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MINERALOGY OF SENSITIVE CLAYS FROM QUEBEC

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ABSTRACT

X-ray diffraction, X-ray fluorescence, thermogravimetry and selective dissolution analyses were performed on six samples of sensitive clays from Quebec. The techniques employed provide improved quantitative data for the crystalline components and an approximate assessment of the amorphous matter. The main layer silicates are illite and chlorite, with subordinate quantities of vermiculite; the total layer-silicate content is generally less than 10%. Quartz and feldspar minerals are abundant; the plagioclase is of a sodic nature. An association between the carbonate minerals and the >2μm fraction is observed. Selective dissolution analyses reveal an important component (15-30%) which is more readily soluble than the crystalline residue but which cannot be identified by XRD; this is interpreted as being amorphous matter with some hornblende. The mineralogical results suggest that the interaction of primary mineral particles and amorphous matter plays an important role in the formation of sensitive clays.

SOMMAIRE

Six échantillons d'argiles sensibles du Québec ont été analysés par diffraction- et fluorescence-X, thermogravimétrie et dissolution sélective. Ces techniques fournissent des données quantitatives améliorées pour les constituants cristallins et une estimation de la proportion de la matière amorphe. Les principaux phyllosilicates sont l'illite et la chlorite; la vermiculite est moins importante. En tout, moins de 10% des échantillons sont des phyllosilicates; le quartz et les feldspaths abondent, et le plagioclase est plutôt sodique. On observe une relation entre les carbonates et la fraction >2μm. Les analyses par dissolution sélective révèlent une fraction importante (de 15 à 30%) plus soluble que le résidu cristallin, mais que l'on ne peut identifier par diffraction-X; nous l'interprétons comme un mélange de matière amorphe avec un peu de hornblende. D'après ces analyses minéralogiques l'interaction des particules cristallines primaires avec la fraction amorphe jouerait un rôle important dans la formation d'argiles sensibles.

(Traduit par la Rédaction)

INTRODUCTION

In the St. Lawrence River valley of eastern Canada clay soils occur which are extremely prone to slope failure. An initial landslip often is followed by retrogression and gravitational remoulding which transforms the clay into a viscous slurry. The degree to which clay can be transformed by remoulding is given by the sensitivity value (Terzaghi 1944). These sensitive clays are commonly referred to as quick clays, although the terms Leda or Champlain clays are also used (Gadd 1975).

The engineer's approach to this problem is one of classical soil mechanics when the behavior of the clay is monitored under laboratory conditions or in specially prepared field situations. However, until the mineralogy and chemistry of the clays are understood, the mechanism for the solid/liquid transformation cannot be modelled satisfactorily, and the most effective methods of stabilization cannot be utilized.

Several theories of sensitivity have been proposed. Rosenqvist (1946, 1953, 1966) envisaged a clay-mineral system in which the particles flocculated into an open "card-house" structure. Subsequent removal of the cations by leaching increased the effective electrical charge on the clay-mineral particles, thereby creating a metastable structure. Smalley (1971) and Cabrera & Smalley (1973) proposed a primary mineral system of clay-sized primary mineral particles (≃2μm) in which the short-range nature of the interparticle bonds was of foremost importance. The interparticle bonds in either model may be augmented by deposition of cementitious material which will assist in the formation of a brittle fabric. The object of this paper is to provide chemical and mineralogical data from which an accurate sensitivity model can be formulated.

REVIEW OF MINERALOGICAL DATA

Authors repeatedly refer to Canadian sensitive clays as being a product of leaching, where a dispersed system is created from a flocculated system by the removal of salts (Bird 1972; Kezdi 1974; Parkes & Day 1975; Peck *et al.* 1974). All the authors assume that clay minerals, which respond to a change in pore-water chemistry, are present in quantities sufficient to have a controlling influence on the system as a whole. Scott (1974) states that both Scandinavian and Canadian quick clays "consist mainly of illites and chlorites". This opinion seems to have originated in 1957 after three samples of clay were examined independently at the Department of Agriculture (Canada) and the Massachusetts Institute of Technology; both institutions recognized illite as the main constituent (Eden & Crawford 1957). "Before this work was done, a rather general opinion existed that Leda clay was composed of inert rock flour with little, if any, clay mineral contents" (Eden & Crawford 1957).

Peck *et al.* (1951), discussing the character of the clay from St. Thuribe, Quebec, quoted the results of differential thermal analyses obtained by Grim *et al.* (1937). It was found that the material consisted largely of very fine quartz grains with little mica and possibly a trace of montmorillonite. A rock-flour constitution for these clays was regarded as logical, since they were derived from Canadian Shield rocks during a glacial era in which physical erosion would have predominated. Only in some areas, such as in the Ottawa Valley where glaciers moved some distance over Paleozoic rocks, would clay minerals be an important component (Gadd 1956).

The mineralogical results which have been published since the late 1950's do not provide a consistent basis for a clay-mineral model. Allen & Johns (1960) described three relevant Quaternary samples; all had abundant quartz and hydrous mica, with subordinate chlorite and vermiculite. Feldspars, amphiboles, calcite and dolomite also were recorded.

Brydon & Patry (1961) thoroughly examined Leda clays from the Ottawa area. They reported no marked differences in mineralogy from one sample to another at a given site, or from one site to another. "The most notable feature was that quartz, feldspars, amphiboles, micas and chlorites were found in the clay fractions as well as in the silt and sand fractions", and that the micas were not necessarily more abundant than the feldspars (Brydon & Patry 1961). Expanding-lattice minerals present in most of their samples could not be identified precisely. Amphibole and feldspar were identified even in size fractions less than 0.2μm and 0.08μm, and on this basis Brydon & Patry concluded that the samples had undergone little chemical weathering. Quantitative manometric determinations of the calcite and dolomite contents (Skinner *et al.* 1959) revealed an association between the total carbonates and the $>2\mu$m size fractions. Calcite seemed to predominate, although not in all cases (Brydon & Patry 1961).

TABLE 1. MINERAL ANALYSES OF THE <2μm FRACTION OF SENSITIVE CLAY FROM OTTAWA (AFTER SODERMAN & QUIGLEY 1965)

Illite (including illite interstratified with chlorite)	55-60%
Chlorite (estimated)	10-15%
Vermiculite	5-10%
Pseudo-chlorite* (citrate-extractable chlorite)	5-10%
Montmorillonite (estimated)	5%
Quartz and feldspar (estimated)	10-20%

*Soderman & Quigley (1965, p. 181) describe pseudo-chlorite as "chlorite formed by weathering processes in which mobile Al and Fe hydroxide cations fix the vermiculite sheets together".

Liebling & Kerr (1965) concluded that quartz, albite, microcline and muscovite were predominant in the sand and silt fractions of quick clays, and that hornblende, biotite and calcite also occur in some samples. The principal minerals in the $<2\mu$m fraction are illite, chlorite, vermiculite and montmorillonite.

Soderman & Quigley (1965) studied a sample of sensitive clay from Ottawa using X-ray diffraction and DTA. Their results for the $<2\mu$m fraction are presented in Table 1. Brady & Dean (1966) investigated more than forty samples of clay and shale from Quebec, some of which were sensitive. They observed a suite of minerals apparently similar to that recognized by Liebling & Kerr (1965), and concluded that illite and chlorite were more abundant than quartz and feldspar.

Gillott's (1971) detailed mineralogical analyses of five samples of Leda clay revealed the same suite of minerals that had been recorded by previous investigators. Gillott (1971) confirmed the observation by Brydon & Patry (1961) that the systems had undergone little chemical weathering.

Suggestions have been made that considerable amounts of amorphous matter are present in Canadian sensitive clays (McKyes *et al.* 1974). Selective dissolution of this material indicated that it constituted 11-12 wt. % of the whole soil. Chemical analyses of the amorphous matter extracted indicated that SiO_2 and Fe_2O_3 were the main components with smaller amounts of Al_2O_3.

X-ray diffraction analyses of untreated sensitive clays were made by McKyes *et al.* (1974) and the results are shown in Table 2. The errors in the total mineral percentages were attributed to amorphous matter coating the particles. A repeat of the analysis after removal of the amorphous matter (Segalen 1968) revealed an apparent enhancement of the primary mineral contents (Table 2). It seems that some physicochemical constraints are active which allow the amorphous matter to adhere to the primary mineral particles in preference to the layer silicates. If amorphous matter is common to all sensitive clays then all previous quantitative X-ray diffraction analyses may have underestimated the primary mineral components.

ANALYTICAL TECHNIQUES

The differences of opinion or of interpretation expressed by the various investigators are most prominent with respect to the $<2\mu m$ fractions. The $>2\mu m$ fractions can be studied by more direct analytical techniques and consequently there is good agreement among much of the mineralogical data.

The $<2\mu m$ fraction constitutes approximately 50% of sensitive clays. This fraction undoubtedly contributes to the geotechnical properties, but quantitative mineral assessment is difficult. In order to gain a comprehensive understanding of the mineralogy, several analytical techniques should be employed. In the past, X-ray diffraction was used extensively; thermal analysis was used only to provide supplementary information (Soderman & Quigley 1965; Brady & Dean 1966; Gillott 1971). The lack of agreement among data for the $<2\mu m$ fractions will be, to some extent, a function of analytical technique and sample preparation. The methods of mounting specimens and the drawbacks of XRD as a quantitative technique are discussed by Pierce & Siegel (1969) and Scott (1973). It also is apparent that direct comparison of XRD data may not be possible because of a variation in the amorphous matter contents from one sample to another.

TABLE 2. MINERAL ANALYSES (AFTER MCKYES *et al.* 1974)

	St. Vallier clay		St. Louis clay	
	untreated	treated	untreated	treated
quartz	7.0%	13.0%	6.0%	8.0%
feldspar	7.5	15.0	6.5	14.0
illite and mica	23.0	29.0	18.0	30.0
hornblende	9.0	32.0	9.0	39.0
chlorite	3.0	3.5	4.0	4.5
	49.5%	92.5%	43.5%	95.5%

A somewhat neglected technique which would seem to overcome the problems associated with amorphous coatings is thermal analysis. Indeed, thermal analysis emphasizes the minerals which are thermally unstable (*i.e.*, the layer silicates) and allows their careful analysis without interference from the thermally inert components. Of the techniques available, thermogravimetry has the advantage of giving weight changes due to decomposition reactions directly, which is most suitable for mineralogical assessments (Keattch & Dollimore 1975; Bentley 1976).

Thermogravimetry alone does not permit one to draw mineralogical conclusions for the whole system because the number of peaks is frequently less than the number of components. However, for quantification of the thermally unstable

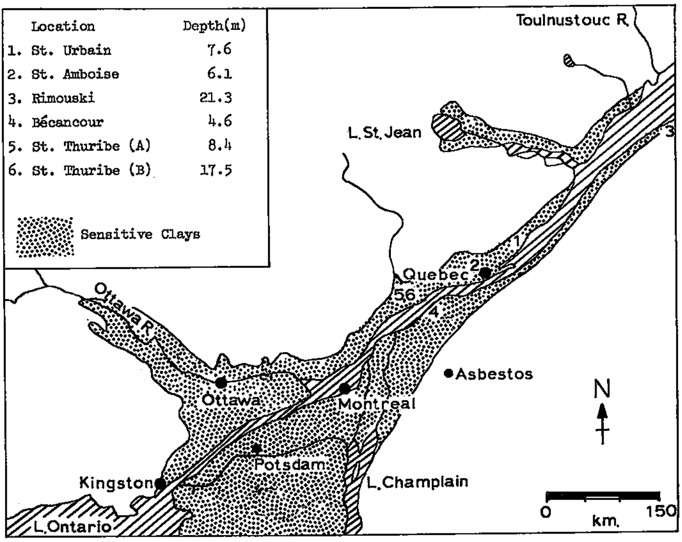


FIG. 1. The distribution of sensitive clays in the St. Lawrence Lowlands, showing locations of the samples.

TABLE 3. PARTICLE-SIZE DISTRIBUTION, DETERMINED BY GRAVITATIONAL SEDIMENTATION – EXPRESSED IN PERCENTAGE BY WEIGHT

	<2µm	>2µm
St. Urbain	39 wt. %	61 wt. %
St. Amboise	23	77
Rimouski	40	60
Becancour	41	59
St. Thuribe (A)	33	67
ST. Thuribe (B)	31	69

components the usefulness of thermogravimetry is undeniable (Smalley *et al.* 1975), especially when used in conjunction with temperature and mineral standards (Smalley *et al.* 1977).

SAMPLES

Six samples of sensitive clay from Quebec were studied (Fig. 1). The samples were disaggregated, dispersed ultrasonically and separated by gravitational sedimentation into two fractions: coarser and finer than 2µm. Results of the mechanical analyses are shown in Table 3. Mineralogical and chemical compositions of the various fractions were determined by X-ray diffraction, X-ray fluorescence, TGA, and selective dissolution analysis.

X-RAY DIFFRACTION

The instrument used was a Philips PW 1310 diffractometer which emitted Cu$K\alpha$ radiation. Smeared mounts were used for the <2µm fractions. X-ray diffraction was used to qualitatively identify the mineral species present in the samples. However, the relative intensities of the peaks allowed grading which may be interpreted in a semi-quantitative manner: grade Ab, abundant; grade P, present and grade Tr, trace (Table 4). Plagioclase recorded in the samples was of a sodic nature. Orthoclase also was recognized in many samples; however, a quantitative assessment of these feldspar minerals was obtained by selective dissolution analysis.

TABLE 4. MINERAL SPECIES IDENTIFICATION BY XRD

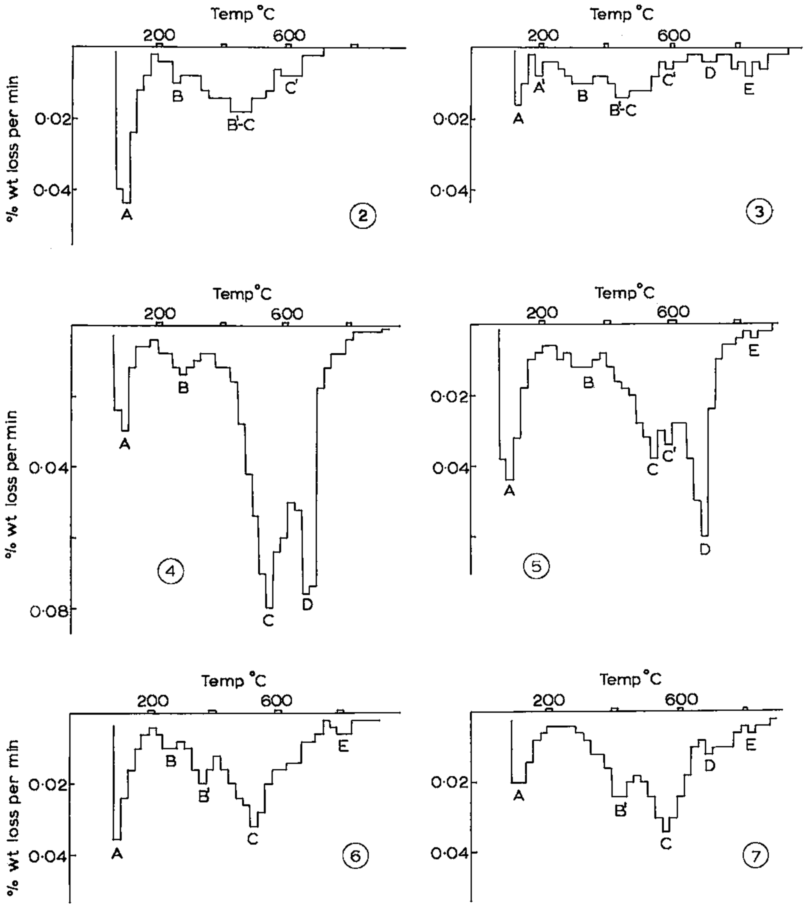
	Illite	Chlorite	Kaolinite	Vermiculite	Hornblende	Calcite	Dolomite	Quartz	Feldspar
St. Thuribe (A)									
>2µm	P	P	Tr		P	Tr	P	Ab	Ab
<2µm	P	P	Tr		P			P	P
St. Thuribe (B)									
>2µm	P	Tr			P			Ab	Ab
<2µm	P	P			Tr		Tr	P	P
St. Urbain									
>2µm	P	Tr			P			Ab	Ab
<2µm	P	Tr			Tr			P	P
St. Amboise									
>2µm	Ab	Tr			P		P	Ab	Ab
<2µm	P	Tr			Tr		Tr	P	P
Rimouski									
>2µm	P	P		Tr	Tr	P	P	Ab	P
<2µm	Ab	Ab		P		Tr		Ab	Tr
Bécancour									
>2µm	P	P	Tr		P	P	P	Ab	Ab
<2µm	P	P	Tr		P			P	P

Ab - Abundant P - Present Tr - Trace

THERMOGRAVIMETRY

The International Confederation for Thermal Analysis (ICTA) standardization committee has prepared a comprehensive list of suggestions for reporting thermogravimetric data (McAdie 1967) and these are followed in this article. Paragraph numbering follows the ICTA formulation.

1. Substances investigated: (i) Sensitive clay samples from eastern Canada. (ii) American Petroleum Institute (API) standard minerals.
2. The sensitive-clay samples were supplied by the Department of Natural Resources, Quebec. The API standard minerals were supplied by Wards Mineral Suppliers and their chemistry has been reported by Molloy & Kerr (1961).
3. Heating rate: 4.7°C/minute. The range was from room temperature (≃20°C) to 1000°C. The temperature scale tended to depart from linearity below 100° and above 850°C.
4. Furnace atmosphere: air at atmospheric pressure.
5. Sample container: "Royal Worcester" porcelain crucibles (diameter 29mm and depth 19mm).
6. Identification of the abscissa: time and temperature were recorded simultaneously.
7. Identification of final products: XRF analyses were performed for each sample, and results are presented here as elemental oxides.
8. Reproducibility of weight change: for the natural clays a reproducibility of ±10% was recorded; there also was fluctuation of the reaction interval temperatures within ±25°C. Because these variations are a function of the inhomogeneity of the samples, the results presented are an average of at least two analyses.
9. XRD and TG analyses of standard minerals allowed the thermal events in the sensitive clays to be identified.
10. Instrumentation: the thermobalance used was a Stanton-Redcroft TR-02.
11. The weight-change axis on the DTG plots is presented as a percentage weight loss per minute.
12. The DTG curve was constructed by plotting the weight change during a predetermined time period; the five-minute interval of the thermobalance timer made this the most convenient in-



FIGS. 2-7. DTG results for whole-soil samples: 2. St. Urbain; 3. St. Ambroise; 4. Rimouski; 5. Bécancour; 6. St. Thuribe (A); 7, St. Thuribe (B).

terval. The system was calibrated at intervals during the test program by the use of potassium hydrogen phthalate as a temperature standard (Smalley *et al.* 1977).

The sensitive-clay samples revealed five distinct thermal events; these are referred to as A-E for convenience, and are discussed separately. Temperatures of occurrence are A, 100°; B, 350°; C, 580°; D, 750°; E, 820°C; these represent the mid-points of the reaction interval in each case. There are also three minor thermal events, denoted by the symbols A^1, B^1 and C^1, which require discussion. Figures 2-7 show the DTG plots for the whole-soil samples.

THERMAL EVENTS: A-E

Peak A

Peak A seems to be due to adsorbed water, and is usually associated with the $<2\mu m$ fractions. The $>2\mu m$ material had been dried at 105°C and the relatively non-adsorbent larger particles had not readsorbed a significant amount of water between drying and testing. The adsorbent materials in the $<2\mu m$ fractions retained and readsorbed sufficient water to make an obvious A peak. Although one would expect a peak due to adsorbed water to be symmetrical and the weight loss to cease at 122°C (Newkirk 1960), the A peak in many of the DTG curves has an extended temperature interval up to approximately 200°C. This event may be due to the gradual evaporation of water from the interstices of the clay fabric, or to the release of water from the amorphous matter present (Mackenzie 1957, p. 102).

Where a distinct A^1 event (100-200°C) occurs it is probably due to the dehydration of some expanding-lattice clay mineral. There is some correlation with the XRD analyses, which indicates that the reaction interval may be due to the decomposition of vermiculite.

The dehydration of vermiculite occurs in three stages: the first two, at ≃150° and 540°C, involve the loss of the interlayer water, and the third, at ≃840°, is due to dehydroxylation. The first stage, which probably represents peak A^1, involves the removal of 94% of the interlayer water. The chemical formula of vermiculite, $(Mg,Fe,Al)_3(Si,Al)_4O_{10}(OH)_2 \cdot 4H_2O$ (Fleischer 1975), shows that the loss of interlayer water involves a 14.7% weight loss, of which 13.8% can be attributed to peak A^1. The second-stage loss of 6% of the interlayer water overlaps with reaction interval C, and this is taken into consideration when peak C is used for quantitative assessment.

Peak B

Gillott (1971) showed that thermograms of untreated Leda clay in an air atmosphere displayed a broad exotherm in the 200-400°C range; this was attributed to organic material. Experiments using hydrogen peroxide treated samples have confirmed event B as being of an organic nature, with a temperature range of 250-375°C. Organic materials necessarily undergo a large percentage weight loss during thermal decomposition; the size of the B peak from samples in this study suggests that the organic content is small, usually less than 0.1%.

In many samples peak B is closely associated with another thermal event B^1, the peak temperature of which occurs in the 370°-420°C range. This peak commonly merges with adjacent ones to form peaks B-B^1 and B^1-C. If the adjacent thermal events B and C are identified, then reaction interval B^1 can be defined precisely. Peak B^1 appears to take the form of a broad event ranging from about 200-500°C. This reaction interval is not consistent with any of the minerals recognized by XRD; however, it is a very large, significant peak. Event B^1 may represent the decomposition of the amorphous matter in these systems.

From DTA experiments of amorphous ferric oxide gels, Mackenzie (1957) noted a very strong exothermic peak in the 250-500°C region, which was attributed to the crystallization of hematite. This possibility is supported by the red color of the heated thermobalance specimens, and the fact that the red coloration intensifies with decreasing particle size of the samples.

An iron–silicon–hydroxyl chemistry has been suggested for this amorphous matter (McKyes *et al.* 1974). Crystallization and/or dehydration of this complex would be accompanied by the loss of water molecules and a corresponding loss in weight. As the precise chemistry of this material is not known, other methods must be used for its quantitative assessment.

Peak C

The temperature of the peak C reaction interval is indicative of a non-expanding layer-lattice mineral; this is consistent with the fact that the $>2\mu m$ fractions usually show only a small trace of this event. The X-ray diffraction data suggest that the mineral may be illite and the C event may represent its dehydroxylation. Thermogravimetric curves of illite (Grim *et al.* 1937) show a considerable weight loss below 100°C, a gradual loss from 100° to about 350°, a relatively abrupt large weight loss from 350° to 600°, and a gradual loss above 600°C. This description is consistent with the observed C events. Several standard illite minerals were used to define the precise temperatures of the reaction interval. The Kerr (1959) illite formula gives a *theoretical* weight loss of 19%; this value has been used in the calculations, but it seems likely that lower values will prove more realistic.

An associate C^1 peak at approximately 600°C is apparent in some samples but its occurrence is not consistent with any other minerals shown by the XRD analyses. The peak may be a higher temperature illite response, perhaps due to structural disorder. However, the peak occurs more commonly in samples containing the coarser fractions, which may indicate that it is due to the delayed decomposition of the illite occurring in particle aggregates.

Peak D

The abrupt drop in the rate of weight loss at the end of the reaction interval is characteristic of carbonate minerals. Peak D occurs at a temperature which does not allow distinction between dolomite and calcite (Liptay 1971). Under ideal conditions dolomite should show a double peak, but at low partial pressures of carbon dioxide the peaks merge (Liptay 1971). These low $p(CO_2)$ conditions may exist in the furnace of the thermobalance. To ensure that the carbonate peak was not obstructing another mineral response the whole soil was treated with 20% acetic acid. Re-examination of the material by thermogravimetry showed that the carbonate peak had been removed and that there was no other peak in that temperature range. Reaction interval D will be assessed as carbonate mineral using an average weight loss of 45.9%.

Peak E

This ill-defined peak occurs at about 820°C.

TABLE 5. RESULTS OF MINERAL ANALYSES

	Amorphous Matter*	Vermiculite	Illite	Chlorite	Carbonate Minerals	Feldspars			Quartz	Total
						K	Na	Ca		
St. Urbain										
<2µm	25.6	1.3	9.6	0.5		26.0	29.4	8.2	0.0	100.6
>2µm	19.1					20.2	30.9	13.2	14.8	98.2
St. Amboise										
<2µm	43.3		6.1	6.8		11.3	20.4	6.8	1.9	96.6
>2µm	8.8					18.3	32.0	14.7	24.3	98.1
Rimouski										
<2µm	46.3	2.6	25.4	1.6		0.7	7.3	4.9	9.0	97.8
>2µm	20.1	0.5	13.1		1.7	2.3	21.9	5.3	34.7	99.6
Bécancour										
<2µm	14.9	2.1	13.4	1.4		9.0	36.4	12.6	30.2	120.0
>2µm	18.3		5.4	3.0	2.4	10.0	27.0	9.6	23.1	98.8
St. Thuribe (A)										
<2µm	50.0		2.9	9.7		10.2	16.1	6.1	1.8	96.8
>2µm	12.7		0.7	4.4	1.2	19.0	51.1	19.6	0.0	108.7
St. Thuribe (B)										
<2µm	38.8		5.8	4.9		13.2	20.5	8.7	3.7	95.6
>2µm	10.3		1.3	0.5	1.3	11.7	30.5	15.0	27.4	98.0

* This is material denoted as $SPSM_2$ in Figure 8; it seems to be amorphous matter with some hornblende.

Its association with the $<2\mu m$ fraction suggests that the reaction is due to the decomposition of a layer silicate. This temperature is consistent with the decomposition of vermiculite, chlorite and illite, although XRD analyses indicate that in most samples the mineral is probably chlorite.

The illite in these samples (C peak) appears to give a single thermal event (550°C) showing a gradual weight loss to about 850°C. Overlap of event C into event E must be accounted for before the chlorite content is calculated.

Chlorite decomposes at 600° and 850°C (Mackenzie 1957). Event E may be the high-temperature response whereas the 600°C event is incorporated into events C and C^1. The weight loss during the dehydration of chlorite is 13%, of which 3.7% is involved in the 850°C event (Brindley & Chang 1974). After calculation of the chlorite content from event E, the weight loss due to chlorite decomposition at 600°C must be subtracted from event C before evaluating the illite content. The second stage dehydration (540°C) and the dehydroxylation (~840°C) of vermiculite also must be evaluated before calculating the illite and chlorite contents. Using the weight losses that take place during decomposition, the vermiculite, illite, chlorite and carbonate mineral contents have been calculated (Table 5).

SELECTIVE DISSOLUTION ANALYSIS

The main constituents that can be identified and quantified by thermogravimetry are layer-silicate and carbonate minerals which decompose between 25° and 1000°C. However, the low overall percentages of these components indicate that other minerals, thermally inert in terms of weight change in the specified temperature range, constitute large proportions of these clays. X-ray diffraction has indicated that quartz and feldspar may be abundant and in order to quantify these the selective dissolution method of Kiely & Jackson (1965) was employed. The method involves fusion with sodium pyrosulfate, which decomposes several of the mineral groups, including layer silicates. The relics of these minerals are removed by hydrochloric acid and sodium hydroxide treatment. The residue can be analyzed for K_2O, CaO, and Na_2O, and these oxide percentages can be assigned to feldspars. Quartz content is calculated from the percentage residue by subtraction of the feldspar content (Table 5).

TABLE 6. X-RAY FLUORESCENCE DATA

	St. Urbain			*St. Amboise*			*Rimouski*			*Bécancour*			*St. Thuribe (A)*			*St. Thuribe (B)*		
	Whole	<2µm	>2µm	Whole	<2µm	>2µm	Whole	<2µm	>2µm	Whole	<2µm	>2µm	Whole	<2µm	>2µm	Whole	<2µm	>2µm
SiO_2	56.0	51.0	61.8	62.5	51.8	66.5	60.9	48.9	68.6	58.7	49.9	64.9	59.5	50.7	64.7	61.8	50.7	66.4
TiO_2	0.9	0.9	0.9	0.8	1.1	0.8	0.9	0.8	0.9	0.9	1.0	0.8	0.9	1.1	0.8	0.8	1.1	0.7
Al_2O_3	15.9	17.1	16.0	15.0	17.4	14.6	16.6	22.8	12.5	15.7	17.9	14.1	15.4	17.3	14.6	15.4	17.7	14.3
FeO	7.0	9.3	5.8	5.7	9.3	4.5	7.0	9.6	5.1	6.7	8.8	5.2	7.2	9.3	5.6	6.1	9.3	4.9
MnO_2	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.8	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.2	0.1
MgO	2.4	3.5	1.8	2.4	4.5	1.7	3.0	4.0	2.4	3.0	4.3	2.1	2.7	3.9	2.0	2.3	3.9	1.7
CaO	4.2	3.3	4.4	3.5	3.1	3.8	1.5	0.8	2.1	3.8	3.4	4.1	3.8	3.0	4.0	3.9	3.1	4.1
K_2O	4.4	4.7	4.2	3.5	4.2	3.4	3.3	5.2	2.1	3.5	4.6	2.7	3.6	4.3	3.1	3.1	4.2	2.7
P_2O_5	0.4	0.4	0.5	0.3	0.3	0.3	0.2	0.2	0.2	0.3	0.3	0.3	0.4	0.4	0.4	0.3	0.4	0.3
Na_2O	4.0	3.2	4.1	3.8	3.4	4.2	1.6	0.7	2.3	2.7	2.7	2.7	3.4	2.9	3.5	3.8	3.4	3.5
TOTAL	95.3%	93.5%	99.6%	97.6%	95.2%	99.9%	95.1%	93.1%	97.0%	95.4%	93.0%	97.0%	97.0%	93.0%	98.8%	97.6%	94.0%	98.7%

The elemental oxides of the residue and untreated samples were analyzed using X-ray fluorescence spectroscopy; this permitted the overall chemistry of the samples to be evaluated simultaneously. The XRF analyses of the untreated samples (Table 6) gave chemical compositions similar to those obtained by Gillott (1971).

A procedure in the Kiely & Jackson (1965) method enables the mica content of the dissolved fraction to be evaluated. The term mica is used in a similar manner to Brydon & Patry (1961), and it includes the term illite. However, the thermal procedures involved in the method ensure that much of the dissolved material will be of the type which would be detected by thermoanalytical techniques. This being the case, the thermogravimetry results were considered to be a more precise assessment of the dissolved fraction. However, the summation of these components does not approach the dissolved percentages. The error would appear to be due to an incomplete assessment of the sodium pyrosulfate-soluble material, $SPSM_1$ (Fig. 8).

The additional soluble material ($SPSM_2$ in Fig. 8) constitutes a large proportion of these clays and is more readily soluble than the residual minerals. Some of the soluble material ($SPSM_2$) may be hornblende; however, in $<2\mu m$ fractions with little of this mineral present, abundant soluble contents still were recorded. These observations suggest that the dissolved material ($SPSM_2$) may include large quantities of amorphous matter (McKyes *et al.* 1974) undetectable by X-ray diffraction.

DISCUSSION

The analytical techniques used in this investigation yield complementary results which provide the complete elemental oxide chemistry of the sample, the quantitative data for the major crystalline components, and an approximate assessment of an amorphous matter content. One of the major problems in the interpretation of thermogravimetric data is the difficulty in obtaining reliable chemical formulae for the mineral species present. In this work the weight-loss values used in the calculations were verified by studying mineral standards that showed the appropriate reaction interval.

X-ray diffraction data published over the past twenty years may be erroneous because of an underestimation of the primary mineral components. This seems to be the result of amorphous matter which preferentially adheres to the electrochemically inert particles (McKyes *et al.* 1974). Direct comparison of XRD data may not be possible because of the variation in the amorphous matter contents from one sample to another.

There is some evidence to suggest that the amorphous matter is active as a cementing agent. After a mineralogical study of the Toulnustouc River material, which Conlon (1966) regarded as highly cemented, Quigley (1968) found no trace of carbonate minerals. The occurrence of carbonate minerals in the coarser fractions ($>2\mu m$) of these samples (Table 5) may indicate that the permeability of the finer fractions had been reduced by an earlier stage of cementation by amorphous matter.

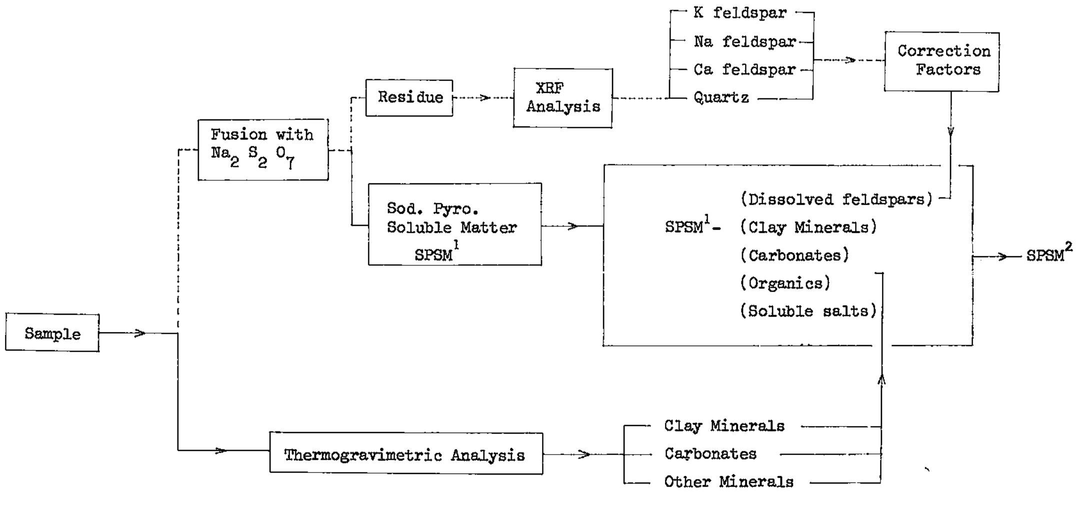


FIG. 8. Flow diagram showing the suggested modification to the Kiely & Jackson (1965) method. Their method is shown with a dashed line.

The mineralogy of the $<2\mu m$ fraction provides an indication of the weathering intensity to which a clay has been subjected. Jackson *et al.* (1948) proposed a weathering sequence and our results show, with quartz and illite present, an indication of weathering stages 6 and 7 respectively. Albite, which may be regarded as making up the majority of the rock flour along with quartz, is indicative of weathering stage 5. Therefore, these mineralogical results support the pre-1957 proposal that sensitive clays have undergone little chemical weathering.

The mineralogical data support Smalley's (1971) postulated primary mineral composition for these clays. However, the essential factor causing high sensitivities is not the predominance of short-range bonds, as Smalley (1971) suggested, but rather the open nature of the particle structure which enables a system to exist at a moisture content in excess of the liquid limit. The results (Table 5) suggest that it is the interaction of primary mineral particles with amorphous matter which plays an important role in the formation of sensitive clay structures.

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