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Marbles from Castagneto Carducci Area (Tuscany, Italy)

Marco Lezzerini¹, Jacopo Civita¹, Andrea Aquino¹, Stefano Pagnotta¹

¹ Department of Earth Sciences - University of Pisa, Via S. Maria 53, 56126 Pisa, Italy

marco.lezzerini@unipi.it

Abstract. In this work, marbles from Castagneto Carducci (Livorno province, Tuscany), which originated by contact metamorphism of the Calcare Massiccio Fm., an Early Jurassic limestone belonging to the Tuscan Sequence, were studied for determining their chemical, mineralogical and petrographic characteristics, and the main physical and mechanical properties. Forty marble samples were sampled and analysed; they are from two inactive quarries on the NW and NE slopes of the Mt. Romitorino, and from natural outcrops in the vicinity of the quarries. The analysed rocks are marbles with high calcite content (> 98% by weight). Optical microscopy observations showed Castagneto Carducci marbles generally have a heteroblastic/granoblastic texture with crystal boundaries from curved-right to lobate. The maximum grain size of the calcite crystals ranges from 0.2 mm to 0.6 mm. The determination of the main physical and mechanical properties of the analysed marbles showed that these rocks are characterised by low porosity and, in general, good physical and mechanical properties.

1. Introduction

Marble is a building and ornamental material that has been widely used over the centuries for its valuable aesthetic features and workability. In Italy, the availability of marbles and their excellent quality favoured a very extensive structural and ornamental use. Italian marbles have been used to build churches, cathedrals, temples and so on, as well as masterpieces of sculpture made by important artists such as Michelangelo Buonarroti, who used marbles extracted from the quarries of the Apuan Alps in Tuscany to create his works of art [1]. It is not a coincidence that Carrara was an important quarrying district since Roman times [2][3][4]. However, there are many other quarries in Italy and around the world from which valuable marbles were extracted for building interesting architectural monuments [5][6][7]. For example in Tuscany, the medieval churches of Pisa and Lucca cities as well as many other buildings of historical and architectural value were built with local stones [8][9] and precious marbles [10][11][12][13].

It is well known that the forces of nature may produce a decay scenario modifying the appearance and structure of marble. Among adverse agents must be included temperature, snow, rain, wind and atmospheric pollutants. These weathering agents generally act in combination with each other to increase the deterioration framework of marble. Rainwater, soluble salts, temperature changes can cause different stresses in texturally and mineralogically different marbles [14][15][16]. For all this, a deep knowledge of the main characteristics of marbles is very important and several authors have researched for this purpose [17][18][19][20], without considering all those works that have been done to create a database of marbles used in antiquity [21][22][23] or for identifying the provenance of them [24][25][26].

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In this work, the marbles coming from the Castagneto Carducci (LI) area were examined. They originated by the contact metamorphism of the calcareous rocks belonging to the sedimentary Tuscan sequence (in this area essentially the *Calcare Massiccio Fm*. according to Costantini et al. [27].

This work aims to determine the chemical, mineralogical and petrographic characteristics, and the main physical and mechanical properties. The further purpose is to investigate the metamorphic conditions that originated the formation of the marbles of the Castagneto Carducci area.

2. Geographic and geological framework of the study area

2.1. Geographic location of the study area

The outcrop area of the studied marbles (Figure 1) is located in the municipality of Castagneto Carducci (Livorno province, Italy), about 5 km E of San Vincenzo and as many kilometres N of Campiglia Marittima, where the extraction of marble followed the extraction of metals from underground and an ancient firing process since the time of the Etruscans [28].



Figure 1 – Detail of the geological map 1:10000 of the SW area of the section n. 305080 Sassetta, Tuscan Region (modified by Cerrina et al. [29]) and location of the sampling points. Quaternary deposits: a3) Undifferentiated debris covers; b) Current or recent alluvial deposits; Ligurian Domain (Upper Cretaceous): MTV – Flysch di Monteverdi; Upper Tuscan Sequence (lower Jurassic-Oligocene): MAS = Calcare Massiccio; RSA = Rosso ammonitico; STO = Scaglia Toscana; Neogenic-Quaternary magmatic Complex: FPO = Porphyritic veins and diches with trachyandesitic and rhyolitic composition.

This area is a part of the NW hilly system of Campiglia Marittima, which also includes Monte Coronato (550 m), Monte Calvi (646 m), Poggio all'Aione (459 m), Monte Rombolo (391 m), Monte Spinosa (386 m) and Monte Valerio (265 m), and the valleys of Temperino, Lanzi, Manienti, Botro ai Marmi, Vallin Lungo and Valle delle Rozze.

Sample	Group	Cc	Do		Texture		Veins	Patches	Grain size (mm)	
Sumpre	oroup	00	DU	Туре	GSU	GBS	(Child	1 atomos	AGS	MGS
CBC 5		Only		G, I	He	Cr-St	Р	А	0.10	0.3
CBC 5-1	W1	Only		G, I	He	Cr-St	Р	А	0.10	0.3
CBC 5-2		Only		G, I	He	Cr-St	Р	А	0.10	0.3
CBC 2		Only		G, I	He	Cr-Lo	А	А	0.20	0.4
CBC 3		Only		G, I	He	Cr-Lo	А	Р	0.09	0.2
CBC 4	W2	Only		G, I	He	Cr-Lo	А	А	0.10	0.2
CBS 1		Only		G, W-A	He	Lo	А	А	0.10	0.3
CBS 8		Only		G, I	He	Lo	А	Р	0.20	0.3
CAC 1		Only		G, W-A	He	Lo	Р	А	0.10	0.4
CAS 5		Only		G, I	He	Lo	Р	А	0.07	0.2
CBC 1		Only		G, I	He	Cr-St	Р	А	0.10	0.5
CBS 2	W/2	Only		G, I	He	Cr-Lo	Р	А	0.20	0.4
CBS 3	vv 5	Only		G, I	He	Cr	А	А	0.20	0.3
CBS 4		Only		G, I	He	Lo	А	А	0.08	0.3
CBS 6		Only		G, I	He	Lo	Р	Р	0.07	0.2
CBS 9		Only		G, I	He	Cr-Lo	Р	А	0.09	0.2
CAC 2		Only		G, I	He	Lo	Р	Р	0.10	0.4
CAC 2-1	W4	Only		G, I	He	Lo	Р	Р	0.10	0.4
CAC 2-2		Only		G, I	He	Lo	Р	Р	0.10	0.4
CAC 3		Only		G, A	He	Lo	Р	Р	0.06	0.6
CAS 8	Y	Only		G, A	He	Lo	Р	Р	0.20	0.4
CBS 7		Only		G, A	He	Lo	Р	А	0.09	0.2
CAS 1		Only		G, W-A	He	Lo	А	А	0.06	0.2
CAS 2		Only		G, I	He	Lo	Р	Р	0.07	0.3
CAS 3	D	Only		G, I	He	Lo	Р	Р	0.10	0.6
CAS 4	1	Only		G, I	He	Lo	А	Р	0.08	0.2
CAS 7		Only		G, W-A	He	Cr-Lo	А	Р	0.10	0.4
CBS 5		Main	Rare	G, W-A	He	Lo	А	Р	0.07	0.2
CAS 6		Only		G, A	He	Lo	Р	A	0.07	0.6
CAS 6-1	G	Only		G, A	He	Lo	Р	А	0.07	0.6
CAS 6-2		Only		G, A	He	Lo	Р	А	0.07	0.6

Table 1 - Main mineralogical and petrographic features of the analysed marble	2S
from Castagneto Carducci area.	

Cc = calcite; Do = dolomite (detected by XRPD and/or SEM/EDS analyses); GSU, Grain Size Uniformity = Ho, homeoblastic; He, eteroblastic; Texture Type: G, granoblastic; S, sutured; I, isotropic; A, anisotropic; S-, strongly-; W-, weakly-; GBS, Grain Boundary Shape = St, straight; Cr, curved; Lo, lobate; AGS and MGS = Average Grain Size of calcite grains and Maximum Grain Size of calcite grains; P = present; A = absent.

2.2 Geological framework

In the study area, lithologies belonging to the sedimentary Tuscan Sequence were intruded by neogenic magmatic bodies. In particular, in the Castagneto Carducci area the sedimentary, metamorphic and magmatic rocks are commonly grouped into the following complexes and structural units [27]:

a) Tuscan Domain, a complex of sedimentary rocks, including at the bottom Upper Trias limestones and dolomitic limestones (the *Calcari neri stratificati Fm*.), and Lower Lias massive limestone (the

Calcare massiccio Fm.), formed by metamorphosed carbonate rocks that are covered by a stratigraphic succession with calcareous-marly, clayey and siliceous formations of Cretaceous-Eocene age, and ending with arenaceous-clayey levels of the Oligocene Macigno;

b) Liguride Domain, including shales, siltstone and calcarenites (Upper Cretaceous);

c) Neoauthochton sediments, made of a sequence of conglomerates, sands and debris deposited in subsiding basins originated during the Neogene and Quaternary post-collisional extension;

d) Neogenic Magmatic Complex, consisting of intrusive granite bodies, acid volcanites and porphyritic veins and diches that placed during the Pliocene, in particular between 6 and 3 Ma [30][31]. In particular, the quartz-monzonitic intrusion [32] of *Botro ai Marmi*, dated to 5.7 ± 0.2 Ma [33], formed a wide metamorphic aureole extending for 6-7 km² within the Mesozoic carbonate sequence [32], which was more or less intensely metamorphosed [34].

The hills of Castagneto Carducci, from the tectonic point of view, belong to the western sector of the Tuscan Metalliferous Hills and represent a stretch of the Apennine chain that was corrugated in the Upper Oligocene - Lower Miocene due to the collision of the European continental margin with the African one (Adria microplate), and that has undergone a collapse and dismemberment in the Neogene due to an intense, typically post-collisional relaxation activity. This distensive tectonic phase, which occurred in the Messinian [35], changed the geostructural order of the area, determining the formation of new structures at Horst and Graben, and the consequent sedimentation in the subsident basins of Neogenic deposits [36].

3. Materials and methods

In this work, 31 marble samples were studied, coming from two inactive quarries, opened on the NW and NE slopes of the Romitorino, and from natural outcrops in the vicinity of these quarries (Figure 1 and Table 1). The geographic coordinates of the two quarries are as follows: 43° 06' 38.1" N – 10° 35' 44.0" E, 43° 06' 50.5" N – 10° 36' 02.7" E and 43° 04' 32" N – 10° 35' 20" E. Additional 9 dolomiterich samples were sampled and selected for applying the geothermometer calcite-dolomite and, more generally, for an evaluation of thermal conditions.

As possible, uncontaminated and un-weathered samples were carefully selected, except for three samples from Quarry A, showing clear feature of weathering that was expressly collected for the sake of comparison.

Chemical analyses were performed by X-ray fluorescence (XRF), according to the procedure suggested by Franzini et al. [37] with the accuracy of measurements of major, minor and trace elements determined by Lezzerini et al. [38][39]. Loss on ignition (LOI) was determined as mass loss in the temperature range 20-950°C.

Mineralogical and petrographic investigations of the samples were performed by optical microscope (OM) on thin sections using a Zeiss-Axioplane polarising microscope. The same equipment was used to study the texture and to determine the mean and maximum grain size (MGS) of the calcite grains. The terms used for describing rock texture are those suggested by Capedri et al. [18] and cited references unless otherwise specified. The crystal sizes of the studied metamorphic rocks are reported according to the grain size scheme of the British Geological Survey.

Insoluble residues were obtained by dissolving about 200 g of marble fragments in a 0.1N HCl solution. Semi-quantitative mineralogical compositions of whole-rock and residue were obtained via X-ray powder-diffraction (XRPD) using a Bragg-Brentano geometry and Ni-filtered CuK_{α} radiation obtained at 40 kV and 20 mA.

Scanning electron microscope (SEM) observations and microanalysis (EDS) of the rock's main and accessory minerals were performed using a Philips XL30 instrument equipped with an energy dispersive spectrometry EDAX (standardless software DXi4) with 20 kV acceleration voltage, 0.1 nA beam current, and 100 s live time.

The MgCO₃ content of the calcite was measured by both XRPD and SEM-EDS analyses on 9 selected samples in which calcite coexisted with dolomite. The X-ray d_{104} -values for calcite, measured using quartz as internal standard, were converted into MgCO₃ mol %, based on the experimental data on some

magnesian calcites collected by Goldsmith & Graf [40]. Electron microprobe analyses were carried out with a defocused beam to minimize the influence of possible sub-microscopic exsolution of dolomite lamellae upon cooling to below the mutual solubility temperature. The core compositions of calcite grains have thus been investigated to estimate the metamorphism peak temperature in such as way as to exclude inter-crystalline diffusion of magnesium due to retrograde resetting. For this reason, the maximum metamorphic conditions were calculated considering the maximum magnesium content recorded in each analysed sample instead of using the average data [41].

Real densities (ρ_r) have been measured through an automatic He-pycnometer on ~10 g of very-finegrained powders, dried at 105 ± 5 °C for 24hr under the following experimental conditions: ultrahigh purity compressed He, target pressure of 100 kPa; equilibrium time: automatic; purge mode: 3 min of continuous flow; maximum runs: 6; number of averaged runs: the last three. Apparent density (ρ_a) and open porosity (to water), which has been measured as water absorption at atmospheric pressure in respect to weight (Ab_w) or volume (Ab_v) of the specimens, were performed on cylinder-shaped specimens with a volume of about 30 cm³ following the prescriptions of the standard UNI 11060:2003 [42]. In particular, apparent density was calculated as the ratio between the mass of the dry sample and its volume, measured through a hydrostatic balance on water-saturated samples [43]. Total porosity (P) and saturation index (SI) were respectively computed as follows: P(%)=100·(1- ρ_a/ρ_r) and SI(%)=100·Ab_v/P. The uniaxial compressive strength was measured on 3 cm cube-shaped specimens, testing three specimens for each sample (EN 1926:2006, [44]).

4. Results and discussion

4.1 Mineralogical and petrographic characteristics of the analysed marbles

From the macroscopic and microscopic point of view, the analysed marbles can be grouped into 4 varieties on the base of both grain size of the calcite crystals and colours, structures and textures of the rocks:

Group W - White marbles (15): these samples are from pure white marble to white marble with grey spots, with or without grey or yellowish veins. Four subgroups were identified: W1, fine-grained pure white marble (MGS <0.3 mm); W2, white fine-grained (MGS <0.4 mm) marbles with grey spots; W3, white fine-grained (MGS <0.5 mm) marbles with grey spots characterised by the presence of yellowish veins; W4, white-yellowish fine-grained (MGS <0.4 mm) marbles with grey spots and veins.



Figure 2 – A detail of the macroscopic features of the analysed marbles.

Group Y - Yellowish marbles (3): they are light-yellow fine-grained (MGS <0.6 mm) metamorphic rocks that are heterogeneous due to the presence of dark yellow-reddish-coloured patches, fractures and thin veins. This marble variety has the typical features of *Giallo Siena* marble.

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Group P - Pink marbles (6): they are white-pink marbles with reddish or yellowish veins, but also grey. Some reddish veins expand to form fading bands, while the yellow and grey ones are generally millimetric in size. The calcite particle size of these samples is fine (MGS <0.6 mm).

Group G - Grey marble (3): they are light grey fine-grained (MGS <0.6 mm) marbles with pink and dark grey millimetric veins.

4.2 Chemical characteristics of the analysed marbles

The chemical data collected by X-ray fluorescence analysis confirms the analysed samples are almost entirely made up of CaO and volatile components (essentially CO₂), which together add up to no less than 99.09 wt% and 98.68 wt% for white marbles and yellowish marbles, respectively (Table 2). The average MgO contents are always less than 1%, varying from 0.53 ± 0.08 wt% for white marbles up to 0.83 ± 0.23 wt% for pink marbles (Table 2). The average non-carbonate fraction ranges from 0.68 wt% to 1.28 wt% for samples belonging to Group W1 and for samples belonging to Group Y, respectively.

Table 2 – Chemical composition of the analysed samples from Castagneto Carducci area.

Group (n.)		LOI	Na ₂ O	MgO	Al_2O_3	SiO ₂	P_2O_5	K ₂ O	CaO	TiO ₂	MnO	Fe_2O_3
W1 (3)	Average	43.85	< 0.01	0.53	0.03	0.07	0.03	0.01	55.46	< 0.01	0.01	0.01
	St. dev.	0.06	-	0.02	0.01	0.02	0.01	-	0.04	-	-	0.01
W2 (5)	Average	43.78	< 0.01	0.6	0.03	0.15	0.03	< 0.01	55.37	< 0.01	0.01	0.03
	St. dev.	0.14	-	0.1	0.03	0.21	0.01	-	0.25	-	0.01	0.04
W3 (8)	Average	43.77	< 0.01	0.64	0.05	0.14	0.03	0.01	55.32	0.01	0.01	0.02
	St. dev.	0.08	-	0.09	0.03	0.14	0.01	-	0.14	-	-	0.01
W4 (3)	Average	43.78	< 0.01	0.53	0.04	0.11	0.03	0.01	55.43	< 0.01	0.01	0.06
	St. dev.	0.02	-	0.08	0.01	0.01	0.01	-	0.05	-	-	0.01
Y (3)	Average	43.58	< 0.01	0.63	0.13	0.36	0.04	0.04	55.1	0.01	0.02	0.09
	St. dev.	0.22	-	0.1	0.1	0.32	0.01	0.04	0.32	-	-	0.04
P (6)	Average	43.66	< 0.01	0.83	0.11	0.24	0.02	0.02	55.05	0.01	0.02	0.04
	St. dev.	0.15	-	0.23	0.08	0.2	0.01	0.01	0.3	0	0.01	0.03
G (3)	Average	43.72	0.02	0.72	0.06	0.11	0.02	< 0.01	55.24	< 0.01	0.04	0.07
	St. dev.	0.07	0.01	0.09	0.03	0.06	0	-	0.1	-	0.01	0.01

n. = number of samples; LOI = loss on ignition at 950°C; Fe_2O_3 = total iron expressed as Fe_2O_3 .

As regards the trace elements, it is interesting to note that all the analysed elements (Ce, Ba, La, Ni, Cr, V, Co, Nb, Zr, Y, Rb) are individually less than 15 ppm, except for the Sr contents varying from 114 \pm 17 ppm (Group Y) to 210 \pm 14 ppm (Group G).

4.3 Physical and mechanical properties of the analysed marbles

The main physical and mechanical properties of the analysed marbles are reported in Table 3. The real density (ρ_r) values of the analysed samples are 2.71 g/cm³, in agreement with the mineralogical composition of the samples consisting of calcite and, sporadically, of calcite and traces of dolomite, while the apparent densities vary from 2.67 to 2.70 g/cm³. The water absorption by total immersion at atmospheric pressure varies from 0.33 to 1.04 % by volume, with an average value of 0.55 ± 0.21 % by volume. Average total porosities, measured on all analysed marbles, vary from 0.53 ± 0.13 to 1.39 ± 0.12 % by volume, with an average value of 0.80 ± 0.24 % by volume. As regards the mechanical resistance, the analysed marble samples have quite similar characteristics: the White marble variety (W1) is the less resistant (69 ± 20 MPa), while the Pink marble variety (P) is the most resistant (115 ± 33 MPa).

Group (n.)		G	$\gamma_{\rm d}$	C ₁ at 0.5h	Ab _w	Ab _v	Р	IS	σ
		(g/cm^3)	(g/cm^3)	$(g/m^2 s^{0.5})$	(%)	(%)	(%)	(%)	MPa
W1 (3)	Average	2.71	2.69	0.63	0.15	0.41	0.67	61	69
	St. dev.	-	-	0.26	0.01	0.02	0.02	3	20
W2 (9)	Average	2.71	2.69	0.89	0.19	0.52	0.83	62	83
	St. dev.	-	-	0.28	0.05	0.12	0.11	12	17
W3 (6)	Average	2.71	2.69	1.66	0.20	0.55	0.71	77	92
	St. dev.	-	-	0.46	0.04	0.11	0.09	9	14
W4 (3)	Average	2.71	2.67	4.77	0.39	1.04	1.39	75	96
	St. dev.	-	-	0.67	0.03	0.06	0.12	2	10
Y (3)	Average	2.71	2.70	0.10	0.15	0.42	0.53	78	97
	St. dev.	-	-	0.05	0.05	0.13	0.13	6	37
P (3)	Average	2.71	2.69	0.69	0.26	0.69	0.73	94	115
	St. dev.	-	-	0.24	0.02	0.04	0.02	7	33
G (3)	Average	2.71	2.69	0.13	0.12	0.33	0.74	45	100
	St. dev.	-	-	0.14	0.01	0.03	-	4	14

Table 3 - Main physical and mechanical properties of the analysed marbles.

4.4 Metamorphism temperature and pressure of the investigated marbles

Based on the geological framework and tectonic evolution of the Campiglia Marittima area, Leoni & Tamponi [45] hypothesized a thickness of 3-4 km, corresponding to a lithostatic pressure of about 1-1.5 kilobar for the sedimentary sequence at the time of intrusion of the Botro ai Marmi granite and related veins, dikes and sills. Applying the calcite-dolomite geothermometer [46] that allows determining the metamorphism temperature by measuring the magnesium content into calcite in the calcite+dolomite assemblage, Leoni & Tamponi [45] and Franzini et al. [17] indicate temperature values of 450-550°C (on 12 marble samples from an exploration well) and about 450°C (on eight samples coming from abandoned quarries in the same area), for the more intense metamorphic events that generated the Campiglia Marittima marbles.

In the present work, the magnesium contents into calcite coexisting with dolomite were estimated by both X-ray diffraction and electron microprobe operating in energy-dispersive mode. The magnesium contents measured on teen analysed samples by means X-ray diffraction technique range from 2.67 to 3.29 wt% (the average d104-value for calcite is $3.026 \pm 0.001 \text{ Å}$), indicating a maximum metamorphic temperature of 417-452°C (mean value of 432 ± 13 °C). Similarly, the magnesium contents into calcite coexisting with dolomite (expressed as MgCO3 mol %) measured using SEM/EDS on eight analysed samples vary from 2.95 to 3.24 mol% (the average MgCO3 value for calcite is $3.08 \pm 0.11 \text{ mol\%}$), indicating a maximum metamorphic temperature of 433-449°C (mean value of 441 ± 6 °C). The estimated temperatures are in good agreement with those induced by the granitic intrusion of Botro ai Marmi in the Mesozoic carbonate rocks of the Tuscan Nappe sequence as estimated by Leoni & Tamponi [45] and by Franzini et al. [17] in the Campiglia Marittima area.

5. Conclusions

The Castagneto Carducci marble samples analysed in this study came from two inactive quarries on the NW and NE slopes of the Mt. Romitorino, and from natural outcrops in the vicinity of the quarries. The analysed marble originated by contact metamorphism caused by granitic intrusion of *Botro ai Marmi* and related veins, dikes and sills within the Mesozoic carbonate rocks of the Tuscan Nappe sequence. The estimated metamorphic temperatures and pressures of the Castagneto Carducci marbles are 420-450°C and 1-1.5 kilobar, respectively.

The analysed samples are fine-grained marbles with high calcite content (> 98% by weight). Based on macroscopic features, the analysed samples can be grouped into four varieties: white marble, yellowish

marble, pink marble and grey marble. From the microscopic point of view, all the varieties of Castagneto Carducci marbles are characterised by heteroblastic/granoblastic texture with crystal boundaries from curved-straight to lobate. The average grain size varies from 0.06 mm to 0.2 mm, while the maximum grain size from 0.2 mm to 0.6 mm. The determination of the main physical and mechanical properties of the analysed marbles showed that these rocks are characterised by low porosity and, in general, good physical and mechanical properties.

In comparison with the marbles outcropping to the south, near Campiglia Marittima, the Castagneto Carducci marbles have generally finer grain size, although they belong to the same formation. This is due to the location of the Campiglia Marittima marbles close to the neogenic magmatic intrusion of Botro ai Marmi. Consequently, the latter have undergone more thorough metamorphism and, therefore, greater recrystallization of the calcite crystals occurred. From a commercial point of view, the Castagneto Carducci marbles are valuable materials, however, the intense fracturing of the rock limits the possibility of extraction.

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