Chemical-physical agents and biodeteriogens in the alteration of limestones used in coastal historical fortifications

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Abstract

The alteration of rocks is usually due to the chemical-physical processes that are initially established on the outer surface of the stone and gradually proceed towards the inner matrix. The chemical alteration generated by the interaction with atmospheric agents (weathering) involves the transformation of the mineral phases constituting the rock that are less stable in the current climatic conditions. That often leads to the formation of new secondary phases more stable with respect to the alteration. However, among these phases are often present some very soluble and hygroscopic phases (i.e., soluble salts, clay minerals) that cause inner degradation of the rock, due to their physical-mechanical actions (inner crystallization pressure, hydration dilation). In the case of carbonate rocks (limestone, sandstone with carbonate cement, etc.), the dissolution is the more frequent process, especially when the monuments were located within the cities, due to the acid meteoric precipitations (with H_2CO_3, H_2SO_4) that lead to the sulfation of carbonate matrix with formation of gypsum, very harmful to the stone. When the rock (e.g., clay-arenaceous limestones) naturally contains hygroscopic phases inside the matrix (i.e., marine salts, phyllosilicates) and they are also porous (> 20%), the physical degradation is accelerated, with decohesion of the mineralogical matrix (between the crystalline granules) and consequent disintegration of the stone. In the rock-atmosphere interaction often occurs the presence of biodeteriogens (plants, fungi, lichens, micro-organisms, etc.), which negatively participate and in various ways in the processes of rock alteration.

The research aims to define the chemical-physical alteration factors on the limestones exposed to different bioclimatic and biogeographic contexts (Mediterranean and Atlantic), taking two study-case monuments located in the Italian and in the Portuguese coasts. In the study presented in this paper the preliminary results of the case-study of Cagliari fortifications have been discussed. In the study the different vascular plants present on stone surface and crevices and their different role in the degradation of limestone rocks have been also studied.

Keywords: Petrography, Chemistry, Botany, Microbiology.

1. Introduction

The sedimentary rocks (e.g., limestone, dolomite, sandstone, etc.) are widely used in the construction of many Italian historical buildings monuments or other Mediterranean countries.

The alteration of carbonate rocks is usually induced by chemical processes. Among these, the main one is the dissolution of the "cement" due to the weathering, which consequently lead to physical type processes, such as the formation of secondary porosity. The latter leads to a decohesion of the matrix, a decrease in mechanical strength and subsequent loss of surface material.

The chemical-physical decay is also due to the presence in the rock of clay minerals and sea salts. In fact, the hygroscopic volume variations of these phases lead to a physical decay with a decrease of mechanical strength, so making the limestone easily degradable.

The decay in the structural stone elements of the monument (e.g., ashlars in the wall, column, jambs, etc.) can lead to a strong retreat of vertical profile of the facade, or detachment of the material portions from the decorative working parts, due to exfoliation and flaking processes, finally with serious static-structural criticality in the buildings.

Moreover, the presence of biodeteriogens (e.g., fungi, bryophyte, lichens, vascular flora) is a further alteration agent that increase the decohesion process especially on the stone surface.

Fig. 1- Aerial view of Santa Croce's area

The project aimed to study the decay of sedimentary rocks used in monuments located in cities with different bioclimatic and biogeographic contexts: Mediterranean and Atlantic.

In the present paper, the first case study on the geomaterials of the Santa Croce's walls (XVI cent., within the *Castello* district), belonging to the low-medieval fortifications of Cagliari (southern Sardinia, Italy) has been presented. From the beginning of the sixteenth century the area of *Santa Croce* (Figs. 1, 2) is interested by a transformation that will lead to the construction of a pentagonal bastion (sector 6 of Fig. 2) integrated in the 18th century by a counter-guard (sector 4, Fig. 2) and low flank (sector 5, Fig. 2, 3). Modifications, collapses and reconstructions in the period between 1568-1578 and recent restorations offer the possibility to observe different lithologies used to realize the works. In particular, the west flank of the bastion preserves the original stone that - as the archival documents indicate – during the Spanish Kingdom of Felipe II had to be cut in three main dimensions (Casu, 2002).

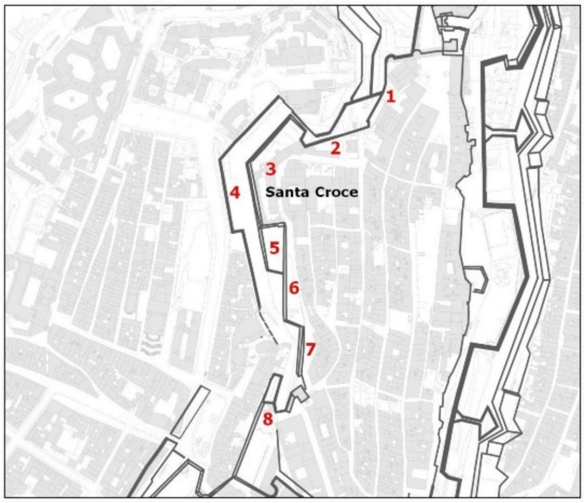

Fig. 2- West front of Castello's fortifications divides in different sector. 1: Bastion of *La Concezione*, 2: Curtain of *San Guglielmo*, 3: Bastion of *Santa Croce*, 4: Counterguard of *Santa Croce*, 5: Low flank of *Santa Croce*, 6: Curtain of *Santa Chiara*, 7: Curtain of *de Cardona*, 8: Bastion of *Balice*

In the ancient walls three main local Miocene sedimentary stones were used: *Pietra Cantone*, *Tramezzario*, *Pietra Forte*. Given the wide availability in the territory around Cagliari and its better workability, these limestones has been widely used in the historical buildings of all periods from Nuragic, to Phoenician-Punic, Roman and medieval (Columbu et al. 2015a; Columbu and Pirinu 2016). However, in the presence of humidity or circulating aqueous solutions, this limestone shows frequently decay problems (Columbu et al. 2016) when it is not protected by other materials (e.g., lime plaster in the wall, etc.). In the present work, the preliminary results and methodological approaches of a study aimed to define the decay processes of limestones and biodeteriogens present in the walls of the fortress are illustrated.

Fig. 3- Low flank of *Santa Croce*

2. Miocene sedimentary rocks

The Miocene limestones outcropping in the Cagliari area are frequently used in the civil and historical architecture. These rocks belong to the sedimentary and volcanic stratigraphic sequence widely outcropping from south to north Sardinia within a complex geological-tectonic context of Sardinia (Advokaat et al, 2014; Casula et al, 2001; Cherchi & Tremolieres, 1984) called *Fossa Sarda* graben (Vardabasso, 1962). The Miocene stratigraphic sequence of Cagliari area mainly consists (from bottom) of the following three facies: clays (*Argille del Fangario*), sandstones (*Arenarie di Pirri*), marly limestones (*Pietra Cantone*), biocalcarenites (*Tramezzario*) and the biohermal limestones (*Pietra Forte*) (Barroccu, 2010; AA.VV., 2005; Barroccu et al, 1981; Cherchi, 1971; Gandolfi & Porcu, 1962; Pecorini & Pomesano Cherchi, 1969). The *Pietra Forte* limestone is compact and it shows a good physical-mechanical resistant, but it is difficult to work.

The *Pietra Cantone* rock is a marly limestone characterised by low cementing degree, high porosity (26-38 vol%). For these reasons, it is more easily workable than the *Pietra Forte*. $CaCO_3$ content generally is about 75-80%, but it can vary between 64 and 89% (Barroccu et al, 1981) depending on the different areas of Cagliari and on the depth of sedimentation. The *Pietra Cantone* generally shows a variable clay component (ranging from 10 to 30%) within the geological formation.

3. Methodological techniques

3.1 Petrographic and physical methods

The mineralogical and petrographic analysis of sedimentary rocks was performed on thin sections under the polarizing microscope (Zeiss photomicroscope Pol II).

For physical tests, cubic specimens (size = 1.5 • 1.5 • 1.5 mm) were dried at 105 ± 5°C and the dry solid mass (m_D) was determined. The solid phases volume (V_S) of powdered rock specimens and the real volume (with $V_R = V_S + V_C$, where V_C is the volume of pores closed to helium) of the rock specimens were determined by helium Ultrapycnometer 1000 (Quantachrome Instruments). The wet solid mass (m_W) of the samples was determined after water absorption by immersion for ten days. Through a hydrostatic analytical balance, the bulk volume V_B ($V_B = V_S + V_O + V_C$ where $V_O = (V_B-V_R)$ is the volume of open pores to helium) is calculated as: $V_B = [(m_W-m_{HY})/\rho_W T_X]100$, where m_{HY} is the hydrostatic mass of the wet specimen and $\delta_W T_X$ is the water density at a temperature T_X. Total porosity (P_T), water and helium open porosity ($\Phi_O H_2O$; $\Phi_O He$), closed porosity to water and helium ($\Phi_C H_2O$; $\Phi_C He$), bulk density (ρ_B), real (ρ_R) and solid density (ρ_S) are computed as:

$$\Phi_T = [(V_B-V_S)/V_B]100$$
$$\Phi_O H_2O = \{[(m_W-m_D)/ \rho_W T_X]/V_B\}100$$
$$\Phi_O He = [(V_B-V_R)/V_B]100$$
$$\Phi_C H_2O = \Phi_T - \Phi_O H_2O$$
$$\Phi_C He = \Phi_T - \Phi_O He;$$
$$\rho_S = m_D/V_S; \rho_R = m_D/V_R; \rho_B = m_D/V_B$$

The weight imbibition coefficient (CI_W) and the saturation index (SI) were computed as:

$$CI_W = [(m_W-m_D)/m_D]100$$
$$SI = (\Phi_O H_2O/\Phi_O He) = \{[(m_W-m_D)/ \delta_W T_X]/V_O\}100$$

The punching strength index was determined with a Point Load Tester (mod. D550 Controls Instrument) according to the International Society for Rock Mechanics (1972; 1985) on the same cubic rock specimens used for other physical properties.

The resistance to puncturing (I_S) was calculated as 2 P/D_e, where P is the breaking load and D_e is the "equivalent diameter of the carrot" (ISRM, 1985), with $D_e = 4A/\pi$ and $A = WD$, where W and 2L are the width perpendicular to the direction of the load and the length of the specimen, respectively. The index value is referred to a standard cylindrical specimen with diameter D = 50 mm for which I_S has been corrected with a shape coefficient (Fs) and calculated as: $I_{S(50)} = I_S F = I_S (D_e/50)^{0.45}$.

The compression and tensile strengths were calculated by punching index values respectively as: $R_C = I_{S(50)} \cdot F_C$; $R_T = I_{S(50)} / 0.8$, where:

F_C (conversion factor) is between 15 and 50 as function of size, characteristics and anisotropy of samples.

3.2 Flora study methods

The basis of the current analysis, mainly for what regards the non-native species, is the latest updated checklist of the Sardinian alien flora (Puddu et al, 2016), supplemented by the recent works about the Italian vascular native and alien floras (Bartolucci et al, 2018; Galasso et al, 2018). Vascular plant taxa have been classified as archaeophytes or neophytes based on their introduction before or after 1492/1500 C.E., respectively. Concerning the taxa for which doubts still persist about their status (alien or native), we have preferred to apply an attribution of doubtful alien (D).

The status of invasiveness has followed that proposed by Richardson et al. (2000) and subsequently elaborated and reviewed by Pyšek et al. (2004) and Richardson et al. (2011). In particular, Sardinian taxa have been attributed to the classes of invasive, naturalized and casual plants on the basis of the cited literature, as well as on our field observations.

Regarding biological forms, Raunkaier life form classification (Raunkaier, 1934) has been followed, using the variations and abbreviations used by Pignatti (1982), while geographic origin of the alien plants is based on what reported by Puddu et al. (2016) or in the relative literature.

3.3 Microbiological methods

The state of conservation of stone materials (such as limestone) present in relation to the characterization of bacterial and fungal populations has been carried out through classical and molecular culture methods. The latter, together with the detection of total microbial counts in the air, were fundamental when the deterioration was not yet visible, representing crucial preventive tools in relation to the altering process induced by microorganisms.

Preliminarily, an inspection was carried out in the St. Croce area, where all the areas were carefully observed and on the surfaces in which an organic patina was visible, sampling was carried out. The latter was conducted by specialized personnel, at different points of the site of historical and artistic interest in the same archaeological area.

For microbiological research, different types of surfaces were considered on which, through the use of the sampling technique with buffer, the microorganisms responsible for the alterations were searched. The samples were transported to the laboratory under controlled temperature conditions, where they were treated for the research and isolation of the microorganisms of our interest. The land used for their research and isolation were Plate count agar (PCA) and Chloramphenicol Glucose, yeast extract Agar (CGYEA).

4. Results and discussion

4.1. Petrographic and physical analysis

Pietra Forte is a cliff limestone (i.e. bioherma or biostroma facies; Pecorini & Pomesano Cherchi, 1969). It consists mainly of calcite with whitish colour and yellowish spots. It is rich in remains of molluscs and especially algae (lithotamins), big foraminifers (Amphistegin, Miogypsina, Elphidium, Rotalia, etc.) and briozoic colonies.

Based on the association of planktonic micro-fauna, the *Pietra forte* was referred to the Tortonian and, according to affinity with other similar formations present in the Gulf of Oristano, Messinian and perhaps partly also Pliocene (Cherchi, 1974).

Pietra Forte shows a high physical-mechanical strength. It is a rock more difficult to work with respect to other *Tramezzario* or *Pietra cantone* limestones. This stone was employed for the ashlars in Santa Croce walls together other *Pietra Cantone* and *Tramezzario* limestones. This rock generally shows a high variability of apparent density (from 2.56 to 2.71 g/cm^3) as function on the porosity (with low values, about 5% vol.) and solid density of calcite (2.71 g/cm^3). The mechanical strength is generally high with indirect compression strength (R_C) ranging from 14 to 61 MPa, but with high variability of values, due to the variable presence of porosity and fissures at different scales. Also the indirect tensile strength (R_T) shows a high variability: from 3 and 10 MPa. Due to its petrographic features and good physical-mechanical resistance, the *Pietra Forte* limestone does not show advanced forms of alteration.

Tramezzario is a clayey limestone with amount of $CaCO_3$ about 85-88% (Barroccu et al. 1981). It generally shows a whitish colour, minute clasts and organogenic fragments. According to Pecorini & Pomesano Cherchi (1969), based on the present macro-fauna (*i.e.* fragments of lamellibranchs and gastropods) and the microfauna this rock was referred to the Tortonian. It is an average compact limestone with both good mechanical characteristics and workability. For this reason, it has been widely used in various ancient buildings until the beginning of the last century. It was also used for the ashlars of walls in the fortification of Santa Croce. In some cases, due to high micro-fracturing processes (Barroccu et al, 1981), this rock has low consistency and poorly physical-mechanical behaviour. It has a high value range of bulk density (from 1.54 to 1.97 g/cm^3), due to the variable incidence of primary and secondary porosity. The compression and tensile strengths show lower values (on average of 9-13 MPa and 1-2.5 MPa, respectively) with respect to the *Pietra Forte*. Due to a greater porosity, the *Tramezzario* limestone shows macroscopic alteration with evident exfoliation and flaking processes on the stone surface.

Pietra Cantone is a "soft" limestone characterized by an easy workability due to a different physical-mechanical behaviour with respect to the other two limestones. For this reason and its wide availability in the territory around Cagliari, this limestone has been widely used to the historical buildings (Fig. 2) of all periods from Nuragic, to Phoenician-Punic, Roman and medieval (references in Columbu & Pirinu 2016). According to Folk (1959) and Dunham (1962) classifications it can be defined as biomicritic limestone and as wackestone, respectively. Considering the microscopic characteristics and the environment of deposition conditions, it is preferable to define this rock as poorly cemented marly limestones. It has a mainly muddy microcrystalline matrix and variable presence of bioclastic components, with a $CaCO_3$ amount about 75-80%, but it can varies between 64 and 89% (Barroccu et al. 1981) depending on the different areas of Cagliari and on the depth of sedimentation. This rock shows a low cementing degree, with high porosity (on average 26-38% vol.) and bulk density from 1.76 to 1.96 g/cm^3 (according to Columbu et al. 2017), as function on the composition and fabric of stone. The compressive strength values range from is lower, ranging from 4.5 to 9.5 MPa. These values are lower with respect to the unaltered quarry samples, but they are greater with respect to those of strongly altered samples (0.4÷0.8 MPa; Barroccu et al. 1981) taken at the surface of the outcrops. The *Pietra Cantone* shows a variable clay fraction (within the geological formation) and the presence of sea salts. These components represented two important factors together the high porosity of rock. In fact, the weathering processes with a variable humidity and circulating aqueous solutions affect this limestone with evident decay problems (Columbu et al, 2017). In the Santa Croce wall the *Pietra Cantone* was used mainly for the two "garitta" (*i.e.* sentry-box) and for the horizontal decorative frame with half-round section located in the upper side of wall. This latter is now absent due to the evident decay.

Fig. 3- Chasmo-comophytic woody and nitrophilous vegetation (*Artemisio arborescentis-Capparidion spinosae*) on the wall of Castello fortification

4.2. Biodeteriogen characterization

The inventory of the vascular flora of Castello's fortifications amounts to 110 taxa, of which 57% are natives (63 taxa) and 43% non-natives (47 taxa). The total flora includes 104 species, 5 subspecies and 1 hybrid, belonging to 43 families and 92 genera. Within the non-natives species 68% are neophytes (32 taxa), 15% are archaeophytes (7 taxa) and 17% are doubtful alien (8 taxa). The invasive status at local level is recognized to 6 taxa (while, according to the Sardinian alien checklist, it amounts to 19); the naturalized taxa at local level are 32 (in the Sardinian alien checklist they are 15); while the casual adventitious are 9 taxa at local level and 10 at regional one.

Fig. 4- Nitrophilous casmophytic vegetation (*Parietarion judaicae*) on the wall of Castello fortification realized with exagonal ashlars of limestone

The biological spectrum of the native flora reveals that therophytes are the most represented (29 taxa), followed by hemicryptophytes (18 taxa) and phanerophytes (9 taxa). On the other hand, the component of non-native plants is mostly characterized by phanerophytes (27 taxa), followed by therophytes (8 taxa) and geophytes (7 taxa). The chorological analysis of native flora shows the dominance of the Mediterranean elements (47 taxa), with rates much lower for what concerns cosmopolitan and subcosmopolitan taxa (4 and 3 respectively). In Figures 4 and 5 some examples of vegetations present in the wall of Cagliari Fortifications are shown.

Regarding the geographical origin of non-native taxa, the major source is represented by the American component (16 taxa), followed by Mediterranean Basin (11 taxa), and South Africa (6 taxa). The biodeteriogenic taxa are in total 27, 12 of which are native and 15 are non-native.

As regards to microbiological characterization, the most frequently isolated microorganisms were represented by both *Gram* positive and *Gram* negative bacteria, *Bacillus* spp, *Pseudomonas* spp, and mycetes with the genera *Penicillium* spp. Furthermore, the presence of *Cyanobacteria* was detected. Molecular methods for species identification are still ongoing.

5. Conclusions

The stones used in the Santa Croce fortification belong to local Miocene formation with three main carbonate limestones: *Pietra Forte, Tramezzario, Pietra cantone*. These three lithologies generally show a chemical alteration for the dissolution of $CaCO_3$ matrix and sulphation processes with the formation of pitting (little pores) and gypsum crusts on the stone surface.

The *Pietra Cantone* lithology, that shows a good workability but with poor resistant, was mainly used for the decorative parts (*i.e.*, cornice, *garitta*). Due to its petrophysical characteristics, with high porosity (often >30% vol.) and the presence of clay minerals and soluble salts, it shows frequently decay problems. In fact, they are hygroscopic phases and so have cyclic hydration / dehydration mechanisms that lead to a physical decay inside the rock matrix and to a decrease of mechanical strength with formation of various macroscopic alteration forms on the stone surface (e.g., decohesion, exfoliation, flaking).

The *Pietra Forte* and *Tramezzario*, more resistant, were used for the wall ashlars. The first limestone shows a high physical-mechanical resistant with respect to the alteration, because it has a lower porosity without clay/salt phases. *Tramezzario* limestone sometimes shows decay process (mainly surface exfoliation), due to a greater porosity with respect to the *Pietra Forte*.

The study of biodeteriogens present in the St. Croce fortification walls has highlighted the massive presence of floral species. The vascular flora inventory shows the presence of 104 species with 110 taxa, of which 63 are natives and 47 non-natives. The action of these several taxa detected involves negative effects with strong degradation of the rocky substrate, with formation of superficial cracking that then develops more in depth. The fissuring creates preferential ways for the degradation action of other chemical and physical factors and processes induced by atmospheric agents. Some floral species insinuate themselves between the mechanically weaker stone ashlars (usually consisting of *Pietra Cantone* and *Tramezzario*), undermining the original bedding mortars and thus annoying the static features in the outermost portions of the masonry.

The results of the biodeteriogen research also showed a microbial activity in the stone surface represented by a multiplicity of both bacterial and fungal genera, the latter sometimes macroscopically visible.

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