

AN X-RAY DIFFRACTION STUDY OF SOME CLAY MINERALS OF THE EASTERN CARIBBEAN, WEST INDIES

M. SWEENEY

Department of Chemistry, University of the West Indies (Trinidad)

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ABSTRACT

X-Ray diffraction patterns were run on 12 soil-clays from Antigua, Dominica, Grenada, Trinidad and St. Vincent, West Indies. The d spacings of the diffraction maxima of these soil-clays saturated with Mg^{2+} and K^+ cations were used for identification of the clay minerals present. The criteria for species differentiation was obtained by diagnostic diffraction maxima from clays specially treated with ethylene glycol and heat treatment up to $550^{\circ}C$.

INTRODUCTION

Recent papers of this author described the identification of clay minerals in some West Indian soils by the DTA method [1–3]. It was pointed out that some of the conclusions arrived at from the DTA patterns were based on supported evidence obtained from X-ray diffraction patterns. These soils, however, contain a number of species, some of which cannot be detected in the DTA method but can be identified from a distinctive diffraction maximum from a single X-ray pattern [4].

This paper is intended to produce the necessary data and discussion of the diffraction patterns of these specially treated soil samples. Since the shape of the X-ray diffraction spectrum is uniquely determined by spatial arrangement of atoms in the scattering solids, the structure can, in principle, be directly deduced from the relative widths, heights and positions of the maxima [5]. Interlayer spacings, d_{001} and d_{002} , and apparent crystallite thickness, L_C , were determined from the position and width of these profiles using $2\theta(CuK_{\alpha})$ radiation [6]. High temperature X-ray diffraction studies have been used for the detection of structural changes [7], and therefore those clay minerals which undergo irreversible structural changes can be identified by this method. The combination of data obtained from five "methods" provide information for the identification of the clay minerals present.

Soil clays often contain two or more layer-silicate species interstratified within a single crystal in either random or regular manner. Diffraction effects from these mixed crystals are quite different from those with only a single species. It has been shown that regular alternation of two species within a

crystal produces repeating diffraction planes at a distance equal to the (001) distances of two species. On the other hand, two species which are randomly interstratified yield a well-defined spacing intermediate between the normal (001) spacing of the two individual members [4].

More complicated interstitial mixtures involving three or more species do occur in soils, but they are practically impossible to differentiate into individual component species. Such mixtures yield only broad, indistinct diffraction effects. A knowledge of the general principles governing diffraction by interstratified mixtures is, however, essential for the study of clay minerals by X-ray diffraction techniques [8].

EXPERIMENTAL

Methods

Preparation of soils (general)

The soils were prepared for X-ray diffraction analyses using a modified procedure of Kittrick and Hope [9]. After the removal of the light materials, soluble salts, exchangeable cations (Ca^{2+} and Mg^{2+}), surface adsorbed or free ions (Fe^{3+} and Al^{3+}) and organic cementing matter, the sediments were dispersed in water, sonified and centrifuged at a convenient speed and time for the $<2\mu$ equivalent spherical diameter particles to settle to 90% of the length of the centrifuge tube.

A. Mg-saturation. Magnesium, which allows relatively uniform interlayer absorption of water by expandable layer-silicates, was used for exchange saturation cation. The $<2\mu$ soil clay suspension was acidified and treated with excess 5 M $\text{Mg}(\text{OAc})_2$. The solid, separated by centrifugation, was washed in 0.5 M $\text{Mg}(\text{OAc})_2$ solution to remove the H_3O^+ ions, and then with 0.5 M MgCl_2 solution, distilled water and alcohol.

(i) Mg-saturated, water suspension, air dried. Water was added to the Mg-saturated clay sample to make enough suspension to cover the slide and permit it to be held by film tension when dried in air.

(ii) Mg-saturated, glycerol solvation, air dried. Glycerol was added to a portion of the Mg-saturated clay to produce both the right amount of gloss as well as to enable the sample to adhere to the slide by film tension when dried in air.

B. K-saturation. Potassium, which specifically restricts interlayer absorption of water, was used as the other exchange cation. The same procedure was followed as for Mg-saturation (above) except that no acid treatment was necessary. The sample was washed several times with 1 M KCl solution and then with water and alcohol to remove the excess salt.

(i) K-saturated, water suspension, air dried. The X-ray slide was prepared as in A(i).

(ii) K-saturated, 300°C. The potassium-saturated specimen was run at room temperature, then heated to 300°C in a muffle furnace for 3 h. The

specimen was cooled just sufficiently to be handled and the X-ray analysis made.

(iii) K-saturated, 550°C. The K-saturated (ii) specimen was heated to 550°C for 3 h. It was cooled slowly in the furnace to approximately 300°C and analyzed immediately.

Analyses

The X-ray diffraction spectra of oriented samples were obtained with a Philips Norelco diffraction spectrometer using filtered $\text{CuK}\alpha$ radiation ($\lambda = 1.5405 \text{ \AA}$) and starting at angle $2\theta = 3^\circ$ (29.425 \AA).

TABLE 1
X-Ray diffraction data — Montserrat soil ($\text{CuK}\alpha$)

Mg/H ₂ O		Mg/G		K/Rm		K/300		K/550	
<i>d</i> (Å)	<i>I</i>	<i>d</i> (Å)	<i>I</i>	<i>d</i> (Å)	<i>I</i>	<i>d</i> (Å)	<i>I</i>	<i>d</i> (Å)	<i>I</i>
14.84	100	26.8	93	23.23	42.6	19.62	24.4	18.3	11.4
9.99	26.6	24.3	85	19.62	36.4	18.02	21.6	9.93	100
9.18	8	22.4	85	18.02	34.2	11.5	21.6	9.30	19.5
7.16	66.7	20.72	90	17.31	33.6	9.99	100	9.13	19.5
5.40	7	19.62	100	16.82	33.0	9.66	25.0	4.96	29.7
4.98	16.7	18.32	100	16.05	31.0	9.20	20.4	4.63	13.8
4.25	8	16.98	76	14.97	34.1	8.30	7.6	4.25	15.8
3.68	5	9.95	26.6	12.80	41.8	7.16	77.0	3.51	14.3
3.58	45.3	7.14	43.5	12.18	44.8	5.0	28.0	3.34	91.0
3.35	53.4			11.68	46.6	4.59	11.0	3.32	86.4
				11.4	42.6	4.26	16.3	3.24	24.5
				11.11	41.7	3.70	12.4	3.18	15.6
				10.62	39.4	3.57	53.3	3.10	18.0
				10.39	38.0	3.34	84.0	3.06	13.8
				9.99	96.6	3.32	79.0		
				9.19	27.4	3.25	7.9		
				7.92	8.6	3.20	7.7		
				7.15	89.7	3.06	18.1		
				4.99	30.0				
				4.25	19.0				
				4.17	11.5				
				4.13	11.5				
				3.57	57.4				
				3.34	100				
				3.24	10.0				
				3.22	9.0				
				3.20	11.6				
				3.18	11.4				
				3.06	20.0				

Mg/H₂O = Magnesium-saturated, air dried; Mg/G = glycolated-saturated, air dried; K/Rm = potassium-saturated, air dried, room temperature.

K/300 = Potassium-saturated, heat treated for several hours at 300°C; K/550 = potassium-saturated, heat treated for several hours at 550°C; *I* = peak intensities.

Patterns were obtained for air-dried samples, samples moistened with glycerol to bring out characteristic montmorillonite spectra, and samples heated to 550°C to collapse expanding minerals and to allow chlorite and vermiculite to show up.

DISCUSSION

The soils studied contain montmorillonite, kaolinite, mica, allophane and/or mixtures of these, as well as small quantities of other minerals. Data relating to the d spacings and corresponding reflection intensities of representative soil clays studied are given in Tables 1–4 for samples high in montmorillonite, kaolinite, mica and allophane, respectively. Table 5 shows a summary of the d spacings of the high intensity reflections, represented in decreasing order of magnitude for each of the five methods employed, and a summary of the mineral contents.

Analyses of the spectra from Montserrat, Cunupia, Fitches and Princes Town clays (Figures 1–4, respectively) show that they contain the following:

TABLE 2
X-Ray diffraction data — Capitol soil

Mg/H ₂ O		Mg/G		K/Rm		K/300		K/550	
d (Å)	I	d (Å)	I	d (Å)	I	d (Å)	I	d (Å)	I
20.53	50.5								
16.66	64	21.0	76	22.07	40.2	10.52	12.4	10.27	73
15.22	75	19.19	90	18.32	32.8	9.93	12.4	9.02	59
14.36	69	18.79	81	15.59	26.5	9.34	12.4	4.60	43
7.30	100	17.80	90	14.72	27.0	8.93	9.8	4.50	43
4.27	19	7.30	100	14.24	26.5	7.25	100	4.14	92
4.11	59			13.8		4.46	13.3	4.10	100
4.06	44			13.59		4.37	14.5	3.80	43
3.59	48			12.62	26.5	4.25	16.3	3.75	53
3.37	29			7.30	100	4.09	26.6	3.39	75
3.22	17			4.41	18	4.06	32	3.36	58
3.20	17			4.27	18.8	3.95	10.6	3.15	45
				4.09	32	3.83	12	3.12	33
				4.06	50	3.76	12		
				3.58	75	3.57	66		
				3.34	32	3.40	11.5		
				3.26	18	3.34	19.7		
				3.22	22	3.22	15.6		
				3.20	20	3.20	12.8		

Mg/H₂O = Magnesium-saturated, air dried; Mg/G = glycolated-saturated, air dried; K/Rm = potassium-saturated, air dried, room temperature; K/300 = potassium-saturated, heat treated for several hours at 300°C; K/550 = potassium-saturated, heat treated for several hours at 550°C; I = peak intensities.

TABLE 3
X-Ray diffraction data — LaPlaine soil

Mg/H ₂ O		Mg/G		K/Rm		K/300		K/550	
<i>d</i> (Å)	<i>I</i>	<i>d</i> (Å)	<i>I</i>	<i>d</i> (Å)	<i>I</i>	<i>d</i> (Å)	<i>I</i>	<i>d</i> (Å)	<i>I</i>
14.72	8.4	21.02	27	14.24	9.2	21.02	8.5	18.39	11
14.24	8.4	18.02	22	13.38	9.2	19.9	5.7	16.35	6.8
13.18	9.6	14.24	22	10.08	9.2	17.66	8.8	15.49	6.4
10.04	10.9	13.22	26	7.31	20.2	16.05	5.2	13.79	6.4
9.82	10.9	7.37	90	7.19	33.3	13.9	5.0	10.04	10
9.60	10.9	7.19	100	5.00	10.5	10.7	6.5	6.51	6.4
7.37	28.5			4.84	13.3	9.97	11.5	4.95	13.5
7.19	32			4.48	18.3	7.31	19.6	4.60	14
4.41	24			4.22	18.3	7.17	32.3	4.42	6.5
4.25	25			4.10	65	6.46	6.0	4.25	10
4.09	49.8			4.06	100	5.32	7.3	4.10	65
4.06	100			3.77	22.5	4.98	5.7	4.06	100
3.58	32			3.58	30.3	4.84	6.1	3.96	8.5
3.56	31.6			3.44	18.8	4.46	5.8	3.91	8.5
3.47	18.8			3.35	31.6	4.38	5.8	3.78	10.6
3.22	18.8			3.25	22.5	4.25	10	3.57	8.9
				3.22	22.9	4.09	58.5	3.36	29
						4.05	100	3.25	8.5
						3.96	8.1	3.22	7.6
						3.78	10.4		
						3.67	8.4		
						3.57	28.8		
						3.53	20.4		
						3.45	15		
						3.34	28.9		
						3.24	11.5		
						3.22	10.2		

Mg/H₂O = Magnesium-saturated, air dried; Mg/G = glycolated-saturated, air dried; K/Rm = potassium-saturated, air dried, room temperature; K/300 = potassium-saturated, heat treated for several hours at 300° C; K/550 = potassium-saturated, heat treated for several hours at 550° C; *I* = peak intensities.

(a) montmorillonite: Montserrat and Fitches are composed essentially of this mineral and the others contain a considerable amount;

(b) kaolinite: it exists as halloysite in Fitches;

(c) illite: with the exception of Fitches, small quantities were found. It exists as an interstratified mineral with montmorillonite and vermiculite in Princes Town;

(d) traces of quartz;

(e) feldspar in Montserrat and Fitches only.

Montserrat

Montmorillonite is identified as the main clay mineral in Montserrat clay by the strong diffraction peak at 14.84 Å (obtained from the Mg-saturated,

TABLE 4

X-Ray diffraction data — Montreal soil

Mg/H ₂ O		Mg/G		K/Rm		K/300		K/550	
<i>d</i> (Å)	<i>I</i>	<i>d</i> (Å)	<i>I</i>	<i>d</i> (Å)	<i>I</i>	<i>d</i> (Å)	<i>I</i>	<i>d</i> (Å)	<i>I</i>
20.53	17.8	19.88	58	21.96	17.4	18.34	18	14.48	16.3
18.79	17.8	17.95	54.5	19.62	10	14.12	18	12.44	17.3
15.49	23.8	16.98	48.6	18.39	12.4	11.38	16.2	10.52	16.3
14.36	100	14.24	100	16.98	15.5	8.18	14.4	10.00	16.3
13.55	25.8	13.18	50	14.48	15.5	8.04	14.4	4.06	28.6
12.53	18	12.6	60.6	13.80	15.5	7.42	12.6	4.02	32
9.93	18	7.94	54	13.59	15.5	7.34	12.6	3.75	32
8.26	19.3	7.43	37	12.95	12.4	7.25	14.4	3.63	30.6
7.16	24.8	7.16	74	12.06	13.6	7.15	37.4	3.54	24.5
4.06	45.5			7.82	10.6	4.54	24.5	3.49	27
4.04	42.6			7.31	8.7	3.95	20	3.40	22
3.22	83.4			7.17	28.2	3.74	21.6	3.36	28
3.18	58.5			4.04	12.8	3.67	19.7	3.22	100
3.13	30.6			3.69	12.4	3.57	21	3.18	75.5
3.02	33.6			3.57	15.5	3.49	20		
2.95	30.2			3.34	18.6	3.34	28		
				3.32	18.6	3.21	100		
				3.22	100	3.18	68.5		
				3.18	54.4	3.01	19		
				3.08	10.5	2.94	15		
				2.95	11.8	2.86	13.5		

Mg/H₂O = Magnesium-saturated, air dried; Mg/G = glycolated-saturated, air dried; K/Rm = potassium-saturated, air dried, room temperature; K/300 = potassium-saturated, heat treated for several hours at 300°C; K/550 = potassium-saturated, heat treated for several hours at 550°C; *I* = peak intensities.

air-dried sample) which expanded to 18.3 Å after glycolation. This peak contracts to 12.2 Å on treatment with potassium at room temperature, and is superimposed on the 10.0 Å illite (001) reflection, which increased in intensity from 27 to 100 from the Mg-treated to K-treated (500°C) samples, while the 14.8 Å reflection has changed from 100(*I*) to zero, being 45(*I*) at 12.2 Å for K-treated, room-temperature analysis (Table 1). The minerals kaolinite, mica (illite), quartz and feldspar are also present.

Kaolinite is identified by its strong characteristic diffraction maxima at 7.16 Å and 3.58 Å which are unaffected by glycolation and thermal treatment to 300°C but disappeared completely at 550°C.

The diffraction patterns [Fig. 1 (1)–(4)] show 10 Å diffraction maxima which are essentially unaffected by glycolation or heat to 300°C and are therefore distinguished as illite. This peak shows a slight contraction at 550°C which is presumably caused by the superimposition of the contracted montmorillonite which is present in higher concentrations.

Quartz is identified by the weak peaks at 4.25 Å and the very strong reflections at 3.34 Å. It is observed that this reflection is strongest for the

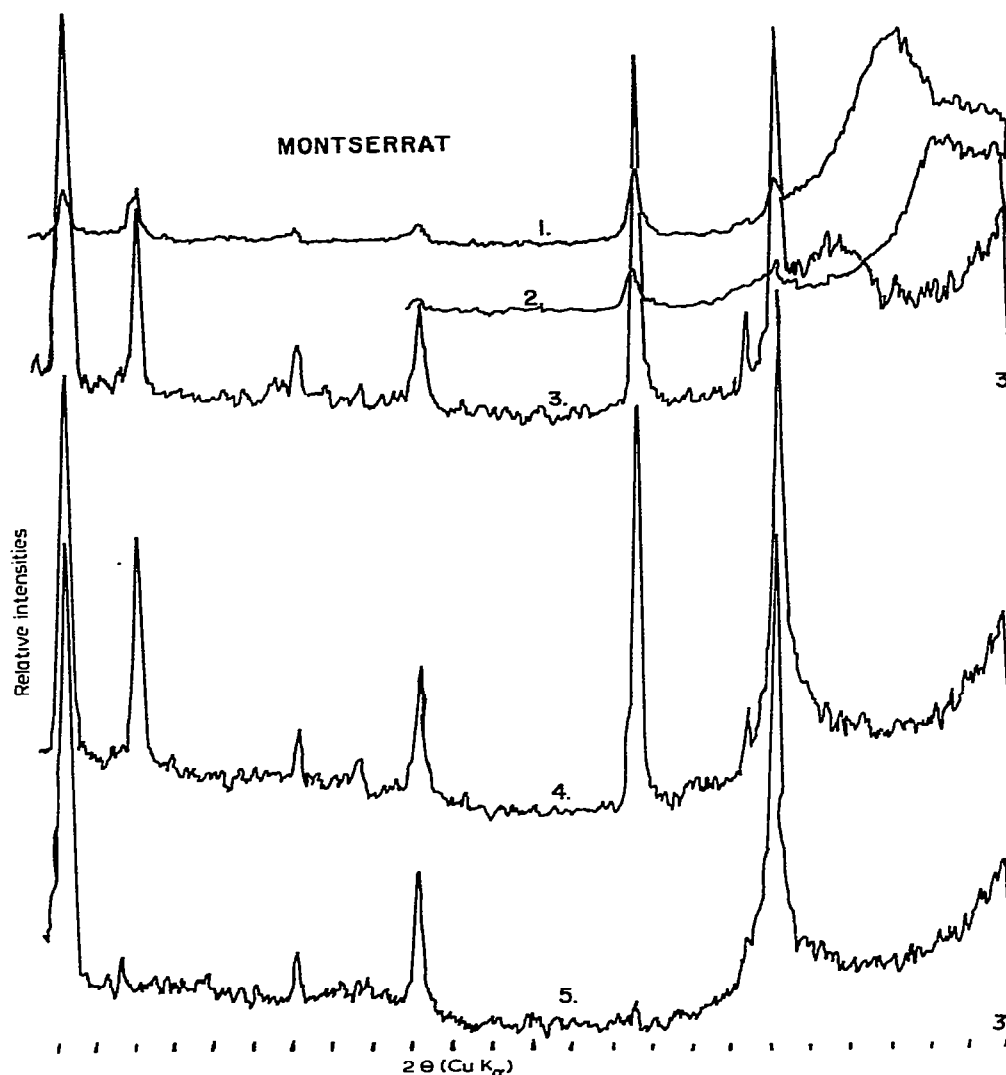


Fig. 1. X-Ray diffraction patterns of Montserrat soil-clay, Trinidad, West Indies.

potassium-treated sample at room temperature but is second to the 10 Å peak in the heated samples where the montmorillonite contraction causes an increase in intensity of this reflection. It should also be noted that the strongest quartz reflection at 3.34 Å is superimposed on the third-order illite reflection. Feldspar is present as a trace mineral, as is evident from the weak diffraction peaks at 3.20–3.24 Å.

Irregular interstratification is deduced from the diffraction patterns of this soil-clay. These appear to be montmorillonite assemblages, interstratified mica–montmorillonite and mica–vermiculite. In the glycolated, Mg-treated sample there are several low 2θ diffuse bands between 26.8 Å and 18.3 Å. The 26.8 Å is believed to be due to the mica–montmorillonite (17 Å + 10 Å), the 24 Å diffraction spacing is the result of mica–vermiculite or mica–chlorite (14 Å + 10 Å), while peaks at 19.6 Å and 18.3 Å show montmorillonite assemblages. The d spacings of K-treated samples support this sugges-

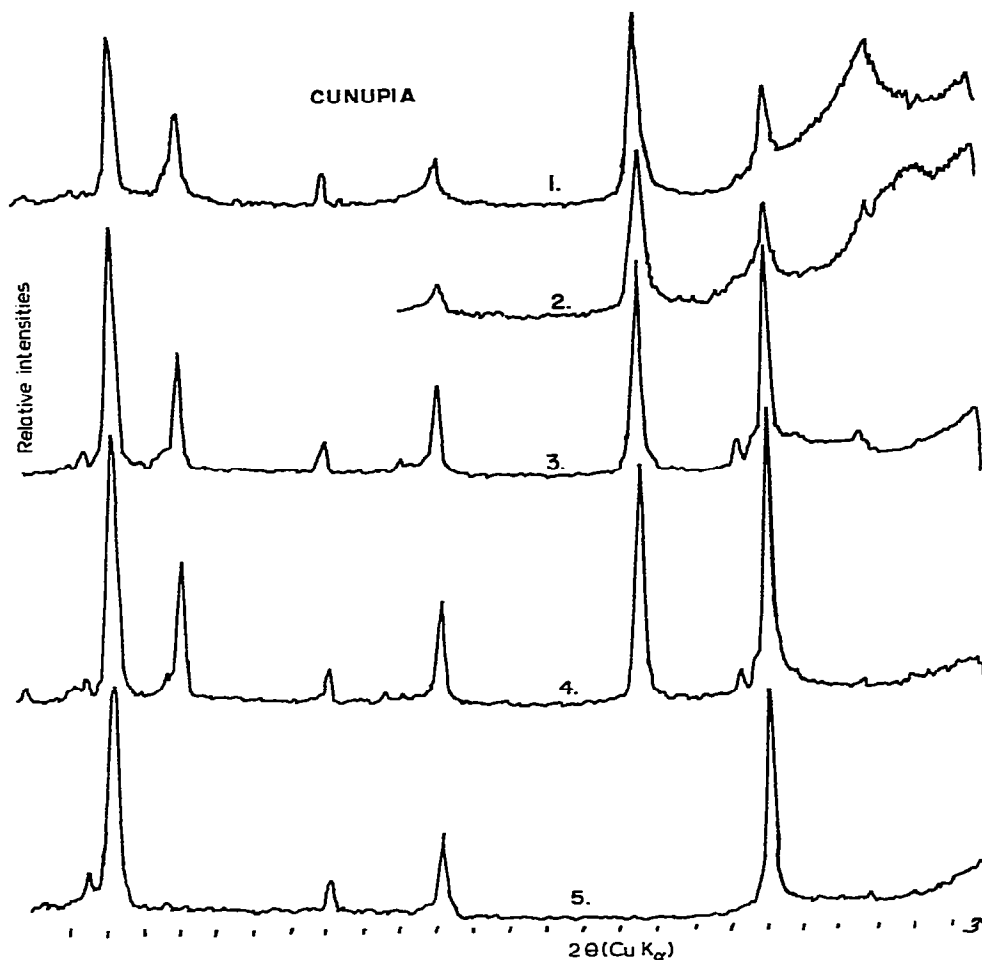


Fig. 2. X-Ray diffraction patterns of Cunupia soil-clay, Trinidad, West Indies.

tion. There are several low angle reflections, but the d spacings for the expandable clays are lower, as expected. For example, the mica—montmorillonite reflection for the K-treated, room-temperature analysis is at 23.2 \AA instead of 26.8 \AA . These reflections decreased both in intensity and d spacings as the K-treated samples were heated. This is the result of the collapse of the montmorillonite and vermiculite structures. Irrational higher order reflections which are not sub-multiples of the first-order reflection also indicate irregular interstratification.

Cunupia

By similar deductive methods, the following minerals were identified in Cunupia: kaolinite, montmorillonite, vermiculite, illite, quartz and traces of irregular interstratifications of kaolinite—vermiculite and mica—kaolinite.

Kaolinite is confirmed by the disappearance of the very strong 7.16 \AA reflection on heating to 550°C [Fig. 2 (5)]. Montmorillonite and vermiculite are confirmed by the splitting of the Mg-treated 14.4 \AA peaks to produce

TABLE 5

Summary of X-ray analysis of West Indian clay minerals (strongest peaks in decreasing order of intensity)

Soil	Mg/H ₂ O (Å)	Mg/ glycerol (Å)	K/Rm (Å)	K/300 (Å)	K/550 (Å)	Minerals ^a
Montserrat	14.84	19.62	3.34	9.99	9.93	Montmorillonite, kaolinite, vermiculite, illite, quartz and feldspar
	7.18	18.32	9.99	3.34	3.34	
Cunupia	7.18	7.18	3.34	9.95	3.34	Montmorillonoid (vermiculite—montmoril- lonite), kaolinite, illite and quartz
	3.34	19.6—18.0	9.95	3.34	9.97	
	14.48	14.3, 10.0	7.16	7.16		
Fitches	14.92	18.9	12.62	11.94—10.6	9.93	Montmorillonite, halloysite, quartz and feldspar
	3.34	17.70	3.34	4.26, 3.34	(3.34—3.18)	
		7.16	3.19	12.8 and 3.2		
Princes Town	14.84	14.84	3.34	10.04	3.34	Montmorillonite, kaolinite, quartz and montmorillonite—vermiculite—illite
	7.19	7.19	7.15	3.34	10.0	
	3.34		10, 3.57, 11—14	7.16	5.0	
Capitol	7.30	7.30	7.30	7.25	4.1	Kaolinite, gibbsite, montmorillonite and quartz
	14.4—16.7	17.80	3.58	3.57	(all weak)	
	4.1, 3.59	18.8	4.06	4.06		
Wood Ford Hill	7.17	7.19	7.17	7.15	10.0	Kaolinite, illite, vermiculite, quartz, halloysite, allophane, chlorite and chlorite— vermiculite
	3.34	10.97	3.57, 4.84	3.59	4.10	
	9.93	14.4	3.34	3.34	3.54	
LaPlaine	4.06, 4.1	7.19	4.06	4.06	4.06	Kaolinite, allophane, montmorillonite— vermiculite—chlorite and illite
	7.19, 7.37	7.37	4.1	4.09	4.10	
	3.58		7.19	7.17	3.36	
Barataria peat	10.04	9.97	9.97	9.95	9.93	Illite, kaolinite, quartz, feldspar, mica (disordered kaolinite—illite), and vermiculite
	3.34	9.7	3.32	3.32	3.31	
	3.2, 9.25		4.98	4.98	4.98	
River Estate	9.98	9.98	10.02	9.98	9.98	Biotite/illite, kaolinite, vermiculite— chlorite, montmorillonite and quartz
	3.33	7.1	3.32	3.32	3.32	
	7.13		5.0, 7.1	5.0, 7.1	4.98	

TABLE 5 (continued)

Soil	Mg/H ₂ O (Å)	Mg/ glycerol (Å)	K/Rm (Å)	K/300 (Å)	K/550 (Å)	Minerals ^a
St. Augustine	10.02	10.02	10.04	10.04	10.00	Illite, kaolinite, vermiculite, chlorite and quartz
	3.34	7.1	3.33	3.32	3.32	
	5.0, 7.1	14.1	5.00	5.00	4.99	
Montreal	14.36	14.24	3.20	3.21	3.22	Biotite, vermiculite—montmorillonite, kaolinite, allophane and quartz
	3.22	7.16	7.17	7.15	3.18	
	4.06, 7.16	12.6	3.34	3.34	(all very weak)	
Talparo	3.34	7.17	7.17	7.17	3.34	Kaolinite, illite, montmorillonite, montmorillonite—vermiculite, montmorillonite—illite
	7.17	10.04	3.34, 3.57	3.34, 3.57	10.02	
	4.27, 10.04		10.04	10.04		

^a The constituents are placed in decreasing order of the amounts found. They may vary from large quantities through small amounts to traces.

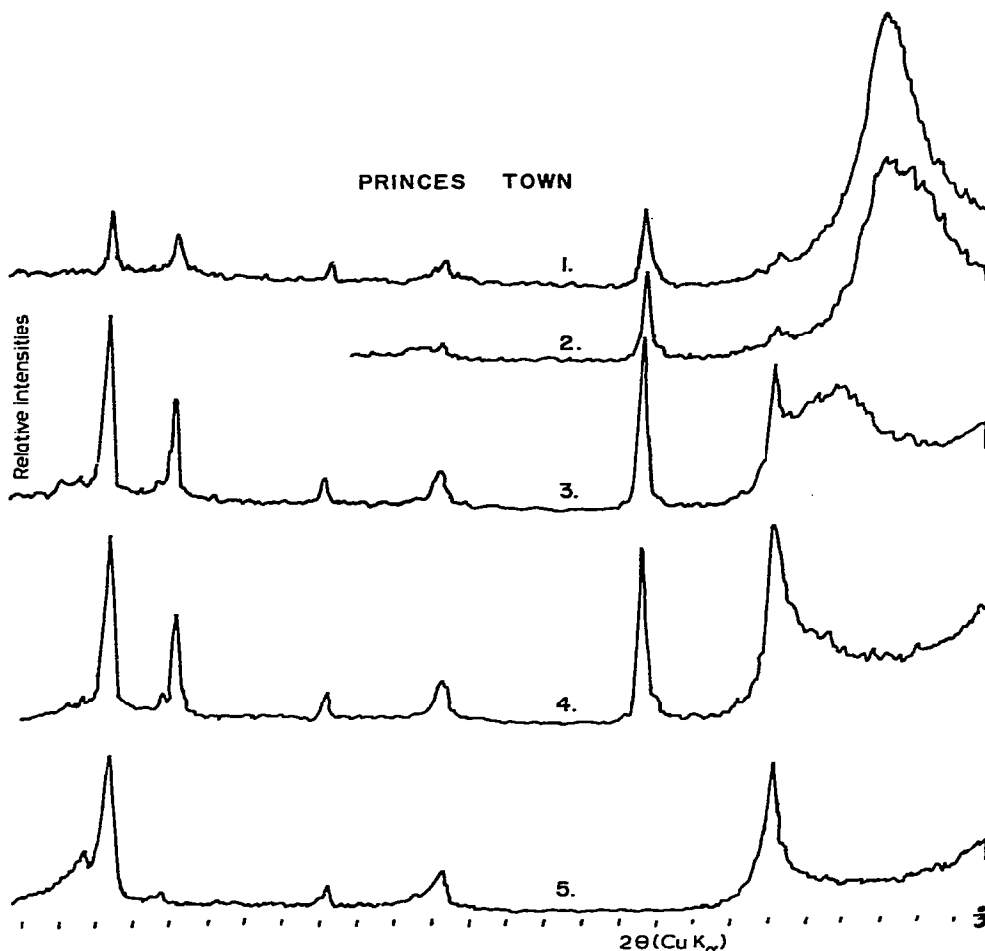


Fig. 3. X-Ray diffraction patterns of Princes Town soil-clay, Trinidad, West Indies.

both a 17.04 \AA peak and a 14.3 \AA peak which collapsed on heating to increase the intensity of the 10 \AA (first order), 5 \AA (second order) and 3.34 \AA (third order) illite reflections. The 3.34 \AA peak has increased in intensity, therefore, from the second order high reflection of quartz, and the third-order illite reflection by the superimposed third-order reflections of the contracted montmorillonoid.

The presence of weak low angle and basal reflections which are not sub-multiples of them indicate irregular interstratifications [10]. The 21.7 \AA peak for the glycolated Mg-treated and the room-temperature K-treated (21.5 \AA) peak seem to be kaolinite-vermiculite interstratification, and the 9.16 \AA peak which disappeared with heating to 550°C is due to mica-kaolinite interstratification. Harrison and Droste [11] found similar behaviour and described it as evidence for random mixing of illite and montmorillonite. The X-ray diffraction patterns (Fig. 2) show a small peak on the high 2θ angle side of the 10 \AA illite reflection between the illite and the 7.16 \AA kaolinite reflections. This peak also disappeared on glycolation and on heating to 550°C . In the glycolated clay however, several diffuse peaks

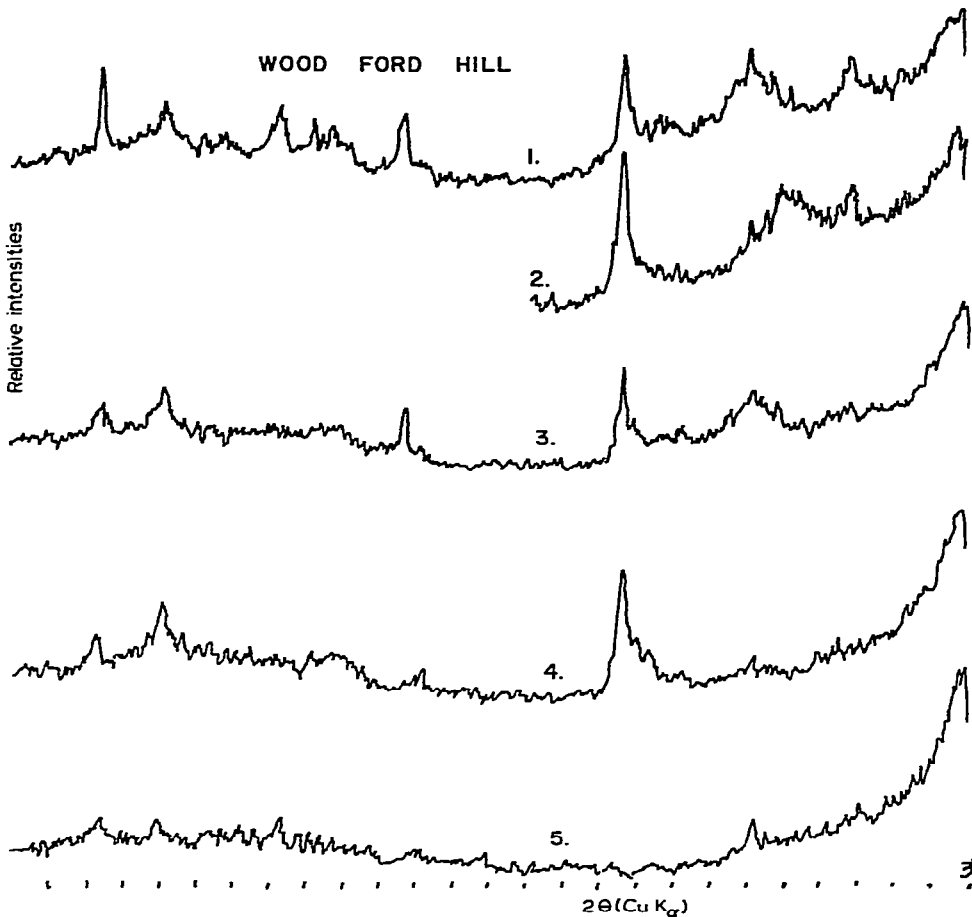


Fig. 4. X-Ray diffraction patterns of Wood Ford Hill soil-clay, Dominica, West Indies.

appeared between 17 Å and 21 Å. These all disappeared at 300°C. This evidence indicates irregular interstratifications and random mixing of illite with kaolinite as well as illite and kaolinite with vermiculite and montmorillonite.

Fitches

Fitches soil is high in montmorillonite (80%), but it also contains traces of quartz, feldspar and kaolinite. The diffraction pattern of the K-saturated sample heated to 300°C shows a broad multi-shouldered peak between 10.6 Å and 20.4 Å, indicative of montmorillonite assemblages and possible trace of irregular mica—montmorillonite interstratification. The samples exhibit peaks at ca. 4.4 Å, which indicates the presence of halloysite [10].

Princes Town

The clay minerals present in Princes Town clay are montmorillonite, kaolinite and quartz together with traces of illite and a randomly interstratified

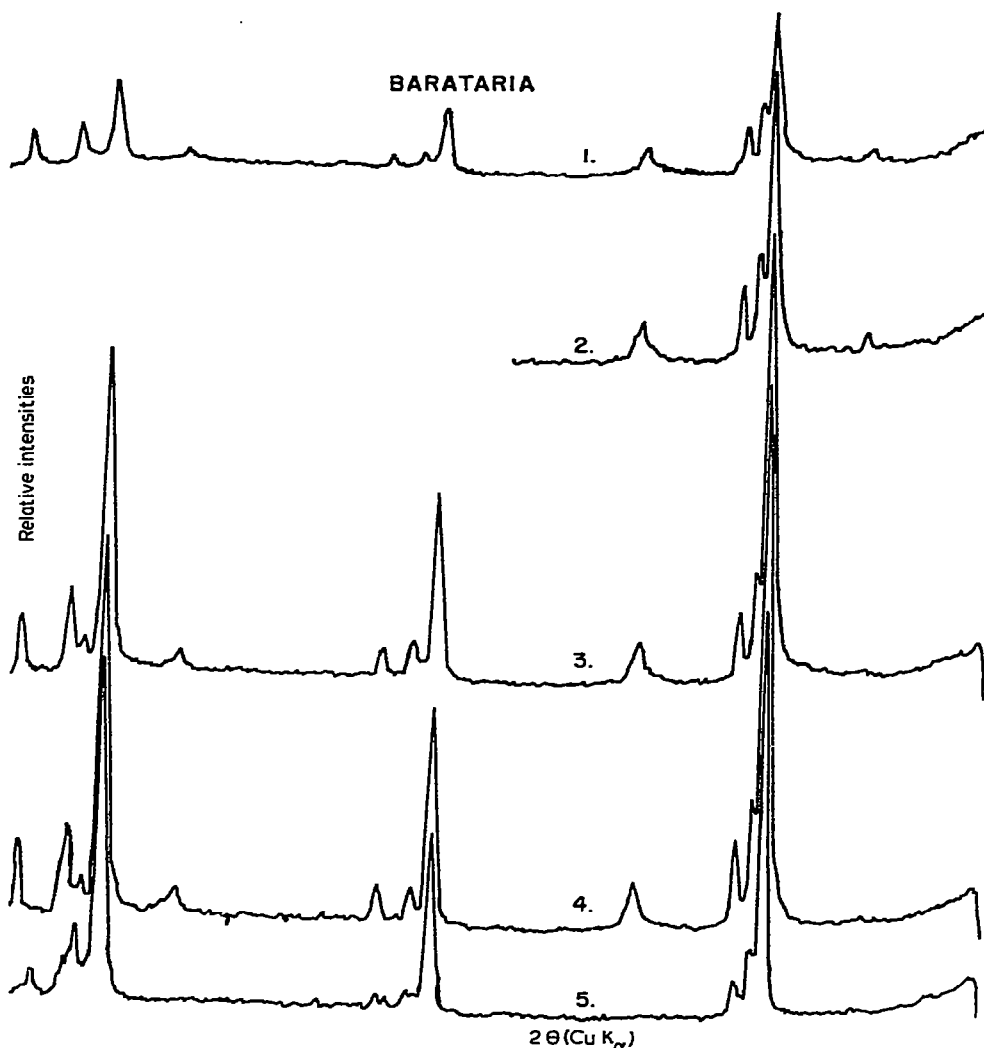


Fig. 5. X-Ray diffraction patterns of Barataria soil-clay, Trinidad, West Indies.

mixture of montmorillonite—vermiculite—illite. Several diffuse bands appeared in the low angle region between 14.4 Å and 23 Å for the glycolated sample; between 10 Å and 19 Å for the K-treated (room temperature) sample which were displaced to between 9 Å and 17 Å when heated to 300°C. Only the 10 Å peak which is due to the presence of contracted montmorillonite, contracted vermiculite, and illite remained at 550°C (Fig. 4). The gradual collapse of the montmorillonite and vermiculite structures and the increasing intensity and symmetry of this peak are supporting evidence for a random mixing of these minerals [11].

Capitol

The Capitol clay contains essentially kaolinite (ca. 85%), as is evident from the high intensities of the first- and second-order reflections at 7.3 Å

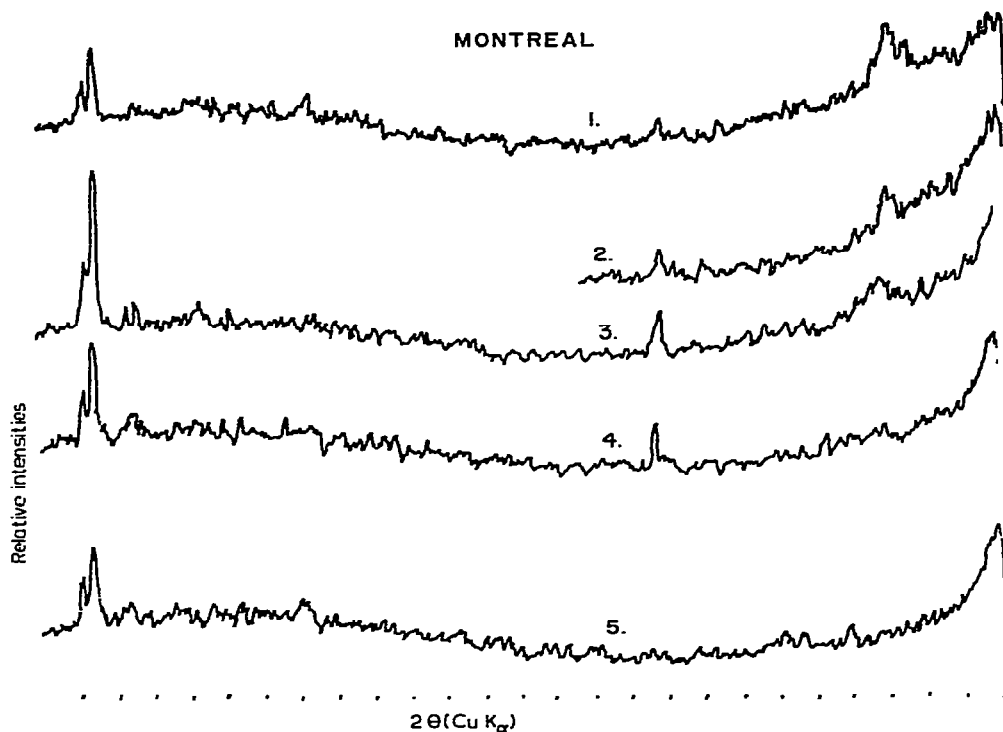


Fig. 6. X-Ray diffraction patterns of Montreal soil-clay, St. Vincent, West Indies.

and 3.6 Å, which disappear as the crystalline kaolin structure is destroyed on heating to 550°C (Table 2). This kaolin mineral appears to be a mixture of antigorite and *meta*-halloysite. The K-treated clay shows a double-peaked reflection at 7.2–7.3 Å, which converge into a single peak of increased intensity at 7.25 Å when heated to 300°C. It also contains some montmorillonite, a trace of quartz, and indications of either an alkali feldspar, metastable tridymite or allophane to account for the relatively high reflections at 4.10 Å on heating the K-treated sample to 550°C [12–14]. It is believed to be due to the presence of allophane, since it is known that allophanic soils occur extensively on volcanic islands such as Grenada [15]. The average intensity peak at 4.06 Å which disappears at 550°C confirms the presence of gibbsite in this clay. This is the only soil among those studied which shows the presence of this mineral.

Wood Ford Hill

This clay is quite similar to that of Capitol clay. It is also essentially kaolinitic (~85%), but does not contain any gibbsite. Other minerals found in it include illite, chlorite, vermiculite, quartz, halloysite, interstratified chlorite–vermiculite and a trace of allophane. The presence of disorganized mica and a randomly interstratified illite–vermiculite–chlorite species are indicated by the several diffuse peaks in the broad band between 10 Å and 14.0 Å present in all but the 550°C diffractogram (Fig. 6).

La Plaine

This is a poorly-crystalline mineral soil consisting of some kaolin minerals, traces of illite and of montmorillonite—vermiculite—chlorite interstratified with kaolinite. The most interesting feature of this clay is the strong double-peaked reflection at 4.06 Å and 4.10 Å resulting from the presence of allophane and minerals whose structures are thermally stable at 550°C (Table 3). This soil is known to contain allophane [15] which gives a peak at 4.06 Å that does not disappear on heating to 550°C. Although the X-ray spectra show that there is little crystalline material present, the DTA thermograms and the X-ray spectra indicate that, mineral percentage-wise, this clay is high in kaolinite, and contains some illite, vermiculite—chlorite—montmorillonite assemblages. There is, however, some doubt concerning the exact identity of this assemblage, because:

(i) the first-order reflection of cristobalite is at 4.04 Å at room temperature [16] and is at 4.15 Å after heating to 500°C [17];

(ii) the second-order tridymite and feldspar reflections are between 4.04 Å and 4.08 Å;

(iii) the third-order kaolinite reflection is at 4.13 Å;

(iv) micas show a low intensity reflection around 4.10–4.12 Å; while,

(v) the seventh-order *d* spacing for a regular alternating vermiculite—chlorite interstratified system is at 4.06 Å.

Barataria et al.

Barataria peat, River Estate, St. Augustine, Montreal and Talparo clays contain mica. Barataria contains essentially illite as is seen from the strong intensity peaks for first-, second- and third-order reflections at 10 Å, 5 Å, ... 3.4 Å ... The peaks at 9.71 Å, 9.25 Å, 4.8 Å, 4.6 Å, etc., indicate the presence of disorganized mica assemblages which, from their positions relative to illite and kaolinite, are probably due to two different mica—kaolinite randomly interstratified (disordered) minerals (Fig. 5). This clay also contains some kaolinite and quartz and traces of feldspar and vermiculite.

River Estate clay contains biotite and illite, some kaolinite, montmorillonite, disordered vermiculite—chlorite and a trace of quartz. The interstratified vermiculite—chlorite assemblage was deduced mainly from the splitting of the 14 Å peak to the doublet at 12 Å and 14 Å on heat treatment to 550°C. The vermiculite contracts at this temperature while the chlorite structure remains unchanged [18].

St. Augustine loam is essentially illite but contains a small amount of disorganized mica, irregularly interstratified mica—kaolinite. This deduction is based on the appearance of a small peak between 9.2 Å and 9.7 Å which persists on glycolation and heating to 300°C but disappears at 550°C. It also contains some kaolinite, vermiculite, chlorite and quartz.

The diffractograms of Montreal clays indicate that it is high in non-crystalline minerals (Fig. 6). Feldspar is identified from the strong 3.2 Å reflection. Alkali feldspar, vermiculite, montmorillonite, kaolinite, irregular interstratifications of vermiculite—montmorillonite and vermiculite—chlorite are also

present. Like La Plaine clay, it contains allophane, as is evident from the 4.10 Å peak which resists heat of 550 Å (Table 4).

Talparo clay, unlike Montreal clay, is high in crystalline clay minerals. The diffraction patterns show several sharp reflections which are first- and higher-order diagnostic reflections of kaolinite, illite, quartz, interstratified montmorillonite-vermiculite, montmorillonite-vermiculite-illite, and kaolinite-illite. A summary of the results obtained for these soils from the Eastern Caribbean, West Indies, is presented in Table 5.

CONCLUSION

Positive identifications of primary clay mineral species and mixtures of secondary layer-silicate minerals of some West Indian soils have been made. The results obtained by this method confirm the results obtained by the DTA method [1-3], and in addition identified allophane, feldspar and gibbsite; gave a clearer differentiation of the montmorillonite-vermiculite, kaolinite/halloysite, and illite/disordered mica species; indicated regular and irregular interstratifications of clay mineral assemblages, and quartz when present in small quantities.

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