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Original article

Assessing surface weathering by revision and implementation of the peeling-test: In situ sampling and integrated analyses

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A B S T R A C T

Weathering diagnostics is mainly focused on the characterization of deterioration patterns in a mostly descriptive and non-quantitative way. Several authors tried to numerically evaluate rate and extent of the decay features highlighted in case studies. Among others, the peeling-test method was developed to recast reliable data in describing decohesion of surfaces. Such methodology is affected by high operator dependency, due to manual application of the tape on the surface and high variability in the patch area and tape typology. Our study was aimed at improving the methodology by the implementation of a device able to warrant a well-defined pressure during the application, a constant strapping angle along with the standardization of the scotch tape area and typology, obtaining reliable semi-quantitative and qualitative analyses. The revised methodology was then tested on a cladded wall in the Staglieno Monumental Cemetery in Genoa. The site was addressed by multi-scale analysis (i.e., weathering maps sketched from macro-scale observation, surfaces analyses and micro-sampling through peeling). The application of the methodology allowed highlighting the presence of capillary rise up to 1–1.5 meters height and a more weathered central area. On the whole, our protocol allowed reproducible factual sampling, and, on statistically significant population, the categorisation of decay intensity.

1. Introduction

Any rock under the exogenous cycle tends to a renewed equilibrium by means of weathering processes. The rock surface in atmospheric conditions is subject to physical, chemical and biological processes that, at different extent affect its properties. As rocks are largely employed as dimension stones, carving material or painting surface, the implementation of noninvasive methodologies to assess the state of conservation of lithic objects is nowadays one of the main topics for conservator scientists [1,2]. Although the determination of the amount of decay affecting a monument is crucial for diagnostic and conservation, this parameter is difficult to be quantified, especially with non- or micro-destructive analyses. The different approaches can be distinguished in two categories, the first based on the visual analysis of the monument; the second based on simple field tests. The so-called Fitzner indexes [3] are based only on the description of decay patterns and, therefore, are a useful tool in understanding the environmental parameters and weathering processes, but do not provide a reproducible quantification of decay [4]. The second approach aims at calculating the intensity of the diverse weathering processes by analysing their integrated evolution [5]. Focusing on surface weathering several testing methods has been formulated to address the extent of different types of decay. For example, surface erosion can be easily studied in different ways [6], e.g. measuring the recession of the original surface from the original structure [7], assessing the recession of the more susceptible minerals in case of differential erosion [8], or calculating the recession of the surface referred to grooves of ornaments and inscriptions [9]. Equally, the formation of accretion weathering like patinas or crusts can be recorded by measuring the increase in thickness of a surface. Conversely, decohesion, being a less defined physical quantity, does not have a direct form of measurement. The extent of decohesion can only be indexed evaluating other physical quantities linked to the loss of cohesiveness of the rock components, like the resistivity to drilling or hammering or the amount of loose material [10–12]. Having assessed the bearing of evaluating the decohesion of lithic artefacts, especially when dealing with carved pieces at risk of loosing figuration, we adopted the peeling-test as noninvasive analytical tools. The test is widespread and well described in literature [13–15], and proved useful in the analysis of the weathering extent and the effectiveness of protective treatments. Despite its successful use in several case studies,

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the methodology is not yet disciplined by a normative protocol for cultural heritage materials, leaving discretion in the interpretation of results. Several authors collect data with a single strap from the detached material [15], eventually repeat the procedure several times over the same sampling point to obtain the decohesion extent [14,16]; others analyse the percentage of weathered area (with one or several straps on the same spot) [16]. The different methods provide samples useful for one determination, and for different analytical needs. For instance, identifying mathematically the decohesion extent in one-point is the correct approach to infer the weathering rate of a sculpted piece; otherwise, to determine weathering rates over a complex architectonical structure, the collection of multiple samples with one strap for each sampling spot would be recommended.

2. Research aims

The aim of our study was to revise the procedures described in literature and develop a methodology suitable when dealing with real case studies. The reviewing procedure pointed out a high operator dependency in the following cases:

- bringing in contact;
- in the strapping dynamic of the tape.

To minimize these variables, the parameterization of applied pressure using a pocket penetrometer was introduced to impress constant given loads to the tape. Moreover, the use of this device lowered the influence of the strapping dynamic, central topic of many other studies [15], by reducing the testing area and defining the strapping inclination angle. The new procedure has been tested on the monumental structure of Galleria Pontasso (Staglieno Monumental Cemetery, Genoa) to evaluate the potential of the implemented methodology.

3. Method

3.1. The traditional peeling-test

The so-called peeling-test was elaborated to assess the severity of the decohesion process [13–15]. The method basically consists in strapping the surface of the object with a duct-tape and analysing the detached parts by weighing or imaging. This test was formulated as an analytical method and applied extensively [14,15,17], but lacks a regulatory protocol for its application in Cultural Heritage (note that some authors cite ASTM D 4214-07 [18] and ASTM D 3359-08 [19] standard testing which are intended for other purposes) probably due to the different possible interpretation of the results or the lack of correlation with other mechanical characteristics. In fact, the decohesion of the material can be evaluated addressing the mass removed by the tape, or the percentage of detached area [15]. Moreover, the test can be carried out once on a point, to recast intensity values, or repeated several times in order to recast the real decohesion extent of the material. The first approach is more useful for understanding weathering trends along a structure and locating deterioration sources, while the latter is to prefer for small objects or for the quantification the going decohesion. The traditional test has several problems in the procedure. In fact, the type and size of the duct-tape and the technique of applying can affect the measurement. To avoid or at least mitigate the drawback, a new method was experienced.

3.2. The new implemented peeling-test (i-PT)

The implementation encompasses the adoption of a pocket penetrometer to stick the tape to the sample surface with constant load (2 kgf/cm^2) on a well-defined area (i.e., SEM round stub with a surface of 1.3 cm^2), thus providing reproducible homogeneous measures, by limiting the sampling variability. A metal piece capable to grip the alumina stub was designed and crafted to be screwed at the tip of a pocket penetrometer. The pocket penetrometer is a lightweight instrument for field use and check of the visual classification of soils. It gives data about consistency, shear strength, and approximate unconfined shear strength. Direct-reading scale, in our tool in kgf/cm^2, corresponds to equivalent unconfined compressive strength. The applied pressure could be selected in relation with the decohesion rate of each surface, and the use of the penetrometer allows maintaining a constant load on the stub surface for the whole measuring time. The crafted device allows exerting the load perpendicular to the sample surface, and the small, round contact surface of the tape (about 1.3 cm^2) allows assuming as negligible the strapping dynamic. Moreover, the circular shape of the tip is thought to avoid tangential stresses and convey evenly the applied pressure on the surface. The current configuration of our device requires a round flat surface of at least 1.5 cm^2 to perform the measurements, in order to get the whole tape equally in contact with the analysed material, and have a significant sampling. In the present work, PELCO Tabs™ of carbon conductive tape with $\Phi = 12$ mm (Prod. No: 16084-1) were employed for their dimensional compatibility with the selected stub, and the possibility to redirect to SEM the acquired sample without further preparation. The selected scotch is a double-sided adhesive tape composed of a sandwich of acrylic adhesive and carbon clothed fibre mat for a total thickness of 125 μm, with an adhesive strength of 0.27 N/m. The amount of time for each sampling was chosen in order to balance the errors that can be induced by a fast (e.g., misplacements, overloads or low pressures, incorrect strap angle, etc.) or a too long (e.g., overloads, sliding, etc.) measurement. The point of the in situ application of the method is collecting and storing the sample without loosing grains, which is fundamental for subsequent elaboration. On the whole, the new procedure (Fig. 1) can be synthetized as follows:

- preparation of stubs for SEM analysis with adherent conductive carbon tape (surface area 1.3 cm^2);
- weighing of stub + tape + its plastic envelope at 0.001 g precision;
- fixing the stub to a pocket penetrometer;
- noninvasive sampling of the incoherent dust, applying a constant pressure of 2 kgf/cm^2 for 1 minute, then packing away the stub without loosing grains;
- weighing of stub + tape + weathering products + their plastic envelope at 0.001 g precision;
- recast the weight of removed material;
- addressing the weathering products to image analysis, electron microscopy and or microanalysis.

The new sampling procedure allows:

- a mass quantification;
- an areal quantification;
- a qualitative characterization of the detached material.

The first yields the weight of detached material; the second is carried out using image analysis software (ImageJ®) and provides the percentage of the detached area, while the latter allows a chemical compositional analysis, for better understanding the involved processes. The mass quantification is a rather simple measure, recast directly from the sample by subtracting the mass of the