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Macigno sandstone from Garfagnana and Vellano (north-western Tuscany): chemical, mineralogical, petrographic and physical characterization of a building material

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***Abstract* – The sandstones belonging to the flysch-like deposits of the Macigno Formation are nearly the most used stones as a building material in north-western Tuscany. From prehistoric times to date, this stone has been employed to produce a wide variety of stone structures: prehistoric statue-stele, medieval monumental churches, railway infrastructures, town paving and civil buildings. The wide use of Macigno sandstones is responsible for the typical appearance of most towns and villages in north-western Tuscany; in fact, they show the grey to grey-yellowish colour of this stone, which lends them a quaint, old-time look. The aim of this work is the chemical, mineralogical, petrographical, physical and mechanical characterization of the Macigno sandstone from Garfagnana (LU) and Vellano (PT) within a wider research project directed to deepen the knowledge about the alteration and the decay of these sandstones.**

I. INTRODUCTION

For several years the Department of Earth Sciences of the University of Pisa has pursued studies and researches aimed at characterizing the Macigno sandstone of Tuscany in order to understand its degradation processes when exposed to natural and/or anthropic atmospheric events [1-4]. Macigno sandstone from Tuscany has been studied in detail: stratigraphy [5-9], structural geology [10-12], paleontology [13-14], mineralogy and petrography [1-3, 15-18].

The physical-mechanical data determined on this stone are scarce and the degradation processes that occur above all with surface exfoliation phenomena [19], which are clearly visible on the products exposed to natural atmospheric agents, are still not fully clarified.

The aim of this work is to carry out a chemical, mineralogical and petrographic characterization of the sandstone from some outcrops of the Garfagnana and from an active quarry in the province of Pistoia (Vellano quarry).

A petrographic characterization of some samples collected in the active quarry of Matraia (LU) was also carried out. Furthermore, a complete physical-mechanical characterization was carried out on the material from the Vellano quarry, according to European standards for natural stones.

II. GEOLOGICAL SETTING

Garfagnana area is located in northern Tuscany and belongs to Lucca district. From a geological point of view, it is characterized by the tectonic overlap of lithological successions of rocks belonging to the External and Internal Ligurian units (allochthonous) and to the Tuscan Series (Autochthonous). It is believed [20] that the Garfagnana represents the internal margin of the Apennines which began its extension during the Piacenzian-Zanclean and which subsequently evolved into a graben. For this reason, the main structures consist of normal faults with SW immersion and antithetical faults with NE immersion, which are part of a single extension system. To the NW this graben is limited by a transversal element which, from Fivizzano going south along the Secchia valley, separates Garfagnana from Lunigiana. The SE graben ends south of Barga, and there is no evidence of any structural and transversal element. In the Pistoia Apennines, Macigno sandstone belonging to the non-metamorphic Tuscany series emerges extensively throughout the ridge. This formation is tectonically superimposed on the Cervarola-Falterona Unit, which is in turn superimposed on the innermost turbiditic successions of the Umbrian domain [21]. The succession of rocks outcropping in Garfagnana includes the following formations (geological map of the Castelnuovo di Garfagnana [22]:

- Tuscan Nappe:
  - Calcare Cavernoso: dark dolomites and

brecciated dolomitic limestones, with a characteristic vacuolar structure (Lower Carnic-Rhaetian);

- Calcari a Rhaetavicula contorta: limestones, dolomitic limestones and dolomites, stratified, from grey-dark to black, fetid to percussion, with intercalations of dark silty marls that become rare or absent in the upper portion of the formation (Rhaetian);
- Calcare massicio: unstratified limestones and dolomitic limestones, from light grey to dark grey (Hettangian);
- Calcari ad Angulati: calcilutites and marly calcilutites with clay or silty marl (Hettangian – Upper Lias);
- Rosso Ammonitico: calcilutites, sometimes marly calcilutites, stratified or nodular, from pinkish-red and light grey or havana, often showing concoid fracture, with rare intercalations of thin levels of marls or red limestone argillites (Sinemurian – Upper Pleinsbach);
- Calcare selcifero di Limano: grey siliceous calcilutites, well layered with nodules and lists of light-grey flint, with intercalations of fine calcarenites, sometimes graded, and thin layers of marl and brown silty-clayey materials (Upper Pleinsbach).
- Calcari e marne a Posidonia: grey or grey-green marl and limestone marl, sometimes with splinter fracture, with intercalations of marly calcilutites, grey or grey-green siliceous calcarenites, also graded, with rare flint lists and nodules (Lower / Medium Toarciano-Middle / Upper Bajocian);
- Calcare selcifero della Val di Lima: calcarenites graded from fine to coarse and siliceous calcilutites from grey to dark-grey, sometimes fetid to percussion, with abundant nodules and lists of black flint and rare intercalations of marls or clayey marls (Middle / Upper Bajocian - Upper Kimmeridgian / Upper Titonian);
- Diaspri: radiolarites and red, green, gray, brick red, or dark-grey flints, intensely fractured, , with very thin intercalations of siliceous red, green or grey-green clayey material (Titonian);
- Maiolica: white or light-gray calcilutiti and siliceous calcilutiti, with concoid fracture. In the upper part of the formation there are nodules and lists of light-gray flint (Upper Titonian / Berriasian - Upper Berriasian / Lower Valanginian);
- Scaglia toscana: mudstones, carbonate pelites, limestones, limestone-siliceous, calcarenites and rudites (Lower Cretaceous - Oligocene);
- Macigno: quartz-feldspathic sandstones, alternating with argillites and siltites (turbidites) (Middle / Upper Oligocene - Upper Miocene);

- Ligurian Units:
  - Breccias and ophiolitic elements (Cretaceous);
  - Heterogeneous complex mainly argillitic, with chaotic structure, with layers and blocks of sandstones, limestone, marly limestones, and siliceous “Palombino” type limestone (Cretaceous - Eocene). Locally included in the complex: light grey and white marly limestones and limestones (Paleocene – Lower / Middle Eocene).
  - Flysh to Helmintoids: marly limestones, marls and limestone sandstones (Upper Cretaceous).
- River-lake deposits:
  - Pebbles with prevalent elements of Macigno (Pleistocene) sandstone.
- Quaternary deposits l.s.

III. ANALYTICAL METHODS

For the purpose of this work, we have collected and studied 47 samples of Macigno sandstone: 35 samples from Garfagnana (Sillano, SI, Scorticchiaia, SC, Tereglio, PG, Isola, FL, and Matraia, PM); 6 samples Matraia (MT) and 6 samples from Vellano (VE).

Below are the analytical techniques used for this work:

- chemical analysis through X-ray fluorescence [22-23] for the determination of major and minor compounds (Na_2O, MgO, Al_2O_3, SiO_2, P_2O_5, K_2O, CaO, TiO_2, MnO, Fe_2O_3). The measurement uncertainty results between 4-7% by weight for concentrations <1%, between 2-4% for concentrations between 1 and 10% and around 1% for concentrations > 10% [24].

- CO_2 content was measured by calcimetry to estimate the amount of $CaCO_3$ in the tested sample [25]. The content of calcite was calculated with reference to a calibration curve constructed by linking the volume of CO_2 developed by acid attack of the powdered rock with the amount of pure $CaCO_3$.

- mineralogical analysis through X-ray diffractometry (XRD) $\lambda = 1.5406$ Å, angle range 4-66°2θ;

- petrographic analyses: transmitted light microscopic observation of thin sections (Zeiss Axioplan microscope);

- physical properties of the stones like real (ρ_r) and apparent (ρ_a) density, water absorption coefficient by capillarity, water absorption at atmospheric pressure, total and open porosity and saturation index have been determined following EN standards [26-28];

- real density (ρ_r) has been determined using a gas pycnometer (Ultrapycnometer 1000 by Quantachrome Corporation) [26]. The measurements were performed on approximately 10 g of very-fine-grained powders dried at 105 ± 5 °C for 24 h under the following experimental conditions: ultrahigh purity compressed Helium with outlet pressure of 140 kPa; target pressure, 100 kPa; equilibrium time, automatic; purge mode, 3 minutes of continuous flow; maximum number of runs, 6; number of averaged runs, the last three;

- apparent density (ρ_a) has been determined by ratio between dry mass and volume of each specimen. The specimens were placed in a stove at 60° C until the dry weight was reached, (i.e. when the difference between two successive weighing at an interval of 24 h is not greater than 0.1 % of the mass of the specimen). Then the specimens were immersed in distilled water following [26]. The volume of the specimens was measured by means of a hydrostatic balance on water-saturated samples [29];

- water absorption coefficient by capillarity has been determined on the same samples used for apparent density determination following EN 1936:2007 [27]. Measurements were taken after 1, 3, 5, 15, 30, 60, 120, 180, 240, 300, 360, 420, 480, 1440, 2880 minutes;

- determination of water absorption at atmospheric pressure has been carried out on the same specimens [28].

- the total porosity has been calculated according to (1)

$$P\ (\text{vol. }\%) = 100 \cdot (1 - \rho a / \rho r) \qquad (1)$$

- dilatation by imbibition: the standard for the physical-mechanical characterization of stone materials does not consider yet the use of determining the volumetric variation of a stone material subjected to imbibition. However, the dilatation by imbibition of some rocks is certainly not to be underestimated and knowing its amplitude can allow to predict the behaviour of the material used.

The behaviour of the sandstone specimens completely immersed in water during the was studied by means of a specially constructed dilatometer [30].

- mechanical properties: uniaxial compressive strength [31]; uniaxial compressive strength after 48 freezing/thaw cycles [32]; flexural strength under concentrated load [33]; flexural strength under concentrated load after 48 freezing/thaw cycles [32]; abrasion resistance [34]; determination of rupture energy [35].

IV. RESULTS AND DISCUSSION

The chemical analysis has been carried out by means of XRF. The average chemical composition of the samples is reported in Table 1.

The rocks studied vary in colour from grey, more or less dark in fresh samples, to yellowish grey in the more altered samples. There are no significant differences in the chemical composition of the samples from the different studied areas, except for the CaO and CO_2 quantity, mainly linked to the carbonate contents.

The main mineralogical phases identified by X-ray diffractometry are quartz, plagioclase, K-feldspar, phyllosilicates, and calcite. The latter phase is present in large quantities with rare exceptions.

The analyses carried out on the fraction <4 µm allowed to identify micas (illite), chlorites s.l. (chlorite s.s. + interlayered chlorite / smectite and, in some samples also corrensite) and kaolinite. The chlorite / smectite interlayers of the studied rocks have a variable content of chloritic layers, generally <50%.

Table 1: Chemical composition (wt%) of the analysed sandstones. Fe_2O_3= total iron expressed as Fe_2O_3. SI: Capanne di Sillano (LU); SC: Scorticchia (LU); PG: Tereglio (LU); FL: Isola (LU); PM: Matraia (LU); VE: Vellano (PT).

Sample	H_2O	CO_2	Na_2O	MgO	Al_2O_3	SiO_2	K_2O	CaO	Fe_2O_3	Other <1%
FL mean	**2,46**	**2,92**	**2,65**	**5,48**	**13.36**	**61,49**	**2,25**	**5,02**	**3,80**	**0,58**
FL st. Dev.	0,22	1,22	0,21	1,14	0,37	2,20	0,26	1,78	0,61	0,07
PG mean	**2,74**	**1,45**	**2,25**	**6,26**	**14,09**	**62,35**	**2,54**	**3,12**	**4,61**	**0,58**
PG st. Dev.	0,14	0,98	0,23	0,37	0,69	2,61	0,11	1,42	0,5	0,08
PM mean	**2.44**	**2,81**	**2,27**	**5,87**	**13,49**	**60,63**	**2,66**	**5,13**	**4,16**	**0,56**
PM st. Dev.	0,31	2,16	0,20	1,13	0,81	3,07	0,07	2,97	0,27	0,10
SC mean	**2,20**	**0,78**	**2,56**	**6,78**	**15,22**	**62,11**	**2,88**	**2,63**	**4,10**	**0,57**
SC st. Dev.	0,31	0,11	0,07	0,69	0,24	1,32	0,10	0,47	0,14	0,11
SI mean	**2,6**	**1.04**	**2,39**	**6,21**	**14,99**	**62,59**	**2,66**	**2,57**	**4,4**	**0,57**
SI st. Dev.	0,31	0,94	0,19	0,79	0,7	1,66	0,23	1,38	0,31	0,1
VE mean	**2,05**	**3,04**	**2,18**	**6,52**	**14,62**	**58,79**	**2,5**	**5,08**	**4,65**	**0,56**
VE st. Dev.	0,52	2	0,24	0,51	0,28	4,28	0,11	2,52	0,3	0,08

Corrensite, the interlayered chlorite/smectite consisting of a regular alternation of smectite layers and chloritic layers (with a 1:1 ratio), has been identified in almost all samples, with the exception of the samples from Tereglio and Matraia although the presence of this phase was observed also in the latter locations [2].

From a petrographic point of view, the rocks coming from the quarries of Matraia and Vellano are felsdpatic sandstones, with a generally medium and medium-fine grain size, moderately classified, having a grain-sustained texture, with detrital grains from angular to sub-angular and of colour variable from grey to bluish-grey. The contacts between the granules are generally point-like, with some linear contact. The matrix is essentially phyllosilicate-bearing.

Modal analysis has shown that the rocks analysed are constituted in order of abundance by quartz and plagioclase, with subordinate quantities of K-feldspar, phyllosilicates and lithic fragments. According to Folk's sandstone classification (Fig. 1) the analysed rocks can be classified as arkose. Regarding the physical properties, it emerges that the real density values vary from 2,680 g/cm^3 to 2,713 g/cm^3. Based on the real density values, the samples coming from the natural outcrops of the Garfagnana can be classified as medium to heavy rocks, while the samples coming from the Vellano quarry can be classified as heavy light.

The average values of the water absorption by atmospheric pressure coefficients vary from 0.7 wt.% to 1.5 wt.%, while the total porosity varies from an average of 1.1% to 4.1% by volume. The water absorption by capillarity, measured on the samples taken from the Vellano quarry, have a rather constant trend, with a strong initial absorption and then gradually decreasing by passing of time. The water absorption coefficients by capillarity, measured perpendicular to the anisotropy planes (C_1) are quite constant and the average is 2.39 ± 0.11 g/m^{2}s$^{0.5}$, while those measured parallel to the anisotropy planes (C_2) have an average value equal to $2{,}36 \pm 0{,}07$ g/m^{2}s$^{0.5}$.

Table 1: Average chemical compositions of Macigno sandstone samples from Gafagnana (FL, PG, PM, SC, SI) and Vellano (VE) determined by XRF analysis: the major elements are expressed in oxides wt.%., st. D., standard deviation.

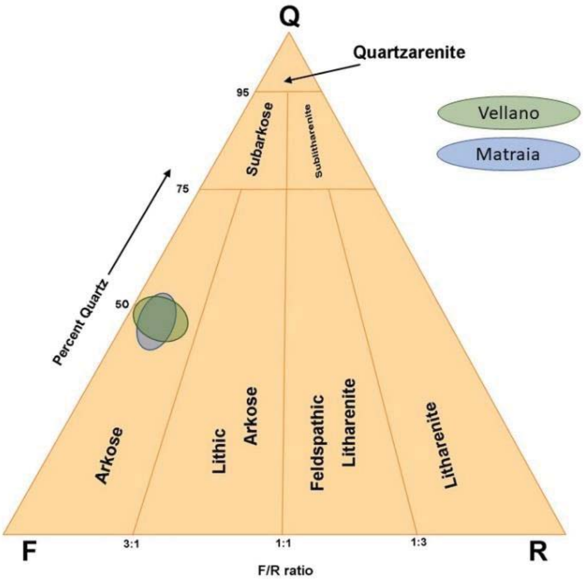

Fig. 1: QFR diagram for Folk's sandstone classification. The analysed samples can be classified as arkose.

By comparing the average linear expansion curves obtained it is noted that the samples measured orthogonally to the direction (O) undergo greater elongations than those measured parallel to them (P). This study also made it possible to observe that, by performing repeated water imbibition cycles and drying in a stove at 60 °C, the extent of the elongation undergoes significant variations between the first and second measurement cycles, while remaining constant in the subsequent ones. cycles. The water absorption values also follow the same trend, that is, they increase between the first and second measurement cycles while they remain constant between the second and third cycles.

V. CONCLUSIONS

The Macigno sandstone, object of this work, is a stone material widely used as a building material in western Tuscany, and it is still widely used to produce urban decorations and paving in historical centres.

For this, we studied samples from outcrops of the Garfagnana and from an active quarry, located near the town of Vellano (Pistoia province). The chemical, mineralogical and petrographic characterization was performed on all the studied samples, while the physical and mechanical characterization according to the European standard, only on the fresh samples taken from the Vellano quarry.

Comparing the absorption capacity at atmospheric pressure and the smectite percentage of the chlorite/smectite interlayers of the studied samples, a positive correlation emerged, confirming what had been observed in previous works [3-4].

Regarding the mechanical properties, it appears that the sandstone samples from Vellano have worse characteristics than those of Matraia and Pian di Lanzola quarries, but above all the sandstone of Vellano fears frost (decrease> 25% in the uniaxial compressive strength, after freezing cycles /thaw). Matraia sandstone remains the one that has better mechanical properties [35].

The data on linear water dilation confirm what was previously observed on samples from Lunigiana [3-4] and on samples from the Matraia quarry [37], namely that these rocks tend to stretch when they are immersed in water and that this elongation is differential, i.e. greater along the stratigraphic planes of the rocks and less along the parallel direction [4].

In conclusion, with this work we characterized some samples of the Macigno sandstone of the Vellano quarry from a chemical, mineralogical, petrographic and physical-mechanical point of view; we confirm the influence of phyllosilicate minerals on the degradation of this rock; we confirm that the linear water expansion of these rocks is greater along the direction perpendicular to the planes of stratification and smaller in the parallel one; moreover, the linear water expansion is greater during the second measurement, but remains broadly unchanged during the third and subsequent others.

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