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Influence of Pore Fluid Composition on Volume Change Behaviour of Clays Exposed to the Same Fluid as the Pore Fluid

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Abstract. This paper reports experimental results relative to the influence of pore fluid composition on volume change behaviour of four different clayey soils: the Ponza bentonite, a commercial kaolin, the Bisaccia and the Marino clays. Oedometer tests were carried out on dry materials and on the materials reconstituted with distilled water, concentrated NaCl solutions and a non polar fluid (cyclohexane). The influence of ethanol was analysed only in the case of the Bisaccia clay. All the tests were carried out by exposing the soils to the same fluid as the pore fluid. The results show that the materials prepared with cyclohexane behave similarly to the dry ones and they both are less compressible than the materials prepared with aqueous solutions. The coefficient of consolidation increases dramatically and swelling is negligible in the whole considered stress range. Volume change parameters strongly depend on the dielectric constant of the pore fluid.

1 Introduction

It is well known that the volume change behaviour of clays is greatly influenced by mineral composition. The influence is particularly strong when the pore fluid is distilled water, as shown by Figure 1 which compares the compression and swelling curves of the Ponza bentonite and a commercial kaolin prepared with and immersed in distilled water. As expected, the behaviour of the two clays – reconstituted at about the liquid limit - is very different. In particular, kaolin compression and swelling curves are more similar to those of a loose sand than to those of the other clay.

The influence of pore fluid composition depends on the type of soil. In the case of very active montmorillonite, experimental results show that compressibility and swelling decrease with increasing pore liquid ionic force, or with decreasing dielectric constant (Bolt, 1956, Kenney, 1967; Kinsky et al., 1971; Olson and Mesri, 1970; Mesri and Olson, 1971; Sridharan, 1991; Mitchell, 1993; Di Maio, 1996b). Changes in compressibility and swelling caused by pore fluid composition variations can be almost the same as those caused by mineral composition variations. In particular, the compression curve of the Ponza bentonite reconstituted with saturated salt solutions is more similar to that of kaolin than to that of the water-saturated bentonite (Figure 2). Depending on the particular value of its specific surface, kaolinite may or may not be influenced by pore fluid composition. Sridharan and Ventakappa Rao (1973) found that kaolinite may undergo consolidation as an effect of an increase in the dielectric



Figure 1. Oedometer compression and swelling curves of water – saturated Ponza bentonite, commercial kaolin and sand (Di Maio et al., 2003).



Figure 2. Oedometer compression and swelling curves of kaolin and Ponza bentonite reconstituted with and immersed in distilled water and saturated NaCl solution (Di Maio et al., 2003).

constant of the pore fluid. The Authors hypothesized that kaolin volume changes are related to the interparticle forces which control particle sliding. If the ionic strength of pore fluid decreases, or if the dielectric constant increases, the resistance to particle movements decreases, thus allowing a reduction in porosity. Chen et al. (2000) observed that the compression index of kaolin varies with the dielectric constant D of the pore organic fluid similarly to the Hamaker constant, and it exhibits a minimum at about D = 24.

Generally, results relative to materials prepared with aqueous ion solutions are explained, at least qualitatively, in terms of diffuse double layer models. The effects of organic non polar solvents - attributed to the suppression of the diffuse double layer - are often analysed in terms of the pore fluid dielectric constant. The purpose of this paper is to compare directly the influence of extreme types of fluids (water, air, concentrated salt solutions, non-polar organic fluids) on different types of soils. To this aim the materials were reconstituted at about their maximum porosity and the results were analysed with reference to a unique parameter: the pore fluid static dielectric constant. The Ponza bentonite and the commercial kaolin, since they are mainly composed of Na-montmorillonite and kaolinite respectively, represent extreme types of clay soils. In order to analyse the influence of the smectitic component on natural soil behaviour, two clays with different smectite percentages, the Bisaccia and the Marino clays, were also tested.

2 Materials

Particle size distribution curves of the tested soils are reported by Figure 3. Table 1 reports mineral composition determined by X-ray powder diffraction analysis, using Cu-K α radiation, and the liquid limit evaluated by means of the fall cone test on the powdered soils prepared with distilled water.



Figure 3. Grain size distribution curves.

	Gs	w _L (%)	kaolinite %	illite %	smectite %	
Ponza bentonite	2.77	390	20	-	70 -80	-
						Clorite 10%
Bisaccia clay	2.78	110	10	20	30	Quartz 15%
						Calcite 10%
						Feldspars 5%
kaolin	2.63	50	75 - 80	8 - 10	<5	Quartz and
						feldspars 10%
Marino clay	2.75	50	30	10	Mixed layer	chlorite 10%
					10	quartz 30%-40%

Table 1. Specific weight G_s, liquid limit w_L, mineral percentages.

The limit water content was evaluated also by mixing the soils with NaCl solutions at various concentrations. The results show (Figure 4) that w_L decreases with increasing salt solution molarity, with the exception of the commercial kaolin which seems to be unaffected by pore fluid composition.

Water retention properties were evaluated more accurately by settling tests carried out following Sridharan & Prakash (1998). Several suspensions were prepared by mixing 25 g dry material with different quantities of distilled water or saturated NaCl solution, and they were left to settle. For any initial water content of the suspension w_i , water retention capability w_f of the Bisaccia clay in the concentrated salt solution is much lower than in distilled water, consistently with the fall cone test results (Figure 5). On the contrary, water retention capability of kaolin in the salt solution is higher than in distilled water. So, pore liquid composition influences the kaolin behaviour, and the type of influence is opposite to that on the smectitic clay, as found by Sridharan & Ventakappa Rao (1973).

The difference in the type of influence is shown also by Figure 6, which reports two photographs relative to the Ponza bentonite and the commercial kaolin at about four years from the beginning of sedimentation tests. The samples were prepared with 40 g/l dry clay. The material on the left of each photograph sedimented in distilled water, whereas the material on the right hand side in a saturated NaCl solution. It can be observed that, in the case of the Ponza bentonite, the sediment volume decreases with increasing pore solution concentration whereas it increases in the case of kaolin. Time trend of consolidation too is strongly dependent on pore fluid composition, as shown by Figure 7 which reports the average void ratio of the sedimentting Bisaccia clay in distilled water and in a 1 M NaCl solution. The material consolidates in a few minutes in a concentrated salt solution, whereas, in distilled water, after 4 years from the beginning of the test, it has not reached the equilibrium yet!

The limit water contents of the Ponza and Bisaccia clays were evaluated also with cyclohexane and ethanol. For the Bisaccia clay also with dimethylsulfoxide, a fluid whose dielectric constant is equal to that of saturated NaCl solution. The obtained values, together with those obtained with the aqueous solutions, seem to be well correlated to the static dielectric constant D of the pore fluid (Figure 8). In particular, it is worth noting that w_L of the Bisaccia clay



Figure 4. Liquid limit w_L against pore solution molarity. The limit, determined by fall cone test, is defined as the weight of water divided by the weight of solid (without salt).



Figure 5. Equilibrium water content of the sediment volume w_f against initial water content of the suspension w_i .



Figure 6. Sediment volume of the Ponza bentonite (on the left) and of the commercial kaolin (on the right) in distilled water and in a saturated salt solution, about four years after the beginning of the test.



Figure 7. Average void ratio against time for the Bisaccia clay sedimenting in distilled water and in 1 M NaCl solution.



Figure 8. Liquid limit against pore fluid static dielectric constant.

reconstituted with saturated NaCl solution is very close to w_L of the material prepared with dimethylsulfoxide.

The intrinsic compressibility $C_c^* = e^*_{100} - e^*_{1000}$ (with e^*_{100} and e^*_{1000} the intrinsic void ratios at $\sigma'_a = 100$ kPa and 1000 kPa respectively) varies almost linearly with the void ratio e_L at the liquid limit (Burland, 1990). So, the results reported by Figure 8 suggest that an analogous dependence on pore fluid dielectric constant will be shown by the compression index.

3 Methods

Compressibility was evaluated by means of fixed-ring oedometer tests on specimens submerged in the same fluid as the pore fluid. Four sets of materials were reconstituted with four different fluids. The first set consisted of the natural soils reconstituted by mixing the air-dried powders with distilled water. The second set was constituted by the air-dried powdered materials mixed with NaCl solutions at various concentrations. Also in this case, the initial solution content was equal to - or higher than - the liquid limit evaluated with the same solution. For the Ponza bentonite and the Bisaccia clay reconstituted with water or with aqueous ionic solutions, the liquid limit state was verified to be a reference state. In fact the compression curves relative to several different initial solution contents higher than w_L were found to converge at low values of σ'_a (Figure 9). Another set of tests was carried out on the materials saturated with cyclohexane, an organic non polar fluid whose static dielectric constant is D = 2.0 at 20°C. The powdered soils were oven-dried at 105°C for three days. In this case it was impossible to test the materials for fluid contents higher than the liquid limit because self-weight consolidation rapidly occurred. So, the materials were rapidly mixed with the maximum liquid content at which apparently there was no self-weight consolidation. They were placed in consolidation cells, immersed in cyclohexane, and the cells were sealed. The last set of tests was carried out on "dry" specimens. The materials, dried at 105°C for 3 days, were prepared in thin layers, with an initial void ratio close to that of the materials prepared with cyclohexane.

The specimens - 2 cm thick - were loaded and subsequently unloaded by steps, doubling and halving respectively the external load. Only in the case of the Bisaccia clay reconstituted with distilled water, a great tendency of the material to extrude required smaller axial stress increments. Each load was sustained long enough for the completion of primary compression or swelling and the development of secondary volume strains.



Figure 9. Compression lines for initial liquid contents higher than the liquid limit.

4 Results

The comparison of the effects of different types of pore fluids is reported by Figure 10.a for the Ponza bentonite. The figure reports the oedometer compression and swelling curves of a specimen reconstituted with distilled water, of a specimen reconstituted with a saturated NaCl solution, one prepared with cyclohexane and the curves relative to dry material. The figure shows that:

- at a given initial void ratio the behaviour of the material prepared with cyclohexane is very similar to that of the dry material. It is worth noting that the two pore fluids have similar static dielectric constant;
- b. the compression curves of both cyclohexane-saturated and dry materials intersect the normal compression lines of the material reconstituted with aqueous solutions, clearly indicating an increase in shear resistance at the particles' contacts;





Figure 10. Comparison among oedometer curves relative to the Ponza bentonite (a) and the Bisaccia clay (b) dry, and reconstituted with distilled water, saturated NaCl solution and cyclohexane.

A behaviour qualitatively similar to that of the Ponza bentonite is exhibited by the Bisaccia and Marino clays (Figures 10.b and 11.a respectively), with the effects decreasing as the smectitic percentage decreases. Contrarily to the other materials, the slope of the compression curve of kaolin reconstituted with cyclohexane is slightly higher than that obtained by using aqueous solutions (Figure 11.b).

The differences are best seen in Figure 12 which reports the compression index C_c - evaluated for an increment of axial stress from 150 kPa to 300 kPa - against static dielectric constants of the considered pore fluids. It can be observed that the dependence of C_c on D is very strong. Furthermore, it can be observed that differences among the different materials become noticeable for values of D higher than about 70. Analogous trend can be observed for the swelling index C_s (Figure 13). Figure 14, which reports C_c^* against e_L for the considered soils reconstituted with water, with the NaCl solutions and with organic solvents, compares these results to those reported by Burland (1990) and to the line $C_c^* = 0,256e_L - 0,04$ found by the Author as the best fit regression line to data reported in the literature. The point relative to the Ponza bentonite reconstituted with distilled water has not been reported because it is out of the range of validity of the relation (which holds for $0.6 < e_L < 4.5$), and in fact it would lie well above the regression line. The figure shows that there is a good agreement among data obtained by mixing different materials with distilled water and those obtained by mixing the same material with other pore fluids. So, the state of the materials at the liquid limit can be considered



Figure 11. Comparison among oedometer curves relative to the Marino clay (a) and the commercial kaolin (b) dry, and reconstituted with distilled water, concentrated NaCl solution and cyclohexane.



Figure 12. Compression index evaluated in the range 150 kPa - 300 kPa against pore fluid static dielectric constant.



Figure 13. Swelling index C_s in the range 150 kPa - 300 kPa against pore fluid static dielectric constant.



Figure 14. Intrinsic compression index $C_c^* = e^*_{100} - e^*_{1000}$ against void ratio e_L at the liquid limit.

	e_L	C_c^*	e_L	C_c^*
distilled water	10.8	3.80	3.06	1.05
0.5 M NaCl			1.97	0.45
0.6 M NaCl	3.43	0.85		
1 M NaCl	2.70	0.59	1.77	0.48
sat. NaCl solution	1.77	0.42	1.41	0.37
ethanol			1.61	0.42
cyclohexane	2.09	0.33	1.03	0.19

Table 2. Intrinsic compression index $C_c^* = e^*_{100} - e^*_{1000}$ against void ratio e_L at the liquid limit.Ponza bentoniteBisaccia clay

as a reference state also in the case of pore liquids different from water. Data relative to Ponza bentonite and Bisaccia clay are reported by Table 2.

A high influence of mineral and pore fluid composition is observed also on time trend of volume change. The influence of mineral composition is best seen on water-saturated materials. The coefficients of consolidation c_v and swelling c_s - determined on the experimental curves of displacements by the log-time method based on the Terzaghi model - are reported against axial stress in Figures 15 and 16 respectively for the extreme types of soils, bentonite and kaolin, and for their mixtures at various percentages. For bentonite contents equal to 50% and 80%, the curves are very close to that of 100% bentonite; furthermore, c_v decreases with axial stress increasing. For bentonite percentages equal to or lower than 20%, c_v and c_s increase with decreasing bentonite content, as expected on the basis of previous experimental results (among others: Yin, 1999), and increase with axial stress. The results obtained for the Bisaccia and Marino clays show that the values of c_v and c_s are close to those relative to the bentonite – kaolin mixtures containing similar percentages of montmorillonite (Di Maio et al. 2003).

The influence of pore solution concentration on c_v and c_s is noticeable in all the considered stress range (Figure 17). The difference between the material reconstituted with distilled water and that reconstituted with 1 M NaCl solution is of about two orders of magnitude in the case of the Ponza bentonite, lower for the Marino clay. The Bisaccia clay behaves similarly to the Ponza bentonite, whereas no influence was found for kaolin. For all the materials prepared with the organic fluid, consolidation occurred in a few seconds. Only under the highest stress levels for the Ponza and Bisaccia clays c_v could be determined by the *log-time* method, and a value of 0.015 cm²/sec was obtained. This value is much higher than that obtained for the materials reconstituted with aqueous solutions.



Figure 15. One-dimensional coefficient of consolidation c_{ν} against axial stress for bentonite-kaolin mixtures reconstituted with distilled water and for the Bisaccia and Marino clays. Curves **a** and **b** were determined by Robinson and Allam (1998) for a montmorillonite and a kaolinite respectively.



Figure 16. One-dimensional coefficient of consolidation c_s against axial stress for bentonite-kaolin mixtures reconstituted with distilled water and for the Bisaccia and Marino clays.



Figure 17. One-dimensional coefficients of consolidation c_v and swelling c_s against axial stress for the Ponza bentonite reconstituted with distilled water, with NaCl solutions and cyclohexane.

Under the hypothesis of the Terzaghi model of one-dimensional consolidation, a first approximate value of the hydraulic conductivity k in the axial direction can be obtained from the relation $c_v = kE_{ed}/\gamma_w$. Figure 18 reports k against void ratio for the considered soils and for mixtures bentonite-kaolin with a given percentage of bentonite. As expected, differences up to four degrees of magnitude are found between kaolin and bentonite; 10% bentonite is sufficient to cause large decrease in permeability with respect to kaolin; the values obtained for the Bisaccia clay and for the Marino clay are very close to those obtained for the artificial mixtures with a similar percentage of montmorillonite. It is interesting to observe that variations due to pore fluid are of the same order of magnitude as those due to mineral composition and grain size. In the case of the Ponza bentonite reconstituted with cyclohexane, the value of k is practically equal to that obtained for kaolin at the same void ratio (Figure 19). Furthermore, analogous variations are found in terms of intrinsic permeability.



Figure 18. Hydraulic conductivity against void ratio of bentonite-kaolin mixtures and of the Bisaccia and Marino clays reconstituted with distilled water.



Figure 19. Hydraulic conductivity of the Ponza bentonite with different types of pore fluids.

5 Conclusions

Oedometric compression experiments are presented with different basic skeleton minerals, different smectite contents, and finally different pore fluids. Compressibility and unloading moduli in one -dimensional strain states, as well as the initial void ratio, are the study focus. The results show that:

a) Volume change behaviour of artificial mixtures and natural soils reconstituted with distilled water is strongly influenced by mineral composition and, in particular, by the smectite fraction. The influence depends on the stress level. With increasing axial stress, the influence on compression and swelling indices decreases, whereas it increases on the coefficients of consolidation and swelling.

b) An increase in pore solution concentration makes the materials compressibility decrease and the coefficients of consolidation and swelling increase, with the exception of c_s at low stress levels and for high smectite content. The differences among the different materials reduce greatly.

c) The materials prepared with cyclohexane behave more similarly to the dry ones than to those prepared with aqueous solutions. Compressibility is much lower, the coefficient of consolidation increases dramatically and swelling is negligible in the whole considered stress range.

d) The great differences in behaviour among different clayey soils are essentially due to their different interaction with water or with high dielectric constant fluid.

Further experimentation with fluids different from water but with the same dielectric constant - or with dielectric constant higher than 80 - is now under examination in order to complete the analysis.

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