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## **Texture and sesquioxide effects on water-stable aggregates and organic matter in some tropical soils**

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### **Short title**

Aggregation and organic matter in kaolinitic soils

### **Abstract**

Many tropical soils include sesquioxides, which influence the stability of soil organic matter (OM) and aggregation to an extent that is not fully characterized. The present study was carried out on a range of 18 topsoil samples (0-10 cm) from low-activity clay (LAC) soils from sub-Saharan Africa and Brazil, and aimed: (i) at characterizing the size distributions of water-stable aggregates and organic constituents, (ii) at studying how these distributions were affected by texture and sesquioxides, and (iii) how they interacted.

The distributions of stable aggregates were generally dominated by macroaggregates (> 200 µm), and those of organic constituents by fine OM (< 20 µm). Aggregation was not clearly affected by soil texture, while total soil carbon (Ct) and the amount of carbon (C) as fine OM increased with soil content in clay plus fine silts (< 20 µm). Stable macroaggregation correlated with Ct and with C amount as fine OM, but each of them correlated more closely with citrate-bicarbonate-dithionite-extractable aluminium (Al), which was not expected.

Stable macroaggregation also correlated with C amounts as coarse- and medium-sized OM (> 200 and 20-200  $\mu\text{m}$ , respectively), but each of them correlated more closely with oxalate-extracted Al. These results suggested that for the LAC tropical soils under study OM and aggregate stability depended closely on Al-containing sesquioxides, on Al-substituted crystalline hematite and goethite especially. These sesquioxides also seemed to play a dominant role in the relations between aggregation and OM. As far as soils rich in sesquioxides are concerned, this confirmed that OM is not the main aggregating agent, and suggested that physical protection within aggregates is not necessarily the main mechanism for OM stabilization. However, as soil sesquioxide content cannot be managed easily, the effect of land use on soil OM and aggregation was determinant at the local scale: indeed, for a given location, stable macroaggregation, Ct and C amount as fine OM generally decreased with land use intensification (i.e. cultivation, tillage, reduced surface cover).

## **Keywords**

Kaolinitic soils; Aggregation; Organic matter; Texture; Sesquioxides; Aluminium

## **1. Introduction**

Soil organic matter (OM) and aggregation are important determinants of soil fertility and productivity, and are key factors in the global carbon (C) cycle. Indeed, soil OM is a source of nutrients, promotes structure and water retention, and is a substrate for soil heterotrophs (Baldock and Skjemstad, 2000). Soil aggregation has a major influence on root development, on water and C cycling, and on soil resistance to erosion (Kay, 1998). Moreover, soil OM and aggregation are closely linked one to another: OM is considered an important aggregate binding agent, on the one hand, and may be physically protected against decomposition within stable aggregates, on the other hand (Feller and Beare, 1997; Angers and Chenu, 1998).

However, the stability of soil OM and aggregation as well as relationships between OM and aggregation are affected by the presence of sesquioxides. Indeed, the aggregating role of OM could be less effective in soils that include large amounts of such inorganic constituents (Tisdall and Oades, 1982; Six et al., 2002). Due to the role of sesquioxides in the stabilization of OM (Dalal and Bridge, 1996; Guggenberger and Haider, 2002), the importance of aggregation in the stability of OM should also be re-examined for soils that are rich in sesquioxides. This is the case for low-activity clay (LAC) soils, which include clay minerals dominated by 1:1 phyllosilicates, as kaolinite, associated with crystallized and

poorly or non-crystallized iron (Fe) and aluminium (Al) oxides and hydroxides. They cover about 60-70% of tropical areas (Segalen, 1994), where they are mainly represented by Alfisols, Oxisols and Ultisols according to the US Soil Taxonomy (Soil Survey Staff, 1999) or by Acrisols, Ferralsols and Nitisols according to the FAO soil classification (FAO-ISRIC-ISSS, 1998). Though they are widely distributed, LAC tropical soils have received much less attention than soils from temperate areas, and knowledge on OM and aggregation in LAC tropical soils still remains fragmentary.

As far as LAC tropical soils are concerned, more information is thus useful to verify the importance of sesquioxides in soil OM and aggregate stability, and complementarily, to characterize the interrelations between OM and aggregation. The influence of texture on soil OM is more clearly established. Several studies, some of them regarding LAC tropical soils, showed that soil C content increases with clay content (Feller and Beare, 1997). However, the role of texture in stable aggregation remains questionable (Le Bissonnais, 1996; Amézqueta, 1999), and requires additional investigations.

Soil OM may first be considered through the determination of total soil C content (Ct), as far as soils do not include carbonates. It can also be characterized by chemical fractionations, but the importance of the obtained fractions (e.g. fulvic and humic acids) towards major soil processes such as aggregation and OM mineralization is not yet well established (Feller and Beare, 1997; Baldock, 2002). Therefore, increasing attention has progressively been paid to procedures that involve physical fractionations of soil OM, e.g. particle size fractionations. Indeed, the characterization of OM associated with soil primary particles (sands, silts, clay) has proved to be a relevant approach to separate fractions that have different morphologies, composition, and dynamics (Christensen, 1992; Feller and Beare, 1997). The characterization of water-stable aggregate distribution, which may be achieved using simple laboratory procedures, also provides useful information on soil quality and behaviour (Tisdall and Oades, 1982; Amézqueta, 1999; Barthès et al., 2000).

The objectives of the present work were (i) to characterize the size distributions of stable aggregates and organic constituents associated with primary particles, (ii) to study how these distributions were affected by soil texture and Al- and Fe-sesquioxides, and (iii) how these distributions were interrelated, for a range of topsoil layers (0-10 cm) from LAC soils from sub-Saharan Africa and Brazil.

## 2. Material and methods

### 2.1. Sites and sampling

Composite soil samples were collected at 0-10 cm depth in 18 plots: five in southern Congo-Brazzaville (two sites in the same region), four in southern Benin (one site), and nine in Brazil, including three in Goiás (one site), three in São Paulo (three sites), and three in Paraná (one site). Table 1 provides information on location, climate, soil type, parent material, texture, clay mineralogy, and land use. The climate is tropical (Congo, Benin, Goiás) or subtropical (Paraná, São Paulo), ranges in mean annual rainfall and temperature being 1100-1600 mm and 23-27°C, respectively. Soils are mainly Oxisols and Ultisols, with sandy to clayey texture, and clay mineralogy is dominated by kaolinite. Further information on sites and soils was given by Kouakoua et al. (1999) for Congo, Goiás and Paraná, Barthès et al. (2000) for Benin, and De Luca (2002) for São Paulo. Samples were generally collected using 0.25- to 0.5-L cylinders, air-dried at room temperature, then gently crushed using a mortar and pestle and sieved through a 2-mm mesh. Aliquots were finely ground to pass a 0.2-mm mesh, also using a mortar and pestle.

### 2.2. Aggregate fractionation

The distribution of water-stable aggregates was determined on 2-mm sieved air-dried soil samples using a test adapted from Kemper and Rosenau (1986). Four grams of soil were rapidly immersed into deionised water for 30 min then wet-sieved during 6 min, using a motor-driven holder lowering and raising 200- $\mu\text{m}$  sieves into containers of water. On the one hand, the fraction  $> 200 \mu\text{m}$  was dried at 105°C then weighed. It was successively sieved into dispersive NaOH solution (0.05 M), dried at 105°C, and weighed to determine coarse sand content. Stable macroaggregate ( $> 200 \mu\text{m}$ ) content was calculated as the difference between fraction  $> 200 \mu\text{m}$  and coarse sands. On the other hand, the fraction  $< 200 \mu\text{m}$  was transferred to a glass cylinder, which was shaken by hand (30 end-over-end tumbings) and left to stand during the correct settling time for the 0-20  $\mu\text{m}$  fraction (pipette method). An aliquot of the fraction  $< 20 \mu\text{m}$  was then siphoned from the upper 10 cm of the suspension, dried at 105°C, weighed, and referred to the cylinder volume to determine microaggregate ( $< 20 \mu\text{m}$ ) content. Mesoaggregate (20-200  $\mu\text{m}$ ) content was calculated by difference between total soil and other fractions. Compared to the macroaggregate fraction ( $> 200 \mu\text{m}$ ), which included genuine aggregates only due to coarse sand-correction, the meso- and microaggregate fractions included aggregates as well as primary particles of the same size. Measurements were made

on four replicates per sample. It should be noticed that the aggregate fractions were not extracted thus were not available for further analyses.

### *2.3. Particle size fractionation of organic matter*

The particle size fractionation of OM requires maximum soil dispersion with minimum particle alteration. It was carried out on 2-mm sieved air-dried soil samples of 40 g (sandy soils) or 20 g (other soils), in duplicate, following Gavinelli et al. (1995). Each sample was pre-soaked overnight at 4°C in deionised water with 2.5 g L<sup>-1</sup> sodium metaphosphate. It was then shaken with agate balls in a rotary shaker for 2 h (sandy soils) or 6 h (others). Indeed, a long shaking duration is necessary to achieve maximum dispersion in clayey samples, but in coarse textured samples it may lead to an alteration of coarse organic particles by sands (Feller et al., 1991). Next the soil suspension was wet-sieved through 200- and 50- $\mu\text{m}$  sieves, successively. The fractions remaining in the sieves were washed with water and the washings added to the 0-50  $\mu\text{m}$  suspension. The suspension < 50  $\mu\text{m}$  was ultrasonicated for 10 min with a probe-type ultrasound generating unit, then it was sieved on a 20- $\mu\text{m}$  screen, and the residual material obtained from the sieve was washed. The three fractions > 20  $\mu\text{m}$  were dried at 60°C and weighed. The suspension < 20  $\mu\text{m}$  was transferred to a 1-L glass cylinder, where water was added to bring the volume to 1 L. Then the cylinder was shaken by hand (30 end-over-end tumblings) and left to stand during the correct settling time for the 0-2  $\mu\text{m}$  fraction (pipette method). The upper 30 cm of the suspension, which included particles < 2  $\mu\text{m}$  only, was siphoned off. The process (i.e. water addition, shaking, settling, and siphoning) was repeated until the supernatant was clear thus the fraction 0-2  $\mu\text{m}$  extracted completely (five cycles at least). The 0-2  $\mu\text{m}$  suspension was then flocculated with strontium chloride, centrifuged, filtered, and the pellets were washed with water, dried at 60°C and weighed. For some samples (Benin, Saõ Paulo), the supernatant from centrifugation and the washing waters from previous sieving operations were grouped, then an aliquot was collected and filtrated at 0.2  $\mu\text{m}$  for the analysis of soluble organic C. The 2-20  $\mu\text{m}$  fraction, which remained at the bottom of the sedimentation cylinder, was also dried at 60°C and weighed. All these fractions, separated from completely dispersed soil samples, were called dispersed fractions, as opposed to aggregate fractions. In the present paper, data regarding the dispersed fractions 50-200 and 20-50  $\mu\text{m}$  have been combined (20-200  $\mu\text{m}$ ), as well as those regarding the fractions 2-20 and 0-2  $\mu\text{m}$  (< 20  $\mu\text{m}$ ).

#### 2.4. Analyses of carbon, pH, and iron and aluminium sesquioxides

Carbon contents of finely ground (< 0.2 mm) soil aliquots and dispersed fractions were determined by dry combustion using a Leco CHN 600 elemental analyzer (St Joseph, MI). In the absence of carbonates, all C was assumed to be organic. Soluble organic C was analyzed using a Shimadzu TOC 5000 analyzer (Kyoto, Japan). The pH in water was determined with a soil:solution ratio of 1:2.5. On whole soil aliquots, “free” Fe ( $\text{Fe}_{\text{CBD}}$ ) and Al ( $\text{Al}_{\text{CBD}}$ ) were extracted with citrate-bicarbonate-dithionite (CBD), “amorphous” Fe ( $\text{Fe}_{\text{OX}}$ ) and Al ( $\text{Al}_{\text{OX}}$ ) with ammonium oxalate, and all were analyzed using an Unicam P 900 (Cambridge, UK) atomic absorption spectrometer (Rouiller et al., 1994).

#### 2.5. Statistical analyses

Simple and multiple linear correlation coefficients were calculated between variables, and their significance was denoted <sup>NS</sup> for  $p \geq 0.05$ , \* for  $p < 0.05$ , \*\* for  $p < 0.01$ , and \*\*\* for  $p < 0.001$ . Principal component analyses (PCAs) were also carried out on the dataset. The objective of PCA is to reduce the number of original variables in a smaller number of representative and uncorrelated factors, and to detect structure in the relationships between original variables (Lebart et al., 1997).

### 3. Results

Studied variables are presented in Table 2, and main correlations between them in Table 3.

#### 3.1. Soil texture and contents in Fe- and Al-sesquioxides

The cumulative yield of dispersed fractions ranged from 95 to 101%, and averaged 99%. Clay plus fine silts (< 20  $\mu\text{m}$ ) ranged from 8 to 87%; they correlated negatively with coarse sands (> 200  $\mu\text{m}$ ), and to a lesser extent, with coarse silts plus fine sands (20-200  $\mu\text{m}$ ). Soil content in  $\text{Fe}_{\text{CBD}}$  ranged from 8 to 99  $\text{g kg}^{-1}$ , and  $\text{Al}_{\text{CBD}}$ ,  $\text{Fe}_{\text{OX}}$  and  $\text{Al}_{\text{OX}}$  from 0.4 to 7.6  $\text{g kg}^{-1}$ . All correlated positively one with another in general, particularly  $\text{Fe}_{\text{CBD}}$  and  $\text{Fe}_{\text{OX}}$ , and  $\text{Al}_{\text{CBD}}$  and  $\text{Al}_{\text{OX}}$ . Moreover, all correlated positively with clay plus fine silts,  $\text{Al}_{\text{CBD}}$  especially. Clay plus fine silts tended to increase and coarse sands to decrease with land use

intensification (i.e. cultivation, tillage, reduced surface cover)<sup>1</sup>. In contrast, the effect of land use on Fe- and Al-sesquioxides was not clear.

### *3.2. Soil aggregate distribution; relations with texture and Fe- and Al-sesquioxides*

The cumulative yield of aggregate fractions plus coarse sands was 100% by definition, the mesoaggregate fraction being calculated by difference. Aggregate distributions were generally dominated by macroaggregates (> 200 µm): they included 42 to 82% of the mass of aggregate fractions (coarse sands being excluded), and represented a higher soil proportion than mesoaggregates (20-200 µm) in 16 out of the 18 samples. The proportion of microaggregates (< 20 µm) was very low. Macro- and mesoaggregate fractions did not correlate significantly (except on a coarse sand-free basis, due to the low proportion of microaggregates). The proportion of microaggregates did not relate clearly with other aggregate fractions.

Considering all 18 plots, the macroaggregate fraction correlated negatively with coarse sands and positively with clay plus fine silts, Al<sub>CBD</sub>, Al<sub>OX</sub> (Figure 1a), and to a lesser extent, with Fe<sub>CBD</sub> and Fe<sub>OX</sub>. It seemed that macroaggregates correlated with clay plus fine silts “indirectly” (relation A), as a consequence of their close negative correlations with coarse sands (relation B), which related negatively with clay plus fine silts (relation C). Indeed, the product of coefficient correlations assigned to relations B and C was similar to the coefficient correlation assigned to relation A. Meso- and microaggregate fractions related less closely with texture and sesquioxides.

### *3.3. Soil carbon; relations with texture and Fe- and Al-sesquioxides*

For the studied samples, Ct ranged from 5 to 43 g C kg<sup>-1</sup>. The cumulative C yield in dispersed fractions ranged from 78 to 101%, and averaged 90%. Rather low recoveries could be attributed, at least partly, to soluble C. Indeed, when it was determined, soluble C accounted for 6 to 9% of Ct (data not shown), which agreed with data reported by Christensen (1992). On average, the dispersed fractions > 200, 20-200 and < 20 µm included respectively 37, 15 and 27 g C kg<sup>-1</sup> fraction (C concentrations). This corresponded to 1.5, 0.8 and 1.9 times

<sup>1</sup> The effects of land use on many soil properties are fairly well established (cf. end of section 4.7) and the present paper did not aim at discussing them extensively. However, increasing topsoil clay content upon land use intensification has not been often reported. It might be due to erosion, which generally increases with land use intensification and leads to the outcropping of sub-superficial layers that are more clayey than superficial ones in many LAC tropical soils.

Ct (C enrichments), represented 1.6, 3.1 and 13.2 g C kg<sup>-1</sup> soil (C amounts), and accounted for 8, 17 and 65% of Ct, respectively. Size distributions of organic constituents were thus dominated by fine OM (46 to 80% of Ct). Considering all 18 soil samples, Ct correlated positively with C amount in every dispersed fraction, especially with that of fraction < 20 µm. Carbon amounts in the dispersed fractions 20-200 and > 200 µm correlated closely one with another, but correlated less closely with that of fraction < 20 µm.

Considering all 18 soil samples, texture related clearly with the characteristics of several C pools (Figure 2). Clay plus fine silts correlated positively with Ct and C amount in the dispersed fraction < 20 µm (Figures 1b and 1c), with C concentration and enrichment in the dispersed fraction > 200 µm, and negatively with C enrichment in the dispersed fraction < 20 µm. Soil content in Al<sub>CBD</sub>, and in Al<sub>OX</sub> to a lesser extent, correlated closely with Ct and C amount in the dispersed fraction < 20 µm (Figures 1b and 1c), and less closely, with C amounts in the coarser fractions. By contrast, Fe<sub>CBD</sub> and Fe<sub>OX</sub> did not relate clearly with Ct and fraction C amounts. Multiple linear correlations between C amount in a given fraction, on the one hand, and two or more variables regarding texture and/or sesquioxides, on the other hand, were not closer than corresponding simple linear correlations or were not significant..

#### *3.4. Possible determinants of soil aggregation and organic matter*

Considering all 18 soil samples, soil content in stable macroaggregates correlated positively with Ct and with C amount in the dispersed fraction < 20 µm (Figure 3), but each of them correlated more closely with Al<sub>CBD</sub> (Figure 1). To a lesser extent, macroaggregate content correlated with C amount in the dispersed fractions > 200 and 20-200 µm, but each of them correlated more closely with Al<sub>OX</sub>. Multiple linear correlations between macroaggregate content, on the one hand, and Al-extracts and fraction C amounts, on the other hand, were not closer than corresponding simple correlations. Relationships between meso- or microaggregate fractions and Ct or fraction C amounts were not significant.

Data thus suggested that Al<sub>CBD</sub>, and Al<sub>OX</sub> to a lesser extent, were major determinants of soil aggregation and OM content in the studied soils. However, when considering each location separately, Al<sub>CBD</sub> and Al<sub>OX</sub> varied generally within narrow ranges, and the above-mentioned correlations were not significant in general. By contrast, the effect of land use became important: land use intensification (i.e. cultivation, tillage, reduced surface cover) generally caused a decrease in the proportion of macroaggregates, in C concentration and amount in every dispersed fraction, thus in Ct. These effects have already been described for

several of the studied sites (Kouakoua et al., 1999; Barthès et al., 2000), and will not be detailed here.

### 3.5. Principal component analysis (PCA)

Three main PCAs were carried out, with active variables that contributed to factor definition, and supplementary variables that did not. Considering supplementary variables helped limiting the over-influence of linked variables (e.g. C amounts of fractions and their sum Ct). Clay plus fine silts, coarse silts plus fine sands, Al<sub>CBD</sub> and Fe<sub>CBD</sub> were always considered active variables. For the first PCA, C amounts in dispersed fractions were also considered active, while Ct, aggregate fractions, Al<sub>OX</sub> and Fe<sub>OX</sub> were considered supplementary. For the second PCA, fraction C concentrations were considered active, and Ct, fraction C amounts and aggregate fractions supplementary. Finally, for the third PCA, macro- and mesoaggregate fractions were considered active, and Ct and fraction C amounts supplementary.

On the whole, the three PCAs showed similar structure in the relationships between variables (the first PCA is presented in Figure 4). The first factor included 58-62% of the total variability, the second factor 20-22%, and the three first factors together 90-92%. Macroaggregate content and Al<sub>CBD</sub> were always very closely associated, and were associated with C amount < 20 µm and Ct, confirming their strong interrelations. The coordinates of these four variables on the first factor ranged from -0.77 to -0.96. Clay plus fine silts were associated with this group of variables, but to a lesser extent. Amounts of C in the dispersed fractions > 200 and 20-200 µm were associated one with another, as well as the corresponding C concentrations. When Fe<sub>OX</sub> and Al<sub>OX</sub> were included in the analysis, they were associated with Fe<sub>CBD</sub> and Al<sub>CBD</sub>, respectively.

## 4. Discussion

### 4.1. Size distribution of stable aggregates

Stable macroaggregates (> 200 µm) dominated aggregate distributions in general. A high content in stable macroaggregates has often been reported for clayey LAC tropical soils (Oades and Waters, 1991; Albrecht et al., 1998; Six et al., 2002) but less frequently for coarse-textured ones (Dalal and Bridge, 1996; Spaccini et al., 2001), where it has been considered that macroaggregation could be less stable (Albrecht et al., 1998). Thus stable aggregation was well developed in the studied soils, and this probably stands for most LAC tropical soils. Within aggregate fractions (i.e. on a coarse sand-free basis), the very close

correlation between macro- and mesoaggregates suggested an aggregate hierarchy: macroaggregate disruption released mesoaggregates mainly, and conversely, macroaggregates resulted mainly from bounding of mesoaggregates. Aggregate hierarchy has not often been reported in LAC soils. Oades and Waters (1991) reported such a hierarchy for an Alfisol and a Mollisol (dominated by 2:1 clay mineralogy), but not for a clayey Oxisol. Six et al. (2000a) also found a higher degree of aggregate hierarchy in 2:1 than in 1:1 clay-dominated soils. However, Feller et al. (1996) reported a tendency to an aggregate hierarchy for an Ultisol, an Inceptisol and an Oxisol.

#### *4.2. Size distribution of organic constituents*

In the present study, the dispersed fractions  $> 200$ , 20-200 and  $< 20$   $\mu\text{m}$  included 8, 17, and 65% of Ct in average, respectively. This was consistent with data from tropical soils reviewed by Feller and Beare (1997), which indicated that proportions of Ct in the dispersed fractions  $> 50$ , 2-50 and  $< 2$   $\mu\text{m}$  generally ranged from 10 to 30%, 20 to 40%, and 35 to 70%, respectively. The present study also showed that Ct correlated positively with C amount in every dispersed fraction (in  $\text{g C kg}^{-1}$  soil), with fine fraction C especially. Thus changes in Ct resulted from changes in every size class, but depended more closely on those in the fine fraction. Studying 21 grassland topsoils from Saskatchewan to Texas, Amelung et al. (1998) similarly reported positive correlations between Ct and C amount in every dispersed fraction, in the fine one especially, and found that the dispersed fraction  $< 20$   $\mu\text{m}$  included 58 to 99% of Ct. For a clayey Oxisol under bush savanna and pasture in Brazil, Roscoe et al. (2001) also found positive correlations between Ct and fraction C amounts. Moreover, the present study showed that C amounts in the dispersed fractions 20-200 and  $> 200$   $\mu\text{m}$  correlated closely one with another but correlated poorly with C amount in the fine fraction. Similar relationships were found between fraction C concentrations (in  $\text{g C kg}^{-1}$  fraction). This suggested that organic compartments  $> 200$  and 20-200  $\mu\text{m}$  had similar dynamics, which differed from those of organic fraction  $< 20$   $\mu\text{m}$ . Indeed, several works supported the evidence that organic constituents  $> 20$   $\mu\text{m}$  are plant debris mainly, the coarser ones being less decomposed and having a more recognizable morphology than the finer ones, whereas organic constituents  $< 20$   $\mu\text{m}$  are more humified and have a slower turnover (Feller and Beare, 1997; Baldock and Skjemstad, 2000; Guggenberger and Haider, 2002).

#### *4.3. Influence of texture on soil aggregation*

Macro- and mesoaggregate fractions correlated positively with clay plus fine silts. However, this seemed to result largely from negative correlations between all three variables and coarse sands: an increase in coarse sands caused simultaneous decreases in clay plus fine silts and aggregate fractions, and there was no evidence of direct influence of clay plus fine silts on aggregate fractions, macroaggregates especially. This contrasted with the rather general agreement that clay content positively affects aggregate stability, as reported in reviews by Le Bissonnais (1996) and Amézketa (1999). However, these authors mentioned contradictory results, especially when considering wide ranges of soils, because significant correlations between clay and aggregation have often been established within narrow ranges of soils. Dalal and Bridge (1996) did not consider that clays were important agents of aggregate stabilization in Oxisols and Ultisols, and Six et al. (2002) did not find any correlation between clay content and aggregate stability over a wide range of soils from tropical and temperate areas.

#### *4.4. Influence of texture on soil organic matter*

The present study confirmed the positive relation between Ct and clay plus fine silts, which was reported for LAC tropical soils by Feller and Beare (1997). When plotting Ct against clay plus fine silts (in %), slope coefficient was very close to that calculated by these authors (0.036 vs. 0.037). Indeed, soil texture is considered a key variable affecting Ct, as a result of electrostatic binding between negatively charged clay surfaces and organic colloids via cation bridges (Ingram and Fernandes, 2001; Paustian et al., 2003). Due to sorption of fine organic constituents to fine mineral particles, C amount in the fine fraction increases with the proportion of fine primary particles, as confirmed by Feller et al. (1996) over a range of tropical soils. As fine fraction C includes the highest proportion of Ct, Ct also increases with clay plus fine silts. Thus clayey soils contain more C, but the present study showed that C enrichment affected their coarse fraction mainly, whereas in coarse-textured soils C enrichment rather affected the fine fraction. Christensen (1992) also reported in his review that C enrichment in the fine fraction was inversely related to its proportion in the whole soil. Dalal and Bridge (1996) similarly noticed preferential C enrichment of the clay fraction in coarse-textured soils. Studying grassland topsoils from Canada to Texas, Amelung et al. (1998) observed that C concentrations in the dispersed fractions 250-2000 and 20-250  $\mu\text{m}$  were inversely related to their respective proportions in the whole soil, and suggested that it was largely explained by a dilution effect. The influence of texture on C concentration in

coarse fractions could also be attributed to encrustation of plant debris by fine mineral particles, which are attached to mucilages produced by microorganisms that feed on debris (Tisdall and Oades, 1982; Angers and Chenu, 1998). This helped explain the positive correlation between clay plus fine silts and C concentration and enrichment in the dispersed fraction  $> 200 \mu\text{m}$ . Additionally, the present study showed that the relationship between the mass of the fraction and the C amount it contains was less clear for the dispersed fractions 20-200 and  $> 200 \mu\text{m}$  than for that  $< 20 \mu\text{m}$ . This suggested that soil OM dynamics were more affected by fine than by coarse primary particles. This was confirmed by Amelung et al. (1998) for a range of North American grasslands, where C amount in the dispersed fraction  $< 20 \mu\text{m}$  correlated positively with clay plus fine silts, whereas C amounts in coarser fractions did not relate to texture.

#### *4.5. Influence of sesquioxides on soil aggregation*

Soil content in stable macroaggregates correlated closely with  $\text{Al}_{\text{CBD}}$ ,  $\text{Al}_{\text{OX}}$ , and to a lesser extent, with  $\text{Fe}_{\text{CBD}}$  and  $\text{Fe}_{\text{OX}}$ . Many authors mentioned the importance of sesquioxides in aggregate stability, in LAC soils especially (Kemper and Koch, 1966; Oades and Waters, 1991; Le Bissonnais, 1996; Six et al., 2000b). The role of these compounds has been attributed to their flocculation capacity, to their binding effect of clay particles to organic molecules, and to their possible precipitation as gels on clay surfaces, but this was reported to affect mesoaggregation mainly (Amézketa, 1999). Electrostatic bindings between clays and oxides has also been mentioned, but due to their limited range of action, it has been hypothesized that these interactions result in macroaggregates that are not stable enough to resist fast wetting (Six et al., 2002).

While the predominant role of free Fe oxides in macroaggregate stability has been reported by several authors, for Oxisols especially (Kemper and Koch, 1966; Dalal and Bridge, 1996), the importance of Al-containing crystalline sesquioxides, as observed in the present study, has not been strongly supported by the literature. The effectiveness of gibbsite in maintaining soil aggregation has been reported to be higher than that of Fe-oxides (El-Swaify and Emerson, 1975). However, correlation between  $\text{Al}_{\text{CBD}}$  and macroaggregation could not be attributed to gibbsite because: (i) gibbsite is little attacked by CBD thus releases little  $\text{Al}_{\text{CBD}}$  (Jackson et al., 1986), and (ii)  $\text{Al}_{\text{CBD}}$  was maximum in Congolese samples, which did not include gibbsite. Aluminium-substituted crystalline hematite and goethite are dissolved by CBD (Jackson et al., 1986), and were probably the main sources of non-oxalate extractable  $\text{Al}_{\text{CBD}}$  in the studied samples. As macroaggregate content correlated moderately

with Fe<sub>CBD</sub>, the aggregating role of crystalline Fe-sesquioxides seemed to result principally from the degree of Al-for-Fe substitution in their structure. Indeed, several authors reported that the specific surface area of crystalline Fe-sesquioxides increases with Al-substitution, mainly due to decreasing crystallite size (Schwertmann and Taylor, 1989; Ruan and Gilkes, 1995). As aggregation relates to specific surface area (Goldberg et al., 1988; Le Bissonnais, 1996; Amézqueta, 1999), the increase in stable macroaggregates with Al<sub>CBD</sub> might thus result from the increase in specific surface area with Al-substitution on crystalline Fe-sesquioxides.

#### *4.6. Influence of sesquioxides on soil organic matter*

The present study also showed close relationships between organic constituents and Al-containing sesquioxides, coarse fractions (> 20 µm) relating with non-crystalline compounds preferentially (Al<sub>OX</sub>), and fine fractions (< 20 µm), much more closely, with both crystalline and non-crystalline ones (Al<sub>CBD</sub>). The affinity of OM to Al- and Fe-sesquioxides, either crystalline or non-crystalline, has been reported in the literature, regarding fine organic fractions as well as incompletely humified residues, which can be stabilized by sorption, by entrapment, or by complexation (Guggenberger and Haider, 2002).

The role of amorphous Al and Fe in soil OM protection against biodegradation, through the formation of organo-mineral complexes, has been highlighted by numerous authors (Boudot et al., 1986; Baldock and Skjemstad; 2000; Guggenberger and Haider, 2002). Baldock and Skjemstad (2000) reported that Al-complexes resulted in a greater stabilization of OM than Fe-complexes, as observed in the present study: C amount in every dispersed fraction correlated more closely with Al<sub>OX</sub> than with Fe<sub>OX</sub>.

Moreover, it is likely that the main sources of non-oxalate extractable Al<sub>CBD</sub> in the studied samples were Al-substituted crystalline Fe-sesquioxides, as mentioned in the previous section. As also mentioned, the increase in Al-for-Fe substitution causes an increase in specific surface area, and this in turn causes an increase in OM stabilization (Baldock and Skjemstad; 2000; Kaiser and Guggenberger, 2003). This helped to explain the correlations between Al<sub>CBD</sub> and Ct or fraction C amounts. Considering that micropores are preferential sites for OM sorption (Kaiser and Guggenberger, 2003), the particular affinity of organic particles < 20 µm to non-oxalate extractable Al<sub>CBD</sub> might be due to the small crystallite size of Al-substituted Fe-sesquioxides, the micropores of which are accessible to small particles only. Studying the mineral association of OM in the surface horizons of an Alfisol and an Oxisol, Shang and Tiessen (1998) also observed the enrichment of the fine fractions in Al<sub>CBD</sub>, while

Al<sub>OX</sub> was particularly concentrated in the light fractions (i.e. including coarse organic particles mainly).

#### *4.7. Determinants of soil aggregation and organic matter content*

Many authors observed positive correlations between stable macroaggregation and OM, which were attributed to the aggregating role of OM and to physical protection of OM within aggregates (Feller and Beare, 1997; Angers and Chenu, 1998; Six et al., 2002). Several works reported that some organic fractions, such as polysaccharides or particulate OM, were particularly involved in these relationships (Tisdall and Oades, 1982; Kouakoua et al., 1999; Baldock, 2002). This was not supported by the present results, macroaggregation being more closely correlated with Ct than with any organic size fraction.

Moreover, considering all 18 soil samples, stable macroaggregation was more closely linked with Al<sub>CBD</sub> than with other parameters, organic ones especially. Tisdall and Oades (1982) already underlined that organic materials were not necessarily the main aggregating agents, especially in soils that included more than 10% sesquioxides. Oades and Waters (1991) also suggested that oxides were dominant stabilizing agents in the Oxisol they studied. Six et al. (2002) similarly hypothesized that stable macroaggregation was less dependent on OM in highly weathered tropical soils than in temperate ones, because the formers had a greater potential to form physicochemical macroaggregates, in the presence of oxides especially. Moreover, C amount in any dispersed fraction was more closely linked with Al<sub>CBD</sub> or Al<sub>OX</sub> than with macroaggregation or texture. This suggested that the stability of OM was more dependent on its association with Al-containing sesquioxides than on physical protection within stable aggregates or sorption to clay particles. It could thus be assumed that Al-containing sesquioxides were the main determinants of stable macroaggregation and OM content in the LAC tropical soils under study.

However, when each location was considered separately, the influence of land use increased markedly. The negative consequences of land use intensification (i.e. cultivation, tillage, reduced surface cover) on many soil properties are fairly well established. As far as tropical soils are concerned, they have been reported for topsoil aggregation (Resck et al., 2000; Spaccini et al., 2001) and for total and fraction C (Feller and Beare, 1997; Solomon et al., 2002; Vågen et al., 2005). This underlines that at the local scale, due to rather homogeneous mineralogical properties, land use remains an important determinant of soil OM content and aggregation.

## 5. Conclusion

Over the range of LAC tropical soils that were studied, aggregate distributions were dominated by stable macroaggregates ( $> 200 \mu\text{m}$ ), and C distributions in primary particles by fine fraction C ( $< 20 \mu\text{m}$ ). Macroaggregation was not clearly affected by texture, whereas total C and fine fraction C increased with clay plus fine silts. The results also suggested that Al-containing sesquioxides were major determinants of macroaggregate and OM stability. Indeed, macroaggregate content correlated positively with  $C_t$  and with C in every fraction, with fine fraction C especially, but it correlated more closely with CBD-extractable Al. This suggested that Al-containing sesquioxides had a more important aggregating role than OM in LAC tropical soils. Moreover, C amounts in fine ( $< 20 \mu\text{m}$ ) and coarse ( $> 20 \mu\text{m}$ ) fractions related more closely with CBD- and oxalate-extractable Al, respectively, than with stable macroaggregate content or texture. Thus the stability of soil OM was more influenced by its association with Al-containing sesquioxides than by physical protection within stable macroaggregates or sorption to clay particles. It could also be assumed that Al-containing sesquioxides played a dominant role in the relations between OM and aggregation. However, considering each location separately, soil content in sesquioxides varied within narrow ranges, and as it cannot be managed easily, the importance of land use on OM and aggregation remained fundamental.

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**Table 1.** Presentation of the studied sites, soils, and plots.

Country (state), city	Latitude, longitude	Mean annual rainfall and temperature	Soil type <sup>a</sup> (and parent material)	Texture	Clay mineralogy				Land use	No
					K	H	Go	Gi		
Congo-Brazzaville, Loudima	04°00'S, 13°30'E	1100 mm 25°C	Typic Haplorthox <i>Orthic Ferralsol</i> (schisto-calcareous sediment)	Clayey	+++	0	++	0	Savanna	1
									Cassava	2
									Natural fallow	3
Congo-Brazzaville, Mantsoumba	04°10'S, 13°30'E	1400 mm 25°C	Typic Haplorthox <i>Orthic Ferralsol</i> (schisto-calcareous sediment)	Clayey	+++	0	++	0	Savanna	4
									Cassava	5
Benin, Cotonou	06°24'N, 02°20'E	1200 mm 27°C	Typic Tropudult <i>Dystric Nitisol</i> (sandy clay sediment)	Sandy clay loam	+++	++	++	0	Maize, no input	6
									Fertilized maize	7
									Maize-legume intercrop	8
									Rotation of maize & maize-legume intercrop	9
Brazil (Goiás), Goiânia	16°S, 49°30'W	1500 mm 23°C	Typic Haplustox <i>Orthic Ferralsol</i> (detritic-lateritic sediment)	Clayey	+++	++	++	++	Bush savanna	10
									Maize/bean rotation	11
									<i>Bracharia</i> pasture	12
Brazil (São Paulo), Pradópolis	21°22'S, 48°03'W	1600 mm 23°C	Typic Hapludox <i>Orthic Ferralsol</i> (basalt)	Clayey	+++	++	0	++	Sugarcane (residue burning)	13
Brazil (São Paulo), Matão	21°36'S, 48°22'W	1500 mm 23°C	Typic Hapludult <i>Orthic Acrisol</i> (sandstone)	Sandy clay loam	+++	++	++	0	Sugarcane (residue burning)	14
Brazil (São Paulo), Serrana	21°12'S, 47°35'W	1500 mm 23°C	Typic Quartzipsamment <i>Ferralic Arenosol</i> (sandstone)	Sandy	+++	+	+	+	Sugarcane (residue burning)	15
Brazil (Paraná), Londrina	23°23'S, 51°11'W	1600 mm 21°C	Typic Haplorthox <i>Rhodic Ferralsol</i> (basalt)	Clayey	+++	++	++	++	Forest	16
									Oat	17
									<i>Citrus</i> orchard with legume intercropping	18

<sup>a</sup> Soil Survey Staff (1999) and *FAO-ISRIC-ISSS* (1998).

K: kaolinite; H: hematite; Go: goethite; Gi: gibbsite.

+++; dominant; ++; present; +; traces; 0: absent.

**Table 2.** Size distributions of aggregate and dispersed fractions, C contents of whole soil and dispersed fractions, and whole soil pH (in water) and contents in citrate-bicarbonate-dithionite- and oxalate-extractable iron and aluminium (for No, see Table 1).

No	Mass of dispersed fractions (g kg <sup>-1</sup> soil)			Mass of aggregate fractions <sup>a</sup> (g kg <sup>-1</sup> soil)			Total soil C content (g C kg <sup>-1</sup> soil)	C concentration of dispersed fractions (g C kg <sup>-1</sup> fraction)			C amount in dispersed fractions (g C kg <sup>-1</sup> soil)			pH (H <sub>2</sub> O)	Fe <sub>CBD</sub>	Al <sub>CBD</sub>	Fe <sub>OX</sub>	Al <sub>OX</sub>
	> 200 μm	20-200 μm	< 20 μm	> 200 μm	20-200 μm	< 20 μm		> 200 μm	20-200 μm	< 20 μm	> 200 μm	20-200 μm	< 20 μm					
1	38	104	857	669	238	55	35.0	76.2	32.1	27.6	2.9	3.3	23.7	5.0	30.4	6.4	1.3	2.1
2	40	188	775	571	354	48	21.8	34.7	11.8	21.5	1.4	2.2	16.7	6.0	35.6	6.8	1.4	2.2
3	31	201	760	621	309	41	36.4	69.0	27.8	36.6	2.1	5.6	27.8	5.5	32.0	7.6	1.5	3.3
4	93	260	632	746	154	7	42.5	72.0	39.5	35.5	6.7	10.3	22.4	5.0	33.7	6.9	2.9	5.8
5	32	179	771	585	366	17	18.8	51.0	13.5	18.6	1.6	2.4	14.4	4.4	nd	nd	1.2	1.9
6	524	202	226	164	207	18	5.1	1.4	4.5	11.1	0.7	0.9	2.9	5.1	9.0	0.7	0.5	0.4
7	625	216	145	210	132	13	6.5	1.3	6.9	22.8	0.8	1.5	3.3	5.2	7.7	0.7	0.4	0.4
8	611	202	175	238	97	11	11.4	2.1	11.4	30.3	1.3	2.3	5.3	5.0	8.6	0.7	0.4	0.5
9	590	208	192	246	148	17	8.5	1.5	7.7	21.4	0.9	1.6	4.1	5.2	8.4	1.0	0.4	0.4
10	81	449	469	615	313	2	22.6	21.3	8.0	34.0	1.7	3.6	15.9	5.5	35.0	4.8	2.2	nd
11	47	383	571	541	382	10	21.4	25.3	3.9	29.9	1.2	1.5	17.1	5.2	38.9	4.7	1.9	4.9
12	67	456	477	623	276	8	22.0	22.8	3.7	34.9	1.5	1.7	16.6	5.9	29.9	5.4	1.9	3.9
13	78	325	597	661	246	20	20.7	11.1	16.9	21.1	0.9	5.5	12.6	nd	98.8	6.0	1.7	3.1
14	503	300	192	329	143	12	7.4	1.4	4.8	27.2	0.7	1.5	5.2	nd	15.1	2.5	0.5	0.7
15	411	505	83	298	320	15	7.0	1.6	4.4	49.2	0.7	2.2	4.1	nd	11.2	1.5	1.0	0.6
16	47	146	766	752	189	13	30.8	118.1	35.3	22.6	2.6	5.2	17.3	6.8	87.1	5.1	5.3	5.4
17	39	101	868	551	388	23	17.8	71.9	21.7	14.9	0.5	2.2	12.9	5.7	70.6	4.5	5.3	2.7
18	29	122	834	639	309	30	23.0	87.4	18.9	18.9	1.1	2.3	15.7	5.9	72.0	5.1	5.2	2.8

nd: not determined.

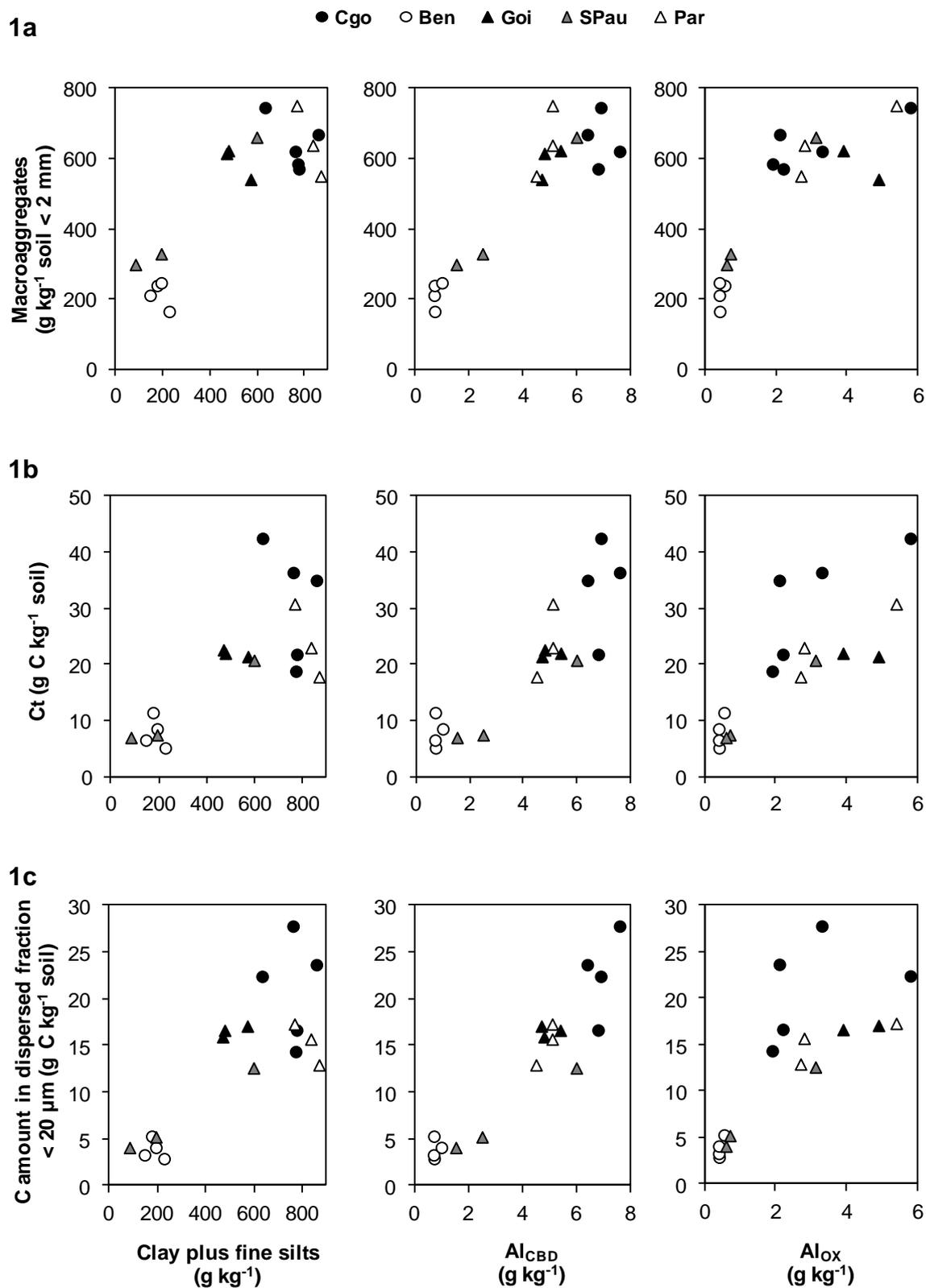
<sup>a</sup> aggregate fraction > 200 μm did not include coarse sands; aggregate fractions 20-200 and < 20 μm included primary particles of the same size.

**Table 3.** Main correlations between the studied variables (C concentration in g kg<sup>-1</sup> fraction, C amount and other variables in g kg<sup>-1</sup> soil < 2 mm).

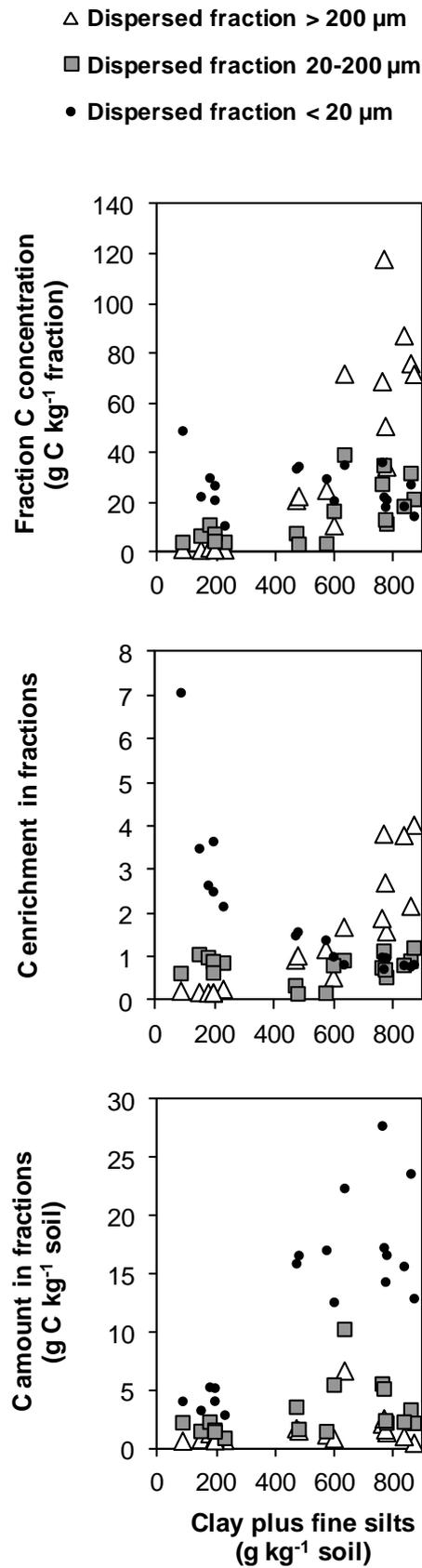
	Dispersed fraction mass			Macroaggregates	Ct	Fraction C concentration			Fraction C amount			Fe <sub>CBD</sub>	Al <sub>CBD</sub>	Fe <sub>OX</sub>	
	> 200 μm	20-200 μm	< 20 μm			> 200 μm	20-200 μm	< 20 μm	> 200 μm	20-200 μm	< 20 μm				
Fraction mass {															
20-200 μm	0.070														
< 20 μm	-0.906***	-0.483*													
Macroaggregates	-0.935***	-0.085	0.852***												
Ct	-0.774***	-0.200	0.759***	0.883***											
Fraction C {															
concentration > 200 μm	-0.712***	-0.541*	0.842***	0.769***	0.771***										
20-200 μm	-0.505*	-0.537*	0.663**	0.676**	0.831***	0.840***									
< 20 μm	-0.023	0.686**	-0.262	0.097	0.221	-0.130	-0.009								
Fraction C {															
amount > 200 μm	-0.345	-0.095	0.332	0.550*	0.782***	0.493*	0.737***	0.297							
20-200 μm	-0.411	-0.048	0.372	0.627**	0.777***	0.490*	0.793***	0.293	0.856***						
< 20 μm	-0.859***	-0.160	0.821***	0.878***	0.953***	0.729***	0.686**	0.210	0.601**	0.597**					
Fe <sub>CBD</sub>	-0.701**	-0.225	0.711**	0.723**	0.446	0.615**	0.481	-0.315	0.057	0.364	0.424				
Al <sub>CBD</sub>	-0.926***	-0.098	0.863***	0.928***	0.889***	0.662**	0.622**	0.137	0.516*	0.601*	0.938***	0.575*			
Fe <sub>OX</sub>	-0.621**	-0.310	0.668**	0.641**	0.468	0.801***	0.546*	-0.207	0.199	0.306	0.423	0.776***	0.460		
Al <sub>OX</sub>	-0.769***	0.049	0.638**	0.854***	0.810***	0.672**	0.601*	0.158	0.623**	0.653**	0.759***	0.611**	0.760***	0.632**	

\* p < 0.05; \*\* p < 0.01; \*\*\* p < 0.001.

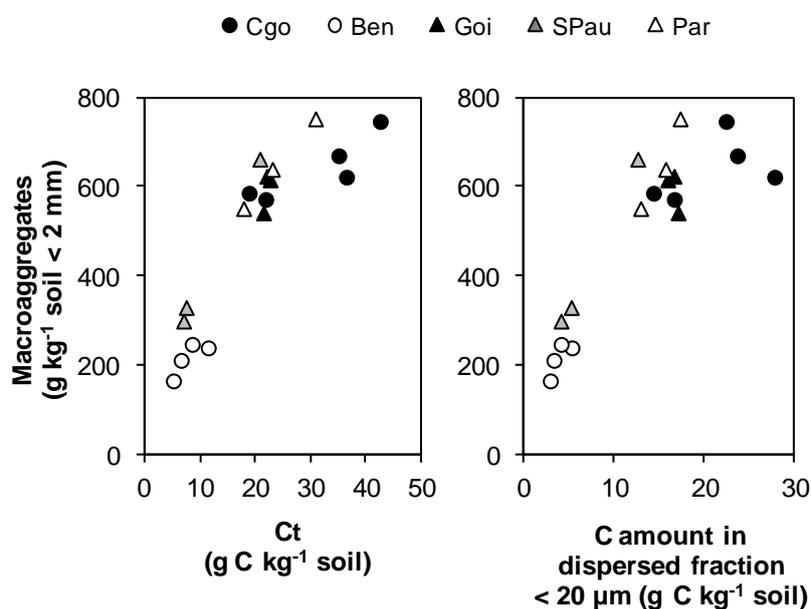
**Figure 1.** Relationships between clay plus fine silts ( $< 20 \mu\text{m}$ ), citrate-bicarbonate-dithionite-extractable Al ( $\text{Al}_{\text{CBD}}$ ), oxalate-extractable Al ( $\text{Al}_{\text{OX}}$ ), on the one hand, and: **1a**, soil content in water-stable macroaggregates ( $> 200 \mu\text{m}$ ); **1b**, total soil C content (Ct); and **1c**, C amount in the dispersed fraction  $< 20 \mu\text{m}$  (Cgo, Ben, Goi, SPau and Par represent samples from Congo, Benin, Goiás, São Paulo and Paraná, respectively).



**Figure 2.** Relationships between clay plus fine silts (<20  $\mu\text{m}$ ) and C concentration, enrichment and amount in dispersed fractions.



**Figure 3.** Relationships between total soil carbon content (Ct), C amount in the dispersed fraction < 20  $\mu\text{m}$ , and stable macroaggregate (> 200  $\mu\text{m}$ ) content (Cgo, Ben, Goi, SPau and Par represent samples from Congo, Benin, Goiás, São Paulo and Paraná, respectively).



**Figure 4.** Principal component analysis (PCA) with seven active variables, in bold, and six supplementary variables, in italics (C>200, C20-200 and C<20 represent C amount in the dispersed fractions > 200, 20-200 and < 20  $\mu\text{m}$ , respectively).

