EFFECT OF pH ON THE RHEOLOGY OF MARINE CLAY FROM THE SITE OF THE SOUTH NATION RIVER, CANADA, LANDSLIDE OF 1971

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Abstract—The pH of Na–saturated, carbonate-containing and carbonate-free Leda clay, at salinities of 2 and 10 g/liter, was decreased from pH 8 to 4 by the addition of HCl. The Bingham yield stress, as determined with a coaxial viscometer, increased in all materials as the pH decreased. Above about pH 7 the 2-g/liter materials had a lower yield stress at any water content than the 10-g/liter materials, whereas, below about pH 6.8 the yield stress of the carbonate-containing soil at a salinity of 10 g/liter was lower. For the carbonate-free material, the change occurred at about pH 6.2. The influence of salinity on the remolded shear strength of these materials was pH-dependent. A yield stress increase with decreasing pH was likely due to a change in ion saturation. The carbonate-free material exhibited a maximum yield stress at about pH 5.5–6.2, depending on salinity. The isoelectric points for oxides and clay mineral edges most probably account for the existence of the maximum.

Key Words—Isoelectric point, pH, Quick clay, Remolded strength, Rheology, Salinity, Yield stress.

INTRODUCTION

The remolded response of sensitive marine clays is an important aspect of their geotechnical behavior and is undoubtedly one of the most important factors in determining whether or not a flow slide develops. Soils which have the combination of high sensitivity and low remolded shear strength required for designation as quick clays are particularly susceptible to flow sliding and to other problems associated with post-failure behavior. A number of investigators (e.g., Rosenqvist, 1953; Bjerrum, 1954; Penner, 1965; Torrance, 1975) have demonstrated that a soil's remolded response is influenced by the chemical state of the system. In the chemical methods of stabilization proposed by Bjerrum *et al.* (1967), Moum *et al.* (1968), and Broms and Boman (1979), salts or lime are added to the soil, and the increased pore water salinity and/or the change in ion saturation increase the remolded shear strength. Because some of these treatments may decrease the pH of the surrounding material, the influence of pH decrease on the remolded behavior of Leda clay material was examined in the present investigation.

LITERATURE REVIEW

It has generally been observed (Mitchell, 1976) that high solution pH tends to disperse clay systems. This phenomenon is undoubtedly related to the dissociation of H ions as the pH increases and the resultant increased negative charge on clay particles which increased repulsion between particles and the probability of the dispersion being stable. The reverse occurs at low pH where positive charges dominate the edges of particles and lead to enhanced flocculation by edge-to-face attraction. Low pH also affects the stability of the clay particles.

van Olphen (1977) indicated that acid attacks clay particles at edges, thereby releasing Al ions. Thus, in post-glacial marine clays, acid attacks the less stable minerals present such as carbonates and chlorite, and releases Ca, Fe, Al, and other ions into solution. These ions encourage flocculation.

Bjerrum *et al.* (1967) noted a region of increased plasticity in the acid environment which developed near the anode during electro-osmosis experiments. They concluded that the acid conditions led to the formation of an unstable proton-saturated clay which subsequently changed to an Al-saturated clay having increased plasticity. A similar explanation was offered by Moum *et al.* (1968) to explain shear-strength increases obtained after diffusion of acidic salts into quick clays.

Bentley (1979) suggested that the yield-stress maximum at about pH 6.5 for a marine clay from Gloucester, Ontario, was associated with the isoelectric point of the mineral assemblage present. Given the mineralogy of the Leda clays—quartz, feldspar, illite, chlorite, and various oxides—it is unlikely that the isoelectric point of the whole soil occurs at pH 6.5 because of the considerable permanent charge on illite and chlorite and the low isoelectric points of quartz and feldspar (Yariv and Cross, 1979). Nonetheless, some of the minerals may have isoelectric points about pH 6.5. When comparing Bentley's results with those reported in the present paper, it is important to note that the Gloucester clay contains 1–2% carbonate (Bentley and Smalley, 1979).

Yong *et al.* (1979) used a fall cone apparatus to test the effect of pH on the remolded strength of a Leda clay from Outardes, Quebec, at field moisture content. With a pore water NaCl concentration of 0.05 N (~2.9 g/liter), the maximum remolded strength was obtained when the soil was buffered at pH 6.0. The observed response was attributed "to the fact that the solubility of iron and alumina is very low at a pH of about 6 and that the solubility of silica is also reduced in the presence of iron and alumina at this pH level." How this explains the maximum was not elaborated. The clay at Outardes contains "randomly distributed disintegrated marine shells" (Dascal *et al.*, 1977), but whether carbonates were present in the soil tested is unclear, although shells of *Macoma balthica* were reported in one of the blocks tested.
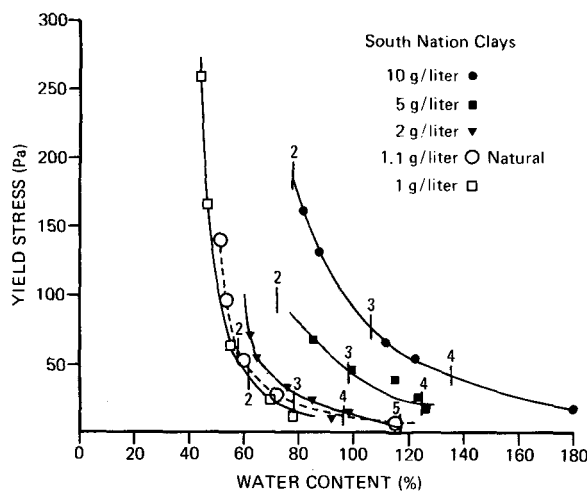
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Figure 1.   Yield stress-water content relationships for Na-saturated clay at selected salinities and for natural South Nation clay.

Table 1.   Pore-water cation composition for natural and Na-saturated samples of Leda clay.

Sample	Salinity (g/liter)	pH	Liquidity index	Na (ppm)	K (ppm)	Ca (ppm)	Mg (ppm)
Natural	1.1	8.6	2.1	380	20	8	6
Na-saturated	1.1	8.6	3.1	380	5	7	2
Na-saturated	2.1	8.5	3.2	750	7	10	3
Na-saturated	5.5	8.6	4.0	2300	18	32	15
Na-saturated	9.0	8.7	5.5	3200	21	59	25

The pH was measured by inserting a combination electrode into the soil material before and after viscometric testing. The pH tended to drift to higher values with time, and the final pH was accepted as the pH of testing. In the carbonate-containing materials the drift was as much as 0.5 units, but normally it was less than 0.2 units per hour; thus, viscometric testing was done under non-equilibrium conditions as far as pH was concerned. In the carbonate-free materials the pH also drifted upward, but the rate of change was slower and was caused by acid attack which released bases from the relatively unweathered minerals.

In these experiments the soil after each pH, water content, or salinity change was mixed using a mechanical mixer until it appeared uniform. This material was placed in the viscometer, and the rotor was maintained at its maximum rotation rate for the 10–15 min required for the temperature to cool to 7°C. The Bingham yield stress was determined from the shear rate-shear stress curve generated by reading the torque required to maintain constant rotation rate 5 sec after the rate had been decreased stepwise by a factor of 2 in each step from 512 to 1 rpm. The yield stress-water content curves for the Na-saturated natural material were found to superimpose in two test runs made six months apart. All other results are on the basis of single tests at each water content, pH, and salinity.

MATERIALS AND METHODS

A Leda clay from the site of the South Nation River landslide of 1971 in eastern Ontario, Canada (Eden *et al.*, 1971) was used in these experiments. The effect of pH on the Bingham yield stress over a range of water contents and salinities was assessed using a Haake Rotovisco RV12 coaxial viscometer equipped with the MVII and MVIII sensors. All experiments were conducted at 7°C. The natural water content of the soil varied between 40 and 47%, and the liquid limit ranged from 34 to 38%. The soil consisted of quartz, feldspar, amphibole, illite, chlorite, and some carbonate minerals and contained ~63% clay-size material.

The soil was homogenized, and the yield stress of subsamples was investigated under the following conditions: (1) At natural pH and natural salinity, over a range of water contents; (2) After Na-saturation by three washings with 1 N NaCl, testing was done at the natural pH over a range of salinities between 1 and 10 g/liter equivalent NaCl; (3) After Na-saturation and HCl addition to adjust the pH to a range of values between the natural pH and pH 4, the material was tested at pore water salinities equivalent to NaCl concentrations of 2 and 10 g/liter as determined by electrical conductivity. Hereafter this sample is referred to as the carbonate-containing sample; (4) After the carbonates were destroyed using HCl, the soil was Na-saturated and the pH and salinity were adjusted in the manner described above. This sample is referred to as the carbonate-free sample; (5) Under conditions of Ca-saturation at the natural pH and at salinities of 2 and 10 g/liter. In all experiments the salinity was monitored by determining the conductivity of a 2-ml sample of pore water squeezed from about 10 ml of soil after the rheological testing.

RESULTS

The yield stress of the natural and Na-saturated samples increased as the water content decreased and, at all water contents, decreased as the salinity decreased (Figure 1). These results are consistent with the increase of the remolded shear strength of the sensitive Canadian postglacial marine clays when their salinity is increased (Torrance, 1975). The natural sample behaved in a manner similar to Na-saturated material of comparable salinity suggesting that at this low salinity it was effectively Na-dominated—a situation which was confirmed by the pore water analysis (Table 1). These results served as the standard against which the yield stress response at altered pH were compared.

Carbonate-containing samples

The yield stress-water content results of the carbonate-containing soil material at 2 and 10 g/liter equiv-
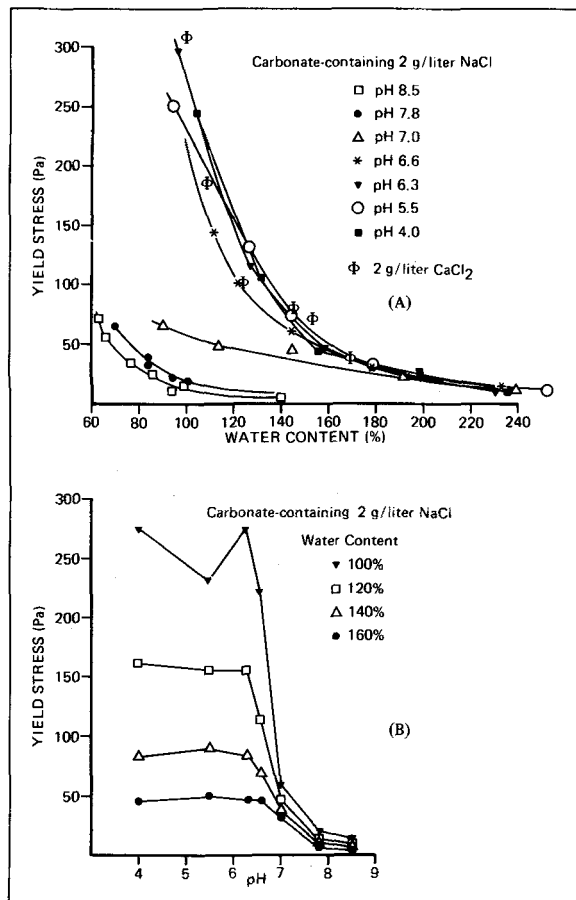
Figure 2. (A) Yield stress-water content relationships for South Nation clay at 2 g/liter salinity and different pHs; (B) derived yield stress-pH relationships at constant water content.
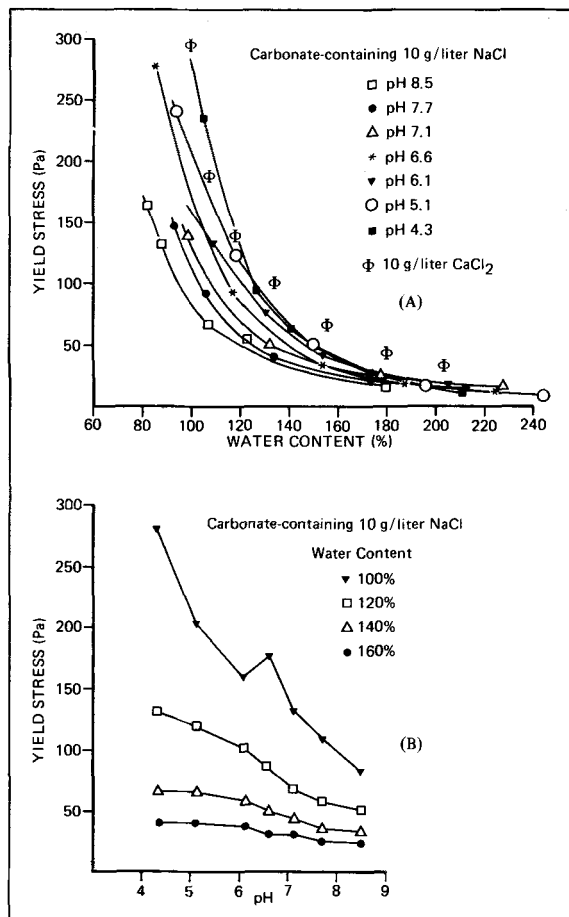

Figure 3. (A) Yield stress-water content relationships for South Nation clay at 10 g/liter salinity and different pHs; (B) derived yield stress-pH relationships at constant water content.

alent salinity, with best-fit curves drawn by eye, are presented in Figures 2a and 3a. At both salinities, higher yield stresses were observed as the pH of the soil material decreased. Figures 2b and 3b show the yield stress-pH relationships at a range of water contents as interpolated, and occasionally extrapolated, from Figures 2a and 3a. The yield stress of all samples increased as the pH decreased. Although the increase commenced immediately upon lowering the pH, the greatest change occurred between pH 7 and 6.5 in the 2-g/liter material. The change was more gradual in the 10-g/liter material. Above pH 7 the 10-g/liter material exhibited a distinctly higher yield stress at all water contents, but below about pH 6.8 the yield stress of the 2-g/liter material was higher. The high salinity moderated the rate of yield-stress increase as the pH decreased. The yield stress remained constant below about pH 6.5 in the 2-g/liter material but continued to rise in the 10-g/liter material, contrary to the results of Bentley (1979) and Yong *et al.* (1979) who found a yield stress max-

imum for carbonate-containing Leda clay, at intermediate pH.

The increased yield stresses were undoubtedly caused by the presence of Na as the dominant pore-water cation at high pH and the presence of Ca, with some Mg and Al, as the dominant cations at low pH (Table 2). The greater flocculating power of the polyvalent cations led to the greater yield stress. At concentrations of both 2 and 10 g/liter the yield stress-water content response changed with decreasing pH from that of a Na-saturated soil to that of a Ca-saturated soil (Figures 2a and 3a).

Carbonate-free samples

The yield stress-water content results for the carbonate-free soil material at 2 g/liter and 10 g/liter equivalent salinity (Figures 4a and 5a) and the derived yield stress-pH relationships (Figures 4b and 5b) also showed a general shift towards higher yield stress as the pH of the soil material decreased. The yield stress
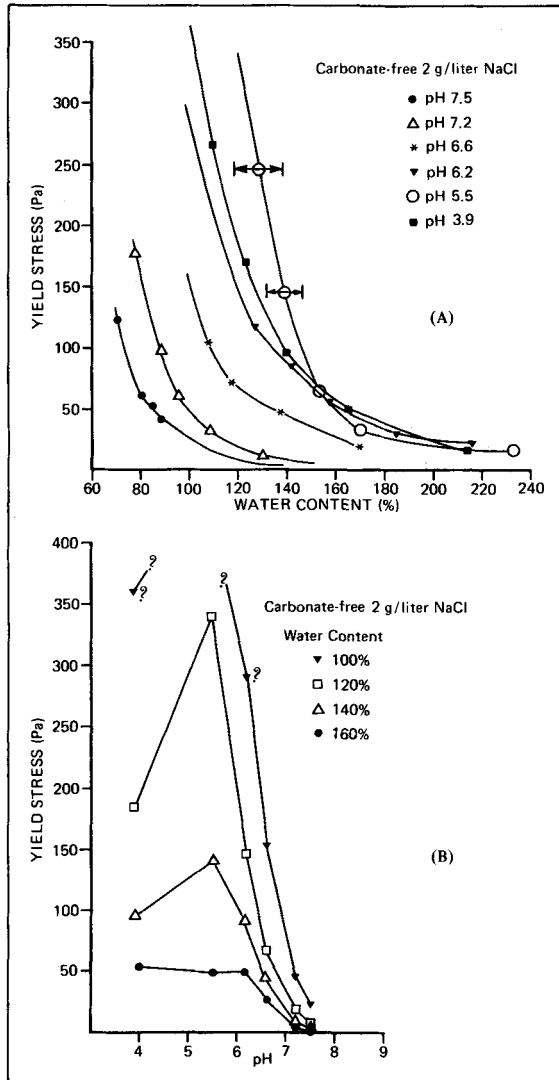
Figure 4. (A) Yield stress-water content relationships for carbonate-free South Nation clay at 2 g/liter salinity and different pHs; (B) derived yield stress-pH relationships at constant water content.
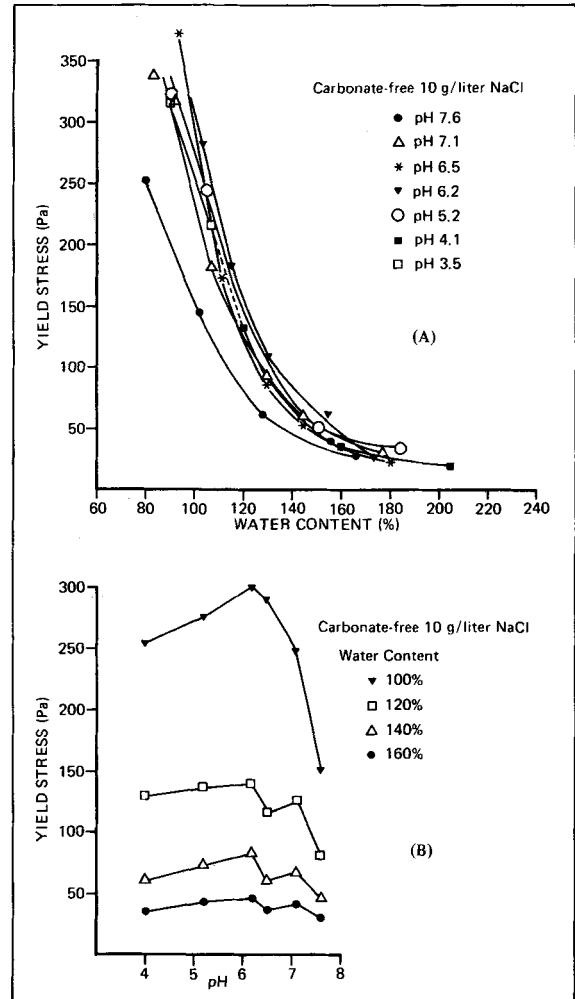

Figure 5. (A) Yield stress-water content relationships for carbonate-free South Nation clay at 10 g/liter salinity and different pHs; (B) derived yield stress-pH relationships at constant water content.

Table 2. Pore-water cation composition for carbonate-containing samples of Leda clay.

pH	Liquidity index	Na (ppm)	K (ppm)	Ca (ppm)	Mg (ppm)	Fe (ppm)	Al (ppm)
			10 g/liter salinity				
7.7	3.9	3600	36	90	46	—	—
7.1	5.4	3300	35	140	61	—	—
6.6	4.5	3200	54	490	100	—	—
6.1	4.1	2100	57	1030	120	—	0.1
5.1	3.6	1200	36	2400	720	24	0.2
4.3	4.3	800	30	1900	900	80	2.1
			2 g/liter salinity				
7.8	3.5	600	10	50	6	—	—
7.0	4.1	700	20	150	35	—	—
6.6	3.6	450	21	270	50	—	—
6.3	3.9	70	15	820	50	—	0.2
5.3	3.3	50	9	540	250	—	0.3
4.0	4.1	70	10	420	190	54	1.9

reached a maximum for the 2-g/liter material at about pH 5.5 at a water content of <150% (Figure 4b). At higher water contents, the yield stress increased to a relatively constant value below about pH 6.2. At water contents <150%, the yield stress at pH 4 was distinctly less than at pH 5.5, suggesting a detrimental effect of low pH on yield stress. The chemical data (Table 3) suggest that with decreased pH and constant pore-water conductivity, the ionic population shifted from dominant Na to dominant polyvalent and hydrogen ions.

For the 10-g/liter material, less change was noted in the yield stress with decreased pH than for the 2-g/liter material. Also, the yield stress of the 2-g/liter material at pH 5.5 and 3.9 and at all water contents was greater

Table 3. Pore-water cation composition for carbonate-free samples.

pH	Liquidity Index	Na (ppm)	K (ppm)	Ca (ppm)	Mg (ppm)	Fe (ppm)	Al (ppm)
		10 g/liter salinity					
7.6	4.1	4500	17	73	10	0.4	0.4
7.1	3.7	4700	23	190	16	0.3	0.4
6.5	3.6	4600	30	430	120	0.2	0.7
6.2	3.5	3800	32	630	100	0.5	0.2
5.2	3.8	2900	24	710	470	0.3	0.3
4.0	3.5	3400	41	490	320	480	3.4
		2 g/liter salinity					
7.5	3.4	700	5	20	2	—	—
7.2	3.9	700	6	30	4	—	—
6.6	4.5	800	9	100	13	—	—
6.2	3.7	600	12	280	76	—	0.2
5.5	3.2	50	8	440	230	—	0.2
3.9	3.4	80	13	270	150	65	3.2

Figure 6. Liquid limit as influenced by pH for the South Nation clay materials.

than that of the 10-g/liter material at the same pH and water content. Whereas the yield stress of the 10-g/liter material at constant water content increased with decreased pH, the increase was less than for the 2-g/liter material, and the tendency for a maximum at approximately pH 6.2 was less pronounced.

DISCUSSION

Regulating the "salinity" by maintaining a constant conductivity at different pH actually decreased the pore water salinity, as normally understood, as the pH decreased, due to the much greater conductance of the hydrogen ion compared with other ions in the system (Castellan, 1964, p. 593). Inasmuch as the measured "salinity" was an overestimate, and because salinity differences affected the yield stresses measured and the magnitude and direction of pH-dependent change, the yield stresses were sometimes underestimated and sometimes overestimated compared with what would have been expected if the ionic strength of the pore water had been held constant. Because the nominally 2-g/liter material exhibited the higher yield stress at low pH, the measured yield stress at low pH may have been an overestimate. This overestimation has little effect on the interpretation of the results for the carbonate-containing material but, for the carbonate-*free* material, the maxima at intermediate pH may be less pronounced than indicated in Figures 4b and 5b. Regardless of the above effect, decreasing the pH caused an increase in the yield stress at constant water content, suggesting that the remolded shear strength at natural water content should have increased as well.

The presence of carbonates influenced (1) whether or not the yield stress-pH relationship exhibited a maximum, and (2) the magnitude of the yield stresses exhibited at any water content. If carbonates were present, the yield stress increased as pH decreased, at least to pH 4; whereas, for carbonate-free material, a max-
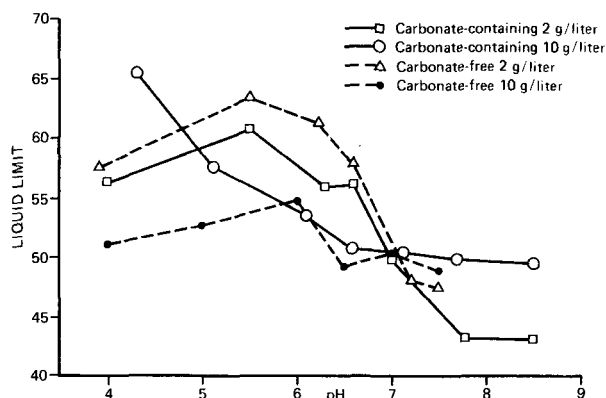
imum yield stress was at pH 5–6 and became more pronounced at low salinities and lower water contents. At 2 g/liter, the carbonate-free material exhibited the lower yield stress above pH 7; between pH 7 and ~6.3 the carbonate-free sample exhibited lower yield stresses at >140% water content, whereas the yield stresses were similar at <120% water content. Below pH 6.3, the carbonate-free material exhibited higher yield stresses for water contents <140%; the yield stresses were comparable at higher water content. At 2 g/liter, the carbonate-free material had the higher liquid limit below about pH 6.8; the liquid limits were similar above pH 6.8 (Figure 6). The responses of these two measures of rheological behavior to pH change under low-salinity conditions were consistent. At 10 g/liter, the carbonate-free material exhibited the higher yield stress at pH 7.6–5 and a similar or marginally lower yield stress at pH <5. At both salinities, carbonate affected the response of the yield stress as the pH decreased.

Increasing salinity at natural pH increased the yield stress at constant water content (Figure 1). As the pH decreased, this relationship changed. When carbonate was present before pH change, the 10-g/liter material exhibited the higher yield stress above pH 6.8, and the 2-g/liter material had the higher yield stress below pH 6.8, with the exception of the lowest pH, lowest water content test. The liquid limits (Figure 6) followed a similar pattern. When carbonate was removed before Na–saturation and pH adjustment, the 2-g/liter material exhibited the higher yield stress below pH 6.2–6.5 and the lower yield stress at higher pH. The liquid limits followed a similar pattern, but the crossover point was at approximately pH 7. This interplay between salinity and pH, as they affected yield stress of the remolded soil, is intriguing, and the greater yield stress below about pH 7 exhibited by the lower salinity material suggests that the hydrogen ions and the polyvalent ions released by acid attack on the mineral par-

ticles, had a greater effect when the competition from other ions was weakest, as in the low salinity, carbonate-free material.

The ion saturation data explain the yield stress increase with decreasing pH, but the isoelectric points may have contributed to the maximum noted for the carbonate-free materials. Flegmann *et al.* (1969) noted for a suspension of 5 g kaolinite per 100 ml a yield-stress maximum at pH 5.75 for Na-kaolinite in 10^{-4} M NaCl, at pH 7.0 for H-kaolinite in distilled water, and at pH 7.95 for Al-kaolinite in distilled water. These maxima coincided with the isoelectric points of the respective kaolinites, as determined by ion-adsorption experiments. The low permanent charge on kaolinite (Grim, 1968) should yield an isoelectric point equal to the pH of maximum interaction between clay particles and, hence, of maximum yield stress. In Leda clay, between pH 8.5 and 4, all primary minerals and clay minerals should bear a negative charge. The aluminum oxides, however, have an isoelectric point, although varying with purity and solution conditions, at about pH 7 (Siffert, 1978). Likewise, edges of the clay mineral particles, where the Al-containing octahedral sheet is exposed, probably have isoelectric points between pH 5 and 9 (I. Th. Rosenqvist, Institute of Geology, University of Oslo, Blindern, Norway, personal communication, 1983).

Because the edges of clay minerals have isoelectric points and are positively charged below this pH, their interaction with negatively charged mineral particles increases below their isoelectric point. Such a reaction, along with changing ion saturation, partially explains the increased yield stresses noted as the pH decreased, but it does not entirely explain the maximum between pH 5 and 6 for the carbonate-free material, unless the isoelectric point of other minerals in the soil was reached and mutual repulsion of particles took place, as at high pH when all particles were negatively charged. The presence of oxide coatings, with isoelectric points around this pH on some otherwise negative mineral particles should increase the possibility of mutual repulsion at low pH.

The influence of pH on the yield stress of Leda clay is not a simple, straight-forward relationship, but a complex function of pH, salinity, water content, and mineralogy. In all of the experiments conducted, decreasing the pH below the natural value increased the yield stress, but the magnitude of the change depended on the material and the salinity. If pH decrease is considered as a means of improving the geotechnical behavior of the marine clays, the remolded strength should be increased thereby reducing the sensitivity. Such treatment does not necessarily represent an improvement because in carbonate-containing materials, the destruction of the carbonates could lead to a drastic drop in the undisturbed strength (Moum *et al.,* 1968), due to the evolution of carbon dioxide and the elim-ination of the cementing properties of the carbonates. For sensitive marine clays of the Leda type, deliberately decreasing the pH should improve the soil only if it is carbonate-free and if disturbance of the original structure is not of consequence.

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Резюме—Величина pH Na-насыщенной, карбонато-содержащей и карбонато-свободной глины Леда, при солености 2 и 10 грамм/литр, уменьшалась от 8 до 4 при добавлении HCl. Величина предела текучести Бингама, определенная при помощи коаксиального вискозиметра, увеличивалась для всех материалов, когда величина pH уменьшалась. Выше значения pH около 7, материалы при солености 2 грамм/литр имели предел текучести более низкий при всех содержаниях воды, чем материалы при солености 10 грамм/литр, хотя ниже значения pH около 6,8 предел текучести почвы, содержащей карбонат при солености 10 грамм/литр, был более низкий. Для материала, несодержащего карбонат, это изменение получилось при pH = 6,2. Влияние солености на прочность на срез материалов зависело от величины pH. Увеличение предела текучести при уменьшении значения pH было, вероятно, результатом изменения насыщения. Материалы, несодержащие карбонае, давали максимальное значение предела текучести при pH между 5,5–6,2, в зависимости от солености. Изоэлектрические точки для окисей и граней глинистых минералов являются, наиболее вероятно, причиной присутствия этого максимума. [E.G.]

Resümee—Der pH-Wert von Na-gesättigtem, Karbonat-haltigem und Karbonat-freiem Leda-Ton nahm bei Salinitäten von 2 und 10 g/l durch die Zugabe von HCl von pH 8 auf pH 4 ab. Die Bingham Fließgrenze die mit einem koaxialen Viskosimeter bestimmt wurde, nahm in allen Substanzen zu, wenn der pH abnahm. Oberhalb von etwa pH 7 hatten die 2 g/l-Substanzen bei jedem Wassergehalt eine geringere Fließgrenze als die 10 g/l-Substanzen. Unterhalb von etwa pH 6,8 war die Fließgrenze des Karbonat-haltigen Bodens bei einer Salinität von 10 g/l jedoch niedriger. Bei dem Karbonat-freien Material trat die Veränderung bei etwa pH 6,2 ein. Der Einfluß der Salinität auf die Restscherfestigkeit dieser Substanzen war pH-abhängig. Eine Zunahme der Fließgrenze mit abnehmendem pH beruht wahrscheinlich auf Veränderungen in der Ionensättigung. Das Karbonat-freie Material zeigte eine maximale Fließgrenze bei einem pH-Wert von etwa 5,5–6,2 je nach Salinität. Die isoelektrischen Punkte für Oxide und Tonmineralkanten sind höchstwahrscheinlich für die Existenz dieses Minimums verantwortlich. [U.W.]

Résumé—Le pH d'argile Leda, saturé de Na, avec et sans carbonate, à des salinités de 2 et 10 g/litre a été diminué du pH 8 à 4 par l'addition d'HCl. La force de Bingham nécéssaire pour faire céder le matériau, determiné avec un viscomètre coaxial, a augmenté dans tous les matériaux au fur et à mesure que le pH diminuait. Au dessus d'à peu près pH 7 la force nécéssaire pour faire céder les matériaux 2-g/litre était plus basse pour toute teneur en eau que pour les matériaux 10-g/litre, tandis qu'en dessous du pH 6,8, la force nécéssaire pour faire céder le sol contenant du carbonate à une salinité de 10 g/litre était plus basse. Le changement s'est produit à à peu près pH 6,2 pour le matériau sans carbonate. L'influence de la salinité sur la résistance à la tension remoulée de ces matériaux était dépendante du pH. Une augmentation de la force nécéssaire pour faire céder les matériaux correspondant à une diminution du pH était probablement due à un changement de saturation d'ions. La force nécéssaire pour faire céder le matériau sans carbonate a atteint un maximum à à peu près pH 5,5–6,2, dépendant de la salinité. Les points isoélectriques pour les oxides et les bords des minéraux argileux rendent probablement compte de l'existence de ce maximum. [D.J.]