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LINKING SMALL-STRAIN STIFFNESS TO DEVELOPMENT OF CHEMICAL REACTIONS IN LIME-TREATED SOILS

--Manuscript Draft--

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Abstract:	<p>In the context of lime stabilization, this note shows how the development of soil-lime reactions can be linked to the variation of small shear stiffness with time. To determine the evolution of small-strain stiffness, the shear wave velocity was measured by means of bender elements (BE) on a compacted clayey soil treated with 3% quicklime, starting from 2 hours after compaction until 98 days of curing. Different methods of signal interpretation were applied with the purpose of highlighting how the peculiarity of lime treated soils affects BE testing results and to provide practical indications for optimizing similar testing on lime-treated soils. The results showed that lime treatment and compaction affect the waveform of the received signal and that measurements should span across a wide range of input frequencies in order to identify an optimal waveform. The small strain shear modulus was found to increase with curing time with a trend that can be related to that of soil-lime chemical reactions, thus representing a promising parameter to monitor the development of soil-lime reactions.</p>
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Francesco Mazzieri: Conceptualization, Writing - Review & Editing, Funding acquisition, Project administration, Supervision

Declaration of interests

☒The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

☐The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

To: prof. Antonio Gomes Correia
Chair Editor
Transportation geotechnics

Ancona, Italy - October 15th 2021

Dear prof. Antonio Gomes Correia,

We submit a manuscript with the title “Linking small-strain stiffness to development of chemical reactions in lime-treated soils” for the possible publication as a Technical Note in Transportation Geotechnics. This note is focused in the field of transport infrastructure geotechnics with particular reference to the lime stabilization technique of soils for earthworks and embankments construction. It shows how the development of soil-lime reactions can be linked to the variation of small strain stiffness with time, identifying G_0 as a key parameter to monitor the reaction development. Moreover, the note highlights how the peculiarity of lime treated soils can affect the Bender Elements testing and provides practical indications to optimize similar testing on lime-treated soils.

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All the authors have seen the manuscript and agree to its submission. The authors declare herewith that the accompanying manuscript is original work, not published elsewhere or under consideration for publication elsewhere.

Yours Faithfully,

Dr. Marta Di Sante
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LINKING SMALL-STRAIN STIFFNESS TO DEVELOPMENT OF CHEMICAL REACTIONS IN LIME-TREATED SOILS

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Abstract

In the context of lime stabilization, this note shows how the development of soil-lime reactions can be linked to the variation of small shear stiffness with time. To determine the evolution of small-strain stiffness, the shear wave velocity was measured by means of **bender elements** (BE) on a compacted clayey soil treated with 3% quicklime, starting from 2 hours after compaction until 98 days of curing. Different methods of signal interpretation were applied with the purpose of highlighting how the peculiarity of lime treated soils affects BE testing results and to provide practical indications for optimizing similar testing on lime-treated soils. The results showed that lime treatment and compaction affect the waveform of the received signal and that measurements should span across a wide range of input frequencies in order to identify an optimal waveform. The small strain shear modulus was found to increase with curing time with a trend that can be related to that of soil-lime chemical reactions, thus representing a promising parameter to monitor the development of soil-lime reactions.

Keywords

Soil stabilization, lime, small-strain stiffness, shear wave velocity, **bender elements**

Introduction

Lime stabilization of clayey soils is a well-known and sustainable ground improvement technique for the construction of earthen structures (e.g. Boardman et al., 2001; Beetham et al., 2014; Gomes Correia et al., 2016; Rosone et al., 2018). Despite its wide application in engineering works, the optimization of the mix design procedure and the search for methods and correlations to predict soil-lime performances are the objective of recent research studies (e.g. Consoli et al., 2014; Robin et al., 2015; Di Sante et al., 2015; Consoli et al., 2017; Di Sante et al., 2020; Fratolocchi et al., 2020). **Moreover, delayed compaction (due to hitches or technical breaks during construction e.g. Osinubi et al., 2006; Di Sante et al., 2015), wetting-drying and freezing-thawing cycles (e.g. Stoltz et al., 2012; Shirmohammadi et al., 2021) and a lack of an extensive experimental laboratory investigation can potentially adversely affect the stabilization outcomes, therefore these occurrences should be taken into account during the design phase.** In this perspective, understanding the sequence, mode and timing of reactions between lime and clayey soils plays an essential role. Table 1 summarizes the reactions that develop in a soil-quicklime-water system and the related effects.

Table 1

Several studies have been carried out to identify the chemo-mineralogical evolution of lime treated soils with time (e.g. Vitale et al., 2017; Guidobaldi et al., 2018). Among the different methodologies proposed to identify

the reactions timing and evolution, those based on pH measurements of the soil-lime-water system are easy to be carried out and turned out to be very effective and reliable (Rao and Shivananda, 2005; Al Muckthar , 2010; Di Sante et al., 2014, Di Sante, 2020).

In the present note a preliminary study to monitor the development of chemical reactions based on stiffness variation of a clayey soil treated with quicklime is presented. The results are compared with those obtained by the pH method (Di Sante et al., 2014). A parameter which can reflect the structure of the porous medium is the small-strain shear modulus, G_0 , that is linked to the stiffness of the material that, in turn, is expected to change with the progress of chemical reactions, thus with curing time. Although G_0 can increase in time even for untreated soils due to ageing phenomena (Schmertmann, 1991; Jovičić et al., 1997; Rammah et al., 2004), laboratory data showed that the maximum rate of increase of G_0 with the logarithm of time (in days) can be equal to 19% in case of secondary compression.

G_0 can be measured by **bender elements** (BE) test in a non-destructive way, which allows testing the same soil-lime sample for the entire curing period. The small-strain shear modulus is related to the shear waves velocity that can be calculated from the travel time measured generating a shear wave at one boundary of the specimen (transmitter BE) and receiving the wave at the opposite boundary (receiver BE).

Piezoelectric transducers applications for soils were originally introduced by Lawrence in '60s (Lawrence, 1963) and, from then on, the experimental setting has been improved as well as the methods of signal interpretation (e.g. Arulnathan et al., 1998; Wang et al., 2017). Some attempts to apply the BE technique to study the stiffness of lime treated soils with several different aims have been recently made. In particular, 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 (with different sulphate levels and curing conditions) by BE tests, finding 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 using a 2% quicklime treated compacted plastic silt at different curing times and saturation degrees. The effects of wetting-drying cycles and of the aggregate sizes on the stiffness of lime treated clayey soils have also been studied (Ying et al., 2021; Tang et al., 2011; Dong, 2013; Wang et al., 2020). The results showed that the treatment of clayey soils (CH and CL USCS class) with 2 to 4% quicklime amount causes a huge increase in G_0 with curing time.

While evaluating the use of G_0 as a key parameter to monitor the reaction developments, the necessity of a proper interpretation of the transmitted signal arose. Therefore, different methods of interpretation were

applied with the additional purpose of highlighting how the peculiarity of lime treated soils can affect the BE testing and in order to provide practical indications to optimize similar testing on lime-treated soils.

Materials and Test Methods

The studied soil is an inorganic clay of high plasticity (CH), according to the Unified Soil Classification System (USCS – ASTM D2487-93). Its characteristics are summarized in Table 2. The soil fully matches the suitable grain size distribution requirements for lime treatment. The amounts of sulphate and organic matter are 0.39% and 2.70% by dry mass of soil, respectively. The lime used in this research is a fine calcic quicklime, classified as CL80-Q (UNI EN 459-01 – $\text{CaO} > 87\%$, $\text{MgO} < 5\%$), completely passing through the ASTM 200 sieve (75 μm sieve opening). The amount of lime added to the soil was 3% by dry mass of soil. This amount is higher than the initial consumption of lime (ICL, Table 2), that represents the minimum lime content to start pozzolanic reactions.

Table 2

The specimens were prepared by crumbling the air-dried soil, adding the amount of lime after soil wetting (wet mix - as usually done in the field, Fratolocchi et al., 2009) until a uniform distribution was achieved. The mixture was then compacted by the Standard Proctor procedure (ASTM D698-12).

In order to investigate the development of soil-lime reactions by pH measurements (methodology proposed and validated by Di Sante et al., 2014), a sample was compacted close to the optimum water content (optimum water content = 23%, dry unit weight = 15.6 kN/m³, details in Di Sante et al., 2016) and was submerged in distilled water, resting on its lateral surface to maximize the contact area with water. The development of soil-lime reactions was monitored by periodically measuring the pH values of the soaking water (pH Electrode Sentix 41, WTW).

The sample for BE test was compacted in three layers (101 mm in diameter and 116.5 mm in height) slightly wet of optimum ($w = 25\%$, corresponding to a saturation degree of 93%; dry unit weight = 15.4 kN/m³). It was immediately coated with paraffin, to prevent loss of water during the test. The sample weight was monitored throughout the entire curing period (98 days): its decrease resulted lower than 1%. The curing temperature was $23 \pm 1^\circ\text{C}$.

The BE test was conducted following the recent ASTM Standard D8295-19. An undersized slot was excavated in the two opposite faces of the specimen to easily insert the BE, as suggested for stiff specimens, paying attention to ensure their alignment; the specimen was placed on a specially designed wooden sample holder. The test was carried out in unconfined conditions. Although some researchers

reported difficulties in data interpretation such as a less efficient grounding and the possibility of a weak coupling of the transducer to the soil (de Paula et al., 2020), conducting BE test in unconfined conditions is usual in the area of stabilized soils (e.g. Puppala et al., 2006; Tang et al., 2011; Chan, 2012; Consoli et al., 2012; Wang et al., 2017; Wang et al., 2020). This test configuration originated from the complementarity of BE test and unconfined compression tests and thereafter became a common practice so much so that it is a possibility contemplated by the relevant ASTM Standard. Moreover, given the very low compressibility of the studied soil after the treatment (i.e. oedometric modulus = 26 MPa for 0-50 kPa of vertical stress range, see Di Sante et al., 2016) the effect of the confining stress is expected to be low.

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. Several frequencies were applied, ranging from 0.5 kHz to 50 kHz. The decision of using this wide span of input frequencies is due to two orders of reasons: (1) there are not consolidated indications in literature on the optimal frequency of BE application to soil-lime materials and (2) considering the development of chemical reactions and the consequent changes in the structure of the sample, optimal testing frequency can vary with curing time and this evolution is not known at the beginning of the test. is not Both the transmitted and the received signals were acquired by a digital oscilloscope (Picoscope 6, Pico Technology LTD) and then processed. The experimental set up is shown in Figure 1.

Figure 1

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 98 days. The shear wave travel times, t , were computed by time domain methods (Peak to peak and Start to start) as suggested in ASTM Standard D8295-19. The Cross correlation method (Viggiani & Atkinson, 1995) was tentatively applied, too.

The small strain shear modulus, G_0 , was calculated through the shear wave speed, V_s , by the formula:

$$G_0 = \rho \cdot V_s^2 = \rho \cdot \left(\frac{L_{tt}}{t}\right)^2 \quad \text{eq.1}$$

where L_{tt} is the BE tip to tip distance and ρ is the density of the sample.

An untreated sample was also prepared at the same compaction conditions and tested with BE in order to identify the contribution of lime to the stiffness of the treated soil. The results of the untreated sample were considered also representative of the initial conditions when analysing the effects of curing time.

Results and Discussion

Travel time interpretation issues

Both time domain and frequency domain methods were initially taken into account for signal interpretation. In particular, Peak to Peak (P-P) and Start to Start (S-S) methods are suggested in the ASTM 8295-19 Standard (S-S measured from the start of the transmitter signal to the horizontal intersection on the receiver signal). The Standard also allows the application of ~~frequency-domain~~ other methods, provided that they have been shown to give reliable results. Therefore, the cross-correlation method, C-C, introduced in Viggiani and Atkinson (1995), was also tentatively applied, to avoid the more subjective visual picking methods.

In the present study the recorded received signal showed a first peak that rarely had the highest amplitude, whereas, after the wave arrival, the amplitude increased with time and then gently decreased (Figure 2). This type of received waveform was observed also by other researchers dealing with cemented soil (e.g. Chan, 2010; Consoli et al., 2012). Probably, due to this particular feature and to a difference between the frequency of the transmitted and received signals, the application of cross correlation method gave the maximum cross-correlation with the maximum peak of the received signal, resulting in a travel time significantly higher than other methods and significantly scattering among the subsequent measurements (as also reported by Ogino et al., 2015). This suggested that, in the case of concern, lime treatment and compaction, by affecting the stiffness of the specimens (as discussed in the following), could play a key role in modifying the ability of the soil to transmit the signal, thus affecting the interpretation of the test by this frequency domain method. Moreover, in the standard procedure for compaction, as per ASTM D698-12, three layers of soil are subsequently placed in the mould, each compacted with 25 blows, resulting in a decreasing applied compaction energy from the base to the top of the sample, causing an inhomogeneity in density across the sample which may have contributed to affect the test results.

In assessing the travel time by the S-S method, difficulties were encountered in following the Standard criterion because, in some of the transmissions, background noise, dispersion and near field effects made the picking of the start of the received signal rather difficult.

Given the above reasons, the P-P method was selected to identify the travel time in order to monitor stiffness variations and thus the development of reactions in the soil-lime specimen.

Figure 2

Effect of the input frequency on G_0

Input signal of several frequencies, f (Hz), ranging from 0.5 kHz to 50 kHz, were applied to the input signal; in Figure 3 G_0 values (calculated through eq. 1) are plotted versus the frequency of the input signal for three curing times (8, 28 and 81 days). For all the curing times considered, the obtained shear modulus increased with increasing frequency, up to 20 kHz and stabilized thereafter. It is worth noticing that at low frequencies (i.e. 0.5 to 5 kHz) the ratio between L_t and the wavelength, λ (computed as V_s/f) is lower than 2. As widely reported in the literature (e.g. Sanchez-Salinero et al., 1986; Arulnathan et al., 1998; Arroyo et al., 2003; Wang et al., 2007), values of $L_t/\lambda < 2$ or greater than 9 are associated to a prevailing near field effect which masks the actual arrival of the received signal that is consequently difficult to be read.

Figure 3

By comparing the high frequency G_0 values at the three curing times (Figure 3) it is also evident the increase in the small strain shear modulus with curing: from 690 MPa at 8 days of curing to 1150 MPa at 81 days. In the first 7 days of curing, only 0.5 to 10 kHz input signals were readable, whereas higher frequency signals (20-50 kHz) resulted undetectable by the receiver BE; on the contrary, after 60 days of curing, the output signal corresponding to 0.5 kHz input signal was not readable. This variation in the sample behaviour is an additional sign of the increase in stiffness with curing; in fact, as reported by Lee & Santamarina (2005), the readability of the signal is optimized when the frequency approaches the resonant frequency of the soil-BE system and the higher the soil stiffness, the higher the resonant frequency, f_R .

In order to verify this occurrence, f_R was measured, by means of Lissajous forms, at different curing times obtaining the values shown in Figure 4 plotted as a function of the shear wave speed, V_s , calculated by eq.1.

An estimate of f_R on varying of the soil stiffness in terms of V_s was proposed by Lee & Santamarina (2005) by combining the mass and the stiffness of the BE and those of the affected soil, assuming for the BE a cantilever beam behaviour and considering that the BE is buried in a soil mass. The involved parameters (defined in Lee and Santamarina, 2005) were determined according to the case of concern (type of BE used and lime treated soil) and the effective length factor, α , ($=1$ for a perfectly rigid anchor, >1 for a soft anchor of the BE) is considered equal to 1.5, as in the reference study. The effective soil mass factor, β , that, in this particular case, represents the soil-lime mass factor, was determined by fitting the experimental data. The best fitting was obtained for $\beta=1.8$ (see the trend of " f_R , calculated" in Figure 4).

Figure 4

The effect of the increase in stiffness is well described by the expression proposed by Lee and Santamarina (2005) even if, in this case, the increase is mainly due to the chemical reactions that develop with curing time, instead of an increase in the effective stress state.

Effect of the curing time on G_0

The input signal at $f=10\text{kHz}$ is the only readable through the entire period of curing, while giving, in the majority of the performed transmissions, acceptable values of the ratio L_t/λ . Therefore, the reactions progress in the tested sample can be monitored during the whole curing period, referring to the trend of G_0 values calculated at $f=10\text{ kHz}$.

By plotting G_0 values as a function of the curing time (Figure 5) it is possible to observe its increasing trend, obtaining indirect information on the development of soil-lime reactions.

The G_0 computed for the compacted untreated soil (20.9 MPa) is considered as the initial value for the soil-lime trend. As observable by Figure 5 (curing days in log-scale zoom), G_0 slowly increased during the first day of curing and was subjected to a sharp increase at 2 days of curing, almost tripling its value between 24 and 48 hours. This trend is clearly observable also for 1 and 2 kHz of frequency of the transmitting signal. The timing of reactions obtained by monitoring the pH of the soil-lime system are reported in Figure 6 (details of pH trend and related comments in Di Sante et al., 2016).

The trend of pH and ion concentration, consistently with that of G_0 , suggests that cation exchange reaction is completed within 2 days of curing (see Figure 5) because during these first 2 days the pH holds steady at high values (>12), suggesting that oxydriol ions are not involved in this reaction phase, as in the cation exchange reaction (see the description of reactions in Table 1). Therefore, the sharp increase in stiffness at 2 days of curing marks the prevailing effect of pozzolanic reactions over the cation exchange reaction. Also this increase in G_0 reflects the timing identified with the pH trend: in pozzolanic reactions 2 hydroxyl ions for each calcium ion reacting with silica or alumina are consumed (see Table 1) causing the pH to decrease.

From then on, measurements at different frequencies consistently give a progressive increase in stiffness, from 265 MPa at 2 days (value calculated for a transmitted wave of 10kHz) and reaching 1535 MPa at 98 days of curing (mean value calculated for $f=10, 20, 30\text{ kHz}$).

The obtained experimental results are in agreement with those of other researchers (Tang et al., 2011; Dong, 2013; Wang et al., 2020; Ying et al., 2021) dealing with BE applications for silty and clayey soils treated with 2-4% quicklime and compacted. They also identified a two-phase change pattern of the G_0 modulus. A first, very slight increasing trend in the first 40-100 hours was attributed to cation

exchange/flocculation and then a sharp increase was registered and related to pozzolanic reactions; the stabilization of G_0 required long curing times (i.e. more than 80 days of curing).

The observed development of stiffness in time is consistent with the pH measurements for which pozzolanic reactions are still in progress at 70 days of curing (Figure 6) although at a lower rate if compared with the first 28 days of curing.

The effect of short term reactions is mainly a re-arrangement of soil particles that causes a modification in the soil structure (see Table 1) but not a significant increase in stiffness; whereas the stiffness is highly incremented by the cementing and bonding effect resulting from pozzolanic reactions. This occurrence can also be justified by investigation of the cementation products at a micro-scale level (Russo et al., 2019; Di Sante, 2016; Kasyap et al., 2021), in fact, the calcium silicate hydrates formed with pozzolanic reactions, coating and bonding the soil grains together, are capable to strengthen and stiffen the treated soil.

Figure 5

Figure 6

Conclusions

With reference to the studied soil-lime mixture (CH soil treated with 3% of quicklime), the following conclusions can be drawn.

- Lime treatment and compaction affect the waveform of the received signal, thus influencing the applicability of some of the possible methods to determine the travel time.
- The stiffness of the studied soil-lime mixture increases with curing time as a result of chemical reactions taking place after the addition of the binder. By observing the trend of the small strain shear modulus with curing time, the two typical stages of reaction can be identified, regardless of the frequency of the transmitted signal; therefore, G_0 is a promising parameter to monitor the soil-lime reaction development.
- Although pH measurements are surely an easier method to be performed in order to have information about the reactions timing, BE measurements offer additional information about the obtainable stiffness of the mixture.
- In order to obtain a representative value of the small strain shear modulus for a soil-lime mixture, BE measurements should be done using a wide range of frequencies of the input signal. In the present

case, the received signal, at low input frequencies (i.e. <10 kHz), was affected by the near field effect and its interpretation gave lower G_0 values than those obtained with high frequencies.

- In light of the obtained results, a suitable method to estimate the resonant frequency of the system as a function of the stiffness is the analytical solution by Lee and Santamarina (2005), adjusting the parameters for the lime treated soil.

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LINKING SMALL-STRAIN STIFFNESS TO DEVELOPMENT OF CHEMICAL REACTIONS IN LIME-TREATED SOILS

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Abstract

In the context of lime stabilization, this note shows how the development of soil-lime reactions can be linked to the variation of small shear stiffness with time. To determine the evolution of small-strain stiffness, the shear wave velocity was measured by means of bender elements (BE) on a compacted clayey soil treated with 3% quicklime, starting from 2 hours after compaction until 98 days of curing. Different methods of signal interpretation were applied with the purpose of highlighting how the peculiarity of lime treated soils affects BE testing results and to provide practical indications for optimizing similar testing on lime-treated soils. The results showed that lime treatment and compaction affect the waveform of the received signal and that measurements should span across a wide range of input frequencies in order to identify an optimal waveform. The small strain shear modulus was found to increase with curing time with a trend that can be related to that of soil-lime chemical reactions, thus representing a promising parameter to monitor the development of soil-lime reactions.

Keywords

Soil stabilization, lime, small-strain stiffness, shear wave velocity, bender elements

Introduction

Lime stabilization of clayey soils is a well-known and sustainable ground improvement technique for the construction of earthen structures (e.g. Boardman et al., 2001; Beetham et al., 2014; Gomes Correia et al., 2016; Rosone et al., 2018). Despite its wide application in engineering works, the optimization of the mix design procedure and the search for methods and correlations to predict soil-lime performances are the objective of recent research studies (e.g. Consoli et al., 2014; Robin et al., 2015; Di Sante et al., 2015; Consoli et al., 2017; Di Sante et al., 2020; Fratolocchi et al., 2020). Moreover, delayed compaction (due to hitches or technical breaks during construction e.g. Osinubi et al., 2006; Di Sante et al., 2015), wetting-drying and freezing-thawing cycles (e.g. Stoltz et al., 2012; Shirmohammadi et al., 2021) and a lack of an extensive experimental laboratory investigation can potentially adversely affect the stabilization outcomes, therefore these occurrences should be taken into account during the design phase. In this perspective, understanding the sequence, mode and timing of reactions between lime and clayey soils plays an essential role. Table 1 summarizes the reactions that develop in a soil-quicklime-water system and the related effects.

Table 1

Several studies have been carried out to identify the chemo-mineralogical evolution of lime treated soils with time (e.g. Vitale et al., 2017; Guidobaldi et al., 2018). Among the different methodologies proposed to identify

the reactions timing and evolution, those based on pH measurements of the soil-lime-water system are easy to be carried out and turned out to be very effective and reliable (Rao and Shivananda, 2005; Al Muckthar , 2010; Di Sante et al., 2014, Di Sante, 2020).

In the present note a preliminary study to monitor the development of chemical reactions based on stiffness variation of a clayey soil treated with quicklime is presented. The results are compared with those obtained by the pH method (Di Sante et al., 2014). A parameter which can reflect the structure of the porous medium is the small-strain shear modulus, G_0 , that is linked to the stiffness of the material that, in turn, is expected to change with the progress of chemical reactions, thus with curing time. Although G_0 can increase in time even for untreated soils due to ageing phenomena (Schmertmann, 1991; Jovičić et al., 1997; Rammah et al., 2004), laboratory data showed that the maximum rate of increase of G_0 with the logarithm of time (in days) can be equal to 19% in case of secondary compression.

G_0 can be measured by bender elements (BE) test in a non-destructive way, which allows testing the same soil-lime sample for the entire curing period. The small-strain shear modulus is related to the shear waves velocity that can be calculated from the travel time measured generating a shear wave at one boundary of the specimen (transmitter BE) and receiving the wave at the opposite boundary (receiver BE).

Piezoelectric transducers applications for soils were originally introduced by Lawrence in '60s (Lawrence, 1963) and, from then on, the experimental setting has been improved as well as the methods of signal interpretation (e.g. Arulnathan et al., 1998; Wang et al., 2017). Some attempts to apply the BE technique to study the stiffness of lime treated soils with several different aims have been recently made. In particular, 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 (with different sulphate levels and curing conditions) by BE tests, finding 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 using a 2% quicklime treated compacted plastic silt at different curing times and saturation degrees. The effects of wetting-drying cycles and of the aggregate sizes on the stiffness of lime treated clayey soils have also been studied (Ying et al., 2021; Tang et al., 2011; Dong, 2013; Wang et al., 2020). The results showed that the treatment of clayey soils (CH and CL USCS class) with 2 to 4% quicklime amount causes a huge increase in G_0 with curing time. While evaluating the use of G_0 as a key parameter to monitor the reaction developments, the necessity of a proper interpretation of the transmitted signal arose. Therefore, different methods of interpretation were

101 applied with the additional purpose of highlighting how the peculiarity of lime treated soils can affect the BE
102 testing and in order to provide practical indications to optimize similar testing on lime-treated soils.

103 **Materials and Test Methods**

104 The studied soil is an inorganic clay of high plasticity (CH), according to the Unified Soil Classification
105 System (USCS – ASTM D2487-93). Its characteristics are summarized in Table 2. The soil fully matches the
106 suitable grain size distribution requirements for lime treatment. The amounts of sulphate and organic matter
107 are 0.39% and 2.70% by dry mass of soil, respectively. The lime used in this research is a fine calcic
108 quicklime, classified as CL80-Q (UNI EN 459-01 – $\text{CaO} > 87\%$, $\text{MgO} < 5\%$), completely passing through the
109 ASTM 200 sieve (75 μm sieve opening). The amount of lime added to the soil was 3% by dry mass of soil.
110 This amount is higher than the initial consumption of lime (ICL, Table 2), that represents the minimum lime
111 content to start pozzolanic reactions.

112 **Table 2**

113 The specimens were prepared by crumbling the air-dried soil, adding the amount of lime after soil wetting
114 (wet mix - as usually done in the field, Fratolocchi et al., 2009) until a uniform distribution was achieved. The
115 mixture was then compacted by the Standard Proctor procedure (ASTM D698-12).

116 In order to investigate the development of soil-lime reactions by pH measurements (methodology proposed
117 and validated by Di Sante et al., 2014), a sample was compacted close to the optimum water content
118 (optimum water content= 23%, dry unit weight=15.6 kN/m^3 , details in Di Sante et al., 2016) and was
119 submerged in distilled water, resting on its lateral surface to maximize the contact area with water. The
120 development of soil-lime reactions was monitored by periodically measuring the pH values of the soaking
121 water (pH Electrode Sentix 41, WTW).

122 The sample for BE test was compacted in three layers (101 mm in diameter and 116.5 mm in height) slightly
123 wet of optimum ($w = 25\%$, corresponding to a saturation degree of 93%; dry unit weight= 15.4 kN/m^3). It was
124 immediately coated with paraffin, to prevent loss of water during the test. The sample weight was monitored
125 throughout the entire curing period (98 days): its decrease resulted lower than 1%. The curing temperature
126 was $23 \pm 1^\circ\text{C}$.

127 The BE test was conducted following the recent ASTM Standard D8295-19. An undersized slot was
128 excavated in the two opposite faces of the specimen to easily insert the BE, as suggested for stiff
129 specimens, paying attention to ensure their alignment; the specimen was placed on a specially designed
130 wooden sample holder. The test was carried out in unconfined conditions. Although some researchers

131 reported difficulties in data interpretation such as a less efficient grounding and the possibility of a weak
 132 coupling of the transducer to the soil (de Paula et al., 2020), conducting BE test in unconfined conditions is
 133 usual in the area of stabilized soils (e.g. Puppala et al., 2006; Tang et al., 2011; Chan, 2012; Consoli et al.,
 134 2012; Wang et al., 2017; Wang et al., 2020). This test configuration originated from the complementarity of
 135 BE test and unconfined compression tests and thereafter became a common practice so much so that it is a
 136 possibility contemplated by the relevant ASTM Standard. Moreover, given the very low compressibility of the
 137 studied soil after the treatment (i.e. oedometric modulus = 26 MPa for 0-50 kPa of vertical stress range, see
 138 Di Sante et al., 2016) the effect of the confining stress is expected to be low.

139 The input signal, a sine pulse of 10 Vpp amplitude, was generated by a function generator (Aim TTI
 140 TG5011A), triggered with a period set to allow enough time for the attenuation of the BE response before the
 141 next pulse. Several frequencies were applied, ranging from 0.5 kHz to 50 kHz. The decision of using this
 142 wide span of input frequencies is due to two orders of reasons: (1) there are not consolidated indications in
 143 literature on the optimal frequency of BE application to soil-lime materials and (2) considering the
 144 development of chemical reactions and the consequent changes in the structure of the sample, optimal
 145 testing frequency can vary with curing time and this evolution is not known at the beginning of the test. is not
 146 Both the transmitted and the received signals were acquired by a digital oscilloscope (Picoscope 6, Pico
 147 Technology LTD) and then processed. The experimental set up is shown in Figure 1.

148 **Figure 1**

149 The first measurement was performed two hours after mixing and compaction of the soil-lime sample and the
 150 measurements were repeated at increasing curing time until 98 days. The shear wave travel times, t , were
 151 computed by time domain methods (Peak to peak and Start to start) as suggested in ASTM Standard
 152 D8295-19. The Cross correlation method (Viggiani & Atkinson, 1995) was tentatively applied, too.

153 The small strain shear modulus, G_0 , was calculated through the shear wave speed, V_s , by the formula:

$$154 \quad G_0 = \rho \cdot V_s^2 = \rho \cdot \left(\frac{L_{tt}}{t} \right)^2 \quad \text{eq.1}$$

155 where L_{tt} is the BE tip to tip distance and ρ is the density of the sample.

156 An untreated sample was also prepared at the same compaction conditions and tested with BE in order to
 157 identify the contribution of lime to the stiffness of the treated soil. The results of the untreated sample were
 158 considered also representative of the initial conditions when analysing the effects of curing time.

159 **Results and Discussion**

160 **Travel time interpretation issues**

161 Both time domain and frequency domain methods were initially taken into account for signal interpretation. In
162 particular, Peak to Peak (P-P) and Start to Start (S-S) methods are suggested in the ASTM 8295-19
163 Standard (S-S measured from the start of the transmitter signal to the horizontal intersection on the receiver
164 signal). The Standard also allows the application of other methods, provided that they have been shown to
165 give reliable results. Therefore, the cross-correlation method, C-C, introduced in Viggiani and Atkinson
166 (1995), was also tentatively applied, to avoid the more subjective visual picking methods.

167 In the present study the recorded received signal showed a first peak that rarely had the highest amplitude,
168 whereas, after the wave arrival, the amplitude increased with time and then gently decreased (Figure 2). This
169 type of received waveform was observed also by other researchers dealing with cemented soil (e.g. Chan,
170 2010; Consoli et al., 2012). Probably, due to this particular feature and to a difference between the frequency
171 of the transmitted and received signals, the application of cross correlation method gave the maximum cross-
172 correlation with the maximum peak of the received signal, resulting in a travel time significantly higher than
173 other methods and significantly scattering among the subsequent measurements (as also reported by Ogino
174 et al., 2015). This suggested that, in the case of concern, lime treatment and compaction, by affecting the
175 stiffness of the specimens (as discussed in the following), could play a key role in modifying the ability of the
176 soil to transmit the signal, thus affecting the interpretation of the test by this frequency domain method.
177 Moreover, in the standard procedure for compaction, as per ASTM D698-12, three layers of soil are
178 subsequently placed in the mould, each compacted with 25 blows, resulting in a decreasing applied
179 compaction energy from the base to the top of the sample, causing an inhomogeneity in density across the
180 sample which may have contributed to affect the test results.

181 In assessing the travel time by the S-S method, difficulties were encountered in following the Standard
182 criterion because, in some of the transmissions, background noise, dispersion and near field effects made
183 the picking of the start of the received signal rather difficult.

184 Given the above reasons, the P-P method was selected to identify the travel time in order to monitor stiffness
185 variations and thus the development of reactions in the soil-lime specimen.

186 **Figure 2**

187 **Effect of the input frequency on G_0**

188 Input signal of several frequencies, f (Hz), ranging from 0.5 kHz to 50 kHz, were applied to the input signal; in
189 Figure 3 G_0 values (calculated through eq. 1) are plotted versus the frequency of the input signal for three
190 curing times (8, 28 and 81 days). For all the curing times considered, the obtained shear modulus increased
191 with increasing frequency, up to 20 kHz and stabilized thereafter. It is worth noticing that at low frequencies
192 (i.e. 0.5 to 5 kHz) the ratio between L_t and the wavelength, λ (computed as V_s/f) is lower than 2. As widely
193 reported in the literature (e.g. Sanchez-Salinero et al., 1986; Arulnathan et al., 1998; Arroyo et al., 2003;
194 Wang et al., 2007), values of $L_t/\lambda < 2$ or greater than 9 are associated to a prevailing near field effect which
195 masks the actual arrival of the received signal that is consequently difficult to be read.

196 **Figure 3**

197

198 By comparing the high frequency G_0 values at the three curing times (Figure 3) it is also evident the increase
199 in the small strain shear modulus with curing: from 690 MPa at 8 days of curing to 1150 MPa at 81 days. In
200 the first 7 days of curing, only 0.5 to 10 kHz input signals were readable, whereas higher frequency signals
201 (20-50 kHz) resulted undetectable by the receiver BE; on the contrary, after 60 days of curing, the output
202 signal corresponding to 0.5 kHz input signal was not readable. This variation in the sample behaviour is an
203 additional sign of the increase in stiffness with curing; in fact, as reported by Lee & Santamarina (2005), the
204 readability of the signal is optimized when the frequency approaches the resonant frequency of the soil-BE
205 system and the higher the soil stiffness, the higher the resonant frequency, f_R .

206 In order to verify this occurrence, f_R was measured, by means of Lissajous forms, at different curing times
207 obtaining the values shown in Figure 4 plotted as a function of the shear wave speed, V_s , calculated by eq.1.

208 An estimate of f_R on varying of the soil stiffness in terms of V_s was proposed by Lee & Santamarina (2005)
209 by combining the mass and the stiffness of the BE and those of the affected soil, assuming for the BE a
210 cantilever beam behaviour and considering that the BE is buried in a soil mass. The involved parameters
211 (defined in Lee and Santamarina, 2005) were determined according to the case of concern (type of BE used
212 and lime treated soil) and the effective length factor, α , (=1 for a perfectly rigid anchor, >1 for a soft anchor of
213 the BE) is considered equal to 1.5, as in the reference study. The effective soil mass factor, β , that, in this
214 particular case, represents the soil-lime mass factor, was determined by fitting the experimental data. The
215 best fitting was obtained for $\beta=1.8$ (see the trend of " f_R , calculated" in Figure 4).

216 **Figure 4**

217 The effect of the increase in stiffness is well described by the expression proposed by Lee and Santamarina
218 (2005) even if, in this case, the increase is mainly due to the chemical reactions that develop with curing
219 time, instead of an increase in the effective stress state.

220 **Effect of the curing time on G_0**

221 The input signal at $f=10\text{kHz}$ is the only readable through the entire period of curing, while giving, in the
222 majority of the performed transmissions, acceptable values of the ratio L_H/λ . Therefore, the reactions
223 progress in the tested sample can be monitored during the whole curing period, referring to the trend of G_0
224 values calculated at $f=10\text{ kHz}$.

225 By plotting G_0 values as a function of the curing time (Figure 5) it is possible to observe its increasing trend,
226 obtaining indirect information on the development of soil-lime reactions.

227 The G_0 computed for the compacted untreated soil (20.9 MPa) is considered as the initial value for the soil-
228 lime trend. As observable by Figure 5 (curing days in log-scale zoom), G_0 slowly increased during the first
229 day of curing and was subjected to a sharp increase at 2 days of curing, almost tripling its value between 24
230 and 48 hours. This trend is clearly observable also for 1 and 2 kHz of frequency of the transmitting signal.
231 The timing of reactions obtained by monitoring the pH of the soil-lime system are reported in Figure 6 (details
232 of pH trend and related comments in Di Sante et al., 2016).

233 The trend of pH and ion concentration, consistently with that of G_0 , suggests that cation exchange reaction is
234 completed within 2 days of curing (see Figure 5) because during these first 2 days the pH holds steady at
235 high values (>12), suggesting that oxydriol ions are not involved in this reaction phase, as in the cation
236 exchange reaction (see the description of reactions in Table 1). Therefore, the sharp increase in stiffness at
237 2 days of curing marks the prevailing effect of pozzolanic reactions over the cation exchange reaction. Also
238 this increase in G_0 reflects the timing identified with the pH trend: in pozzolanic reactions 2 hydroxyl ions for
239 each calcium ion reacting with silica or alumina are consumed (see Table 1) causing the pH to decrease.
240 From then on, measurements at different frequencies consistently give a progressive increase in stiffness,
241 from 265 MPa at 2 days (value calculated for a transmitted wave of 10kHz) and reaching 1535 MPa at 98
242 days of curing (mean value calculated for $f=10, 20, 30\text{ kHz}$).

243 The obtained experimental results are in agreement with those of other researchers (Tang et al., 2011;
244 Dong, 2013; Wang et al., 2020; Ying et al., 2021) dealing with BE applications for silty and clayey soils
245 treated with 2-4% quicklime and compacted. They also identified a two-phase change pattern of the G_0
246 modulus. A first, very slight increasing trend in the first 40-100 hours was attributed to cation

247 exchange/flocculation and then a sharp increase was registered and related to pozzolanic reactions; the
248 stabilization of G_0 required long curing times (i.e. more than 80 days of curing).

249 The observed development of stiffness in time is consistent with the pH measurements for which pozzolanic
250 reactions are still in progress at 70 days of curing (Figure 6) although at a lower rate if compared with the first
251 28 days of curing.

252 The effect of short term reactions is mainly a re-arrangement of soil particles that causes a modification in
253 the soil structure (see Table 1) but not a significant increase in stiffness; whereas the stiffness is highly
254 incremented by the cementing and bonding effect resulting from pozzolanic reactions. This occurrence can
255 also be justified by investigation of the cementation products at a micro-scale level (Russo et al., 2019; Di
256 Sante, 2016; Kasyap et al., 2021), in fact, the calcium silicate hydrates formed with pozzolanic reactions,
257 coating and bonding the soil grains together, are capable to strengthen and stiffen the treated soil.

258 **Figure 5**

259

260 **Figure 6**

261

262 **Conclusions**

263 With reference to the studied soil-lime mixture (CH soil treated with 3% of quicklime), the following
264 conclusions can be drawn.

- 265 - Lime treatment and compaction affect the waveform of the received signal, thus influencing the
266 applicability of some of the possible methods to determine the travel time.
- 267 - The stiffness of the studied soil-lime mixture increases with curing time as a result of chemical
268 reactions taking place after the addition of the binder. By observing the trend of the small strain
269 shear modulus with curing time, the two typical stages of reaction can be identified, regardless of the
270 frequency of the transmitted signal; therefore, G_0 is a promising parameter to monitor the soil-lime
271 reaction development.
- 272 - Although pH measurements are surely an easier method to be performed in order to have
273 information about the reactions timing, BE measurements offer additional information about the
274 obtainable stiffness of the mixture.
- 275 - In order to obtain a representative value of the small strain shear modulus for a soil-lime mixture, BE
276 measurements should be done using a wide range of frequencies of the input signal. In the present

277 case, the received signal, at low input frequencies (i.e. <10 kHz), was affected by the near field
278 effect and its interpretation gave lower G_0 values than those obtained with high frequencies.

279 - In light of the obtained results, a suitable method to estimate the resonant frequency of the system
280 as a function of the stiffness is the analytical solution by Lee and Santamarina (2005), adjusting the
281 parameters for the lime treated soil.

282

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413

414

Table 1. Reactions in soil-lime water system and main effects in the short and long term -
*C-S-H and C-A-H are Calcium Silicate and Aluminum Hydrates, respectively. (e.g. TRB, 1987; Beetham et al., 2014)

Table 2 – Soil characteristics (ICL = Initial Consumption of Lime – ASTM C977-00(18)).

Figure 1 – Experimental set up for BE test

Figure 2 – Comparison between Peak to Peak and Cross-Correlation methods -
Transmitted and received signals at 2 hours of curing – frequency of 2 kHz.

Figure 3 – G_0 values determined at different frequencies and related signals at (a) 8 days, (b) 28days, (c) 81 days of curing. For each received signal the travel time, t , is reported.

Figure 4 – Soil-lime stiffness effect on resonant frequency– diamond points are the experimental results, the dashed line corresponds to the analytical solution by Lee and Santamarina (2005) for $\alpha=1.5$ and $\beta=1.8$

Figure 5 – G_0 values of the soil-lime sample with curing time (log scale in the zoomed plot for better visualization of short curing times)

Figure 6 – Timing of reactions as inferred by pH method (CER=cation exchange reaction, PR=pozzolanic reactions)

Fig 2

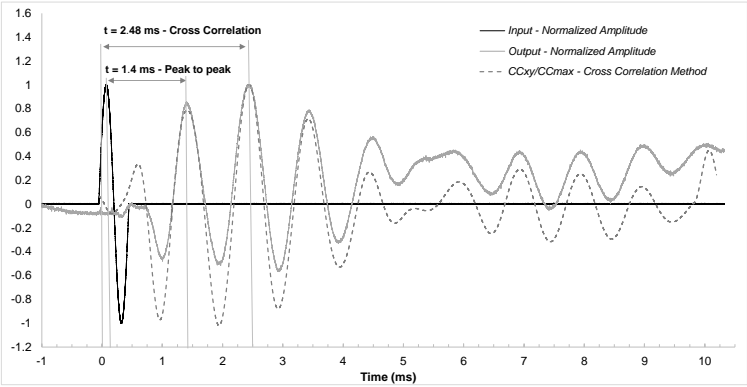


Fig 4

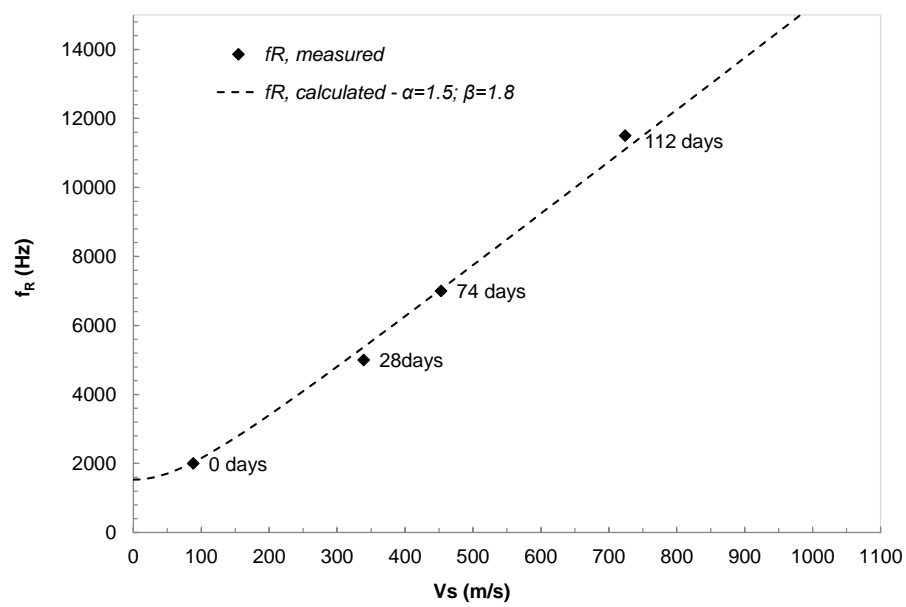


Fig 5

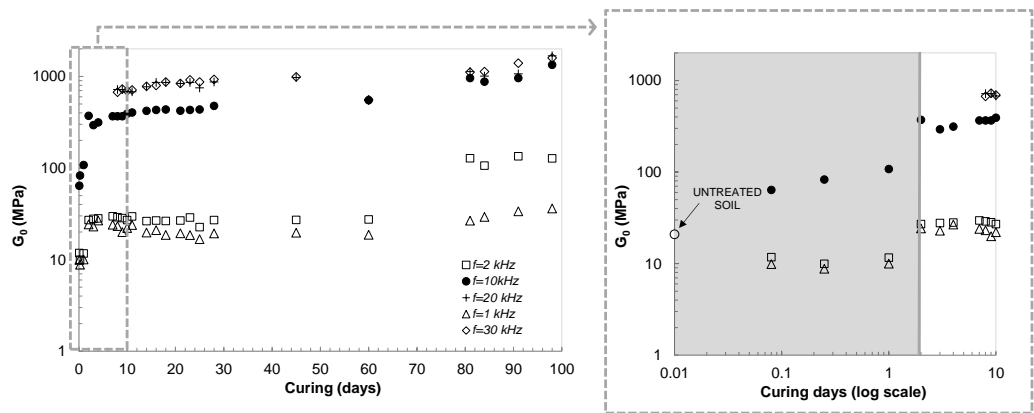
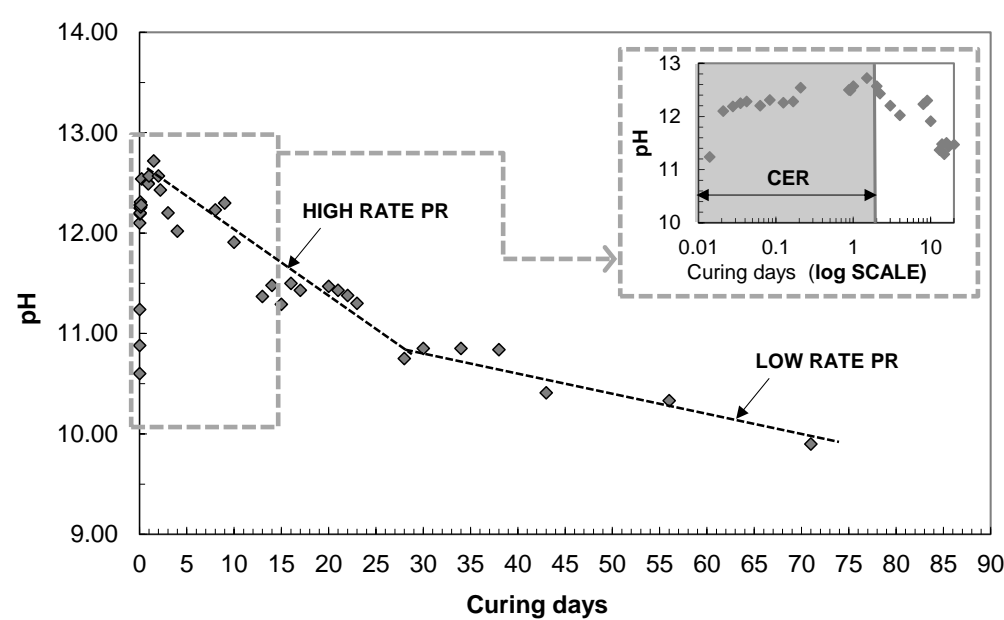
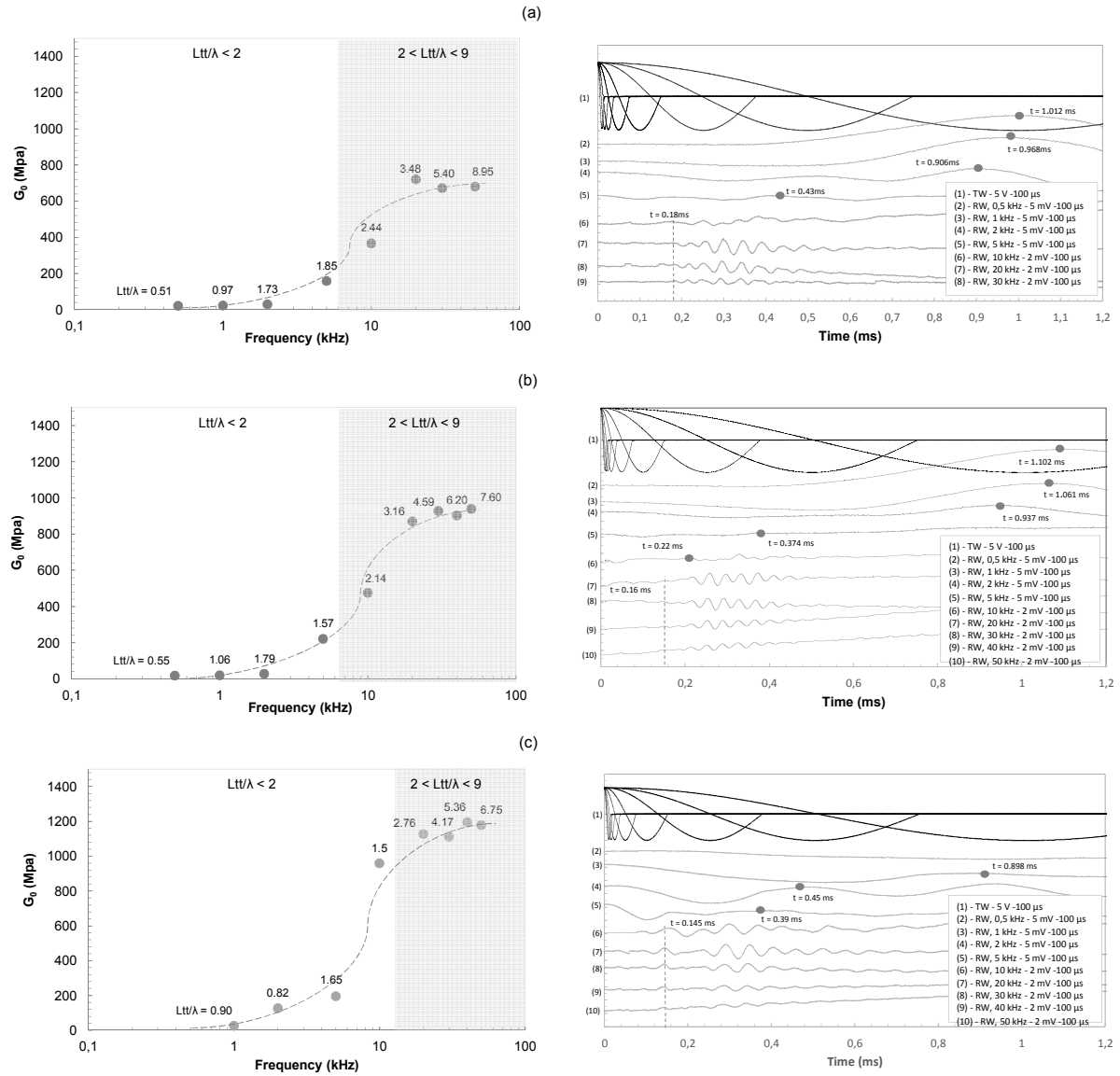


Fig 6





- In BE testing lime treatment affects the waveform of the received signal
- measurements should cover a wide range of input frequencies in order to identify an optimal waveform
- low input frequencies give lower G_0 values than those obtained with high frequencies
- G_0 was found to increase with curing time following a two-phase trend
- G_0 is a promising parameter to monitor the development of soil-lime reactions

REACTIONS		MAIN EFFECTS
"short term"		
quicklime hydration	$\text{CaO} + \text{H}_2\text{O} \rightarrow \text{Ca(OH)}_2$	soil drying
hydroxide dissociation	$\text{Ca(OH)}_2 \rightarrow \text{Ca}^{++} + 2\text{OH}^-$	rising of pH and of electrolyte concentration in pore water
cation exchange reaction (CER)	Ca^{++} replace K^+ , Na^+ , H^+ on clayey particles	Flocculation/aggregation of clay particles: reduction in plasticity and soil-water affinity, increase in hydraulic conductivity
"long term"		
pozzolanic reactions (PR)	$\text{Ca}^{++} + 2 \text{OH}^- + \text{SiO}_2 \rightarrow \text{C-S-H}^*$	Cementation: higher strength, reduced deformability, higher durability
	$\text{Ca}^{++} + 2 \text{OH}^- + \text{Al}_2\text{O}_3 \rightarrow \text{C-A-H}^*$	

Sand (<2mm%)	3
Fine (<0.075mm,%)	97
Clay (<0.002mm,%)	52
Specific Gravity (-)	2.65
Liquid limit (%)	57
Plasticity Index (%)	33
ICL - %CaO	1.5

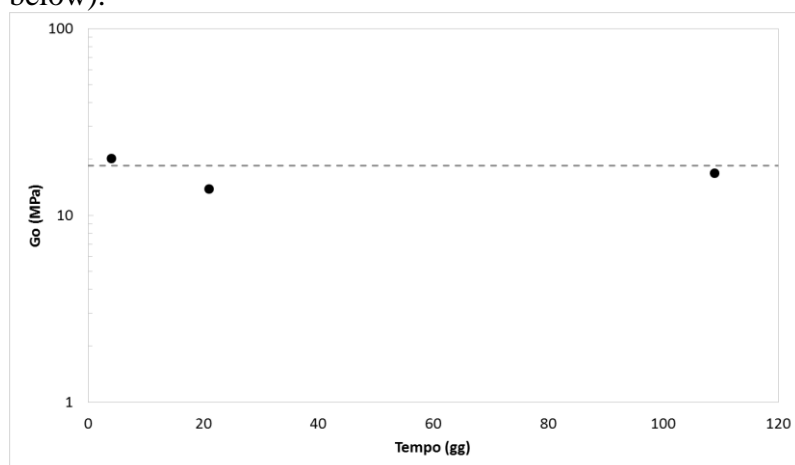
RESPONSE TO EDITOR AND REVIEWERS

We thank the Editor and the two reviewers for the time spent in reviewing the paper and for the valuable comments. In the marked version of the manuscript we highlighted in red the changes/additional comments suggested by the Reviewers.

In the following all the comments of each reviewer are specifically addressed.

Reviewer #1

- 1) We presented “bender elements” in lower-case letters across the text.
- 2) We added the discussion of the main limitations from the use of lime in the Introduction section, citing the related literature – lines 60-65, as suggested.
- 3) We made the two paragraphs as a unique paragraph.
- 4) We added the requested brief discussion in the Introduction section at lines 78-81
- 5) We added the specific gravity of solids of the soil in Table 2.
- 6) The creep in soil is a rearrangement of soil particles caused by an applied effective stress that remains constant for a long period of time. Creep is expected to be significant in soft or organic clays. In the case of concern, during the test, the sample is unconfined, therefore no external stress is applied. In any case, the effect of long term (i.e. pozzolanic) reactions can possibly to mask the effect of the creep, whenever significant. In order to investigate this issue, we also tested in unconfined conditions an untreated compacted sample for more than 100 days observing no significant effect of creep on small strain stiffness (see the figure below).



- 7) We increased the size of the axes in the graphs, as requested.
- 8) We added the brief discussion about unconfined conditions at lines 128-136, also supported by newly added references.
- 9) We explained the reason for the wide span of input frequency used for the tests at lines 140-144.
- 10) We added the brief discussion and references, as per reviewer's suggestions, at lines 254-257.

Reviewer #2

- 1) As suggested by the reviewer, we no more cited the cross-correlation as a frequency domain method.

- 2) By varying the input frequency, the signals were mainly out of phase with the exception of the resonant frequency cases, discussed at lines 206-215.
- 3) We added the literature on the cation exchange reaction in the captions of Table 1 in which the reactions are summarized. We recognized that additional explanation is necessary across the text when commenting on the timing of reactions, therefore we better explained the correlation between the pH and the soil-lime reactions at lines 234-236 and 237-239.