

Chapter 3

Nature of Sensitive Clays from Québec

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Abstract Quantitative mineralogical analyses of sediment samples from 18 sites in Québec indicate that plagioclase is the most abundant mineral in all soils and that chlorite is generally the dominant clay mineral followed by illite and expandable clays consisting of mixed-layer clays minerals involving vermiculite. The study also illustrates how specific surface area and constitutive water content can be used to evaluate departures from average in the mineralogical composition of sensitive clays. The relationship between activity and specific surface area of sensitive clays shows that when compared to soils from different sedimentary basins that have different mineralogy, they are characterized by a much lower activity and specific surface area.

Keywords Sensitive clays • Quantitative mineralogy • Specific surface area • Activity • Québec • Oxides • Postglacial marine sediments

3.1 Introduction

Sensitive clays in eastern Canada are mostly found in Québec but also occur in Ontario and Labrador. In Eastern Canada, sources of sediment are mostly controlled by glaciations and de-glaciation patterns and by the trends of the major topographic features (Locat 1995). North of the St. Lawrence River, ice-flow directions were southerly while north of the water divide boundary in the Appalachian Mountains,

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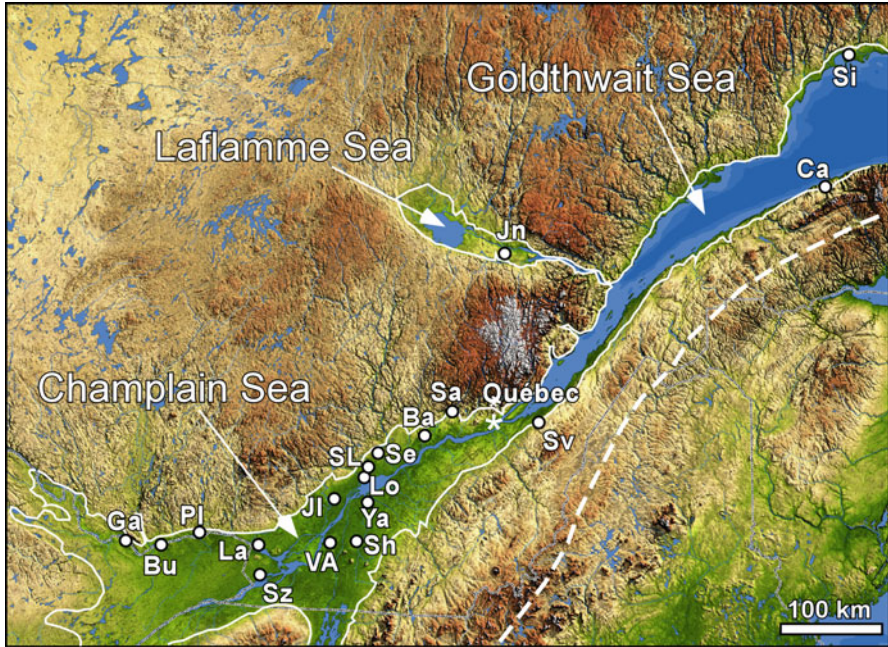


Fig. 3.1 The postglacial seas of the St. Lawrence valley and the location of the sensitive clay sample sites: (*Ba* Batiscan, *Bu* Buckingham, *Ca* Cap-Chat, *Ga* Gatineau, *Jl* Joliette, *Jn* Jonquière, *La* Lachute, *Lo* Louiseville, *Pl* Plaisance, *Si* Sept-Îles, *Sa* St. Alban, *Se* St. Étienne, *Sh* St. Hyacinthe, *SL* St. Léon, *Sv* St. Vallier, *Sz* St. Zotique, *Va* Varennes, *Ya* Yamaska). *Dashed line*: water divide where in the final de-glaciation phase ice-flow direction were to the north and south of that boundary

the ice-flow was towards the north near the end of de-glaciation (see dashed line in Fig. 3.1). In the St. Lawrence Basin in Québec, the source rocks can be roughly divided into three geological provinces: (1) the Precambrian shield (mostly metamorphic rocks), (2) the Appalachian (volcanic and sedimentary rocks) and (3) the Lower St. Lawrence Platform (mostly limestone and some shale). Most of the sediments coming into the various postglacial seas resulted from glacial grinding of the bedrock with little post-depositional weathering. The nature of the sensitive clays from Québec reflects this. Postglacial marine sedimentary basins, along the St. Lawrence River system (Fig. 3.1) consist of the Goldthwait Sea (actual Gulf of St. Lawrence, since 13,500 yBP), the Champlain Sea (St. Lawrence Valley, upstream of Québec City, 13,100–10,600 yBP), and the Laflamme Sea (at the northwestern tip of the Saguenay Fjord, 10,300–8,000 yBP).

Investigation of the mineralogy and chemistry of sensitive clays from Québec, are quite limited. Mineralogical analyses have been presented by Brydon and Patry (1961), Ballivy et al. (1971), Gillot (1971), Gravel (1974), Bentley (1976, 1980), Martel et al. (1978), Foscal-Mella (1976), Babineau (1977), Quigley (1980),

Lebuis et al. (1983), Locat et al. (1984), Torrance (1988, 1995), Locat 1995, and Berry and Torrance (1998). Most of these results can be described as semi-quantitative. We are taking the opportunity of this workshop to present the quantitative mineralogical analyses of sensitive clays from various sites in Québec (Fig. 3.1) that were conducted by St. Gelais (1990). We will commence by describing the methodology. It will not be possible to review the role of mineralogy (sediment source) on the behavior of sensitivity clay sediments. To that effect, the reader is referred to work Quigley (1980), Torrance (1983), Torrance and Ohtsubo (1995), Locat (1995), and Locat et al. (2003).

3.2 Methodology

Samples of sensitive clays were collected, using Shelby tubes, from 1.5 to 2.5 m depths to assure that they were from below the surface oxidizing zone. Great care was given to maximize analyses on the same sample (St. Gelais 1990). Physico-chemical properties, reported in Table 3.1, were acquired according to BNQ (Bureau de la normalisation du Québec) standards including grain size analysis on wet samples. The CEC was measured according to the method of Chapman (1965) using an ammonium acetate solution at a pH of 7, the organic content by calcination (Ball 1964), and the salinity by electric conductivity of the interstitial water (Torrance 1975). The specific surface area was determined using three methods: EGME (Diamond and Kinter 1958), and methylene blue (Tran 1977) for both internal and external surfaces, the BET (nitrogen method) for the external surfaces.

The main mineral phases for all samples were identified using X-ray diffraction. Samples were dried at 25 °C and lightly crushed for the powder diagrams and a wet subsample was put in a centrifuge to extract the <2 µm fraction used for natural, glycolated, and heated X-ray diffraction of oriented samples and to obtain the >2 µm for constitutive water analysis (H₂O_c). Water loss measurements, on both size fractions were determined using a thermogravimetric method (TGV) with temperature intervals as proposed by Jackson (1969), i.e. 105–110 °C for H₂O_h (hygroscopic water), 110–300 °C for interlayer water (as for montmorillonite and vermiculite), and from 300 to 900 °C for constitutive water (H₂O_c). As suggested by Jackson (1969), the water loss for constitutive water was computed with respect to the loss at 300 °C. By doing so, the relative amounts of H₂O_c for vermiculite, illite and chlorite are 5, 4.5, and 13 % respectively. For a Na-montmorillonite and K-montmorillonite the value are 2.7 and 6.2 % respectively (Bérend et al. 1995). For the TGV measurements, samples were pre-treated with hydrogen peroxide to remove the organic matter and the treatment lasted for as long as effervescence was visible (McKeague 1978). H₂O_c results are compiled in Table 3.1 for an average of three tests (St. Gelais 1990).

For the quantitative evaluation of the main mineral phases, the approach proposed by Engler and Tyengar (1987) was used and the results are presented in Table 3.2. It incorporates various methods in parallel. So, for quartz, K-feldspar, and

Table 3.1 Physico-chemical properties of selected sensitive clay sediments from Québec

Site	w	mo	wL	wp	II	Ip	CF	CEC	Act	Sal	BI	BET	EG	H+F	noA	H+>F	n0B	A+B	H+to
Champlain Sea																			
Batiscan	63	0.8	64	21	0.97	42.6	78	12.6	0.53	0.8	77	29	112	4.3	3.5	3.4	0.7	4.1	4.0
Buckingham	39	0.6	42	17	0.86	25.1	50	12.3	0.55	0.2	61	19	101	5.2	2.6	2.6	1.3	3.9	4.0
Gatineau	60	1.0	68	25	0.81	42.2	75	16.0	0.56	0.1	90	32	131	5.2	3.9	2.6	0.6	4.5	4.6
Joliette	40	0.5	42	17	0.94	24.4	48	8.0	0.49	2.3	39	15	53	5.0	2.5	1.5	0.7	3.3	3.1
Lachute	47	0.8	57	24	0.69	33.4	78	14.6	0.42	0.6	93	31	106	5.6	4.2	3.8	1.0	5.2	5.1
Louiseville	73	0.8	76	24	0.94	52.5	80	13.0	0.66	1.9	91	29	97	5.1	4.1	2.2	0.4	4.5	4.5
Plaisance	70	0.9	77	25	0.86	52.3	80	16.6	0.65	0.4	102	36	133	5.9	4.7	3.0	0.6	5.3	5.3
St-Alban	74	0.9	55	21	1.57	33.8	78	12.6	0.42	0.5	98	32	119	5.6	4.5	2.6	0.5	5.0	4.9
St-Étienne	34	0.7	34	19	0.98	14.7	30	10.6	0.49	0.4	41	9	67	4.4	1.3	0.7	0.5	1.8	1.8
St-Hyacinthe	74	0.7	70	22	1.08	48.1	80	12.0	0.60	1.3	88	32	125	5.2	4.2	4.6	0.9	5.1	5.2
St-Léon	66	0.6	71	23	0.90	48.1	73	13.3	0.69	0.5	76	29	102	4.4	3.1	2.6	0.8	3.9	4.0
St-Zotique	86	1.2	71	23	1.32	47.6	83	15.6	0.56	0.9	98	32	112	5.9	5.0	4.2	0.6	5.6	5.7
Varenes	57	0.9	67	27	0.74	40.1	82	15.3	0.50	1.3	102	34	125	5.6	4.5	3.0	0.6	5.1	5.1
Yamaska	41	0.7	40	16	1.04	23.5	40	7.6	0.59	1.8	49	17	63	3.6	1.5	3.5	2.1	3.5	3.4
Goldthwait Sea																			
Cap Chat	32	0.4	40	22	0.58	17.2	30	6.3	0.57	1.5	26	10	35	6.4	1.9	3.8	2.7	4.6	4.5
Sept-Îles	48	0.6	63	27	0.60	36.1	75	12.0	0.48	11.8	38	24	73	7.0	5.2	2.9	0.7	6.0	5.9
St-Vallier	34	0.5	36	15	0.92	20.6	52	8.6	0.41	3.8	46	16	65	4.5	2.2	2.3	1.1	3.4	3.4
Laflamme Sea																			
Jonquière	32	1.0	41	20	0.58	20.5	48	11.3	0.41	0.3	48	18	88	5.5	2.5	1.1	0.6	3.0	3.1

Note: *w* water content (%), *mo* organic matter (%), *wL* liquid limit (%), *wp* plastic limit (%), *IL* liquidity index, *CF* clay fraction (%), *Act* activity, *Sal* salinity (g/L), *BI* surface area (m²/g), *Bleu* method (m²/g), *BET* surface area, nitrogen method (m²/g), *EG* surface area, ethylene glycol (m²/g), *H+F* H₂O_c of clay fraction, *n0A* H₂O_c normalized for the clay fraction, *H+>F* H₂O_c for non-clay fraction, *n0B* H₂O_c normalized for non-clay fraction, *H+to* total constitutive water

Table 3.2 Mineralogical composition of selected sensitive clay sediments from Québec (all values are in %)

Site	Cal	Dol	Cal	Ca2	Am	Qtz	KFd	Pla	I	V	M	C	aAl	aFe	aSi	aTo	CTo	PTo	MTo
Champlain Sea																			
Batiscan	2.0	1.0	3.0	9.3	12	10	11	34	8	5	0	9	0.2	0.5	0.2	0.9	23	70	93
Buckingham	1.0	0.0	1.0	0.5	9	20	7	36	8	4	0	11	0.2	1.2	0.4	1.8	22	73	97
Gatineau	1.0	0.0	1.0	1.0	8	11	12	36	8	9	0	9	0.2	1.0	0.4	1.6	27	68	96
Joliette	0.0	0.0	0.0	1.7	10	17	13	40	10	3	0	4	0.1	0.6	0.3	1.0	16	80	98
Lachute	2.0	1.0	3.0	8.4	12	11	11	33	9	5	0	14	0.2	0.7	0.4	1.3	28	70	100
Louiseville	1.0	0.0	1.0	3.6	9	9	15	39	5	5	0	16	0.2	0.9	0.4	1.5	26	72	100
Plaisance	1.0	1.0	2.0	4.8	10	13	11	32	10	6	0	13	0.1	0.9	0.4	1.5	29	67	98
St-Alban	2.0	0.0	2.0	7.3	8	10	14	34	8	7	0	12	0.2	0.7	0.4	1.2	27	69	98
St-Étienne	1.0	0.0	1.0	4.0	8	18	17	39	2	2	0	5	0.3	0.9	0.5	1.7	9	81	92
St-Hyacinthe	3.0	1.0	4.0	10.3	11	12	8	32	8	5	0	15	0.2	0.5	0.3	0.9	28	67	97
St-Léon	1.0	0.0	1.0	3.3	8	10	15	40	4	5	0	11	0.2	0.9	0.3	1.4	21	74	97
St-Zotique	3.0	0.0	3.0	9.8	10	10	9	31	10	7	0	15	0.2	0.8	0.4	1.4	32	62	95
Varenes	2.0	1.0	3.0	8.3	8	10	13	33	9	7	0	13	0.2	1.0	0.3	1.6	29	66	97
Yamaska	4.0	1.0	5.0	11.1	10	19	10	33	5	6	0	6	0.1	0.5	0.3	0.8	17	77	94
Goldthwait Sea																			
Cap Chat	5.0	2.0	7.0	8.3	16	3	28	20	2	2	0	20	0.1	0.6	0.2	0.9	24	74	99
Sept-îles	2.0	0.0	2.0	1.1	6	8	6	40	13	8	0	13	0.2	1.0	0.3	1.5	34	62	98
St-Vallier	1.0	1.0	2.0	5.3	8	21	10	37	10	3	0	5	0.1	0.5	0.3	0.9	18	77	96
Laflamme Sea																			
Jonquière	2.0	0.0	2.0	3.3	6	17	22	35	2	2	0	11	0.3	1.3	0.3	1.9	15	82	98

Note: Cal calcite, Dol dolomite, Cal from Chittick, Ca2 from chemical analysis (CO₂/0.44), Am amphibole as actinolite, Qtz quartz, KFd K-feldspar, Pla plagioclase, I illite, V vermiculite, M montmorillonite, C chlorite, from DCB aAl₂O₃, aFe Fe₂O₃, aSi SiO₂, aTo total, CTo total clay minerals, PTo total primary minerals, MTo all minerals (including minerals)

plagioclase, the pyrosulfate method was used (Kiely and Jackson 1965). Plagioclase was determined by assigning the Na_2O of the chemical analysis to anorthite (%An) indexed using the method proposed by Van der Plas (1966). Illite was determined by attributing the K_2O obtained from selective dissolution in HCl at 825 °C (Reynolds and Lessing 1962). Vermiculite and montmorillonite were determined using CEC after removing the iron oxides (Alexiades and Jackson 1965). Total carbonates (calcite and dolomite) were determined using the Chittick apparatus (Dreimanis 1962; Locat and Bérubé 1986). For chlorite and amphibole there are no quantitative dissolution methods. Therefore, amphiboles (referred to here as actinolite) were computed by attributing the residual CaO from chemical analysis after re-distribution to carbonates and plagioclases (calcite: 56 %, dolomite: 30.4 %). For plagioclase the distribution depends on the %An (percent anorthite). In the same way, the chlorite was determined by allowing the residual value of MgO after attribution to amphibole: 13.8 %, illite: 2.8 %, vermiculite: 22 % and dolomite: 21.9 %. This re-distribution of MgO is based on an average chemical composition of these minerals present in various Québec soils (Gravel 1974). Amorphous silica (Si), and Fe and Al oxides were determined using the dithionite-citrate-bicarbonate method (DCB, Mehra and Jackson 1960). For each sample, the mineralogical analysis was verified by chemical mass balance. For SiO_2 and Al_2O_3 , differences in concentration computed from mineralogical composition and chemical analyses are on average -0.24 ± 0.8 and 1.1 ± 0.6 % respectively. For more details on the various procedure and test results (including chemical analyses), the reader is referred to St. Gelais (1990).

3.3 Results and Analysis

3.3.1 Physico-chemical Properties

Physico-chemical properties for the soils from 18 sites (Fig. 3.1) are presented in Table 3.1. Water contents values are mostly below or near the liquid limit. This reflects using samples been collected near the surface, within the crust (mechanical) but below the weathered crust. In that of the crust they were likely exposed to soluble weathering products coming from above. Three samples have clay fraction below 50 % (Cap Chat, St-Étienne, and Yamaska) and are consider as clayey silts. The organic matter content is less than 1.2 %. The activity of most soils varies between 0.4 and 0.7 which is within the range (0.25 and 0.75) noted by (Leroueil et al. 1983) for soils from Québec. The Sept-Îles, Joliette, and Saint-Vallier have a pore water salinity above 2 g/L which is also reflected by their higher Na^+ exchangeable cations but not clearly by their activity (St. Gelais 1990). Locat (1982) has shown that increasing the salinity to 35 g/L for 12 soils from Québec resulted in an average activity coefficient of 0.5 ± 0.09 and the increase was higher for soils with an initial activity below 0.3. Specific surface areas, for both internal and external surfaces, were measured by the Blue and EGME methods. They both show

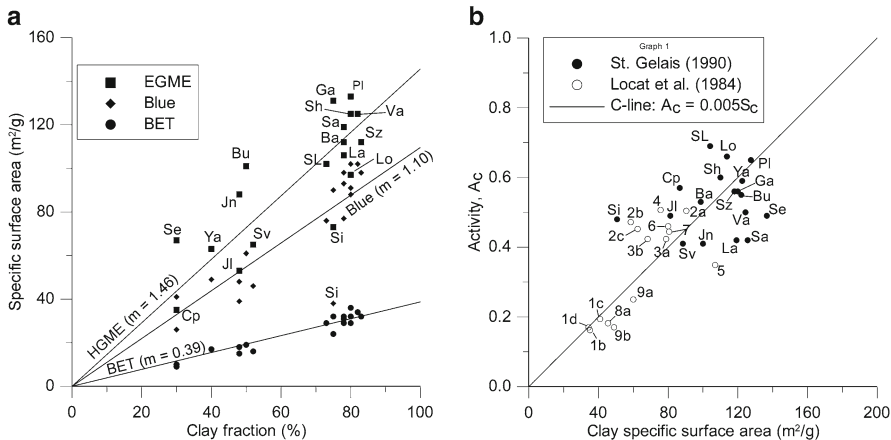


Fig. 3.2 (a) Specific surface areas and clay fraction for total (EGME and Blue methods) and external (BET method) surfaces. (b) Activity as a function of clay specific surface area (C-line from Locat et al. 2003, C stands for clay), sites with numerals are from Locat et al. (1984), 1 Grande-Baleine, 2 Olga, 3 St. Marcel, 4 St. Léon, 5 St. Alban, 6 St. Barnabé, 7 Shawinigan, 8 Chicoutimi, and 9 Outardes

a relationship between surface area and clay fraction, with the EGME values consistently higher than with the Blue method (Table 3.1, Fig. 3.2a). For a linear relationship going through the origin, the r^2 values are 0.97 for both. The BET method (external surfaces only) is less influenced by the mineralogy (St. Gelais 1990) and shows an even better relationship ($r^2=0.99$, Fig. 3.2). For the Blue method, results for the Sept-Îles sample (Si in Fig. 3.2) are much lower than the others when using the Blue method. It is believed that this may be due to its high pore water salinity (11.8 g/L) which may reduce the adsorption of the Blue on the particles (Pimol et al. 2008).

From these analyses, Locat et al. (2003) introduced a concept relating the Skempton Activity (A_c) to the clay specific surface area S_c (Blue or total surface area/ $<2 \mu\text{m}$) to illustrate the effect of clay mineralogy on index and surface properties. The S_c value is equivalent to consider that all the surface area is due to the particles in the clay fraction. Results for sensitive clays from Québec are shown in Fig. 3.2b, also including data from Locat et al. (1984). The relationship has been used to introduce the C-line (Locat et al. 2003). As will be discussed below, the spread in Fig. 3.2b represents the variability of the mineralogical composition of sensitive clays from Québec characterized by a low activity and a relatively low value of clay specific surface area (S_c).

Locat et al. (1984) determined that the constitutive water (H_2O_c) could be a good indicator of the presence of clay minerals in sensitive clays from Québec. As part of the work by St-Gelais (1990), this approach was further developed and then tested on the clay fractions, the non-clay fractions and the bulk samples of the 18 soils, after the removal of organic matter. Results (Table 3.1) show that the method is

quite robust with the total of constitutive water content of both clay and non-clay fraction being nearly the same as the one computed for the whole sample (standard deviation of $\pm 0.08\%$).

3.3.2 Primary Minerals (Non-clay)

Results of the mineralogical quantification are presented in Table 3.2. The suite of primary minerals, by decreasing concentration, is: plagioclase (19–40%), K-feldspar (6–28%), quartz (3–20%), amphiboles (actinolite, 6–16%), calcite (1–5%) and dolomite (0–2%). Taken globally, the amounts of primary minerals and their relative abundances are similar to what has been estimated by Brydon and Patry (1961), Locat et al. (1984) and by Berry and Torrance (1998). The primary mineral content ranges from 62 and 80% while inversely the amount of clay minerals ranges from 9 to 34%. The total amount of dithionite-citrate-bicarbonate (DCB) extractable material ranges from 0.82 to 1.87% (Table 3.2), which is comparable to the range of 1.09–2.06% reported by Berry and Torrance (1998).

3.3.3 Clay Minerals

Clay minerals are compiled in Table 3.2. The suite of clay minerals, by decreasing order of concentration is: chlorite (4–20%), illite (2–13%), and vermiculite (2–9%) with an (illite + vermiculite)/chlorite ratio varying between 0.5 and 1.5. Only traces of montmorillonite was found (<0.1%). The total amount of clay minerals varies from 9% (St. Étienne) to 34% (Sept-Îles). The highest amount of chlorite is found in Cap Chat (20%), because of a large portion of chloritic shale in the non-clay fraction, and the lowest is in the Joliette sample (4%). For illite, the maximum is found in the sample from Sept-Îles (13%) and the lowest at St. Étienne, Cap Chat and Jonquière (2%) with the first two having the lowest clay fraction (30%). For vermiculite, inter-layered with illite and/or chlorite, the maximum is for the Gatineau sample (8%) and the minimum again for Cap Chat and St. Étienne at 2%. The abundance of clay minerals is clearly correlated with the clay-size fraction, except for Cap Chat (Cp, Fig. 3.3a). For the Cap Chat sample, the presence of clay minerals in the non-clay fraction is also in agreement with the higher values of H_2O_c in the non-clay fraction relative to the clay fraction (Table 3.1).

The amount of constitutive water (H_2O_c) in bulk samples, shows an excellent relationship to the abundance of the clay minerals, (Fig. 3.3b). However, when computed from the clay mineral composition, the estimated amount of constitutive water is significantly lower but still shows a positive relationship. The good relationship for the bulk samples, may be due to (St. Gelais 1990): (1) the (illite + vermiculite)/chlorite ratio only varies between 0.5 and 1.5; (2) the small amounts of hydrous oxides present; and (3) only small amounts of calcite are present (0–5%).

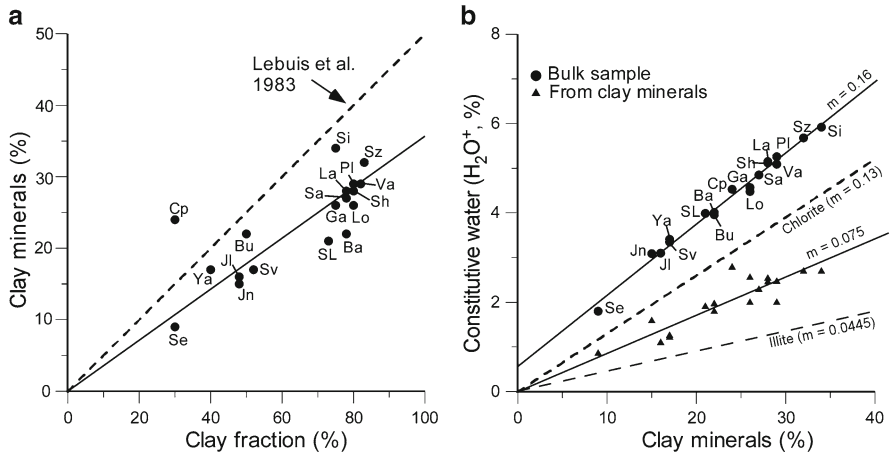


Fig. 3.3 (a) Clay minerals as a function of the clay fraction. Note here that results from Cap Chat (Cp) which contains clay minerals as part of shale particle in the silt fraction. (b) Amount of constitutive water as a function of the amount of clay minerals obtained from bulk samples measurements and computation from clay mineral composition

The relationship between constitutive water and mineral abundance can be a useful tool to evaluate if a clay sample in Québec has a mineralogical composition which departs from typical values.

Berry and Torrance (1998) note that both montmorillonite and vermiculite (in mixed-layer form) are expandable minerals. The impact of vermiculite, as an expandable clay, cannot have the same effect as that of montmorillonite at the same concentration (Locat et al. 2003). Still we consider, like Berry and Torrance (1998), that using the word ‘expandable’ is inclusive and should include mixed-layered minerals (illite/vermiculite and/or chlorite/vermiculite).

When compared to other clays, it is clear that Québec clays have a very different signature than samples formed in different environments and containing a significant amount of swelling clays. As reported by Locat (1995) and Locat et al. (2003) the sedimentary environments found in Québec are also found in other regions like in Ontario, British Columbia (Geertsema and Torrance 2005), Labrador, Scandinavia and parts of Russia and Alaska. Results from various studies carried out at Laval University and reported by Locat et al. (2003) are presented in Fig. 3.4 which encompasses data for soils with very different mineralogical composition from kaolinite soils to smectite dominated soils. The C-line relationship, introduced above, presents both the plasticity index and the specific surface area normalized by the clay fraction (<2 µm). The C-line in Fig. 3.4 illustrates the overall link between these two parameters with a slope of 0.005. The C-line indicates that a sediment with an activity of 0.5, a clay specific surface area (S_c) of about 100 m²/g should be expected and it would fall within the illite/chlorite dominated zone. Figure 3.4 puts the Québec sensitive clays in a broader perspective with respect to soils in Europe

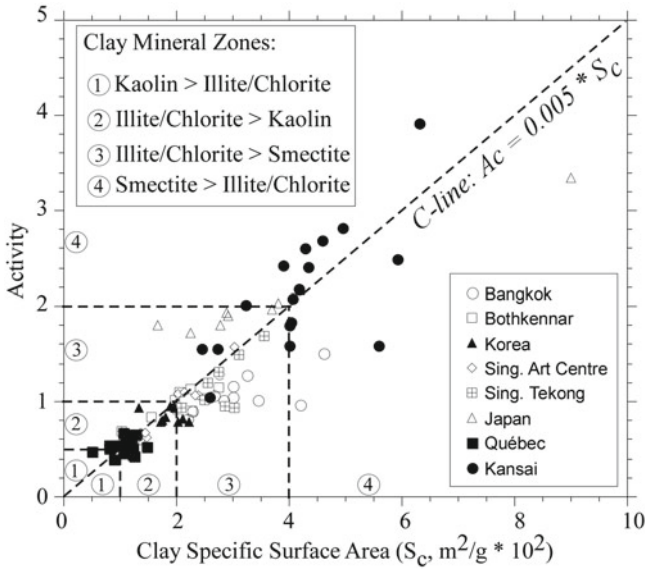


Fig. 3.4 The relationship between the Activity (A_c) of soils and their clay specific surface area (S_c). The dashed line from the origin represents the 'clay line' (C-line, modified after Locat et al. 2003)

and Asia where the geological history and source of sediment may be quite different, but similar to places in Europe and Asia where the geological history is similar. Ariake clay (Torrance and Ohtsubo 1995), which has a low activity for a Japanese clay (varying from 0.75 to 2.0), would fall in zone 3 but close also to boundary between zone 3 and 2. Figure 3.4 also illustrates the fact that sensitive clays from Québec are of low activity and contain little or no swelling clays. In this diagram, Québec clays fall within the illite/chlorite dominated zone, which for Québec could read as chlorite/illite.

3.4 Concluding Remarks

As indicated by Locat et al. (1984), it is difficult to develop a clear mineralogical signature of the soils as a function of their sedimentary basin but the sediments formed in these postglacial seas can be seen as a product of the concentration of fines from the till (Locat 1995). The end result is a clay soil with a low activity and a relatively low clay specific surface area (S_c). This may be due to various reasons including variability in the grain size distribution (e.g. in various profiles as shown by Berry and Torrance 1998) and also because some mixing may have taken place between sediment plumes coming from the various rivers entering the basins.

Although each measurement method has its own uncertainty, this paper has presented what we consider as the quantitative analysis of 18 samples from Québec, most of them within the Champlain Sea basin. The set of data can certainly be explored in more details and should provide a reference to help evaluate the performance of simpler methods.

As part of this work, we have underlined the interesting potential of the specific surface area measurements (all methods) and the amount of constitutive water (H_2O_c) to reflect on the clay mineralogy of the soils, even more when compared to soils from other environments. This work has also illustrated the use of the C-line to reflect on the nature of sensitive clays relative to other clays from around the World.

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