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Monitoring small-strain stiffness in cementitious porous materials and amended soils for geo-environmental applications

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ABSTRACT

The paper illustrates the use of Bender Element (BE) technique to monitor the development of small-strain shear modulus, G_0, in cement-bentonite (CB) mixtures and compacted soil-lime (SL) mixtures, both commonly employed in various geo-environmental applications. Three CB mixtures of different composition and one SL mixture were investigated. The G_0 of the CB mixtures was found to increase with curing time, while the hydraulic conductivity significantly decreased. The cement-to-water ratio of CBs appeared to be the controlling factor on G_0 whereas the hydraulic conductivity is also significantly influenced by other variables in composition (e.g. use of additives). The small-strain shear modulus of the soil-lime was found to increase with curing time with a trend that can be related to that of soil-lime chemical reactions. The results suggested that G_0 can represent a useful parameter to monitor the progress of chemical reactions in soil-lime mixtures.

Keywords: small-strain shear modulus, cement-bentonite, soil-lime, permeability

1 INTRODUCTION

Cementitious porous materials and binder-amended soils are often employed in geo-environmental applications, such as cut-off walls (water-cement-bentonite or “CB” mixtures, Jefferis, 1982), landfill sidewall liners (e.g. cement-soil mixtures, Bellezza & Fratalocchi, 2006) and reuse/stabilization of excavated soils (e.g. soil-lime mixtures, TRB, 1987; Beetham et al., 2014; Di Sante et al., 2020). One common aspect of these materials is the change of some of their properties (e.g., stiffness, shear strength, hydraulic conductivity) with time as a result of the pozzolanic reactions, even in the absence of interaction with chemicals. In laboratory testing and/or field application of these materials, it may be necessary to monitor the above mentioned properties to assess the progress of hydration reactions in absence or presence of chemicals, to make decisions about the test conduction or assess the achievement of the required performance at a given time.

A mechanical parameter which can reflect the properties of the porous medium is the small-strain shear modulus, G_0, linked to the material stiffness that is expected to vary with the progress of pozzolanic reactions, thus with the age of the material (curing time). Moreover, micro and macrostructure alterations of the porous medium caused by chemical reactions with solutes (e.g., formation of expansive phases and cracking of cementitious materials in contact with sulphates) should be reflected by a change in G_0. In laboratory testing, G_0 can be non-destructively measured by the Bender Elements (BE in the following) technique (Divick & Madshus, 1985), which allows testing the same sample for the entire curing period.

BE testing has been recently used to monitor the stiffness increase due to binder addition in lime-treated soils and cement-treated soils. Puppala et al. (2005) studied the small strain shear moduli of lime-cement treated expansive clays for deep mixing applications; Puppala et al. (2006) monitored the stiffness variation of cement and lime-treated sulphate bearing soils by BE tests and observed a significant increase in G_0 with curing time due to binder addition. Wang et al. (2017) finalized a novel method for determining G_0 based on the comparison of S-wave and P-wave received BE signals on a 2% quicklime-treated plastic silt at different curing times and saturation degrees. Verastegui Flores et

al. (2010) used BE testing and conventional unconfined compression tests to monitor the hardening of cement-treated clay with time. The results showed that G_0 and strength of cement-treated samples increases logarithmically with time. For different cement types, G_0 and the compressive strength, when normalized with reference to the relevant values at a specific curing time (28 d), were found to increase with a common trend.

Applications of the BE technique to cement-bentonite (CB) mixtures are very limited. Verastegui Flores et al. (2013) investigated by BE the small-strain shear modulus and the hydraulic conductivity (k) of a cement-bentonite sample in contact with water or sodium sulphate. During permeation with water G_0 increased and *k* decreased gradually with time as a result of cement hydration. However, after prolonged permeation with sulphates, a decrease of G_0 and a gradual increase of *k* were recorded, suggesting degradation of the cemented structure with consequent loss in strength and development of a network of interconnected fissures affecting the hydraulic conductivity. Li et al. (2019) used BE to study the influence of curing, type of cementitious material and slag substitution rate on the shear wave velocity V_s of slag-cement-bentonite mixtures (S-CB). The results showed that V_s (and G_0) of S-CB increased with age and content of cementitious materials whereas it decreased with the increase of slag substitution. It was also observed that the temporal trend of the normalized modulus ($G_0/G_{0,28\ d}$), where $G_{0,\ 28d}$ denotes the value at 28 days of age, depends on the slag substitution rate rather than the total cementitious material content.

This paper presents selected results of an ongoing research activity concerning the engineering properties of geomaterials used for environmental applications. In particular, the paper focuses on the use of BE technique to monitor the evolution of small-strain shear modulus of CB mixtures and soil-lime mixtures. The first objective was to outline an optimal testing procedure, taking into account the continuous change of material properties with time. Three CB mixtures of different composition, representing poor to high quality mixtures, and one soil-lime mixture with a typical lime dosage (3% quicklime) were investigated. Secondly, BE testing was coupled with permeability testing on the same specimen or on separate specimens to compare the evolution of hydraulic and stiffness properties and support the interpretation of test results.

2 MATERIALS AND METHODS

2.1 CB Mixtures

Three different mixtures of cement, bentonite and water (CB) were investigated. The cement-to-water ratio and bentonite type were changed to assess the influence of the composition on the small-strain shear modulus (G_0) of the mixture. The main characteristics of the three mixtures are listed in Table 1. Two different bentonites (a calcium and a sodium bentonite), a simulated III/B type cement consisting of 30% Ordinary Portland Cement and 70% blast furnace slag and tap water were used. A polymeric liquid additive, commonly utilized to increase the workability of fresh mixtures, was added to CB2 and CB3. In short, CB1 represents a low quality mixture (calcium bentonite, no additive), whereas CB2 and CB 3 represent higher quality mixtures (sodium bentonite, use of additive). The mixtures were prepared by adding the bentonite to water (CB1) or to water and additive (CB2 and CB3) while mixing with a high-speed shear mixer for 15 minutes at a speed of 1000 rpm and allowing the bentonite to hydrate for about 24 hours. Then, the cement was added to the bentonite slurry and mixed for 5 minutes at a speed of 1500 rpm. The amount of mixture prepared for each batch allowed measuring Marsh viscosity, unit weight (γ) and bleeding. The fresh mixtures were poured in cylindrical split plastic moulds of the same size of the test specimens (diameter=10.0 cm, length= 6.0 -7.0 cm). The moulds containing the testing specimens were stored under water to allow curing and prevent desiccation. At curing times of no less than 7 days, the hardened specimens were extracted from the moulds for subsequent testing.

Table 1. *Main physical properties of CB mixtures*

Mixture	Bentonite		Cement	Additive			
	Type	Amount	c/w		Marsh Test	γ	Bleeding
[-]	[-]	[g/l]	[-]	[%]	[s]	[kN/m^3]	[%]
CB1	Ca	65	0.27	-	35	11.6	6.5
CB2	Na	60	0.27	0.2	46	11.8	<1
CB3	Na	60	0.22	0.2	53	11.8	<1

2.2 Soil-lime mixture

An inorganic clay of high plasticity (w_L=57%, PI=33% classifying as CH in USCS – ASTM D2487-17) was used for the soil-lime mixture. The lime is a fine calcic quicklime, classified as CL80-Q (UNI EN 459-1:2010), completely passing through the ASTM 200 sieve (75 μm sieve opening). The amount added to the soil was 3% by dry mass of soil. The soil-lime mixture was prepared by crumbling the air-dried soil, adding moisture (tap water) to a target water content of 26%. Lime was added to the moist soil and a uniform distribution was ensured. The soil-lime mixture was then compacted by the Standard Proctor procedure (ASTM D698-21) in three layers. The specimen used for measurement of G_0 had a void ratio e= 0.74 and an initial degree of saturation, Sr = 93%. The compacted specimens were cured in a closed plastic container, stored in a room maintained at controlled temperature (T=22 ± 2°C) and extracted temporarily from the container only to perform BE testing. In order to minimize the loss of water in the curing stage and during the execution of the test, the specimens were completely coated, except for the BE slots, with paraffin immediately after compaction. The weight of the specimens was monitored throughout the curing period and the weight loss due to evaporation was less than 1%.

Using the aforementioned procedure (except for paraffin coating), a different specimen was prepared for permeability testing. After 7 days of curing in a closed plastic bag, the specimen was transferred to a flexible wall permeameter, consolidated under an isotropic effective stress of 35 kPa and permeated with tap water. The low stress level was selected with the purpose of maintaining confinement conditions close to those of BE testing, that was performed without confinement, while ensuring the contact between the specimen and the latex membrane. Tap water was used as permeant liquid instead of deionized or distilled water in order to simulate common operational conditions of construction sites. Moreover, it should be considered that in lime-treated soils the porewater composition is dominated by lime dissociation and induced exchange of adsorbed cations for calcium (Ca^{2+}). Therefore, no significant difference is expected from using distilled or tap water as permeant liquid. At the end of hydraulic conductivity test, Scanning Electron Microscope (SEM) observations were performed by means of VEGA3 – TESCAN, Czech Rep. and FESEMSUPRA40–ZEISS, Germany after air dewatering of samples and their gilding (by an Emitech K550 sputter coater).

2.3 BE apparatus and testing

The BE testing was conducted following the ASTM Standard D8295-19. The input signal, a sine pulse of 10 Vpp amplitude, was generated by a function generator (Aim TTI TG5011A), triggered with a period set to allow enough time for the attenuation of the BE response before the next pulse. Both the transmitted and the received signals were acquired by a digital oscilloscope (Picoscope 6, Pico Technology LTD, UK) and then processed. The shear wave travel times were computed by the time domain method (Peak-to-peak). Signals of several frequencies were applied, ranging from 0.5 kHz to 50 kHz.

As regards CB mixtures, the slots to insert the BE were made by a thin plastic plate of the same size as the transducer, that was pushed into the fresh and relatively soft mixture and extracted prior to BE testing. Attention was paid to ensure the alignment of the slots on the opposite faces of the specimens. After 7 days of curing in water, the specimen was transferred to a triaxial cell in order to carry out tests in confined conditions. The cell pedestal and top cap of the triaxial cell are equipped with BE (Controls, Italy). An effective confining stress of 35 kPa was applied to the specimen and BE testing was alternated with permeation stages with water to monitor the hydraulic conductivity, too.

The test on the lime-treated soil was carried out in unconfined conditions, a commonly adopted approach for lime-treated soils (e.g. Puppala et al., 2006; Tang et al., 2011; Wang et al., 2017; Wang et al., 2020). An undersized slot was excavated immediately after compaction (as suggested for specimens with increasing stiffness) on the two opposite faces of the specimen to easily insert the BE, paying attention to ensure their alignment; to execute the test, the specimen was placed horizontally on a specially designed wooden sample holder. The first measurement was performed two hours after mixing and compaction of the soil-lime sample and the measurements were repeated at increasing curing time until 81 days.

3 RESULTS AND DISCUSSION

3.1 BE testing: signal interpretation

Fig. 1 shows an example of result of BE testing on the CB mixture. The travel time (t_{p-p}) was determined as the time difference between the peak of the transmitted and the first peak of the received signal (Fig 1a). The G_0 of the specimen at the given curing time was calculated as follows:

$$G_0 = \rho\left(\frac{L_{tt}}{t_{p-p}}\right)^2 \quad (1)$$

where ρ= specimen density and L_{tt} =tip-to-tip distance between the BE elements. Several frequencies were generally applied in order to identify the most readable signal. In the case of CB2, both the transmitted and received signals became weaker with curing time, making the identification of the travel time more difficult (Fig. 1b). The exact reason for this occurrence, probably related to a malfunctioning of the electronics was not identified. In any case, the readable signals were acquired.

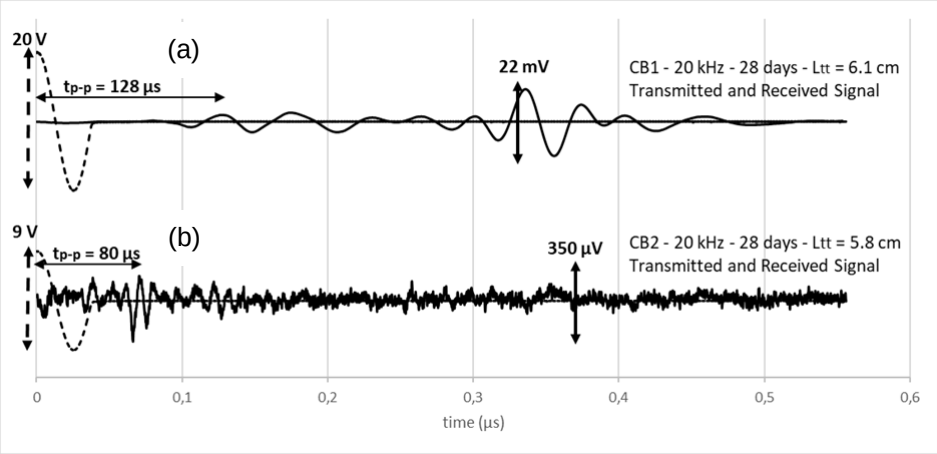


Figure 1. *Examples or transmitted and received signals during BE testing of CB mixtures.*

3.2 CB Mixtures

3.2.1 G_0 vs. curing time

In Fig. 2 the G_0 modulus of the CBs vs curing time in the interval 14-60 days is shown. Only a few data points are plotted for CB2 due to difficulties in obtaining readable signals. The G_0 of all CB mixtures increases with curing time, well beyond the typical curing time of 28 days often adopted in compliance assessment of cementitious materials. The increase of G_0 with curing time is consistent with the increase in stiffness of the solid skeleton due to cement hydration and formation of hydration products (e.g., C-S-H). The trend of G_0 with curing time in the investigated time interval can be well fitted by a logarithmic law (Verastegui Flores et al., 2010) for CB1 and CB3 (Fig. 2). The available data for CB2 are more scattered but relatively close to those of CB1. CB1 and CB2 differ essentially in the bentonite type and the use of additive but have the same content of cementitious components (cement plus slag), which appears to be the controlling factor in determining the small strain stiffness of CB mixtures under the testing conditions adopted. CB3 has a lower cement content (c/w=0.22) than CB1 and CB2 (c/w=0.27) and significantly lower G_0.

The G_0 values obtained in this study are significantly different from those reported in literature on CB mixtures of similar composition. For example, Verastegui Flores & Di Emidio (2013) obtained $G_{0,28\ d}$ = 150 MPa on a CB mixture with 4% sodium bentonite, 16% III/B cement (slag 66-80%), 80% water by weight versus $G_{0,28\ d}$=38 MPa obtained in this study on CB3 which has 5% sodium bentonite, 17% cementitious materials (70% slag) and 78% water by weight. Li et al. (2019) obtained $G_{0,28\ d}$ = 0.25 MPa on a CB mixture with 15% cementitious material (30% OPC and 70% slag). The large scatter in G_0 values from different studies for relatively similar composition of the mixture at the same curing time suggests that the measured G_0 of CB mixtures is probably very sensitive to the testing conditions and to the quality of components.

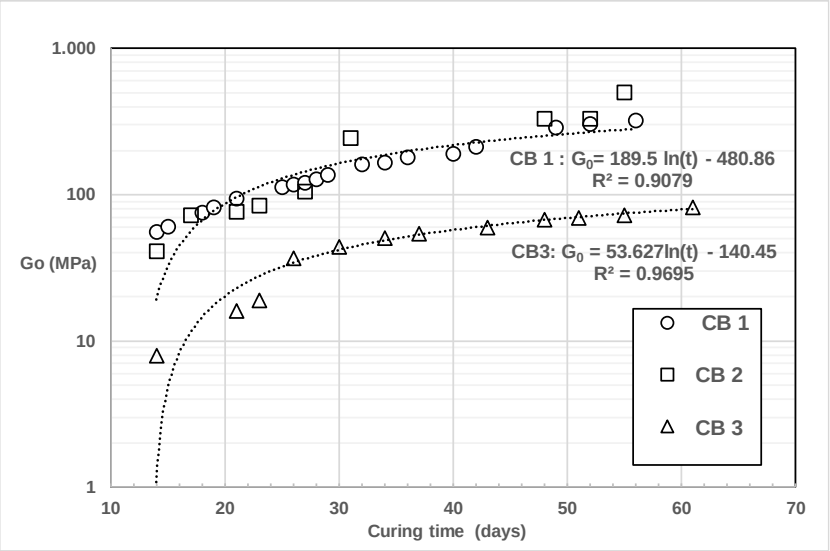


Figure 2. *G_0 versus curing time for the CB mixtures investigated. Dashed lines are the best-fit curves of experimental data for CB 1 and CB 2*

3.2.2 Hydraulic conductivity vs curing time

Fig. 3 shows the (saturated) hydraulic conductivity of the tested CB mixtures as a function of the curing time (counted in days from the preparation) in the curing interval 14-60 days. The k of CB mixtures is known to decrease with time as a result of cement hydration (Fratalocchi &Pasqualini, 1998).The trend of k in water-permeated CB mixtures with curing time, t, can be well modelled by a power law (Fratalocchi & Pasqualini, 1998; ICE, 1999):

$$k(t) = k_{ref}\left(\frac{t}{t_{ref}}\right)^{-\alpha} \tag{2}$$

where k_{ref} = hydraulic conductivity at a reference time t_{ref}, α = the permeability reduction coefficient. The fitting parameters in Eq. 2 depend on curing time at short curing but tend to stabilize within 40-60 days. In Fig. 3 the experimental data of k with time are fitted with Eq. 2 by assuming t_{ref} = 14 days and the resulting expressions for each curve are also shown.

The k of CB1 and CB2 differed by about two orders of magnitude throughout the investigated time interval: the difference is to be ascribed mainly to the presence of the additive and partly to the bentonite type, since the total cement content and slag substitution rate (70%) are the same. Noting that the total porosities of the mixtures are very close, the lower k of CB1 is to be ascribed to a different size distribution and tortuosity of the conductive pores, presumably induced at the initial slurry state by the combined effect of the additive and sodium bentonite, that keep the cement particles more dispersed than in CB2. On the other hand, the α coefficients of CB 1 and CB2 are very close, being mainly related to the cement-to-water ratio in the mixture (e.g. Fratalocchi et al., 2013), that is equal for the two mixtures (c/w=0.27). The difference in k between the two mixtures can be expected to persist even at longer curing times, as predicted by the expressions for k(t) in Fig. 3. CB2 and CB3

have similar k_{ref} at t=14 days but different α values, which can be explained by the different cement-to-water ratio.

Comparing the results in Fig. 2 and Fig. 3 it can be observed that CB1 and CB2 have similar G_0 but differ in k by two orders of magnitude. Therefore, is not possible to establish a unique correlation between G_0, k and curing time for CB mixtures. However, monitoring the G_0 of a given CB mixture, besides permeability, may provide a more comprehensive characterization of the material behaviour, necessary for design purposes. A significant deviation from the expected trend in G_0 (e.g., a sudden decrease or lack of increase at short curing times) can be utilized, coupled with k, as an indicator or a warning for anomalies in the mixture properties or in laboratory procedures. Conversely, the results of this study showed that, for a given curing time, the G_0 is higher for the CB mixture with the higher cement-to-water ratio. The G_0 could then be used as an indicator of the cement content for CB mixtures of the same age (type of cement being equal).

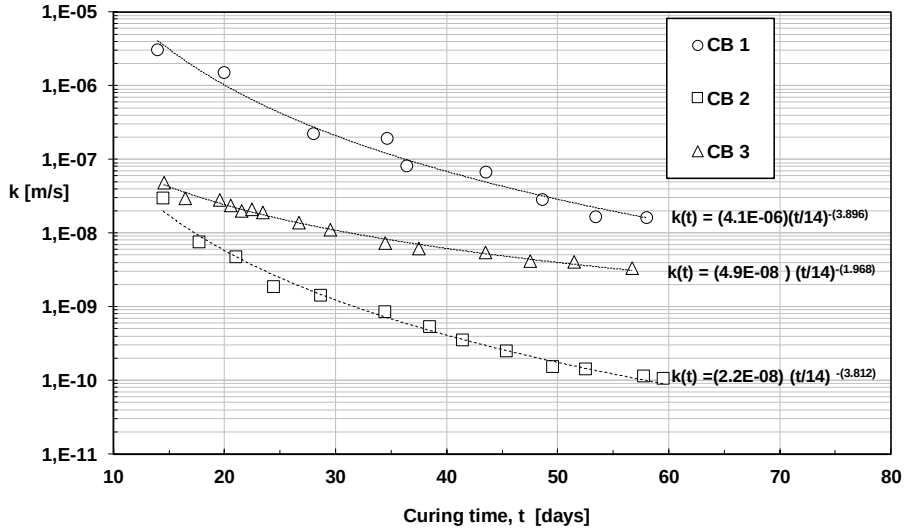


Figure 3. *Hydraulic conductivity vs. curing time of the three CB mixtures investigated. Dashed lines are the best fit curve of experimental data according to Eq.2*

3.3 Soil-lime mixtures

3.3.1 *G_0 versus curing time*

The trend of G_0 for the lime-treated soil versus curing time is shown in Fig. 4. As expected, there is an increasing trend with curing, that can be relatively well described by a logarithmic law. The trend of G_0 at early stages of curing (0-10 days) is enlarged in Fig. 4. The initial value of G_0 (21 MPa) for the soil-lime refers to the compacted untreated soil. The G_0 showed a sharp increase at t=2 days and reached a value of 679 MPa at 10 days of curing; thereafter, a slower increase up to the value of 1127 MPa at 81 days was observed. This trend can be interpreted as initially due to cation exchange, caused by Ca^{2+} released by calcium hydroxide dissociation, and to immediate flocculation and rearrangement of soil particles, which is responsible for the slight increase in stiffness with respect to the untreated soil on the short term (0-2 days).

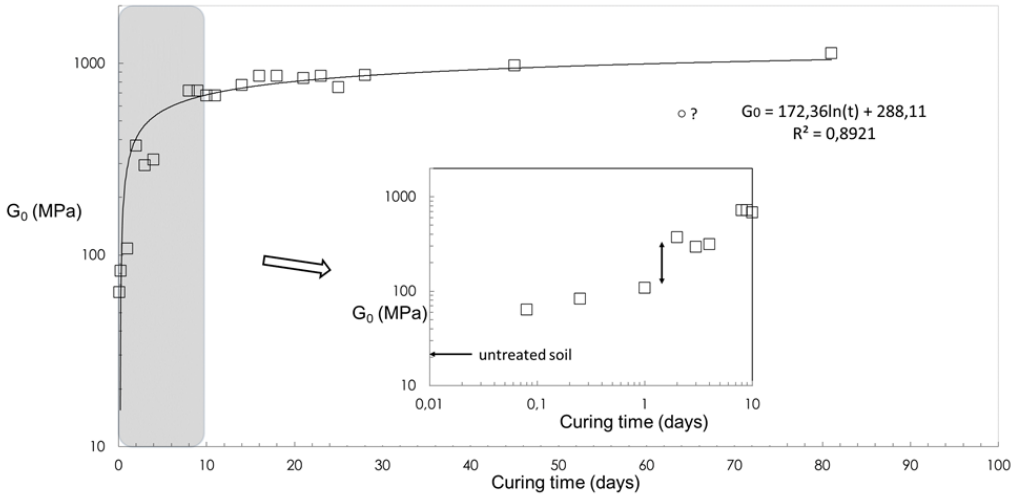


Figure 4. *G_0 vs. curing time for the soil-lime mixture investigated.*

The sharp increase in G_0 at 2 days of curing (see enlarged plot) marks the prevalence of pozzolanic reactions over cation exchange. The increase in G_0 from 2 days until 81 days of curing can be reasonably ascribed to the cementation occurring in the sample due to the development of pozzolanic reactions. The results obtained are in agreement with other studies (e.g., Wang et al. 2020; Liu et al. 2021) on stiffness development in lime-treated soils, in which a two-phase change pattern of the G_0 modulus was also identified. Further, the relationship between the trend of G_0 and short-term and long-term soil-lime reactions has been recently supported by parallel monitoring of porewater chemistry (Di Sante el al. 2022).

3.3.2 Hydraulic conductivity versus curing time

Fig. 5 shows the (saturated) k values of the lime-treated compacted soil in the curing interval 7-55 days. The k of the untreated compacted soil (determined on a separate specimen under the same confining stress) is also shown. It is possible to observe that lime addition induced a substantial increase in hydraulic conductivity (k = 4×10^{-7} m/s) with respect to the untreated compacted soil (k=6–8×10^{-11} m/s). This increase in k is consistent with the release of Ca^{2+} in porewater, subsequent cation exchange on the clay surface and flocculation of clay particles into larger aggregates, with the formation of conductive inter-aggregate pores (e.g., TRB, 1987). During the 7- 55 days curing time interval, a modest reduction of hydraulic conductivity (to k=1×10^{-7} m/s) was observed. Since only long-term pozzolanic reactions are expected to be still active, the modest reduction in k can be ascribed to the formation of amorphous pozzolanic products that partially close the inter-aggregate porosities (Wild et al.,1987). In short, the trend of k within the investigated interval of curing time basically supports the long-term stage of development of soil-lime interactions inferred by the trend of G_0. In Fig. 6 SEM images of the untreated soil (Fig.6a) and of the soil-3%lime mixture (Fig.6b) at the end of permeability test are displayed. Lime addition induces aggregation, thus an open soil fabric of the treated sample, if compared with the untreated one. In Fig. 6b it is evident the presence of both crystalline (needle-like), and gel-form (spongy appearance) pozzolanic products distributed across the sample, confirming the long term reactions development. The bonding and cementing action of these products is able to increase the stiffness of the soil-lime mixture, resulting in the increasing trend of G_0.

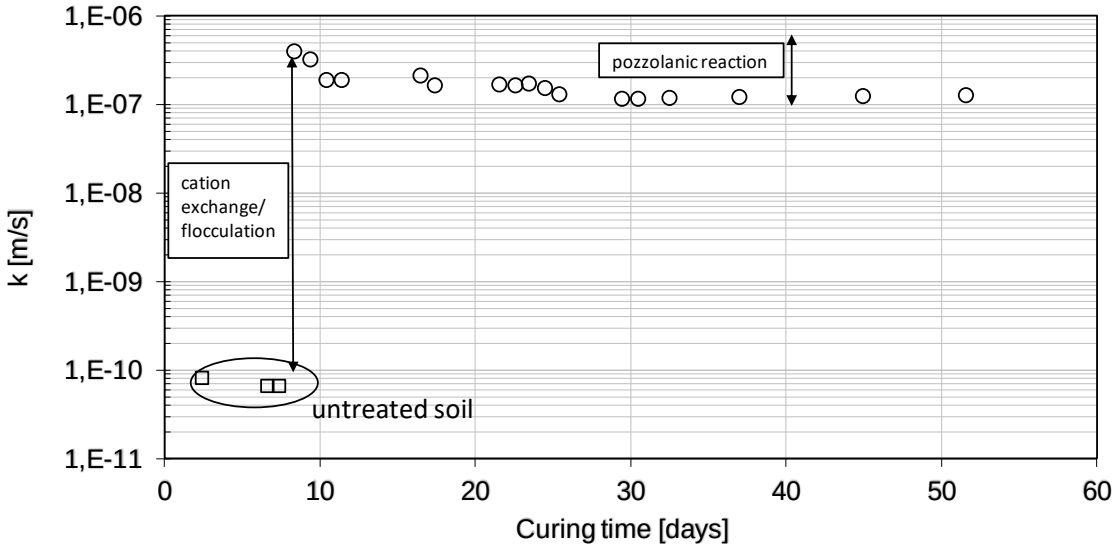


Figure 5. *Hydraulic conductivity versus curing time for the soil-lime mixtures investigated. The hydraulic conductivity of the compacted untreated soil is reported for comparison.*

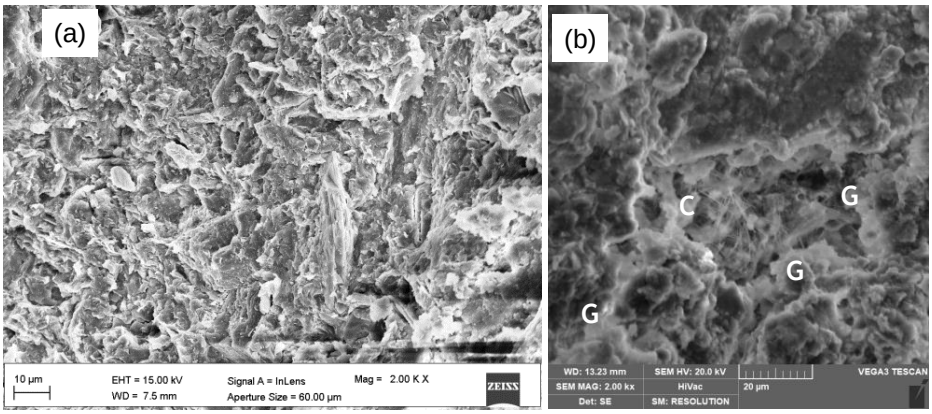


Figure 6. *SEM images of (a) untreated soil (b) soil+3% quicklime at the end of permeability test (C=crystalline pozzolanic products; G=Gel-form pozzolanic products).*

4 CONCLUSIONS

The paper illustrates the use of Bender Element (BE) technique to monitor the development of small-strain shear modulus, G_0, in cement-bentonite (CB) mixtures and compacted soil-lime (SL) mixtures, both commonly employed in various geo-environmental applications. Three CB mixtures of different composition and one SL mixture were investigated.

The G_0 of the CB mixtures was found to increase with curing time, while the hydraulic conductivity significantly decreased. The bentonite quality and the use of additive were the controlling factors on the hydraulic conductivity of CB mixtures of a given cement content. The cement-to-water ratio of the CB mixture appeared to be the controlling factor on G_0 for CB mixtures of different dosages, prepared using the same components. The quality or origin of the CB components also plays a significant role since a large scatter in G_0 values for CB mixtures of the same age and relatively similar composition emerged from the results of the present study and literature data.

The small-strain shear modulus of the SL mixture was found to increase with curing time with a trend that can be related to that of soil-lime chemical reactions and consequent formation of hydration

products, as supported by SEM imaging of the specimens at the end of the tests. The results suggested that G_0 can represent a useful parameter to monitor the progress of chemical reactions in soil-lime mixtures.

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