Minero-Petrographic Features and Physico-Mechanical Properties of the Macigno Sandstone in the Vellano Area (Pistoia, Tuscany, Italy)

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Abstract

The sandstones belonging to the terrigenous deposits of the Macigno Formation (Late Oligocene-Early Miocene) were widely used as building stones in Tuscany (Italy) for the wide distribution of their outcrops and the good qualities of the extracted stone. This research reports the petrographic and mineralogical data, and the physical and mechanical test values collected to evaluate the main technical properties of the Macigno sandstones from the Vellano area, with the purpose of comparing the quality of the stones extracted in this area with those from other quarries in north-western Tuscany. The results, obtained analysing 21 samples from the Vellano quarry and its surroundings, show that sandstones cropping out here are characterized by medium to medium-fine sand-sized grains made up of quartz, K-feldspar, plagioclases, phyllosilicates, lithic fragments, and accessory. Clayey materials and calcite are present as matrix and cement, respectively. The clay fraction is made up of mica-like minerals, chlorite, chlorite/smectite interlayers and, in some samples, corrensite and kaolinite. From the physical and mechanical point of view the analysed samples show low porosity and high flexural and compressive strengths. Compared to the other Macigno sandstone samples from north-western Tuscany, the best samples of Vellano stone show rather comparable mechanical resistance than those quarried at Matraia in Lucca province, which today is another active quarry of sure interest for the good quality of the extracted material.

Keywords: sandstone classification, turbidites, geochemistry, Northern Apennines, Oligocene-Miocene

Introduction

The siliciclastic foredeep turbidites of the Macigno Fm. (Late Oligocene-Early Miocene) belonging to the Tuscan Nappe sequence outcrop extensively in Tuscany [1]. The Macigno siliciclastic rocks are represented by alternating beds of different thickness and grain size forming a succession of strata characterised by the presence of prevailing turbiditic sandstone with minor siltstones, mudstones, marls and shales [2][3]. There are numerous research works that have characterized the Macigno sandstone for defining the stratigraphic-depositional framework and the origins [4][5][6][7][8][9][10][11]. Some authors have highlighted how the westernmost and oldest outcrops, indicated as *Coastal Macigno*, have different characteristics from the *Apennine Macigno*, such to represent an independent depositional system [10][12]. Chemical, mineralogical, petrographic characteristics of the Macigno sandstone have been reported in many papers [13][14][15] and degradation processes of this stone have been studied [16][17][18][19][20] as part of research activities devoted to understanding the mechanisms of degradation of this stone and improving the conservation and protection of objects built with it. Scarce information is available for the Macigno sandstones outcropping in the Pistoia area despite the fact that they have been extensively used in local historical buildings and, today, there is one of the few active quarries in Tuscany.

The aim of this work was to carry out a chemical, mineralogical and petrographic characterization of the Macigno sandstone samples coming from the Vellano area in Pistoia province (Fig. 1) together with a determination of physico-mechanical properties of the material coming from the Vellano quarry and its surroundings. A comparison with the characteristics and properties of the sandstones quarried in western Tuscany will be made as a further purpose of the work.

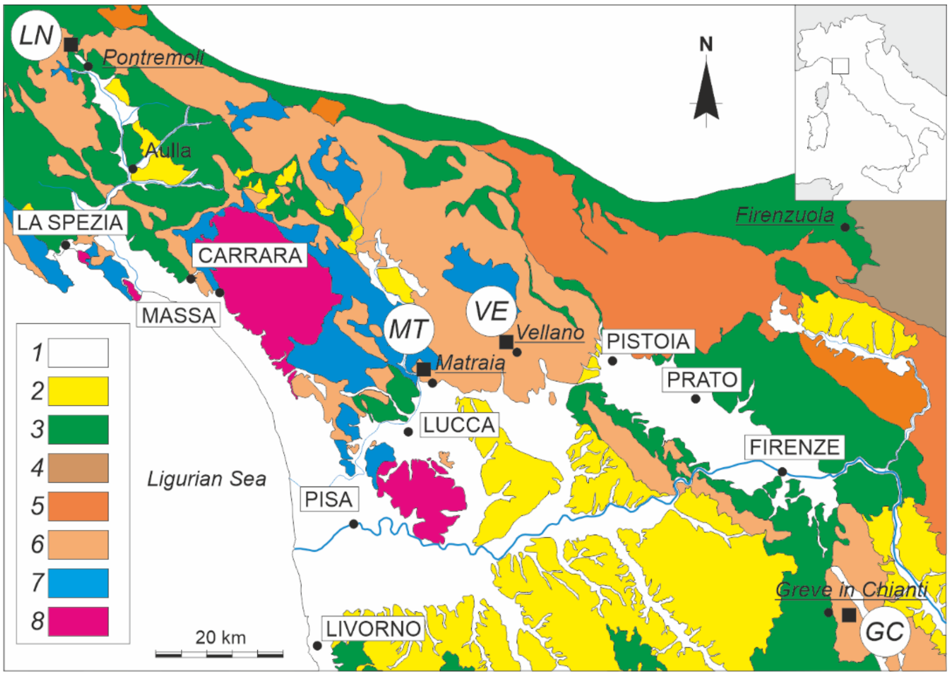

Fig. 1: Geological maps of the northern Tuscany with the main outcrops of Macigno sandstone Fm. 1 = Pliocene-Quaternary continental and coastal deposits; 2 = Pliocene-Quaternary marine deposits; 3 = Ligurian-Piedmont Domain (Oceanic succession affected by HP metamorphism; Internal Ligurian Domain; External Ligurian Domain; Sub-Ligurian Domain); 4 = Umbro-Marchean Domain (Sandstones-marly flysch: sandstones, and marls with olistostromes); 5 = Tuscan Domain, Non Metamorphic Succession, External sandstone flysch: sandstones, siltstones, shales and marls with olistostromes (Cervarola Unit), Chattian-Langhian; 6 = Tuscan Domain, Non Metamorphic Succession, Internal sandstone flysch: sandstones, siltstones with olistostromes (Macigno sandstone and M. Modino sandstones), Chattian-Langhian; 7 = Tuscan Domain, Other formations of Non Metamorphic Succession from Early Cretaceous to Late Triassic; 8 = Tuscan Domain, Metamorphic Succession from Cambrian to Early Miocene (modified by Carmignani & Lazzarotto, 2004 [1]).

Materials And Methods

Twenty-one samples of Macigno sandstone from Vellano quarry and its surroundings have been collected and analyzed. Chemical analysis through X-ray fluorescence [21][22] were used for determining major chemical components (Na_2O, MgO, Al_2O_3, SiO_2, P_2O_5, K_2O, CaO, TiO_2, MnO, Fe_2O_3). The measurement uncertainty results less than 47% by weight for concentrations <1%, between 2-4% for concentrations between 1 and 10%, and around 1% for concentrations > 10% [23]. The CO_2 content was measured by using the calcimetry method to estimate the amount of $CaCO_3$ in the analysed samples [24]; the $CaCO_3$ content was estimated with reference to a calibration linear regression among the volumes of CO_2 developed by acid attack of the powdered rock and the amounts of pure $CaCO_3$.

Phase identification was conducted with XRPD data obtained using a Bragg-Brentano θ–θ geometry X-ray diffractometer (Phaser D2, Bruker) with a Cu X-ray source (1.5418 Å) and a Si strip detector (LYNXEYE, Bruker) in the 2θ range from 5° to 65°. In a typical measurement, a powder sample was illuminated with radiation from the X-ray source excited at 30 kV with 10 mA, passing through a 1.0 mm primary slit module, 2.5° soller slit module, and a 0.5 mm Ni filter using a step size of 0.02° and an integration time of 1 s. The Crystallography Open Database (COD) implemented in the Bruker DIFFRAC.EVA 4.1 software was used to identify the crystalline phases.

Petrographic analysis was carried out thought transmitted light microscopic observations of thin sections (Zeiss Axioplan microscope). Ten medium- to coarse-grained sandstone samples were selected for modal analysis. More than 300 points were counted by using a minero-petrographic microscope for each thin section according to the Gazzi-Dickinson method (Ingersoll et al., 1984 [25]; Zuffa, 1985 [26]). Recalculated grain parameters are defined according to Dickinson (1970) [27], Ingersoll and Suczek (1979) [28], Zuffa (1985) [26], Critelli and Le Pera (1994 [29], and Critelli and Ingersoll (1995) [30], Amendola et al., 2016 [4].

Physical properties of the stones like real density (ρ_r), apparent density (ρ_a), water absorption coefficient by capillarity, water absorption at atmospheric pressure, total and open porosity, and saturation index have been determined following the current European Norms [31][32][33]. In particular: real density (ρ_r) has been determined using a gas pycnometer (Ultrapycnometer 1000 by Quantachrome Corporation). 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 He 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 have been immersed in distilled water [31] and the volume of the specimens was measured by means of a hydrostatic balance on water-saturated samples [34]. The water absorption coefficient by capillarity has been determined on the same samples used for apparent density determination [31]. 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 [32]. The total porosity has been calculated according to (1):

$$P\ (\text{vol. \%}) = 100 \cdot (1 - \rho_a / \rho_r) \qquad (1)$$

where ρ_a and ρ_r are the real density and the apparent density, respectively. Finally, the following mechanical properties have been measured according to the in use European Norms: uniaxial compressive strength [35]; flexural strength under concentrated load [36]; flexural strength under concentrated load after 48 freezing/thaw cycles [37]; determination of rupture energy [38]; abrasion resistance [39]; Knoop hardness [40].

Results And Discussion

Mineralogical and petrographic features

From the macroscopic point of view, the analysed samples vary in colour from dark grey to light grey, in fresh samples, to yellowish-reddish brown, in the altered ones. The samples are from fine- to medium- grained rocks with clearly visible lamellar phyllosilicate minerals flattened along the stratification. The chemical composition of the analysed samples is reported in Tab. 1. For the sake of comparison, the chemical compositions of other turbiditic sandstones belonging to the Tuscan and Umbro-Marchean Domains have been reported in Tab. 2.

TAB. 1: Chemical composition (wt%) of the analysed Macigno sandstone samples.

Sample	H_2O	CO_2	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	MnO	Fe_2O_3
VE (n=21)	2.08	3.40	2.14	6.38	13.72	59.18	0.11	2.47	5.53	0.38	0.07	4.54
St. dev.	0.30	2.16	0.18	0.45	1.05	3.63	0.01	0.16	2.75	0.03	0.01	0.27

Fe_2O_3 = total iron expressed as Fe_2O_3.

TAB. 2: Chemical compositions (wt%) of both the Macigno sandstone samples from Lunigiana, Garfagnana, Matraia, Monti d'Oltre Serchio, Gonfolina, Greve in Chianti and the Marnoso Arenacea samples (Colombino, Masso Grosso and Filaretti).

Sample	LOI	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	MnO	Fe_2O_3
MAC L (41)	4.55	2.73	5.20	13.34	62.16	0.11	2.63	4.39	0.42	0.07	4.39
St. dev.	2.38	0.38	0.98	0.98	4.18	0.02	0.23	3.42	0.04	0.01	1.32
MAC G (28)	3.93	2.39	6.21	14.55	62.32	0.12	2.60	3.02	0.40	0.07	4.38
St. dev.	1.05	0.23	0.74	0.86	1.96	0.02	0.24	1.50	0.06	0.01	0.45
MAC M (7)	5.24	2.27	5.87	13.48	60.63	0.11	2.66	5.13	0.38	0.07	4.16
St. dev.	1.95	0.20	1.13	0.81	3.07	0.01	0.07	2.97	0.07	0.02	0.27
MAC S (19)	6.36	2.12	5.08	12.49	60.67	0.10	2.45	6.20	0.48	0.07	3.98
St. dev.	4.77	0.27	0.84	2.07	8.98	0.03	0.34	7.90	0.07	0.02	0.56
MAC F (2)	4.61	2.96	3.05	12.71	65.98	0.11	2.64	3.73	0.49	0.06	3.65
St. dev.	0.01	0.13	0.11	0.16	0.27	0.01	0.07	0.10	0.01	0.01	0.08
MAC C (2)	5.24	2.64	4.09	11.99	64.41	0.11	2.48	4.82	0.45	0.06	3.71
St. dev.	0.53	0.06	0.05	0.11	0.92	0.01	0.02	0.69	0.01	-	0.06
PFC (3)	27.52	0.53	1.69	5.31	24.31	0.09	1.18	36.55	0.24	0.09	2.49
St. dev.	1.80	0.01	0.08	0.52	2.65	0.02	0.09	2.12	0.01	0.01	0.54
MG (2)	8.24	2.22	4.18	11.45	60.52	0.14	1.91	7.59	0.49	0.08	3.18
St. dev.	0.34	0.07	0.01	0.13	0.80	0.01	0.03	0.59	-	-	0.06
FIL (n=8)	10.70	1.82	4.66	10.55	56.58	0.15	1.66	9.97	0.57	0.08	3.26
St. dev.	3.21	0.21	0.58	1.09	5.12	0.03	0.11	3.42	0.11	0.01	0.47

LOI = loss on ignition at 950°C; Fe_2O_3 = total iron expressed as Fe_2O_3; MAC L = Macigno sandstones from Lunigiana (Di Battistini et al., 2008 [3]); MAC G = Macigno sandstones from Garfagnana (Aquino et al., 2020 [41]); MAC M = Macigno sandstones from Matraia (Lezzerini et al., 2008 [14]); MAC S = Macigno sandstones from Monti d'Oltre Serchio (Aquino et al. 2020 [42]); MAC F = Macigno sandstones from Gonfolina (Gioncada et al., 2011 [18]); MAC C = Macigno sandstones from Greve in Chianti (Gioncada et al., 2011 [18]); PFC = Pietra Forte Colombino (ScCd, Bargossi et al., 2002 [43]); MG = Masso Grosso (CaF and CaM, Bargossi et al., 2002 [43]); FIL = Filaretti (CuG, CuR, Bor13, Na11, CaC, B10, B11 and B12, Bargossi et al., 2002 [43]).

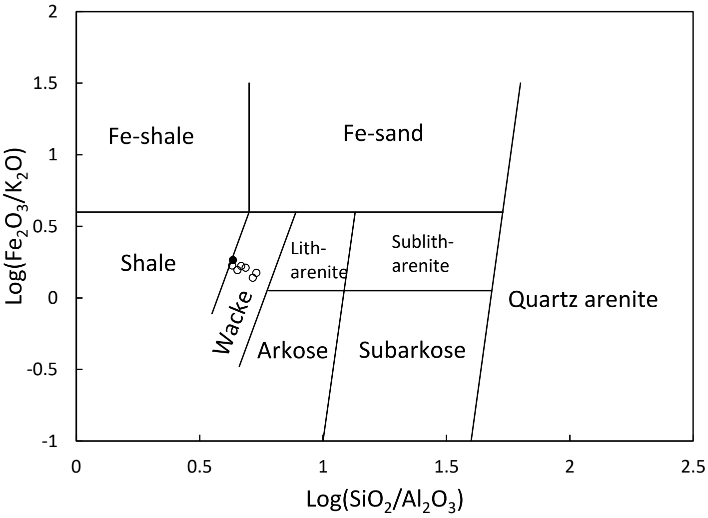

Fig. 2. Chemical classification of the Macigno sandstone samples based on log (SiO_2/Al_2O_3) vs. log (Fe_2O_3/K_2O) diagram of Herron, 1988 [44]. Full circle and empty circles are VE samples (n=21) and MAC samples, respectively.

Observing the collected data, it is interesting to note that no evidence of significative differences there are in the chemical compositions of the analysed samples, except for the contents of the CaO and CO_2 components, due to the different presence of carbonates, essentially calcite.

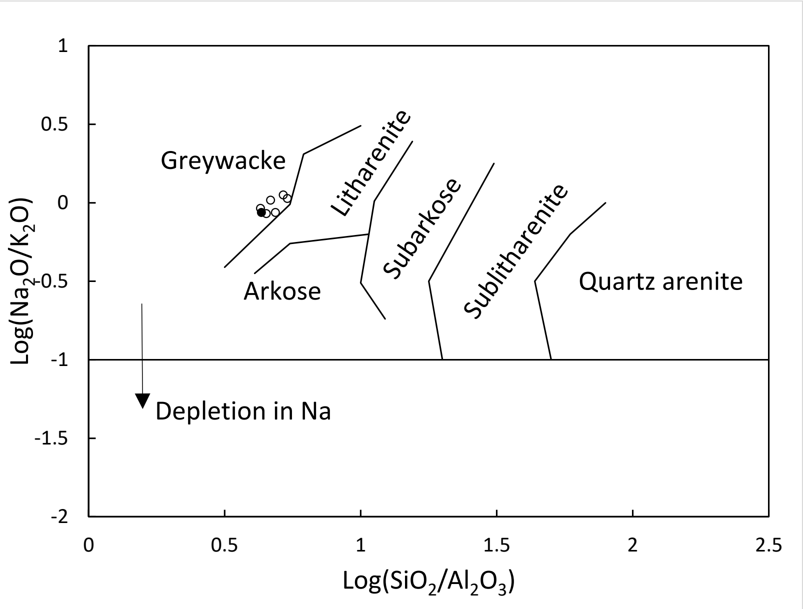

Fig. 3. Chemical classification of the Macigno sandstone samples based on log (SiO_2/Al_2O_3) vs. log (Na_2O/K_2O) diagram of Pettijohn et al., 2012 [45]. Full circle and empty circles are VE samples (n=21) and MAC samples, respectively.

The geochemical classification diagram (Fig. 2) by Herron (1988) [44] classifies the selected sandstones mainly as wacke. Pettijohn et al., 2012 [45] classification scheme for these sandstones (Fig. 3) shows that they plotted mainly in the greywacke field. Using the ternary diagram proposed by Blatt et al., 1972 [46], the selected sandstones plotted mainly in the ferromagnesian potassic sandstones field with similar occurrence in the sodic field (Fig. 4).

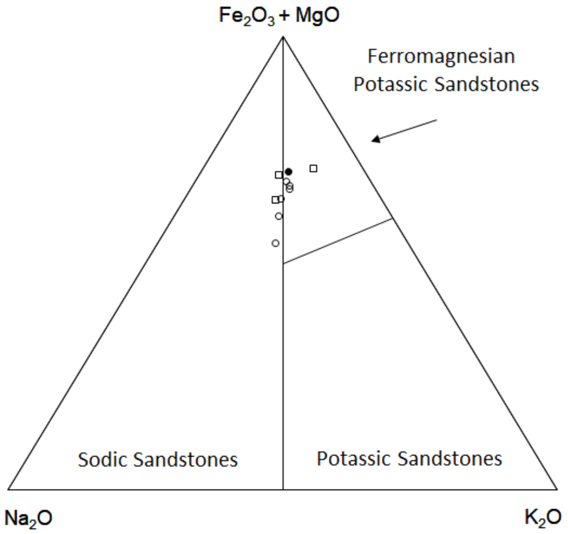

Fig. 4: Ternary diagram of Na_2O-K_2O-(Fe_2O_3+MgO) from Blatt et al., 1972 [46]. Full circle and empty circles are VE samples and MAC samples, respectively; empty squares are PFC, MG and FIL samples.

The mineralogical phases identified by X-ray diffraction are quartz, plagioclases, K-feldspar, phyllosilicates, and calcite. Micas-like minerals (illite), chlorites s.l. (chlorite s.s. + interlayered chlorite / smectite and, in some samples also corrensite, a regular 1:1 interstratification of trioctahedral chlorite and trioctahedral swelling smectite [47]) and kaolinite are the major constituents of the fine fraction (< 4µm).

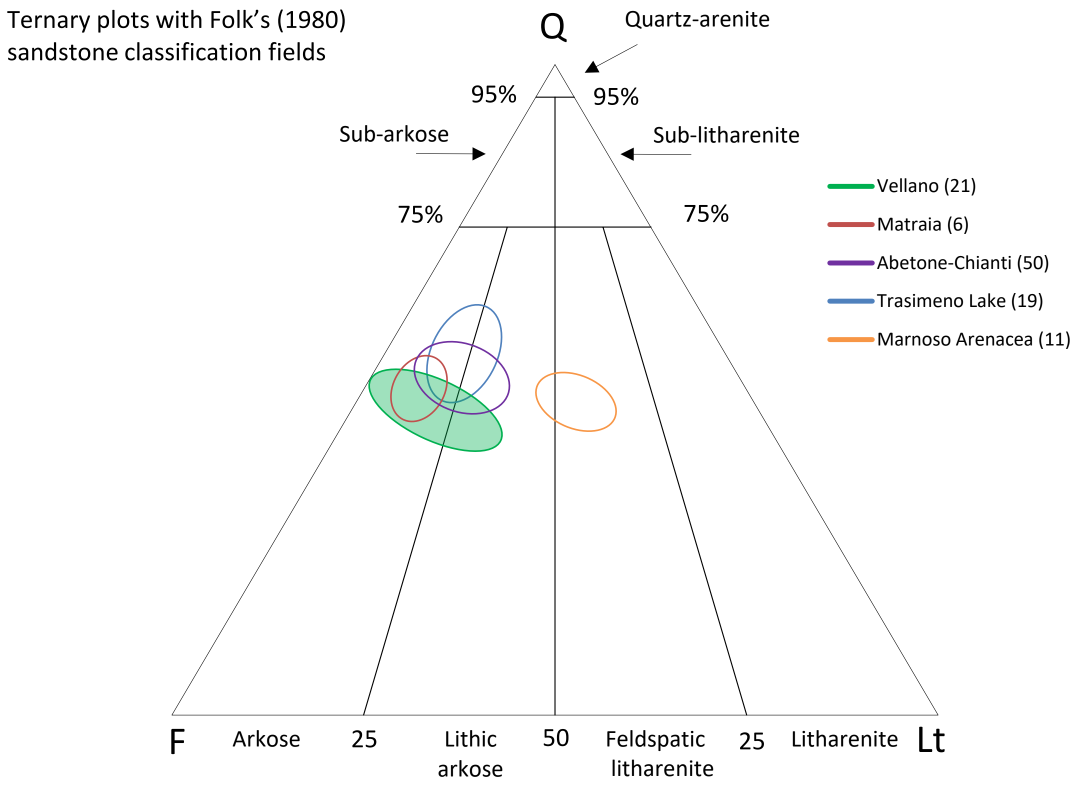

Fig. 5: QFL diagram for Folk's sandstone classification.

From the petrographic point of view (Fig. 5), the analysed rocks from the Vellano area are feldspatic sandstones, more precisely greywackes or arkosic arenites in accordance with the matrix (clasts <30 µm) contents that must be more than 15% by volume or more frequently less of this value. The analyzed sandstones are well-consolidated, from medium to medium-fine grained, poorly to moderately sorted, with angular to sub-angular detrital grains and colour of fresh surface variable from grey to bluish grey. Modal analysis showed that the rocks analysed are constituted in order of abundance by quartz and plagioclase (Ab_{20}), with subordinate quantities of K-feldspar, phyllosilicates (muscovite, biotite and chlorite) and lithic fragments. Heavy minerals include zircon, garnet, apatite, epidote, pyrite and magnetite. Small contents of carbonates occur as detrital clasts and authigenic cement. The rock fragments, less than 10% by volume, include metamorphic (70-80%), igneous (15-30%) and sedimentary (<5%) clasts. The clay matrix in the analysed sandstone samples range from 5 to 20 % by volume.

Physical and mechanical properties

Regarding the physical properties, it is interesting to note that the real density values and the apparent density values of the analysed sandstones differ quite a bit from each other, depending on the state of deterioration of the stone. The average values of the water absorption by atmospheric pressure, measured on 35 specimens, vary from 0.16 wt.% to 1.11 wt.%, with an average value of 0.75 ± 0.26, while the total porosity varies from 0.7% to 3.1% by volume, with an average value of 2.3 ± 0.7 vol.% [33][31]. The water absorption coefficients by capillarity at 24h, measured on 18 specimens perpendicularly to the anisotropy planes (C_1), are not similar in the analysed samples and the average value is 2.078 ± 0.516 $g/m^2s^{0.5}$ [32].

Uniaxial compressive strength [35], measured on 35 specimens, ranges from 91 MPa to 196 MPa, with an average value of 127 ± 19 MPa. Flexural strength under concentrated load measured on 25 specimens before and after 48 cycles of freeze/thaw [36][37], are 13.5 ± 2.7 MPa and 12.1 ± 2.7 MPa, respectively. Rupture energy [38] and Abrasion resistance (Method A - Wide Wheel Abrasion Test) [39] values are 9.7 ± 1.6 J and 20.0 ± 1.5 mm.

The Knoop microhardness [40], measured on 3 polished specimens, ranges in average from 3036 and 4674 MPa, with an average value of 4286 ± 422 MPa.

The collected data shows that from the physical and mechanical point of view the fresh Vellano sandstone samples are characterized by similar properties than those measured by Lezzerini et al. on fresh Matraia samples [22].

Conclusions

The “Macigno” sandstones are building materials widely used in the constructions of western Tuscany and, still today, they are appreciated to realize objects of urban interest and for paving the streets of historic centres.

In this work, Macigno sandstone samples from Vellano area (Pistoia province) were studied to evaluate their physical and mechanical properties and to compare the collected data with those measured on samples extracted from nearby quarries.

The studied rocks vary in color from grey, more or less dark in the fresh samples, to yellowish-grey in the more weathered samples. No significant differences were observed in the chemical composition of the selected samples, apart from the contents of CaO and CO_2, mainly due to the carbonate content. The main mineralogical phases identified by X-ray diffraction are quartz, plagioclases, K-feldspar, phyllosilicates and calcite. The analyses of the fraction < 4 μm allowed us to identify mica-like minerals (illite), chlorites s.l. (chlorite s.s + interstratified chlorite/smectite and, in some samples also corrensite) and kaolinite. The chlorite/smectite interlayers of the rocks studied have a variable content of chlorite layers, generally more than 50%. Corrensite, the interstratified chlorite/smectite, consisting of a regular alternation of smectite and chlorite layers (with a 1:1 ratio), was identified in almost all the samples.

From a petrographic point of view, the rocks from the Vellano area were found to be feldspathic sandstones, with generally medium or medium-fine grain size, moderately sorted, angular to sub-angular detrital grains. The matrix is essentially of phyllosilicate composition. The modal analysis showed that the analysed rocks consist in order of abundance of quartz and plagioclase, with subordinate quantities of K-feldspar, phyllosilicates and lithic fragments, mostly of metamorphic and sedimentary origin. The good physical and mechanical data measured on the Macigno sandstone samples from Vellano area make possible the use of this stone not only as a building material, but also for ornamental and artistic purposes.

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