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Hydraulic and mechanical behaviour of cement-bentonite mixtures containing HYPER clay: impact of sulfate attack

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Abstract. Cemented clays are regularly employed as cut-off walls to isolate polluted soils or in ground improvement technologies. The objective of this research was to evaluate the performance of a polymer-treated bentonite (HYPER clay) and its impact on hydraulic and mechanical properties of cement-bentonite (CB) mixtures in contact with sulfates. In this research, bender elements were installed in a hydraulic conductivity cell, to simultaneously monitor the small-strain shear modulus (G_0) and the hydraulic conductivity (k) of cement-bentonite mixtures. Initially, during permeation with deionized water, an increase of G_0 with time was observed, due to cement hydration (hardening). Conversely, after prolonged permeation with sulfates, a decrease of G_0 and a gradual increase of k are expected due to degradation of the cemented material. Preliminary results show that k of mixtures containing untreated bentonite increases due to contact with sulfates; conversely, k of mixtures containing HYPER clay remains unaffected. The stiffness modulus G_0 was not significantly affected after a sulfate contact period of about 100 days in none of the cement-bentonite mixtures. More extensive monitoring is required for evaluating long-term mechanical degradation. These observations suggest that the use of polymer-treated bentonites could improve the resistance of cement-bentonite mixtures to sulfate attack maintaining a low hydraulic conductivity.

Keywords. Clay, cement, sulfate attack, hydraulic conductivity, stiffness

1. Introduction

Cement-clay grouts are often used in contaminated soils either as parts of barrier systems (e.g. cut-off walls) or for general ground improvement applications [1]. Deterioration of concrete by sulfates of an external source is a commonly observed durability problem in cement structures exposed to seawater, contaminated soils or groundwater containing high concentrations of sulfate ions. This durability problem, also known as sulfate attack, occurs after a series of chemical reactions between sulfate ions, cement paste and moisture [2].

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The two manifestations of sulfate attack are expansion, caused by formation of ettringite and gypsum, and loss of strength and mass, caused by the deterioration of the cohesiveness of the cement matrix [3].

For the design and the quality control of cut-off walls, cement-clay samples are studied to evaluate their strength, hydraulic conductivity and chemical compatibility [4]. The small-strain shear modulus (G_0) could be used to correlate the strength of a cement-clay mixture [5]. G_0 may be obtained from shear-wave velocity measurements using bender elements. This technique was pioneered by [6], improved and studied by many subsequent researchers [7,8, 9, 10].

Bender elements have been installed in a flexible-wall hydraulic conductivity cell, to monitor (in a nondestructive way) G_0 as well as the hydraulic conductivity, simultaneously. In a previous work, in a cemented sample permeated with deionized water, an increase in G_0 with time was observed with increasing interparticle cementation [11]. Conversely, a decrease of G_0 may be expected due to the impact of aggressive solutions, such as sulfates.

In this study, experiments were carried out to analyze the impact of sulfate attack on cement-bentonite mixtures containing untreated bentonite and polymer-treated bentonite using the HYPER clay technology [12] to evaluate their performance.

2. Materials and sample preparation

For this research a sodium-activated bentonite clay was treated with HYPER clay technology, by mixing the clay with a polymeric solution containing an anionic polymer, CarboxyMethyl Cellulose (CMC), and dehydrating the slurry at 105°C for about 24 hours. The chosen polymer dosage was 8% by dry weight (8 g of polymer for 100 g of clay).

The samples studied in this research consist of a mixture of bentonite clay (treated and untreated), cement and water. Blast furnace slag cement of the type CEM III/B 32.5 N-LH / SR (26% Portland clinker, 70% blast furnace slag, 4% limestone) and purified water with an electrical conductivity EC $\leq$ 10 μS/cm and a pH of about 7.6 were used.

Deionised water, produced using a water purification system, was used to prepare the samples and as reference permeant solution. During hydraulic conductivity testing, the cement bentonite sample was initially permeated with purified water for 1 month to allow for further hydration of the cement products. After that period, the sample was permeated with a 25 g/L solution of Na_2SO_4 to study the impact of sulfate attack. Such high Na_2SO_4 concentration was chosen here to accelerate the degradation process; however, it may be too high to represent common sulfate exposure levels in the field.

Some physical properties and chemical composition of the clays and cement used here are summarized in Table 1 and Table 2.

The prepared samples consist of 80.4 % water, 16.1 % cement and 3.5% bentonite (by weight). This composition is in agreement with other studies in the literature [1, 13, 14]. First, a slurry of bentonite and water was mixed with a high-speed shear mixer for 5 minutes. The slurry was poured in a closed container and allowed to hydrate for about 24 hours. Subsequently, the cement was added to the 24-hour hydrated bentonite slurry and mixed with a high-speed shear mixer for about 2 minutes.

Then, the fresh CB slurry was poured in stainless-steel molds to prepare cylindrical specimens. The bottom and top ends of the mold were sealed with plastic foil and silicon grease to prevent moisture loss. Then, the samples were allowed to cure for about 7 days in a conditioned room at 18°C. After that period, when the samples showed enough strength to be handled, they were carefully extruded out of the molds.

Three different sample sizes were produced with a diameter D=100mm and height H=70mm (for hydraulic conductivity cells modified with bender elements), D=70mm and H=70mm (for standard hydraulic conductivity tests), D=50mm and H=100mm (for unconfined compression tests).

Table 1. Physical properties of the treated and untreated bentonite clays.

	clay	HYPER clay
Liquid Limit [%]	527.15	592.10
Plastic Limit [%]	90.10	171.90
Plasticity Index [%]	437.05	420.20
Swell Index [%]	22 (M)	28
CEC [meq/100g]	50.21	64.96
(M) = Macropores		

Table 2. Chemical composition of the cement and of the untreated bentonite clay.

	cement	clay
SiO_2 [%]	29.6	52.8
Al_2O_3 [%]	8.4	3.44
Fe_2O_3 [%]	1.2	1.16
MgO [%]	5.6	24.25
CaO [%]	47.8	1.95
Na_2O [%]	0.16	3.75
Other	7.24	16.4

3. Methods and procedures

The hydraulic conductivity and small-strain shear modulus of CB samples were studied in flexible-wall hydraulic conductivity cells provided with bender elements (Permeabender cells). These cells were provided with a cathetometer to monitor sample height changes (Fig. 1). In addition to that, replicate hydraulic conductivity tests were also performed in standard flexible wall permeameters. Furthermore, the unconfined compressive strength (UCS) of CB samples cured in pure water was monitored through unconfined compressive tests.

Samples with a diameter of 100 mm and a height of 70 mm were used for hydraulic conductivity tests in the permeabender cells; samples with a diameter of 70 mm and a height of 70 mm were used for replicate standard hydraulic conductivity tests and for UCS tests after permeation; samples of 50 mm diameter and 100 mm height were used for standard UCS tests cured in deionized water.

The hydraulic conductivity k was evaluated out of a falling-head test performed in a conditioned room at constant temperature (18°C) and at an isotropic effective stress of 30 kPa. The sample was first permeated with deionized water for about 1 month (1.93 and 0.26 pore volumes of flow for the untreated and treated clay respectively). Next, the deionized water was replaced with the 25 g/L solution of Na_2SO_4 and the test was continued for a period of about 4 months (about 2.70 and 0.30 pore volumes of flow, respectively for the untreated and treated clay respectively).

Figure 1. Flexible-wall hydraulic conductivity cell provided with bender elements and a cathetometer.

The small-strain shear modulus of the CB samples (starting at 7 days of age) was evaluated by bender element testing. The bender elements used here are of the type T220-A4–203Y (Piezo Systems, Inc.). The effective bender element length penetrating in the sample was about 4.5 mm.

The bender element transmitter and receiver, fixed to top plate and base pedestal of the hydraulic conductivity cell, are vertically aligned. Bender element measurements were performed on a regular basis from the first day of permeation up to 5 months (the tests are still ongoing to analyze the long-term behavior).

In bender element testing, G_0 is determined out of the propagation velocity (V_s) of shear waves generated and detected by the transmitter and receiver bender elements installed on opposite sides of a sample. G_0 is estimated as:

$$G_0 = \rho\ Vs^2 \tag{1}$$

where ρ is the density of the sample. V_s is evaluated as follows:

$$V_s = L / t_s \tag{2}$$

where L is the tip-to-tip distance between the transmitter and receiver bender elements, and t_s is the travel time of the shear waves from the transmitter to the receiver; t_s is evaluated out of the signal recordings. In this research, t_s was evaluated by means of two methods. The first one consists of visually identifying the first direct arrival from the output signal [e.g. 7, 15, 16]. The second method used here was the cross-correlation method, first introduced by [7]. The cross-correlation analysis measures the level of correspondence or interrelationship between two signals of similar nature and it produces the time shift between them. An example of the recorded signals is given in figure 2. Both interpretation methods produced similar outcome.

Lastly, the unconfined compressive strength (UCS) of some specimens immersed in deionized water for 7, 28, 60 and 90 days was determined.

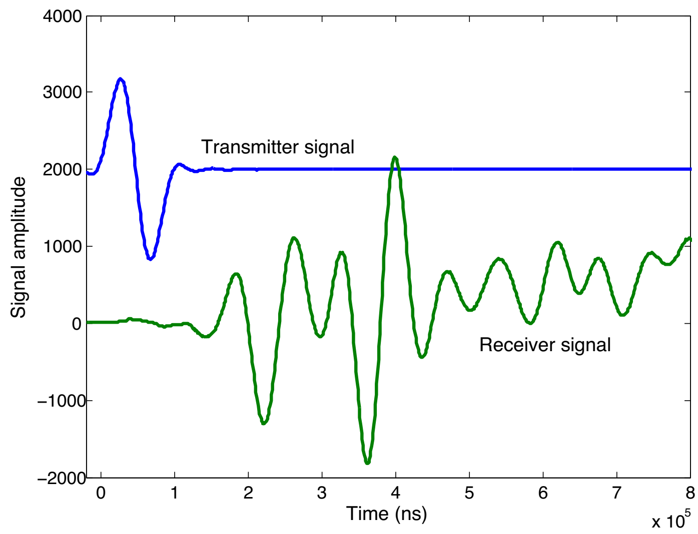

Figure 2. Example of bender element signals recorded in the hydraulic conductivity cell

4. Results and discussions

4.1. Hydraulic conductivity

Figure 3 shows the hydraulic conductivity of the cement-bentonite mixtures. As expected, the hydraulic conductivity to water of both type of mixtures decreased with time according to the correlation reported by [17]:

$$k = k_r(t/t_r)^{-\alpha} \tag{3}$$

where k_r is the hydraulic conductivity at an age of 28 days (t_r), t is the age of the sample in days and α is a constant.

After about 30 days of curing, the permeant solution was substituted with a solution containing sulfates (25 g/L Na_2SO_4). As shown in Fig. 3, the hydraulic conductivity of the cement-untreated clay mix increased after permeation with sulfates. Conversely, the hydraulic conductivity of the cement-bentonite mixtures containing HYPER clay continued decreasing with time.

A decrease of the hydraulic conductivity of the cement-untreated clay mixture was noted immediately after contact with sulfates. This behavior was likely due to clogging of the pores due to gypsum and ettringite formation. When the formation of these products becomes significant fissures could appear leading to an increase of the hydraulic conductivity.

4.2. Small-strain shear modulus

Figure 4 shows the small-strain shear modulus of the cement-bentonite mixtures. As expected, a gradual increase of G_0 was observed during the permeation with deionized water as a consequence of the cement hydration process. The two mixtures showed similar values of G_0.

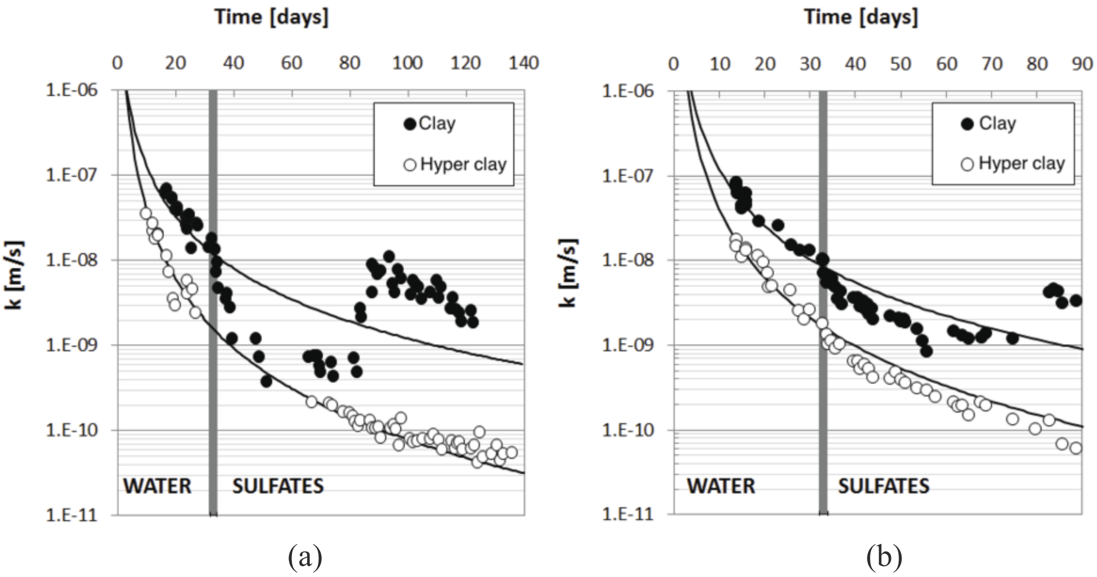

Figure 3. Impact of sulfate attack on the hydraulic conductivity of CB mixtures containing untreated clay or polymer-treated bentonite (HYPER clay) permeated with a 25 g/L Na_2SO_4 solution. (a) Hydraulic conductivity vs. time in the permeabender cell. (b) Replicate test results of hydraulic conductivity vs. time in a flexible wall cell.

After contact with sulfates for a period of about 100 days, no significant changes were registered even if a decrease of the small-strain shear modulus was expected as a result of the cement degradation caused by the expansion of gypsum and ettringite, as observed in a previous study [18]. These tests are still ongoing to monitor the long-term behavior.

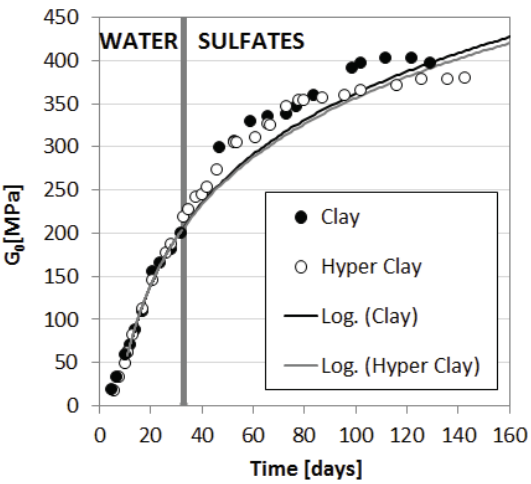

Figure 4. Impact of sulfate attack on the small-strain shear modulus of cement-bentonite mixtures containing untreated clay or polymer-treated bentonite (HYPER clay) .

4.3. Unconfined compression tests

Figure 5 summarizes all UCS measurements carried out on samples immersed in deionized water.

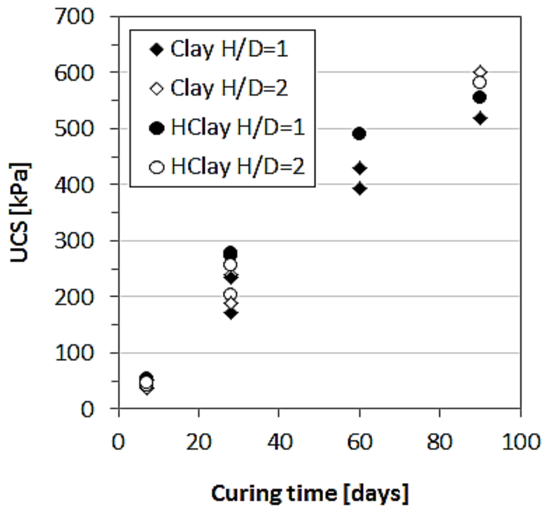

Figure 5. Compressive strength versus time of the CB samples (immersed in deionized water) with untreated clay (Clay) and polymer-treated bentonite (HClay) with different height/diameter (H/D) ratios.

Both types of mixtures showed a similar increase of unconfined compressive strength with time. These results suggest that the polymer treatment on the clay did not have negative effects on the strength of CB mixtures yet maintaining a lower hydraulic conductivity.

5. Conclusions

The hydraulic and mechanical properties of cement bentonite samples are usually studied separately. In this research the hydraulic conductivity and the small-strain shear modulus of samples prepared with untreated and polymer-treated clays were simultaneously measured using a flexible wall permeability cell provided with bender elements.

Test results showed that the cement-HYPER clay mixtures were less permeable and less sensible to the microstructural changes caused by the interaction between cement hydration products and sulfates.

Samples prepared with HYPER clay technology achieved a strength and stiffness comparable to CB samples containing untreated clay, still maintaining a lower permeability compared to the cement-untreated bentonite mixtures.

The results and all the considerations about the hydraulic conductivity, small-strain shear modulus and unconfined compressive strength are referred to a short study period of about 150 days. Longer monitoring periods are necessary to evaluate the long-term behavior of CB samples.

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