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Non-linear decay of building stones during freeze–thaw weathering processes

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HIGHLIGHTS

- ▶ 102 Samples of carbonate rocks were tested during 100 cycles of freeze–thaw.
- ▶ Decay evolution was characterized at micro/meso-scale and petrophysically.
- ▶ Ultrasonic attenuation is the most useful parameter for studying damage evolution.
- ▶ Strength, porosity and weight decrease are not useful for damage quantification.
- ▶ Standard procedures are revealed as not effective for rock durability diagnostics.

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ABSTRACT

This paper studies the resistance of rocks to freeze–thaw and their petrophysical evolution during weathering. Moreover, the accuracy of existing standards regarding frost durability is discussed. A long-term test was established with these purposes, in which 102 samples of six different dimension stone types were tested (carbonates).

Samples were divided into five groups and each group was tested after 0, 12, 24, 48 and 96 freeze–thaw cycles. At the end of the cycles several properties were measured: volume loss, open porosity variation, visual damage, mechanical properties evolution (measuring strength and elastic modulus) and ultrasonic propagation (quantifying both P-wave velocity and spatial attenuation). The micro-textural evolution was also studied using SEM in polished samples.

Results display that the rocks with the highest open porosity values (>10%) are the least durable. These rocks show a non-linear decay pattern, with long periods of apparent stability followed by rapid and catastrophic decay. Microscopic observation reveals that during the stable period, isolated microcracks appear from where new ones nucleate and grow as the test progresses. When a critical threshold is exceeded, microcracks turn into cracks and grow rapidly, causing rock breakdown after a low number of cycles. Most of the measured petrophysical parameters do not predict the ultimate breakdown of rocks. However, spatial attenuation of ultrasonic waves reveals as the most sensitive parameter, detecting the critical decay threshold of rocks and their imminent breakdown. Results suggest an important review of standardized durability tests since they do not reflect the reality of frost weathering of rocks: to increase the number of freeze–thaw cycles and to monitoring the weathering process of samples by means ultrasonic measurements.

1. Introduction

Frost durability has traditionally been viewed as an important rock weathering process and one of the first ones that historically was tested to characterize building material performance. As early as 1828, De Thury devised a test using salt crystallization as a way of modelling ice crystallization within rock pores. Ice crystallization in the porous system of rock can cause important damages [41,42], not only in very cold regions, but also in temperate climates in which could episodically reach freezing temperature especially at night-time. However, although the effects of ice crystallization in rocks are well-known, the weathering physical process is until under discussion in the scientific community.

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Freeze–thaw process in rock pores is perhaps more complex than traditional views might suggest, for example, the temperature at which water freezes in pores is variable and difficult to ascertain (it cannot be inferred from ambient temperature data), and the precise mechanisms by which freeze–thaw actually causes damage is a matter of debate [18].

As for salt crystallization, frost resistance of natural stone is going to depend very much on its initial porosity and how it evolves after freezing. This behaviour will obviously change with time and as stone undergoes successive cycles of freeze–thaw. This is why, frost resistance is determined through accelerated ageing tests in which stones are frozen in a chamber after being loaded with water by immersion. Samples are thawed again in water, which loads the samples again for the next freezing cycle. Although these tests are standardized [8–10], the number of freeze–thaw cycles the samples undergo and the physical property quantifying frost action are not the same across the different existing standards.

Frost resistance can be evaluated either by comparing the initial and final state of the samples, or preferably by monitoring non-destructively the evolution of samples during laboratory tests. In addition to visual inspection, standards propose several properties for this evaluation, including weight loss, porosity, apparent volume, dynamic elastic modulus (by measuring the fundamental resonance frequency), flexural strength (under concentrated load) or compressive strength.

Some of these evaluation parameters present problems for being considered as the most appropriate procedures for weathering control. On the one hand, visual inspection, weight loss, porosity or apparent volume may be highly restrictive as it only reflects the variable and surface loss of material without measuring the overall decrease in rock strength, which is not necessarily visible. On the other hand, measurements of the fundamental resonance frequency has found important problems of reproducibility, mainly by the coupling between the specimen and emitting and receiving transducers, and the isolating from external vibrations. This parameter is therefore becoming increasingly obsolete in the natural stone characterizations.

The variation of flexural and compressive strength after freeze–thaw cycles is used to quantify frost action in natural stone depending on material requirement. Thus, flexural strength is required for example for paving slabs whereas compressive strength is used for kerbs.

Otherwise although ultrasonic test is not considered by standards to evaluate the decay state, this technique has been employed with this purpose on the evaluation of laboratory decay tests for decades, offering interesting and useful results [5,6,19,26,27,40,21,28]. Mainly, P-wave velocities have been measured, but, previous works have been revealed that the spatial attenuation of the ultrasonic wave is a much more sensitive parameter, offering a more complete view of the weathering state of the rock [20].

Ultrasounds allow performing non-destructive monitoring of each individual sample throughout the test. This overcomes one of the shortfalls of most of the indicators included in standards that act on 'black box' type experimentation (i.e. they only take into account initial and final state) and do not take into account the individuality of each block [14]. By using non-destructive monitoring of individual samples throughout decay tests, the individuality of each block can be tested and may give and "early warning" of surface modifications that appear in tested blocks prior any visible decay happens can be identified [14].

This is important as stone decay is a dynamic process and evolves into an inherently complex system in which processes evolution and rates may change due to many factors both intrinsic and extrinsic to the material. Most standardized accelerated stone decay tests are based on the assumption that decay will occur always linearly and that an indicator based on the comparison between the final and the initial values of certain physical property or parameter will give a figure proportional to the durability of certain stone type that can serve as comparison to other stone types. However, stone decay behaves most often as a non-linear system, i.e. those in which outputs do not change proportionally to a change in inputs over the entire range of inputs [37]. As Viles [37] points out some of the reasons why stone decay may behave as a non-linear system include (but are not limited to) the existence of decay thresholds, self-reinforcing positive feedbacks, and 'memory effects'. Non-linearity is behind the apparently chaotic behaviour observed in many buildings in which some blocks show an extraordinarily rapid rate of decay that leads to so-called 'catastrophic decay' [31]. The seemingly unpredictable, episodic and sometimes catastrophic breakdown is particularly common in sandstones [32] but also found in limestones [33].

The existence of thresholds has been pointed out specifically for freeze–thaw as a cause of non-linear behaviour [37]. Samples remain initially apparently unalterable until the stress exceeds the strength of the stone and an episode of noticeable decay takes place. This threshold-based episodic decay pathway has been extensively described in the context of stone decay by Smith et al. [33,43,44]. In this model, every time a threshold is reached, the balance between negative feedbacks (opposed to decay) and positive feedback (promoting decay) will control if there is another episode of apparent stability or not. If in any of these steps positive feedbacks well exceed negative feedbacks, decay may be considered 'catastrophic'.

Positive feedbacks include, for example, anisotropy of the decay process, energy decrease from crack formation to crack propagation and pore form and size variations throughout the decay process. Each of these positive feedbacks may not lead itself to catastrophic decay, but will leave a trace in the rock that can be expressed later on as a 'memory effect'. McCabe et al. [22,23] have shown how the combination of background and 'exceptional' stresses during the decay history of stone may change the possible decay pathways. McCabe et al. [23] show how combining decay processes that may seem to lead initially to similar decay pathways, after a long-enough number of cycles may diverge greatly in time.

Therefore, although Trudgill and Viles [35] point out weathering laboratory rates cannot be used to the predict the precise field weathering rates, knowing how the weathering process takes place, at what rates and if it is linear, episodic or chaotic, is the only way of understand and predict the behaviour of a building material. The aim of this paper is to characterize minutely the weathering process of rocks during freeze–thaw conditions. This characterization is carried out from both a petrographic point of view, checking the evolution of rock texture at micro and mesoscale, as well as from a petrophysic field, analyzing the changes produced in the porous system of rocks and in their ultrasonic and mechanical response. Moreover, the effectiveness of existing standards will be questioned, taking into account both the decay mechanisms of rocks during freeze–thaw test and the sensitivity of the monitoring parameters suggested by the standard procedures. Finally, a new weathering inspection criterion is proposed in order to increase the effectiveness of the rock damage evaluation.

2. Materials

Six different limestones have been selected for this study: a travertine (LP-Tr), a calcite marble (LP-Mb), two biosparite limestones (LP-Bs and P-Bs), an oosparite limestone (P-Os) and a biomicritic limestone (P-Bm) (Figs. 1 and 2). All of them are quarried in Spain and commercialized as building stones for cladding and paving.