



# Factors contributing to aggregate stability at different particle sizes in ultisols from Southern China

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## Abstract

**Purpose** The main objective of this study was to investigate the effects of abiogenic and biogenic factors, and their interaction, on aggregate stability determined at different particle sizes.

**Materials and methods** Soil samples with the same land use pattern were collected and fractioned into five aggregate sizes: 10–15 mm, 5–10 mm, 2–5 mm, 0.25–2 mm, and < 0.25 mm. Contents of iron/aluminum (Fe/Al) oxides, soil organic carbon (SOC), clay, and mean weight diameter (MWD) values for aggregates at different sizes were determined. The respective contributions of these factors were further estimated using path analysis.

**Results and discussion** The results showed that SOC contents in A horizon declined with the increase of aggregate size. Highest amorphous iron oxide (Fe<sub>o</sub>) contents were observed in 0.25–2 and 2–5 mm aggregates, but highest amorphous aluminum oxide (Al<sub>o</sub>) contents were found in 5–10 mm aggregates. Abiotic factors (Fe/Al oxides, clay) played a more important role in determining the formation of < 0.25 mm aggregates, whereas both abiotic and biotic factors play an effective role in stabilizing larger aggregates (0.25–2, 2–5, 5–10, and 10–15 mm). The organo-mineral complexes played a certain role in the stability of soil aggregates, especially the larger aggregates.

**Conclusions** We conclude that abiotic and biotic factors play variable roles in soil aggregates at different sizes, and more studies are needed to better assess their respective roles to improve our understanding of soil aggregation.

**Keywords** Aggregate size · Mean weight diameter · Path analysis · Sesquioxides · Soil organic carbon · Ultisols

## 1 Introduction

Soil structure is one of the important soil properties, which can affect the movement and storage of soil water, air, heat, and nutrients, and also influence the ability of soils to resist

erosion (Saygin et al. 2012; Chaplot and Cooper 2015; Peng et al. 2015; Wu et al. 2016). Generally, the stability of soil aggregates is commonly adopted to measure the soil structure (Bronick and Lal 2005; Regelink et al. 2015; Almajmaie et al. 2017). Recent researches on the formation and stability of soil aggregates have been gaining attention (Wu et al. 2016).

Soil aggregate stability may be affected by many factors including soil organic matter, soil microbes, land use, iron and aluminum (Fe/Al) oxides, and tillage. Both the inorganic (e.g., sesquioxides and clay) and organic (e.g., soil organic carbon) agents involved in soil aggregation have been studied intensively (Six et al. 2004; Bronick and Lal 2005; Igwe et al. 2009). It has been widely accepted that positive correlations exist between soil aggregate stability and sesquioxides or soil organic matter (Boix-Fayos et al. 2001; Six et al. 2004; Noellemeyer et al. 2008; Zhao et al. 2017). Several studies indicated that sesquioxides may play a more important role in stabilizing aggregates in oxisols (Pinheiro-Dick and Schwertmann 1996; Six et al. 2002; Wang et al. 2016; Igwe

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et al. 2009). Barthès et al. (2008) reported that Al-containing sesquioxides (particularly Al-substituted crystalline hematite and goethite) rather than SOC were the main aggregating agent in tropical soils. It was also reported that the Fe and Al oxides played a more significant role in binding particles together in water-stable aggregates ( $> 100 \mu\text{m}$ ) in the oxide-rich soils (Tisdall and Oades 1982; Oades and Waters 1991). The biotic and abiotic aggregation processes are often interactional, whereas there is a lack of knowledge about the difference in their roles in aggregate stability.

The coexistence of these inorganic and organic agents in soil makes assessment of their respective contributions to soil aggregation difficult. Until now, no standard method has been proposed to weigh the contributions of biological and abiotic processes to the formation and stability of soil aggregates (Peng et al. 2015). By means of structural equation models, Barto et al. (2010) assessed the contributions of biotic and abiotic factors to soil aggregation, and suggested that abiotic factors played a more important role in stabilizing aggregates than did biotic factors. Unfortunately, they did not investigate the respective contributions of sesquioxides and SOC to stability of aggregates at different sizes. Assessing the aggregate size distribution of the soil extracted by dithionite–citrate–bicarbonate (DCB), oxalate, and  $\text{H}_2\text{O}_2$ , Peng et al. (2015) found that the Fe and Al oxides played a more significant role in determining the stability of  $< 0.25 \text{ mm}$  aggregates, whereas the SOM can be more important for determining stabilizing larger aggregates (0.25–2.00 mm). However, they also failed to assess exactly the ratios of contribution of biotic and abiotic agents to stabilizing aggregates of different size classes.

Until now, most studies have focused on identifying major factors determining soil aggregation, but researches on the causal relationships among these factors remain lacking. Path analysis, which was proposed by Wright (1934), permits analysis of specific indirect relationships between these factors as well. Here, we introduce the path analysis to explore the effects of abiogenic and biogenic factors, and their interaction, to determine their respective contribution to the stability of aggregates at different sizes.

Thus, the objectives of this study were to (i) investigate the spatial and profile distribution of sesquioxides in the bulk soils and aggregates at different sizes, (ii) determine the stability of soil aggregates at different sizes, and (iii) assess the contributions of sesquioxides and SOC to the stability of aggregates at different sizes using path analysis.

## 2 Materials and methods

### 2.1 Sample collection

Five soil profiles (S1, S2, S3, S4, and S5) have been studied in Jiangxi Province, Southern China (Fig. 1). At each sampling

site, we dug a soil profile and collected soil samples in three different profile horizons, including eluvium (A horizon), illuvial (Bt horizon), and parent material horizon (C horizon) in April 2016 (Table 1). Fifty samples were collected from Jinxian county (S1 and S4), Xinjian county (S2 and S3) of Nanchang city, and Gongqingcheng county (S5) of Jiujiang city. In addition, we also obtained undisturbed soil cores ( $200 \text{ cm}^3$ ) using the cutting ring method to determine the bulk density and soil moisture. Once collected, the samples were transported to Jiangxi Provincial Key Laboratory of Soil Erosion and Prevention in Jiangxi Eco-Science Park of Soil and Water Conservation ( $115^\circ 42' 38'' \text{ E}$ – $115^\circ 43' 06'' \text{ E}$ ,  $29^\circ 16' 37'' \text{ N}$ – $29^\circ 17' 40'' \text{ N}$ ), which is located in De'an county, Jiujiang City, northern Jiangxi. The soils were gently broken up with hands in order to ensure no damage to the original structure. The soil samples were then fully air-dried.

The texture can be classified as clay or clay-loam according to the USDA (Soil Survey Staff 2010) (Table 1). The clay mineralogy is dominated by kaolinite. All soils are derived from quaternary clay (Zhao and Shi 1983). Terrain conditions for all sampling sites were of gentle slope or plain with slope gradients smaller than 16%. All sampling sites were wasteland covered with grass and not affected by human activities, so they preserved intact genetic soil profiles. The climate is subtropical with mean annual temperature and precipitation ranging from 16 to 18 °C and from 1366 to 1700 mm, respectively. The soils were classified according to the United States Department of Agriculture soil taxonomy as Plinthudults (Ultisols).

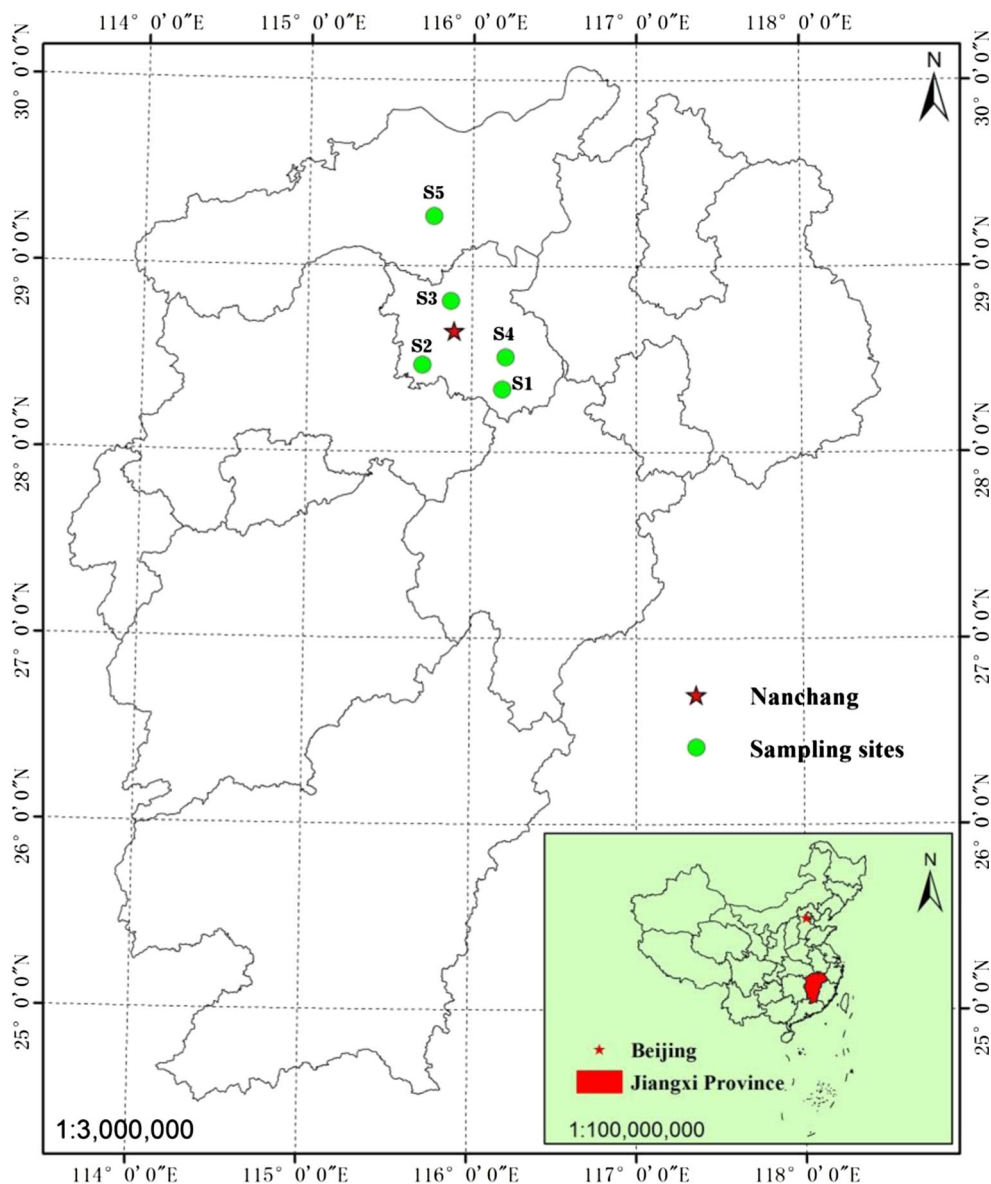
### 2.2 Physical and chemical analysis

The physicochemical properties of the bulk soils and aggregates were determined by standard analytical methods, including:

#### (a) Chemical properties

CEC was determined using ammonium acetate exchange method (Thomas 1982). pH was determined at 1:2.5 soil/water ratio in mass using a pH meter. Fe/Al oxides and SOC were determined for the bulk soils and the aggregate size fractions. Briefly, SOC was determined after acid digestion with potassium-dichromate (Walkley and Black 1934); free Fe/Al oxides ( $\text{Fe}_d/\text{Al}_d$ ) were extracted by DCB at 80 °C with a persistent disturbance for 10 min, according to the extraction procedure described by Jackson et al. (1986). Amorphous (non-crystalline) Fe/Al oxides ( $\text{Fe}_o/\text{Al}_o$ ) were extracted by ammonium oxalate (McKeague and Day 1966) in dark with shaking for 2 h on a reciprocating shaker. Inductively coupled plasma optical emission spectrometry (ICP-OES) (VISTA-MPX, Varian, USA) was used to determine Fe and Al concentrations in the extracts collected after centrifugation. Clay mineralogy was determined by powder X-ray diffraction

**Fig. 1** Location of the study area and sampling sites



(XRD) on a Bruker D8 Advance diffractometer (Bruker AXS GmbH, Karlsruhe, Germany) equipped with a LynxEye detector. The conditions for XRD were CuK $\alpha$  radiation ( $\lambda = 0.154$  nm), test voltage for 40 kV, test current for 40 mA, step scan with a speed of 10°/min, and step length of 0.01°.

#### (b) Physical properties

Soil moisture and bulk density (BD) was determined by the cutting ring method. Texture was determined by the hydrometer method (Gee and Bauder 1979).

#### (c) Aggregate fractionation

Before size fractionation, we removed the visible gravel and plant roots. After gentle, manual crumbling to < 15 mm, soils

were sieved by stacking sieves (10, 5, 2, and 0.25 mm) to partition the aggregate sizes. Sieves were shaken at 30 Hz on a constant temperature shaker. Each bulk soil sample was fractionated into five aggregate sizes: 10–15 mm, 5–10 mm, 2–5 mm, 0.25–2 mm, and < 0.25 mm through the routine dry-sieving procedure.

#### (d) Aggregate stability analysis (mean weight diameter (MWD))

For 10–15 mm, 5–10 mm, and 2–5 mm aggregates, soil aggregate stability was measured using wet-sieving method. In brief, the aggregate analyzer with four identical cylindrical water containers was used. Each cylindrical water container of the aggregate analyzer has a column of four sieves: 2, 1, 0.5, and 0.25 mm. In brief, 30 g of aggregates at each size was accurately weighted and placed on the topmost sieve, then immersed

**Table 1** Basic description of the sampling sites

Sampling sites	Longitude/latitude	Parent material	Soil horizon/depth (cm)	Slope	Altitude (m)
S1	116° 11' 06.1" E, 28° 20' 12.7" N	Quaternary red clay	A/0~75 Bt/75~100 C/80~	8–10%	47
S2	115° 41' 57.9" E, 28° 27' 52.1" N	Quaternary red clay	A/0~90 Bt/90~130 C/130~	10–12%	43
S3	115° 52' 11.4" E, 28° 48' 50.5" N	Quaternary red clay	A/0~30 Bt/30~50 C/50~	13–16%	51
S4	116° 12' 11.8" E, 28° 30' 41.5" N	Quaternary red clay	A/0~30 Bt/30~100 C/100~	11–14%	30
S5	115° 45' 49.6" E, 29° 17' 02.2" N	Quaternary red clay	A/0~30 Bt/30~140 C/140~	12–14%	57

completely in water. After 10 min, the motor-driven system was opened and the sieves were shaken with a vibration amplitude of 4 cm and a frequency of 30 cycles per min for 30 min. The remaining soils on each sieve were all collected and oven dried at 105 °C, weighed, and corrected for sand contents (Elliott et al. 1991). For 0.25–2 mm aggregate, 30 g of aggregates at such size were accurately weighted and firstly fractionated to 0.25–2.0 mm, 0.053–0.25 mm, and < 0.053 mm fractions by wet sieving. The remaining soils at 0.25–2.0 mm and 0.053–0.25 mm size were all collected and oven dried at 105 °C, weighed, and corrected for sand contents. The < 0.053 mm fraction was further transferred to a 1000-ml graduated cylinder, and diluted to 1000 ml by adding water. The proportions of 0.02–0.053 mm, 0.002–0.02 mm, and < 0.002 mm aggregates were determined using pipette method. Similarly, < 0.25 mm aggregate was fractionated to four aggregate sizes: 0.053–0.25 mm, 0.02–0.053 mm, 0.002–0.02 mm, and < 0.002 mm using wet sieving and pipette method. All analyses were run in triplicate and averaged for statistical analysis.

The MWD was calculated to characterize the stability of aggregate at different sizes, which can be expressed as follows (Kemper and Rosenau 1986):

$$MWD = \sum_1^{n+1} \frac{r_{i-1} + r_i}{2} \times m_i \tag{1}$$

where,  $r_i$  means the aperture of the  $i_{th}$  mesh (mm),  $r_0 = r_1$ ,  $r_n = r_{n+1}$ ;  $m_i$  is the sand-corrected mass percentage of aggregates remaining on each sieve; and  $n$  indicates the number of the sieves.

### 2.3 Data analysis

All data analyses were performed with IBM SPSS Statistics 19 software. All figures and tables were created in Microsoft Excel 2013 and SigmaPlot 12.5. One way ANOVA was used

to assess the least significant difference (LSD at  $p < 0.05$ ) among different treatments.

There are several factors affecting the aggregate stability, for example, Fe/Al oxides, SOC, and clay. Moreover, interrelationships exist among these factors. Therefore, each factor could influence the aggregate stability both directly and indirectly through other factors. To investigate the direct and indirect contributions of factors to the stability of aggregates at different sizes, the path model proposed by Wright (1934) was introduced as described elsewhere (Gui et al. 2017).

For each aggregate size, stepwise multivariate linear regression was adopted to identify all determinants that could account for the majority of MWDs (Xue 2013). We selected six independent variables including free iron oxide ( $Fe_d$ ), amorphous iron oxide ( $Fe_o$ ), free alumina oxide ( $Al_d$ ), amorphous alumina oxide ( $Al_o$ ), SOC, and clay content (clay). Then, the causal relationship between the dependent variable and the independent variables ( $X_1, X_2, \dots, X_m$ ) can be described in the path diagram in Fig. 2.

According to the path analysis method, the direct path coefficients of the determinants on the MWD of each aggregate size can be calculated as (Gui et al. 2017):

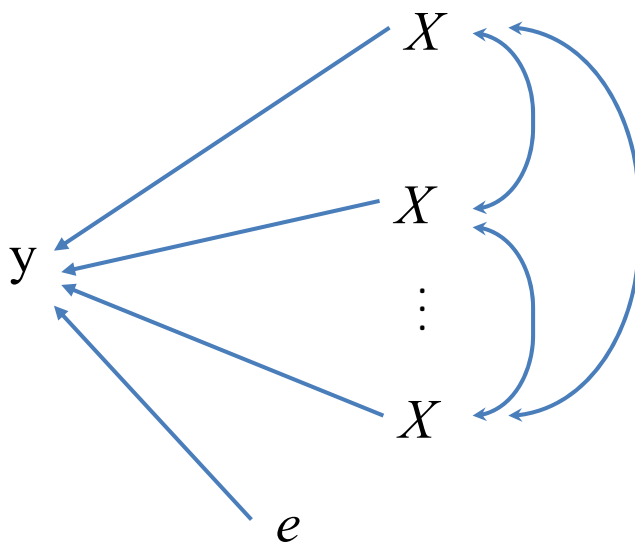
$$P_{m,y} = \alpha_m \times \frac{S_m}{S_y} \tag{2}$$

where,  $P_{m,y}$  indicates the direct path coefficient, representing the direct effect of independent variable  $m$  on dependent variable  $y$  (MWD);  $\alpha_m$  is a partial regression coefficient; and  $S_m$  and  $S_y$  mean the standard deviations of the corresponding independent variable and MWD, respectively.

Then, the indirect path coefficient can be described as:

$$IP_{m,n,y} = r_{m,n} \times P_{n,y} \tag{3}$$

where  $IP_{m,n,y}$  means the indirect path coefficient, representing the indirect effects of the determinant  $m$  through  $n$  ( $m \neq n$ ) on



**Fig. 2** Path diagram, where  $y$  indicates the dependent variable ( $MWDs$ );  $X_1, X_2, \dots, X_m$  indicate the independent variables affecting the  $MWDs$  for each aggregate size;  $e$  indicates the residual error

the dependent variable  $y$ ; and  $r_{m,n}$  indicates the correlation coefficient between  $m$  and  $n$ , where  $r_{m,n} = r_{n,m}$ .

Then,

$$r_{m,y} = P_{m,y} + \sum IP_{m,n,y} \quad (4)$$

where  $r_{m,y}$  is the total effects of independent variable  $m$  on dependent variable  $y$ .

### 3 Results

#### 3.1 Basic physicochemical properties of soils

Table 2 shows the basic physicochemical properties of the bulk soils. The pH values varied from 3.95 to 4.91, indicating that all soil samples were acid. CEC values ranged from  $9.30 \pm 1.45$  to  $19.92 \pm 2.96$   $\text{cmol kg}^{-1}$  with a mean of  $13.38 \pm 2.67$   $\text{cmol kg}^{-1}$ . The lowest CEC value ( $9.30$   $\text{cmol kg}^{-1}$ ) was found in S3-A and the highest value ( $19.92$   $\text{cmol kg}^{-1}$ ) was observed in S4-C. For S2, S3, and S4, the CEC values increased gradually with depth in the soil profile, whereas the highest CEC value was found in the A horizon for S1 and in the Bt horizon for S5. The soil moisture varied from  $21.72\% \pm 0.40\%$  to  $27.41\% \pm 1.17\%$  with a low CV of 10%. For S1–S3, the soil moisture decreased in the sequence  $A > Bt > C$ . For S4 and S5, the highest soil moisture was found in the Bt horizon. Bulk density ranged between  $1.29 \pm 0.00$ – $1.57 \pm 0.02$   $\text{g cm}^{-3}$  with a mean of  $1.43$   $\text{g cm}^{-3}$  and the lowest CV of 6%. The percentage of 5–10 mm aggregates was the highest for most of soils except S3-A, and the percentage of < 0.25 mm aggregates was the lowest. Similar percentage of aggregates at different sizes was found for all samples from different sites and soil horizons. For clay mineralogy, kaolinite was the dominant mineral for all soil samples (Table 3). A certain amount of vermiculite (expansive clay mineral) was also found in these soils, ranging between 13–23% with a mean of  $16.2\% \pm 4.1\%$ .

**Table 2** Basic physicochemical properties of bulk soil samples

Sampling sites	Soil horizon/ depth (cm)	pH	CEC ( $\text{cmol kg}^{-1}$ )	Soil water content (mean $\pm$ std, %, $n = 3$ )	BD (mean $\pm$ std, $\text{g cm}^{-3}$ , $n = 3$ )	Texture (%)			USDA texture class	Main mineralogy
						Sand (> 50 $\mu\text{m}$ )	Silt (2–50 $\mu\text{m}$ )	Clay (< 2 $\mu\text{m}$ )		
S1	A/0~75	4.08 $\pm$ 0.12	12.71 $\pm$ 0.89	23.84 $\pm$ 0.04	1.30 $\pm$ 0.09	30.51	32.79	36.70	Clay-loam	Kao-Ver-1.4 nm
	Bt/75~100	4.02 $\pm$ 0.31	11.89 $\pm$ 1.26	23.29 $\pm$ 0.04	1.47 $\pm$ 0.02	26.22	32.62	41.16	Clay	
	C/80~	4.01 $\pm$ 0.25	11.89 $\pm$ 1.09	21.78 $\pm$ 0.54	1.55 $\pm$ 0.02	28.18	38.67	33.15	Clay-loam	
S2	A/0~90	4.61 $\pm$ 0.16	11.15 $\pm$ 1.54	28.19 $\pm$ 0.06	1.34 $\pm$ 0.03	31.35	37.96	30.70	Clay-loam	Kao-Ver-III
	Bt/90~130	4.91 $\pm$ 0.18	12.87 $\pm$ 1.65	25.60 $\pm$ 2.17	1.50 $\pm$ 0.05	35.79	36.00	28.21	Clay-loam	
	C/130~	4.91 $\pm$ 0.42	13.09 $\pm$ 1.82	25.75 $\pm$ 2.75	1.47 $\pm$ 0.05	33.46	31.54	35.00	Clay-loam	
S3	A/0~30	4.01 $\pm$ 0.36	9.30 $\pm$ 1.45	23.75 $\pm$ 1.84	1.41 $\pm$ 0.09	32.62	12.05	55.33	Clay	Kao-Ver-III
	Bt/30~50	4.01 $\pm$ 0.54	15.50 $\pm$ 2.31	22.87 $\pm$ 0.55	1.52 $\pm$ 0.02	34.54	32.68	32.78	Clay-loam	
	C/50~	3.97 $\pm$ 0.59	17.77 $\pm$ 2.56	20.75 $\pm$ 0.38	1.57 $\pm$ 0.02	32.24	20.13	47.63	Clay	
S4	A/0~30	4.01 $\pm$ 0.34	12.39 $\pm$ 1.87	27.41 $\pm$ 1.17	1.37 $\pm$ 0.06	34.99	35.20	29.80	Clay-loam	Kao-Ver-1.4 nm
	Bt/30~100	3.96 $\pm$ 0.29	15.68 $\pm$ 1.59	29.74 $\pm$ 2.41	1.29 $\pm$ 0.00	32.81	30.93	36.27	Clay-loam	
	C/100~	3.95 $\pm$ 0.35	19.92 $\pm$ 2.96	26.06 $\pm$ 1.74	1.47 $\pm$ 0.00	28.55	37.09	34.35	Clay-loam	
S5	A/0~30	4.52 $\pm$ 0.19	10.73 $\pm$ 0.99	21.72 $\pm$ 0.40	1.35 $\pm$ 0.12	37.00	26.76	36.23	Clay-loam	Kao-Ver-III
	Bt/30~150	4.00 $\pm$ 0.17	14.33 $\pm$ 0.96	25.00 $\pm$ 1.38	1.40 $\pm$ 0.05	29.06	35.64	35.30	Clay-loam	
	C/150~	3.97 $\pm$ 0.24	11.55 $\pm$ 1.06	24.41 $\pm$ 0.65	1.44 $\pm$ 0.04	27.08	33.69	39.23	Clay-loam	
	Mean	4.20	13.38	24.68	1.43	31.63	31.58	36.79		
	CV/%	8	20	10	6	10	22	18		

SOC, soil organic carbon; CEC, cation exchange capacity; BD, bulk density; Kao, kaolinite; Ill, illite; Ver, vermiculite; 1.4 nm = 1.4 nm intergrade mineral



**Table 3** Soil aggregate proportions at different sizes (%) in each sample

Sampling sites	Soil horizon/depth (cm)	Proportion of aggregates at different sizes (%)				
		10–15 mm	5–10 mm	2–5 mm	0.25–2 mm	<0.25 mm
S1	A/0~75	28.44	24.25	22.59	20.05	4.67
	Bt/75~100	24.69	30.20	30.12	13.10	1.89
	C/80~	23.57	34.56	26.57	13.25	2.05
S2	A/0~90	23.03	30.98	25.38	18.13	2.47
	Bt/90~130	22.28	36.10	26.37	13.52	1.73
	C/130~	20.30	40.09	27.44	10.90	1.27
S3	A/0~30	36.49	28.68	19.68	12.06	3.09
	Bt/30~50	28.67	33.17	23.06	12.84	2.26
	C/50~	21.15	37.98	26.63	12.14	2.10
S4	A/0~30	16.67	31.28	26.47	20.48	5.10
	Bt/30~100	23.78	28.66	25.43	19.31	2.82
	C/100~	29.52	39.92	22.16	7.58	0.81
S5	A/0~30	14.74	29.20	25.61	24.99	5.46
	Bt/30~150	15.79	32.08	26.04	21.44	4.66
	C/150~	22.75	35.50	25.00	14.80	1.95

### 3.2 Profile distribution of sesquioxides in bulk soils and aggregates at different sizes

$Fe_d$  contents were much higher than  $Al_d$  contents in all soil samples. For bulk soils, relatively uniform distribution of soil  $Fe_d$  was observed in the soil profiles ( $p > 0.05$ ). Similar results were observed for  $Fe_d$  contents in the 0.25–2 and <0.25 mm aggregates. For 2–5, 5–10, and 10–15 mm aggregates,  $Fe_d$  contents increased with soil profile depth. Five to approximately 10 and 10–15 mm aggregates contained highest  $Fe_d$  contents ( $p < 0.05$ ).  $Al_d$  content in C horizon was much higher than in A and Bt horizons ( $p < 0.05$ ), but no significant difference was found between A and Bt horizons. There were no significant differences among the  $Al_d$  contents of the aggregates at different sizes.

Soil  $Fe_o$  and  $Al_o$  contents ranged from 2.10 to 4.47 g kg<sup>-1</sup> and from 2.33 to 5.38 g kg<sup>-1</sup>, presenting higher CVs of 41.96–53.09% and 43.15–64.82% respectively (Fig. 3). Contrary to the  $Fe_d$  and  $Al_d$  contents, the  $Fe_o$  and  $Al_o$  contents displayed an overall declined trend with soil profile depth ( $p < 0.05$ ). For A horizon, highest  $Fe_o$  contents were observed in 0.25–2 and 2–5 mm aggregates, and lowest  $Fe_o$  contents were found in <0.25 mm aggregates ( $p < 0.05$ ). However, highest  $Al_o$  content was observed in 5–10 mm aggregates ( $p < 0.05$ ). For Bt and C horizons, there were no significant differences among the  $Fe_o$  and  $Al_o$  contents of the aggregates at different sizes.

Except S5, the  $Fe_o/Fe_d$  ratios of all bulk soils decreased as the soil profile depth increased (Fig. 4). The  $Al_o/Al_d$  ratios for most bulk soils presented a similar downward trend with the  $Fe_o/Fe_d$  ratios. For <0.25 mm aggregates, the  $Fe_o/Fe_d$  ratios declined gradually with depth in the soil profile except S2

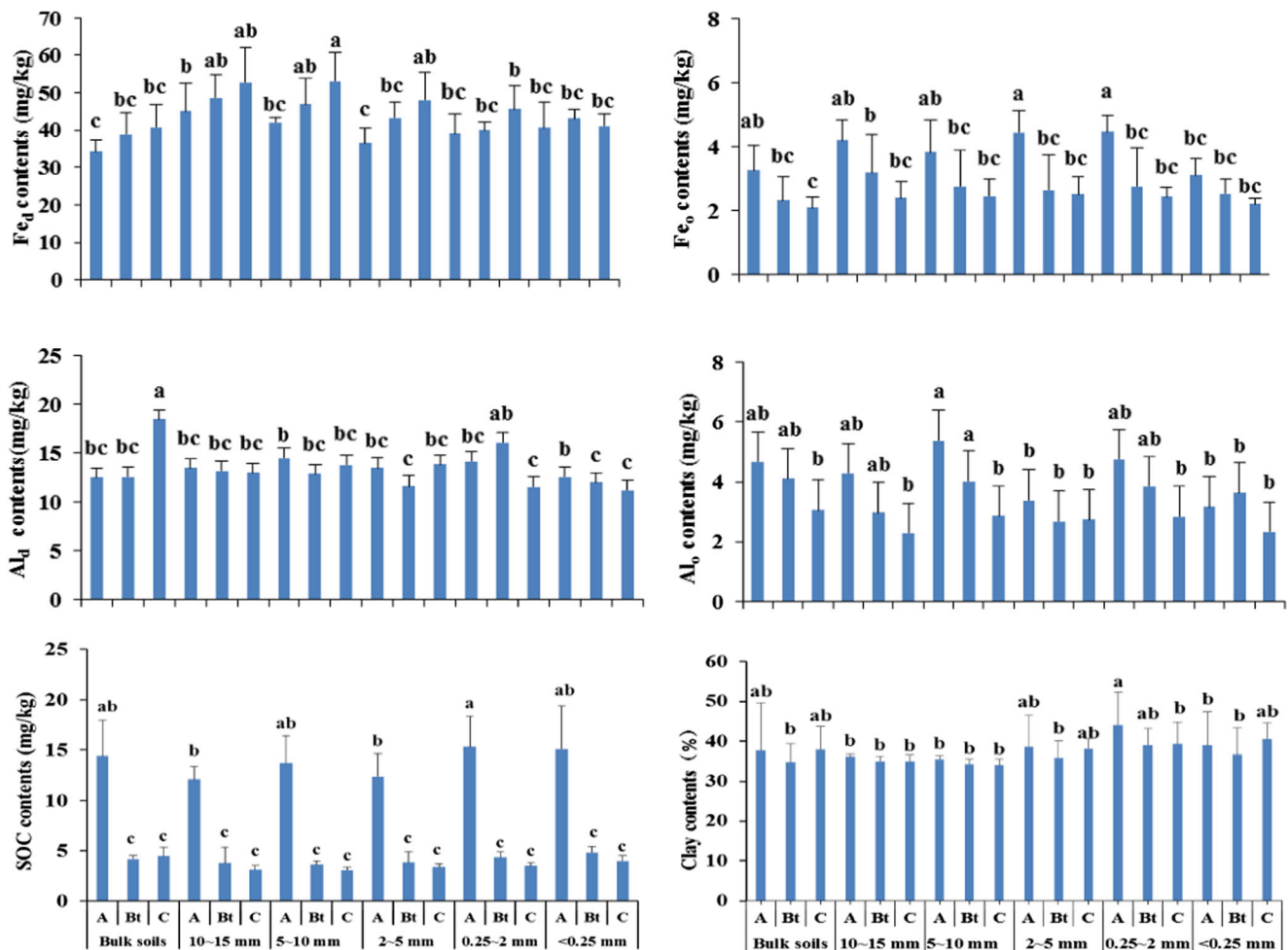
(Fig. 4). Except S5, the  $Al_o/Al_d$  ratios for <0.25 mm aggregates showed a similar variation trend. For 0.25–2 mm aggregates, the  $Fe_o/Fe_d$  ratios for S1–S4 and the  $Al_o/Al_d$  ratios for all five samples declined with the increase of soil depth. For 2–5 mm aggregates, the  $Fe_o/Fe_d$  ratios for S1, S3, S4, and the  $Al_o/Al_d$  ratios for all five samples decreased with soil depth. For 5–10 mm aggregates, the  $Fe_o/Fe_d$  and  $Al_o/Al_d$  ratios of S1–S4 declined with soil profile depth. For 10–15 mm aggregates, the  $Fe_o/Fe_d$  and  $Al_o/Al_d$  ratios for most samples declined with soil profile depth.

### 3.3 Profile distribution of SOC and clay contents in bulk soils and aggregates at different sizes

SOC contents in the bulk soils and aggregates at different sizes varied from 3.09 ± 0.43 to 15.31 ± 3.00 g kg<sup>-1</sup> (Fig. 3). SOC contents in A horizon was much higher than in Bt and C horizons ( $p < 0.05$ ), but no significant difference was found between Bt and C horizons. For A horizon, SOC contents declined with the increase of aggregate sizes. However, no significant differences were observed among the SOC contents of the aggregates at different sizes for Bt and C horizons. Clay contents in the bulk soils and aggregates at different sizes varied from 34.02% ± 1.54% to 44.04% ± 8.20% (Fig. 3). Highest clay contents were observed for 0.25–2 mm aggregates in A horizon.

### 3.4 Stability for soil aggregates at different sizes

Profile variations of the mean weight diameters (MWDs) for soil aggregates at different sizes have been listed in Fig. 5. The



**Fig. 3** Profile variations of iron and aluminum oxides ( $Fe_d$  and  $Al_d$  indicate free Fe and Al oxides;  $Fe_o$  and  $Al_o$  indicate amorphous Fe and Al oxides), SOC, and clay contents for bulk soils and their aggregates at different sizes. Different lowercase letters indicate the significant difference at  $p < 0.05$ ,  $n = 5$

MWDs of 10–15 mm aggregates varied from 1.44 to 5.39 mm with a CV of 33%. MWD values of 10–15 mm aggregates for all soil samples (except S3) decreased with soil depth. For S3, the MWD value of 10–15 mm aggregates decreased in the sequence: Bt > A > C. For 5–10 mm aggregates, the MWDs ranged from 1.14 to 3.96 mm with the CV of 37%. Except S5, MWD values of 5–10 mm aggregates for all soil samples declined with soil depth. For 2–5 mm aggregates, the range of MWDs was 0.68–2.14 mm with a CV of 40%. MWD values of 5–10 mm aggregates for S1, S2, and S4 tended to decline with the soil depth. For S3 and S5, the sequence of MWD values for 5–10 mm aggregates in the soil profile depth was Bt > A > C. MWDs for 0.25–2 mm aggregates ranged from 0.25 mm (S5-Bt) to 0.67 mm (S4-A) with a CV of 25%. MWD values of 0.25–2 mm aggregates for all soil samples declined with soil depth. MWDs for <0.25 mm aggregates varied from 0.08 mm (S3-Bt) to 0.17 mm (for S4-Bt) with a CV of 20%. The profile distribution of MWDs for <0.25 mm aggregates displayed a distinct trend different from that of other particle-size aggregates (10–15 mm, 5–10 mm, 2–5 mm, and 0.25–2 mm). For S1 and S4, MWDs for <

0.25 mm aggregates showed a declined trend in the order of Bt > A > C, while the order was C > A > Bt for S3 and was C > Bt > A for S5.

### 3.5 Identifying determinant of MWDs of aggregates at different sizes

Stepwise multiple-regression results showed that the determinants for <0.25 mm aggregate stability were  $Fe_o$  and clay (Table S1, Electronic Supplementary Material (ESM)), which were positively correlated ( $p < 0.01$ , Table S2, ESM). For 0.25–2 mm aggregates, the factors impacting the stability were  $Al_o$  and SOC (Table S3, ESM). Moreover, positive correlation existed between these two factors ( $p = 0.21$ , Table S4, ESM). The factors impacting the stability of 2–5 mm aggregates were SOC and clay (Table S5, ESM). In addition, they were positively correlated ( $p = 0.16$ , Table S6, ESM). For 5–10 mm aggregates, the determinants affecting the aggregate stability were  $Fe_o$ , SOC, and clay ( $R^2 = 0.84$ ,  $n = 15$ ,  $p = 1.8E-4$ , Table S7, ESM). Moreover,  $Fe_o$ , SOC, and clay were positively correlated with each other (Table S8, ESM), but

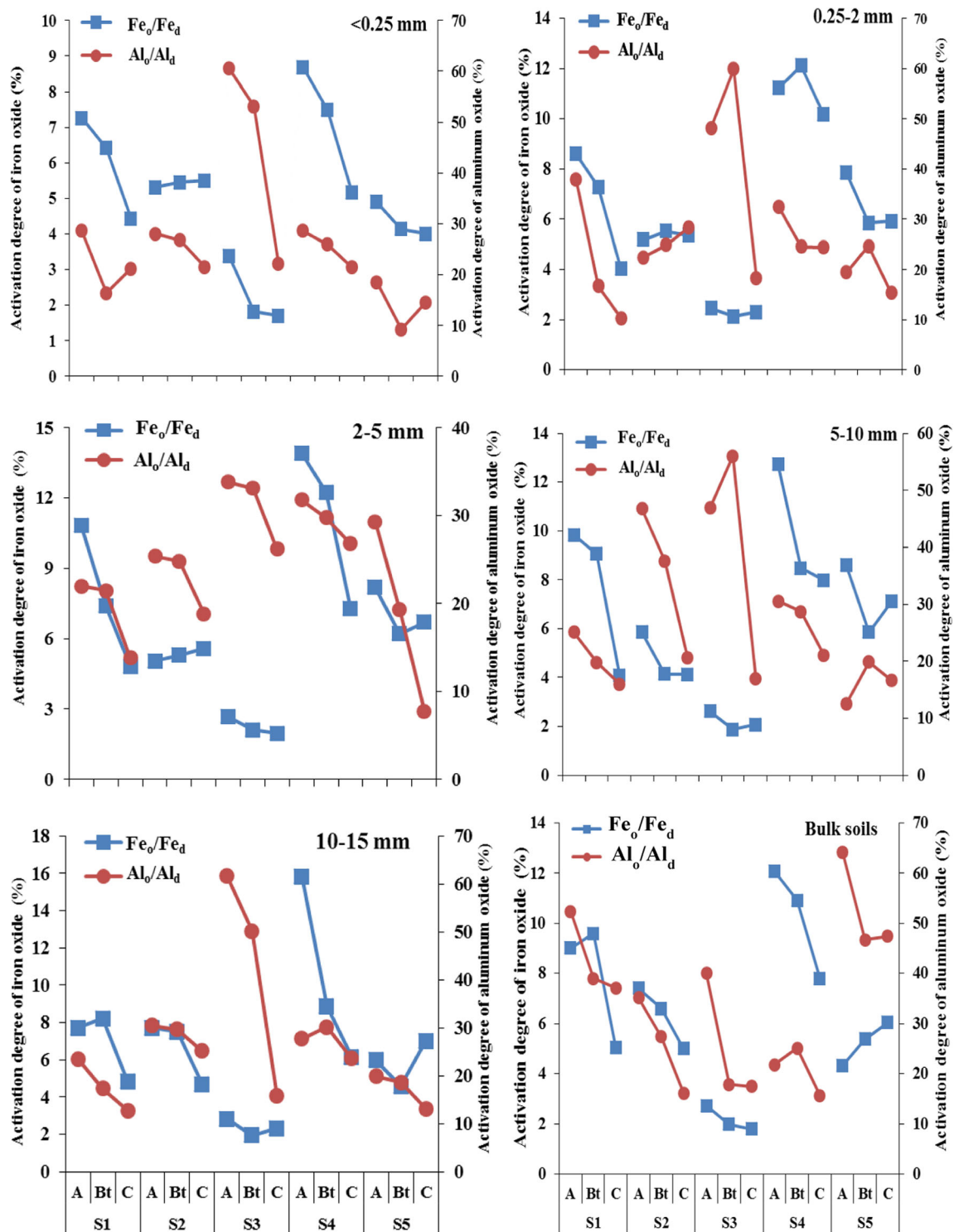


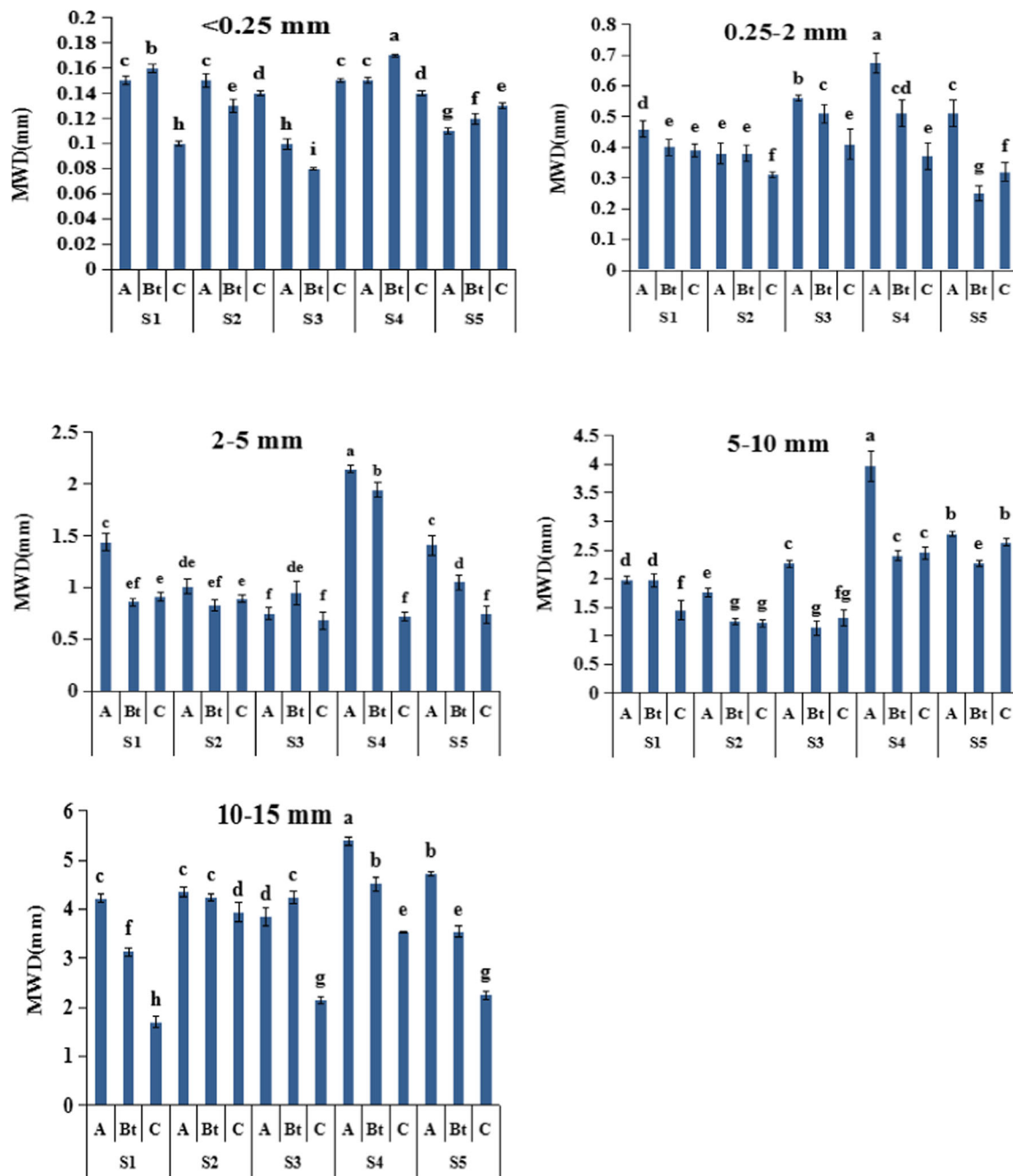
Fig. 4 Spatial and profile variations of activation degree ( $Fe_o/Fe_d$ ,  $Al_o/Al_d$ ) for bulk soils and their aggregates with different sizes

only  $Fe_o$  was significantly related to clay ( $p < 0.05$ ). The determinants impacting the 10–15 mm aggregate stability were  $Fe_o$  and SOC ( $R^2 = 0.52$ ,  $n = 15$ ,  $p = 0.013$ , Table S9, ESM).  $Fe_o$  were negatively correlated with SOC ( $p = 0.24$ , Table S10, ESM).

### 3.6 Path analysis

For <0.25 mm aggregates, the direct contribution of each factor ( $Fe_o$  and clay) and their indirect contributions were positive (Fig. 6). Both the direct and indirect contributions



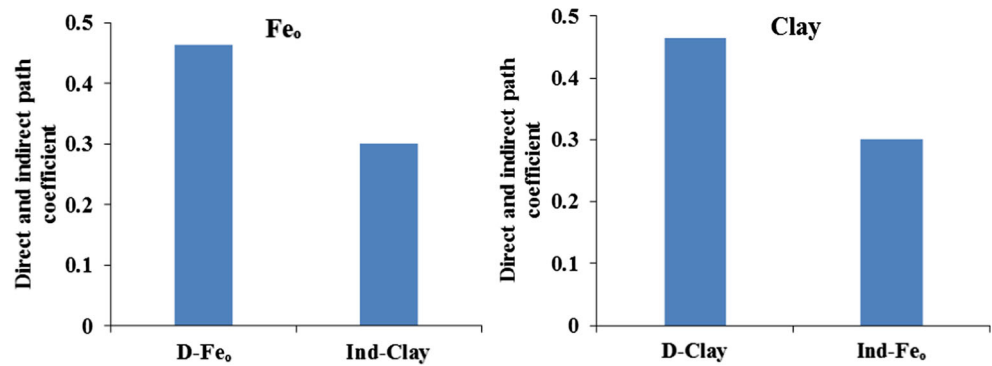


**Fig. 5** Spatial and profile variations of the mean weight diameters (MWD) for the aggregates with different sizes. Different lowercase letters indicate the significant difference at  $p < 0.05$ ,  $n = 5$

of  $Fe_0$  were equivalent to those of clay. The effect of each factor mainly came from its direct contribution. The direct and indirect contributions of  $Al_0$  and SOC to MWDs of 0.25–2 mm were positive (Fig. 7). For 2–5 mm aggregates, the direct and indirect contributions of each factor (SOC and clay) were also positive (Fig. 8). The direct contribution of each factor was higher than its indirect effect. The effect of SOC was higher than that of  $Al_0$ . The direct and indirect contributions of  $Fe_0$ , SOC, and clay to MWDs of 5–10 mm were positive (Fig. 9). The direct contribution of SOC was

higher than those of  $Fe_0$  and clay, so was the total effect. The main effect of  $Fe_0$  and SOC was attributed to its direct impact, while the main effect of clay arose via indirect contributions through  $Fe_0$  and SOC. The direct and indirect contributions of each factor to MWDs of 10–15 mm aggregates are shown in Fig. 10. D- $Fe_0$  indicates the direct contribution of  $Fe_0$  to MWDs, and Ind-SOC represents the indirect contribution of  $Fe_0$  through SOC. From Fig. 6, both the direct contributions of  $Fe_0$  and SOC to MWDs of 10–15 mm were positive, while the indirect contributions of  $Fe_0$  through SOC and

**Fig. 6** Direct and indirect contributions of factors to the stability of < 0.25 mm aggregates. D-Fe<sub>o</sub> and D-clay represent the direct contribution of Fe<sub>o</sub> and clay, respectively; Ind-clay and Ind-Fe<sub>o</sub> represent the indirect contribution of Fe<sub>o</sub> through clay and the indirect contribution of clay through Fe<sub>o</sub>, respectively



SOC through Fe<sub>o</sub> were negative. The effect of Fe<sub>o</sub> was lower than that of SOC, but the direct contributions of Fe<sub>o</sub> and SOC were higher than their indirect contributions.

## 4 Discussion

### 4.1 Size distribution of related soil properties associated with aggregates

SOC associated with both macro-aggregates and micro-aggregates decreased with depth (Fig. 3). Similar trends were also reported by several other authors, including Shrestha et al. (2007), Haile et al. (2008), and Gelaw et al. (2015). Gelaw et al. (2015) showed that SOC contents in 2.00–4.75 mm, 1.00–2.00 mm, 0.50–1.00 mm, and 0.25–0.50 mm aggregates declined with depth under different land uses in Tigray, northern Ethiopia. Decrease of SOC contents with soil depth is mainly due to the higher input of root exudates and plant residues in the upper layer of soil (García-Orenes et al. 2010). We demonstrated that micro-aggregates contained higher SOC concentrations than macro-aggregates in A horizon, while no significant differences were found in Bt and C horizons. The higher SOC concentration in micro-aggregates in our study indicates its high potential to stabilize SOC. The results obtained by us were different with several other experimental investigations. Qiu et al. (2015) reported that the

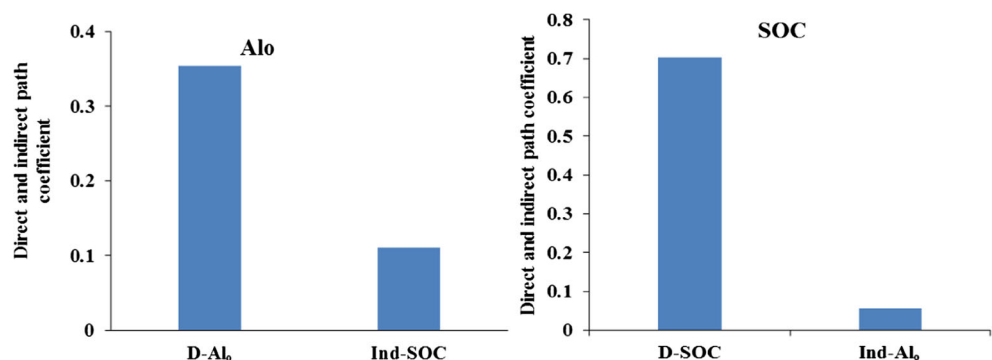
accumulation of organic C in soils after afforestation on abandoned farmland was mainly due to the accumulation of OC in macro-aggregates. Similar results were reported by Shrestha et al. (2007) and Gelaw et al. (2015). An explanation for the elevated SOC concentrations in macro-aggregates reported in these studies is that larger aggregates are composed of small particles plus organic binding agents (Elliott 1986), which is supported by the theory of hierarchical aggregation.

The Fe<sub>o</sub> and Al<sub>o</sub> contents in soil and aggregates decreased with soil depth, while the Fe<sub>d</sub> and Al<sub>d</sub> contents displayed an opposite trend (Fig. 3). Moreover, higher Fe<sub>o</sub>/Fe<sub>d</sub> and Al<sub>o</sub>/Al<sub>d</sub> ratios were observed in the surface soil horizon, which contained higher storage of organic C. The results suggested that soil organic C might have inhibitory effects on the crystallization of Fe<sub>o</sub> and Al<sub>o</sub> (Chi et al. 2016). Our results demonstrated a significant accumulation of Fe<sub>d</sub>, Fe<sub>o</sub>, and Al<sub>o</sub> in macro-aggregates. The results indicated that sesquioxides could play an effective role in binding micro-aggregates to form larger aggregates.

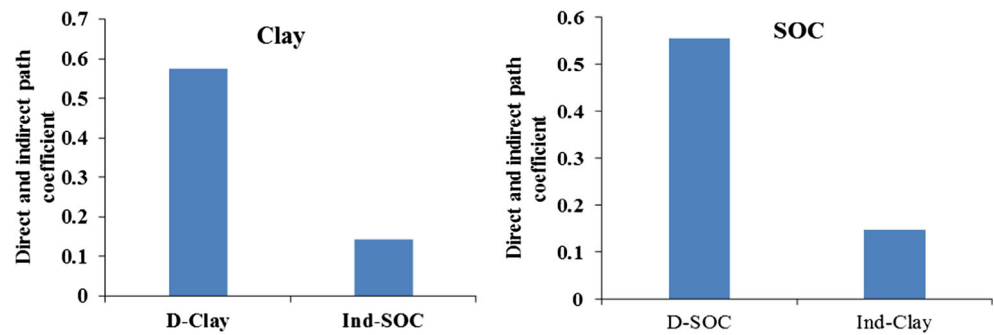
### 4.2 Related soil properties contributing to stability of soil macro- and micro-aggregates

In tropical and subtropical soils (e.g., ultisols), the sesquioxides and SOC coexist and act as the main inorganic and organic binding agents of soil aggregation, respectively (Six et al. 2004; Bronick and Lal 2005). Many scholars have studied

**Fig. 7** Direct and indirect contributions of factors to the stability of 0.25–2 mm aggregates. D-Al<sub>o</sub> and D-SOC represent the direct contribution of Al<sub>o</sub> and SOC, respectively; Ind-SOC and Ind-Al<sub>o</sub> represent the indirect contribution of Al<sub>o</sub> through SOC and the indirect contribution of SOC through Al<sub>o</sub>, respectively



**Fig. 8** Direct and indirect contributions of factors to the stability of 2–5 mm aggregates. D-clay and D-SOC represent the direct contribution of clay and SOC, respectively; Ind-SOC and Ind-clay represent the indirect contribution of clay through SOC and the indirect contribution of SOC through clay, respectively



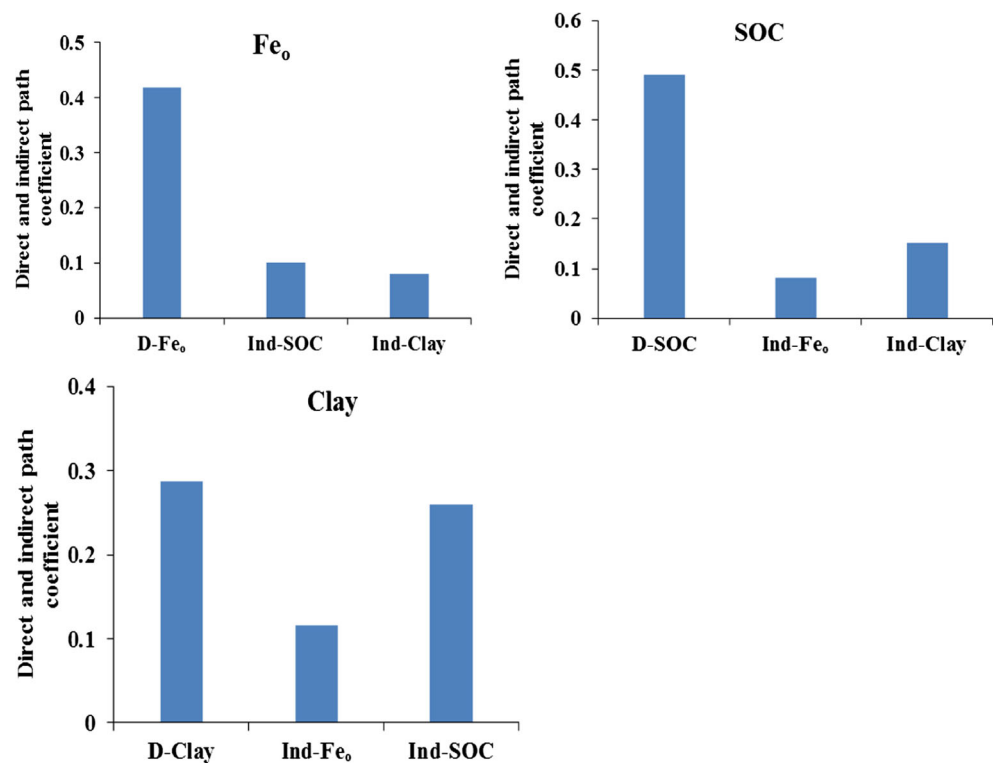
cementation process of the sesquioxides and SOC in depth (Deneff et al. 2002; Kögel-Knabner et al. 2008; Pronk et al. 2012; Martins et al. 2013). However, it is still very difficult for us to assess the respective contributions of the two binding agents to soil aggregates with different sizes. Assessing the changes of the MWDs after the soils were extracted by DCB, oxalate, and  $H_2O_2$ , respectively, Peng et al. (2015) concluded that the sesquioxides may act as the primary binding agents of the micro-aggregates (<0.25 mm) in an oxide-rich ultisol, but the SOC seemed to play a major role in stabilizing 0.25–2 mm aggregates. However, they still have not quantified the respective contributions of sesquioxides and SOC to the stability of aggregates at different sizes.

Our results suggested that the soil properties such as Fe/Al oxides and SOC differed significantly in terms of fraction size, which was consistent with the hierarchical theory of soil aggregates (Tisdall and Oades 1982; Puget et al. 2000; Six et al.

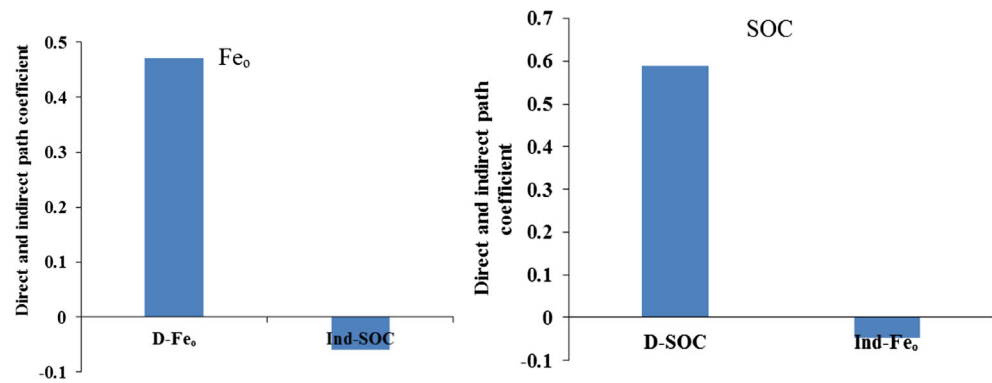
2000; John et al. 2005). Analyzing the indirect and direct contributions of determinants to the stability of aggregates at different sizes using step multiple-regression and path analysis, we found sesquioxides especially  $Fe_o$  played a most important role in stabilizing the <0.25 mm aggregates (Fig. 10), while SOC acted as the primary binding agents of 0.25–2, 5–10, and 10–15 mm aggregates (Figs 6, 7, and 9). In addition, amorphous oxides, especially amorphous iron oxides, made a greater contribution to aggregate stability according to their correlations with MWDs. Amorphous oxides tend to have a much larger and more reactive surface area than crystalline oxides, so they played a bigger role in stabilizing aggregate (Duiker et al. 2003).

According to Six et al. (2004), oxides in the oxide-rich soils can act as binding agents of soil aggregation in three ways: (1) formation of organo-mineral complexes through adsorbing organic materials on oxide surfaces, (2)

**Fig. 9** Direct and indirect contributions of factors to the stability of 5–10 mm aggregates. D- $Fe_o$ , D-SOC, and D-clay represent the direct contribution of  $Fe_o$ , SOC, and clay, respectively. Ind-SOC and Ind-clay represent the indirect contribution of  $Fe_o$  through SOC and clay, and so on



**Fig. 10** Direct and indirect contributions of factors to the stability of 10–15 mm aggregates. D-Fe<sub>o</sub> and D-SOC represent the direct contribution of Fe<sub>o</sub> and SOC, respectively. Ind-SOC and Ind-Fe<sub>o</sub> represent the indirect contribution of Fe<sub>o</sub> through SOC and the indirect contribution of SOC through Fe<sub>o</sub>, respectively



electrostatic binding between positively charged oxides and negatively charged clay minerals, and (3) a coat of oxides on the surface of minerals. In our study, Fe<sub>o</sub> and clay were the determinants playing profoundly important roles in the stability of <0.25 mm aggregates. The indirect and direct contributions of these two factors to the MWD of <0.25 mm aggregates were positive. Moreover, the proportions of direct and indirect contributions for each factor to the total effects were 61% and 39%, respectively. The results suggested that the formation of <0.25 mm aggregates involved the latter two ways. For 0.25–2 mm aggregates, SOC and Al<sub>o</sub> contributed positively to the MWD through direct and indirect ways. Organo-mineral complexes formed between SOC and Al<sub>o</sub> played a certain role in stability of 0.25–2 mm aggregates. Organo-mineral associations were also found to play an effective role in stabilizing the 2–5 mm and 5–10 mm aggregates. Our results are similar to Amézketa (1999) and Harbour et al. (2007), who suggested that the interactions between the sesquioxides and SOC may play a more important role in stabilizing soil aggregation in many cases. However, our results were different from Peng et al. (2015) and Wu et al. (2016). Wu et al. (2016) suggested that complex oxides played a more important role in smaller water-stable aggregates. Peng et al. (2015) indicated that aggregates were predominantly formed by either SOC or Fe/Al oxides, but not by both simultaneously. For 5–10 mm aggregates, their formation may involve all three ways. Moreover, the first process may be more effective than the second way in stabilizing 5–10 mm aggregates. For 10–15 mm aggregates, the negative correlation between SOC and Fe<sub>o</sub> indicated the effect of one specific factor could be more significant than their interactions.

## 5 Conclusions

To gain insights into the contributions of sesquioxides and SOC to stability of aggregates at different sizes in ultisols,

the correlations of soil aggregate stability with sesquioxides and SOC at different sizes have been investigated. The respective contributions of sesquioxides and SOC to aggregate stability were further estimated using path analysis. In summary, we have shown that abiotic factors (Fe/Al oxides and clay) can be more important for determining the formation of <0.25 mm aggregates, whereas both abiotic and biotic factors (SOC) play an effective role in stabilizing larger aggregates (0.25–2, 2–5, 5–10, and 10–15 mm). The organo-mineral complexes performed a certain role in aggregate stabilization for the ultisols tested. Further studies on abiogenic and biogenic processes involved in stabilizing the aggregates at different sizes by means of some microscopic instruments are required to improve our understanding of soil aggregation stabilization. Furthermore, their respective contribution ratios should be assessed with high precision.

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