Agropedology, 1999, **9**, 47-53

Nature and relative abundance of smectite in clays of Vertisols derived from different parent materials

I.Y.L.N. Murthy[*], *S.C. Datta, T.G. Sastry*[**] *and R.K. Rattan*

Division of Soil Science and Agricultural Chemistry, IARI, New Delhi 110 012

Abstract

Qualitative and quantitative studies on clay minerals in the clay fraction (<2 μm) of Vertisols developed on different parent materials such as grey shale, basalt, granitic-granodiorite, and limestone, were made by differential thermal analysis (DTA), thermogravimetric analysis (TG) and x-ray diffraction (XRD) analysis. The results indicate the dominance of smectite in the clay fractions of Vertisols. However, the crystallinity of smectite varied. An improvement in the existing semi-quantitative estimate of smectite in presence of chlorite and vermiculite has been proposed.

Additional keywords: Smectite, Vertisols, differential thermal analysis, thermogravimetric analysis.

Introduction

It is a well known fact that clay of Vertisols is dominant in smectite, but there is very little work on the nature, i.e. crystallinity and/or true particle size (number of units per crystalloid which produce coherent X-ray scattering) of smectite derived from different parent materials. Width at half-maximum of a peak or its integral breadth is a characteristic of crystal strain and particle size distribution (Datta 1996). But during peak measurements of smectites a common problem is encountered. Usually the smectite peak of Vertisol clay is very large and in most of the cases masks and encompasses adjoining small peaks (say, 1.4 nm peak of chlorite or vermiculite). So it is not only very difficult to measure the width concurrently but also a problem for the estimation of smectite content correctly.

In the present investigation a method is adopted to avoid these two difficulties to study the nature of crystalloids from integral breadth and relative abundance of smectite in relation to other minerals, particularly, 1.4 nm minerals which otherwise get overlooked due to masking effect.

Materials and methods

Soil samples from five Vertisol pedons of different parent materials viz. grey shale (Pedon 1), basalt (Pedon 2), granitic-granodiorite (Pedon 3), granodiorite (Pedon 4) and limestone (Pedon 5) were collected from Suhagpur,

---

[*] Present address : Directorate of Oilseed Research, Rajendranagar, Hyderabad 500 030

[**] Deceased.

Dhana (Betul Distt., M.P.), CICR Farm (Nagpur Distt., Maharashtra), Baklavanvalli, Makthal and Undevalli (Mehboobnagar Distt., A.P.), respectively. The physical and chemical properties of the soils were presented elsewhere (Murthy *et al.* 1994).

Clay fraction (<2 μ) from these soil samples was separated by using the standard procedure (Jackson 1956). Calcium saturated clays were prepared for differential thermal analysis (DTA) and thermogravimetric analysis (TG). Total surface area of clay was determined by the method of Carter *et al.* (1965).

For X-ray diffraction analysis preferred oriented slides were prepared and scanned by CuKα radiation for the following treatments: 1) Mg saturated and air dried, 2) Mg saturated and glycerol solvated, 3) K saturated and air dried and 4) K saturated and heated to 550°C. Clay minerals were identified by analysing the diffractograms corresponding to all the above treatments.

Results and discussion

The DTA and TG curves of clay fraction of pedon 1 is presented in figure 1 and the peak temperatures of DTA of all clay samples are listed in table 1. The dominance of smectite in the clay fraction is clearly indicated by the low temperature endothermic peak at about 120°C with a distinct shoulder peak at 192°C in all the pedons studied which is due to loss of sorbed water varying from 42.6 to 62.0 per cent (Fig. 1). The broad dehydroxylation peak, the flatness of the curve and a broad weak endothermic-exothermic inversion system in the region of 850-910°C also indicate the presence of kaolinite and mica besides smectite.

The minerals present were identified as smectite, chlorite and interstratified chlorite, mica and kaolinite. Semi-quantitative analysis of the samples were done following the principles of Gjem (1967) i.e., measuring the area under a given peak over a suitably drawn background curve and applying some correction factor obtained by regression analysis of large number of samples. Correction factors are attributed to the angular variation of mainly Lorentz-Polarisation factor. Conventionally, the peak area is measured in diffractogram of magnesium saturated-glycerol solvated specimen. But in the present investigation the area was measured in the diffractogram of K saturated-heated to 550°C. This was done to avoid overlapping large smectite peak and any 1.4 nm peak. During heating to 550°C smectite peak collapses to 1.0 nm. Substracting the area of original mica peak area (in glycerol solvated specimen) from that of ignited sample, the contribution of smectite to 1.0 nm peak is obtained. As a result of shifting of smectite peak to 1.0 nm, 1.4 nm peak and the reflection in the region of 1.0 to 1.4 nm become well separated and measurement of their area become easier and reproducible and by principle these areas in the ignited samples correspond to chlorite and interstratified chlorite and/or hydroxy interlayered smectite, respectively. This mixed layer mineral cannot be mica-chlorite because in the glycerol solvated specimen there was no peak in between 1.0 and 1.4 nm. The area of the 1.4 nm peak in the ignited sample was always

Table 1. DTA, TGA and total specific surface area of clays (<2 µm)

Pedon No.	Horizon	Differential thermal peak maximum (°C)		% Moisture loss at peak maximum		
		Endothermic	Exothermic	I	II	III
1	Ap	120 (s), 192 (sh), 512 (m), 722 (m)	915 (m)	49.25	24.02	21.32
	A	120 (s), 204 (sh), 521 (m), 722 (m)	915 (m)	46.4	26.30	22.70
	AC	120 (s), 192 (sh), 495 (m), 722 (m)	900 (m)	50.00	24.30	17.50
	C	122 (s), 192 (sh), 522 (m), 730 (m)	915 (m)	51.20	23.07	21.56
2	Ap	133 (s), 205 (sh), 612 (m), 730 (m)	900 (m)	62.45	22.01	12.17
	A	120 (s), 192 (sh), 512 (m), 722 (m)	900 (m)	62.41	21.66	12.41
	AB	120 (s), 192 (sh), 512 (m), 755 (m)	900 (m)	42.66	18.90	33.72
	AC	133 (s), 192 (sh), 512 (m), 755 (m)	900 (m)	42.66	18.90	32.21
	C	120 (s), 215 (sh), 512 (m)	900 (m)	59.60	31.67	--
3	Ap	133 (s), 192 (sh), 512 (m), 722 (m)	900 (m)	53.47	21.43	18.51
	A	120 (s), 215 (sh), 512 (m), 739 (m)	900 (m)	47.51	17.93	30.90
	AC	120 (s), 192 (sh), 512 (m), 725 (m)	900 (m)	53.42	21.14	22.57
	C	120 (s), 192 (sh), 512 (m), 688 (m)	915 (m)	61.90	21.57	13.39
4	Ap	120 (s), 192 (sh), 512 (m), 722 (m)	988 (m)	53.72	26.22	15.03
	A	120 (s), 192 (sh), 512 (m), 688 (m)	900 (m)	58.43	25.72	15.38
	AC	120 (s), 192 (sh), 512 (m), 755 (m)	900 (m)	36.13	19.30	25.86
	C	105 (s), 192 (sh), 512 (m), 730 (m)	915 (m)	30.57	20.27	20.34
5	Ap	120 (s), 192 (sh), 602 (m), 722 (m)	900 (m)	58.73	19.97	16.50
	A	120 (s), 215 (sh), 438 (m), 739 (m)	900 (m)	53.50	21.99	20.65
	AC	120 (s), 192 (sh), 530 (m), 739 (m)	900 (m)	53.25	18.85	20.00
	C	120 (s), 192 (sh), 530 (m), 705 (m)	884 (m)	62.06	21.14	11.19

S = strong peak Sh = shoulder peak M = medium peak w = weak peak

found to be greater than that in glycerol solvated specimen (measured with eye estimation). This rules out the presence of any appreciable amount of vermiculite in the sample (Gjem 1967). The principle followed in the estimation of mica, chlorite, smectite and mixed layer minerals from peak areas in the diffractogram of ignited sample is based on two assumptions (Gjem 1967) viz., 1) the content of expanding minerals is in proportion to the contribution each makes to the 1.0 nm reflection in ignited sample. It is thus assumed that the intensity of the 1.0 nm reflection in the ignited sample is a linear additive function of those in the glycerol solvated sample and 2) it is assumed that the 1.4 nm chlorite reflection in ignited sample is directly comparable with the intensity of the mica reflection at 1.0 nm and that the same applies to interstratified chlorite.

Table 2 shows the semi-quantitative mineralogical composition of the clay fraction and integral breadth (peak area/peak intensity) of 1.0 nm peak in the ignited specimen. It is seen that smectite of pedon 4 having granodiorite as parent material has lowest value (0.84° 2θ) of integral breadth, thus having more

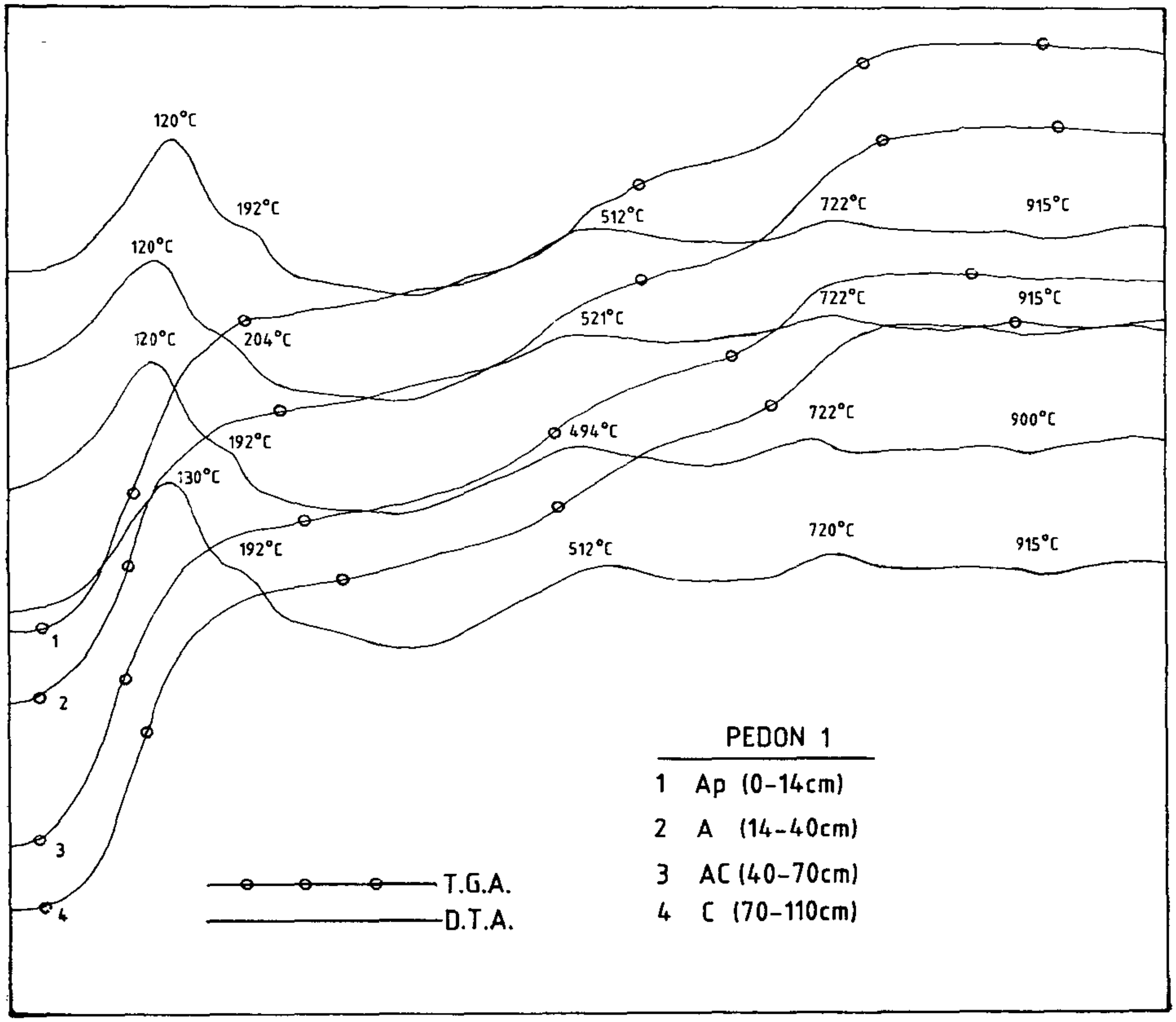

Fig. 1. Representative DTA and TGA curves of clay fractions (<2 μm) of Vertisols.

crystallinity and coarser particle size in terms of coherent x-ray scattering. This is also supported by the less specific surface area of all the clay samples of this pedon ranging from 455 to 530 m^2/g. On the other hand, smectite of pedons 1, 3 and 5 of parent materials gray shale, granite-granodiorite and limestone is less crystalline and finer particle size as indicated by the values of the integral breadth of 1.55, 1.55 and 1.47, respectively. These are also having more specific surface area than those of pedon 4.

Smectite content is maximum in pedon 5 having limestone as parent material ranging from 69 to 79 per cent and minimum in case of pedon 2 (40 to 62%) having basalt as parent material. Consequently, chlorites and interstratified chlorite (or hydroxy interlayered smectites) are minimum (18 to 28%) in case of pedon 5 and maximum (26 to 48%) in case of pedon 2. From this inverse relationship between abundance of smectite and chloritic minerals, it appears that there exists some sort of derivational relationship between these two minerals.

Table 2. Semi-quantitative analysis, specific surface area of clay minerals and integral breadth of 1.0 nm peak in ignited samples

Horizon	Parent material	Smectite	Chlorite & int. Chlorite	Mica	Kaolinite	Integral breadth (°2θ)	Specific surface area (m^2/g)
Ap	Grey shale	59	28	8	5	1.25	560
A	"	69	25	3	3	2.25	615
C	"	58	36	3	3	1.16	752
Mean		**62**	**29**	**5**	**4**	**1.55**	**642**
Ap	Basalt	48	46	2	4	1.25	687
A	"	40	48	10	2	1.09	636
C	"	62	26	8	4	1.12	720
Mean		**50**	**40**	**7**	**3**	**1.15**	**681**
Ap	Granite-granodiorite	58	39	1	2	2.1	490
C	"	49	44	5	2	1.06	510
Mean		**54**	**41**	**3**	**2**	**1.55**	**500**
Ap	Granodiorite	51	31	11	7	0.57	455
AC	"	64	25	8	3	0.85	480
C	"	60	31	6	3	1.11	530
Mean		**58**	**29**	**8**	**4**	**0.84**	**488**
Ap	Limestone	76	21	1	2	1.44	650
A	"	75	23	1	1	1.37	696
AC	"	79	18	1	2	1.7	706
C	"	69	28	1	2	1.36	720
Mean		**75**	**22**	**1**	**2**	**1.47**	**694**

The relative amount of chloritic minerals reported in the present investigation seems to be quite high. Earlier studies either reported nil amount (Chatterjee and Rathore 1976) or small amount (Pal and Deshpande 1987, Prakasa Rao and Swamy 1987) of chlorite in black soil. The reason might be that in the present investigation the measurement of peak area of smectite minerals has been done in the ignited sample rather than in the glycerol solvated specimen where the large smectite peak masks smaller peaks in the region of 1.0 to 1.4 nm and gives over estimation of smectite and under estimation of chloritic minerals.

The nature of the chloritic minerals whose reflections occupy the region of 1.0 and 1.4 nm in the diffractogram of the ignited samples (Fig. 2) needs to be further examined to explore the possibility of these minerals to be hydroxy interlayered smectites of their interlayer filling to different extent.

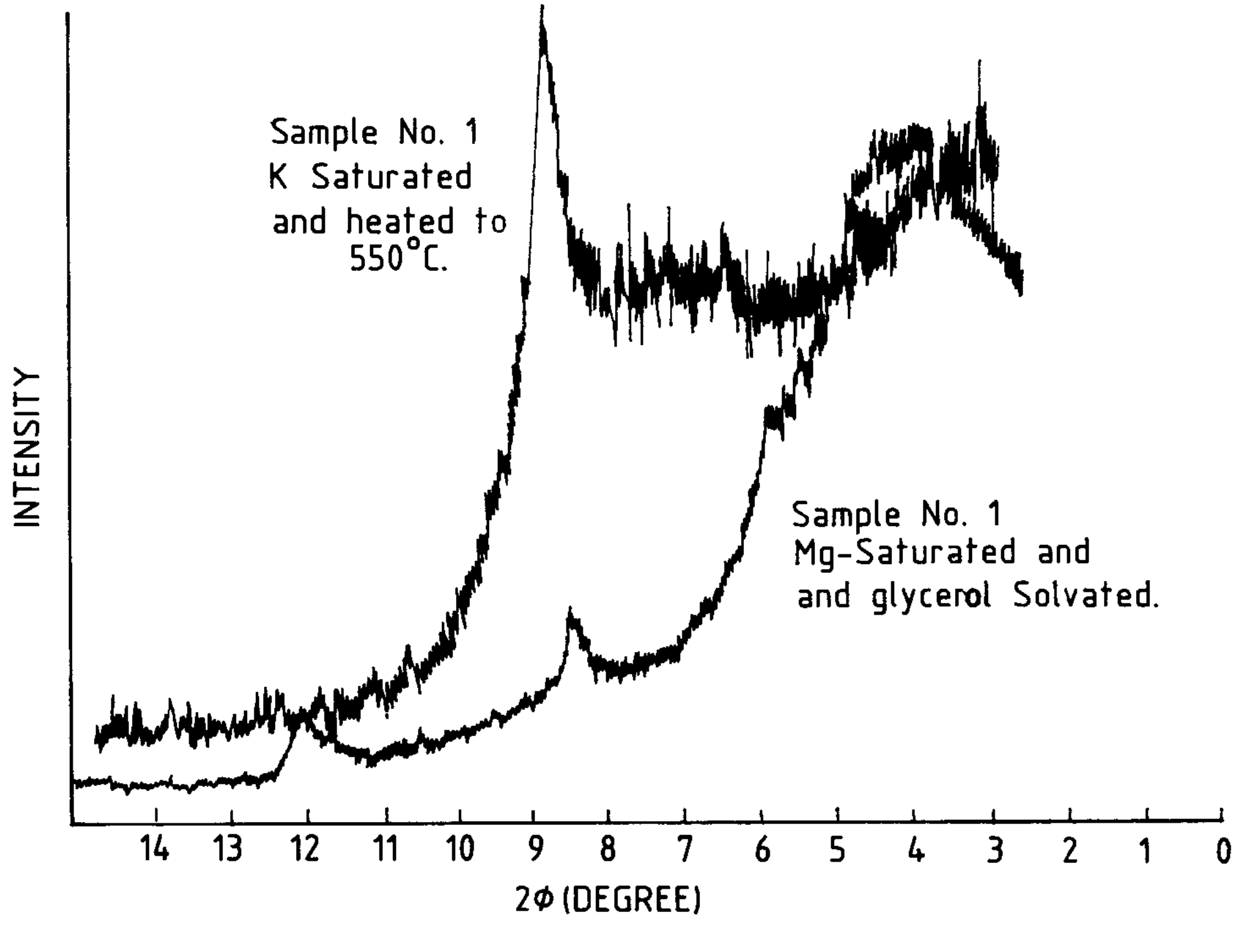

Fig. 2. Representative XRD diagram of the clay fractions of Vertisols.

Acknowledgement

The first author is thankful to IARI for awarding senior research fellowship to conduct the above studies.

References

Carter, D.L., Heilman, M.D., and Gonzalez, C.L. (1965). Ethylene glycol monoethyl ether for determining surface area of silicate minerals. *Soil Science* **100,** 356-360.

Chatterjee, R.K., and Rathore, G.S. (1976). Clay mineral composition, genesis and classification of some soils developed from basalts in Madhya Pradesh. *Journal of Indian Society of Soil Science* **24**, 144-157.

Datta, S.C. (1996). Characterisation of micaceous minerals in soils for strain and size of crystallites through deconvolution and curvefitting XRD profile. *Clay Research* **15**, 20-27.

Gjem, O. (1967). Studies on clay minerals and clay mineral formation in soil profiles in Scandinavia. *Meddelelser fra det Norsk skogforsokvesen,* **21**, 303-415.

Jackson, M.L. (1956). 'Soil Chemical Analysis Advanced Course'. (Published by the author : Univ. of Wisconsin, Madison, Wisconsin.)

Murthy, I.Y.L.N., Sastry, T.G., Datta, S.C., Narayanasamy, G., and Rattan, R.K. (1994). Characterisation and classification of Vertisols derived from different parent materials. *Agropedology* **4**, 107-112.

Pal, D.K., and Deshpande, S.B. (1987). Genesis of clay minerals in a red and black soil complex of Southern India. *Clay Research* **6**, 6-13.

Prakasa Rao, M., and Swamy, A.S.R. (1987). Clay mineral distribution in the mangroves of the Godavari delta. *Clay Research* **6**, 81-86.

Received : March, 1997; Accepted : June, 1999