MINERALOGY OF LEDA CLAY

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ABSTRACT

The paper describes the mineralogical composition of some samples of Leda clay. The clay is a predominantly marine deposit of post-glacial age which accumulated in an arm of the ocean existing from 12,000 to 8,000 B.P., known as the Champlain Sea. Analyses of five clay samples were made by examination on the petrographic microscope, by *x*-ray diffraction and by differential thermal analysis; chemical analyses were also made.

Four of the samples contain mixed-layer clay minerals with an expansible component. All samples contain a significant proportion of chlorite as well as a layer structure silicate with a spacing of about 10Å. Primary minerals such as quartz, feldspar and amphibole comprise a significant proportion of the clay ($< 2\ \mu m$) as well as of the non-clay fractions. Smaller amounts of pyroxene, biotite, garnet, staurolite, zircon, titanite, andalusite, magnetite and hematite were also found.

Three of the samples were quite similar in mineralogy, a fourth sample did not contain two of the mixed-layer minerals, and a fifth sample showed the greatest differences. The minerals are thought to be largely detrital and differences may reflect differences in original source area.

INTRODUCTION

The object of the present paper is to describe the mineralogical composition of some samples of Leda clay. This sediment is a predominantly marine deposit of post-glacial age which accumulated in an arm of the ocean known as the Champlain Sea. Some of the upper layers of sediment may have been redeposited under fresh-water conditions (Gadd 1963). The Leda clay, which often contains considerable quantities of silt and sand-sized particles, is of variable thickness and exceeds 200 ft in places.

A considerable amount of work has already been done on the mineralogy of the Leda clay, some of the most recent being by Brady & Dean (1966), Soderman & Quigley (1965), Brydon & Patry (1961) and Allen & Johns (1960).

The Leda clay is of geotechnical as well as mineralogical interest because of its unusual engineering properties. Flow-type slides are common; the soil is classified as "quick clay" and is defined as being highly "sensitive" (Crawford 1968). Sensitivity is the ratio of the undisturbed strength

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of soil to its remoulded strength at the natural moisture content. Leda clay also commonly shows a considerable irreversible shrinkage on dewatering (Bozozuk & Burns 1960).

Theoretical work indicates that the behaviour of quick clays may in part be dependent upon changes in soil fabrics (Gillott 1970; Penner 1963; Quigley & Thompson 1966). In addition, engineering properties of soils are known to be affected by the chemistry of the pore fluids and by the mineralogical composition of the material. It is hoped that future studies of fabric and composition will throw light on the geotechnical properties of Leda clay.

SAMPLES

Five samples were studied and their locations are given as a footnote to Table 1. The fabric and engineering properties of these same samples have been previously reported (Gillott 1970).

Samples were disaggregated, dispersed with NaOH and separated by gravity or centrifugal sedimentation into three fractions: coarser than 2 μm e.s.d.*, 2 to 0.2 μm and <0.2 μm. The coarser than 2 micron fraction was itself separated into two fractions by sink-float techniques in a liquid of density 2.89 gm/cc. The clay fractions were separated into duplicate batches and flocculated in $MgCl_2$ and $CaCl_2$ solutions respectively and rinsed. Results of the mechanical analysis are shown in Table 1.

Mineralogical composition of the various fractions was determined by optical examination on the petrographic microscope, by *x*-ray diffrac-

TABLE 1. LEDA CLAY : MECHANICAL ANALYSIS

	A	B	C	D	E
> 2 μm	25.6	29.1	35.1	20.8	72.9
< 2 μm	74.4	70.9	64.9	79.2	27.1
2–0.2 μm	47.2	35.2	28.5	42.1	16.0
< 0.2 μm	27.2	35.7	36.4	37.1	11.1

A. Ottawa sewage plant, depth 45 ft.
B. Ottawa sewage plant, depth 53 ft.
C. Walkley and Russell Rd., Ottawa, depth 33 ft.
D. C.F.S. Gloucester, Ontario, depth 16 ft. 8 ins.
E. St. Joachim de Tourelle, Québec, depth 20 ft.

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* e.s.d. = equivalent spherical diameter.

tion analysis and by differential thermal analysis; chemical composition was estimated by wet chemical methods.

ANALYSIS BY X-RAY DIFFRACTION AND OPTICAL MICROSCOPY

Samples in the "as-received" condition and as fractionated material were examined on the petrographic microscope and by the powder method of *x*-ray diffraction analysis by means of a Hilger and Watts diffractometer, a Nonius Guinier quadruple-focusing camera and a Philips 11.46-cm diameter camera.

Mineralogical analyses were not generally made on a quantitative basis because of well-known technical difficulties (Pierce & Siegel 1969; Gibbs 1967; Taylor & Norrish 1966; Norrish & Taylor 1962; Brindley & Kurtossy 1961; Copeland & Bragg 1958). Quantitative mineralogical data for some minerals are shown in Table 2. The percentage of quartz in the "total sample" was derived by an *x*-ray diffraction procedure using the method of known additions (Brown 1961, p. 495) and the 4.26Å *x*-ray reflection.

Non-clay fractions

The coarser than 2 μm fractions of samples A, B, and D contained no appreciable heavy residue; this residue amounted to about 7% of the coarse fraction of sample E and about 17% of the coarse fraction of sample C. The mineralogical composition of corresponding fractions of different samples was quite similar. The heavy residues (sp.gr.>2.89) of samples C and E were subdivided into two fractions, one magnetic and the other non-magnetic. The dry powder was passed through a Frantz isodynamic separator using a current of 1 amp and slope of 10°.

The concentrates were examined on the petrographic and binocular microscopes, and powder *x*-ray diffraction photographs were taken. In the magnetic fraction, the most abundant mineral was green hornblende; in lesser amounts were pyroxenes, biotite, chlorite, garnets, and a few crystals of staurolite. In the non-magnetic fractions, zircon, sphene and andalusite (sample C) crystals were recognized. Amphibole may also be present. Magnetite and hematite were present in the heavy residue prior to magnetic separation.

The light residues (sp. gr. < 2.89) of all five samples were composed of alkali feldspar, plagioclase feldspar and quartz. Samples were stained (Reeder & McAllister 1957), and the proportions of the chief minerals present were estimated semi-quantitatively by optical microscopy (Table 2). Assuming that the feldspars in the Leda clays were derived from regionally metamorphosed terranes, the plagioclase will be in the low temperature state (Smith 1956, p. 63). Values of lattice spacings obtained by measurements of x-ray diffraction photographs taken with a Guinier camera indicate that much of the plagioclase present falls within the oligoclase-albite composition range.

Clay fractions

Microscopic examination of the 2 to 0.2 μm fractions indicated that feldspars, quartz and amphiboles together with aggregations of clay minerals were present.

TABLE 2. LEDA CLAY : MINERALOGICAL COMPOSITION PER CENT

Sample	Fraction	Calcite *	Albite *	Orthoclase **	Plagioclase **	Quartz ***
A	Total	2.1	24.2			18.5
	>2 μm ; Sp. G. < 2.89			20	15	65
	2–0.2 μm		27.9			
	< 0.2 μm		23.2			
B	Total	3.3	22.9			15.3
	>2 μm ; Sp. G. < 2.89			25	20	55
	2–0.2 μm		27.9			
	< 0.2 μm		17.8			
C	Total	5.9	22.0			21.4
	>2 μm ; Sp. G. < 2.89			30	15	55
	2–0.2 μm		26.5			
	< 0.2 μm		15.0			
D	Total	2.4	24.7			13.8
	>2 μm ; Sp. G. < 2.89			35	15	50
	2–0.2 μm		28.6			
	< 0.2 μm		16.9			
E	Total	8.2	11.2			38.9
	>2 μm ; Sp. G. < 2.89			10	25	65
	2–0.2 μm		8.5			
	< 0.2 μm		7.4			

* Computed from chemical analysis
** Optical microscopy
*** X-ray diffraction

X-ray diffraction analysis was carried out on oriented samples and on randomly oriented samples. Oriented samples were prepared by allowing a suspension of the clay to slowly evaporate on a glass slide in a high-humidity atmosphere. Difficulties with this technique, due to size segregation of minerals during gravity settling on the slide, were not found in this work.

Powder photographs were used in identifying non-clay minerals and in attempts to recognize polytypic modifications between the clay minerals; they were also used to establish whether the clay minerals were dioctahedral, trioctahedral or were represented by members of both groups.

Samples A and B had an almost identical mineralogical composition. Sample C was very similar in composition, but the diffraction patterns of the clay minerals were weaker and the lines broader, suggesting that the clay minerals were less well crystallized. There were some differences in mineralogical composition between these samples and sample D and differences were even more marked in the case of sample E. In samples A, B and C, a significant proportion of the total composition was made up of mixed-layer clay minerals, whereas minerals of this sort were absent or present in much smaller quantities in the other two samples (D, E). Relatively sharp diffraction maxima were recorded from both size-fractions of sample E, suggesting that the layer structure silicates were better crystallized in this sample from St. Joachim de Tourelle than in the others.

Samples from the Ottawa sewage plant (A, B) and Walkley Road, Ottawa (C) contained at least two mixed-layer clay minerals. One of these had a long spacing of about 32Å and was most plentiful in the 2 to 0.2 μm fraction. The other mineral had a spacing of about 12Å and was present in about equal amounts in both the 2 to 0.2 μm and the $<$ 0.2 μm size fractions (Fig. 1).

An x-ray reflection with a spacing from about 32Å could result from combined scattering of a mixed-layer combination of vermiculite and montmorillonite. The peak at a position corresponding to a spacing of about 30Å on the diffractogram of the 2 to 0.2 μm material at 95% relative humidity, showed a slight shift to about 32Å, and was enhanced and sharpened following treatment with glycerol. The strong probability of interference with reflections from other layer structure silicates, feldspars and other minerals, made it almost impossible to identify an integral series of higher orders of the 32Å reflection. It was, therefore, not certain whether or not the mineral had a regular mixed-layer structure. The long spacing was not in itself proof of regularity though the sharpness of the peak seemed to favour this interpretation. Heating at 300°C and 600°C caused the 32Å reflection to vanish, and the peak at about 10Å to be enhanced considerably (Fig. 1). Both vermiculite and montmorillonite display structural collapse to somewhat less than 10Å when heated. No reflection corresponding to a spacing in the 20Å range was detected;

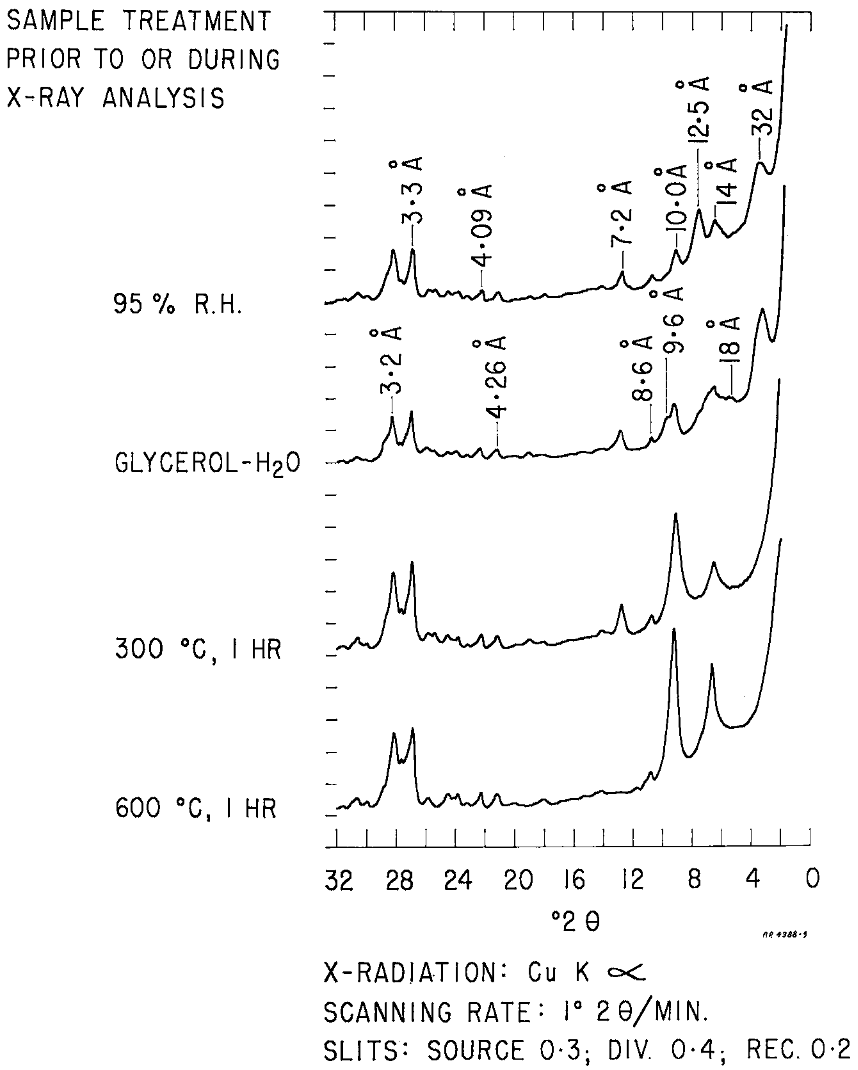


Fig. 1. X-ray diffractograms of sample A . 2–0.2 μm fraction.

possibly in the collapsed state odd-order reflections were absent because of space group criteria. Exactly out-of-phase scattering from two very similar layer structures would have led to cancellation of a possible x-ray reflection. The possibility that the mineral represents a regular mixed-layer containing montmorillonite and chlorite seemed to be excluded since no reflection occurred at an angle that corresponds to a spacing of either 24Å or 12Å after heat treatment.

Diffractograms of both the 2 to 0.2 μm, and less-than-0.2 μm fractions of samples A, B and C showed a strong reflection at a position corresponding to an interplanar spacing of about 12.5Å (Fig. 1). This shifted to about 13.5Å after the sample had been treated with glycerol. A reflection corresponding to a variable spacing of this value could have resulted from combined scattering from the 001/001 planes of a random mixed-layer mineral composed of about 20 to 25% of 10Å layers and 75 to 80% of 14Å layers. Such a mineral could correspond to an intergrowth of mica/vermiculite in these proportions (Brown 1961, p. 406). Consideration of curves of mixing functions given by Brown (p. 404) suggested that the other possible combination, mica/montmorillonite, is much less probable.

A variable spacing material, probably a random mixed-layer mica/montmorillonite, was present in samples A to D but not E. It gave peaks at about 18Å and 9.6Å on diffractograms of glycerol-treated samples and caused marked enhancement of the peak at about 3.34Å on treatment with ethylene glycol (Weaver 1956, p. 205). Heat treatment of the sample at 300°C and 600°C caused structural collapse and marked enhancement of the 10Å reflection (Fig. 1).

All samples contained a significant proportion of chlorite in the clay fractions. There was a peak at about 14Å and an integral series of orders on diffractograms. The 14Å peak showed marked enhancement, and higher orders vanished, following heat treatment of the sample at 600°C (Fig. 1). Kaolinite can often be recognized in the presence of chlorite by slow scans over the angular range, in which the second order kaolinite reflection and fourth order chlorite reflection occur (Biscaye 1964). In the five Leda clay samples, this technique showed little or no kaolinite to be present; just a slight tail occurred on the peak at 3.54Å of samples A, B, C and D, and sample E gave a sharp peak with no tail.

Diffractograms of all samples showed a moderate peak corresponding to a spacing of about 10Å and integral series of higher orders indicate the presence of a non-swelling mica-type clay mineral. This mineral has generally been described as illite by previous workers. It was the most abundant clay mineral in sample D and it made up a significant proportion of the clay mineral fraction in all the other samples. At 95% relative humidity, the sharpest *x*-ray reflections were registered from sample E. In all samples except that from St. Joachim, E, heat treatment enhanced the 10Å reflection. This was attributed to collapse of the expanding layer structure minerals to an interplanar spacing of about this value.

In summary, samples A, B and C were quite similar in clay mineralogical composition; sample D differed in that the 32Å and 12.5Å phases were absent. The sample from St. Joachim de Tourelle, E, showed the greatest differences in that swelling clay minerals were absent, and there were sharp diffraction peaks suggesting that the layer structure silicates were better crystallized. It is possible that the minerals in this sample were derived from an Appalachian source whereas the other samples may have had a provenance in the Canadian Shield or in Paleozoic rocks derived elsewhere (Brady & Dean 1966, p. 40).

Powder patterns of samples A to D were complex and this precluded attempts to identify polymorphic forms of the mica-type minerals. A weak reflection at 1.503Å suggested that some of the clay minerals are dioctahedral. Positive recognition of trioctahedral minerals was prevented by a powder line at 1.54Å, which was attributed to quartz. Sample E gave a better powder pattern, which when analyzed, suggested that the mica polytype was a 2*M*-muscovite.

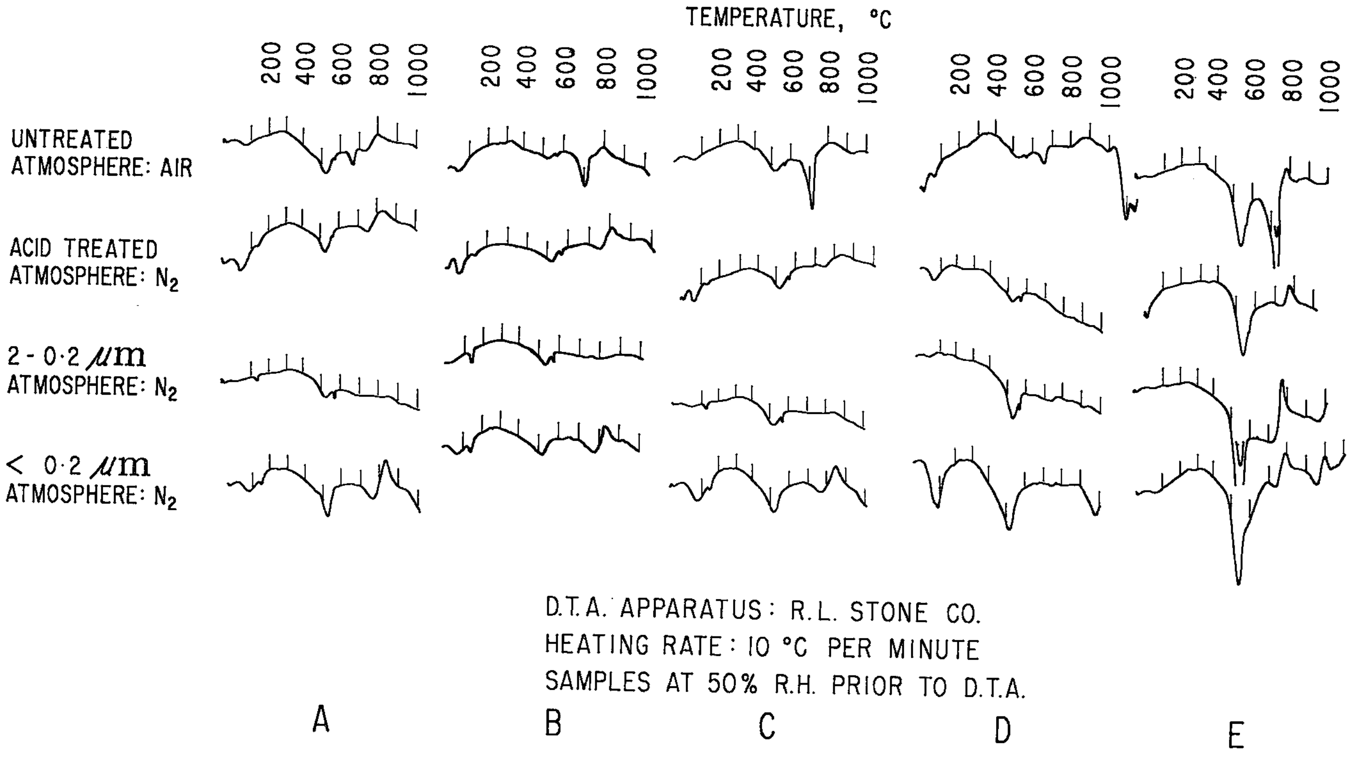
As was reported by previous workers (Brydon & Patry 1961), the Leda clay examined contained primary minerals within the clay-size range (< 2 μm e.s.d.). Quartz, amphibole and feldspar were present in all samples.

Differential Thermal Analysis (dta)

Differential thermal analysis was carried out on five fractions of each sample. These fractions were: (1) total sample (untreated, unsized material); (2) acid-treated, unsized material; and fractionated material of particle size ranges; (3) > 2 μm; (4) 2 - 0.2 μm; and (5) < 0.2 μm. A selection of the thermograms is shown in Fig. 2.

When untreated material was analyzed in an air atmosphere, thermograms displayed a broad exotherm in the 200° to 400°C temperature range; this was attributed to oxidation of organic material. When analyzed in an N_2 atmosphere, this material was found to have the same thermograms except for this exothermic loop.

Thermograms of unsized, untreated material, displayed an endothermic peak at about 650° to 725°C, which was attributed to decomposition



D.T.A. APPARATUS: R.L. STONE CO.
HEATING RATE: 10 °C PER MINUTE
SAMPLES AT 50% R.H. PRIOR TO D.T.A.

FIGURE 2 THERMOGRAMS OF LEDA CLAY

of calcite. The relative size of this peak on thermograms of different samples agreed with the relative proportions of calcite calculated from the chemical analyses (Table 2). The largest amount was present in sample E and the minimum quantity in samples A and D (Fig. 2). Other peaks on thermograms of the unsized samples matched those on thermograms of size-fractionated material where, however, they were often seen to better advantage.

The thermogram of the coarser than 2 μm fraction (not shown in Fig. 2) generally showed few inflexions apart from a strong peak at about 573°C, which was attributed to quartz. Sample E, however, gave a large endotherm between 500° to 600°C, that was interpreted as evidence of mica-type minerals which exceed 2 μm e.s.d.

Thermograms of the 2 to 0.2 μm size fraction showed a sharp endotherm at about 573°C attributed to quartz but were otherwise similar to thermograms of the $<$ 0.2 μm size fractions; the fines generally gave more intense peaks (Fig. 2).

An endothermic peak or peak system at about 100°C was attributed to loss of surface moisture; its greater intensity on thermograms of the $<$0.2 μm fractions supports this conclusion because they should have the largest specific surface area and greatest sorptive capacity.

Thermograms of all clay-grade material showed the major endotherm at about 525°C which was attributed to dehydroxylation of the layer structure silicates. As several mica-type minerals and chlorite are known to be present, this peak may well be complex. This was partly confirmed on the thermogram of the $<$ 0.2 μm fraction of sample E where there is a distinct change in slope of the high temperature portion of the 500 - 600°C peak (Fig. 2E).

A peak system at 750 - 800°C consisting of an endotherm followed by an exotherm occurred on thermograms of the fines of all samples except D. It was attributed to decomposition of chlorite though similar inflexions are often present on thermograms of vermiculite. The gap between the endotherm in the 500° to 600°C region and the 800°C exotherm is a characteristic of the thermal breakdown of 14Å, rather than 7Å, chlorites (Phillips 1963). Endothermic loss of hydroxyl at two different temperatures from a chlorite may reflect a difference in binding energy between OH ions in the brucite layer and in the silicate layer.

The endotherm-exotherm peak system at 900°C to 1000°C, commonly reported on thermograms of most clay minerals, was seen clearly only on the thermogram of the St. Joachim sample (Fig. 2E).

CHEMICAL COMPOSITION

The chemical composition of the unfractionated samples, and of the 2 to 0.2 μm and < 0.2 μm fractions is shown in Table 3. The values for the unfractionated material are quite similar to one another and to those reported by Clarke (1924, p. 631) for an average clay. In sample E, the much larger amount of coarser-than-2 μm materials (Table 1), which includes a significant proportion of silt-sized quartz, is reflected chemically in the proportion of silica which reaches a maximum in the unfractionated material; this material also contains the minimum quantity of alumina and potash, which reflects the smaller relative proportion of potash-containing layer-structure silicates. In the other four samples, there are only small differences in the proportion of potash between the fractionated and unfractionated material.

Comparison with Clarke's (1924) "average values" showed that in unfractionated Leda clay samples A to D, there was a higher than average percentage of iron, and the iron concentration was even higher in the < 0.2 μm fraction. The proportion of iron was close to "average" in sample E (unfractionated) and the < 0.2 μm fraction contained no more than the 2 to 0.2 μm fraction. Lambe & Martin (1955) concluded that "chlorites in natural clays can cause engineering problems from their high compressibility [and] water sensitivity". They also found that high-iron chlorites differed from low-iron chlorites in their plastic properties. The MgO content was somewhat higher than "average" and showed little change either between fractions or from one sample to another.

In all samples the presence of calcite in the unfractionated material was reflected in the CaO analyses. In sample E there was, however, a much sharper drop in CaO content between the unfractionated material and the size fractions (which were acid-treated) than in the other four samples. By far the largest proportion of CaO, evidently, was present as calcite in sample E, whereas there were significant amounts of lime in non-carbonate minerals in the other four samples. The CO_2 analysis indicated the relative amount of calcite in the five samples; the calculated proportion of this mineral present in the total sample is shown in Table 2.

The Na_2O content in the unfractionated material represented the combined total of sodium contained in the pore solution and in the minerals of which the clay was composed. The contribution from the solution phase was fairly small, and after correction, the Na_2O content of the first four samples was not much changed. Sample E had only about half the quantity of Na_2O found in the other samples. In all samples the minimum amount of Na_2O was found in the < 0.2 μm fraction.

TABLE 3. LEDA CLAY : CHEMICAL COMPOSITION PER CENT **

Sample	Fraction	SiO_2	Al_2O_3	FeO *	MgO	CaO	Na_2O	K_2O	L.O.I.	TiO_2	CO_2	P_2O_5	Total
A	Unfractionated	52.3	19.3	7.9	4.8	3.4	3.3	3.8	5.2	—	0.91	0.15	101.1
	2–0.2 µm	55.8	17.0	7.4	4.2	2.8	3.0	3.8	2.6	1.3	—	—	97.9
	< 0.2 µm	46.8	19.5	9.3	5.1	1.8	2.7	3.0	6.8	1.2	—	—	96.2
B	Unfractionated	52.2	18.1	7.8	5.0	4.7	3.1	3.8	4.8	—	1.5	0.17	101.2
	2–0.2 µm	55.2	16.1	7.7	4.2	3.5	3.3	3.7	1.8	1.7	—	—	97.5
	< 0.2 µm	47.0	16.9	11.4	6.0	2.0	2.1	3.8	4.5	1.8	—	—	95.5
C	Unfractionated	53.4	17.8	7.4	4.2	5.2	2.7	3.6	6.2	—	2.6	0.17	103.3
	2–0.2 µm	59.1	13.1	6.4	4.5	3.1	3.1	3.6	2.0	1.5	—	—	96.4
	< 0.2 µm	49.8	18.2	9.5	5.0	1.7	1.8	3.5	4.9	1.4	—	—	95.8
D	Unfractionated	48.1	24.1	8.1	4.6	3.5	3.2	3.4	5.8	—	1.0	0.08	101.9
	2–0.2 µm	56.9	15.8	7.0	3.9	3.1	3.4	3.2	2.3	1.4	—	—	97.0
	< 0.2 µm	48.5	19.3	9.6	5.2	1.5	2.0	2.9	4.8	1.2	—	—	95.0
E	Unfractionated	55.1	16.4	5.7	4.4	5.3	1.4	2.5	8.1	—	4.3	0.08	103.3
	2–0.2 µm	52.5	18.1	8.7	4.1	0.6	1.0	3.4	5.6	2.1	—	—	96.1
	< 0.2 µm	44.7	24.0	8.2	4.5	0.2	0.9	4.9	7.5	1.0	—	—	95.9
F	Average clay * *	54.28	14.51	6.39	2.99	5.04	1.21	2.12		0.42	3.53	0.09	

* Fe'' not differentiated from Fe'''

** Analyzed by : Analytical Section, Division of Chemistry, N.R.C., Ottawa

** Data from Clarke, 1924, p. 631

— Indicates no analysis available

This suggests that much of the Na_2O is contained within the albite molecule as x-ray evidence indicates that even though feldspar is present it forms a smaller proportion of the < 0.2 µm fraction than of the coarser fractions. X-ray diffraction analyses indicated that the plagioclase falls within the albite-oligoclase composition range. The assumption was made that the Na_2O present in the solid was contained within the albite molecule. Na_2O was recalculated as albite to give an approximate estimate of the proportion of plagioclase (Table 2). There are at least two possible sources of error in this estimate of the plagioclase content. Chemical analyses reported by Deer, Howie & Zussman (1962) give an average Na_2O-content of 1.2% for illites, 0.24% for smectites and 0.07% for chlorites. If appreciable Na_2O is present in the clay minerals this calculation would overestimate the plagioclase-content. Conversely the plagioclase-content may be underestimated since no allowance was made for the inclusion of Ca-ions which might have been present in the feldspar to the amount represented by the oligoclase composition range.

All samples contained small amounts of phosphorus and titanium due to the presence of apatite and rutile. The mass spectrograph showed trace amounts of Ba, Ce, Zr, Sr, Rb, Cu, Mn, Cr, V, S, P, F, N and B.

The chief compounds present in the pore solutions are shown in Table 4. Engineering properties of clays are affected by the chemistry of the pore solutions; for example plasticity is affected by the ratio of monovalent to divalent cations (Penner 1965). Composition of the solutions will depend upon the original composition of the water of deposition, amount of leaching and chemical composition and properties of the minerals such as exchange capacity.

CONCLUSIONS

The five samples of Leda clay have a complex mineralogy. The clay-size fractions contain a significant proportion of primary minerals.

TABLE 4. LEDA CLAY : CHEMICAL COMPOSITION OF PORE SOLUTION

Sample	MgO	CaO	K_2O	Na_2O	Cl^-	SO_4^{--}
A	29.2	8.9	81.4	400.3	31.3	178.4
B	27.8	9.8	64.9	363.4	19.6	171.2
C	31.1	117.1	72.8	73.8	18.8	228.9
D	15.7	4.99	32.0	289.0	32.8	107.2
E	37.6	156.6	41.6	51.2	16.0	310.1

Composition in mgm per 100 gm, dried clay

This affects the engineering properties of clay soils because primary minerals have a smaller specific surface area and weaker colloid chemical properties than clay minerals. The swelling clay minerals in samples A to D may affect the shrinkage characterictics of the clays.

Some of the primary minerals present have only a low resistance to chemical weathering; since unstable minerals do occur this indicates that the sediments have suffered only very little from this form of attack. Many of the minerals are certainly detrital, though chemical weathering or diagenesis may have contributed to formation of some of the mixed-layer and expanding-lattice clay minerals. The mineralogical differences between samples A to D and sample E suggest that the material from St. Joachim de Tourelle was supplied from a source area different from the other group of samples.

Engineering properties of soils are known to be affected by the chemistry of pore solutions, by the chemical composition of the minerals and by the exchange ions and ion ratios.

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