Canadian Geotechnical Journal
Revue canadienne de géotechnique

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Journal:	<i>Canadian Geotechnical Journal</i>
Manuscript ID	cgj-2017-0490.R1
Manuscript Type:	Note
Date Submitted by the Author:	19-Oct-2017
Complete List of Authors:	Spagnoli, Giovanni; BASF Construction Solutions GmbH Stanjek, Helge; RWTH Aachen University, Clay and Interface Mineralogy Sridharan, Asuri; Indian National Science Academy
Is the invited manuscript for consideration in a Special Issue? :	N/A
Keyword:	pore fluid, Atterberg limits, undrained shear strength, clays

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Some observations considering undrained shear strength, liquidity index and fluid-solid ratio of mono-mineralic clays with water/ethanol mixtures

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Abstract

The mechanical properties of clays are also influenced by the characteristics of the fluid in the pore space. Liquid limit reacts different to the permittivity ϵ of the fluids: For smectites the slopes are positive, for kaolinite and illite they are negative and smaller. This dissimilarity can be explained by the structural differences between swelling smectites with solvated interlayer cations and non-swelling clay minerals such as kaolinite and illite. Undrained shear strengths of Ca-smectite, but not Na-smectite, correlate with the actual fluid ratio. Regressing c_u against the liquidity index I_L yields two different regression lines for Na- and Ca-smectite. For the first time, it is shown that when c_u is normalized to the ϵ of the pore fluid, this results in a single regression line for both smectitic clay types. As kaolinites and illites possess significantly less exchangeable cations than smectites, this yields significantly smaller ranges for Atterberg limits and reduces the impact of ϵ to almost pure particle-particle interactions. Besides, the much larger particle sizes of the kaolinite and illite, respectively, may dominate the undrained shear strengths, as normalization of c_u to ϵ did neither change the relationships to the actual water content nor to the liquidity index.

Keywords Undrained shear strength; pore fluid; Atterberg limits; clays

Nomenclature

W_L	Liquid limit (mass %)
W_p	Plastic limit (mass %)
I_p	Plasticity index (mass %)
I_L	Liquidity index (-)
I_C	Consistency index (-)
a	intercept of the regression line
b	slope of the regression line
c_u	Undrained shear strength (kPa)
n	sample numbers
p	probability value (-)
R^2	coefficient of determination (-)
ε	Dielectric constant (relative permittivity) of pore fluid (-)

Introduction

Leakage and spillage of diesel and gasoline from pipelines, processing plants or from abandoned fuel stations contaminates the surrounding soil. Clay minerals in these soils may mitigate the extent of contamination because of their chemical interaction with these hydrocarbons (Fine *et al.* 1997), but organic fluids will also change the mechanical properties when water in the pore space is partially replaced by non-polar fluids. In fact, according to the Diffuse Double Layer (DDL) theory, the extension of the double layer decreases with increasing electrolyte concentration (or decreasing dielectric constant) (Mitchell and Soga, 2005). Besides, low polar fluids tend to make weak electrostatic interactions with clay surface oxygen and interlayer-water molecules which result in an increase in interlayer spacing (e.g. Amarasinghe *et al.* 2009). Clay in contact with non-polar fluids tend to flocculate, causing a change in mechanical (e.g. Sridharan and Jayadeva 1982; Sridharan *et al.* 1986, 1988; Spagnoli *et al.* 2012a; Estabragh *et al.* 2014) and hydraulic properties (e.g. Anderson *et al.* 1985; Bowders and Daniel 1987; Fang 2013; Di Matteo *et al.* 2016). The impact of such hydrocarbons on compaction characteristics, friction angle and Atterberg limits has been studied, but a survey of Khosravi *et al.* (2013) showed that there are still unexplained variations in the data. Furthermore, the addition of pure ethanol to gasoline (an increasing trend in the EU, US and Brazil) (RFA, 2012) requires attention, because ethanol mixes completely with water, whereas most gasoline components do not.

Since the pioneering work of Atterberg (1911) it is known that soils change their mechanical properties significantly with their water content, w . Starting at high water contents and reducing w continuously, the liquid limit w_L is approached, where a transition from the viscous to the solid state is observed. With further decrease of w , the plastic limit w_p is reached, where the still ductile consistency changes to a brittle behaviour (e.g. Carter and Bentley 1991; Bolton and Cheng 2002; Haigh *et al.* 2013). Both w_L and w_p are widely used as index parameters, from which the plasticity index I_p can be calculated as:

$$I_p = w_L - w_p \quad (1)$$

I_p can be used to predict, e.g., the undrained shear strength, the ratio of strength of the Standard Penetration Test (SPT) blow count and for critical state Cam-Clay parameters (cf. Vardanega and Haigh 2014, for further details). According to Skempton and Northey (1952) w_L and w_p of soils with a similar mineralogy correspond approximately to fixed mechanical strengths $F_{L,p}$, which are related as $F_{L,p} \sim 1/100$, although

recently O'Kelly (2013) and Vardanega and Haigh (2014) showed that this value is inadequate in the semi-logarithmic space. Considering the current water content w of a soil, the liquidity index I_L is defined as:

$$I_L = (w - w_p) / I_P \quad (2)$$

Undrained shear strength has been related to I_L (with H_2O as the pore fluid) (see Skempton and Northey 1952; Leroueil *et al.* 1983; Locat and Demers 1988; Wroth and Wood 1978; Yilmaz 2000; Vardanega and Haigh 2014; Kuriakose *et al.* 2017). Replacing water progressively with ethanol this had no effect on kaolinite and illite, but for a smectitic clay a significant correlation between undrained shear strength and ethanol/water ratio fraction was found (Spagnoli *et al.* 2012b). In contrast to these findings, Di Matteo *et al.* (2016) observed that increasing amounts of ethanol in gasoline increased the hydraulic permeability of a kaolinitic soil up to two orders of magnitude. Albeit undrained shear strengths relate somehow to Atterberg limits, data for Atterberg limits as a function of ethanol/water ratios are sparse. In this work, we determined therefore Atterberg limits for kaolinite, illite and Ca-smectite, respectively, as a function of ethanol/water ratio.

Materials and Methods

The materials used in this research are purified kaolinite, illite and Na- and Ca-smectite, respectively, which have been described in Spagnoli *et al.* (2010; 2012a;b) and Spagnoli (2011). The dielectric constant ϵ of the pore fluid was adjusted by mixing distilled water with increasing amounts of ethanol in volume fractions between 0 and 1 in steps of 0.25 $m^3 m^{-3}$. The dielectric constants of the ethanol/water mixtures were taken from Wohlfarth (2008). For Na-smectites it was not possible to determine the Atterberg limits for ethanol fractions above 0.5, because the Na-smectites flocculated (see explanation given by Kaya and Fang 2005 for details). Tab. 1 shows the values of the soils tested. The Atterberg limits were determined according to the DIN 18121 (2012) and DIN 18122 (1997). The undrained shear strengths for the standard clays, mixed with the fluids from table 1, have been determined using the vane shear test according to the German standard DIN 4094-4 (2002). The paste was made by mixing the powder clay with the fluids and then for attaining equilibrium kept in a closed bucket to avoid fluid loss. The water (or fluid) content was then measured to determine the consistency value. After that, vane shear tests were carried out for clay specimens (diameter 100 mm, height 120 mm) with indices of consistency, I_C from 0.3 to 0.85, compacted

with a Proctor standard test (DIN 18127, 2012) with an average compaction energy of 0.6 MNm/m^3 , then a vane was inserted for 3 cm and a torque with a velocity of 2 cm/s was applied until the specimen failed. I_C is defined as:

$$I_C = (w_L - w) / I_p \quad (3)$$

Results

Smectites have both the highest liquid and plastic limits compared to kaolinite and illite (Fig. 1). Note that the variances of w_L and w_p for kaolinite and illite are significantly smaller than for smectites, but for all four groups, significant correlations between w_p and w_L were found.

The liquid limit w_L changes significantly with ε of the pore fluids, but for smectites in a different way than for kaolinite and illite, respectively. For smectites w_L increases with increasing ε (Fig. 2), for kaolinite and illite w_L decreases significantly with ε .

Figure 1: Plastic limits w_p versus liquid limits w_L for smectites in water. Key: ($\diamond$) Ca-smectite, ($\blacklozenge$) Na-smectite, kaolinite (+) and illite ($\square$).

Figure 2: Liquid limits versus ε for smectites (A) and kaolinite and illite (B). Key: $\diamond$ Ca-smectite, $\blacklozenge$ Na-smectite, kaolinite (+) and illite ($\square$).

The plasticity indices I_p (Fig. 3) show relationships similar to those of w_L versus ε (Fig. 2), but for kaolinite and illite, the slopes of the correlation are not significant.

Figure 3: Plasticity indices I_p versus ε for smectites (A) and kaolinite and illite (B). Key: $\diamond$ Ca-smectite, $\blacklozenge$ Na-smectite, kaolinite (+) and illite ($\square$).

The undrained shear strengths correlate only for Ca-smectite with the liquid limits (Fig. 4). For all other samples, c_u varies considerably, but without relation to the liquid limit.

Figure 4: Undrained shear strengths c_u versus w_L for smectites (A) and kaolinite and illite (B). Key: $\diamond$ Ca-smectite, $\blacklozenge$ Na-smectite, kaolinite (+) and illite ($\square$).

Kuriakose *et al.* (2017) observed relationships between $\log(c_u)$ and the actual fluid content of the samples, which had been normalized to the liquid limit w_L . For our samples, only Ca-smectites correlate with this normalized water contents, whereas for Na-smectite, kaolinite, and illite, respectively, the R^2

values were below 0.4 and the slopes had significance levels smaller than 98% (Fig. 5, left). The same applies for plots against the liquidity index (Fig. 5, right). In Figs. 5A and 5B, a and b are the intercept and the slope of the regression line respectively.

Figure 5: Logarithmic values of undrained shear strengths $\log c_u$ versus actual water content of samples normalized to the liquid limit (V_{H2O}/w_L) (left figures) or versus liquidity index (right figures) of smectites (top) and kaolinite and illite (bottom). Key: $\diamond$ Ca-smectite, $\blacklozenge$ Na-smectite, kaolinite (+) and illite ($\square$). a and b are the intercept and the slope of the regression line respectively.

Normalizing $\log(c_u)$ to the ε of the pore fluid and plotting it against actual fluid content still separates Ca-smectites from Na-smectites (Fig. 6A), but there is no significant correlation between both variables. Using the liquidity index, however, results in two major changes: Both Ca- and Na-smectites merge into a single group (Fig. 6B) and as a novel finding, $\log(c_u)/\varepsilon$ correlates now significantly with the liquidity index (with $p < 0.0001$) (Fig. 6B).

Figure 6: Logarithmic values of undrained shear strengths ($\log c_u$) normalized to ε versus actual fluid content (A) or versus liquidity index I_L (B) for smectites ($\diamond$: Ca-smectite, $\blacklozenge$ Na-smectite). The correlation between $(\log c_u)/\varepsilon$ and the liquidity index (B) is with $R^2 = 0.719$ ($n = 24$) significant, the regression equation for both Na and Ca-smectites reads: $y = 0.0326(22) - 0.0887(118)x$. a and b are the intercept and the slope of the regression line respectively.

For kaolinites and illites, $\log(c_u)/\varepsilon$ results also in linear correlations with the normalized fluid content and the liquidity index, respectively, although with much less significance. For both plots, the slopes and the intercepts of the regression lines for kaolinite and illite are identical within their statistical errors (Fig. 7).

Figure 7: Logarithmic values of undrained shear strengths ($\log c_u$) normalized to ε versus actual fluid content (A) or versus liquidity index I_L (B) for kaolinite (+) and illite ($\square$). The correlations are all significant. a and b are the intercept and the slope of the regression line respectively.

Discussion

A general tendency of c_u to decrease with increasing actual water content (Fig. 5, left side) or with the liquidity index (Fig. 5, right side) has been observed already by Wroth and Wood (1978). In our work,

changes in the particle-particle interactions, reflected here in varying Atterberg parameters, resulted in highly significant correlations between the liquid limit and ϵ , although w_L reacts different to ϵ : for smectites the slopes are positive (Fig. 2A), for kaolinite and illite they are negative and smaller (Fig. 2B). The negative trends for kaolinite and illite show that in the ethanol-water systems other forces than the attractive van der Waals forces may be present, because a decreasing permittivity of the fluid would result in a decreasing Hamaker constant for two like particles (Israelachvili 2002). However, the expected net cohesion could also result from a reduction of repulsive forces between particles. Kaya and Fang (2000) calculated a net reduction of repulsive forces for fluids lower in ϵ than water, but care has to be taken, because they investigated flocculation in comparably dilute suspensions. Alternatively, the particle-particle distances may have changed with increasing amounts of ethanol, but unfortunately, the bulk densities of the systems were not measured.

The two distinctive behaviors for Na- and Ca-smectites (Fig 5A and B) result from different hydration states: Ca-smectite produces a $d(001)$ of around 19 Å in liquid water, whereas Na-smectites can even delaminate to infinite particle distance in the absence of confining pressures, higher ionic strength, or ethanol (Brindley and Ertem 1971; Berkheiser and Mortland 1975). This explains the poorer correlation of $\log(c_u)$ with the liquidity index of the Na-smectites (Fig. 5B). For smectites, ethanol has at least a two-fold impact. First, ethanol replaces water molecules of the hydrated interlayer cations and it changes the interlayer spacings depending on the arrangement of the ethanol molecules around the interlayer cations (Bissada et al. 1967; German and Harding 1971). Dowdy and Mortland (1968) report that Ca- and Na-montmorillonite produced 001 peaks around 16.5 and 13.5 Å, respectively, when the smectites were equilibrated via a gas phase of ethanol. Saturation of Ca- and Na-smectites in liquid ethanol, however, resulted in 001 d-values around 17 Å (Brindley et al. 1969). This agrees with the findings shown in Fig. 6B, where Na- and Ca-smectites merged into one group, because their solvation state becomes similar in ethanol. Part of the variance of $\log(c_u)$ versus the liquidity index shown in Fig. 5B is therefore due to the variance of ϵ , which is eliminated by recalculating $\log(c_u)$ to $\log(c_u)/\epsilon$. The findings of Kuriakose et al. (2017) that the actual water content, and not the liquidity index, might be a better proxy for c_u is not supported by our data (Fig. 5A and 6A).

Conclusions

Undrained shear strengths, c_u , of Na- and Ca-smectites correlate with the actual fluid ratio normalized to the liquid limit (proposed by Kuriakose *et al.* 2017), but this correlation can be improved significantly, first, by normalizing c_u to the ε of the pore fluid and, second, by relating it to the liquidity index I_L . At least for Na and Ca as interlayer cations, it could be shown that a single regression results regardless of the kind of interlayer cation. This will facilitate the prediction of the mechanical behavior of soils in hydrocarbon-contaminated sites.

Acknowledgements

The results presented in this research come from a PhD research, which was funded by the German Federal Ministry of Education and Research (BMBF) in the framework of the special program “Geotechnologien”. The project was “*InProTunnel - Grenzflächenprozesse zwischen Mineral- und Werkzeugoberflächen - Ursachen, Probleme und Lösungsansätze am Beispiel des maschinellen Tunnelbaus*“. The authors would also like to thank the anonymous reviewers, who considerably improved the quality of the manuscript.

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- the dielectric constant and electrolyte concentration of the pore fluids on the undrained shear strength of smectite. *Soils and Foundations*, **50(5)**, 757–763.
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Figure captions

Figure 1: Plastic limits w_p versus liquid limits w_L for smectites in water. Key: ($\diamond$) Ca-smectite, ($\blacklozenge$) Na-smectite, kaolinite (+) and illite ($\square$).

Figure 2: Liquid limits versus ε for smectites (A) and kaolinite and illite (B). Key: $\diamond$ Ca-smectite, $\blacklozenge$ Na-smectite, kaolinite (+) and illite ($\square$)

Figure 3: Plasticity indices I_p versus ε for smectites (A) and kaolinite and illite (B). Key: $\diamond$ Ca-smectite, $\blacklozenge$ Na-smectite, kaolinite (+) and illite ($\square$)

Figure 4: Undrained shear strengths c_u versus w_L for smectites (A) and kaolinite and illite (B). Key: $\diamond$ Ca-smectite, $\blacklozenge$ Na-smectite, kaolinite (+) and illite ($\square$)

Figure 5: Logarithmic values of undrained shear strengths $\log c_u$ versus actual water content of samples normalized to the liquid limit (V_{H2O}/w_L) (left figures) or versus liquidity index (right figures) of smectites (top) and kaolinite and illite (bottom). Key: $\diamond$ Ca-smectite, $\blacklozenge$ Na-smectite, kaolinite (+) and illite ($\square$). a and b are the intercept and the slope of the regression line respectively.

Figure 6: Logarithmic values of undrained shear strengths ($\log c_u$) normalized to ε versus actual fluid content (A) or versus liquidity index I_L (B) for smectites ($\diamond$: Ca-smectite, $\blacklozenge$ Na-smectite). The correlation between $(\log c_u)/\varepsilon$ and the liquidity index (B) is with $R^2 = 0.719$ ($n = 24$) significant, the regression equation for both Na and Ca-smectites reads: $y = 0.0326(22) - 0.0887(118)x$. a and b are the intercept and the slope of the regression line respectively.

Figure 7: Logarithmic values of undrained shear strengths ($\log c_u$) normalized to ε versus actual fluid content (A) or versus liquidity index I_L (B) for kaolinite (+) and illite ($\square$). The correlations are all significant. a and b are the intercept and the slope of the regression line respectively.

Figure 1: Plastic limits wP versus liquid limits wL for smectites in water. Key: (◇) Ca-smectite, (◆)Na-smectite, kaolinite (+) and illite (□).

97x70mm (300 x 300 DPI)

Figure 2: Liquid limits versus ϵ for smectites (A) and kaolinite & illite (B). Key: $\diamond$ Ca-smectite, $\blacklozenge$ Na-smectite, kaolinite (+) and illite ($\square$)

265x373mm (120 x 120 DPI)

Figure 3: Plasticity indices I_p versus ϵ for smectites (A) and kaolinite & illite (B). Key: $\diamond$ Ca-smectite, $\blacklozenge$ Na-smectite, kaolinite (+) and illite ($\square$)

260x376mm (120 x 120 DPI)

Figure 4: Undrained shear strengths c_u versus w_L for smectites (A) and kaolinite & illite (B). Key: $\diamond$ Ca-smectite, $\blacklozenge$ Na-smectite, kaolinite (+) and illite ($\square$)

251x367mm (120 x 120 DPI)

Figure 5: Logarithmic values of undrained shear strengths $\log c_u$ versus actual water content of samples normalized to the liquid limit (VH_2O/w_L) (left figures) or versus liquidity index (right figures) of smectites (top) and kaolinite and illite (bottom). Key: $\diamond$ Ca-smectite, $\blacklozenge$ Na-smectite, kaolinite (+) and illite ($\square$). a and b are the intercept and the slope of the regression line respectively.

297x245mm (120 x 120 DPI)

Figure 6: Logarithmic values of undrained shear strengths ($\log c_u$) normalized to ϵ versus actual fluid content (A) or versus liquidity index IL (B) for smectites ($\diamond$: Ca-smectite, $\blacklozenge$ Na-smectite). The correlation between $(\log c_u)/\epsilon$ and the liquidity index (B) is with $R^2 = 0.719$ ($n = 24$) significant, the regression equation for both Na and Ca-smectites reads: $y = 0.0326(22) - 0.0887(118)x$. a and b are the intercept and the slope of the regression line respectively.

244x344mm (120 x 120 DPI)

Figure 7: Logarithmic values of undrained shear strengths ($\log c_u$) normalized to ϵ versus actual fluid content (A) or versus liquidity index IL (B) for kaolinite (+) and illite ($\square$). The correlations are all significant. a and b are the intercept and the slope of the regression line respectively.

343x315mm (120 x 120 DPI)

Sequence	ε (-)	Undrained shear strength, c_u (kPa)	Material	Fluid content (%)	w_L (mass %)	w_P (mass %)	LI
1	79.50	4.12	Kaolinite	50	57	35	0.68
2	68.15	4.94	Kaolinite	51	58	35	0.70
3	55.07	1.37	Kaolinite	54	60	39	0.72
4	38.28	2.2	Kaolinite	54	61	42	0.64
5	79.50	6.59	Kaolinite	46	57	35	0.50
6	68.15	7.41	Kaolinite	46	58	35	0.48
7	55.07	2.47	Kaolinite	49	60	39	0.48
8	38.28	3.02	Kaolinite	49	61	42	0.37
9	79.50	8.66	Kaolinite	43	57	35	0.36
10	68.15	9.47	Kaolinite	40	58	35	0.22
11	55.07	2.70	Kaolinite	41	60	39	0.10
12	38.28	3.79	Kaolinite	41	61	42	-0.05
13	79.50	12.9	Kaolinite	42	57	35	0.30
14	68.15	18.29	Kaolinite	39	58	35	0.17
15	55.07	8.13	Kaolinite	37	60	39	-0.10
16	38.28	2.2	Kaolinite	40	61	42	-0.11
17	79.50	25.43	Kaolinite	39	57	35	0.18
18	68.15	19.75	Kaolinite	35	58	35	0.00
19	55.07	11.36	Kaolinite	38	60	39	-0.05
20	38.28	5.41	Kaolinite	35	61	42	-0.37
21	79.50	14	Na-smectite	126	455	70	0.15
22	68.15	21.69	Na-smectite	72	201	58	0.10
23	55.07	32.67	Na-smectite	58	127	51	0.09
24	79.50	11	Na-smectite	161	455	70	0.24
25	68.15	21.1	Na-smectite	90	201	58	0.22
26	55.07	31.6	Na-smectite	63	127	51	0.16
27	79.5	2.7	Na-smectite	207	455	70	0.36

28	68.15	1.6	Na-smectite	102	201	58	0.31
29	55.07	0.8	Na-smectite	70	127	51	0.25
30	79.50	3.79	Ca-smectite	93	143	76	0.25
31	68.15	5.77	Ca-smectite	74	119	53	0.32
32	55.07	8.24	Ca-smectite	61	99	48	0.25
33	38.28	12.36	Ca-smectite	47	77	42	0.14
34	24.95	12.08	Ca-smectite	42	73	43	-0.03
35	79.50	4.33	Ca-smectite	88	143	76	0.18
36	68.15	7.96	Ca-smectite	67	119	53	0.21
37	55.07	12.63	Ca-smectite	55	99	48	0.14
38	38.28	16.47	Ca-smectite	44	77	42	0.06
39	24.95	15.92	Ca-smectite	41	73	43	-0.07
40	79.50	5.41	Ca-smectite	84	143	76	0.12
41	68.15	11.53	Ca-smectite	63	119	53	0.15
42	55.07	14.55	Ca-smectite	46	99	48	-0.04
43	38.28	18.4	Ca-smectite	40	77	42	-0.06
44	24.95	17.57	Ca-smectite	39	73	43	-0.13
45	79.50	6.86	Illite	52	58	39	0.72
46	68.15	6.32	Illite	53	61	41	0.58
47	55.07	4.39	Illite	52	66	43	0.40
48	79.5	17.57	Illite	48	58	39	0.46
49	68.15	12.63	Illite	48	61	41	0.36
50	55.07	6.86	Illite	49	66	43	0.25
51	79.50	25.97	Illite	45	58	39	0.32
52	68.15	15.15	Illite	45	61	41	0.20

53	55.07	13.52	Illite	45	66	43	0.09
54	79.50	40.64	Illite	43	58	39	0.22
55	68.15	32.92	Illite	43	61	41	0.10
56	55.07	23.16	Illite	43	66	43	0.00
57	79.50	186.09	Illite	41	58	39	0.11
58	68.15	98.46	Illite	41	61	41	0.00
59	55.07	30.84	Illite	40	66	43	-0.13

Tab. 1 Geotechnical properties for the clays investigated for this research

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