N₂-BET SPECIFIC SURFACE AREAS OF SOME LOW ACTIVITY CLAY SOILS AND THEIR RELATIONSHIPS WITH SECONDARY CONSTITUENTS AND ORGANIC MATTER CONTENTS

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Size fractions (2000–20 μm, 20–2 μm, 2–0.2 μm, and 0.2–0 μm) were obtained after Na-resin treatment of five top horizons from low-activity clay soils. Their specific surface areas (SSA) were measured by the N₂-BET method. The same method was applied after organic matter removal by H_2O_2 treatment to two of the bulk soils and their size fractions. The weighted sum of the SSA of the fractions fitted well to the SSA of the bulk soils, indicating that the association of particles, destroyed by the fractionation procedure had no effect on the SSA. The SSA of the coarsest fractions were greater than theoretical values from a sphere model, because of the presence of microaggregates. A good relationship was found between the SSA of the soils and subfractions and their total contents in R_2O_3 ($R_2O_3 = Al_2O_3 + Fe_2O_3$), i.e., an estimation of the importance of the secondary minerals present. Organic matter removal from two of the soil samples and their subfractions resulted in systematical but weak increase in SSA (≤ 8 m²·g⁻¹). This increase was not correlated to the amount of C removed from the samples. It was mainly due to changes in SSA occurring in the clay fractions where amorphous organic matter was assumed to act as a glue binding some clay plates together.

The specific surface area (SSA) developed by its colloids is a basic soil property that is related to many others: ion exchange capacity, reactivity during adsorption phenomena, organization of constituents in aggregates, porosity, and thus hydrodynamic characteristics, etc. The measurement of this property has received much attention in the past. More specifically, at-

tempts have been made to evaluate which constituents, or family of constituents, present in the soil play the major role in this property. Simultaneously, problems have also arisen concerning the methods to select in order to appreciate correctly the specific surface area of soils.

The objective of this paper is to evaluate the role of organic matter on SSA developed in the top, humus-rich horizons of Low Activity Clay (LAC) soils (i.e., soils with low CEC values of their colloids in their subsurface diagnostic horizon), varying by their contents in different particle size fractions. The mineralogy of these soils, as well as the nature of the question to be examined here, prompted us to rely, for this purpose, on SSA measured by the N₂-BET method for the reasons summarized below.

Due to their mineralogy, no clay interlamellar surface accessible to polar compounds is, in principle, present in LAC soils. Therefore, SSA measured by methods based on the physisorption of an inert gas (N₂, Ar, Kr) should theoretically be similar to those evaluated through the use of compounds that interact chemically at the interface, such as Ethylene glycol (EG) (Hendricks and Dyal 1950) or Ethylene glycol methyl ether (EGME) (Heilman et al. 1965). However, the theoretical prediction that both methods would be adequate to appreciate SSA of LAC soils was not verified in previous studies. Gallez et al. (1976), for instance, reported that EGME SSA of such soils could be two or three times higher than those measured by the N₂-BET method. Similarly, Schwertmann (1988) noted that some reported SSA of iron oxides evaluated from EGME measurements appeared to be unrealistically high. On the other hand, Chiou et al. (1990) pointed out that a polar compound like EG can partition into soil organic matter, hampering its use for measuring reliable SSA. This view is also supported by the data of Dejou (1989) who showed that the discrepancies in SSA measured on soil clays by EGME and BET methods depended not only on their mineralogy, but also on their organic matter content.

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In the present paper we will examine i) whether SSA determined by the N_2-BET method are reliable for the LAC soils under study, and ii) whether it is possible with this method to understand the effects of organic matter on SSA developed by soils.

MATERIALS AND METHODS

Soil Samples

They were collected in the top A horizon (0–10 or 0–20 cm) of seven tropical soils of different origins. The main physical and chemical properties of the samples as well as some characteristics of the soils from which they originate are given in Table 1.

Particle Size Determination

Particle size fractions were obtained by the method described by Feller et al. (1991a). After dispersion in water in the presence of a cationic Na resin (Amberlite IR 120), the 2–0-mm samples were sieved at 200 and 50 μm. The 50–0-μm suspension was sonicated, and sieved at 20 microns. A 20–2 μm (fine silt) fraction was separated by sedimentation from the 2–0-μm supernatant. The latter was centrifuged to obtain a 2–0.2 μm fraction (coarse clay) and a 0.2–0 μm (fine clay) fraction. The dispersion state obtained by this method, and for these samples, was virtually identical (Table 1) to that obtained by mechanical analysis, H_2O_2 treatment, followed by dispersion by Na hexametaphosphate and sonication. The fractions were then dried at 50°C until constant weight was reached. The fractions greater than 20 μm were gathered. The following four fractions were thus obtained: 2000–20 μ

Destruction of Organic Matter

The treatment was applied to each bulk soil and different size fractions. A quantity of 3 grams of sample was placed in contact overnight with 50 ml of H_2O_2 (50 vol) at 20°C and then at 50°C until dry. For the samples richest in organic matter, this operation was repeated. About 85% of the initial organic matter was destroyed by the H_2O_2 treatment.

Measurement of Specific Surface Areas

Nitrogen gas adsorption isotherms at liquid nitrogen temperature (77°K) were obtained with a laboratory-built volumetric apparatus equipped with a Datametrics 0–1000 Tor pressure gauge. The dried samples were mechanically ground in an agate mortar (Pulverisette Fritsch) for 10 minutes and degassed overnight at 20°C under a 0.03 Tor vacuum. Five to seven measurements of adsorbed gas volume were performed for relative pressures, P/P_o, ranging from 0.05 to 0.25. Specific surface areas were calculated after the linearized BET equation.

SSA measurements performed after different grinding times confirmed that grinding for 10 minutes does not create new surfaces. Degassing at 20° saved the organic matter from thermal evolution and provided results similar to those obtained after degassing at 60°C or 100°C for 12 hours. The variation coefficients ranged between 4 and 6% for 30 to 56 $m^2 \cdot g^{-1}$ area samples, and the standard deviations were 1.5 to 2.8 $m^2 \cdot g^{-1}$. For the nontreated soils samples, the reported SSA values are average results of triplicate measurements. Only one measurement was performed for the soil fractions and for the H_2O_2-treated soils and subfractions.

Other Analyses

Carbon and nitrogen contents of the samples were determined with a CHN Carlo Erba 1106 elemental analyzer. Total analyses (SiO_2, Al_2O_3, Fe_2O_3) of the soils and their size fractions were determined after fusion with Na metaborate after Jeanroy (1974). The mineralogy was assessed by X-ray diffraction performed either on powdered samples (coarse fractions) or on oriented films (clay fractions).

RESULTS

Specific Surface Areas of the Bulk Soils and Subfractions

Table 2 reports the values of SSA measured on the different types of materials collected for this study: bulk soil samples <2 mm, soil samples <2 mm after H_2O_2 treatment, subfractions, and H_2O_2-treated subfractions.

Whatever the soil and the treatment applied, the SSA values of the bulk samples are very close to those calculated from the SSA of their subfractions. The differences $\Delta SSAa = SSA_{measured} - SSA_{computed}$ vary only from −0.4 to +5 $m^2 \cdot g^{-1}$, and this agreement can still be improved (see $\Delta SSAb$) by considering that the small weight losses occurring during the fractionation procedure are totally due to losses in

TABLE 1

Origin and characteristics of the samples selected in this study

Sample	TNR101	V1	V3	T85	MSP41	MSP61	MPR51
Origin	Terres Neuves, Eastern Séné-gal	Booro-Borotou, Ivory Coast	Idem V1	Le Galion, Marti-nique	Piracicaba (S.P.), Brazil	Idem MSP41	Brazil, Ponta Grossa (P.A.)
FAO classification of the soil	Ferric Lixisol	Ferric Acrisol		Ferralic Cambisol	Rhodic Ferralsol		Rhodic (dystric) Ferralsol
Reference	Feller and Milleville (1977)	Fritsch et al. (1989)		Chevignard et al. (1987)	Cerri (1986)		
C %	0.9	1.7	1.0	2.2	4.3	1.5	4.5
C/N	15	17	14	12	10	13	19
2–0 μm % (*)	7.6 (8.2)	18.6 (nd)	20.7 (22.4)	49.3 (51.3)	56 (52)	58 (57.2)	65 (nd)
20–0 μm % (*)	17.4 (16.4)	28.4 (nd)	28.4 (28.2)	68.5 (67.5)	65 (58.8)	70 (66.9)	83 (nd)
SiO_2 g·100 g^{-1}	91.6	78.0	82.5	44.0	49.8	50.6	24.8
Al_2O_3 g·100 g^{-1}	3.2	8.8	8.7	21.7	17.4	20.9	28.7
Fe_2O_3 g·100 g^{-1}	1.1	3.9	2.2	14.0	11.6	12.9	17.8
R_2O_3 g·100 g^{-1}	4.3	12.7	10.9	35.7	29.1	33.8	46.5
Ki (2–0 μm)	2.56	nd	nd	2.28	1.62	nd	0.76
Mineralogy (2–0 μm)	K/Qz	K/Hm/Go	K/Hm/Go	K/H/Go	Hm/K/Go/Gi	Hm/K/Go/Gi	Gi/Hm/Go/K
Mineralogy (20–2 μm)	Qz/K/Il	nd	nd	Qz/Cr/H/Fe	nd	Qz/K/Hm/Gi	Qz/Gi/Hm/Go/K

Abbreviations: Qz = Quartz, Cr = Cristobalite, Fe = Feldspar, Il = Illite, K = Kaolinite, H = Halloysite, Go = Goethite, Hm = Hematite, Gi = Gibbsite, Ki = SiO_2/Al_2O_3 molar ratio, R_2O_3 = (Al_2O_3 + Fe_2O_3), nd = not determined.

(*) in parentheses, values corresponding to mechanical analysis; without parentheses values corresponding to the size fractionation method used in this study

TABLE 2

Carbon (C), R_2O_3 contents, and specific surface areas (SSA) of the soils and their size fractions, before (SSA1) and after (SSA2) H_2O_2 treatment

| Soil	Fraction (μm)	Before H_2O_2 treatment				After H_2O_2 treatment		Difference After − Before	
		C	R_2O_3	SSA1		SSA2		ΔSSAc = SSA2 − SSA1	
		g/100g	fraction	$m^2 \cdot g^{-1}$ fraction	$m^2 \cdot g^{-1}$ soil	$m^2 \cdot g^{-1}$ fraction	$m^2 \cdot g^{-1}$ soil	$m^2 \cdot g^{-1}$ fraction	$m^2 \cdot g^{-1}$ soil
TNR101	2000–20	0.42	1.5	0.9	0.8				
	20–2	3.5	9.2	6.9	0.7				
	2–0.2	4.33	30.8	24.0	1.1				
	0.2–0	1.81	36.5	47.8	1.4				
	Sum (1)	0.94	4.6		4.0				
	Bulk soil (2)	0.89	4.3		4.3		3.4		−0.9
	ΔSSAa = (2)–(1)				0.3				
	ΔSSAb[b]				0.3				
T85[a]	2000–20	1.3	32.4	8.3	2.0	6.8	1.6	−1.5	−0.4
	20–2	2.58	30.3	32.3	6.2	34.6	6.7	2.3	0.4
	2–0.2	2.47	39.1	41.7	13.1	53.3	16.8	11.8	3.7
	0.2–0	1.97	40.7	73.2	13.0	94.7	16.9	21.5	3.8
	Sum (1)	1.95	33.2		34.4		42.0		7.5
	Bulk soil (2)	2.18	35.7		39.4		46.2		6.8
	ΔSSAa = (2)–(1)				5.0		4.2		
	ΔSSAb[b]				3.6		2.1		
MSP41	2000–20	2.22	5.1	3.1	1.0				
	20–2	10.6	20.5	11.5	1.0				
	2–0.2	5.89	45.6	36.5	9.4				
	0.2–0	3.35	46.0	48.2	14.6				
	Sum (1)	4.15	29.1		26.0				
	Bulk soil (2)	4.25	29.1		25.6		30.0		4.4
	ΔSSAa = (2)–(1)				−0.4				
	ΔSSAb[b]				−0.4				
MSP61	2000–20	0.78		1.7	0.5	1.5	0.4	−0.2	−0.04
	20–2	2.98		32.0	4.0	35.6	4.6	3.6	0.6
	2–0.2	1.68		52.8	18.5	57.5	20.1	4.7	1.6
	0.2–0	1.33		62.7	14.2	69.8	15.8	7.1	1.6
	Sum (1)	1.48			37.2		40.9		2.6
	Bulk soil (2)	1.54	33.8		40.9		43.2		2.3
	ΔSSAa = (2)–(1)				3.7		2.3		
	ΔSSAb[b]				3.7		2.3		
MPR51[a]	2000–20	1.86	27.3	20.1	2.3				
	20–2	5.37	42.8	44.4	7.8				
	2–0.2	4.45	54.9	59.4	25.6				
	0.2–0	3.34	55.9	71.7	15.9				
	Sum (1)	3.81	46.7		51.6				
	Bulk soil (2)	4.09	46.5		54.7		62.3		7.6
	ΔSSAa = (2)–(1)				3.1				
	ΔSSAb[b]				1.8				
V1	Bulk soil	1.7	12.7		11.6		13.2		1.6
V3	Bulk soil	1.0	10.9		10.5		11.9		1.8

[a] Weight losses during fractionation of 2.7% and 2.1% respectively for T85 and MPR51.

[b] ΔSSAb is a modified ΔSSAa value when the losses occurring during the fractionation are assumed to be due to clay losses.

FIG. 1. Plot of the specific surface areas (SSA1) of the soils and their subfractions against R_2O_3 contents of the H_2O_2 untreated samples. Regression equation, with (a) or without (b) T85 sample and its subfractions:

(a) SSA $(m^2 \cdot g^{-1})$ = 1.21 R_2O_3 $(g \cdot 100\ g^{-1})$ − 5.49; (R = 0.89, P < 0.01)

(b) SSA $(m^2 \cdot g^{-1})$ = 1.16 R_2O_3 $(g \cdot 100\ g^{-1})$ − 4.47; (R = 0.96, P < 0.01)
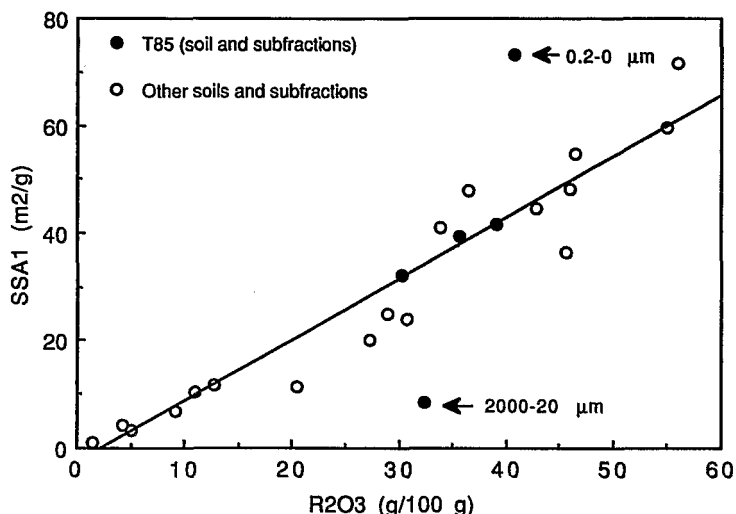
clay fractions. Then, ΔSSAb fall almost completely in the range of the standard deviation value due to the method (ΔSSAb = −0.4 to +3.8 $m^2 \cdot g^{-1}$).

The agreement between the SSA of the soils and those obtained from their particle size fractions, whether the samples were H_2O_2-treated or not, indicates that the association of particles destroyed during the fractionation procedure has no effect on the SSA developed. In other words, with the soils of this study and the fractionation procedure adopted, the BET-N_2 SSA measured on their fractions are additive.

Relationships between Specific Surface Areas and Secondary Constituents

In the collection of soils under study (Table 1), the regression equation between SSA and the clay content in each bulk sample is the following:

$$SSA\ (m^2 \cdot g^{-1}) = 0.77\ clay\ (g \cdot 100\ g^{-1}) - 3.64$$
$$(R = 0.93,\ P < 0.01)$$

This equation is remarkably similar to that proposed by Pope (1976) and is in good agreement with the regression equation that can be calculated from the data of Gallez et al. (1976). However, Table 2 shows that the sand and silt fractions of our samples develop SSA that cannot be neglected. In agreement with Bartoli et al. (1988), this indicates that these fractions are made not only of coarse particles but also of aggregates in which constituents developing high SSA are present. Therefore, an attempt

was made to take into account also the SSA due to the secondary constituents present in these fractions.

Figure 1 plots where the SSA measured on the H_2O_2-untreated materials of this study (soils and subfractions) are related to their R_2O_3 contents. With the exception of sample T85 and its subfractions (Fig. 1) all the selected samples originate from soils poor in weatherable aluminum- and iron-bearing primary minerals. Their R_2O_3 contents are thus related to the amounts and nature of their secondary constituents such as kaolinite, iron oxides and occasionally gibbsite (see Table 1).

We verified that the coarse fractions of the T85 sample are rich in amphiboles and pyroxenes, whereas its fine clay fraction contains, in addition to crystalline iron oxides, some ferrihydrite (François 1988). Thus, in this sample, R_2O_3 does not reflect exclusively the crystalline secondary constituents present.

Figure 1 can thus be interpreted as showing that the secondary constituents present in most of the samples, whether located in the clay fractions or not, develop SSA proportional to their R_2O_3 content.

Effect of Organic Matter Removal on SSA

For the bulk samples (<2 mm) the SSA measured after destruction of organic matter are systematically higher than these measured before H_2O_2 treatment, except for sample TNR 101 (Table 2). However ΔSSAc (SSA after H_2O_2 − SSA before H_2O_2) is poorly correlated

($r = 0.67$, $P > 0.05$) to the amount of C lost during the H_2O_2 treatment.

When the effect of organic matter removal on subfractions is examined (Table 2), it appears that the increase in SSA observed for the H_2O_2-treated samples is mainly due to changes in SSA occurring in their coarse and fine clay fractions. Only small increases are observed for the fine silt, and no change was found for the 2000–20 μm fraction. In fact, the two clay fractions accounted for more than 85% of the change in SSA.

DISCUSSION AND CONCLUSIONS

The additivity of SSA measured on subfractions that is observed here, although expected, is a result that points out the suitability of the N_2-BET method not only to determine the areas developed by the soils but also to interpret the changes due to organic matter removal. In this last respect, it must be recalled that conflicting opinions have already been reported concerning i) the magnitude of the surface area to be attributed to soil organic matter (SOM) and ii) the interpretation of changes in SSA associated with the destruction of SOM by H_2O_2. Chiou et al. (1990) have clearly pointed out that the BET-N_2 SSA of SOM is low. Therefore, its contribution to the total SSA of soils should be negligible. On the other hand, using a BET-N_2 method, Sequi and Aringhieri (1977), Aringhieri and Sequi (1979), and Turchenek and Oades (1979) reported increases in SSA after SOM removal that were substantial ($4 < \Delta SSAc < 200$ m$^2 \cdot$g^{-1}) but very scattered. It should be noted that in these reports high activity clay soils (i.e., soils containing 2:1 clay minerals) were used. Recently, Van Damme and Ben Ohoud (1990) showed that, contrary to kaolinite, the BET-N_2 external surface areas of smectites were strongly dependent on their mode of preparation. Thus, it is doubtful that the BET-N_2 method selected to show the effect of organic matter removal on soils containing 2:1 clay types can give reliable results. Such minerals are not present here (Table 1), and the effect of organic matter removal was found to be low ($\Delta SSAc \leq 8$ m$^2 \cdot$g^{-1} and $\leq$ 15% of the bulk soil SSA$_2$) and restricted mainly to an increase in SSA in the clay fractions. We believe that such an effect can possibly be explained by considering the different nature of organic matter located in the different fractions of our samples. As shown elsewhere by Feller et

al. (1991b) for the subfractions of soil T85, the organic matter associated with the coarse silt + sand and the fine silt fractions is mainly made of fungal and plant debris, whereas organic matter in the clay is more amorphous. From the electron microscope observation of these last fractions, it seems also that organic matter in the clay can act as a glue, binding clay plates together. A removal of organic matter in the clay fraction may thus recover SSA available to N_2 by a process of microdisaggregation. Thus, in this study, the "organic matter effect" on SSA seems to be better understood in the clay fractions, organic matter hides a part of available SSA by binding some clay particles together.

Concerning the relationships between R_2O_3 and SSA shown in Figure 1, two further comments can still be made. This relationship suggests that the richer in sesquioxides the secondary constituents, the higher their SSA. As a matter of fact, this suggestion seems to be justified by the data available for the kaolinite-iron oxide pair. In LAC soils, kaolinite develops BET-N_2 SSA that are generally smaller (30 to 60 m$^2 \cdot$g^{-1}) (Herbillon and Rodrique 1979) than those of the crystalline free iron oxide (50–300 m$^2 \cdot$g^{-1}) (Gallez et al. 1976; Bigham et al. 1978; and Borgaard 1982). However, in this relationship, the role of fine-grained gibbsite is unclear because data concerning this mineral are still lacking. Finally, it is clear that more data are needed to confirm the validity of this relationship. If verified, it would be possible to get a rough, but nonetheless useful, estimation of the SSA of LAC soils, not only from their clay contents (Pope 1976), but also from their chemical composition (i.e., their R_2O_3 content).

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