In-Situ Testing of a Cement-Stabilized Metallurgical Residue

Marcos Arroyo
UPC, Barcelona, Spain. E-mail: marcos.arroyo@upc.edu

Sara Amoroso
Istituto Nazionale di Geofisica e Vulcanologia, L'Aquila, Italy. E-mail: sara.amoroso@ingv.it

Marcelo Devincenzi, Norma Pérez
Igeotest, Figueres,Spain. E-mail: marcelo@igeotest.com

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ABSTRACT: A large deposit of cement-stabilized jarosite was investigated by means of DMT as well as other in-situ tests (e.g. CPTu) and laboratory tests on core and block samples retrieved from the deposit. The material of dominant silt size, contains no silicates, has a large porosity and a relatively high (11%) cement content that results in a significant –but fragile- mechanical improvement. The deposit was built without resource to systematic compaction and the in-situ tests reveal significant inhomogeneities despite the industrial origin of the material. This paper examines the in situ test results focusing on basic index and state properties of the residue. The performance of correlations and interpretation methods well proven in conventional soils (i.e. silicate-based) is examined in detail. Particular attention is given to cement-induced apparent overconsolidation. The classical K_D based correlation for clay appears to perform relatively well.

1 INTRODUCTION

Because of the large quantity of data that they provide, their speed and simplicity and good repeatability, in situ probing tests such as the DMT and the CPTu are now the major tool for geotechnical soil investigation. It is complicated to find analytical interpretations of these probing tests that take into account the installation procedure and therefore empirical interpretations of their results still play a large role. Empirical interpretations in this context are correlations between some in situ test result and a laboratory determination. The limit of this approach is that the applicability of any such correlation outside the initial range of materials for which it was established is questionable. Mineralogy is always cited as one of the main determinants of soil behaviour (e.g. Wroth 1984). It is then interesting to explore the behaviour of correlations established for silicate soils in a case in which the soil contains no silicates at all.

2 CASE DESCRIPTION

2.1 *Material*

Sludges are typical wastes from the metallurgical industry (Zheng & Kozinski 1996), and often a major cause of environmental concern. This is the case, for instance, of the leached iron residues from electrolytic zinc production. One of their most common form is jarosites, hydrated ferric sulphates rich in heavy ions that may be easily washed away in the porewater that permeates the residue. Jarofix (Seyer et al. 2001, Chen and Dutrizac 1996) is a mixture of jarosite, lime and cement made at the end of the zinc electrolytic process resulting on chemical stabilisation of the jarosite residues. It has now been adopted by several large plants world-wide as the method of choice for jarosite treatment. After treatment, the Jarofix mixture may be safely landfilled.

The Jarofix here studied was industrially produced mixing sodium jarosite with lime and Portland cement in a proportion of 1 % and 12.5 % of, respectively, lime and cement weight to dry residue weight. The mixture takes place in a specially designed unit at the end of the factory residue disposal line, after filtering and drying. Even after that process, the residue is akin to a slurry, and the Jarofix here studied had mixing water contents between 92 % and 105 %. After set-up the water content reduced to values near 70%.

X-ray diffraction of the hardened product reveals a composition were the dominant species are sodium jarosite, $NaFe_3(SO_4)_2(OH)_6$, and gypsum

$CaSO_4.2H_2O$. Gypsum is already present in the residue but it does also appear as a reaction product of sodium jarosite and Portland cement. The reaction does also produce silicate-sulfate-hydrates that incorporate the soluble heavy ions present in the jarosite residue (Chen and Dutrizac 1996, Seyer et al. 2001).

Under the scanning electron microscope the general aspect of Jarofix is that of an open skeletal, largely isotropic, very porous microstructure. The microstructure main elements are sand and silt sized gypsum particles and silt sized sub-rounded aggregates composed of partially reacted jarosite crystals (Fig. 1, Arroyo et al. 2006)

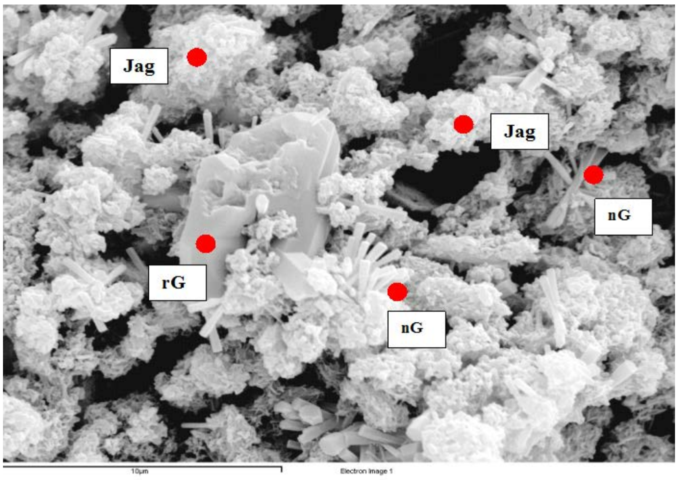

Fig. 1. SEM of a Jarofix sample. Dots indicate representative reacted Jarosite aggregates (Jag), residual Gypsum (rG) and newly formed Gypsum (nG). Graphic scale 10 µm.

2.2 *Site conditions*

Within the development plan of a large zinc smelter a large Jarofix landfill was projected at the site of an old quarry, about 600 m x 250 m in plan, located next to a harbour. Landfill stability assesment required a detailed study of Jarofix geotechnical properties.

At the deposit here sampled the mixture was left to set during 18 to 24 h before road transport to the disposal area. Once at the disposal area it was simply unloaded and extended using bulldozers.The quarry floor and walls were impermeabilized with a geomembrane before the material was landfilled.

Close to its main entrance the quarry included an area, 200 by 100 m in plan, that had been excavated up to 12 m below the general quarry base level. That area was where the landfilling operation started and where the site investigation took place. It is noted that the site was being continuously landfilled and there were up to 6 m difference in elevation between the different site investigation points. All the probes were fully within the Jarofix deposit and therefore all the “in situ” data refer only to this material.

2.3 *Geotechnical site investigation*

A site investigation campaign including DMT (5) and CPTu (5) soundings. The DMT and CPTu soundings were performed in pairs. In four of these locations results from FVT are also available.

A variety of samples were also recovered for laboratory testing, including 8 large blocks which were tested in several laboratories (Arroyo 2003, Ripamonti & Sala 2005). Some results from the laboratory campaigns that are relevant for the DMT interpretation are summarized in Table 1. Jarofix large specific weight is in agreement with its mineralogy. The large void ratio and uniform silt size are in agreement with what is suggested by microscopic inspection.

Table 1. Summary of laboratory results.

Parameter	Unit	Value
Specific Weight	-	3.3
Liquid Limit	%	60-90
IP		4-25
D50	µm	9
CU	-	5
Void ratio		2.5
Unit weight		1.65
Oedometric yield	kPa	375

3 IN SITU TEST RESULTS

3.1 *Overview*

An example of DMT profile (DMT2), in terms of material index I_D, constrained modulus M and horizontal stress index K_D (Marchetti 1980), from the deposit is illustrated in Fig. 2. The parallel CPTu profile, by means of corrected cone resistance q_t, sleeve friction f_s and pore pressure u_2, is illustrated in Fig. 3. Despite the industrial origin of the material the probes reveal significant mesoscale inhomogeneity in the deposit. The material investigated appears layered, with stronger and stiffer levels alternating with softer ones. Layering is also apparent on the CPTu pore pressure profile with small draining and/or dilative episodes punctuating a general trend of positive values.

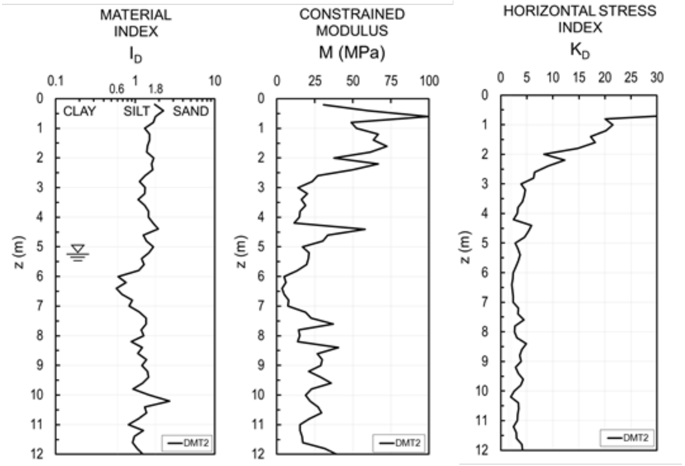

Fig. 2 Example DMT profile from the Jarofix deposit.

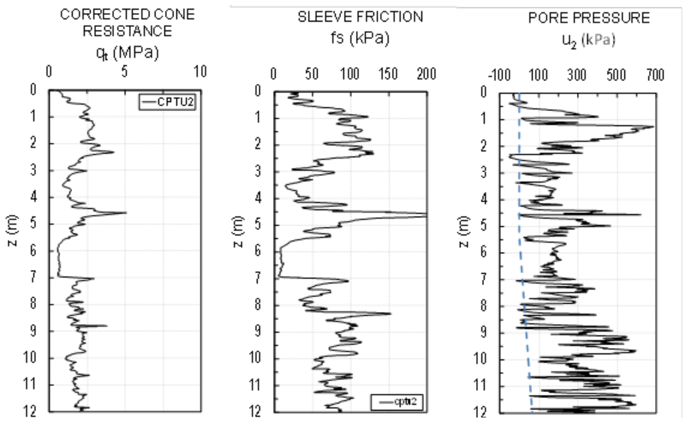

Fig. 3. Example CPTu profile from the Jarofix deposit.

The most likely origin of this meso-structure in the deposit is the irregular filling process, in which deposition and extension was not homogenous. Some of this profile features are similar to those observed in fine tailing deposits (e.g. Salehian & Kalinski 2014). Fine tailings, however, are not mixed with cement and they segregate during hydraulic driven deposition. In this case a possible interpretation of the observed profile variability is that either because of being less mechanically reworked during filling or because they were exposed to the atmosphere for a longer period, some layers seem to have cemented strongly than others.

3.2 *Soil type*

I_D profiles from all the 5 DMT soundings available are presented in Fig. 4. An average soil behavior type (SBT) profile is presented in Fig. 5 (Robertson 2010).

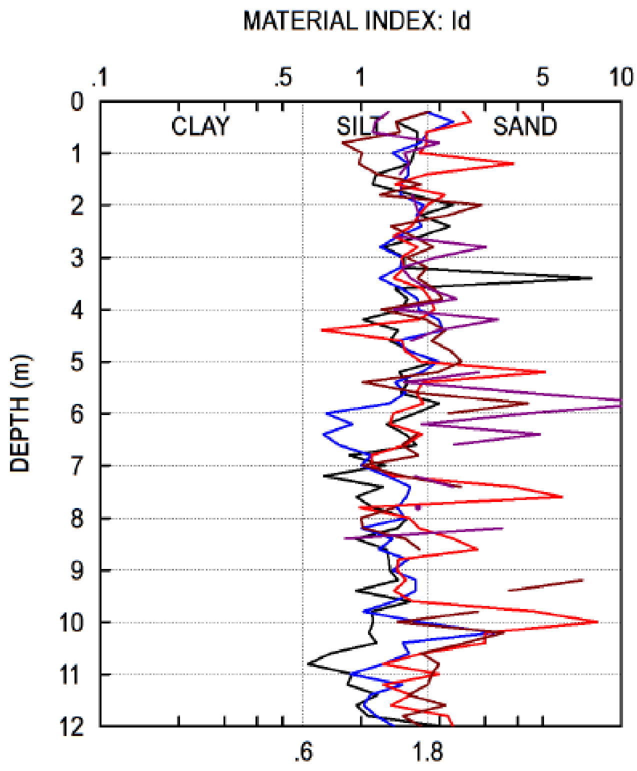

Fig. 4. Material index (I_D) of the Jarofix deposit.

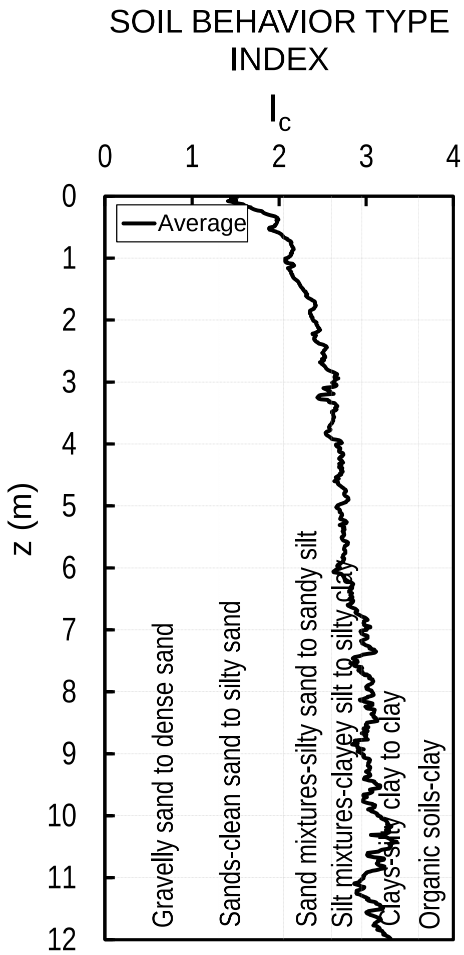

Fig. 5. SBT index (I_C) average profile data from CPTu the Jarofix deposit.

Both kinds of profile indicate a deposit that is finer in depth that close to the surface However, to the DMT the Jarofix deposit appears as silt with frequent spikes of sand. To the CPTu it appears somewhat finer, with most data plotting on zones 4 (Silt mixtures) and 3 (Clays) of the soil classification graphs.

One possible explanation for this difference is that CPTu results will be more directly affected by crushability than DMT. It is interesting to note, in this respect, that some laboratory results indicated the presence of a sandy fraction (20-30%) that was sensitive to the sample preparation procedure (e.g. disappearing when the sample was thoroughly crushed with the rubber hammer). A similar effect of grain size evolution was observed if the material was compacted. This is coherent with what will be expected of a lightly cemented material.

3.3 *Unit weight*

Marchetti & Crapps (1981) proposed an empirical correlation to estimater unit weight from DMT results. Similar efforts but using the CPTu were later made by Mayne et al. (2010) and Robertson (2010). The results obtained for the DMT1-CPTu1 at the Jarofix deposit are presented in Fig. 6, alongside the mean value obtained from the laboratory measurements. Table 2 summarizes the results for all the 5 probes available. All the correlations overestimate the unit weight. The Mayne et al. (2010) correlation is slightly more accurate but less precise than the Marchetti & Crapps (1981) correlation. The Robertson (2010) correlation does not perform as well as the other two.

Table 2. Summary of laboratory results.

Correlation		Normalized probe mean	Normalized standard error
		%	%
Marchetti-Crapps (1981)	DMT	105	7
Mayne (2010)	CPTu	104	9
Robertson (2010)	CPTu	109	11

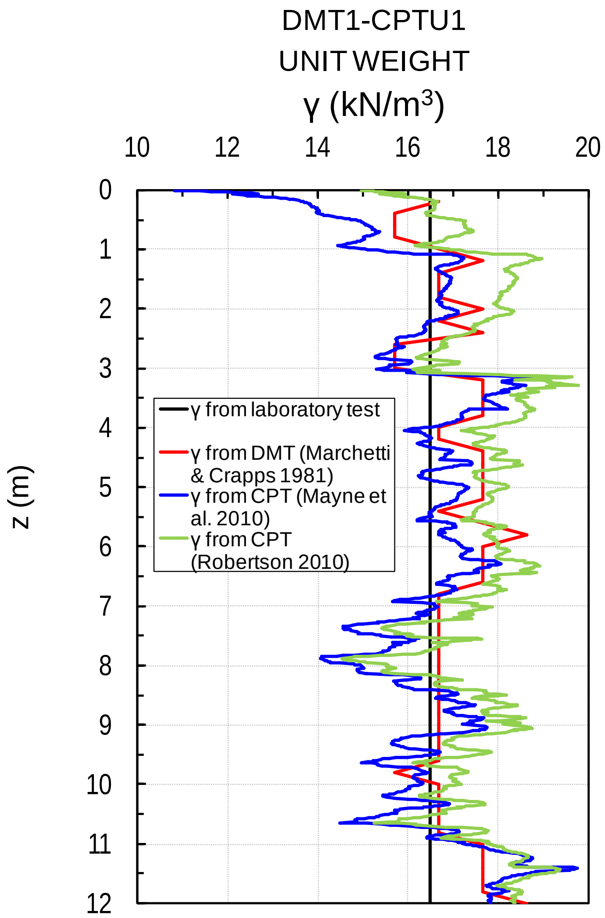

Fig. 6. Profiles of soil unit weight deduced from empirical DMT and CPTu based relationships for one probe on the Jarofix deposit.

3.4 *Overconsolidation*

As stated before, the Jarofix deposit was built without systematic compaction and the stress history was then that of a normally consolidated deposit. However, it is well known that cementation either natural or artificial endows soils with an apparent overconsolidation (i.e. an ability to sustain larger stress at a given void ratio than what is possible for the same material without cement).

3.4.1 *Laboratory results*

Block samples, approximately cubical, 30 cm on side, were recovered on open air excavations reaching depths varying between 1.5 and 3.0 m depth. Both incremental and continuous rate of strain oedometers were performed on different specimens retrieved from the samples. Typically yielding was not abrupt but rather gradual (Fig. 7). The average effective vertical stress at yield is 375 kPa, with a coefficient of variation $CoV \approx 0.2$, and no clear influence of sample depth or type. Most lab tests fall within a 280-450 kPa range.

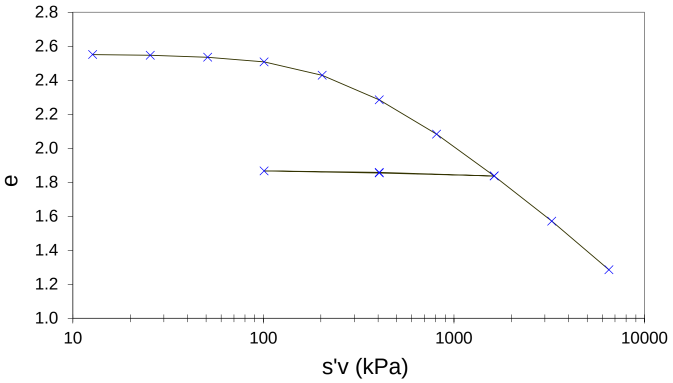

Fig. 7. Incremental loading oedometer on Jarofix.

3.4.2 *CPTu based estimates*

Demers & Leroueil (2003) reviewed CPTu based correlations for the sensitive structured clays of Eastern Canada. Such materials bear some resemblance to Jarofix in that they are fine and show some bonding (of natural origin). Their study concluded that the stronger and less biased correlations were those with net tip resistance, of the form:

$$N_{\sigma t} = \frac{q_t - \sigma_{v0}}{\sigma'_p} \tag{1}$$

For the Canadian clays Demers & Leroueil (2003) found a overconsolidation CPTu number $N_{\sigma t}$ = 3.4, which was well in line with previous results for silicate soils. The average value of this parameter for the Jarofix deposit is shown in Fig. 8. The estimated interval is 5-8 in the upper half (roughly where SBT < 2.5), reducing somewhat in the lower half of the deposit.

3.4.3 *DMT on cemented soils*

Marchetti (1980) proposed specific relations, confirmed later by other Authors (Finno 1993, Kamei & Iwasaki 1995, Yu 2004), to estimate K_0 and *OCR* in clays from K_D. Specifically:

$$OCR_{DMT} = (0.5\, K_D)^{1.56} \tag{2}$$

It was then emphasized that the relations proposed were intended for uncemented soils only. However, it was also stated there that the tell-tale of cementation was an anomalous K_D profile: the profile of this parameter in the Jarofix deposit (Fig. 2) is very similar to the "simple unloading" overconsolidated profiles that Marchetti identified. Later, Marchetti (1997) introduced the idea of "extended" *OCR*, noting that K_D was a combined result of *OCR* ("stricto sensu" i.e. due to stress history) and cementation. That combined value, identified by oedometric yield in the laboratory, could be predicted using OCR_{DMT}.

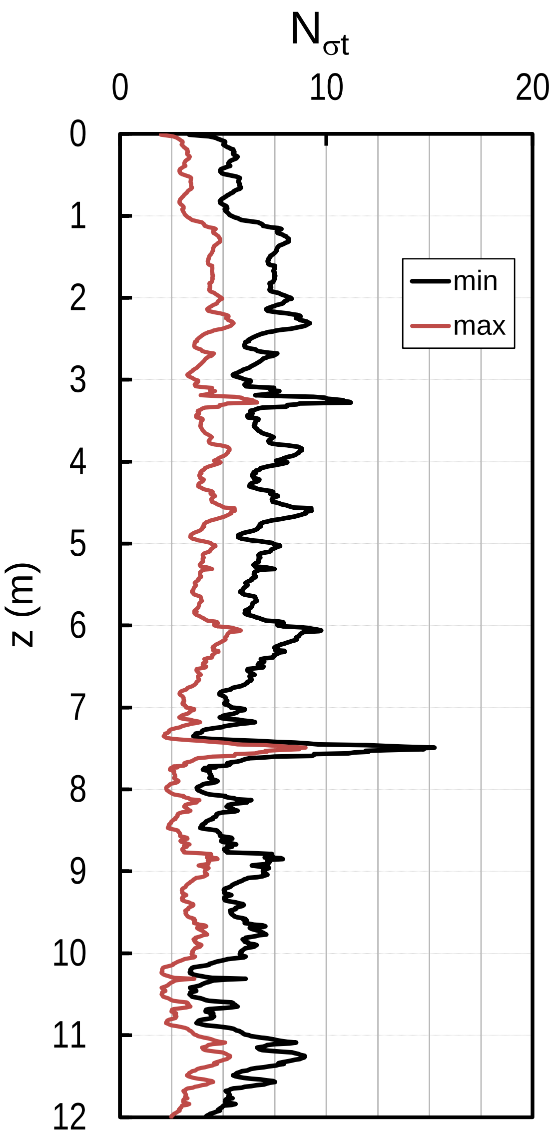

Fig. 8. Average profile of overconsolidation CPTu number $N_{\sigma t}$ in the Jarofix deposit.

The average prediction for the Jarofix deposit data is shown in Fig. 9, as well as the range that will be deduced from laboratory tests results. At the sampling depths (1.5-3.0 m) there is good coincidence. At higher depths the DMT indicates smaller yield stress than what would have be observed if all the deposit maintained the same level of bonding as the surface.

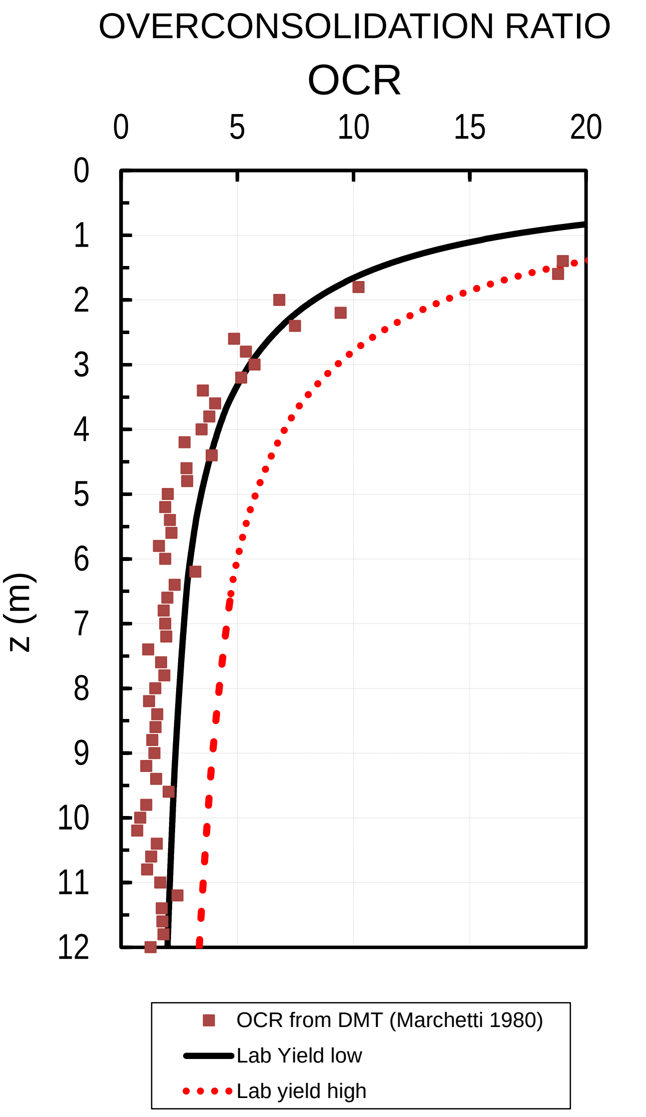

Fig. 9. *OCR* prediction from the original Marchetti (1980) relation and bracket profiles from laboratory measurements. Average profiles for Jarofix deposit.

3.4.4 *DMT-CPTu based relation*

Recently Monaco et al. (2014) introduced a possible estimation of *OCR* in sand combing DMT and CPTU data, by means of the ratio M/q_t (Eq. 3):

$$OCR = 0.0344(M/q_t)^2 - 0.4174M/q_t + 2.2914 \quad (3)$$

In addition Monaco et al. (2014) proposed a tentative correlation of *OCR* in sand also considering only DMT data, in terms of K_D (Eq. 4):

$$OCR = -0.0135K_D{}^2 + 0.4959K_D - 0.0359 \quad (4)$$

Although the material index I_D of the Jarofix deposits indicates silty sands and sandy silts, while Eq. (3) and Eq. (4) referred to sand layer having I_D > 1.8, Monaco et al. (2014) formulations were applied to the cement-stabilized metallurgical residue since this material is a landfill industrially produced.

Fig. 10 compares the regression curve M/q_t vs K_D, obtained from Monaco et al. (2014) DMT-CPTu dataset, with Jarofix M/q_t - K_D coupling, considering all the DMT1 and CPTu1 test results. The agreement is quite consistent for low values (i.e. $M/q_t \leq 10$ and $K_D \leq 5$). *OCR* prediction from the original Marchetti (1980) relation were matched with OCR prediction from Eq. (3) and Eq. (4) for Jarofix deposit (DMT1- CPTu1), as shown in Fig. 11. The combination of DMT and CPTu results (Eq. 3) provided a *OCR* profile similar to the one estimated by Marchetti (1980). Instead at shallow depths the tentative correlation *OCR*-K_D from Monaco et al. (2014) did not fit properly with the OCR profile from Marchetti (1980).

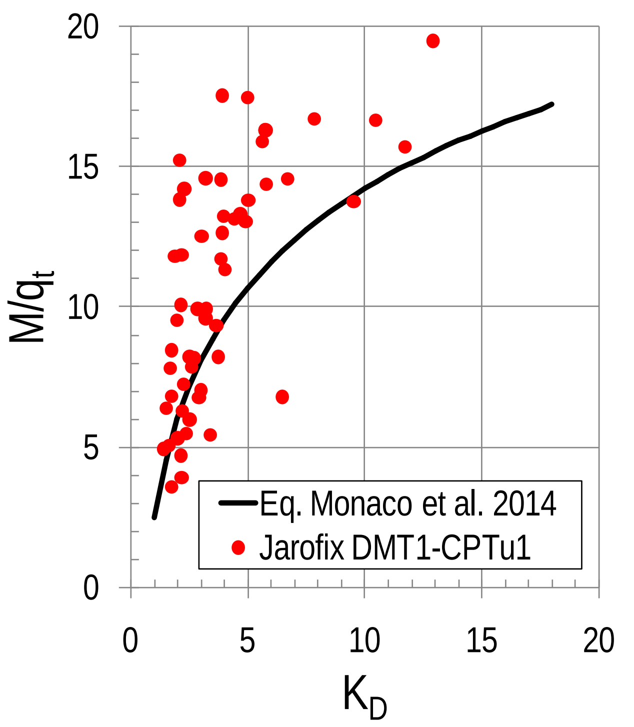

Fig. 10 Regression curve M/q_t vs K_D (Monaco et al. 2014) compared with Jarofix M/q_t - K_D coupling (DMT1-CPTu1).

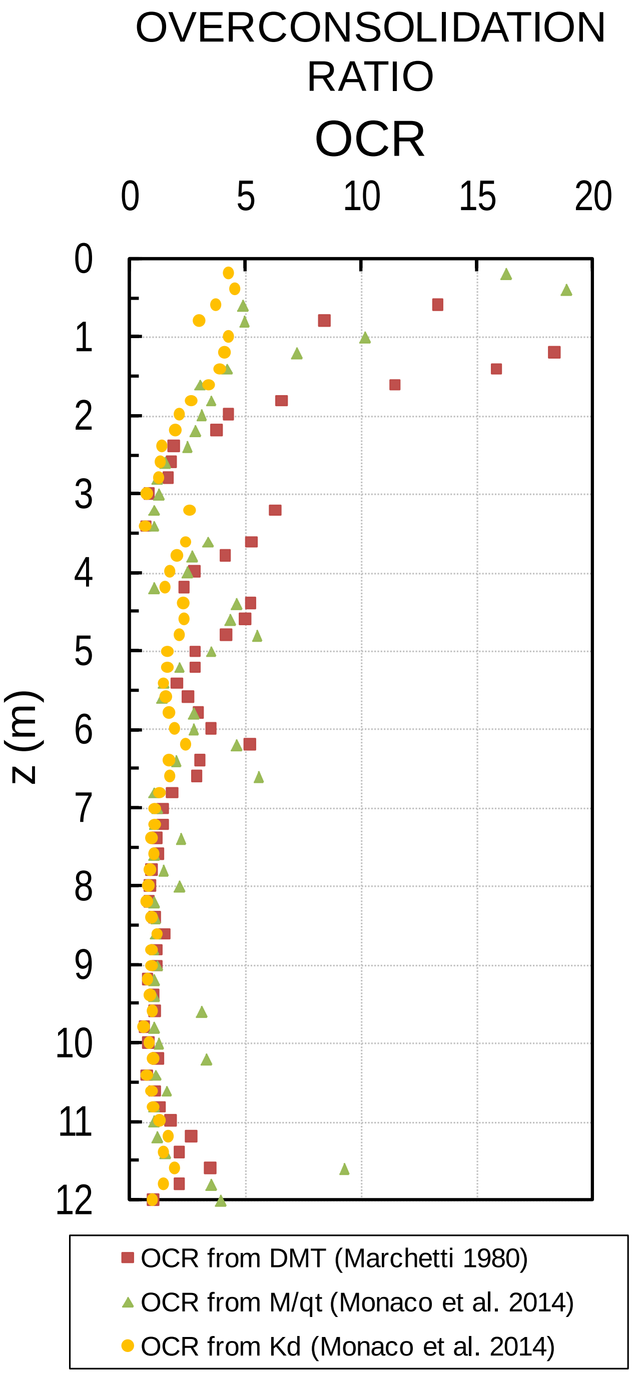

Fig. 11 OCR prediction from the original Marchetti (1980) relation and Monaco et al. (2014) correlation for Jarofix deposit (DMT1- CPTu1).

3.5 *Lateral earth pressure K_0*

Marchetti (1980) postulated the following relation between K_D and K_0 for uncemented soils

$$K_0 = (\frac{K_D}{1.5})^{0.47} - 0.6 \quad (3)$$

For structured clays Mayne & Martin (1998) reported that the previous equation overpredicted K_0. They also argued that a more general relationship would have the form:

$$K_0 = (\frac{K_D}{\theta_1})^{\theta_2} \quad (3)$$

Where, even for uncemented soils, the coefficients θ_1 and θ_2 are dependent on rigidity index and friction.

Several oedometers with measurement of lateral confinement were performed on specimens retrieved from block samples on the Jarofix deposit. The values obtained on loading paths for the range of effective stress present in the deposit are shown in Fig. 12, alongside the DMT-based average prediction.

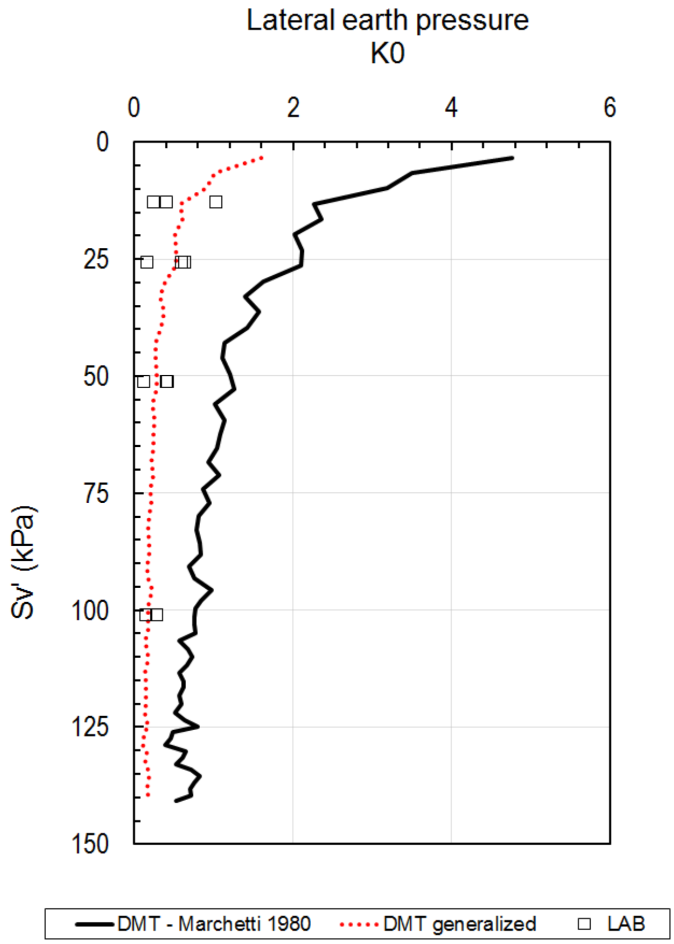

Fig. 12. K_0 prediction from the original Marchetti (1980) relation and Mayne & Martin (1998) generalized expression for Jarofix deposit (average all DMT).

It appears that the original relation does indeed overestimate K_0 in this cemented material. The adjusted generalized relation has parameters θ_1 = 30 and θ_2 = 0.75.

4 CONCLUSIONS

In this paper we have explored the performance of several classic DMT based correlations in a geotechnical context that is very different from the one in which they were originally established. Jarofix is a cemented soil with no silicates in its composition. Several CPTu based correlations have been also used for reference. The main conclusions obtained are:

1. CPTu classifies ("senses") this soil as somewhat finer than DMT. It is speculated that such difference is related to the presence of highly crushable intergranular bonds.
2. The DMT classical estimate of soil unit weight overpredicted unit weight by 5% on average, being less volatile than CPTu-based estimates.
3. The DMT classical K_D based estimate of OCR for clays is within the range of apparent oedometric yield of the Jarofix deposit close to the surface, but below it at depth. The latter discrepancy can be a consequence of "in situ" destructuration. Adding CPTu information to the correlation does not improve the fit.
4. The DMT classical K_0 estimate for clays is way above the values that are obtained from laboratory testing on Jarofix samples.

It is relatively surprising that some of the correlations explored perform correctly in a material that lies so far from its mineralogic origins. Further work needs to be done to evaluate and contrast DMT predictions of stiffness and strength in Jarofix. In the meantime, these results may perhaps be taken as an indication that the control that mineralogy exerts on soil mechanical behavior is less important than that of other physical features like grain size or shape.

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