ORGANO-MINERAL INTERACTIONS IN TROPICAL SOILS. IN SEARCH OF "FUNCTIONAL" ORGANIC MATTER POOLS: THE PARTICLE-SIZE FRACTIONATION APPROACH

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The objective of this paper was to illustrate how a particle size fractionation approach for characterizing soil organic matter (SOM), enables or not the identification of " functional " SOM pools.

A recall on historical aspects of SOM physical fractionation was presented, and some methodological problems underlined.

It appeared that the particle size fractionation was *a priori* an interesting approach for identifying " organic functional pools ", based upon their very different origin, composition and dynamics; three main fractions were then proposed: a " plant debris compartment " (fraction 20-2000 or 50-2000 µm), an " organo-silt complex " (fraction 2-20 or 2-50 µm) and an " organo-clay compartment " (fraction 0-2 µm). All examples presented concerned low activity clay (LAC) soils.

These pools were tested as for their " functionality ". The notion of functionality has been defined and quantified. Each property was defined by its " descriptor value " (i.e. the mineralized C per unit of total C). A " functionality index (FI) " quantifies the distribution of a studied property within the different soil fractions, and a " functionality scale " was proposed. This approach was applied to three types of functions: a biological function such as short-term C mineralization, a mineral exchange function such as CEC, and an organic sorption function such as atrazine adsorption. The results showed that:

- the only descriptor value is insufficient to evaluate the role played by a given organic compartment for a given soil property, for the distribution of the property within the soil fractions must also be considered,
- a same fraction does not exhibit a same level of functionality depending upon the characteristics of the soil,
- a same fraction, for a given soil, does not exhibit a same level of functionality depending upon the type of the studied function,
- the " plant debris compartment " is an important functional pool for a biological function such as short-term C mineralization in sandy soils (but not in the clayey ones); the " organo-silt complex " is a functional pool mainly for sorption processes, particularly for sandy soils, but not for clayey oxisols; the " organo-clay compartment " is a functional pool for all the studied functions in clayey soils, and also for the exchange function in sandy soils.

This approach must be extended to others SOM functions, under different environmental conditions.

I. Introduction

Total SOM (expressed as total carbon Ct) exerts essential and different functions in soil:

- biological functions, such as easily mineralizable carbon or nitrogen (Cm, Nm), microbial nitrogen immobilization, enzymatic activities.
- exchange and sorption functions, such as cation exchange capacity or sorption of pesticides,
- function of aggregation,
- functions of medium- to long-term storage (" sequestration ") of elements and/or nutrients for plants and soil organisms, such as total organic carbon, nitrogen, phosphorus, sulfur, non-exchangeable bases associated to SOM.

It is thus important to identify, for a given function, which part(s) of the total SOM represent(s) most of the considered function. This is the notion we shall refer to when functionality of SOM pools (or reactive sites) will be discussed throughout this paper.

The notion of soil organic matter (SOM) or soil organic carbon (SOC) functional pool is often quoted in the literature (Tiessen et al., 1984; Duxburry et al., 1989; Theng et al., 1989; TSBF, 1989; Bonde et al., 1992; Christensen, 1992; Cambardella and Elliott, 1993, 1994; Feller, 1993; Woomer, 1993; Herrick and Wander, 1997; Monreal et al., 1997; Sternberg, 1998). But this notion is generally poorly defined, never quantified and is more generally restricted to the dynamics of SOM than applied to the different and numerous functions that SOM plays in the soils or in the soil-plant-atmosphere system.

In a previous paper (Feller et al., 1999 in press), we gave a quantitative definition of the functionality of SOC or SOM pools. In this communication we want to illustrate how a particle size fractionation of the soil allows, or not, to study some major functions of SOM involving different types of interactions.

This paper is divided in three parts:

- recalls on some historical aspects of the physical fractionation of SOM,
- some methodological considerations on SOM particle size fractionation of SOM and main characteristics of the fractions,
- applications of particle size fractionation to the identification of functional pools in low activity clay (LAC) tropical soils, for the following functions: short-term C mineralization, cationic exchange capacity and pesticide adsorption.

II. Materials and Methods

A. Sites, soils and land use

The results reported here were obtained from the pedological situations summarized in Table 1.

All the soils were low activity clay (LAC) soils with a mineralogy of the clay fraction dominated by kaolinite or halloysite associated with iron and/or aluminium oxyhydroxides. As the texture is an important factor in the SOM content and soil functioning, we choosed sandy (Ft1 and Fl1) and clayey (Fi6 and Fo8) soils. The main site and soil characteristics are presented in Table 1. Others analytical data are detailed in Feller (1995). All the samples originated from the surface horizon, generally the 0-10 cm layer, except for the Fo8 site (0-6 cm). Each soil sample was constituted from 6 to 12 replicates.

- fraction 2-20 µm (f2-20), the "organo-silt complex": consisting of very humified plant and fungi debris associated with stable organomineral microaggregates which have not been destroyed during the fractionation, with medium to low C/N ratio, medium XYL/MAN ratio and turnover rate y %,
- fraction < 2 µm (f0-2), the "organo-clay fraction": with predominance of amorphous OM acting as a cement for the clay matrix, with low C/N ratio and XYL/MAN ratios and low turnover rate y %. Sometimes, under forest or savanna, presence of plant cell walls occurs in the coarse clay fraction (f0.2-2.0) but usually not in the fine clay (f0-0.2). Very often, bacterial cells or colonies at different stages of decomposition can be observed in both fractions.

Table 2. Some characteristics of the particle size fractions of an Oxisol (Fo8 site) under sugarcane (50 yrs) after forest:
- carbon to nitrogen ratio C/N is an indice of the stage of decomposition,
- xylose to mannose ratio XYL/MAN is an indice of the origin (vegetal vs microbial) of OM: high or low for plant or microbial origin,
- y is the C of the fraction derived from sugarcane and expresed in percentage (y %) of the total C of the fraction (sugarcane+forest). y % was determined from natural ^{13}C abundance determinations.

Adapted from Feller, 1995.

Fraction	C/N	XYL/MAN	y %
f20-2000	23.2	6.1	60
f2-20	14.9	2.3	48
f0-2	11.5	2.3	34

C. Definitions of different functionality parameters: descriptor value, functionality index, functionality scale and functional SOM pools

These parameters were defined in Feller et al. (1999, in press) and will be summarized here:

- " descriptor value " (DV). The SOM functions derive from properties of organic entities or molecules that can be described and quantified by a " descriptor value ". For every fraction, the following examples of such descriptors were used in this paper : the coefficient of mineralization of the carbon (CM-Cm) expressed in g C mineralized . kg^{-1} C fraction, the organic cation echange capacity (orgCEC) expressed in cmole(+) . kg^{-1} C fraction and the normalized soil sorption coefficient Koc for atrazine expressed in L . kg^{-1} . g^{-1} C fraction,
- " functionality index " (FI). According to the value of the descriptor of a given property and the distribution of carbon within the soil sample fractions, it is easy to calculate the participation of each fraction (in %) to the total property expressed by the whole sample or the sum of the fractions. The participation of each fraction to a given soil property was called functionality index (FI),

Table 1. Some soil, climatic, and land use characteristics of the studied sites.

Locality[a]	Site[a]	Climate		Soil order	Samples[a]	Vegetation or crops	Horizon 0-10cm		
		P (mm)	T° C				Clay	Carbon	C/N
							(g.kg^{-1} soil)		
Sites studied for C mineralization									
Senegal	Ft1	700	29	Entisol	Mi6	millet	51	2.2	10.6
Martinique	Fi6	1820	26	Inceptisol	Ca50	sugarcane	493	19.2	12.0
Sites studied for organic CEC									
Senegal	Fl1	800	29	Alfisol	SA	tree savannah	76	9.3	15.0
Martinique	Fi6	1820	26	Inceptisol	Ca50	sugarcane	493	21.8	12.0
Sites studied for Atrazine adorption									
Senegal	Fl1	800	29	Alfisol	SA	tree savannah	76	8.9	15.0
Martinique	Fi6	1820	26	Inceptisol	Ca50	sugarcane	493	21.8	12.0
Brazil	Fo8	1200	21	Oxisol	F	Forest	560	42.5	9.9

[a] The symbols refer to the nomenclature used by Feller (1995). For the sample symbol, the number refers to the last duration (years) of the agricultural system.

B. Particle-size fractionation method

The particle-size fractionation method used in this study was described in Feller et al. (1991, “ method R/US ”). Briefly, it consists in shaking for 2 to 16 hours (duration depending upon the soil texture) the 0-2 mm soil sample (40 g) in water (300 ml) in presence of a cationic resin (R) saturated with Na+ to improve the soil dispersion. This was followed by wet sieving at 200 and 50 µm to separate the coarse (200-2000 µm) and fine (50-200 µm) sand fractions. An ultrasonic treatment (US) of the 0-50 µm suspension (100J/ml) improved the clay dispersion. The coarse silt fraction (20-50 µm) was obtained by sieving. The fine silt (2-20 µm) was separated from clay (0-2 µm) by repeated sedimentation-centrifugation operations.

The above method provided a high dispersion of the soil constituents.

C and N analyses were performed by dry combustion with a CHN Analyser (Carlo Erba, Mod. 1106). SOM solubilized during the fractionation procedure (less than 4 % of total SOM) was not considered in this study.

By simplification, and according to previous studies (Feller, 1995; Feller et al., 1996; Feller et Beare, 1997) on the morphology, carbon to nitrogen C/N ratio, xylose to mannose XYL/MAN ratio, and C dynamics (y %) of different particle-size fractions, we shall only consider here (below and Table 2) the three following fractions:

- fraction 20-2000 µm (f20-2000), the "plant debris fraction ": predominance of plant debris at different stages of decomposition, with high C/N and XYL/MAN and a high turnover rate (y %),

- " functionality scale ". The functionality index level will depend upon the number of considered fractions. Therefore, it is necessary to define a functionality scale taking into consideration the number of fractions. For a number n equal or higher than 2, we define:

* a low functionality (L),	when	$FI_i = 75/n$
* medium functionality (M),	when	$75/n < FI = 125/n$
* high functionality (H),	when	$Fi_i > 125/n$

- a " functional SOM pool ". A SOM fraction will be considered as a " functional " pool for a given function if its functionality index FI is " high " according to the functionality scale.

D. Soil carbon mineralization (whole soil and particle-size fractions)

For the whole soil (0-2 mm), 25 g were moistened at 80 % of their field capacity (pF 2.5) and incubated in 125 ml flasks for 28 days at 288 C. Evolved CO_2 was measured at 0, 2, 7, 14 and 28 days (Nicolardot 1988) and cumulated CO_2 evolved after 28 days was defined as *Cm*. For the size fractions, the fractions larger than 20 µm were incubated alone, but each of the 2-20 and 0-2 µm fractions were mixed (1/1, w/w) with coarse commercial sand. The incubation conditions were similar to those applied to the whole soil. All determinations were conducted in triplicate.

E. Total and organic cation exchange capacity

The CEC_{iso} used here is a measure of the CEC obtained from results (Feller, unpublished) on the selective K/Ca exchange properties of the size fractions, according to the method of Dufey et Delvaux (1989). This CEC determination is based on exchange isotherms curves on the separated fractions. It was shown (Feller, 1995) that CEC_{iso} is equivalent to the effective cationic exchange capacity ECEC for the LAC soils. SSA is the surface specific area determined by the BET-N_2 method and described in Feller et al. (1992). The organic cationic exchange capacity (orgCEC) was determined for each fraction by the difference between the measured CEC_{iso} and a calculated mineral CEC. For the clay fraction of the Fi6 and Fl1, a value of 10 cmol(+) . kg^{-1} fraction was used. For the other fractions, mineral CEC was deduced from the measured surface specific areas according to Fran¡ois (1988).

F. Atrazine adsorption

The batch equilibration method described in Barriuso et al. (1992a) was used for the bulk soil. For the fractions (Barriuso et al., 1992b) labelled ^{14}C-atrazine solutions were prepared in 0.01 M CaCl2 at 5 different concentrations: 10.0, 3.0, 1.0, 0.3 and 0.1 µg . L-1. A 10 ml aliquot of each solution was added to 10 g fraction sample and mechanically shaken for 24 h at 25±28C. Then the suspentions were centrifuged for 20 min at 3000 x *g*. and the supernatant filtered through a 0.45 µm filter (HV Millipore). The amount of atrazine in solution was determined by radioactivity measurement in the supernatant. The amount of atrazine adsorbed to the soil or to the fractions was determined by the difference between the atrazine concentration in soil- or fraction-and the blanks and atrazine in solution after equilibration. All adsorption studies were run in duplicate for each initial atrazine concentration. The distribution coefficient, K_d, was calculated as follows :

$$K_d = (x/m)/C_e$$

where x is the mass of adsorbed atrazine (µg), m the mass of soil (g), and C_e the atrazine concentration ($mg.L^{-1}$) of the supernatant solution at equilibrium. The distribution coefficient (K_{OC}) was related to the soil organic carbon content (% C), by expressing the concentration of the adsorbed herbicide per unit of organic carbon :

$$K_{OC} = (x/m)_C/C_e = K_d.100/\% C$$

where $(x/m)_C$ is the mass of adsorbed herbicide (µg) per gram of soil organic carbon (Hamaker and Thompson, 1972).

III. Results and Discussion

A. Historical aspects on SOM particle-size fractionation

A.1. Before 1950

Since the early 19th century, with the extraordinary development of chemistry, the techniques used to study soil organic matter up to now, have been inevitably dominated by chemical characterizations (alkali-acid extraction, and generally, chemical fractionations). The humus fractionations have represented, and still represents, an essential tool in soil pedogenesis and soil ecology in temperate (Duchaufour, 1970) as well as tropical areas (Thomann, 1964 ; Perraud, 1971 ; Turenne, 1977 ; Dabin, 1980/81). Its application to the study of water-soluble OM is essential.

However, in so far as soil functioning (aggregation, exchange dynamics, mineralization potential) is concerned, it is often difficult to ascribe definite and measurable functions to organic fractions obtained on the basis of solubility characteristics, and especially for the study of bio-organo-mineral interactions in soils.Yet Gasparin (1843) questioned the significance of humic extractions. Accordingly, over the last 20 years, as the validity of the SOM chemical approaches was questioned (Tiessen and Stewart, 1983 ; Anderson et al., 1983) for the study of agropedological processes, the use of a different characterization - based on particle-size and/or density fractionations of soil organic pools - was developed.

In contrast with the chemical or biological approaches (Kononova, 1961, Vaughan and Ord 1985), there do not exist many historical papers on the physical SOM fractionation that would be exhaustive enough ,especially before 1970. So we shall refer here to some more ancient references. Additionnally, more details were published recently in two papers (Feller 1997a,b and 1998).

In 1829, Martin described a method of separation of "humus" by sedimentation : it was the solid material remaining in the supernatant, after shaking the soil in water (1:8, w/v) for one minute. The precipitate contained "silica" and "alumina". But the first particle-size fractionation of OM was carried out by Schloesing in 1874 under suitable experimental conditions and criticism (Feller, 1998). This work could practically be published nowadays. Schloesing questioned the distribution of OM between sand and clay in natural samples to understand some physical soil properties. Schloesing thus set out a particle-size fractionation of OM in five fractions : " coarse sand ", " fine sand ", " scales ", and " two clay deposits ". The results were shown in two tables with recoveries (99,98 %, w/w), C and N content per gram of fraction, C and N content per gram of soil. It would not be better done today. One of Schloesing's conclusions was that " clay contains 69 % organic matter ; it is enough to be actually modified in the way it acts as a cement ". Using a separation technique of sands, Gain (1918) briefly described a physical separation of soil OM after trituration of soil with Ca $(NO_3)_2$ in two fractions : (i) " humus " that was plant debris floating after the treatment and (ii) " humic materials " extracted afterward from the material precipitated with acid (oxalic acid, boiling 1 hr).

In fact, except in Schloesing's work, before 1940, no concept has been established for the adequacy of " fractionation methods/nature of OM ". From 1940 onwards, the need for a separation of the " soil plant debris " (or " free organic materials ") from the remaining and more humified OM (" stable organic material "), has been clearly shown.

Between 1930 and 1950, some isolate works attempted to separate soil plant debris by densitometry (Finnell, 1933 ; Lein, 1940 ; Harris, 1941), particle-size fractionation (Shively and Weaver, 1939) or particle-size/density fractionation (McCalla et al., 1943).

A.2. Henin and Turc's works (1950) : a density fractionation method

But the most important work was that of Henin and Turc (1950) based on a density fractionation method. These authors distinguished two types of fractions :

- " light " fractions ($d < 1.75$), named " free OM ", characterized by a very high OM content (30-35g C/100g fraction), C/N ratios higher than 16, and essentially constituted of plant debris. These fractions account for 15-20 % of the total organic matter ;
- " heavy " fractions ($d > 1.75$), named " OM bound to inorganic constituents " characterized by a lower OM content (0.7-16g C/100g fraction), C/N ratios lower than 11, more humified than the light fractions and constituted in particular by the clay-humus complex.

This densitometric approach has been essentially developed between 1950 and 1970 by French scientists (Monnier et al., 1962 ; Duchaufour and Jacquin, 1966 ; Dabin, 1971), as a preliminary step in humus fractionation. However, the use of heavy liquids, either inorganic or organic, involves a range of methodological problems: contamination and/or denaturation of the fractions. This probably explain why this approach was not widely adopted. This approach was further advanced by Greenland and Ford (1964), and Ford et al. (1969) who improved the separation of " free OM " by combining sonication (for disrupting aggregates) with densitometry ($d = 2.0$).

A.3. Edwards and Bremner's works (1964-67): sonication and particle-size fractionation methods

Edwards and Bremner in 1964 and 1967a, referred to and developed earlier works in order to show the effectiveness of sonication in dispersing the soil in water (Whittles, 1923), with no need for a preliminary extraction of OM or the addition of a dispersing agent. Particle-size fractions can thus be separated together with the associated organic materials . This work is of cardinal importance as it provides a practical method for locating OM among particle-size fractions, with no interfering chemical reaction likely to denature the organic or inorganic constituents. At the same time, the study of clay-organic fractions highlights the concept of "clay-humus complex" (Greenland, 1965). Finally, depending on the energy level imparted, information can be obtained regarding OM associated with aggregates or microaggregates that have different stabilities (Edwards and Bremner, 1967b). With regard to history, let us point out that Edwards and Bremner in their first publications (1965, 1967a, b), demonstrated that Na-resins could be as successful as ultrasounds in dispersing strongly - aggregated samples; they also reported that shaking soils with beads may be used to disrupt aggregates. The fractionation schemes proposed by Feller et al. (1991a), and Bruckert et al. (1978), respectively, rely on these two former notions.

A.4. Numerous works on particle-size fractionation after 1970

It then appeared very rapidly (Chichester, 1969 ; McKeague, 1971 ; Watson and Parsons, 1974 ; Feller, 1979 ; Tiessen et al., 1984) that OM associated with size fractions higher than 20 or 50 μm, consisted largely of plant debris in varying stages of decomposition, with C/N ratios often higher than 15, whereas OM associated with clays were characterized by C/N ratios generally lower than 10. Generally, the turnover of OM associated with the different particle-size fractions strongly decreased from the coarse fractions (plant debris) to the fine fractions (organo-clay complexes) (Anderson and Paul, 1984 ; Balesdent et al., 1987, 1988). In other words, it is possible by simple particle-size fractionation in water, to easily separate forms of OM " natural " - adjective used occasionally when comparing this approach to the "humic" one (McKeague, 1971 ; Turchenek and Oades, 1974) - very different from the point of view of their nature, properties and dynamics. In order to specify the nature of the fractions, densitometric separations can be applied to some or to the whole size-fractions (McKeague, 1971 ; Turchenek and Oades, 1974, 1979 ; Spycher and Young, 1977).

Bruckert et al. (1978) and Bruckert (1979) have proposed particle-size fractionations of OM employing shaking of the soil in water with beads, instead of sonication, and Feller et al. (1991) proposed to associate the use of cationic resin (form Na^+) and ultrasonication. Most of the works published actually, use sonication (Elliott and Cambardella, 1991 ; Morra et al., 1991) but do not always take into account its modifying effect on soil plant debris (Balesdent et al., 1991). Alternative methods were proposed involving ultrasonication only on the fractions inferior to 50 μm (Balesdent et al., 1991; Feller et al., 1991).

Presently, numerous studies are being developed through the physical approach to analyze the role of SOM in biogeochemical cycles and macro- or microaggregations. They are often associated to microscopic and ultramicroscopic techniques, chemical, biochemical, spectroscopic and isotopic analyses to establish the precise nature of SOM, its relationships with biological and mineral constituents in the particle-size fractions and its turnover.

B. Some methodological problems involved in SOM fractionation

B.1. Organic matter size fractionation (OMSF) is not Aggregate size fractionation (AGSF)

It is important to remember that objectives of OMSF are not those of AGSF even if they both involve a particle size approach. The " philosophy " of OMSF is (or has) to isolate, as quantitatively as possible, the forms of natural OM associated to sand, silt and clays. The " philosophy " of AGSF is to study the distribution of soil aggregates, these aggregates representing generally associations between OM and mineral particles (sand, clay and silt). Therefore, one methodological constraint in OMSF is to achieve a complete disaggregation and dispersion of the soil, without alteration of the natural forms of SOM, including the minimum of SOM solubilization.

B.2. " Desaggregation " versus " alteration of fraction "

Most of the works published actually, use sonication to achieve soil desaggregation and dispersion (Elliott and Cambardella, 1991 ; Morra et al., 1991) but do not always take into account its modifying effect on soil plant debris. Balesdent et al. (1991) showed that ultrasonication applied as usual to the whole soil sample 0-2 mm conducted to a transfer of up to 50 % of the carbon of the plant debris fraction larger than 50 µm to the finer one (< 50 µm). Feller et al. (1991) have shown a similar effect on the plant debris fraction for methods involving desaggregation by shaking the soil in water with glass beads, especially for coarse textured soils. In terms of decomposition process this can lead to errors in interpreting the results and could explain that, in some studies the participation of the fraction > 50 µm to the total SOM appeared very low (Feller and Beare 1997). Alternative methods were proposed by Feller et al. (1991) and Gavinelli et al. (1995) involving the following sequence:

* a shaking time less than 2 hours (50 rpm mn^{-1}) of the whole soil sample in water, in the presence or not (according to the soil aggregate stability) of a chemical dispersant (sodium hexametaphosphate HMP) or a sodic resin (R),

* a separation by sieving of the fractions > 50 µm,

* an ultrasonication of the 0 - 50 µm suspensions to achieve a dispersion of clay before separation of silt from clay.

B.3. Control of desaggregation and dispersion level

This control can be easily done by:

- optical observations. With optical microscopy it is possible to have a first qualitative evaluation of the nature of the separates larger than 50 µm, the objective being to have minimum particles in the form of organo-mineral aggregates,
- comparisons of the mineral mass distribution between the OMSF and the particle size fractionation obtained after oxidation (by H_2O_2) of the SOM (" mechanical analysis ", MEAN). The two distibutions have to be the most similar as possible. Often, with OMSF the complete separation between fine silt and clay is difficult to obtain, but a total disaggregation of aggregates larger than 50 or 20 µm is necessary and has to be controlled,
- comparisons between the C:N ratios of the different fractions. Generally, but not always, the C:N ratios decrease drastically from the coarse sand fraction to the clay one. Therefore, for a given soil, an unusual low C:N ratio of the fractions > 50 µm can indicate the incomplete destruction of aggregates > 50 µm.

Finally, even if a complete dispersion was not achieved, it is important for a good discussion of the results, to know which fractions can be considered or not, as completely disaggregated.

B.4. Proposition of a simplified method based on aliquots of the 0-20 µm suspension

To avoid the time-consuming of quantitative separation of fine silt and clay (" the decanting method "), we evaluated (Gavinelli et al., 1995) the possibility to achieve an acceptable C and N balance for the sum of the fractions with aliquots extraction of the 0-20 µm suspension (" the aliquot method "). We operated as follows:

- separation of coarse sand, fine sand and coarse silt by sieving at 200, 50 and 20 µm,
- extraction from the 0-20 µm suspension, of 0-20 and 0-2 µm aliquots.

All fractions were dried, weighed and analyzed for C and N. The C and N concentrations (g C, N per kg fraction) and amounts (g C, N per kg soil) of the 2-20 fraction were calculated by differences between the C and N determined on the 0-20 and 0-2 µm fractions.

The results were compared with the " decanting " method and were in agreement for the C balance. For N balance, the correlation was not so good. Finally, if the " decanting " method is preferable, the " aliquot method " can be successfully used for C balance when numerous samples have to be fractionated and/or when a complete separation (for other analysis) of the fine silt and clay fractions is not necessary.

B.5. Balances in mass, carbon and nitrogen

The quality of the interpretation of the results will depend on the mass, C and N balances obtained after fractionation. For that purpose, the sum of the fractions (SUM), including the water-soluble fraction when possible, has to be compared to the non fractionated soil sample (NF Soil). From our experience, the fractionation can be considered as acceptable if the range in percent of recuperation is: 97 - 102 % for mass, 90 - 110 % for carbon and 80 - 120 for nitrogen.

C. Functionality of particle-size fractions for carbon mineralization, cationic exchange capacity and atrazine adsorption

For samples described in Table 1, we studied the respective role of the different particle size fractions in relation to three SOM functions: short-term carbon mineralization, cationic exchange capacity and atrazine adsorption.

The characteristics of the bulk soils (non fractionated sample NF Soil) are presented in Table 3, and the different balances (Sum fractions in % NF Soil) after fractionation in Table 4.

Amounts of C, Cm, CEC and Q-ATRA are higher for the clayey samples Fi6-Ca and Fo8-F than for the sandy sample Fl1-SA. The differences were much higher for Cm and CEC than for Q-ATRA for (434 mg.kg^{-1}soil) than for the sandy one (Table 3). The organic part of the total CEC (orgCEC) was calculated by difference between the total measured CEC and an estimated mineral CEC (see • II.E). This orgCEC represents 68 an 41 % of total CEC for Fl1-SA (sandy sample) and Fi6-Ca (clayey sample), respectively.

Table 3. Contents in total carbon (C), mineralized carbon (Cm), cation exchange capacity (CEC) and adsorbed atrazine (Q-ATRA). Soil layer 0-10 cm.

Sample	C g.kg^{-1}soil	Cm mg.kg^{-1}soil	CEC cmole(+).kg^{-1}soil	Q-ATRA mg.kg^{-1}soil
Ft1-Am	2.2	122.5		
Fi6-Ca	19.2	434.0		
Fl1-SA	9.3		4.3	
Fi6-Ca	21.8		11.7	
Fl1-SA	8.9			10.2
Fi6-Ca	21.8			12.8
Fo8-F	42.5			15.1

All the mass and C balances are acceptable. The relative low values (c.a. 78 %) for Cm are probably due to the fact that the water soluble fraction, rich in easily mineralizable compounds, was discarded and not studied for C mineralization in this work. The balances for CEC are acceptable. The balances for adsorbed atrazine was good for the clayey samples Fi6-Ca and Fo8-F but was relatively low (71.8 %) for the sandy sample Fl1-SA. This suggests an over-estimated adsorption for the NF Soil.

Table 4. Balances after fractionation in mass, total carbon (C), mineralized carbon (Cm), cation exchange capacity (CEC) and adsorbed atrazine (Q-ATRA) of the sum of the fractions (Sum). Results expressed in % NF Soil. Soil layer 0-10 cm.

Sample	Mass	C	Cm	CEC	Q-ATRA
				Sum % NF Soil	
Ft1-Am	99.9	86.4	78.2		
Fi6-Ca	103.7	108.8	78.3		
Fl1-SA	100.0	104.3		88.8	
Fi6-Ca	96.9	88.8		94.0	
Fl1-SA	100.0	104.3			71.8
Fi6-Ca	99.6	89.1			99.8
Fo8-F	100.1	97.6			96.4

All these determinations were done on the fractions isolated after dispersion of the soils. It is interesting to observe that all balances for Cm, CEC and Q-ATRA were lower than 100. It means that the dispersion due to the fractionation method does not create an important part of reactive sites. In this case, the Sum ought to be higher than 100.

C.1. Fraction functionality in relation to short-term mineralization of soil C (Table 5)

The descriptor value DV-Cm is the mineralization coefficient of the C expressed in g Cm . kg^{-1} C fraction. For both samples DV-Cm varied in the order : f20-2000 > f0-2 > f2-20. The two soils differed more by the value of the f20-2000 fraction (higher for Ft1) than by the value of the f0-2 and f2-20 fractions. This trend towards higher DV-Cm values (or an equivalent index) for the sand-size fractions with regard to silt- and clay-size fraction was also observed by Christensen (1987), Gregorich et al. (1989), Hassink (1995) but not by Bernhardt-Reversat (1987, 1988) in the case of tropical sandy soils under savannah or tree plantation.

Table 5. Functionality parameters of the different fractions and the sum of the fractions (Sum) of sandy (Ft1-Am, Fl1-SA) and clayey (Fi6-Ca, Fo8-F) samples for the functions of mineralization (Cm), cation exchange capacity (org.CEC) and atrazine adsorption (ATRA).

Function	Sample	Fraction (μm)	DV	FI (% Sum)	Functionality Scale
Cm	Ft1-Am	20-2000	95.3	85.5	**H**
		2-20	10.4	3.3	L
		0-2	13.8	11.3	L
		Sum	50.5	100.0	
	Fi6-Ca	20-2000	25.2	31.8	M
		2-20	8.4	9.8	L
		0-2	15.6	58.4	**H**
		Sum	16.3	100.0	
orgCEC	Fl1-SA	20-2000	199	15.3	L
		2-20	219	28.8	M
		0-2	469	45.9	**H**
		Sum	280	100.0	
	Fi6-Ca	20-2000	99	6.8	L
		2-20	188	20.9	L
		0-2	287	72.3	**H**
		Sum	231	100.0	
ATRA	Fl1-SA	20-2000	274	37.2	M
		2-20	423	41.9	**H**
		0-2	202	21.0	L
		Sum	112	100.0	
	Fi6-Ca	200-2000	103	2.1	
		50-200	236	5.2	
		20-50	268	3.5	
		2-20	341	41.9	
		0.2-2.0	86	33.5	
		0-0.2	32	7.5	
		20-2000	186	17.1	L
		2-20	341	41.9	**H**
		0-2	69	41.0	M
		Sum	98	100.0	
	Fo8-F	20-2000	202	20.7	L
		2-20	237	19.3	L
		0-2	77	60.0	**H**
		Sum	58	100.0	

According to its very high functionality index FI-Cm (FI % = 85.5), the plant debris fraction (f20-2000) appeared to be the functional pool for the sandy soil Ft1 but not for the clayey soil Fi6 (FI % = 31.8). In this soil indeed, the functional pool was the organo-clay fraction f0-2 (FI % = 58.4).

The difference in the functionality of the same fraction according to the type of soil might be attributed to the distribution of the total C within the fractions (textural effect) rather than to the differences of the descriptor values DV-Cm. For example, even if we applied the corresponding DV-Cm values of sample Fi6 to the Ft1 fractions, the plant debris fraction (f20-2000) would have remained the functional pool for soil Ft1. Hassink (1995) also observed that DV-Cm did not differ significantly among size fractions in soils of different texture for temperate grasslands.

This emphasizes the fact that the only descriptor value seems insufficient to evaluate the role played by a given organic compartment for a given soil property; distribution of the property within the soil fractions must also be considered.

C.4. Fraction functionality in relation to cation exchange capacity (Table 5)

The descriptor value DV-orgCEC is the cation exchange capacity of OM in the fraction expressed in cmole(+) . kg^{-1} C fraction. In both samples, DV-orgCEC increased from the plant debris fraction (20-2000 µm), with values of 199 and 99 cmole(+) . kg^{-1} C fraction, to the clay fraction (469 and 287 cmole(+) . kg^{-1} C fraction). In comparison, with coarse textured tropical Alfisols Guibert (1999) gives values of 270 cmole(+) . kg^{-1} C fraction for the sand and silt fractions and about 1000 cmole(+) . kg^{-1} C fraction for the clay one. For temperate situations Leimweber et al. (1993) give a value of 560 cmole(+) . kg^{-1} C fraction for the clay fraction, of the same order than the others fractions

Both soils exhibited high functionality index for the clay fraction (FI % = 45.9 and 72.3). This fraction appeared thus as the functional pool for LAC soils, whatever the soil texture, even if the functionality index for sandy soil was lower.

C.5. Fraction functionality in relation to atrazine adsorption (Table 5)

The descriptor value DV-ATRA is the distribution coefficient (K_{OC}) related to carbon unit of atrazine. In the three samples, K_{OC} varied in the order : f2-20 > f20-2000 > f0-2. In the Fi6-Ca sample, K_{OC} was studied for the plant debris subfractions f200-2000, f50-200, f20-50 and for the coarse clay and fine clay. Considering all the fractions and subfractions, it appeared that the Koc values increased from the coarse sand fraction (f200-2000, K_{OC} = 103) to a maximum in the fine silt (f2-20, K_{OC} = 341) then decreased to low and very low values in the coarse clay (f0.2-2.0, K_{OC} = 86) and fine clay (f0-0.2, K_{OC} = 32). These lower values of coarse and fine clays were also observed for Fl1-SA (K_{OC} values of 243 and 50) and Fo8-F (K_{OC} values of 101 and 40).

The global variation of K_{OC} within the different fractions, characterized by a maximum for the f2-20, can neither be explained by the global variation of orgCEC (this paper), nor the specific surface areas (BET-N_2 method, Feller et al., 1992), that which increased from the coarse to the fine soil fractions, nor by the C:N ratio which decreased from the coarse-sand to the fine-clay fractions. Martin-Neto et al. (1994a) did the hypothesis that the mechanism involves in atrazine sorption (by humic acids) is not a charge-transfer mechanism but more weak interaction mechanisms as hydrogen bonding or proton-transfer. Different sorption processes have to be considered in relation to the fractions higher than 2 µm and the clay fractions:

- for fractions higher than 2 µm, K_{OC} increased with the increasing humification degree as it appeared with the C:N ratio variation or contents in humic material (Feller, 1995).

These results are in agreement with those of Walker and Crawford (1968) who showed that for atrazine (propazine) the K_d of soils incubated with straw was increasing during the humification process of the straw. This can be related to the chemical structure of OM, in particular to the free radicals semiquinone and aromaticity distibutions. Thus, for a Mollisol and a Gley-humic soil, Martin-Neto et al. (1994b, 1998) and Bayer et al. (submitted, pers. com.) shown that the aromaticty and the free radicals semiquinone reach maximum values for the 2-20 µm fraction,

- the K_{OC} of the organo-clay fractions were always lower than those of the fractions > 2µm in comparison to the fractions higher than 2 µm, and the K_{OC} of the fine clay fraction (< 0.2 µm) was always lower than that of the coarse clay. Several hypotheses can be invoked:

  * the OM quality differs strongly between the > 2 µm and < 2 µm fractions. In experiments of humic polymer synthesis, Andreux et al. (1992) showed that ATRA was more easily incorporated to fulvic than to humic acids. Barriuso et al. (1991) also showed, in field experiments, that ATRA-residues concentrations were higher in fulvic than in humic acids. In contrast, in sandy soils, Payaperez et al. (1992) observed an opposite distribution. In the absence of humic fractionation of our own samples, it is difficult to conclude. But the general composition observed by ^{13}C-NMR[1] for the different size fractions (see review of Mahieu et al., 1999) show that composition of clay fractions differs from the other fractions especially for their higher aliphatic and lower O-alkyl and aromatic groups. On the opposite, the O-alkyls are the most abundant group in the silt-size fractions.These observations can partly explain the lower atrazine adsorption for clay fraction,

  * the ATRA adsorption sites are protected within organo-clay microaggregates and are not easily accessible to the ATRA molecule during the sorption experiment. This could explain both the K_{OC} differences between the fractions > 2 µm and the < 2 µm and the differences between the coarse (0.2-2.0 µm) and fine (0-0.2 µm) clay fractions. Effectively, we have shown for the clayey samples Fi6 and Fo8 (Feller et al., 1992), that specific surface areas hindered to N_2 sorption in these soils were mainly associated to organoclay microaggregates and that this phenomenon was much higher for the fine clay than for the coarse-clay.

In terms of functionality, the fine-silt fraction (f2-20) represented the functional pool for the sandy sample Fl1-Sa (FI % = 41.9) as compared to the clay fraction for the brazilian Oxisol Fo8-F. The clayey soil Fi6 with an intermediate sorption capacity was characterized by two functional pools, the fine-silt and the clay fractions.

IV. Conclusions

1. It is always interesting to discover that some so-called " recent " approaches were already considered one century ago. Why were not they broadcasted ? This is an other subject for the science historians .

2. The particle size fractionation of SOM is *a priori* an interesting approach to identify " organic functional pools " because some large groups of fractions differ strongly by their

origin, composition and dynamics. On this basis, and for tropical LAC soils, a distinction in three main fractions was proposed: a " plant debris compartment " (fraction 20-2000 or 50-2000 µm), an " organo-silt complex " " (fraction 2-20 or 2-50 µm) and an " organo-clay compartment " (fraction 0-2 µm).

3. These three pools were tested for their " functionality ". The notion of functionality has been defined and quantified. We proposed to separate the notions of " descriptor value (DV) ", that refer to the property of the fraction, from that of " functionality (FI index) " which quantifies the distribution of this property within the different soil fractions. Based on the FI index, a " functionality scale " was proposed. This approach was applied to sandy and clayey LAC soil and for three types of functions: a " biological " function (C mineralization, a " mineral exchange " function (CEC) and an " organic sorption " function (Atrazine adsorption). The results showed that:

- the only descriptor value is insufficient to evaluate the role played by a given organic compartment for a given soil property, for the distribution of the property within the soil fractions must also be considered,
- the same fraction did not exhibit the same level of functionality according to the soil characteristics,
- the same fraction, for a given soil, did not exhibit the same level of functionality according to the type of the studied function,
- the " plant debris compartment " is an important functional pool for a biological function as short-term C mineralization in the sandy soils but not in the clayey ones; the " organo-silt complex " is a functional pool mainly for sorption process and particularly for sandy soils, but not for the clayey oxisols; the " organo-clay compartment " is a functional pool for all the studied functions in clayey soil and also for the exchange function in sandy soils.

This approach must be extended to others SOM functions and environmental conditions.

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