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A MINERALOGICAL STUDY OF SOME WEST INDIAN SOIL-CLAYS

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ABSTRACT

Identification of primary clay mineral species and mixtures of secondary layer-silicate minerals and of allophane have been made on some West Indian Soils. These soils are high in kaolinite, low in gibbsite, and contain montmorillonite, illite, vermiculite, quartz, allophane and chlorite. Some soils, however, are composed essentially of organic materials.

INTRODUCTION

Since the type of clay in a soil has a great effect on its productivity, identification of clay minerals is of academic and practical value. Differential thermal analysis (DTA) has been used extensively in the study of soils and clay minerals from most regions excluding the West Indies. The DTA thermograms and X-ray diffraction spectra were used to identify the West Indian soil-clay constituents.

The DTA method of analysis has several advantages. It provides a simple, rapid and sensitive method, and specificity for mineral species which may not be readily differentiated by diffraction properties. Differences in the temperature of hydroxyl-water loss provide a means of differentiating mineral species within any isomorphous series of minerals, and because of the characteristic shapes of DTA curves of certain minerals (e.g. kaolinite), it is often possible to detect their presence by this method alone. However, clays may exist either as a combination of discrete particles or an interstratification of layers of separate minerals. The interstratification may exist either as a regular alternation of two or more layer-silicate species within a single crystal, or the layers may be dispersed at random. These soil types contain a number of mineral species some of which may not be detected by the DTA method but can be identified by a distinctive X-ray diffraction maximum [1].

EXPERIMENTAL

Samples

The samples were prepared for analyses by:
A. DTA

TABLE 1

Clay minerals of selected West Indian soils

Soils	Location	Mica (I/B)	K	M/V	Heat of reaction ΔH (kJ kg^{-1})[a]		A	Other trace	Interstratified
					10^{-5}d	10^{-4}r			
Barataria[b]	Trinidad	25/45 ± 5						K; F; Q; V	K—I
Capitol	Grenada		85 ± 5	15 ± 5	2.36	4.67		Q	
Cunupia	Trinidad	20	35 ± 5	25 ± 2/5	1.20	2.67		5% Go; Q	M—V
Fitches	Antigua			75 ± 5				Q; F; H	
LaPlaine[c]	Dominica		45 ± 5		3.17	4.00	35 ± 5	6% Go; V; I	C—V; M—C—V
Montreal[b]	St. Vincent	/85 ± 5			0.094	4.94	15 ± 5	K; M; V; F	V—M
Montserrat	Trinidad		10	45 ± 5/15				15% Go; F; I; Q	
Princes Town	Trinidad		35 ± 5	35 ± 5	8.79	1.50		Q	M—V—I
River Estate	Trinidad	/50 ± 5	25 ± 5	4	0.746	0.514		8% Go, Q	C—V
St. Augustine[b]	Trinidad	55 ± 5						Q; C; K; V	
Talparo	Trinidad	25 ± 5	35 ± 5	25 ± 2/5	1.04	1.52		4% Go; Q	M—V; M—V—I
Wood Ford Hill	Dominica	15 ± 5	85 ± 5		2.45	3.75	Some	V; C; H; Q	C—V

A = Allophane; B = biotite; C = chlorite; F = feldspar; Go = goethite; H = halloysite; I = illite; K = kaolinite; M = montmorillonite; V = vermiculite.

[a] d = Dehydroxylation; r = recrystallization.

[b] Essentially organic soils.

[c] High in organic matter.

(i) Removal of unwanted substances (e.g. dry leaves).
(ii) Removal of organic matter and substitution of magnesium ions as the replaceable cations [2].

B. X-Ray diffraction analyses
(i) Air-dry slides of the magnesium saturated samples.
(ii) Slides of glycolation of the magnesium saturated samples.
(iii) Substitution of potassium ions as the replaceable cations, and making slides for analyses as follows:
(a) air-dry slides were prepared at room temperature (~25° C);
(b) the air-dry slides were heated for several hours at 300° C;
(c) the 300° C slides were heated for several additional hours at 550° C [3].

Analyses

The DTA instrument was a DuPont 900 analyzer, with Pt/Pt—13% Rh thermocouples, Pt sample holders, and a heating rate of 20° C min^{-1} [2].

A Philips Morelco diffractometer equipped with a potentiometric recorder and AMR monochromator was used for obtaining the X-ray spectra. The patterns of the oriented samples [B (iii) (a)—(c)] were obtained using CuK_α radiation. The samples were moistened with glycerol (ii) to bring out the characteristic montmorillonite lines, and heated to 550° C [(iii) (c)] to collapse certain mineral structures such as kaolinite and gibbsite and to allow chlorite, vermiculite and allophane lines to remain visibly more distinct.

The soils used in this work are all treated specimens from the Eastern Caribbean (Table 1). The clay minerals were identified from the diagnostic DTA thermograms and the X-ray diffraction spectra. The proportion of the minerals present was estimated from the intensity of the diffraction peaks, and the heats of reaction from the areas of the DTA curves. These results, though qualitatively reliable, are only semi-quantitative since various factors may influence the intensity of the diffraction peaks and the areas of the DTA curves.

RESULTS AND DISCUSSION

Montmorillonite, kaolinite and mica are the main clay minerals found. These names indicate mineral groups rather than specific minerals. Allophane was found in La Plaine, Wood Ford Hill and Montreal soils (Table 1).

Vermiculite was distinguished from montmorillonite, halloysite from kaolinite, and mica (biotite and illite) from gibbsite by the changes in the positions and intensities of the X-ray diffraction peaks as a result of changes in chemical treatment and in thermal conditions. For instance, although the *d* spacings of ca. 14 Å of the air-dry magnesium-treated sample may be due to the presence of either montmorillonite, vermiculite, chlorite or a mixture of these species, the solvation with glycerol allows the separation and positive identification of montmorillonite whose lattice is expanded to ca. 18 Å while the others remain essentially unchanged. Saturation with potassium ions allows separation of vermiculite from chlorite since the chlorite lattice does not collapse on heating while vermiculite and montmorillonite undergo lattice contraction — heating to 550° C also destroys kaolin minerals.

Chlorite usually yields a second-order maximum at nearly the same position as the first-order maximum of kaolinite (at 7.15 Å). Thus, if a peak at ca. 7 Å disappears or decreases in intensity on heating to 550° C, the presence of kaolinite is confirmed. The peak for gibbsite (ca. 4.06 Å) disappears on heating to 550° C.

Illite is identified by a diffraction peak at 10 Å which is unaffected by glycolation or heat to 300° C, but shows contraction at 550° C.

Quartz is confirmed by strong reflections at 3.34 Å; feldspar shows a reflection at ca. 3.20 Å; allophane (little crystalline) gives ill-defined peaks at ca. 4.10 Å which remain at 550° C; halloysite gives various peaks in the 7 Å region, the position being dependent on the amount of interlayer water present. Peaks at ca. 4.4 Å are indicative of halloysite [4].

It is known that regular alternation of two species within a crystal produces repeating diffraction planes at a distance equal to the (001) distances of the two species. For example, a regular alternation of montmorillonite—mica, or chlorite—mica yields a diffraction spacing at 14 Å + 10 Å = 24 Å, while montmorillonite—vermiculite, montmorillonite—chlorite or vermiculite-chlorite give 14 Å + 14 Å = 28 Å spacing in each case. Two species which are randomly interstratified, however, yield a well-defined spacing intermediate between the normal (001) spacing of the two individual members. Thus, mica—vermiculite yields, an average spacing between 10 Å and 14 Å in which the exact spacing distance will depend upon the relative proportions of the two species in the mixture, etc. [1].

Clay mineral groups were also identified by the DTA method. For example mica (biotite, brucite and illite), which is high in hydronium ions and water, gives characteristic fairly well-defined DTA curves. These thermograms show low—medium temperature dehydroxylation endotherms and an S-shaped endothermic—exothermic system at ca. 900° C, of which the exothermic section represents formation of a spinel.

Clays which contain montmorillonite have a single endotherm in the 100—200° C range. The structural hydroxyl is lost in two stages, the first results from the breaking of the cation-hydroxyl bond and the second is due to the complete breakdown of the montmorillonite structure [5]. These occur at 627° C and 930° C for standard bentonite but appear at slightly different temperatures in soils.

Soil kaolins show marked endothermic effects at ca. 540—600° C resulting from the evolution of structural hydroxyl groups. Grimshaw et al. [6] found that this reaction occurred at ca. 585° C for kaolinite. In this work, the peak temperature appeared at 566 ± 2° C for standard kaolinite and rose to 575° C in the mixture with standard illite. The soils produced peaks in this temperature range, as well as the diagnostic high temperature recrystallization exotherm. In the treated Princes Town clay a second exotherm appeared. It should be remarked that the X-ray diffraction spectrum of Fitches soil did not support the presence of kaolinite in this soil. This is the only case in which both methods did not complement each other. However, the X-ray data showed the presence of halloysite, a member of the kaolin family.

Quartz is usually identifiable from its α—β transition endotherm which occurs at 573° C. However, these peaks are interferred with by broad endothermic effects of kaolinite and montmorillonite in soils in which they are all present. Evidence of the presence of quartz in Fitches and Princes Town clays was obtained from X-ray patterns. The results given in this paper were, therefore, deduced and calculated from a combination of the data obtained from the DTA thermograms and X-ray diffraction spectra of these soils. The relative amounts of the clay minerals present together with the heats of reaction of the kaolinitic clays of the $<2\mu$ fraction are given in Table 1.

Barataria peat and St. Augustine loam from Trinidad, and Montreal clay from St. Vincent are essentially organic soils, containing mica (biotite and illite) as their main mineral constituents. They also contain small amounts of quartz, kaolinite, vermiculite and feldspar or chlorite (Fig. 1). Montreal clay also contains allophane and traces of montmorillonite and interstratified vermiculite—montmorillonite. La Plaine clay from Dominica is high in organic matter and allophane. The treated soil also contains a large percentage of kaolinite, some goethite, and traces of illite, vermiculite, interstratified chlorite—vermiculite, and vermiculite—montmorillonite—chlorite assemblages (Fig. 2). This soil can be classified as a "mixed" mineral. The organic

Fig. 1. X-Ray, Barataria, T and T (organic).

Fig. 2. X-Ray, La Plaine, Dominica (allophanic).

matter is evidenced in DTA thermograms by large, low temperature oxidation, exothermic peaks [7], and in X-ray diffraction spectra by very diffuse, ill-defined patterns [8]. Allophane is "little" crystalline, and therefore, yields ill-defined X-ray spectra.

Capitol (Grenada) and Wood Ford Hill (Dominica) clays contain a large percentage of kaolinite and gibbsite (Fig. 3) as evidenced by both DTA and X-ray techniques. Capitol clay contains some montmorillonite and a trace of quartz, while Wood Ford Hill clay contains some illite, vermiculite, allophane, traces of quartz, chlorite and interstratified chlorite—vermiculite. The presence of quartz was not evident from the DTA thermogram of either soil, but was clearly identified from their X-ray diffractograms. They can be classified as "mixed-mineral" soils which are high in kaolinite/gibbsite.

Cunupia and Talparo (Trinidad) clays are quite similar in texture and composition. They contain more "poorly" crystalline kaolinite than the other clays which are classified as kaolinitic. They contain significant amounts of montmorillonite, quartz, illite, traces of goethite and montmorillonoid (interstratified montmorillonite—vermiculite in both samples, and montmorillonite—vermiculite—illite in Talparo clay). Although the quartz content was not determined quantitatively, it is apparent that the Talparo clay contains a larger quantity of quartz. The quartz inversion temperature curve is more prominent in the DTA thermogram, and its reflection (3.34 Å) intensity is

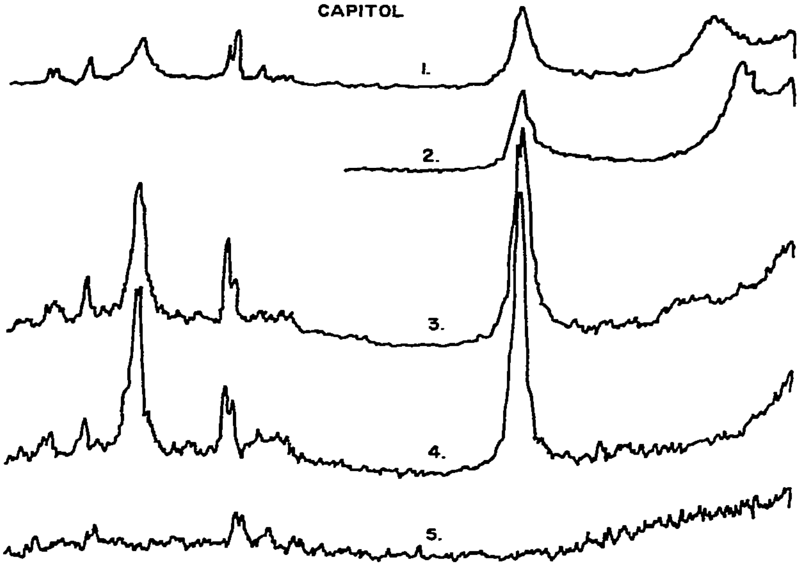

Fig. 3. X-Ray, Capitol, Grenada (kaolinitic).

stronger in the X-ray pattern (Fig. 4) for Talparo clay than for Cunupia clay. Princes Town (Trinidad) clay shows some similarities to these but it contains illite (as in Talparo clay) only as interstratified species with montmorillonite and vermiculite, and it contains a larger percentage of montmorillonite than they do.

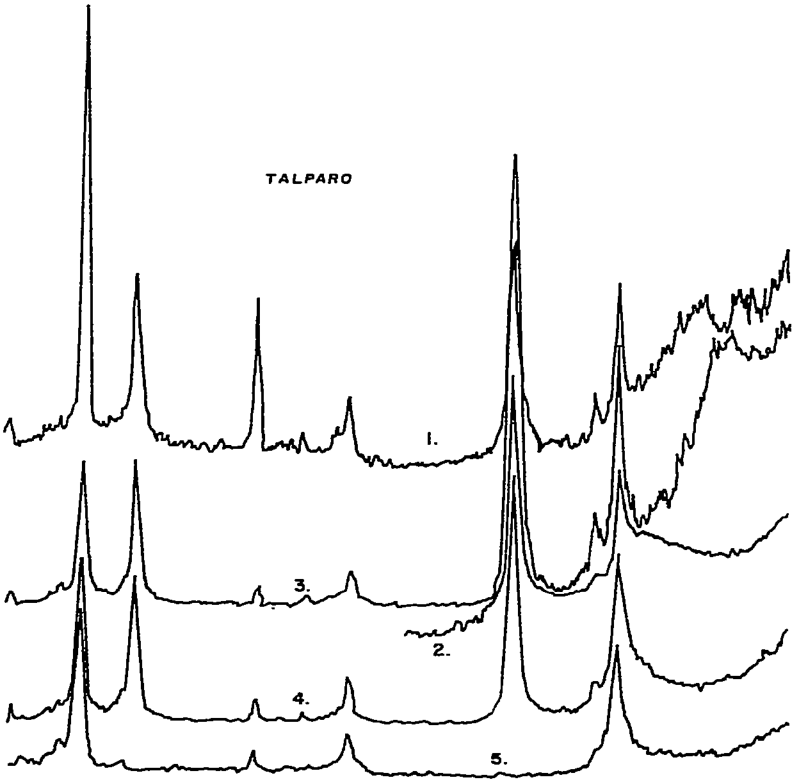

Fig. 4. X-Ray, Talparo, T and T (mixed Mineral).

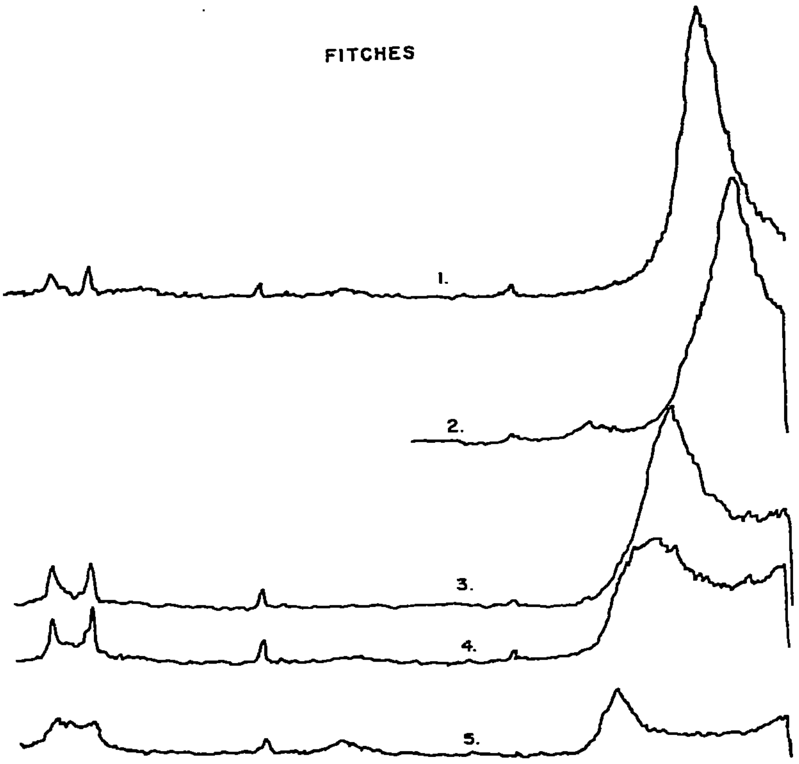

Fig. 5. X-Ray, Fitches, Antigua, (montmorillonitic).

Fitches (Antigua) and Montserrat (Trinidad) clays contain large quantities of montmorillonite. Fitches clay is composed essentially of montmorillonite (Table 1; Fig. 5), but it also contains traces of quartz, feldspar and halloysite. Montserrat clay contains, in addition to montmorillonite, vermiculite, goethite, kaolinite and a trace of illite.

River Estate (Trinidad) clay contains a large amount of biotite, a considerable quantity of kaolinite, small amounts of goethite, interstratified chlorite—vermiculite, and traces of montmorillonite and quartz.

CONCLUSION

Characteristic effects of many clay minerals and organic constituents of a selected group of West Indian soils were studied from their DTA and X-ray diffraction patterns. The information gained from the combination of data from these methods was used to identify the composition of these soils.

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