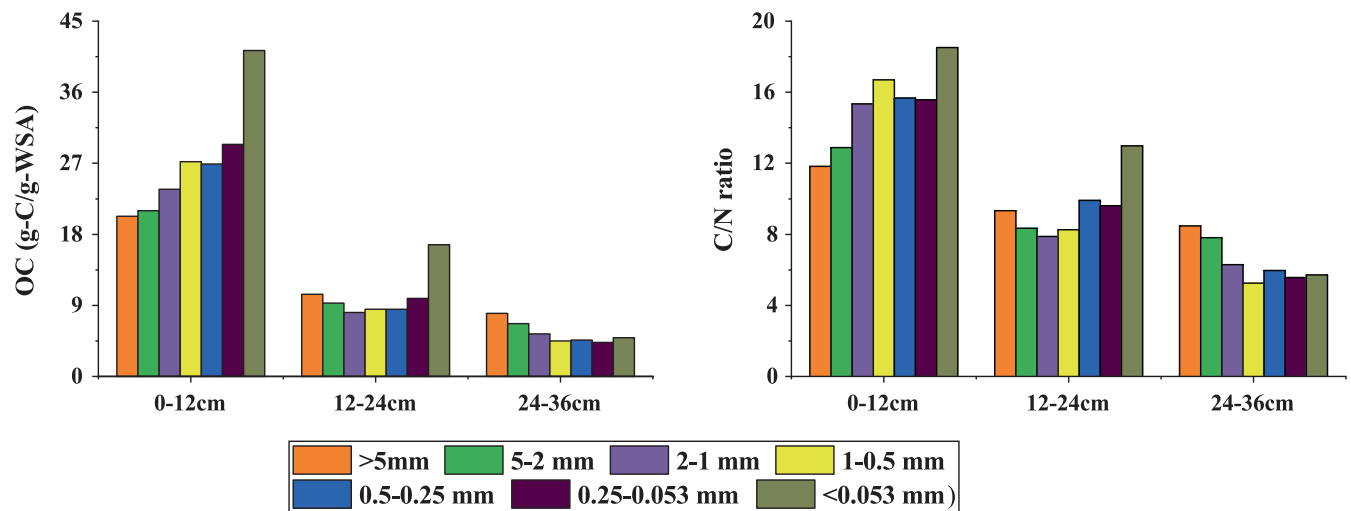


FIGURE 2 Concentrations of the organic carbon (OC, g-OC/kg-WSA) and C/N ratio in water stable aggregates (WSA) size fractions

depth, Al_{AO} decreased from 2.05 g-Al_{AO}/kg-aggregate in >5 mm sized aggregate to 1.85 g-Al_{AO}/kg-aggregate in <0.053 mm sized aggregate. Both at 0–12 cm and 24–36 cm

depths, a consistent but limited decreasing trend with decreased aggregate size were observed for Fe_{DCB} concentration. For example, at 24–36 cm, the Fe_{DCB} decreased from

70.43 g-Fe_{DCB}/kg-aggregate in >5 mm aggregate size to 64.32 g-Fe_{DCB}/kg-aggregate in 0.25–0.053 mm sized aggregate with decreasing aggregate sizes.

3.3 | Organic carbon concentrations and C/N atomic ratios of water stable aggregates

Recovery of OC and C/N ratio in WSA size fractions were in the range of 107%–117% and 83%–88%, respectively (Table S3). The OC concentrations varied significantly across the macro-aggregate size fractions, and the relationships between the OC concentration and aggregate size varied significantly across soil depths (Figure 2 and Table S3). For example, at 0–12 cm depth, the OC concentrations in >5 mm and 0.25–0.053 mm aggregates were 21.1 and 29.6 g kg⁻¹, respectively, and increased with decreasing aggregate size; however, at 24–36 cm depth, OC concentrations were 8.2 g kg⁻¹ in >5 mm aggregates to 4.3 g kg⁻¹ in 0.25–0.053 mm aggregates and decreased as aggregate size decreased. At 12–24 cm depth, the OC concentration decreased firstly and then increased with decreasing aggregate size, and was found to be lowest in 2–1 mm aggregate. At three soil depths, OC concentrations were higher in aggregate groups of <0.053 mm than in aggregate groups of 0.25–0.053 mm.

The relationships between soil C/N atomic ratio and aggregate size also varied across soil depths and were analogous to those observations for OC concentrations (Figure 2). Namely, the C/N ratio increased and decreased at 0–12 cm depth and 24–36 cm depth, respectively, as the aggregate size decreased.

OC concentration ($r \geq 0.83$, $p < .001$) and C/N ratio ($r \geq 0.84$, $p < .001$) were found to be significantly positively correlated with Al/Fe-(hydr)oxide (especially Al_{DCB}, Fe_{AO} and Al_{AO}) concentration in WSA size fractions (Figure 3). Significant positive correlation of C/N ratio and OC concentration was also observed among WSA size fractions across three depths ($r = 0.97$, $p < .001$; Figure 3). However, Fe_{DCB} concentration was found to be weakly correlated with OC concentration ($r = 0.44$, $p < .05$; Figure 3) and C/N ratio ($r = 0.57$, $p < .01$; Figure 3).

4 | DISCUSSION

4.1 | Roles of soil organic carbon and Al-/Fe-(hydr)oxides in soil aggregation

The OC concentrations and C/N ratios of WSA size fractions generally increased with WSA size because the fine roots and hyphae exhibit significant contributions to macroaggregate formation (Elliott, 1986; Maltoni et al., 2017). This relationship is most common in soils with high SOC and permanently charged phyllosilicate clay (Oades & Waters, 1991). In contrast, Asano and Wagai (2014) reported little difference in OC content and C/N ratios among the macroaggregate size fractions in an allophanic Andisol characterized by both high numbers of SOC and short-range-order (SRO) minerals. In this study, the OC concentration decreased and increased with the size of the WSA at 0–12 cm and 24–36 cm depths of the soil profile, respectively. In line with our study, Zhang et al. (2016) also reported diverse relationships between OC concentrations and WSA sizes across three soil profiles, that is



FIGURE 3 Pearson's correlation coefficients between Al-/Fe-(hydr)oxides, OC and C/N atomic ratio in WSA size fractions (sample number $n = 21$, including three depths of soil samples and 7 aggregate fractions for every soil depth). Notes: Levels of statistical significance: * for $p \leq .05$, ** for $p \leq .01$, and *** for $p \leq .001$)

OC concentration increased as aggregate size decreased at depths of 0–10 and 10–20 cm, but not at the 20–30 cm depth. The inconsistent relationships between OC concentration and aggregate size among soil depths may be owing to Al-/Fe-(hydr)oxides, which play a critical role in a variety of processes in SOC stabilization (Lalonde et al., 2012; Wagai & Mayer, 2007; Zhao, Adhikari, et al., 2017) and soil aggregation (Barthes et al., 2008; Six et al., 2002).

The observed decreased OC concentration (or C/N ratio) with decreasing WSA size at 24–36 cm depth was analogous to previous studies. This effect may be associated with the aggregating role of SOC and the physical protection of aggregates to SOC (Six et al., 2002). The observed negative correlation of OC concentration (or C/N ratio) and WSA size at 0–12 cm depth may be because of the presence of Al-/Fe-(hydr)oxides. As proposed by Barthes et al. (2008) and Xue, Huang, Huang, Zhou, et al. (2019), some Al-/Fe-(hydr)oxides, especially the ones extracted by DCB and ammonium oxalate solutions, play a dominant role in the relationships between soil aggregates and SOC. In studies of relationships of Al-/Fe-(hydr)oxide and aggregation, Xue, Huang, Huang, Zhou, et al. (2019) have reported that Fe_{DCB} is important in aggregate stability in two paddy soils, but Xue, Huang, Huang, Yin, et al. (2019) also have suggested that Fe_{DCB} is the weakest binding agent for all aggregate fractions. These different results may be owing to the different soil types and redox conditions (i.e. paddy soils and upland soils).

4.2 | Roles of organo-oxide associations in soil aggregation

Individual Al-/Fe-(hydr)oxides participate in the aggregation of high oxide soils (Barthes et al., 2008; Peng et al., 2015; Six et al., 2004). However, Six et al. (2002) revealed that aggregates bound by individual Al-/Fe-(hydr)oxides might easily break down by slaking despite strong resistance to mechanical stress. In addition, Figure 3 shows that Al-/Fe-(hydr)oxides and SOC are associated with each other rather than separated phases. For example, although particulate organic carbon may be excluded (Lalonde et al., 2012; Wagai & Mayer, 2007; Zhao, Adhikari, et al., 2017), Wagai and Mayer (2007) and Zhao et al. (2016) found that more than 40% of total SOC attributes to Fe-bound SOC. Grand and Lavkulich (2011) reported that organically complexed metals, SRO inorganic phases and clay content together explain 58% of the total variance in SOC concentration. Al-/Fe-(hydr)oxides accounted for a greater proportion of OC in this study because POM materials were removed before OC determination (Section 2.2). The association of OC and Al-/Fe-(hydr)oxides is usually mediated by the crystallinity

of Al-/Fe-(hydr)oxides. The association of OC with SRO ferrihydrite is especially important because of the high hydration, surface area and variable charges of SRO ferrihydrites (Dublet et al., 2017; Fritzsche et al., 2021; Torn et al., 1997). This study demonstrated the significant positive correlations of OC (as well as C/N ratio) and Al_{DCB} , Fe_{AO} , and Al_{AO} among the WSA size fractions from all three depths (Figure 3). In this regard, it is reasonable to assume that in wet sieving, SOC and Al-/Fe-(hydr)oxides associates contribute to aggregation rather than individual SOC and/or Al-/Fe-(hydro)oxide phase.

4.3 | Role of Organic carbon/(Fe + Al) ratio in aggregation

It is suggested that organo-oxide associations (OOAs) built aggregates in this soil. Thus, the force strength between OOAs affects the stability of aggregate, which in turn determines whether aggregates are destroyed during wet sieving. When the force between intra-aggregates is less than the destructive force caused by water dispersion, larger aggregate will break down to smaller ones. The OC/(Fe + Al) ratio provides information about the possible nature of associated OC and the types of SOC-mineral interactions (Huang et al., 2021; Kaiser & Guggenberger, 2007) and indicates interaction strength between two OOAs. Kleber et al. (2007) suggested that low OC content is retained through strong interactions with minerals (e.g. adsorption), and as OC content increases, the OC is further stabilized through weak forces, such as hydrophobic interactions, cation bridging, hydrogen bonding and other interactions. In this study, we calculated the OC/(Fe + Al) ratios of WSAs to evaluate the strength of OC and Al-/Fe-(hydr)oxide association. Fe_{DCB} concentrations in WSA size fractions were found to be weakly correlated with OC in the preceding discussion (Figure 3). Therefore, Fe_{DCB} was excluded from the OC/(Fe + Al) ratio calculations in this study.

Monolayer OC load on the sedimentary mineral surface is often in the order of 0.5–1.0 mg-OC/m²-mineral (Kleber et al., 2007), approximately equivalent to an atomic ratio of 1.1–2.2 mol-C/mol-Fe, assuming the specific surface area of the mineral is 224 m²g⁻¹ (freshly precipitated ferrihydrite (Fe₅H₉O₈·4H₂O [Wagai & Mayer, 2007])). Al-(hydr)oxides also acted as a protective layer for OC preservation in natural soils. Taking into account the Al-(hydr)oxides, the atomic ratio ought to be greater than 2.2 mol-C/mol-(Fe + Al). In this study, OC/(Fe + Al) atomic ratios of different WSA size fractions ranged from 1.89 to 3.13 at 24–36 cm depth (Table 2), suggesting the presence of a large fraction of OC in the form of a monolayer on the minerals. The OC/(Fe + Al) ratios of WSA size fractions

at 0–12 cm depth ranged from 6.22 to 9.27 (Table 2) and were higher than the monolayer load of OC by Al-/Fe-(hydr)oxide phases, implying that most OC may be associated with Al-/Fe-(hydr)oxides in multilayer form and stabilized via complex inter- and intra-molecular binding mechanisms (Kleber et al., 2007) at 0–12 cm depth. Souza et al. (2017) discovered that SOC overloads the reactive minerals in the topsoil of Oxisols, and they attribute SOC to multilayer accumulation in minerals. Additional layers of OC will be retained in Al-/Fe-(hydr)oxides by relatively weak forces (e.g. hydrogen bonds). Therefore, OC readily dissociates from Al-/Fe-(hydr)oxides (Souza et al., 2017). In this regard, the amount and stability of OC on Al-/Fe-(hydr)oxide may influence soil aggregation. This suggestion is supported by laboratory experiments.

TABLE 2 OC/(Fe + Al) molar ratios of aggregate size fractions in three horizons of the yellow soil

| Aggregate size (mm) | OC/(Fe + Al) (Mol/Mol) | | |
|---------------------|------------------------|----------|----------|
| | 0–12 cm | 12–24 cm | 24–36 cm |
| >5 | 6.22 | 3.64 | 3.13 |
| 5–2 | 6.49 | 3.38 | 2.67 |
| 2–1 | 7.26 | 2.89 | 2.15 |
| 1–0.5 | 7.96 | 2.95 | 1.89 |
| 0.5–0.25 | 7.77 | 2.98 | 1.98 |
| 0.25–0.053 | 7.79 | 3.58 | 1.91 |
| <0.053 | 9.27 | 5.53 | 2.11 |

For instance, Guhra et al. (2019) discovered that excessive biogenic OC coatings could induce electrostatic repulsion between (micro-)aggregate forming materials, thereby inhibiting liquid phase aggregation. Narvekar et al. (2017) have reported weak and strong aggregations of haematite-extracellular polymeric substances associations and silica at high and low EPS loadings, respectively.

4.4 | Mechanism of aggregation

Given the differences in OC/(Fe + Al) ratios in WSA size fractions in different depths, three types of organic-Al-/Fe-(hydr)oxide associations (OOAs) are suggested to exist arbitrarily in yellow soils (Figure 4). These types included Type A, in which OC is associated with Al-/Fe-(hydr)oxides in the form of multilayer, with a high OC concentration and a high OC/(Fe + Al) ratio; Type C, in which OC is highly unsaturated on/in Al-/Fe-(hydr)oxides, with a low OC content and a low OC/(Fe + Al) ratio; and Type B, with a medium OC concentration and a medium OC/(Fe + Al) ratio. Three types of OOAs were all hypothesized to be structural units and bound together into soil WSAs, and three scenarios of aggregation were assumed to exist for simplicity (Figure 4). In practice, WSAs formed by scenario I were found to be unstable because of the high concentration of OC on, or in, the Al-/Fe-(hydr)oxides. As previously stated, the outer regions of OC stabilized in OOAs of Type A were held together by relatively weak forces (e.g. hydrogen bonds) and easily detached from

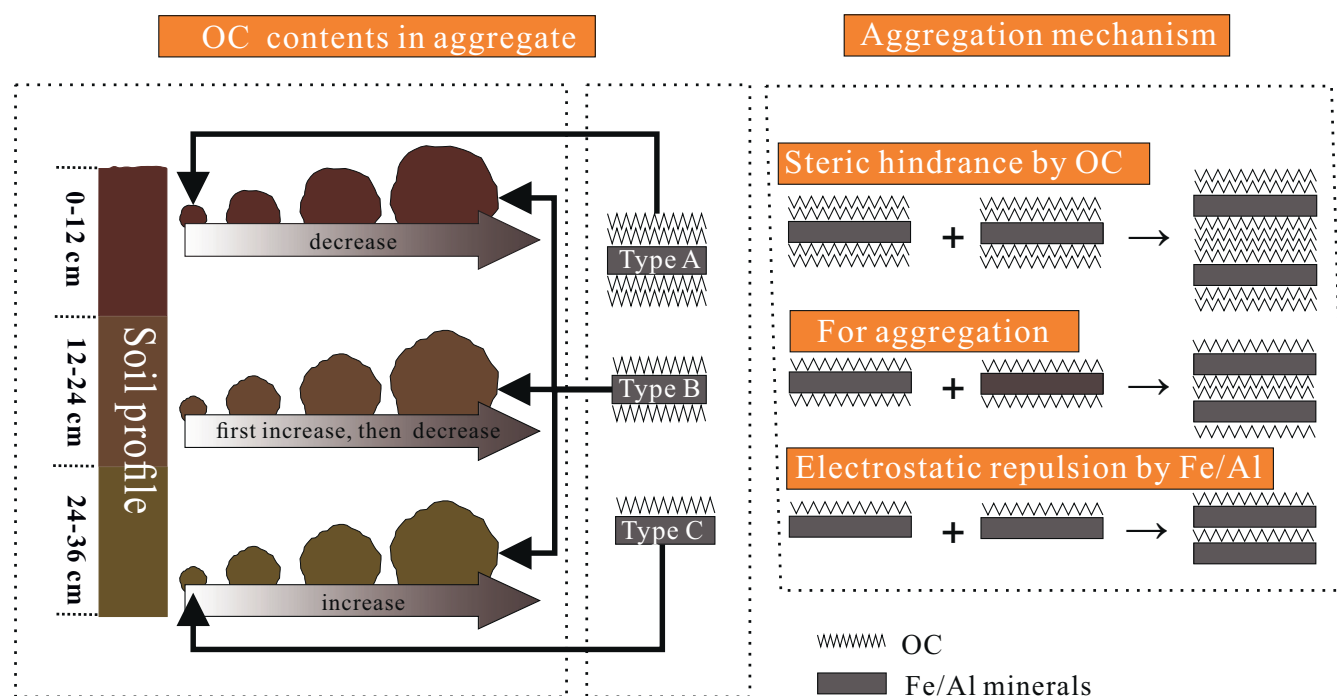


FIGURE 4 Aggregation mechanism for yellow soil

Al-/Fe-(hydr)oxides (Souza et al., 2017). Consequently, WSAs formed by the scenario I may be unable to resist water dispersion during wet sieving. While because of steric hindrance or electrostatic repulsion between Al-/Fe-(hydr)oxides, Scenario III is also unstable. In addition, low OC coverage exposes large numbers of Al-/Fe-(hydr)oxide surfaces in OOAs of type C. Consequently, the aggregates formed by scenario III likely resemble the aggregates formed by individual Al-/Fe-(hydr)oxides and will have low 'slake' stability (Six et al., 2002). In this context, the most stable and prevalent aggregates in soils are likely to be formed by scenario II, which involves OOAs of type B (with a medium OC/(Fe + Al) ratio).

The disparate relationships between OC concentration and WSA size at 0–12 and 24–36 cm depths (Figure 2 and Table S3) may be because of the variation in OC/(Fe + Al) ratios at 0–12 cm depth and 24–36 cm depth (Table 2). The used soil showed a significant decrease in the OC/(Fe + Al) ratios with soil depth (Table 2), indicating that OOAs of Type A reduced and OOAs of Type C increased with soil depth. When OOAs of Type B (with a medium OC/(Fe + Al) ratio) were preferentially bound to large aggregates, OOAs of Type A (with a high OC/(Fe + Al) ratio) were left in smaller aggregates at 0–12 cm depth, resulting in a decrease in OC concentrations (or OC/(Fe + Al) ratio) with increasing of aggregate sizes. In comparison, few OOAs of Type A and an increasing number of OOAs of Type C existed in the soil of 24–36 cm depth because OOAs of Type B were preferentially chosen to form large aggregate, while Type C of OOAs remained in smaller aggregates. Therefore, the OC concentration increased as the aggregate size increased.

Rumpel and Kögel-Knabner (2011) suggested that the mechanisms of aggregation in subsoils may be quite distinct from those in topsoil and that physical processes, such as root twine are thought to be more important in subsoils than biological aggregation. In this study, plant debris (including leaves and roots) was removed as much as possible before the determination of the OC and C/N ratio. To a large extent, we excluded the aggregation caused by root twine. Therefore, the proposed aggregation mechanism can account for soil aggregation at three depths. According to this mechanism, an abundance of OOAs of type A in the soil is harmful to soil aggregation. Although OC/(Fe + Al) ratios of WSAs at 0–12 cm depth were 2–4 times than those at the 24–36 cm depth, OOAs of type B were likely to be abundant, rather than OOAs of type A, because OC in OOAs of type A stabilized in the outer region of minerals and was retained by relatively weak forces and only existed for a short time (Kleber et al., 2007). Therefore, it stands to reason that the number of water-stable macro-aggregates decreases with depth (Table 1) because the number of OOAs of type B and Al-/

Fe-(hydr)oxides (primarily involving Al_{DCB}, Fe_{AO} and Al_{AO}; Table S1) decreases with soil depth.

4.5 | Suggestions for soil management

Organic amendment (compost, manure, crop straw, etc.) is an important soil management practice for carbon sequestration and soil erosion mitigation in the context of global climate change and land degradation. However, inconsistent results have been reported in the literature regarding the responses of organic material application to soil aggregation. Generally, a low level of organic material input promotes macro-aggregation, whereas a high level tends to reduce the proportion of macroaggregates (Xie et al., 2015). The effects of organic incorporation on soil aggregation may be related to the initial SOC saturation of organo-mineral association (Blanco-Moure et al., 2012; Das, Chakraborty, Singh, Aggarwal, Singh, Dwivedi, & Mishra et al., 2014), specifically the capacity to stabilize exogenous OC. The OC/(Fe + Al) ratio was an important indicator of soil aggregation stability in this study. The organic material application increases the content of OOAs of Type B in soils with a medium OC/(Fe + Al) ratio, which increases opportunities for macro-aggregation. However, if the OC/(Fe + Al) ratio of soil is initially high, the soil often maintains a good aggregation structure because of an adequate amount of OOAs of Type B and an appropriate amount of OOAs of Type A. Additional OC input will result in initial OOAs of Type B accruing more OC and converting to OOAs of Type A, thereby affecting soil aggregation.

5 | CONCLUSIONS

This study integrated the roles of OC and Fe-/Al-(hydr)oxides in soil aggregation and proposed a mechanism of soil aggregation that emphasized the importance of the OC/(Fe + Al) ratio in the formation of soil aggregates in yellow soils. Briefly, only an appropriate amount of OC associated with Fe-/Al-(hydr)oxides (with medium OC/(Fe + Al) ratios) could effectively form large aggregates that are stable against breakdown during wet sieving. A less or greater amount of OC associated with Fe-/Al-(hydr)oxides (with low and high OC/(Fe + Al) ratios, respectively) will inhibit availability because of steric hindrances of OC and electrostatic repulsion of Al-/Fe-(hydr)oxide. Although some questions in terms of soil processes can be explained by this mechanism, the generality of the proposed mechanism should be confirmed in further studies of different soils with high Fe and Al contents. Furthermore, attention must be paid when applying this mechanism to other

soil types because soil-type-specific mineral phases, especially 2:1 clay, can have a significant impact on SOC stabilization and aggregation. Nevertheless, given the sustainable development goals, deep studies are needed to guide soil organic management to healthy soil and carbon sequestration.

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CONFLICT OF INTEREST

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

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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