Int. Agrophysics, 2004, 18, 83-90

INTERNATIONAL
Agrophysics
www.ipan.lublin.pl/int-agrophysics

Characteristics of the specific surface area of Vertisols from the Gezira region in Sudan

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Received July 9, 2003; accepted November 13, 2003

Abstract. In this work the investigations of water vapour adsorption and calculation the specific surface area of soil samples of Sudanese Vertisols in North (NG), Central (CG) and South (SG) Gezira are presented. The Brunauer-Emmet and Teller (BET) equation was used to analyze experimental adsorption data of water vapour and to calculate the specific surface area (S_{BET}) from both the adsorption and the desorption branches of the adsorption isotherm. The values of the S_{BET} estimated from the adsorption isotherms ranged from 106 to 120, from 141 to 155 and from 165 to 187 $m^2 g^{-1}$ for the soil from North, Central and South Gezira, respectively. If the desorption isotherms were used, the ranges of S_{BET} were 117-140, 158-183 and 179-206 $m^2 g^{-1}$, respectively. Linear regression plot for the electric conductivity (EC), soluble Na^+ ions and the difference ΔS=S(des)-S(ads) was found. The specific surface area slightly increased with the depth of the soil profile. The specific surface area of Vertisols from Sudan increased from the north to the south and was related to the water regime and the intensity of cracking. The correlation between selected chemical and physical characteristics of soils and the values of the specific surface area was found.

Keywords: water vapour adsorption, specific surface area, Vertisol, Sudan

INTRODUCTION

Studies of the surface chemistry of solids frequently include determinations of surface area. Many investigators have attempted to measure surface area as a means of describing better the soil materials under study or understanding better a particular process or reaction.

Surface area is significantly related to various chemical and physical properties of soils [3-5,9,15,17,18,23-26,29]. The method of determining the surface area of textured solids, including soil materials, is based on the adsorption of any of a number of gases or vapours, and on the application of the Brunauer-Emmet and Teller (BET) equation [10,14].

Adsorption of gases or vapours and surface area depend on the nature of the soil solids. Various kinds of polar and non-polar functional groups have been identified in mineral and organic soil constituents. The most important are the carboxylic-, phenolic-, hydroxylic-groups. These groups create primarily hydrogen bonding or van der Waals forces or -bonding. Several polar functional groups serve as sorption sites for water molecules. Because of a high dipole moment and the ability to form hydrogen bonds, water can form quite complex adsorbed layers that can exhibit different properties depending on the kind of surface functional groups binding water molecules. The hydration or coordination reactions between surface cations and water molecules also influence adsorption of water vapour.

Soil colloids are primarily of permanent charge type in temperate regions, but the tropical soils consist predominantly of variable charge colloids. This is one of the reasons why the transfer of temperate region experience and technology to the tropics may succeed in some cases and fail in others [28]. Vertisols have a distinct morphology, exhibiting wide and deep cracks in dry season, wedge-shaped structural aggregates with polished faces, slickensides, and other features caused by the swelling and shrinking of smectite clays, and resulting in movements in the soil due to shear failure [2]. In highly weathered soils of the tropics, the surface charge arises from the adsorption of potential determining ions. The most important are hydrogen and hydroxyl ions.

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Hematite is a typical example of a constant surface potential, variable charge, pH-dependent charge mineral. Organic matter, non-crystalline materials, oxides and hydrous oxides, slightly soluble carbonates and sulphates can be treated as variable charge soil constituents. Stones and gravel are common on the surface of Vertisols [11,21]. This feature has been explained as originating from the pedoturbation of the soil mass. In this process two incompletely balanced movements of the soil mass take place: an upward movement with the soil swelling, and a downward one when the soil shrinks. Blokhuis [2] reported that on the degraded clay plains of central Sudan stones were often found on the surface of stone-free Vertisols at sites well away from rocky inselbergs. In the Central Clay Plain he found an upward coarsening of the soil texture in a number of profiles. In some of those soils silt content remains the same irrespective of the pedoturbation depth.

Properties of tropical soils result from several processes, mainly from weathering and soil formation. One consequence of rock weathering and soil formation is the synthesis of extremely fine grain clay minerals at the expense of coarser grained rock-forming minerals. This change causes the specific surface area (per unit mass) of soil to increase markedly with weathering [28]. In tropical soils, seasonal changes in specific surface area range from 9 to 28%. This means that in Vertisol with 50% clay and a moist specific surface area of 300 m^2 g^{-1}, a change in specific surface of 30 m^2 g^{-1} between wet and dry season is possible [28].

The objective of this work was to determine the specific surface area of selected soil samples of Vertisols and to verify the existence of correlations between the chemical and physical characteristics of the soils and the values of the specific surface area.

MATERIALS AND METHODS

The experiments were carried out for the Sudan Gezira Vertisols. The selected sites represent South, North and Central Gezira. Central Gezira is represented by a profile from the farm of Gezira Research Station at Wad Medani, referred to as GRS. This profile was taken from irrigated and cropped plots. One plot at that farm, referred to as PF, was left permanently fallow representing the virgin land, so that it could be used as a control as changes in properties of soils resulting from long-term cropping were monitored. The investigated soil samples were taken at the end of the dry season. The soil samples were air-dried and sieved through a screen of the 2 mm mesh. Chosen characteristics of the studied sites and the soils are given in Tables 1 and 2.

The adsorption-desorption isotherms of water vapour were measured by gravimetric method. Before the adsorption measurement, the soil samples were dried in a vacuum chamber with concentrated sulphuric acid until the weight of the samples reached constant values. Soil sample of a weight of approximately 3 g per sample were put into a glass vessel and placed over sulphuric acid solution. The samples were equilibrated with water vapour during two days. The amount of adsorbed water vapour was computed as the difference between the weight of the sample with water and the dry sample (dried in an oven at 105 C). The relative water pressures were obtained from the density of sulphuric acid solutions. Twenty levels of relative pressure were selected in the range from 0.05 to 0.97. The adsorption measurements were replicated three times keeping the temperature constant at T=20°C±0.5. Twenty levels of relative pressure were selected in the range from 0.015 to 0.95. The variation in replicated data did not exceed ±5% at the lowest vapour pressure and ±1% at the highest vapour pressure. The averaged values were used to obtain the specific surface areas and to prepare the figures.

The surface area of soil samples was evaluated from adsorption-desorption isotherms in the BET range of relative water vapour pressure, using the Brunauer-Emmett-Teller (BET) method [10,14,22]. The first step in the application of the BET method is to obtain the monolayer capacity (N_m) from the BET plot. The second step is to calculate the surface area S from the dependence $S = N_m M^{-1} L \omega$, where L is the Avogadro number ($6.02\ 10^{23}$ molecules per mole), M is the molecular weight of water (gram per mole) and ω is the molecule cross-sectional area ($10.8\ 10^{-20}$ m^2 for water molecule). If N_m is expressed in grams of adsorbate (water) per gram of solid, the specific surface area S (m^2g^{-1}) is estimated from the monolayer capacity as $S=3612\ N_m$. This procedure agrees with the Polish Standard PN-Z-19010-1 for measuring the surface area of soil [16].

Table 1. Description of the studied sites

Specification / Location	North	Central	South
Name of soil profile	NG	GRS	SG
Parent material	Blue Nile alluvial	Blue Nile alluvial	Blue Nile alluvial
Rainfall annual (mm)	292	354	430
Temperature annual (°C)	36.2	36.7	36.7
Physiography	plain	plain	plain
Slope	< 1%	< 1%	1%
Last crop	fallow	wheat	fallow

Table 2. Chosen properties of the investigated soil profiles

Depth (cm)	pH (H_2O)	CEC (mM kg^{-1})	EC (S cm^{-1})	Sand	Silt	Clay	$C_{org.}$	C_{total}	N_{total}
					(%)			(g kg^{-1})	
North Gezira, NG									
0-25	8.4	434	243	29	32	40	4.79	10.79	0.30
25-45	8.9	463	407	28	30	42	4.01	9.40	0.20
45-70	9.0	429	700	27	31	43	2.46	6.60	0.19
70-90	8.7	518	943	20	33	47	5.31	8.85	0.18
90-110	8.6	525	924	19	32	49	5.54	9.04	0.18
110-150	8.8	504	914	16	39	45	3.99	11.15	0.14
Central Gezira, GRS									
0-10	7.4	594	325	18	30	52	6.35	11.64	0.34
10-35	8.4	626	275	16	29	55	5.44	10.59	0.23
35-65	8.9	618	490	15	29	56	4.79	9.84	0.19
65-85	8.7	621	731	15	30	55	5.96	10.47	0.18
85-115	8.3	682	1371	13	29	58	6.30	10.74	0.22
115-150	7.6	679	3605	12	30	58	6.43	11.77	0.22
South Gezira, SG									
0-3	7.4	691	651	11	32	57	7.50	13.22	0.43
3-35	8.5	631	406	16	26	58	4.99	15.74	0.26
35-88	8.6	720	597	13	28	59	6.00	13.79	0.24
88-100	7.9	804	1893	8	29	63	8.81	12.21	0.22
110-150	7.7	784	1895	5	30	65	8.83	13.08	0.24
Central Gezira, permanent fallow, PF									
0-7	8.3	669	301	17	31	52	4.00	9.99	0.25
7-35	8.5	658	345	14	29	57	4.61	10.39	0.22
35-65	8.8	601	433	13	30	57	4.67	10.77	0.19
65-90	8.7	585	676	10	30	59	5.77	10.46	0.21
90-120	8.1	678	2475	8	29	61	6.34	10.84	0.29
120-150	7.9	671	3970	8	31	61	6.05	11.30	0.18

Abbreviation: CEC – cation exchange capacity; CE – electrical conductivity.

For determination of cation exchange capacity (CEC), soluble salts were removed by means of 95% ethanol. The exchangeable cations were extracted using 1M NH_4Ac at pH 7. The ammonium exchanged was than extracted by 1M KCl at pH 7 after washing off excess ammonium with 95% ethanol. Total carbon and nitrogen contents were measured with the Carlo Erba CNS Analyser. The pH and EC were measured at 1:5 soil to water ratio.

RESULTS AND DISCUSSION

A number of Vertisols have uniform percentages of sand, silt and clay throughout the profile. The most striking aspects of the granulometric composition of the clay plain Vertisols is the great uniformity. The variation in sand, silt and clay separates within any one profile is small, and often negligible, and between Vertisols from different sites the granulometric composition of the soil is very similar [2].

Figure 1a shows an example of experimental adsorption isotherms obtained for upper layer of soil profiles from North (NG), Central (CG, GRS) and South (SG) Gezira and the hysteresis loop, i.e., adsorption-desorption isotherm, for soil sample from the SG profile. In general, the shapes of the adsorption and desorption curves are similar. According to the BET classification of the adsorption isotherms [10,22], all curves belong to the same class, namely to the type II. However, the detailed course of the curves, and the amount of adsorbed or desorbed water in particular, vary from sample to sample, which is obviously related to the different basic properties of particular samples.

The experimental data have been described using the BET equation. Figure 1b shows an example of applying the transformed BET equation or the so-called BET plot to the adsorption data shown in Figs 1a and 1b. The values of the correlation coefficient, R^2, indicate that the BET equation provides a good fit to the experimental data. In all cases the values of R^2 varied from 0.996 to 0.998 if the calculations were performed at the relative pressures below $p\,p_0^{-1} \approx 0.35$. Generally, better fits have been obtained for experimental desorption than for adsorption data.

The qualitative interpretation of the isotherms provides information about the structure of the investigated soil samples. As we have already noted, all the isotherms belong to the second type of the BET classification scheme, which indicates a non-microporous nature of these adsorbents. Example of the hysteresis loop for an upper layer sample from profile SG is given in Fig. 1a. The hysteresis loop corresponds to the type H3 according to the IUPAC classification [22] and is usually associated with capillary condensation in a mesoporous structure. The rather narrow hysteresis loops confirm this hypothesis.

The adsorption-desorption data have been used to evaluate the values of the specific surface area. Figure 2 shows that the specific surface area increases slightly with depth. Small variations in surface area suggest relative similarity between horizons at the same site. For all the samples studied, the BET specific surface area estimated from the adsorption isotherms ranged from 106 to 120, from 141 to 155 and from 165 to 187 $m^2 g^{-1}$ for the soil from North, Central and South Gezira, respectively. If the desorption isotherms were used, the BET specific surface area was 117-140, 158-183 and 179-206 $m^2 g^{-1}$, respectively.

In the case of the samples taken from the permanent fallow plot (PF), the values of the surface area varied from 145 to 159 and from 162 to 189 $m^2 g^{-1}$ for adsorption and desorption. Note that the profiles from the permanent fallow plot (PF) and from the profile CG (GRS) are situated in Central Gezira (farm of Gezira Research Station at Wad Medani). These results show that the agricultural use of soil does not influence significantly the values of the specific surface area (see Fig. 2). The differences in the specific surface area of samples from profiles PF and CG (GRS) may be a result of cultivation and the water regime (the GRS plot was irrigated). The agricultural use, i.e., mechanical tillage, organic or mineral farming and irrigation, must have an influence on the condition and properties of soil. Salah *et al.* [20] showed the effect of tillage systems on some physical properties of Vertisols, root growth and yield of cotton. Also, under warm and humid conditions, when the soil is cultivated, the organic matter content is difficult to determine [28]. Mechanical tillage leads to more rapid decline in organic matter content, suggesting more severe soil degradation [19]. The specific surface area and surface charge density in tropical soils dominated by permanent charge minerals are managed by manipulation in the concentration and composition of the soil solution. In soils dominated by variable charge minerals, farming practices often unintentionally alter the surface charge density [12,28]. Figure 3a

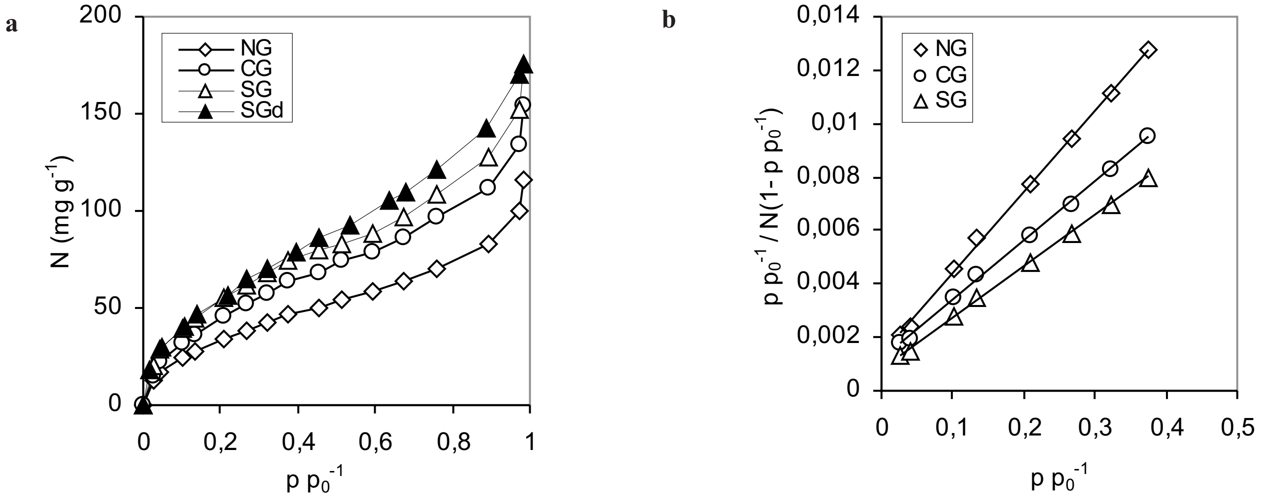


Fig. 1. Examples of water vapour adsorption data: original adsorption isotherms (a) and the BET plot for investigated soil samples (b). Abbreviation: NG, CG, SG – North, Central and South Gezira, N – amount of adsorbed water, $p p_0^{-1}$ – relative pressure, white symbols – adsorption, black symbols – desorption.

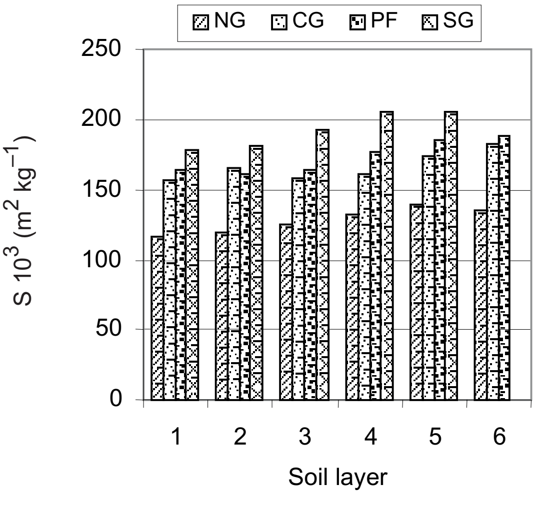


Fig. 2. Specific surface area (S) of different soil horizons for investigated soil samples. The labels along the x-axis correspond to the soil depths from Table 2. Abbreviation: see Fig. 1, PF – profile situated in Central Gezira, permanent fallow.

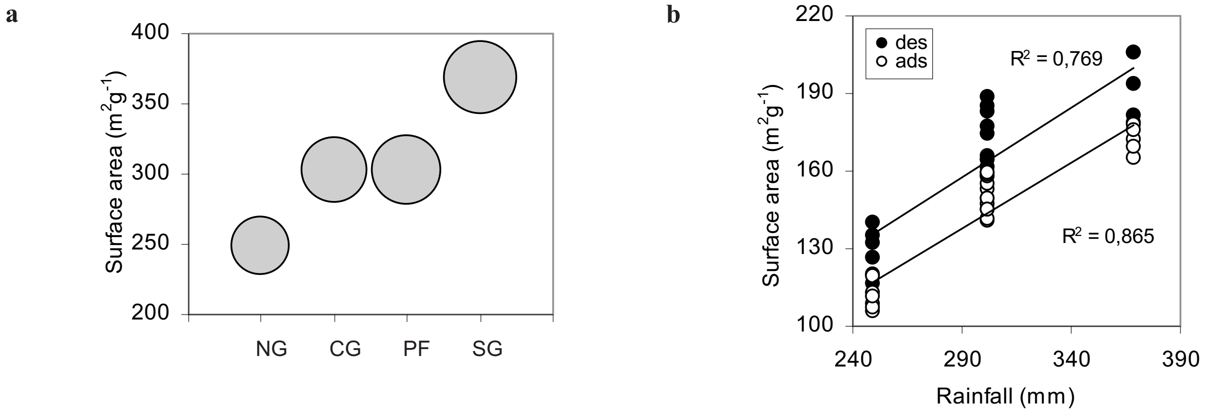


Fig. 3. Specific surface area of Vertisols versus soil profile location (a) and mean rainfall recorded at each site (b). Abbreviation: see Figs 1 and 2.

shows that the value of the specific surface area of Sudanese Vertisols increases from the north to the south. The data representing mean rainfall recorded at each site (Table 1) and the analysis of the specific surface area for the four profiles investigated, show a clear trend in the result – an increase in the specific surface area with increasing rainfall (Fig. 3b).

In general, the values of the surface area obtained from desorption data are higher than those obtained from adsorption data. Similar results have been found by Alekseev *et al.* [1] for latosol, the red and yellow soils from China. This difference is due to adsorption hysteresis [10,14]. There exist several evidences which suggest some criteria for testing the correctness of the monolayer capacity evaluation from the BET equation. In particular, the BET linear equation should lead to reasonable estimation of the monolayer capacity if its prediction is close to the position of the inflection point of the isotherm. In several cases desorption data may better satisfy the last requirement [14]. We have found the linear correlation between the adsorption- and desorption-surface areas (Fig. 4), but the correlation coefficient R^2 was rather low 0.64. The difference between the values of the specific surface area estimated from adsorption and desorption data may be related to the content of soluble salts in the soils [6]. The experimental data suggest the existence of a lineal regression between the electric conductivity (EC), soluble Na^+ ions and the difference ΔS=S(des)–S(ads), with the correlation coefficients close to 0.6 (Fig. 5).

We have investigated the correlations between the chemical and physical characteristics of the soils, and the values of the specific surface area. Figure 6 exhibits the linear regression plots of the specific surface area (S) vs. the percentage content of clay (part a) and of sand fractions (part b). In the case of clay fraction the correlation coefficient R^2 was as high as 0.94 and 0.85 for the desorption- and adsorption-surface area, respectively. It is generally known that there is a good correlation between surface area and the content of the clay fraction [15,25,26]. In our case, a reasonably good correlation has been also found for the sand

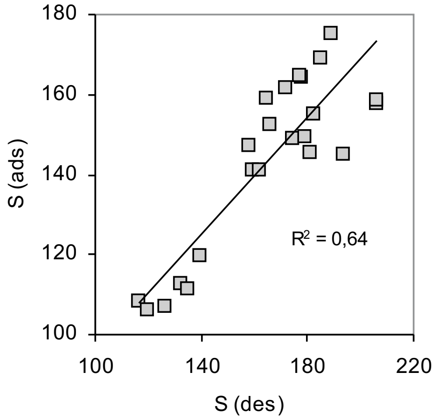


Fig. 4. Relationship between specific surface area (S) estimated from adsorption and desorption data of water vapour.

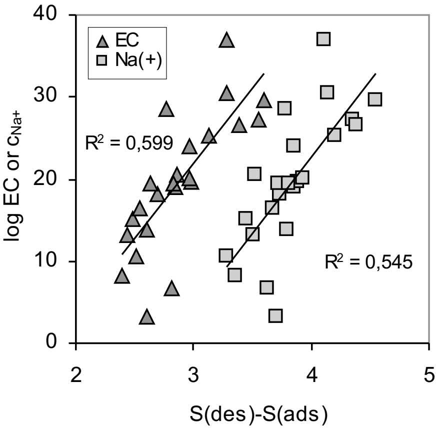
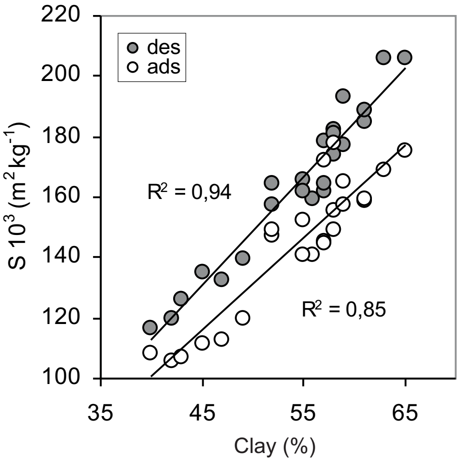


Fig. 5. Electrical conductivity (EC) or concentration of Na+ versus the difference between values of the specific surface area (S) of investigated soil samples estimated from adsorption and desorption data of water vapour.

a



b

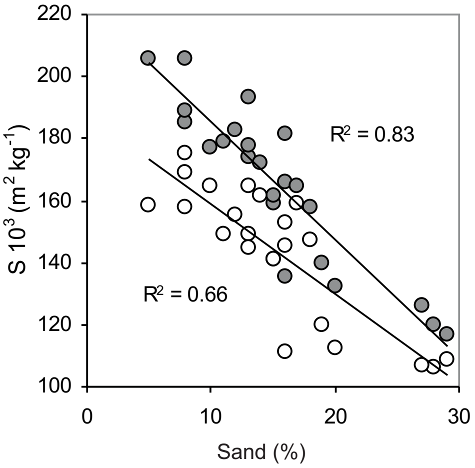


Fig. 6. The correlation between the specific surface area (S) and the percent of clay (a) and sand fraction (b) for investigated soil samples. Abbreviation: see Fig. 1.

fraction. The correlation coefficients R^2 were 0.83 for desorption-surface area and 0.66 for adsorption-surface area. No correlations exist for the silt fraction. Data in Table 2 show that the silt fraction content varies within very narrow range. No correlation can be found with a value that does not change much. Because the silt content does not change much, the sand content is well correlated with the clay content. The above facts should be interpreted taking into account the mineralogical, chemical and granulometric properties of the Sudanese Vertisols.

In tropical soils with variable charge clays, cation exchange capacity (CEC) is the product of the specific surface area and the surface charge density. We have also verified the existence of a correlation between the specific surface area and the cation exchange capacity (CEC), see Fig. 7. In this case we found that the correlation coefficient was quite high (R^2=0.91 and R^2=0.80 for the desorption- and adsorption-surface area, respectively). Torres Sanchez and Falasca [27] showed a similar good correlation between the specific surface area and CEC of untreated Argentinian soils. Several authors [5,17,18,23,29] reported the existence of a good correlation between CEC and the surface area of mineral soils. Blokhuis [2] suggested that in soils with smectite clay mineralogy, like in the Central Clay Plain, the CEC of the clay fraction should exhibit only a small variation and that the lack of the variations may be the result of properties of the clay fraction other than its mineralogy. In order to understand the variation in CEC-clay between sites, their sedimentation history, mineral weathering and soil formation must be known. However, even if not fully understood, the variation in CEC-clay may contribute to the defining of regional units within Central Plain Clay.

No correlation between the content of organic matter and the value of the specific surface area has been found for the investigated Vertisols from Gezira. It should be noted that Blokhuis [2] found no difference in the organic matter content between horizons A and B in several Vertisols. The horizon BC had a lower organic matter amount, and the horizon C – the lowest. Such organic matter profiles suggest that pedoturbation has homogenized the solum. Additionaly, the organic matter in Vertisols occurs bound to the large surface area of smectite clays, and small amounts of organic matter have strongly pigmenting effects. The results reported in the literature concerning the relation between the surface area and the amount of soil organic matter do not allow for any unequivocal conclusion. According to Feller *et al.* [9], changes in the surface area are not correlated with the amount of organic carbon removed from samples. They observed that partial removal of organic carbon by means of

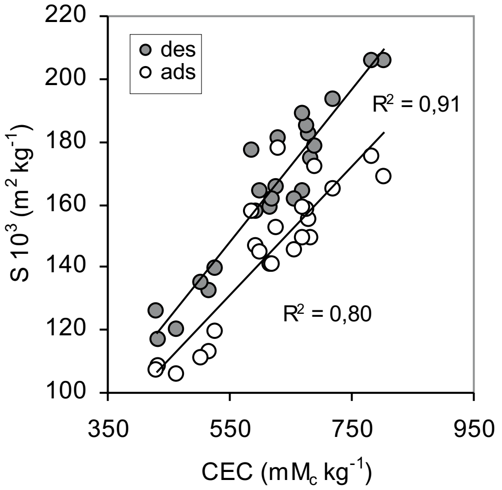


Fig. 7. The correlation between the specific surface area (S) and the cation exchange capacity (CEC) for investigated soil samples. Abbreviation: see Fig. 1.

treatments with H_2O_2 increased the surface area of clay-sized organo-mineral complexes, while the surface area of silt and sand was less affected. Theng *et al*. [26] reported that the surface area evaluated for initial samples, as well as for samples from which organic matter was removed, was identical. Torres Sanchez and Falasca [27] reported that the specific surface area of Argentinian soils, determined by means of ethylene glycol and water adsorption, obtained for soils agreed with the initial carbon content. Sokołowska *et al*. [25] found that the surface area increased after organic matter removal from the clay fraction of loess soils. Yijie and Chaoliang [30] reported similar results for black soil, cultivated yellow loess soil and yellow brown earth, rich in clay minerals at a ratio of 2:1. Contrary to that [30], the specific surface area of colloids of red earth and latosol decreased after the removal of organic matter. They concluded that the effect of organic matter on specific surface is related not only to the organic matter content but also to the composition of clay minerals in soil. Results reported by Elias *et al*. [8] confirmed the last conclusion. They found that humified organics in Vertisols were positively correlated with the clay content.

The relationships between the surface area and the properties of Vertisols are complicated. The chemistry, fertility and physics of Vertisols, and therefore their management, are altered by their mineralogy. If a significant proportion of tropical soils differs from soils of temperate regions, one should look for the difference in the specific surface of clays. Swelling, cracking and crusting of soils are also related to specific surface and charge density. Kaszubkiewicz *et al*. [13] stressed that the clay content, specific surface and the kind of parent rock are the parameters influencing the swelling index. They found a linear correlation between the total surface area of soils and the swelling index (R = 0.886). Our results also indicate the existence of a relationship between the specific surface area of Sudanese Vertisols and the cracking process. We have found that the value of the specific surface of Sudanese Vertisols increases from the north to the south. In our previous paper [7] we showed that the intensity of cracking increased from the north to the south and the cracking intensity in the Gezira Vertisols was associated with the soil clay content and the water regime. The difference between the adsorption and desorption results can also be attributed to some irreversible changes occurring in the soil samples during adsorption (e.g., swelling or hydration of cations).

CONCLUSIONS

1. The shapes of the experimental adsorption isotherms for Vertisols from the Sudanese Gezira are similar, but the amount of adsorbed or desorbed water varies from sample to sample. This is obviously connected with the different basic properties of particular samples. The values of the correlation coefficient, R^2, indicate that the BET equation provides a good fit to the experimental data.

2. The BET surface areas for the studied Vertisols locate in high ranges of surface areas of mineral soils. This may be caused by the accumulation of highly weathered poorly crystalline or amorphous material in these soils. The specific surface area slightly increases with the depth of the soil profile. Small changes of surface area suggest similarity between horizons at the same site. The values of the specific surface area obtained from adsorption and desorption data are different. Specific surface area evaluated from desorption data is greater. The values of the surface area differ significantly between the soils studied, with a clear trend of increasing their values in the soil profiles from North (NG), Central (CG) and South (SG) Gezira. We have found and increase in the value of the specific surface area with increasing rainfall from North to South in Gezira.

3. A linear relationship between the surface area and the content of the granulometric fractions has been found. In the case of the clay fraction, the correlation coefficient, R^2, was very high. Similarly, a linear relationship has been found for the sand fraction. A strong correlation (with the correlation coefficient $R^2 = 0.91$ and $R^2 = 0.80$) exists between the surface area and the cation exchange capacity, but no correlation between the content of organic matter and the value of specific surface area has been found.

ACKNOWLEDGMENTS

The Alexander von Humboldt Foundation partially sponsored this work as a Post-Doctorate Fellowship to E.A. Elias.

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