Micro-drilling studies in azulejo consolidation

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SUMMARY: The consolidation treatment of azulejos is indispensable when there is a debilitated or disaggregated ceramic biscuit and/or one wishes to reestablish a weakened glaze-ceramic adherence.

In this communication the impregnation profiles obtained via mass consolidation on ceramics conservation are presented. An acrylic resin, an ethyl silicate and a nanolime were used to determine the micro-drilling relevance for the evaluation of consolidation treatments applied on ceramics.

This technique allowed to observe the impregnation patterns which cannot be determined by the mechanical strength tests (flexural and compression) commonly used to evaluate the consolidation effect.

KEY-WORDS: azulejo, consolidation, micro-drilling, profiles, hardness in depth

INTRODUCTION AND OBJECTIVES

The consolidation actions on glazed tiles are diverse and must meet the final objective which is defined according to the most relevant problems faced. The loss of glaze is a substantial aspect in the conservation of such materials but sometimes it is necessary to act on the ceramic body and provide for its consolidation. Salt laden walls can contaminate glazed tiles and, due to it, ceramic bodies can show powdering, crumbling, granular disintegration, scaling or flaking that justifies the need for a mass consolidation action, after desalination.

Mass consolidation requires the use of different solutions and products from those used as adhesives to fix the connection of the glaze to the ceramic body. When mass consolidation is required the consolidant must have different requirement and characteristics. Namely the ability to penetrate into the ceramic voids without creating rigid interfaces is usually considered a key-parameter. For this reason, it is important to evaluate the penetration depth of the product and its distribution after curing. The formation of sharp interfaces is considered a harmful effect because due to differential behaviour it is able to produce, in the short or in a medium term, splitting where the consolidated and non-consolidated substrates meet and consequently damage of the material may occur.

When mass consolidation is the objective, the increase of the cohesion of the material is expected after consolidation. Conventionally, this effect is measured by the increase of mechanical resistance and destructive methods are used for the purpose (such as the compressive or bending strength). However, very often, the consolidant has some difficulty to percolate inside the voids and surface accumulation can occur. Or, in other cases, the product can accumulate somewhere after curing and both features cannot be detected with conventional tests which are bulk analysis and not depth discriminant. Moreover, this differential consolidation can lead to an overestimation of the consolidation effect since an increase of the mechanical strength is measured and a potentially harmful effect may be wrongly interpreted as a beneficial one.

The development of a technique such as DRMS that measures the hardness in depth is considered particularly relevant for the assessment of bulk consolidation. Developed in the frame of stone conservation [1] it has been extensively used on other materials and for other purposes where hardness in depth is relevant, although some adequate adaptation is sometimes/usually necessary. In this particular case, the objective is to evaluate the penetration characteristics of some products when applied to ceramic bodies, as a first step evaluation of the consolidation action in these substrates. This method can also be used to characterize the resistance of the material itself, allowing the comparison among different materials [2].

In this paper, some results of micro-drilling tests performed to evaluate the penetration depth and the distribution of the consolidant in ceramics (similar to the biscuits of glazed tiles) are presented aiming to show the relevance of this method for a global evaluation of consolidation effect in this particular material.

PRODUCTS AND METHODS

For this program, the selection of the products to be tested was based on three criteria: vast use in practice; claimed high penetration depth; and some compatibility with the composition of the biscuit to be consolidated. The most commonly used material applied nowadays for consolidation is the acrylic resin Paraloid B72®. The ethyl silicate based products, acid silicate esters such as TEOS – tetraorthosilicate [3] also vastly applied, fulfils also the second requirement. Nanolime was also considered due to the fact that the pastes used to manufacture azulejos did have large contents in calcite and the high porosity of the material can overcome the limitation regarding the penetration capability recognized in this type of products, at least for some stone materials [4], [5]. Paraloid B72® was tested in 1: 3 and 5% (wt) dilution conditions, using acetone as solvent. Nanolime (CaLoSil E50®) diluted with ethyl alcohol (5g/l) and TEOS (Tegovakon V®) as ready-to-use product.

The products were applied by capillarity, method recommended for tests in laboratory conditions as it is considered to avoid the influence of the operator during the application by brush, at this level of evaluation.

The ceramic bodies selected are semi artisanal tile biscuits produced by New Terracota Lda. The physical characteristics of these materials are presented in Table I.

Table I – Open porosity, real and bulk density and mechanical resistance characteristics of ceramic bodies

Open porosity (%)	37,2
Maximum water content (%)	22,1
Real density ($kg.m^{-3}$)	2673
Bending strength (MPa)	16

Small pieces (65 x 30 x 9mm) were cut from larger square units (130 x130 x 9mm). A small area was coated with an epoxy resin aiming to control the contact area of the specimen with the liquid (see photos in Figures 3 to 5) during capillary absorption of the product.

The products were applied for 24 hours; from time to time, the process was interrupted to weigh the specimens. After curing for at least 20 days, the specimens were dried (at 70±1ºC) and tested.

THE USE OF DRMS ON CERAMICS

The Drilling Resistance Measuring System (DRMS) [1] is a power drill with constant feed and a force transducer that measures the thrust as a function of the drilling depth. During the test a hole with 5mm is produced and the results (force and depth) are registered by the system; the output are graphs similar to those in Figure 1 that compares soft and very homogeneous stone with homogeneous and heterogeneous old paving tiles. On ceramics, quite often intermediate hardness materials are heterogeneous and typical curves are in between b) and d) in figure 1. In this particular case, the ceramic can be considered rather homogeneous, similar to b).

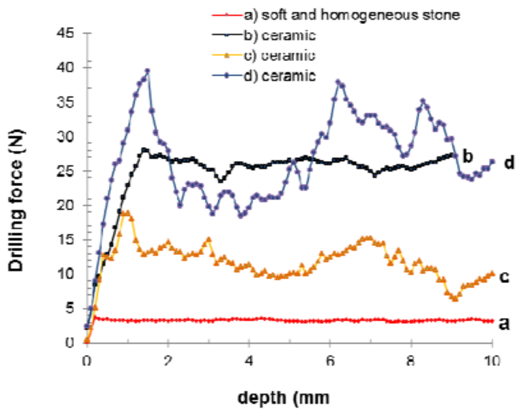
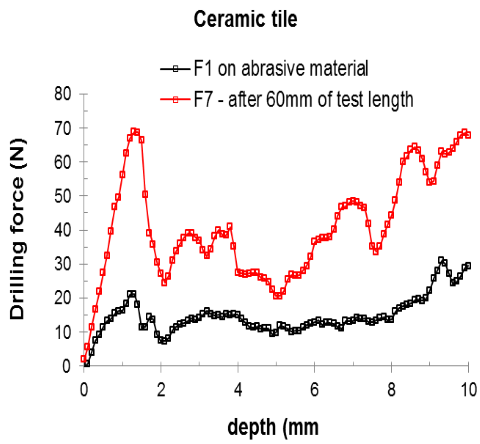


Figure 1 – Examples of drilling resistance curves obtained on materials: a) soft and very homogeneous stone; b) a homogeneous ceramic; c) and d) heterogeneous old paving tiles

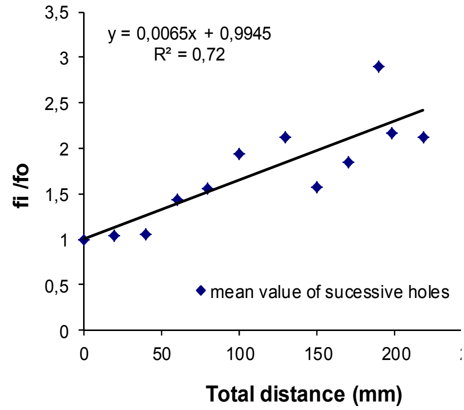
Besides stone characterization in depth, drilling resistance was the parameter used to quantify the consolidation effect promoted by the treatment and to identify the impregnation depth. Although the method is considered very useful, on ceramics or in similar materials where hard grains (usually quartz) are present, some drill bit wear is expected. This effect depends on the material, not only on the composition but also on the texture and indirectly on the porosity. For these reasons, it must be assumed that wear effect abrasiveness is not universal, must be evaluated and, if it is the case, corrected.

The abrasive effect on ceramics can be illustrated in Figure 2. In this case, two successive holes were performed on reference material (a similar ceramic) used to evaluate abrasive effect during drilling tests performed on ceramic bricks. The increase of the force (from 20N to 40N expressed in terms of mean values, as seen in a) is due to the drill bit wear effect but sometimes it has also some consequences in the range of the values. In the last 2mm, the increase of force can be the result of dust accumulation. This drawback can be solved by drilling over a previous pilot-hole of smaller diameter [6].

To account for the increase in force due to the abrasive effect, a calibration line (exemplified in Figure 2b) can be computed and the values corrected using a simple method [7].



a) effect of abrasivity on drilling test results measured on two successive holes performed on ceramic material



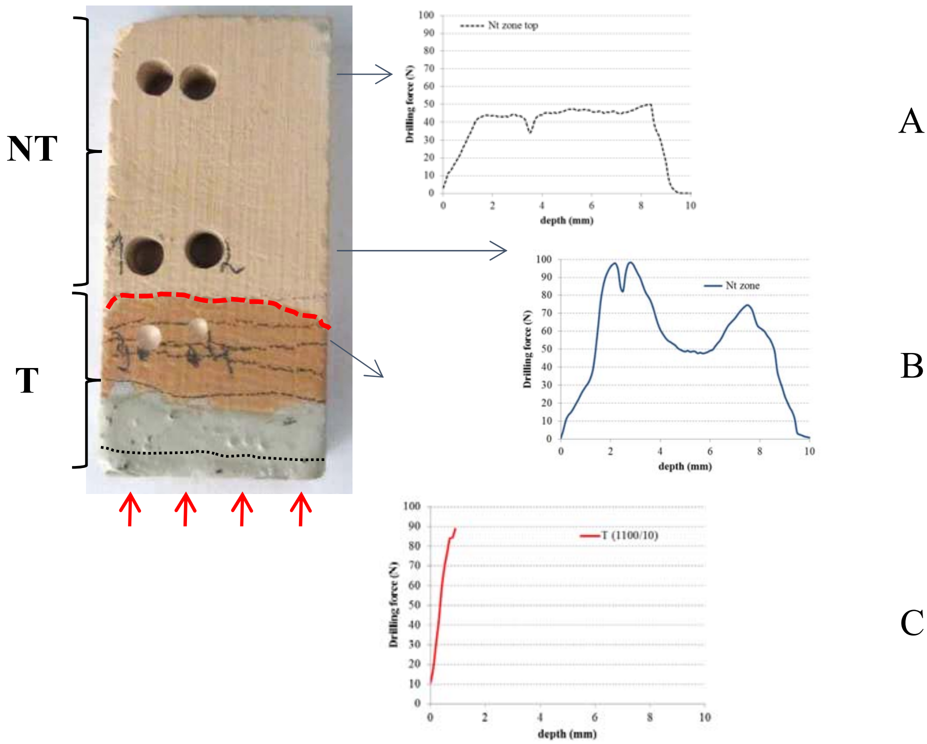
b) abrasive law determined for a ceramic sample; “fo“ and “fi - mean value of force at stage “0” and “i”, respectively

Figure 2 – Drilling resistance on abrasive ceramic materials

The past experience showed that even in “hard conditions” of testing the results can be useful and relevant [8]. The micro-drilling assessment of old ceramics can be highly complex, mainly due to the high heterogeneity and some hard spots encountered on the profiles of the materials and whose origins are not clear.

SOME RESULTS

The consolidation products often promote colour changes which are hepful to define the test areas. In some cases the product has a darkening effect such as in Paraloid (5%). In this case the darker area has a boundary at about 25mm from the bottom (Figure 3a). Two holes were tentatively performed at a level of 20mm, inside the “treated zone” (C), two above the treated zone at about 30mm from the bottom (B) and then two at the top of the specimen (A), as is presented in Figure 3.



a) specimen treated with Paraloid 5% in acetone (w/w)

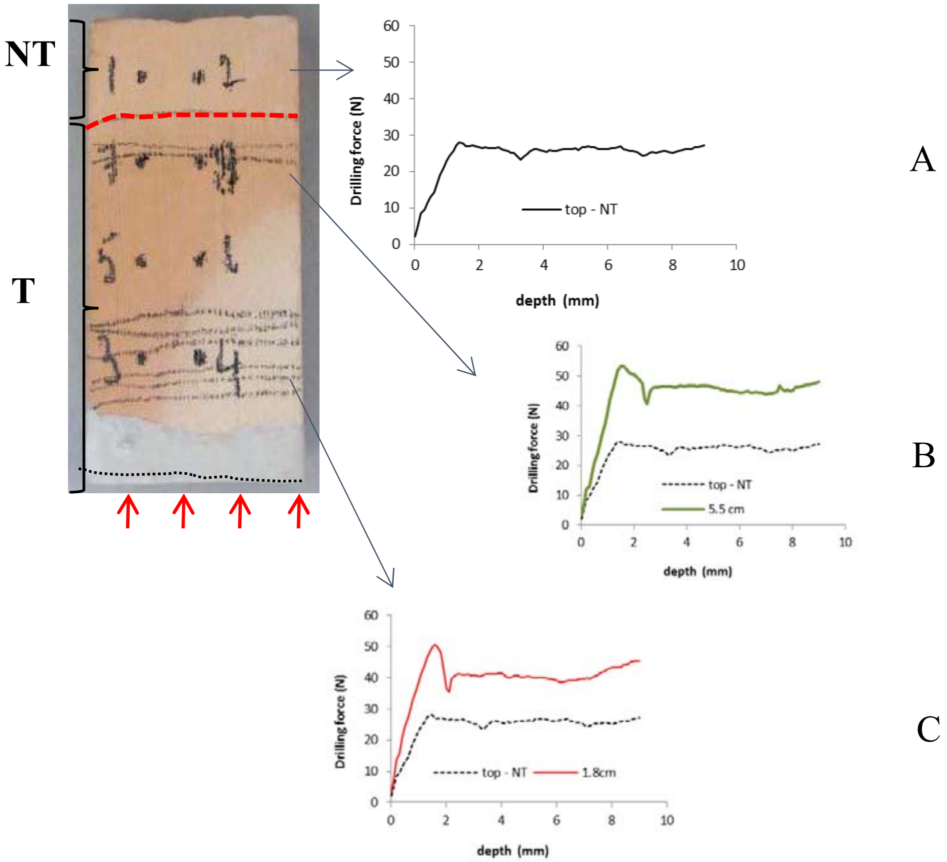
b) graphs obtained at different levels

Figure 3 – Drilling resistance curves obtained on a specimen treated with Paraloid (5%)

The original hardness is defined by the curve obtained at the level A with a resistance to drilling of about 40N using the standard conditions (rotation at 600 rpm and a penetration rate of 10mm/min). The product promoted a very strong effect on the original hardness and at the C level it was not possible to make holes, meaning that the resistance to drilling was superior to 100N (the limit of the load cell installed on this equipment). However, another interesting conclusion can be drawn from the results obtained at B level: in fact, the absence of a visual colour change suggested that this was an untreated zone; however the results clearly show that the product reached this level well above the visually set boundary.

Another important conclusion is that at this level the consolidated material is quite heterogeneous and external layers present peaks of force in comparison with the internal part. The effect is considered to be the result of drying and polymerization of the product at the surface as no direct contact with the liquid during treatment did occur.

The colour effect obtained with ethyl silicate is not so evident (see Figure 4), but the computed fringes during treatment allow to consider that the product has reached the level indicated by the line (in red). Considering so, the untreated zone (A) has a very different result when compared with B and C. A peak at the surface (of about 10N) is also detected and it is present at different levels.



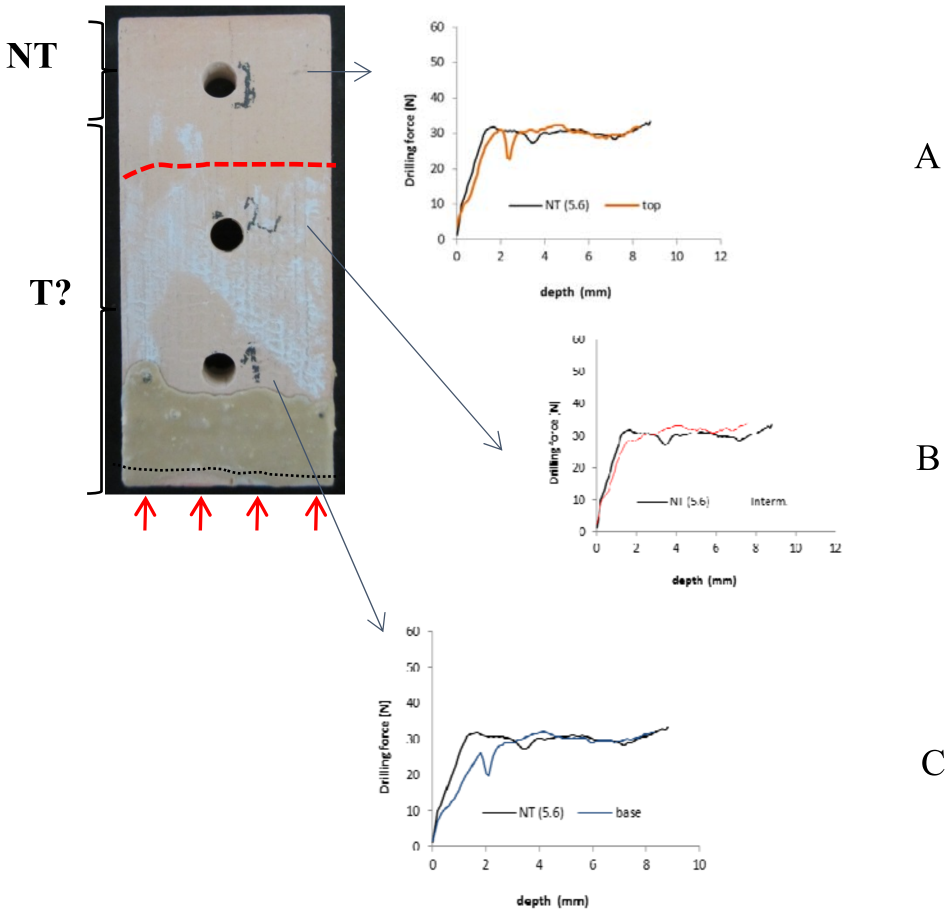
a) specimen treated with ethyl silicate

b) graphs obtained at different levels

Figure 4 – Drilling resistance curves obtained on a specimen treated with ethyl silicate

Note: the whitening zone seen on the right (at the bottom) was already present in the original biscuit and stems from some heterogeneity of the ceramic or firing.

Nanolime promotes a heterogeneous colour effect and white zones are visible, more concentrated in some parts and more dispersed in others (Figure 5). Assuming that the product had reached the line (marked in red), the holes performed at the three levels did not show significant differences and it is not possible to assert any increase in the original hardness derived from the product using this method.



b) graphs obtained at different levels

a) specimen treated with CaLoSil

Figure 5 – Drilling resistance curves obtained on a specimen treated with CaLoSil E50 (5%)

CONCLUSIONS

The use of the drilling resistance on ceramics characterization or on the evaluation of consolidants in this type of materials can be time consuming and present difficulties imposed by the heterogeneity of the materials and the wear effect of the drill bit during the test. This is a direct consequence of the compositions of the ceramics and specifically due to the presence of hard components (such as quartz grains) that are abrasive and promote heterogeneity and peaks of resistance force.

But the results obtained on ceramics treated with three types of consolidants allow to conclude that this method can also produce very meaningful and useful information.

The experience allows considering that it can be successfully used to detect the distribution profile of the product where it is not evident to have resulted any consolidation at all, but also to discriminate different behaviours after consolidation treatment.

The hard zones observed, in particular when Paraloid B72 is used, will be investigated further as they represent signs of potential risk. High porosity materials are able to absorb large quantities of consolidant and this fact can be seen as a positive indication but, in some circumstances, it can also represent an increase of the risk associated with this conservation action, if high gradients in the physical properties led to internal stress and possible fissures in the material. Further investigation will also address this issue, aiming to find the way to promote a more homogeneous distribution of the consolidant.

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