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FABRIC OF LEDA CLAY INVESTIGATED BY OPTICAL,
ELECTRON - OPTICAL, AND X - RAY DIFFRACTION METHODS

BY

J. E. GILLOTT

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TEXTURE DE L'ARGILE LEDA ETUDIEE PAR LES METHODES
OPTIQUES, LES METHODES OPTIQUES-ELECTRONIQUES
ET LA DIFFRACTION AUX RAYONS-X

SOMMAIRE

Les données de texture, de paléosalinité et de génie sont données pour cinq spécimens d'argile Leda. On démontre que les couches de silicates dans deux des spécimens sont orientées parallèlement au lit, mais que dans les trois autres spécimens, les minéraux stratifiés ont un arrangement peu organisé. Les études par diffraction aux rayons-x démontrent qu'il n'y a pas de différence dans l'orientation des couches de silicates $7\text{\AA}$ et $10\text{\AA}$.

Les valeurs de paléosalinité obtenues par le contenu en bore indiquent que trois des spécimens ont été déposés dans une eau plus douce que l'eau marine normale. Les deux autres spécimens ont été déposés dans une ambiance marine normale. Le rapport entre les données de la texture et de la paléosalinité est assez précis malgré qu'une étude sur un plus grand nombre d'échantillons soit requis avant que la méthode puisse être évaluée d'une façon satisfaisante pour l'argile Leda.

L'enlèvement de l'humidité par quatres méthodes différentes démontre que la texture originale peut être affectée par la technique de préparation des spécimens. Les résultats indiquent que la texture résulte beaucoup plus des conditions à la période du dépôt que des conditions subséquentes au dépôt.

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FABRIC OF LEDA CLAY INVESTIGATED BY OPTICAL, ELECTRON-OPTICAL, AND X-RAY DIFFRACTION METHODS

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SUMMARY

Fabric, palaeosalinity and engineering data are given for five samples of Leda clay. It is shown that the layer structure silicates in two of the samples are oriented parallel to the bedding; that in the other three samples the platy minerals have a near random arrangement. X-ray diffraction studies showed that there was no difference in the orientation of 7Å and 10Å layer structure silicates.

Palaeosalinity values deduced from boron content indicate that three of the samples were deposited in water that was fresher than normal marine. The other two samples were deposited in a nearly normal marine environment. The correlation between fabric and palaeosalinity data is fairly good, though information on a larger number of samples is needed before the method can be satisfactorily evaluated for Leda clay.

Moisture removal by four different methods shows that original fabric can be affected by the technique of sample preparation.

The evidence is in agreement with the idea that fabric was more affected by conditions at the time of deposition than by subsequent events.

INTRODUCTION

The present paper has a two-fold objective: to describe the fabric of samples of Leda clay and attempt correlation with engineering properties and palaeosalinity; to give the results of an investigation of whether different methods of moisture removal have a detectable influence on fabric.

Leda clay, of Postglacial age, is a predominantly marine sediment that occupies much of the St. Lawrence lowland in eastern Canada. It is classified in soil engineering terminology as a "quick clay"; disturbance can convert clay of this type from a relatively strong brittle material to a viscous liquid. Such soils are said to be highly sensitive. Sensitivity is the ratio of the undisturbed strength of a soil to its remoulded strength at the natural water content. It is thought that the degree of sensitivity is related to soil fabric, which is the "orientation in space of the elements of which a rock is composed" (AMERICAN GEOLOGICAL INSTITUTE, 1960,

p.104). It is the closest English equivalent of the German word Gefüge (SANDER, 1930) and embodies also "a functional or behavioural aspect which is concerned with the directional physical properties that are a necessary correlate of geometrically regular organization of matter" (DENNIS, 1967, p.40).

Fabric data on five samples of Leda clay were obtained by optical, electron-optical and X-ray diffraction methods. Particle size analyses were carried out by pipette and hydrometer methods. Previous studies of the fabric of Leda clay have been made by PENNER (1963), QUIGLEY and THOMPSON (1966), and O'BRIEN and HARRISON (1967). There are many additional studies of clay fabric reported in the literature (MARTIN, 1966; PUSCH, 1966; SMART, 1967). Mineralogical analyses of the same samples of Leda clay will be reported separately. The study forms a part of the detailed investigations of the engineering properties of Leda clay being carried out in the Division of Building Research.

GEOLOGICAL BACKGROUND

In eastern Canada the earth's crust was depressed during the Pleistocene by the continental ice sheet. When the ice melted the area was inundated by an arm of the ocean known as the Champlain Sea, which existed about 8,000–12,000 years ago (KARROW, 1961). Streams, shoreline erosion, and ice ablation supplied detritus, which accumulated as sediment known as Leda clay. This is of variable thickness and exceeds 200 ft. in places. Some of the uppermost layers of the Leda clay may have been re-deposited under fresh-water conditions due either to release of melt-water from glacial sources or to influx of fresh water from the Great Lakes region (GADD, 1963). The Champlain Sea episode was terminated when the isostatic uplift of the land exceeded the eustatic rise in sea level. The predominantly salt water deposited sediments then came under the influence of a fresh water regime and were subjected to attack by subaerial erosion.

GEOTECHNICAL BACKGROUND

Quick clays such as Leda clay are commonly subject to flow-type landslides. These often leave pear shaped craters and the sites of ancient slides are revealed by the characteristic topography. Such scars are readily recognized on aerial photographs. Slides of this sort have resulted in wasteful and costly damage to property and sometimes in loss of life. The abrupt and serious decrease in shear resistance when the soil is disturbed has been a major focus of engineering interest.

In addition to the stability problem there are other questions of engineering concern. For example, Leda clay shows a considerable shrinkage on dewatering, and a large part of this volume decrease is not recovered on rewetting. Transpiration from trees has been held responsible for vertical ground movements (BOZOUK and BURN, 1960). Apart from shrinkage problems, compressibility

characteristics are often hard to evaluate, and there has been disagreement in the settlements predicted by computation and those found by field observation of actual structures (CRAWFORD, 1968).

The unusual engineering properties have stimulated theoretical discussions aimed at drawing a mechanistic picture to describe the behaviour of quick clays, which differ from thixotropic systems in that only a small fraction of the original strength is regained if a sample is allowed to age undisturbed after being remoulded. It is thought that fabric changes are important. Theoretical and experimental evidence indicates that clay minerals should assume an open arrangement in marine deposited clays as colloidal particles are rapidly flocculated in water with a moderate electrolyte content (LAMBE, 1953, 1958; MITCHELL, 1956; ROSENQVIST, 1962). This loose fabric tends to persist because of physical restraints that hamper reorientation of the particles even when their arrangement is out of equilibrium with the present environment. Disturbance causes breakdown of the open fabric and the particles become oriented in a more close-packed configuration. As the water content remains the same for the short time interval involved, the deposit becomes over-saturated and flows like a viscous liquid. It has been suggested that metastability of the open fabric results from a decrease in salt content of pore solutions due to leaching or diffusion (ROSENQVIST, 1953, 1962). There are probably other factors involved in the case of the Leda clay, since there is generally no correlation between salt concentration and sensitivity. An interrelationship with electrokinetic potential has, however, been demonstrated (PENNER, 1965), supporting the general conclusion of repulsive forces between particles suggested in the hypothesis. Fabric and composition may also be involved in determining the shrinkage and compressibility characteristics of Leda clay.

PALAEOSALINITY

The water in the Champlain Sea is thought to have varied in composition from near-normal marine to relatively fresh. Clay minerals like other hydrophobic colloids are flocculated by water containing a moderate concentration of electrolytes, but form a dispersion under fresh-water conditions. The salt-content of the water in the Champlain Sea, therefore, probably had an important influence on whether single crystals or agglomerations of clay minerals were the settling units at the time of sedimentation of the Leda clay. The fabric displayed by different clays would be better understood if the palaeosalinity at the time of deposition was known. This may be estimated from data derived by at least two geochemical techniques; but both have serious limitations.

Salt content of pore water has been determined for a number of samples in the Ottawa district and is generally less than 2 g/l. Because of leaching and ionic diffusion since deposition, it is improbable that these values will give either

an absolute measure or even an accurate relative indication of the salinity of the water in which sedimentation of different samples took place.

Boron content has been used as a means of estimating the palaeosalinity of water in which illitic clays were deposited. Boron is sorbed by illite (and to a smaller extent by kaolinite and montmorillonite) in proportion to the amount present, to the salinity of the water, to the specific surface area and crystallinity of the clay and other factors. Hence the boron content of marine clays is generally higher than that of fresh-water deposits (FREDERICKSON and REYNOLDS, 1960; FLEET, 1965; LERMAN, 1966; WALKER, 1968; COUCH and GRIM, 1968).

Boron content was determined for size-fractionated samples of the Leda clay studied, and values are shown in Table I. A recalculation of these results according to the method described by WALKER (1968) and WALKER and PRICE (1963) leads to the designations of palaeosalinity shown in the last column of Table I. The

TABLE I

PALAEOSALINITY DATA

<i>Sample</i> ¹	<i>Fraction size (μ)</i>	<i>Carbonate</i> ² (%)	<i>Organic carbon</i> (%)	<i>Boron</i> (p.p.m.)	<i>Palaeosalinity designation</i>
A	total	2.1	0.27		
	2 - 0.2		0.21	100	
	< 0.2		0.50	50	fresher than normal marine
B	total	3.3	0.37		
	2 - 0.2		0.54	10	
	< 0.2		0.98	50	fresher than normal marine
C	total	5.9	0.31		
	2 - 0.2		0.30	100	
	< 0.2		0.43	150	near normal marine
D	total	2.4	0.19		
	2 - 0.2		0.24	50	
	< 0.2		0.53	25	fresher than normal marine
E	total	9.7	0.24		
	2 - 0.2		0.24	250	
	< 0.2		0.40	250	near normal marine

¹ A. Ottawa Sewage Plant; B. Ottawa Sewage Plant; C. Walkley Rd., Ottawa; D.H. M.C.S. Gloucester, Ontario; E. St. Joachim de Tourelle, Quebec.

² Data are given only for the 'total sample' as carbonates are removed by acid treatment prior to separation of the 2-0.2 μ and < 0.2 μ size fractions.

quantity of boron contained in a sediment is affected by many factors, particularly by the amount already present in detrital clay minerals and possibly by the amount of soluble organic matter. The state of flocculation at the time of sedimentation

is thought to be one of the most important factors affecting clay fabric and hence palaeosalinity is likely to be of major significance. Among other things, however, fabric is also likely to be influenced by the amount of carbonates present so these values are also given in Table I.

Many of the minerals in the Leda clay are known to be detrital. Mica-type minerals derived from the igneous and metamorphic rocks of the Canadian Shield would be expected to be poor in boron (GOLDSCHMIDT, 1954). The illite from the Palaeozoic limestones and other sediments that outcrop to the south, however, would be expected to have had significant boron contents when they entered the Champlain Sea. Any boron picked up in the Champlain Sea would be added to that already present in the clay mineral structure.

The situation is further complicated by the possibility of redeposition of older Champlain Sea sediment under more nearly freshwater conditions towards the end of the marine phase. Furthermore, size fractions are not monomineralic. It seems inevitable that correlation will be imperfect between fabric data and palaeosalinity values deduced from boron-content, particularly for samples collected near the margins of the sedimentary basin. Hence, great care is required in using these results, and many more observations are needed before a satisfactory evaluation of the method can be made for Leda clay.

SAMPLES

Samples A and B were collected from a depth of 45 and 53 ft. at the site of the Ottawa sewage plant during its construction. Sample C was collected from a depth of 33 ft. at the junction of Walkley Road and Russell Road, Ottawa. Sample D came from a depth of 16 ft. 8 inches at H.M.C.S. (C.E.S.) Gloucester¹, Ontario, and sample E was collected from a depth of 20 ft. at St. Joachim de Tourelle in eastern Quebec.

The four Ontario samples were "undisturbed" blocks; the one Quebec sample was an "undisturbed" block collected from the scarp of a landslide. Some mass properties for the samples are shown in Table II. Engineering tests showed that samples A, B, C and E had been preconsolidated, and that sample D was almost normally consolidated. The value shown for sample E is an estimate. Sensitivity values and natural moisture contents are also given in Table II. Undrained shear strength varied from 0.2–2.0 tons/sq. ft., the normally consolidated sample being the weakest. The undrained shear strength of the two oriented samples (A, B) is similar to but somewhat greater than the strength of two of the randomly oriented samples C, D. The strength of the randomly oriented silt sample E is about the same as that of the two oriented samples A, B.

¹ Land-based station of Canadian Forces near Ottawa.

TABLE II.

ENGINEERING PROPERTIES

Sample ¹	A	B	C	D	E
Depth(m):	13.716	16.154	10.058	5.08	6.096
Natural water content (%)	60	45	58	70	22
Preconsolidation load ²	4.5 (4.39)	5.0 (4.88)	2.4 (2.34)	0.7 (0.68)	5 ³ (4.88)
Strength (undrained) ³	1.4 (1.36)	1.5 (1.46)	0.6 (0.58)	0.22 (0.21)	1.5 -2.0 (1.46-1.95)
Sensitivity	56	500	21	100	-
Liquid limit (%)	49	30.6	53.2	43	29.1
Plastic limit (%)	23	23.4	24.7	23.5	21.4
Plasticity index	26.3	7.2	28.5	19.5	7.7
Liquidity index	1.4	3.0	1.1	2.3	0.1

¹ A. Ottawa Sewage Plant; B. Ottawa Sewage Plant; C. Walkley Rd., Ottawa; D. H.M.C.S. Gloucester, Ontario; E. St. Joachim de Tourelle, Quebec.

² Values in tons/sq. ft.; values in parenthesis are in kg/cm².

³ Estimate

These small differences will probably not result directly from fabric differences. The plasticity index of samples B and E is low in relation to that of the other three. This may be because of the coarseness of sample E and may be a consequence of a higher ratio of monovalent to divalent cations in sample B than in sample A (PENNER, 1965). The liquidity index indicates that the natural moisture

Fig.1. Particle size analyses (hydrometer).

A. SEWAGE PLANT, OTTAWA

B. SEWAGE PLANT, OTTAWA

C. WALKLEY RD. AT RUSSELL RD.,
OTTAWA

D. HMCS GLOUCESTER, ONTARIO

E. ST. JOACHIM de TOURELLE,
QUEBECFig. 2. Transmission electron micrographs of dispersions of $< 2\mu$ fraction of Leda clay.

content was very close to the liquid limit in samples A and C; that natural moisture content was in excess of the liquid limit in samples B and D; and apparently less than the liquid limit in sample E, due possibly to desiccation of the sample. Particle size analyses by hydrometer and pipette methods show that sample A is predominantly of clay size; samples B, C and D are silty clays; and sample E is a silt (Fig.1).

Electron micrographs of dispersions of the 2–0.2 μ fraction of samples A–D and smaller than 2 μ -size fraction of sample E are shown in Fig.2. All samples contain a predominance of thin platy particles of irregular shape, presumably layer-structure silicates related to illite. In one sample, platy particles were observed that have a tendency to hexagonal outline (Fig.2D). Lath-like particles were present in smaller amount (Fig.2E). Small quantities of irregular grains and tabular particles are present, probably representing quartz, feldspar and other primary minerals reduced to the clay size range by ice abrasion.

SAMPLE PREPARATION FOR FABRIC ANALYSIS

The fabric of non-lithified materials such as clay soils may be changed drastically by remoulding, drying, or shearing. In the present work undisturbed block samples were collected, sealed in wax to retain moisture, and stored in a room held at high R.H. to decrease the possibility of drying. It is not possible to analyse the fabric of wet samples by optical or electron optical methods. The natural water, therefore, had to be replaced or removed. Four techniques were used: (1) air-drying; (2) critical point drying; (3) freeze-drying; and (4) carbowax impregnation.

Dimensional change was at a maximum in air-dried samples; in samples prepared by the other three methods dimensional change was minimized but not entirely eliminated. The true fabric of the soil in its natural, undisturbed condition is, of course, unknown but one would anticipate that fabric damage would be at a maximum in samples which showed the greatest change in dimensions. Likewise, the most satisfactory method of sample preparation would be expected to be that which induced the minimum volumetric change in the sample. By making a comparison between the fabric observed in samples prepared by the four different methods it was hoped that both the true fabric and most satisfactory method of sample preparation would become apparent.

Air-drying

Samples were dried at room temperature at 50% R.H. Leda clay shows a considerable decrease in volume when air dried (GILLOTT, 1969). Fabric damage would be expected to be at a maximum in such samples and fabric analyses were compared with the results obtained on samples dried by other methods. Dimensional change due to drying is thought to be caused by migration of moisture,

by surface tension forces and by change in surface-free energy. Increase in surface-free energy necessarily accompanies moisture removal so shrinkage from this cause is difficult or impossible to avoid if the sample has to be dried. It is possible, however, to reduce shrinkage caused by surface tension forces and moisture migration by using critical point drying and freeze-drying techniques.

Critical point drying

The critical point is defined by a temperature and pressure above which the physical properties of a liquid and its vapour are the same. As there is no longer a boundary layer, surface tension forces vanish (ANDERSON, 1950; GILLOTT, 1969). Fabric damage from these forces should therefore be much reduced if the moisture is removed above the critical point. Damage may, however, not be entirely eliminated because of inherent limitations in the method. The critical point of the pore fluids is not accurately known for at least two reasons: (a) exchange and other ions in solution will cause the physical properties of the fluid to differ from those of the pure liquid; (b) close to a surface the physical properties of a liquid deviate considerably from those of the material in bulk.

About four molecular layers of water are thought to be significantly affected. Surface tension forces are greatest in small pores, in the smallest of which it is improbable that critical point conditions will be established because of the increased importance of surface effects. The perturbation of physical properties is thought to result from increased orientation in successive layers of water molecules near the surface. This orientation is believed to be induced by two main factors: (a) hydrogen and hydroxyl bonds between the surface atoms and water molecules and between the water molecules themselves; and (b) hydration of exchange ions held at the surface.

Owing to these effects quite high temperatures and pressures may be needed to attain the critical point. This is undesirable because, apart from other reasons, mineralogical changes can be induced. In the present work water was replaced by ethyl alcohol, which has a lower critical point (H_2O : 374°C , 3266 p.s.i.; $\text{C}_2\text{H}_5\text{OH}$: 243°C , 946.5 p.s.i.). Replacement by a non-polar liquid may be even better because surface effects would presumably extend through fewer molecular layers and near-normal physical properties should exist closer to the surface. Critical point conditions would thus be more predictably induced closer to actual crystal-to-crystal junctions.

Freeze-drying

Moisture was also removed by freeze-drying. Ice glass formed by rapid cooling was removed by sublimation under vacuum. This method also has known limitations. Fabric damage may result from dimensional change caused by freezing, from recrystallization of ice glass, and from failure to freeze the last few monolayers of water because of perturbation of physical properties close to a surface (GILLOTT, 1969).

Impregnation

Moisture can be replaced by impregnation with a water-soluble compound (Carbowax 6000). Leda clay shows a small volume decrease as a result of the slow diffusion of the carbowax into the pore water to form a uniform mixture.

Samples dried by all four techniques were examined by X-ray diffraction and on the scanning electron microscope. Samples shown in Fig.4 were dried by the critical point method. Optical microscopic examination was generally restricted to carbowax impregnated samples.

Once moisture had been removed or replaced, sample preparation varied depending on the technique of fabric analysis to be used.

Thin sections for study on the petrographic microscope were prepared from carbowax-impregnated samples by a procedure similar to that described by MITCHELL (1956). The slide is made by conventional techniques, but is ground dry or in an organic vehicle instead of in water. The clay is generally bounded to the glass by a cold-setting epoxy cement. Two sections were prepared from each sample, one oriented normal to the sedimentary bedding, and the other oriented parallel to bedding.

The scanning electron microscope gives information about a surface. It is therefore essential that the surface examined should be representative of the fabric in the body of the sample. Because of the very large depth of focus in the image produced by the scanning electron microscope, the sample surface need not be perfectly flat. In this work surfaces were exposed by fracture. Samples normal and parallel to bedding were examined. Samples dried by all methods have been examined; those illustrated were dried by the critical point method and fractured after drying (GILLOTT, 1969).

Clay soils are sometimes very weak when the water has been removed and loose crystals may fall on the exposed surface. Platy particles have a high ratio of face-to-edge area, so that seen face-on they have a masking effect. This can give an erroneous impression of a greater abundance of crystals with this morphology or of a more frequent orientation in the plane of the section than is realistic. Care is required and some experience needed in recognizing such artifacts, which obscure the true fabric.

X-ray diffraction was used to evaluate quantitatively whether any difference in fabric could be detected between samples dried by the four methods. Samples were air-dried in a conditioned room at 50% R.H., 22°C; impregnated with carbowax; freeze-dried; and dried by the critical point method. Two sections oriented at right angles were cut from each sample, one parallel, the other normal to the bedding. A smooth surface for exposure to the X-ray beam was prepared by grinding. The samples to be freeze-dried were held in a jig and ground under liquid N₂ in a cold room prior to removal of the ice by sublimation under vacuum. The carbowax-impregnated samples were ground by routine procedures used in the preparation of microscopic thin sections. The air-dried samples were quite robust

and were dry-ground. Two methods of grinding were tried for the samples to be dried by the critical point method. Dry grinding subsequent to moisture removal was tried, but many samples were extremely fragile and it was difficult to obtain reproduceable results, probably because of surface irregularities and distortion. Results were better if the sample was wet-ground under alcohol, after replacement of water but prior to drying.

RESULTS OF FABRIC ANALYSES

Optical microscopy

Clay minerals are generally too small to be individually resolved in the optical microscope, but the main features of their aggregate orientation may be deduced because of their combined influence on polarized light. A microscopic thin section cut normal to a plane of preferred orientation and rotated between crossed polars displays four positions of maximum light transmission. These alternate with four corresponding positions of maximum darkness; each light and dark maximum is separated by an angular interval of 45° . A thin section parallel to the basal surfaces of oriented clay minerals shows uniformly low colours for all positions of rotation. Intensity of light transmitted remains approximately the same at all angular positions if the clay minerals are randomly oriented. In the five Leda clay samples studied preferred orientation was observed in samples A and B, being most marked in sample A (Fig.3A,B). Qualitative evaluation of the remaining three samples suggests that the fabric is close to random (Fig.3C,D,E). The significant amount of angular, silt-sized grains in sample E is evident on the micrograph (Fig.3E).

Scanning electron microscopy

The scanning electron micrographs (Fig.4) show that many particles have an irregular outline and platy morphology, as was found on examination of size-fractionated dispersions on the transmission electron microscope. Lath-like platy crystals and irregular equiaxial silt-sized grains occur in subordinate amount, the silt grains being most abundant in the St. Joachim sample. In the two Ottawa sewage plant samples more crystal edges are visible (Fig.4A,B, normal) than in the micrographs of the corresponding samples oriented parallel to the bedding, where proportionately more crystal faces are visible. This supports the conclusion based on optical microscopy that the layer-structure silicates in these two samples have preferred orientation parallel to sedimentary bedding. Nonetheless, orientation appears far from perfect and crystals that lie at large angles to the dominant direction are quite common in both normal and parallel-oriented samples. In both views of the other three samples, edge and face surfaces are visible in about equal proportion, as is to be anticipated when platy particles are stacked in an open, near-random type of configuration. Some platy minerals can be seen

Crossed Polarizers

Fig.3. Optical micrographs of Leda clay.

Fig.3. D,E. (Legend see p.144.)

adhering to the surfaces of silt grains (Fig.4E, parallel), although the extent of such coatings is hard to evaluate and some silt grains have relatively clean surfaces (Fig.4A, normal).

In kaolinite compacted in the laboratory, SLOANE and KELL (1966) described a random "bookhouse" and oriented "parallel packet" fabric. SMART (1967) reported turbostratic structure and particle orientation in failure zones, and a "stepped face-to-face" arrangement was found by SMALLEY and CABRERA (1969). Such structures were not conspicuous in Leda clay, although natural analogues may be represented by what appeared to be "books" of mica-type minerals sometimes partially separated along crystal cleavages; in some instances a marked

Normal to Bedding

Parallel to Bedding

A. SEWAGE PLANT, OTTAWA

B. SEWAGE PLANT, OTTAWA

C. WALKLEY RD. AT RUSSELL RD., OTTAWA

Fig.4. Scanning electron micrographs of Leda clay.

Normal to Bedding

Parallel to Bedding

D. HMCS GLOUCESTER, ONTARIO

E. ST. JOACHIM de TOURELLE, QUEBEC

Fig.4. D,E. (Legend see p.146.)

preferred orientation of platy minerals parallel to a surface of weakness, making a large angle with sedimentary bedding in the Leda clay, has been observed.

X-ray diffraction

A quantitative evaluation of clay mineral orientation was made by X-ray diffraction using a modified version of the procedure previously employed by ODOM (1967).

X-ray diffraction data were recorded at 4-minute angular intervals (20) by incremental steps using a Hilger and Watts diffractometer. A North American Philips X-ray generator was employed. Data recorded on punched paper tape were processed on an I.B.M. 360 computer. Integrated areas were derived for two peaks corresponding to an interplanar spacing of $10\text{\AA}$ and $7\text{\AA}$, respectively. The values were used to calculate the "fabric indices" from the following expression:

$$\text{Fabric index} = V/(P + V)$$

where: V = area of a basal diffraction peak from the perpendicular section;
 P = area of the same diffraction peak from the parallel section.

Theoretically the fabric index can range in numerical value from 0 for perfect preferred orientation to 0.50 for a completely random fabric. In practice when the fabric is close to random the index may exceed 0.50 because of statistical variation of the X-ray result about the true value.

The large shrinkage caused by air-drying suggests that in air-dried samples fabric relations would be different from those in samples dried by the other techniques. This suggestion was examined by comparing results statistically by an analysis of variance procedure (Tables III, IV). The comparison showed that there

TABLE III

X-RAY DIFFRACTION FABRIC INDICES

Drying technique	Sample ¹ :	A	B	C	D	E	TM ²	Fabric index evaluation	
								index	preferred orientation
Air dried	10Å	0.30	0.31	0.38	0.34	0.48		0.00	
	7Å	0.33	0.37	0.37	0.38	0.49			very good
	mean	0.315	0.34	0.375	0.36	0.485	0.348	0.10	
Carbowax	10Å	0.14	0.29	0.48	0.40	0.61			good
	7Å	0.17	0.20	0.45	0.40	0.58		0.20	
	mean	0.155	0.245	0.465	0.40	0.595	0.316		fair
Freeze dried	10Å	0.25	0.24	0.48	0.46			0.30	
	7Å	0.27	0.28	0.48	0.39				poor
	mean	0.26	0.26	0.48	0.425		0.356	0.40	
Critical point	10Å	0.14	0.31	0.45	0.53				very poor
	7Å	0.23	0.29	0.50	0.57			0.50	
	mean	0.185	0.30	0.475	0.55		0.378		
Sample mean		0.229	0.286	0.449	0.434				

¹ A. Ottawa Sewage Plant; B. Ottawa Sewage Plant; C. Walkley Rd., Ottawa; D. HMCS Gloucester, Ontario; E. St. Joachim de Tourelle, Quebec.

² Technique mean.

is a very significant difference between the fabric of the different samples as indicated by their mean from all four treatments. This confirms the conclusions of the qualitative optical and electron optical analyses. This distinction was masked, however, in the air-dried samples A-D. In these it appears that the technique of air-drying has obscured the true fabric relations. The result implies, rather surprisingly, that in samples A and B the degree of preferred orientation is decreased

TABLE IV

ANALYSIS OF VARIANCE¹

<i>Effect</i>	<i>df</i>	<i>F</i>	<i>Significance (%)</i>
Sample (S)	3	93.32	99.9
Technique (T)	3	4.96	95
Spacing (Sp)	1	1.00	N.S.
S × T	9	8.98	99
S × Sp	3	0.86	N.S.
T × Sp	3	7.07	99

¹*df* = degrees of freedom; *F* = variance ratio; N.S. = not significant. The higher the value of *F* the more significant is the effect.

by drying, whereas randomness is diminished somewhat in samples C and D. Apart from the air-dried samples, the fabric indices indicate a fair-to-good degree of preferred orientation of the platy minerals parallel to the bedding in samples A and B. Samples C, D and E have a fabric which is close to random.

No significant difference exists between the fabric indices of the minerals with a spacing of 10Å and 7Å, respectively. Evidently the chlorite and "illite" clay minerals have a similar arrangement. A significant interaction was demonstrated between sample and technique and between technique and spacing; the meaning of this is not easy to visualize.

The technique of sample preparation does influence results obtained, as is shown by their means in Table III. The analysis of variance in Table IV proves that their differences were significant at the 95% confidence level. (This value would only occur 1 time in 20 by chance.) As the true fabric is unknown, it is not possible to say which of the methods of sample preparation was most satisfactory. The absence of a clear distinction between the fabric of air-dried samples A and B versus C and D suggests, however, that air-drying is more likely to give suspect results.

DISCUSSION

The optical and scanning electron micrographs and X-ray diffraction data show that there is fair-to-good preferred orientation of the layer-structure silicates in the two Ottawa sewage plant samples. In the other three samples the fabric is close to random.

At high pressures and on shear planes where considerable movement has occurred layer-structure silicates tend to assume a preferred orientation. In many sediments it has been found, however, that there is no systematic correlation between depth and degree of preferred orientation (GIPSON, 1966). Shales and

mudstones occur in close association in rocks at least as old as Carboniferous (BOSWELL, 1961). Factors other than compaction also influence clay mineral orientation. It appears that the most important variables include particle size of the minerals, nature of exchange ion, concentration of interstitial electrolyte, acidity, temperature, organic content and, probably most significant, the fabric developed at the time of, or shortly after, deposition (MEADE, 1964). The platy minerals in clays that accumulated in the deflocculated state tend to be preferably oriented in planes parallel to the bedding. Clay mineral orientation also tends to improve with increase in organic content of the sediment. Conversely, a loose, open arrangement of particles is expected in clays deposited under flocculating conditions. Increase in quantity of silt-size particles and the presence of more than about 10% of carbonate minerals also favours disorder (ODOM, 1967). Particle size distribution is given in Fig. 1 and the quantity of carbonates in the total sample and of organic carbon in the size fractions is shown in Table I.

Four of the five samples of Leda clay contain a rather small quantity of carbonate minerals and silt-sized particles. The fifth sample contains almost 10% carbonates and includes sufficient coarse particles to be classed as a silt; it is also different in clay mineralogy. In all five samples the organic carbon¹ content is small, being less than 1%; and tends to be concentrated in the very fine fraction. It seems most likely that carbonates, organics and coarse particles, had only a minor influence on fabric though in the St. Joachim, Quebec, sample (E) the considerable quantity of silt may have favoured a disordered clay mineral orientation.

The preconsolidation load shows that all of the samples have been consolidated only under low pressures and that one sample (D, H.M.C.S. Gloucester, Ontario) is almost normally consolidated. The two Ottawa sewage plant samples (A, B), in which the clay minerals show preferred orientation, have been more preconsolidated than samples C and D, which have a random fabric. It is improbable, however, that the magnitude of this difference is sufficiently large to account for the difference in fabric.

The palaeosalinity data deduced from boron content indicates that three of the samples (A, B, D) were deposited in water fresher than normal marine, so that deflocculating conditions may have existed at the time of sedimentation. This may account for the oriented fabric observed in the two Ottawa sewage plant samples (A, B), but fails to account for the random fabric of the H.M.C.S. Gloucester, Ontario, sample (D). Clay minerals can be flocculated by water, which contains a good deal less electrolyte than that found in normal ocean water. Hence although the "fresher than normal marine" designation of salinity deduced from the boron data may be correct it is still possible that the electrolyte content of the water was high enough to cause flocculation of the clay minerals during deposition

¹ 'Organic carbon' does not include carbon in carbonates.

of the Leda clay. Palaeosalinity data are not sufficiently precise at this time for a positive distinction to be made between flocculating and non-flocculating conditions at the time sediments were deposited, particularly as it is not possible to take into account the influence of many of the other variables that affect fabric. Palaeosalinity designations suggest that sedimentation of the two remaining samples (C: Walkley Rd., Ottawa, and E: St. Joachim de Tourelle, Quebec) took place in a near-normal marine environment, so that flocculating conditions at the time of deposition would be indicated. Both samples have a random fabric, so that the two sorts of data are in agreement. In general, there is fairly good correlation between the fabric and palaeosalinity data, though many more observations are required before confidence can be placed in the method.

Engineering, fabric and geochemical results support the contention that a flocculating or deflocculating condition in the environment of deposition was the main factor responsible for the fabric now observed in these samples of Leda clay.

A close correlation between fabric data and engineering properties has not been attempted. Many factors affect strength and deformation characteristics, among them composition of minerals and pore fluids, and differences in particle size, shape, and orientation. Perhaps the most important factors are the nature of intercrystalline junctions and the effective area of interparticle contact resulting from the amount of overconsolidation of the clay.

CONCLUSIONS

(1) Sample preparation can affect original fabric and give spurious results. It appears that air-drying causes distortion of the original fabric of Leda clay.

(2) Some samples of Leda clay contain phyllosilicates, which are preferably oriented parallel to the bedding. Other samples have an essentially random fabric.

(3) There is no detectable difference between the orientation of 7Å and 10Å layer-structure silicates.

(4) Palaeosalinity values deduced from boron content are subject to uncertainty because of inherent limitations. Nonetheless, the fabric data of the Leda clays examined correlate fairly well with the palaeosalinity derived by the boron method. Further work is required to verify this suggestion.

(5) The evidence supports the contention that fabric was more affected by conditions at the time of deposition than by subsequent events such as consolidation history.

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