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Influence of physical and mechanical properties on the durability of limestone subjected to freeze-thaw cycles

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HIGHLIGHTS

- Qualitative and quantitative analysis of the frost sensitivity of five limestones.
- Investigation of different decay processes of stones related to freeze-thaw cycles.
- The decay constant values were correlated with different fresh stone parameters.
- Frost damage is correlated with the ratio of the volume fraction of water to that of air.

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ABSTRACT

The frost sensitivity of five French limestones (Massangis (MA), Lens (LS), Savonnières (SA), Saint-Maximin (MX) and Migné (MI)), with different physical and mechanical properties, was studied. The total porosity ranges from 10% to 35% and the uniaxial compressive strength ranges between 10 MPa and 60 MPa. The freeze-thaw tests were applied on the samples saturated in natural condition. During freeze-thaw cycles we recorded the evolution of temperature and volumetric strain of a specimen for each stone and after the freeze-thaw cycles, the weathering evolution in stone samples was monitored by measurement of different physical and mechanical parameters (porosity, P-wave velocity, fracture toughness, compressive strength and elastic static modulus). Two behaviors were observed: a volumetric expansion during freezing phase accompanied by a very important damage from the first cycle until the failure of specimen for MI and MX stones, and a volumetric contraction accompanied with a light damage for the high number of freeze-thaw cycle in the cases of MA, LS and SA. Following the freeze–thaw tests, the durability of stones was evaluated using a decay function model. The decay constant values determined from the evolution of P-wave velocity were correlated with different fresh stone properties. Contrary to generally accepted ideas, the correlation coefficients between the decay constant and total porosity or degree of saturation are very low. Porosity in natural condition seems to have the strongest influence on the decay constant. However, its negative impact can be offset by a bigger part of trapped porosity. The results indicate that it is possible to predict the frost damage of the stones, with a better level of confidence, from the ratio of the volume fraction of water to that of air rather than from only the total porosity or degree of saturation.

1. Introduction

Conservation of historical cultural heritage built in stone requires a thorough understanding of different decay processes of stones related to mechanical and environmental conditions. The mechanisms governing these decay processes and the real effect of intrinsic properties of the stones on the development of these processes must be studied. Among the different environmental conditions responsible for alteration in building materials, we are interested in freeze-thaw cycles on different types of limestone.

Indeed, during the water freezing in porous medium, two types of stresses are applied within the stone [1–3]. The first is as a result of the ice volume expansion, which occupies a volume about 9% more than liquid water. The second source of stress is due to the

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migration of unfrozen water toward ice front in the pore network [4–5]. All the water in the stone does not freeze at the same instant and the freezing point of pore water decreases with pore size. This liquid water can migrate toward ice front due to various reasons: osmotic pressure [1], capillary pressures [2] and causes overpressure in the pores.

The influence of different intrinsic properties and environmental conditions on the frost damage sensitivity of stones was studied in previous works. The degree of saturation is usually considered such as the most important parameter [4,6–8]. Different authors point out a critical degree of saturation, beyond that threshold the material is damaged by frost, for example 80% proposed by [6] and 70% proposed by [7]. Some authors [4,9] underline the importance of the porous network characteristics such as the distribution of the pore size. There is almost no or very little information regarding the effect of mechanical parameters on frost sensitivity of stones.

The freeze-thaw cycles lead to physical and mechanical degradation of natural stones. Different intrinsic property changes, during freeze-thaw cycles, are reported in previous studies. Among the different parameters, P-wave velocity drop is mostly reported by various authors. Some authors indicate the increase of the porosity [7] and a decrease of different mechanical properties such as the Young's modulus, compressive strength and tensile strength [10–11].

Many researchers have studied freezing and thawing of stones through mathematical modeling [12–17]. A descriptive-behavioral model is proposed by [12]. This is a decay function model to evaluate the loss of integrity of stones due to freeze-thaw cycles. For these authors, the concept of rock integrity involves both the hardness and the structural wholeness of the rock. Consequently, different parameters can be used as proxies for rock integrity. In their study, the Shore hardness (SH) was used as the measure of rock integrity. This model has been taken up by many authors [13–14,17] to evaluate decay constant value for different types of stones. [14] proposes a model, generated by multiple regression analysis, to evaluate an index property (P-wave velocity, uniaxial compressive strength and Schmidt rebound hardness) from the initial property and porosity of rock after 20 freeze-thaw cycles. A statistical model was developed by [16] for predicting the loss in uniaxial compression strength as a function of freeze-thaw cycles from the impact strength, elasticity modulus and water absorption of intact stones using a multiple regression. Some authors have used a causative approach for the modelling of different physical frost damage mechanisms [5,15]. [5] develop a model of the volume expansion of the ice growing inside the cavity and the disjoining forces across a thin pre-melted film that develop when the ice has grown to be close enough to the rock. [15] establishes a simple formulation to estimate the stress profiles result from a coupling between thermal propagation, water phase shift and water flow in the notch and porous rock matrix.

This study aims to complement previous research [18] with data on two additional stones. All these results make it possible to gather a sufficient database to carry out a quantitative parametric study. The main focus of this work is to determine the relative part of different processes involved in frost damage of limestone, on the one hand, and to highlight the most relevant properties of materials to predict the kinetics of degradation of the studied stones on the other. The ultimate objective of this research is to propose predictive models for a better visibility of the lifetime of limestones as a function of freeze-thaw cycles. For this purpose, after characterization of all samples, the freeze-thaw cycles were applied on the samples saturated in natural condition. During freeze-thaw cycles the evolution of temperature and volumetric strain of a specimen were monitored during freeze-thaw cycles. The weathering evolution in stone samples was investigated by regular measurements of different physical and mechanical parameters throughout freeze-thaw cycles. Following the freeze–thaw tests, the durability of stones was evaluated using a decay function model. Finally, the correlation between decay constant values with different fresh stone properties was discussed.

2. Materials and methods

2.1. Origin of studied stones and petrography

Five French limestones, taken in the active quarries of Massangis (MA), Lens (LS), Migné (MI), Savonnières (SA) and Saint-Maximin (MX), located in different French regions were used for this study (Table 1). These stones have been chosen because they have been widely used in the construction of historic monuments and are used as restorative stones, on the one hand (Table 1), and, on the other hand, they have very largely different physical and mechanical properties (Tables 2 and 3), allowing to understand the influence of these parameters on stones frost sensitivity. The selected stones are oolitic limesones with similar mineralogical composition made of 99% of calcite, except the Saint-Maximin and Massangis that are marked by the presence of 10% and 3% of quartz in its composition, respectively. Oolitic are cemented by microsparitic cement in the case of Savonnières and a micritic matrix for others stones. The oolitic size ranges from 100 μm to 700 μm with a heterogeneous distribution for MA and SA and a homogenous distribution for MI, LS and MX stones.

2.2. Freezing-thawing cycles and monitoring

40×80 mm cylinders were cored perpendicular to bedding planes and saturated in natural condition (cf. Section 2.3). In the case of SA, the freeze–thaw cycles were carried out on the samples with two different degrees of saturation, $SA(59\%):59\%$ for a natural saturation during 48 h and $SA(73\%):73\%$ for a period more than 48 h. They were exposed to freezing–thawing cycles in a chamber whose temperature is regulated by a Eurotherm 2500 control unit. The air temperature during a freeze-thaw cycle ranged from 10 °C to −10 °C and vice versa at a rate of 4 °C/h followed by a stage, where the temperature was maintained constant for one hour at −10 °C or +10 °C (Fig. 1). The samples were sealed in plastic bags in order to keep constant their moisture state, as shown in Fig. 1.

Twenty-five intact cores were prepared from each stone for freeze-thaw tests and physical and mechanical properties were evaluated at the end of each 5 freezing-thawing cycles for Migné and Saint-Maximin stones, at the end of each 10 freezing-thawing cycles for Savonnières and 50 cycles for Lens and Massangis stones. In the case of Savonnières stone 50 specimens were prepared, 25 samples for each degree of saturation. It should be specified that, this programmer was fixed after preliminary tests and a first evaluated of freeze-thaw sensitivity for each type of stones. At the end of each stage, P-wave velocity measurements were carried out on all saturated specimens. And four (in cases of steps 1357) or three specimens (in cases of steps 2, 4, 6) for each stone were dried in an oven at 60 ± 5 °C before mechanical tests and the remaining samples continue the freezing-thawing cycles. Cyclic uniaxial compression tests were carried out on three previously dried specimens. Another dried cylinder is used for the Semi-Circular Bending test to investigate the fracture toughness (Fig. 2). It should be note that compression tests were carried out at all steps and Semi-Circular Bending tests were performed only at steps 1, 3, 5 and 7 (Fig. 2).

A specimen from each stone was monitored with two thermocouples for the following of temperature evolution in the center