EVALUATION OF THE STRENGTHENING EFFECT OF CONSOLIDANTS APPLIED ON POROUS AND FISSURED SUBSTRATES

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Abstract

The evaluation of the efficacy of stone consolidant treatments is a very important step in the selection process of the product to be used in a real situation. The procedure to select the appropriate product should be a step-by-step program. Sonic measurements and drilling resistance are currently used as non- or micro-destructive methods to evaluate the cohesion increase promoted by the treatment but they can also be used in each step of the evaluation program.

Based on authors' experience drilling resistance technique is a very useful tool for porous stone like limestone and soft sandstones. On fissured rocks, ultrasonic methods are considered very sensitive, allowing for the comparison of treatments, especially in laboratory conditions.

Keywords: consolidation, drilling test, ultrasonic pulse velocity, fissured and porous materials

1. Introduction

The evaluation of effectiveness is a decisive step when selecting the products to be used in stone consolidation. The assessment of the mechanical resistance of the consolidated stone is a key-step in this process.

To evaluate the mechanical resistance increase due to mass consolidation destructive methods, like compressive and bending strength, are usually considered as the most reliable tests.

Looking at literature, quite often the evaluation of consolidants is expressed by the mechanical resistance increase. An illustrative example of data in included in Table 1. These data were obtained in granite and limestone after ethyl silicate consolidation. It is worth mention that it is very important to complement this information with the quantity of the product responsible for consolidation action.

Ançã limestone	Before consolidation	After consolidation
Open porosity (%)	27.1	24.0
Ultrasound pulse velocity (m.s^{-1})	3120 - 3280	3590 - 3750
Compressive strength (MPa)	33.1 - 39.7	54.7 - 62.3
Bending strength (MPa)	4.8 - 5.6	9.5 - 11.7

Product applied (g/100g)	Dry matter * (%)
12.4 - 13.4	4.5

Évora granite

Open porosity (%)	1.9 - 2.1	1.8 - 2.0
Ultrasound pulse velocity ($m.s^{-1}$)	2460 - 2580	3440 - 3580
Compressive strength (MPa)	nd	nd
Bending strength (MPa)	7.3 - 7.7	8.3 - 11.9

Product applied (g/100g)	Dry matter (%) *
0.4	0.1- 0.3

*calculated by the amount of the product retained after curing/ weight of the specimen before treatment*100

Table 1. Some examples of data with relevant parameters to evaluate strengthening effect of ethyl silicate on two substrates: Ançã limestone (*data from Ferreira Pinto 2002, 2012)* and granite of Évora *(data from Costa, 2007)*

Looking at these results, it can be concluded that strengthening effect of ethyl silicate was effective in both lithotypes. A higher amount of product was retained inside the network of the porous limestone: the original mechanical resistance was doubled while the ultrasonic velocity was slightly increased. On the contrary, a very low amount of consolidant had also an effective consolidation effect on granite and it can be quantified by both methods used to characterize this type of material.

Although this information give a global idea of the performance of the consolidants when applied in these very different substrates, other aspects must be considered relevant not only to complement the evaluation of the efficacy but also to evaluate the potential harmfulness when used in practice.

It is also relevant to say that these type of data can only be clearly understood when a fully impregnation of the specimens can be guaranteed. The incomplete impregnation or accumulations of the product near the surface create very heterogeneous specimens that end up giving nonrealistic results. Even when non-destructive methods are also used as complement, it is quite difficult to be sure that strength increased was properly assessed using these tests. For all these reasons, it is very important to have adicional information when the objective is to evaluate strengthening action.

2. The penetration depth: a relevant parameter to characterize the consolidation action

Besides strength increase *per se,* the **penetration depth** of the consolidant, a measure of the capability of the product to go deep and travel inside the system of voids, has been pointed out as key-parameter in stone consolidation since long time ago.

Schaffer (1933), in the emblematic text about weathering of natural building stones reports the failure of stone preservatives observed in practice and considers that “a common cause of failure is that, even in porous materials …, the preservative penetrates only to relatively small depth and a surface skin is formed which differs in physical properties from the underlying material”. Later, in a different context, when presenting the behavior of one Wacker product available at that time, Bosch (1972) considers that “decisive for the quality of the stone strengthener is the depth of penetration”. In his paper, elucidative pictures of specimens after consolidation were also presented to prove the high penetration depth of the product when it was applied to a German sandstone.

However, the methodology to assess this important parameter is still in debate and probably it should not be considered as universal but dependent on the type of substrate.

The direct observation of presence of the product at a certain depth can be done using the scanning electronic microscopy (SEM); besides the morphology of the product after polymerization, this approach is qualitative and the success is clearly dependent on the experience of the observer.

The indirect detection of presence of the consolidant allows the quantification of the effect produced by it; water properties (in liquid or vapour phase) are usual changed due to impregnation and they can be used to evaluate the depth of penetration of the product. Even in small amounts, the consolidants can promote effects measurable by these type of changes in granites (Delgado Rodrigues, 1996).

Our preference goes to a more direct" evaluation, which includes the evaluation of properties related with mechanical resistances increase as it is the aim of the treatment.

Immediately after treatment, specimens frequently show variations in colour induced by the consolidation product. Although these variations may coincide with the consolidated depth, this is not always the case, and very often this coloured front may be higher or even substantially higher than the depth effectively reached by the consolidation action.

In our lab, ultrasound pulse velocity (UPV) has been used for this purpose and more recently a new method was introduced to evaluate resistance in depth with accurate micro-drilling profiles. From our experience, sonic measurements and drilling resistance can be both used for this purpose, but their performance depend on the type of substrate and therefore a selection of the most appropriate tool has to be considered.

Ultrasound test is a real nondestructive measurement, while drilling is a micro-destructive method. While both allow the evaluation of the cohesion increase promoted by the treatment they do it in different ways, which implies that the options for one or the other may not be innocuous.

Both are suitable to characterize materials, but they are not equivalent, and frequently they can be considered as complementary. Both show advantages and drawbacks that will be briefly addressed in this paper.

3. The step by step approach to evaluate the consolidation action

The choice of a product to consolidate stone elements is a key step in cultural heritage conservation, but it is far from being perceived as well defined procedure. Very often, the process is a long time consuming, frequently following approaches with little incidence in the key aspects of the problem and leading to decisions that hardly can be taken as evident demonstrations.

In a recent publication, Tabasso & Simon (2006) present an interesting review on the literature about testing products for stone conservation. A global evaluation of consolidation treatments was proposed by Sasse & Snethlage (1997); besides the exhaustive listing of tests that should be used, the penetration depth is included and can be evaluated by capillary soaking for 5 minutes. The proposed methodology includes the evaluation of biaxial flexural strength, the modulus of elasticity and even drilling hardness to be evaluated in profiles. The practice had demonstrated that it not easy to perform the two first tests in profiles but the criteria indicated are very relevant for a good performance of consolidant treatments due to the fact that they recommend to avoid strong rigid interfaces between treated and non- treated zones.

Ideally, the samples to be tested in this type of studies should be in a weathered condition, as similar as possible to those present in the object to be treated. Such samples would adequately represent the real situations, but it is not evident that all testing protocols would necessarily replicate the onsite treatment and ageing conditions, and consequently that the observed lab behaviors would fully represent the onsite performance. From our experience, the amount of samples available for this purpose is always a serious limitation in real situations, and therefore it has been considered as preferable to split the study in elementary steps, adopting the protocols that are deemed as more adequate to find answers for that step, and building up an overall rational evaluation process. At each step, the results obtained should determine the progression in the program or the return to a preceding level, trying to find better conditions of application to improve the product performance. For limestones, a useful and detailed chart was already proposed as the result of an intensive program of laboratory and onsite tests (Ferreira Pinto, 2002). Figure 1 presents the general layout of the process. In some cases, Phase III and IV can be evaluated simultaneously, reducing the lapsed time in the evaluation process.

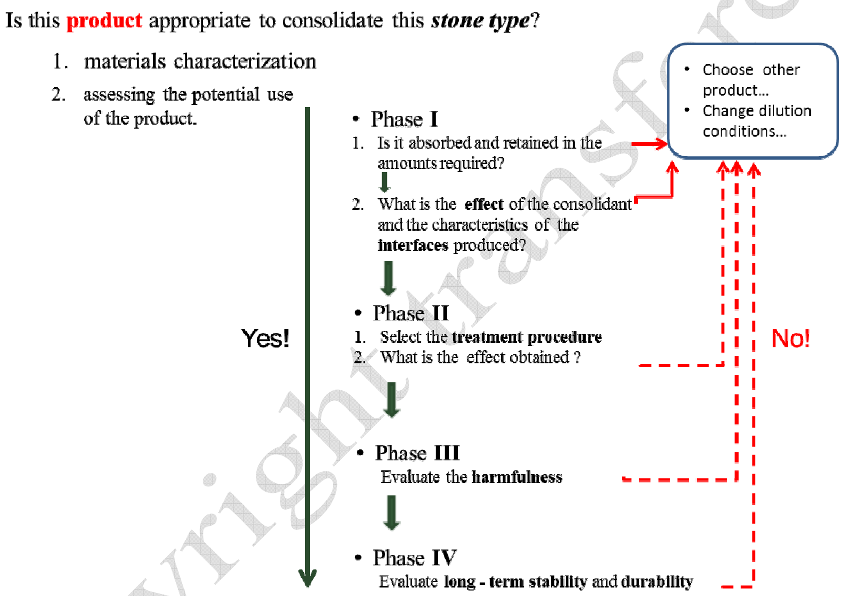


Figure 1. Simplified flow-chart to evaluate the potential use of a product as consolidant (Adapted from Ferreira Pinto 2002)

One important aspect to be noticed is the fact that a decision to eliminate a product under scrutiny can take place since the very first step, thus avoiding to spend resources in its testing in subsequent stages. The first question to be answered is “Is the product absorbed by the stone and retained in its interior?” This question may show some difficulties to be fully answered, but when negative answers are obtained, there is no justification to continue the study of that product.

The evaluation of strength is required in the four phases here considered, but it is particularly important in phase I where the use of nondestructive methods to answer the relevant questions is strongly recommend.

The impregnation capability of the product and the consolidation effect that results from it are the key parameters to be characterized in this first phase. The object of this paper is to highlight how this phase can be solved in fissured rocks like granites and in porous stones like limestones with the two testing methods under discussion.

To assess the impregnation capability our experience recommends to apply the product by direct contact capillarity and to follow the evolution (curing process) until all the solvent has been released. Dry mass after consolidation and the quantity of the product absorbed during treatment shall be computed. In some cases, it is also useful to compute the advancing "velocity" of the wet front, especially to be able to transfer data from lab application to onsite treatment specifications. Ethyl silicate absorption curves obtained on two varieties of granites (AI is a 2.4% and FI a 1.1% water porosity) clear demonstrate the influence of stone properties on the absorption characteristics of the product when in contact with them (Figure 2). When possible, these data should be complemented by the progression of the wavefront determined by naked eye.

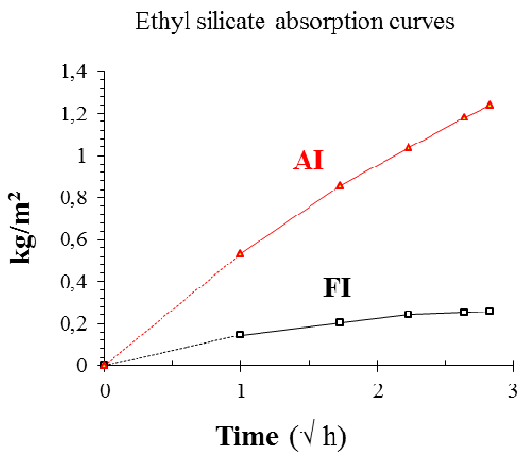


Figure 2. Ethyl silicate absorption curves on two types of granites

4. Testing consolidants on different substrates

For the characterization of the consolidation effect two methods have been used, depending on the characteristics of the stone type we are testing. The *type of voids* and the *hardness/abrasiveness* are the aspects to be considered when the testing method is to be selected.

It is known that P waves propagate differently in materials with pores or fissures. In practice, it is possible to evaluate the type of voids using ultrasound velocity, in dry and wet conditions. The works by Tourenq & Fourmaintraux (1971), and by Delgado Rodrigues (1983) give the necessary hints on how to identify the type of voids present.

Generally speaking, it can be said that spherical voids (pores) are present in most sandstones and limestones. On the order hand, sheet like voids are present in almost all metamorphic and igneous rocks, generally called crystalline materials, including silicate rocks and marbles.

For the selection of the method, the hardness/abrasiveness has also to be considered. This fact is especially relevant for the micro-drilling methods since a fast rate of abrasiveness may jeopardise the excellent performance they show when testing non-hard, and non- and low-abrasiveness materials. Following this concept, as illustrated in Figure 3, studies involving limestones should consider the use of micro-drilling techniques to evaluate consolidation effect, while in fissured materials the ultrasound pulse velocity may provide excellent results, particularly in laboratory studies when testing conditions can be tailored to get more precise measurements.

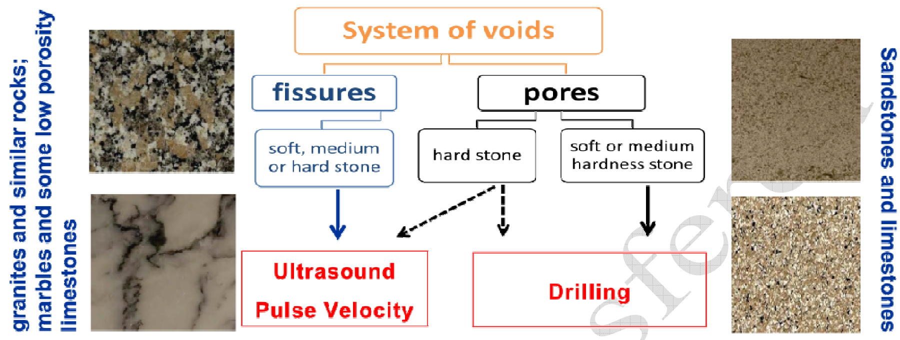
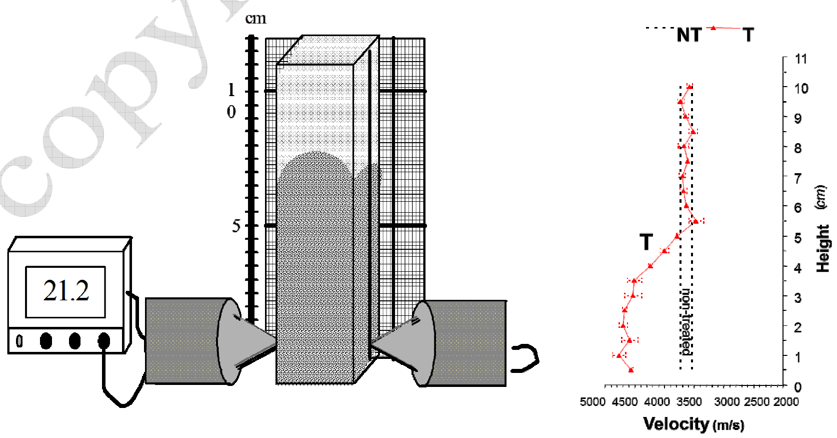
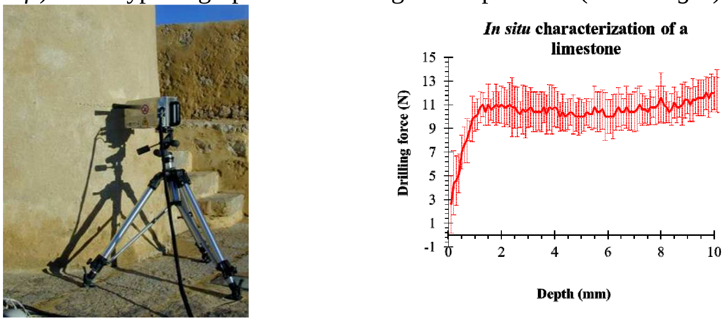


Figure 3. Criteria to select the method to characterise the increase of cohesion after consolidation in fissured and porous stones.

Figure 4 illustrates the equipment and methods considered here. The evaluation of penetration depth and mechanical resistance increase due to consolidation can be made in laboratory conditions using UPV with the set up shown in Figure 4a; it is a comparative measurement, to be done before and after consolidation and it is particularly useful in crystalline materials. For limestones, drilling resistance is recommended, since it can be considered as the best option to evaluate the resistance of the material in depth in laboratory as well as in the field, as exemplified in Figure 4b. Both methods require some training and both show advantages and drawbacks that need to be taken into account when interpreting the obtained results.



a) Ultrasound pulse velocity profiling (direct or transparency measurements) (*on the* *left*) and a typical graph in a treated granite specimen (*on the right*) .



b) drilling resistance equipment and typical graph of drilling resistance in depth.

Figure 4. Evaluation of the characteristics of the material in depth in fissured stones (a) and in porous, non- or low-abrasive materials (b).

The Drilling Resistance Measuring System (DRMS) (Tiano et al. 2000) is a power drill with constant rotation speed and advancing rate equipped with a load transducer that measures the force as a function of the drilling depth. During the test a hole with 5 mm (other sizes are possible) is produced and the results (force and depth) are registered by the system; the output is a graph similar to Figure 4b, in this case obtained in a soft and very homogeneous stone.

The drilling resistance quantifies the consolidation effect of the treatment and identifies the impregnation depth, two relevant parameters on the evaluation of stone consolidants.

4.1. UPV profiling to evaluate the consolidation action

This method is particularly useful when the substrate is of the fissured type and the evaluation is performed in laboratory conditions.

The test is typically a two-phase process. Several variables influence the results and for this reason the use of the same specimens before and after consolidation is considered a very important condition to eliminate the influence of heterogeneity inside the specimens and variability among specimens.

Profiles of ultrasound velocities in granite specimens partially consolidated could clearly identify the treatment boundary, in spite of their low porosity.

One typical example is reported in Figure 5 (a1); in this case, 0.16 kg/m^2 of ethyl silicate was applied by direct contact capillarity, allowing a 0.06 % of consolidant expressed as dry mass per total weight of the specimen. This is a low porosity granite (1.3 % porosity) whose fissures allow the uptake and migration of the low viscosity ethyl silicate. After curing, the original porosity of the material was slighted decreased and the sonic waves travelled at much faster velocity. The graph of the ultrasounds velocities identify clearly the boundary promoted by the treatment. The sharpness of the interface depends on the type of product and also on the application procedure.

As comparison we also present other example (a2) where the product was not able to penetrate into the system of voids and the graph clear represents this situation.

In some particular cases, UPV discriminating profiles can also be obtained on limestones, as exemplified in Figure 5 b), although the differences are systematically smaller than in granites. To be compared are the 400 m/s in difference in this limestone (with 28 % porosity), while it was in the order of 1200 m/s in the preceding example of granite (1.3 % porosity). To reach this result in the limestone specimen, a very high amount of product had to be applied (7.1 kg/m^2), corresponding to a retained 3.6 % of dry mass, which represents an extreme situation that could hardly be conceived to be applied in practical situations.

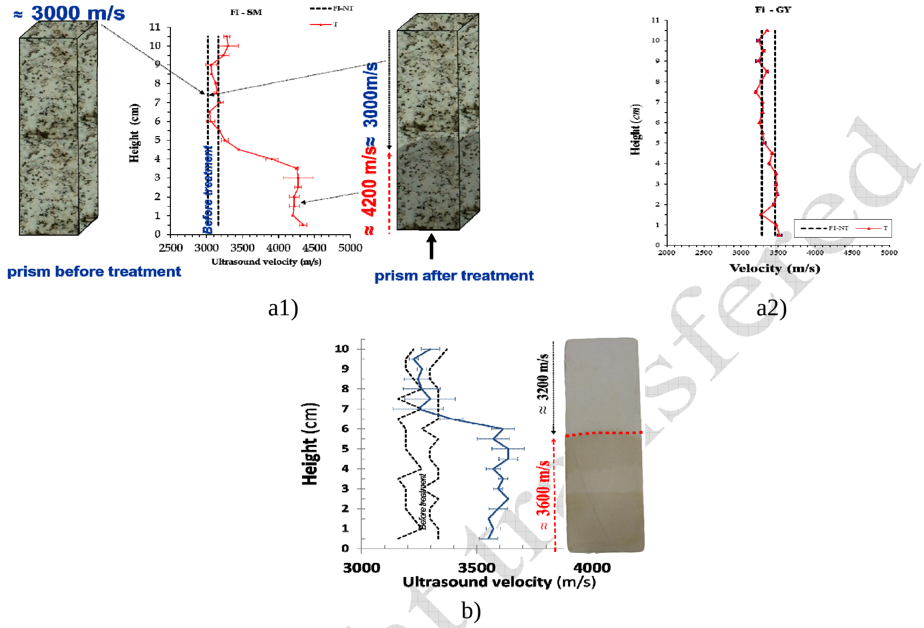


Figure 5. Consolidation effects characterised with ultrasound pulse velocity on granites (a1 and a2) and on limestone (b).

4.1 Drilling resistance on the evaluation of consolidation action

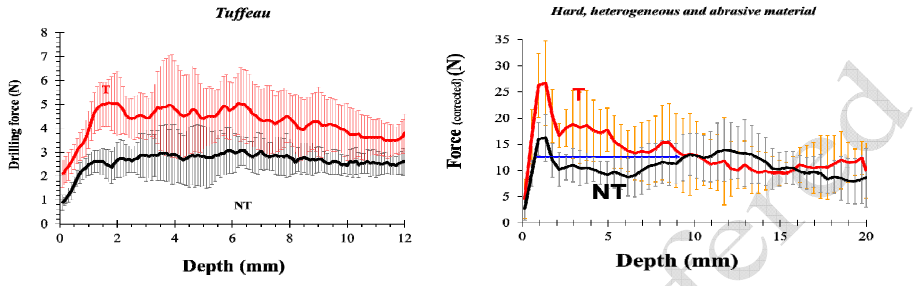
Drilling resistance allows evaluating the hardness in depth; it is possible to analyze both strength increase and the homogeneity of the product distribution.

It is a very sensitive method, although the results can only be compared when the drilling conditions used to perform the test are the same.

The method was used here to evaluate the consolidation action of ethyl silicate applied on a very soft stone (Tuffeau, with 49 % porosity) by capillarity. The high porosity specimen was fully impregnated and 160 kg/m^3 were absorbed. The result (Fig 6a) shows a full impregnated situation with a fairly homogeneous increase throughout the specimen. The sharp initial increase in resistance is an artifact due to the triangular shape of the drill bit tip.

In other materials, the use of this method may become more complex, not only to perform the test but also to interpret the results. When the material is hard, heterogeneous and abrasive it may result in a difficult task: it may be difficult to drill it, the resistance profiles may become very irregular and the absolute values only can be obtained after correction for the effect of the drill bit wear. Figure 6b) illustrates a situation of an abrasive glazed tile where an amount of 0.6 kg/m^2 of ethyl silicate was applied by brush, leading to a 1.7 % of dry mass as consolidation matter. The specimen is a 34.3 % porosity glazed tile and the penetration depth determined by drilling is 9 mm.

When dealing with abrasive materials some specific procedures must be used in order to compensate the wear effect (Delgado Rodrigues & Costa 2004). In some situations drill cuttings may find difficulty to escape and may accumulate inside producing an artificial increase in resistance. A hole-in-hole technique can be followed (Mimoso & Costa, 2006) and backwards vacuum suction can be used through the initial pilot hole when the test is performed in laboratory samples.



a) b)

Figure 6. Consolidation effects measured with DRMS on soft and homogeneous stones (on the left) and abrasive and heterogeneous materials (on the right).

5. Conclusions

Natural stone surfaces loose cohesion down to a certain depth due to decay; consolidation is needed to reestablish the cohesion between particles of deteriorated material and ideally the product needs to reach the non-decayed zone to avoid inconvenient sharp interfaces.

The procedure to select the appropriate product to be used is a laborious and complex process. In our experience we have been using a step-by-step program to avoid the execution of expensive and/or long lasting tests with products that can be discarded early in the selection procedure when assessment logic is set since the beginning of the process.

The comparative evaluation of the performance of products is an important step of this selection and it is better done when sensitive techniques are used to characterize the relevant parameters.

In the authors' experience, a specific approach based on the type of the porous system of the stone substrate to consolidate should be the starting point of the selection procedure.

Sonic velocities and drilling resistance are the parameters recommended for evaluating the strength increase due to mass consolidation. Sonic measurements are widely used since long in both lab and field studies and it has been used in all types of stones, but they are much more suitable on fissured substrates, like granites and marbles, than in porous materials, like limestones and sandstones.

Stone resistance measured with micro-drilling equipment is a very sensitive technique to evaluate the effectiveness of consolidation treatments on limestones or even in sandstones, in laboratory and in situ. The ratio of resistance gives an indication of the consolidation effect, and the consolidation depth is directly determined from the graphs.

Profiling with ultrasound values in fissured materials partially treated is able to identify the treatment boundary. Since the ultrasound values are a good estimate of the materials strength and deformability, the differences in ultrasound values can be also considered as a good estimate of the consolidation action. Specific calibration curves can be drawn when necessary

Summing up, the drilling resistance measurement is a very useful tool for laboratory and field studies when we have to deal with soft porous materials. On fissured rocks, ultrasound methods (UPV) are appropriate, especially in laboratory conditions.

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