



Preliminary laboratory characterization of serpentinite rocks from Calabria (southern Italy) employed as stone material

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ABSTRACT - Serpentinite rocks are employed and traded as building and ornamental stones as well as for decorative jewels worldwide. In Calabria (southern Italy), extensive ophiolite outcrops, made of serpentinite and metabasite rocks, allowed serpentinite exploitation and marketing since prehistorical times. For this reason, we chose some serpentinite-key outcrops, such as those ones located at quarries and road cut in the area of Sila Piccola (northern Calabria), to collect representative samples for specific laboratory analyses.

The petrographic features of the serpentinite rock samples have been investigated in detail by means of Polarized Light Microscopy (PLM), X-ray powder diffractometry (XRPD), Scanning Electron Microscopy combined with Energy-Dispersive Spectrometry (SEM/EDS). Moreover, some tests have been carried out on serpentinite specimens to establish their physical-mechanical properties such as the Uniaxial Compressive Strength (UCS), porosity and seismic behavior, before and after cycles of salt crystallization tests. This work aims to study serpentinites from Gimigliano and Conflenti quarry Calabria region (Italy) attempting to shed light on the variation of main physical-mechanical and petrophysical properties that occur after these rocks undergo ageing tests, in order to predict their performances upon emplacement in monuments. Results showed that microfractures play a key-role in affecting the whole behavior of these rocks, since the combined action of filling by salt and expansion implicates weaker physical-mechanical properties.

Keywords: serpentinite; physical-mechanical properties; petrophysics; historical quarries; Naturally Occurring Asbestos; Calabria (Italy).

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1. INTRODUCTION

Serpentinite is one of the metamorphic rocks used as building materials and ornamentals stones for its appealing appearance and color (Pereira, 2012). From the mineralogical point of view, serpentinite rocks are mostly composed of serpentine-group minerals (i.e. antigorite, lizardite and chrysotile; Deer et al., 2009). They may also contain other accessory minerals such as magnetite, talc, chlorite, brucite, carlosturanite, balangeroite, carbonates as well as remnants of the precursor mineralogy such as olivine and pyroxene (Moody, 1976; Compagnoni et al., 1985; Belluso and Ferraris 1991; Bloise et al., 2009 a,b; Bloise et al., 2010). Serpentinites are formed after the transformation (i.e., serpentinization) of ultramafic rocks such as harzburgite, dunite and lherzolite, the main minerals of which are olivine and pyroxenes. Serpentinization process has been exhaustively discussed

in the past years (Moody, 1976; Evans 2004; Bloise et al., 2017a). The rocks can be partially or totally serpentinized and therefore exhibit different varieties of texture, which will involve different mechanical and physical properties, such as different performances upon emplacement. Moreover, once moved from the outcrop to the outdoor urban context, serpentinites are highly susceptible to weathering (Pereira et al., 2012), which is one of the main factors affecting the mechanical behavior of rocks (e.g. Basu et al., 2009; Cirrincione et al., 2013; Mineo et al., 2015; Pappalardo and Mineo, 2017). For example, the natural alternation of seasons and weather patterns, such as temperature changes, freezing and thawing, and washing away by rain and snow melt water, may affect the chemical and physical properties of the serpentinite rocks (Schreier, 1989; Bloise et al., 2017b). However, since antiquity, they have been used around the world in the Mediterranean, Asia-Oceania, and the Americas

to make jewelry and ceremonial and ornamental carvings (Guillot and Hattori, 2013). In many different civilizations, serpentinite was probably among the first materials to be used in tool making, in decoration, or for its supposed magic properties, such as protection from snakebites. For example, a spectacular illustration is provided by cylinder seals-small items that may have been used as much as 5 millennia ago as administrative tools or amulets. Their deep olive-green color and smooth but scaly appearance is the basis for their name, which comes from the Latin *serpentinus*, meaning “serpent” (i.e. snake). The unique thermal (Bloise et al., 2016a) and mechanical properties of serpentine may have been instrumental in helping develop such objects: serpentine is an easy worked materials, but hard enough to keep the information engraved on it from becoming blunted over time (Chernak and Hirth, 2010). Serpentinites are referred to as “Green Marble” commercially; this name has no correspondence to their actual geochemistry, mineralogy and/or physical properties of serpentinites (Ismael and Hassan, 2008; Pereira and Peinado, 2012). Currently, the most commonly used serpentinites for construction come from India, Pakistan, Guatemala and Egypt and the principal countries producing ornamental stones in descending order are China, Italy, India, Egypt and Spain (Ismael and Hassan, 2008). Chrysotile, one of the three main forms of serpentine, is unfortunately the main constituent of asbestos, and its dust is thought to be hazardous to the human health (Bloise et al., 2016b). The Calabria region (southern Italy) is characterized by wide occurrence of serpentinite rocks (Punturo et al., 2015; Bloise et al., 2014; Bloise et al., 2016c), that are removed from their natural place of origin to be used as building and ornamental stones as well as for stoup, vases, hatchets etc (Fig. 1). The main localities of exploitation, some of

which are still active, have been detected in a lot of outcrops (“Verde Calabria” is the local stone commercial name). These sites are located nearby Gimigliano (South-eastern Sila Piccola), Martirano and in the Mount Reventino area (South-western Sila Piccola) (Fig. 2). This work aims to study the microstructural, compositional and physical-mechanical properties of serpentinite outcrops in Sila Piccola (Calabria region, Southern Italy) attempting to shed light into the features of serpentinite after their “change in service” from natural outcrops to manufactures and on their state of decay. Taking into account their composition and textures, rocks with such differences will evolve in different ways in a weathering environment. As a consequence of this, serpentinite rock samples have been characterized in detail through petrographic and mineralogical investigations. Moreover, the strength under compression is the main mechanical parameter to investigate, because it strongly conditions the employment of such rocks as building stone and, in turn, is affected by their physical properties (e.g. Miller, 1965; Palchik and Hatzor, 2002; Mineo and Pappalardo, 2016; Pappalardo et al., 2016a; Pepe et al., 2018). Therefore, uniaxial compression tests were carried out, even on specimens after salt crystallization, and results were correlated to porosity and seismic properties of such rocks in order to study their relationships. Data, presented in this study, can be also used to better plan future restoration works of artifacts built with serpentinite rocks.

2. AREA DESCRIPTION

The study area is located in Calabria (southern Italy), where an ophiolitic sequence known as Liguride Complex (Jurassic-Early Miocene in age; Vitale et al., 2013) is tectonically interposed between upper



Fig. 1 - Examples of use of serpentinite rocks for artefact. a) pillars in altar; b) baptismal font.

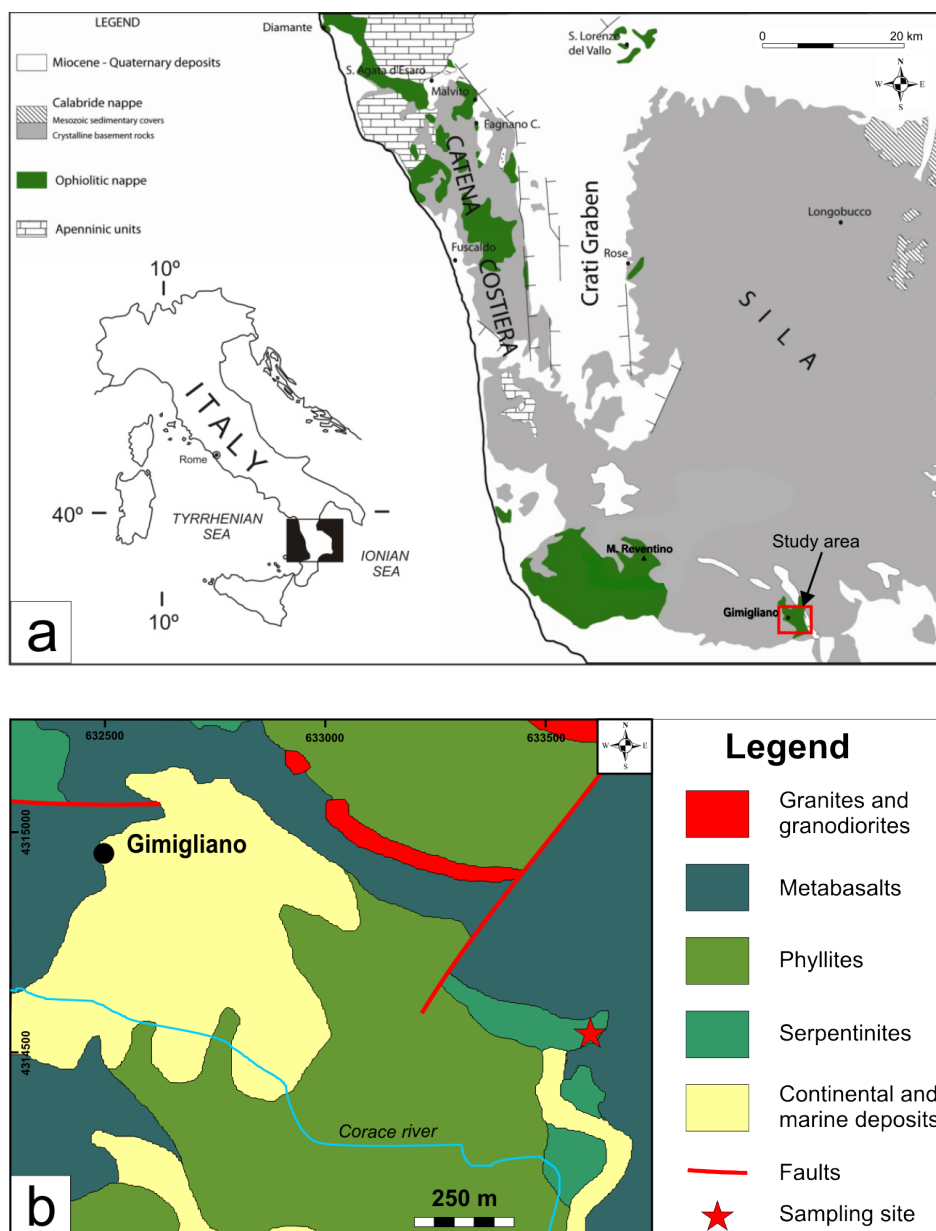


Fig. 2 - a) Geological sketch map of the northern sector of the Calabrian-Peloritani Orogen and study area; b) detailed geological map of the study area and sampling site at the abandoned quarry.

metamorphic and magmatic acid rocks (Calabrian Complex; Cirrincione et al., 2015) and lower carbonate level of Apulian platform belonging to Apennine Units (Monaco et al., 1998; Ciarcia et al., 2012). Ophiolites exposures (often outcropping as tectonic melanges) trace an important geological boundary consisting of an ocean-continent paleomargin, roughly corresponding to the Catanzaro trough, a Neogene-Quaternary sedimentary basin, which separates two main sectors of the Calabrian Peloritani Orogen (CPO; Cirrincione et al., 2015): the Sila Massif northward, mainly consisting of plutonic rocks, and the Serre Massif southward (Punturo et al., 2017; Caggianelli et al., 2007), made by metamorphic rocks intruded by various pulses of magmatic intrusive bodies (Late Hercynian in age; Fiannacca et al., 2017).

The ophiolite sequence can be considered as a multilayer tectonically sliced complex forming an accretionary wedge (Piluso et al., 2000; Ciarcia et al., 2012) and comprises different HP/LT metamorphic rock types ($0.9 \text{ GPa} < P < 1.1 \text{ GPa}$; $T \text{ ca. } 350 \text{ }^\circ\text{C}$; Piluso et al., 2000): from mantle-derived ultramafic serpentinites to massive and foliated metabasites with T-MORB affinity of the basaltic protoliths (Liberi et al., 2006; Punturo et al., 2004). Serpentinites crop out as dark green colored massive bodies, sometimes weakly foliated, cut by serpentine (Fig. 2b) and calcite veins. The sedimentary cover is essentially made by meta-radiolarites and meta-limestones (Lanzafame and Zuffa, 1976; Spadea, 1979) and by flyschoid terrigenous deposits consisting of metapelites and metarenites of uncertain age.

Samples were collected near the village of Gimigliano, nearby an abandoned quarry of serpentinites and

ophicalcites (Fig. 2 a,b) where Gimigliano-Mount Reventino Unit occurs (Sila Piccola, Calabria, southern Italy; Fig. 2a). It is essentially made of metabasites (i.e. meta-basalts, -gabbros and -dolerites) and serpentinites covered by marbles alternating with calc-schists and quartzites (Piluso et al., 2000; Punturo et al., 2015).

3. METHODS AND MATERIAL STUDIED

Serpentinite rocks have been collected at one abandoned quarry where this lithotype was formerly exploited (Fig. 2b).

For the petrographic investigation, serpentinite rocks were characterized by PLM on thin sections prepared from representative part of samples.

Salt crystallization tests were carried out on serpentinites in order to study their resistance to decay and the development of microfractures, if any. Given the lithotype, the procedure followed was modified from the one proposed by UNI EN 12370 (1999). Five cm edged cubes of known mass were subjected to at least thirty cycles consisting of 2 h at 20 °C full immersion in an aqueous solution of decahydrate sodium sulphate ($\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$) at 14%v/v and subsequent heating for 16 h at 105 °C. The mass of each test sample was measured again after 35 cycles in order to determine the resulting variation in mass.

Direct seismic measurements were performed to investigate on a set of 12 cubical specimens, 6 of which underwent a preliminary salt crystallization process; all the specimens were previously oven-dried at 50 °C overnight. The non-destructive ultrasonic wave velocity test was carried out at standard conditions according to the ASTM designation (D2845-00). The device used for measurements of transit time (ms) is the A5000M pulse transmitter (MAE s.r.l.), with transducers operating at 50 kHz and an accuracy of ± 0.3 ms. P-wave measurements on three mutually perpendicular axes of each cube permitted also calculating the related seismic anisotropy, where $A\% = 100(V_{\text{max}} - V_{\text{min}})/V_{\text{mean}}$ (Birch, 1961).

Physical-mechanical characterization of the selected rock specimens was carried out at the Laboratory of Applied Geology of University of Catania according to ISRM (2007) specifications. Bulk and real density and total porosity were estimated on the specimens whose seismic properties were previously measured. The same specimens were also employed for the estimation of the Uniaxial Compressive Strength (UCS) by an automatic 3000 kN hydraulic press machine. Results were statistically analyzed and correlations between properties are provided with the aim of investigating on the relationship between such properties.

4. RESULTS AND DISCUSSION

4.1. Petrographic and mineralogical features of samples

From mineralogical point of view, the investigated serpentinite rocks are mostly constituted of antigorite,

chrysotile, lizardite, tremolite-actinolite; calcite, magnetite±chlorite occur in minor amounts (Bloise et al., 2016c). At the microscope scale (Fig. 3) the serpentine group minerals, together with small magnetite grains, constitute the typical fine mesh textured matrix (Fig. 3 c,e); sometimes they also appear as pseudomorphic aggregates after former olivine, pyroxene and spinel (Fig. 3 a,c and d). At the mesoscopic scale, samples sometimes are affected by serpentine and calcite veins (Fig. 3f). In detail, serpentine fibers are oriented either perpendicular to the vein selvages or parallel to their elongation directions with different veins crosscutting each other (Fig. 3 a,c). Detailed microstructural description of serpentinites of the abandoned quarry at Gimigliano village is also reported in Punturo et al. (2015) and Bloise et al. (2016c).

4.2. Physical-mechanical behavior

Salt crystallization tests carried out on the serpentinite sample cubes revealed that, after 35 cycles, salts tend to crystallize both within microfractures and veins, thus promoting increase in pressure that caused the breakage of some specimens at 13-15 cycle. In some case, substitution of soluble salts after calcite within microfractures was observed as well (Fig. 4). Moreover, some decay forms such as exfoliation of surface of the studied specimens also occur.

Seismic velocity tests have been carried out on sample cubes not involved (NCT) and post (PCT) the salt crystallization test. Measurements were done on three mutually perpendicular directions, since the studied serpentinite rocks show nearly isotropic fabric (Fig. 4), these directions have been arbitrarily chosen; results also permitted to calculate the seismic anisotropy AVp related to compressional waves. Before crystallization tests, average compressional velocity values are in the range between 6.00 and 7.50 km/s, while after crystallization tests, average values decrease in the range 5.19-6.78 km/s (Fig. 5), indicating fracture development inside the rock.

As far as the Vp related seismic anisotropy, results highlighted that in almost all specimens after the crystallization test, values (AVp) increase from 14% to 17%; this is likely an effect due to microfracture widening, as a consequence of salt crystallization inside them. From a general point of view, tested specimens are characterized by a low porosity grade (<5%) with an evident variability (Fig. 6b). The greatest values (from 1.9 to 4.8%) are related to specimens tested in their natural condition, i.e. not involved into crystallization test (NCT), whose porosity is represented by cracks at the micro scale. This is a key aspect, since it has been observed that the cracks and microcracks play a structural control on the mechanical behavior of rocks and are responsible of their anisotropy (e.g. Sousa et al., 2005; Pappalardo and Mineo, 2016; Pappalardo et al., 2016b). On the other hand, the lowest porosity affects post-crystallized specimens (PCT), where cracks are likely to having been filled by crystals of salt. Indeed, PCT are characterized by an average reduction of porosity of 23% than NCT, with values ranging between 0.8 and 4.5%.

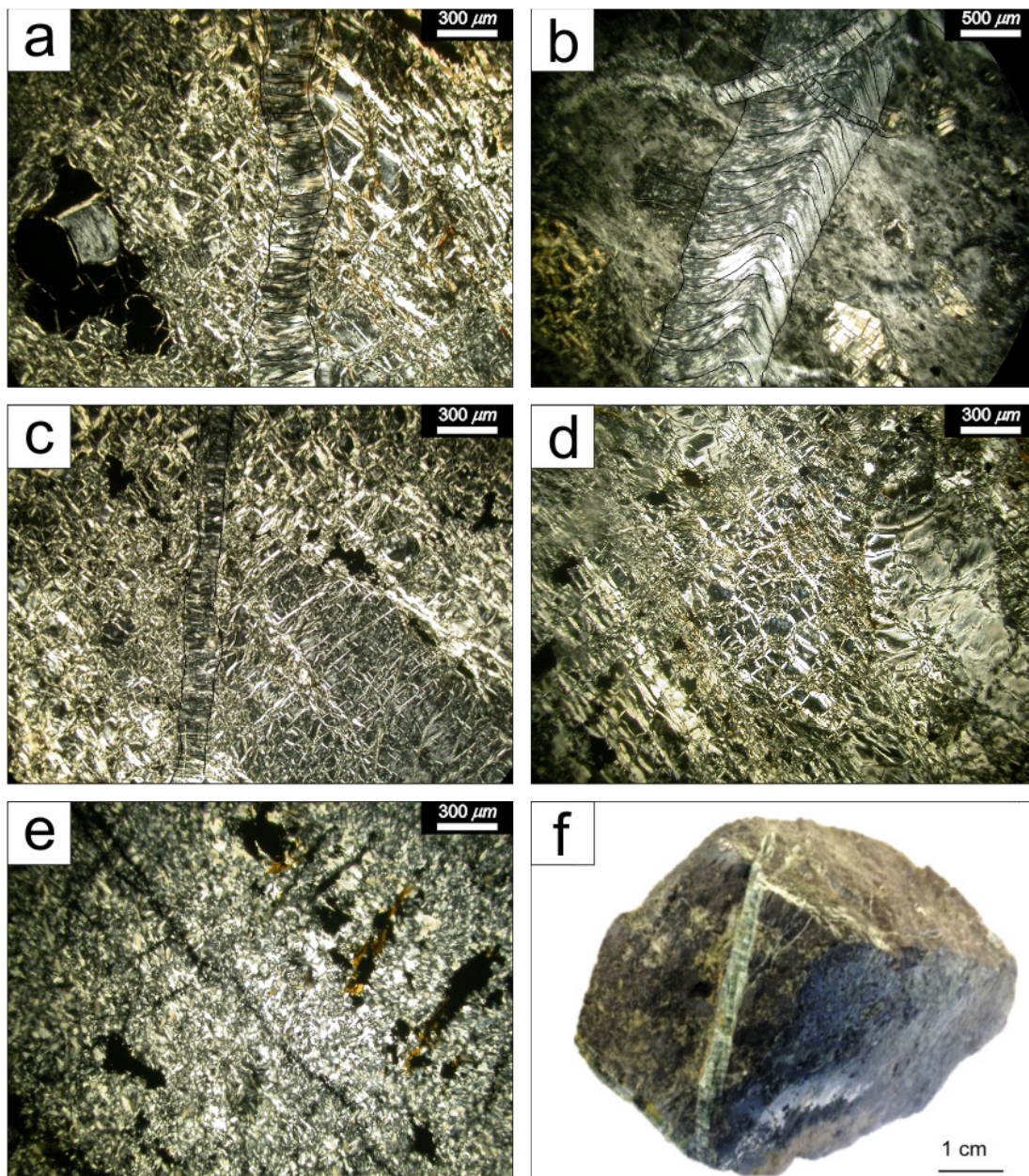


Fig. 3 - Photomicrographs showing the petrographic features of the studied serpentinite rocks. a) mesh texture with relict of spinel and cross-cutting vein; b) various veins cross-cutting each other; c) vein cutting the mesh texture, with an large pseudomorph of olivine; d) pseudomorphic aggregate of olivine, spinel and pyroxene, new formed magnetite is also evident; e) mesh textured matrix with magnetite crystals and microfracture alignment; f) serpentinite with mm-sized serpentine vein.

A similar difference occurs taking into account UCS, which is usually function of porosity, as more porous rocks offer a lower strength to uniaxial failure. In this case, UCS ranges from about 76.9 to 191.3 MPa, thus highlighting the extremely variable mechanical behavior of studied rocks (Fig. 6a). In particular, NCT are on average characterized by higher UCS and a great variability probably due to the random orientation of microcracks. PCT specimens show a reduction of UCS of about 14% than NCT, testifying their greater weakness although showing a less porous structure. This is a key result, proving the relevant influence that weathering

plays on the physical-mechanical behavior of studied rocks.

The statistical correlation between failure strength and porosity returned no satisfactory relation for both NCT and PCT specimens (Fig. 7a). This is a peculiar result, since usually UCS decreases as porosity increases, due to the weakening action of voids (e.g. Al-Harti et al., 1999; Pappalardo, 2015; Pappalardo et al., 2017). Nevertheless, this is mainly true when porosity is represented by voids, while when cracks occur, their influence on UCS is likely to be conditioned by their orientation. Moreover, investigated specimens fall within a narrow range of

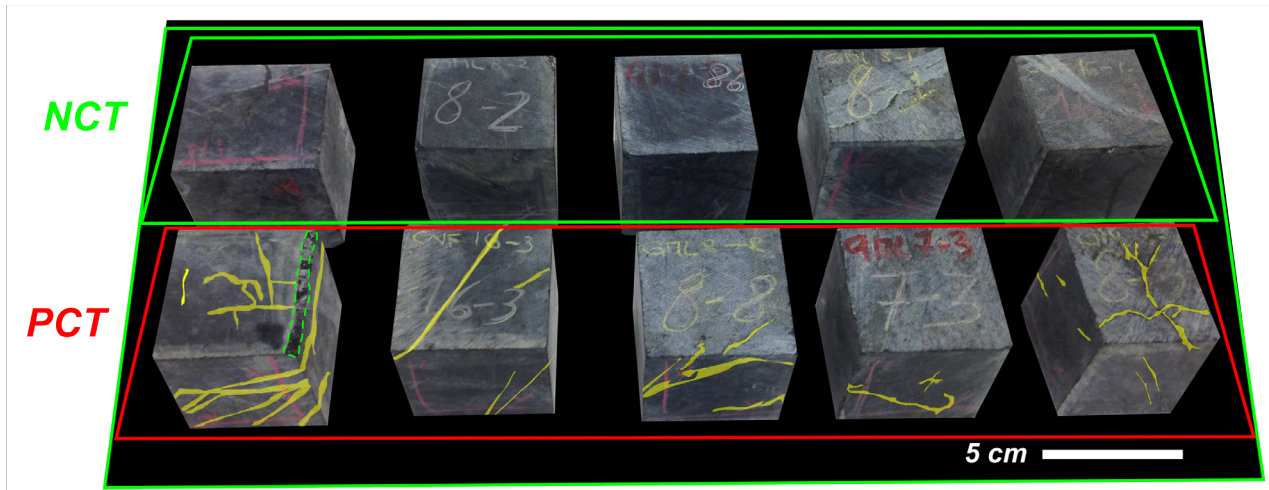


Fig. 4 - Sample cubes of serpentinite rocks without crystallization test (NCT – Not Crystallization Test; upper row) and after crystallization test (PCT- Post Crystallization Test; lower row).

porosity (0.8-4.8%) and the variability of UCS cannot be strictly related to relevant porosity variation. Indeed, Pappalardo et al. (2017) found similar results for the massive variety of lavas from Mount Etna, which showed

a great UCS variability within a narrow range of porosity (2.0-6.6%).

A similar outcome is achieved by taking into account the average P-wave velocity (V_p) values, which are closely dependent on the physical and mechanical properties of intact rocks (e.g. Tugrul and Zarif, 2000; Torok and Vasarhelyi, 2010; Pappalardo et al., 2016a). The correlation between V_p and UCS is described herein by a positive, although scattered, trend (Fig. 7b). Such outcome is in accordance with recent literature works, underlining how there is a poor relation between such two properties, especially for crystalline rocks (e.g. Pappalardo et al., 2016b).

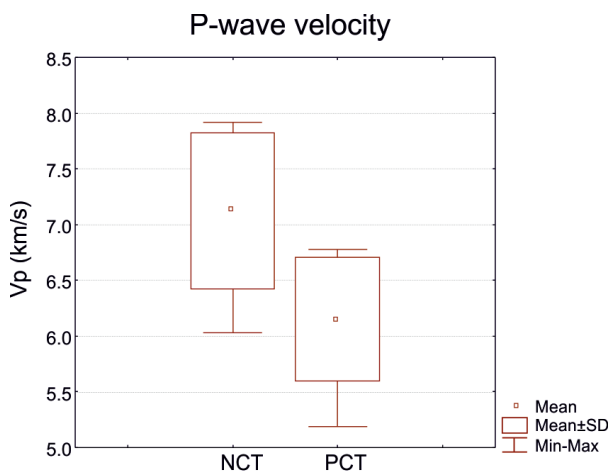


Fig. 5 - Compressional wave velocity values before and after the salt crystallization tests.

5. CONCLUSION

The present study focuses on serpentinite rocks named “Verde Calabria”, which are widely employed and marketed for building and ornamental purposes since pre-historical times.

Although characterized by the same mineralogical content and microstructural features, the behavior of the

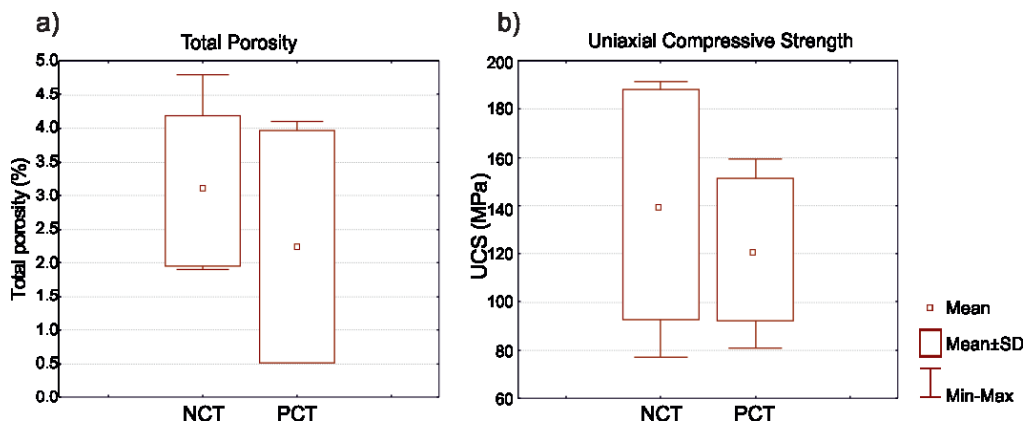


Fig. 6 - a) Box plots showing the statistical variability of UCS; and b) porosity of the studied serpentinite rocks.

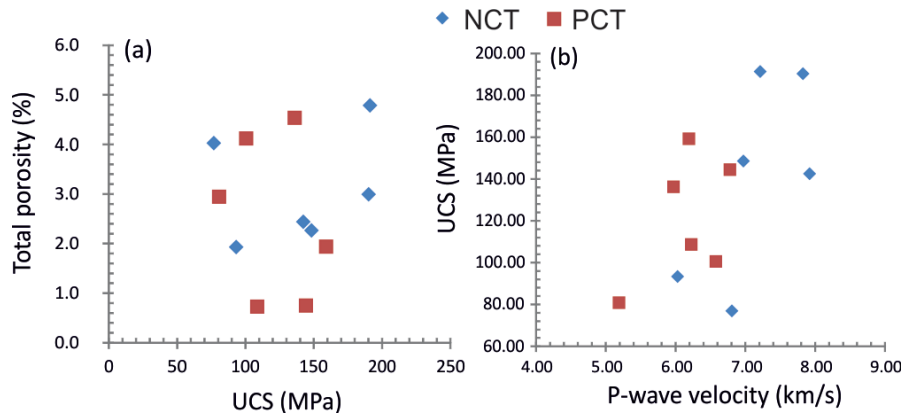


Fig. 7 - a) Porosity vs UCS; b) UCS vs Vp plot.

studied serpentinite rocks is strongly affected by veins and microfractures distribution and by crystallization of salts. Indeed, the obtained results showed that the transformation of the porous system within the serpentinite rock due to the crystallization of salts inside the fractures may trigger and/or accelerate the rock decay process, also due to the worsening of the main mechanical properties of the rocks.

As a matter of fact, this process (crystallization of salts and microfracturing) particularly refers to the stone materials outdoor (e.g. exposed to the action of rain, wind, sun, cold) and, at a lesser extent, to the ornamental materials that are employed indoor.

Results highlighted the importance of defining in detail the petrographic, mineralogical and physical-mechanical properties of these rocks, in order to better plan their suitability for different uses and for construction purposes, as well as for taking right decisions when interventions of restoration have to be carried out on monuments and buildings.

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