The Piedmont white marbles used in Antiquity: an archaeometric distinction inferred by a minero-petrographic and C-O stable isotope study

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THE PIEDMONT WHITE MARBLES USED IN ANTIQUITY: AN ARCHAEOMETRIC DISTINCTION INFERRED BY A MINERO-PETROGRAPHIC AND C-O STABLE ISOTOPE STUDY

A. Borghi[1], G. Vaggelli[2], C. Marcon[1] and L. Fiora[1],

[1] Dipartimento di Scienze Mineralogiche e Petrologiche, Via V. Caluso, 35, Torino, Italy
[2] CNR Istituto di Geoscienze e Georisorse. Via V. Caluso, 35, Torino, Italy.

ABSTRACT

The metamorphic rocks outcropping in the Western Alps are characterised by a great variety of white marbles which have been poorly studied in the archaeometric field even though they have been used since the antiquity. Typical examples are the Arc of August of Roman time in Susa, (Piedmont, Italy) and lots of monuments and historical buildings of Turin (Italy).

A multi-analytical approach based on petrographic (optical and scanning electron microscope), electron microprobe and stable isotope analysis of white Piedmont marbles have been performed in order to carry out a detailed description, summarizing their main micro-textural, mineralogical and isotopic features.

Eight historical Piedmont marbles have been sampled from well-known quarry sites belonging to different metamorphic geological units of the Western Alps (Ornavasso, Crevola, Pont Canavese, Foresto, Chianocco, Prali, Brossasco and Garessio marbles). Their different metamorphic conditions, ages and structural evolution allowed drawing a discriminative flow chart based on microscopic and minero-chemical data.

Key words: Marble, Western Alps, mineral chemistry, stable isotopes, Cultural Heritage

INTRODUCTION

The word marble derives from the ancient Greek *"marmario"* which means *"to bright"* and was used for any stone whose surface could be polished by a mechanical smoothing. Thanks to its peculiar brightness the marble has been widely used since antiquity like material for sculpture and architecture.

The production of the first marble objects dates from the Neolithic age. In the Cyclades, where marble is abundant (as in the islands of Paros and Naxos), small idols and later on larger sculptures, dated from the end of the IV and the III millennium BC appear as typical artistic production of the "Cycladic" civilization (Lazzarini, 2004).

In ancient Greece, lots of marble quarries were active and numerous marble varieties were extracted as the famous Penteli, Thasos, Naxos and Paros marbles. Since the origin of Greek and Classical ages, the use of the marble was, therefore, widely diffused.

In Rome, the extensive use of marble began in the first century BC and, at the time of Augustus, most buildings were erected in marble. During the first century BC, the Luni marble (now well-known as Carrara marble) replaced the Greek white marbles as it was more inexpensive and of good quality.

Moreover, in Roman age the use of marble for buildings and sculptures spread throughout the whole Roman Empire. In Western Alps of Northern Italy, indeed, local marbles were quarried and used for public employments (see for example the grey marble coming from Aymaville used for the "Porta Praetoria" in Aosta; Borghi et al., 2006).

The identification of the quarry site for ancient marble artefacts covers a great archaeological and historical interest, but it is also significant for the conservation and the restoration aspect.

Up to now, no scientific technique can be decisive by itself for marble classification, and so combining the petrographic study with stable isotope determinations ($\delta^{18}O$ and $\delta^{13}C$) is the most frequently used procedure for marble identification (Gorgoni et al., 2002). Other techniques for marble characterisation are Cathodoluminesce (Barbin et al., 1992), Electronic Paramagnetic Resonance Spectrometry (Attanasio, 2003) and Inductively Coupled Plasma Mass Spectrometry (Green et al., 2002).

The first studied marbles are the white marbles from the Mediterranean area used by the ancient Greeks and Romans (Lepsius, 1890). In the last decades, a widespread research interest on these marbles has produced a detailed petrographic and geochemical characterisation (see for example Asgari and Matthews 1995 on the Prokonnesos marble; Bruno *et al*. 2002 on Thasos; Bruno *et al*. 2000 on Paros; Lazzarini et al. 2002 on Aphrodisias; Matthews 1997, Pike 1999 and Goette et al. 1999 on Penteli marble).

Many geological units of the Piedmont Western Alps (Italy) have also provided a great variety of white marbles. These marbles have been largely employed both in the antiquity as the Augustus Arc of Roman times in Susa (Piedmont, Italy) and more recently for monuments and historical buildings of Turin town (Piedmont, Italy). However, in most cases the attribution of the stone material to its quarry site or geological units was based on macroscopic data (Frisa and Gomez, 1998) or on historical documents without an archaeometric support. Only Barbin et al., 1992 report on the cathodoluminescence data for Crevola D'Ossola marble. In addition, few cases, as the Saint Cristina and Saint Filippo Churches (Turin, eighteen century), were investigated and the provenance site of the stone material hypothesised (Chiari et al.1992; 1994).

The present work aims to study the most used white Piedmont marbles quarried in historical ages, in order to create a data base containing the main minero-petrographic and isotopic features for archaeometric purposes, performed by a multianalytical approach which combined traditional techniques (optical microscopy and stable isotope analysis) to SEM-EDS analytical facilities which have been up to now rarely applied in the archaeometric field.

The results here reported are referred to an average of five samples collected in the Piedmont historical quarry. The present work is therefore a first report on the main textural and minero-petrographic data which allows a comparison between the white Piedmont marbles.

GEOLOGICAL FRAMEWORK OF ALPINE MARBLES

Most Piedmont marbles from the Western Alps generally outcrop as small lenses intercalated with schists and gneisses belonging to various geological units characterised by different metamorphic conditions (Fig. 1). They can be grouped in two classes: the marbles belonging to the Pre-Triassic (Palaeozoic) crystalline basement of the Western Alps characterised by a polymetamorphic evolution and the Triassic marbles coming from the meta-sedimentary cover with a monometamorphic evolution. The Palaeozoic marbles include the Ornavasso marble (from Ivrea-Verbano zone in Ossola valley), the Prali and Brossasco marbles (from Dora Maira Massif) and the Pont Canavese marble (from Sesia Lanzo zone). The Triassic ones consist of Foresto and Chianocco marbles (from the meta-sedimentary cover of Dora Maira Massif), Crevola (from the deep Penninic unit in Ossola valley), and finally the Garessio marble (from Brianconnaise zone). These marbles can be also grouped according to the metamorphic evolution suffered from the host unit. Therefore, they can be divided in three quarrying districts characterised by the following metamorphic conditions: medium-high temperature (Ossola Valley: Ornavasso and Crevola marbles), low temperature and high pressure (Dora Maira and Sesia zones: Chianocco, Foresto, Prali and Pont Canavese), and low temperature and low pressure (Brianconnaise zone: Garessio). Finally, the Brossasco marble belongs to a small geological unit which suffered ultra-high pressure (UHP) and high temperature metamorphic conditions (P>30 kbar for T>700°C). A summarizing sketch of the main geologic features is reported in Table1.

ANALYTICAL TECHNIQUES

Petrographic analysis by optical and scanning electron microscope, minero-chemical analysis of main and accessory minerals by EDS electron microprobe, and finally mass spectroscopy for the determination of stable isotope ratios were applied to the samples collected from various outcrops of well-known historical quarries.

The petrographic analysis was carried out to define the mineralogical assemblage and the microstructural features. The accurate description of the marbles by petrographic investigation allowed to deduce the pressure-temperature conditions suffered during the metamorphic process and to determine textural parameters as the Grain Boundary Shape (GBS) and the Maximum Grain Size (MGS) according to the terminology reported by Heinrich (1956), Best (1982) and Gorgoni et al. (2002). Finally, the presence and the relative amount of minerals occurring as subordinate or accessory phases were also determined.

Electron microprobe analyses were performed with an EDS-SEM system of Oxford Instruments. Natural silicates and oxides were used as standards. The accelerating voltage was 15 kV and the dwell time 50 s. A ZAF data reduction program was used. All the analyses were recalculated using the MINSORT computer program of Petrakakis and Dietrich (1985). The mineral compositions are expressed as atoms per formula unit (a.p.f.u.). The mineral symbols are from Kretz (1983).

The electron microprobe analysis has provided the chemical composition of the main mineral component (calcite and/or dolomite), and the chemical composition of subordinate or accessory minerals useful for discriminative porpoises.

The stable isotope analysis (i.e. $\delta^{13}C$ and $\delta^{18}O$) has been carried out on calcite and on dolomite for studied marble type. The protocol reported in McCrea (1950) was followed. In particular, 10 mg of powered calcite or dolomite was reacted with 100 % orthophosphoric acid under vacuum condition. The produced CO_2 was analysed, for its oxygen and carbon isotopic composition, with a Mat 250 Finningan Mass Spectrometer. The results are expressed as isotopic ratio relative to the PDB standard (Craig, 1957), following the convention defined by the international atomic energy agency.

RESULTS AND DISCUSSIONS

Petrographic analysis of microstructure and texture

The minero-petrographic features of the investigated marbles are reported in Table 2 and shown in a series of photomicrographs of representative marble samples (Fig. 2).

A first division of the studied marbles may be done according to fabric, which is diagnostically important since they are related to type and degree of metamorphism suffered by the sedimentary protolith (Lazzarini et al., 1980).

The Piedmont marbles are mainly isotropic; only Crevola Grey variety, Prali and Foresto show a clearly foliated (anisotropic) fabric, reflecting a syn-kinematic recrystallization developed during an oriented-pressure deformation.

The fabric discrimination based on the equal or different size of calcite or dolomite crystals is referred to homeoblastic (HO) or heteroblastic (HE). Only Crevola Grey variety and Chianocco are definitely homeoblastic. All the other marbles are heteroblastic.

Respect to the grain boundary shape (GBS), the Piedmont marbles mainly display irregular crystals marked by curved – sutured to embayed boundaries (Fig. 2). Moreover, single carbonate crystals show polysynthetic twinning and undulate extinction, which reflect the large amount of ductile deformation suffered by the rock under syn-kinematic conditions, rarely followed by recrystallization under static conditions. Only Crevola Grey variety, Chianocco and Brossasco marbles shows a more granoblastic fabric, defined by straight to curved boundary shape of the crystals.

Comparing different microscopic features of the white Piedmont marbles, only Chianocco marble shows microstructural evidences of a pervasive recrystallization under static conditions (Table. 2). All other marbles reflect the polyphasic structural evolution developed during the Alpine or Pre-Alpine orogenic cycles.

MGS parameter (Table 2, Fig.3) can also provide useful information for the studied marble classification. It is an important diagnostic parameter strictly related to the maximum temperature reached by the marble during its metamorphic evolution (Moens et al., 1988). Indeed, the marbles of Ornavasso and Brossasco, equilibrated under granulite and UHP eclogite facies metamorphic conditions, respectively, are characterised by significantly high MGS values (>3 mm). All other marbles are from medium to fine-grain sized (MGS < 2 mm) reflecting lower grade metamorphic conditions ranging from green schists to eclogite facies. More in detail, MGS of medium/fine-grained marbles show comparable and partially overlapping ranges preventing their unambiguous identification based on this parameter.

Mineral chemistry

Examination of representative polished thin section for the analysed marbles by SEM/EDS system, using back-scattered electron (BSE) and X-ray signals, allowed clearly defining the carbonate composition. Brightness signal in BSE images is sensitive to differences in mean atomic number, so the different carbonate phases (i.e. calcite and dolomite) appear as grains with different grey levels: those ones with higher mean atomic number (e.g. calcite) being brighter than minerals with lighter forming-elements (e.g. dolomite). See for example the different brightness signal between the coexisting calcite (grey) and dolomite (black) in Prali marble (Fig. 2F).

The chemical composition and the abundance of the main carbonate component (Table3) allow a main classification of the studied marbles in:

a) *calcitic marbles* (Ornavasso, Brossasco and Garessio) marked by a dolomite content < 20 wt. %.

b) *mainly calcitic marbles* (Prali) with a dolomite content ranging from 20 to 50 wt. %.

c) *dolomitic marbles* (Crevola, Pont Canavese, Foresto and Chianocco) with calcite content < 20 wt%.

The chemical composition of calcite and/or dolomite is a further discriminating parameter (Table 4a).

In calcitic marbles, the composition of calcite is pure (Ornavasso marble) or contain very small amount of Mg (< 0.010 atoms per formula unit: a.p.f.u.) in Brossasco and Garessio. In Prali marble calcite contains a constant Mg content (0.040-0.045 a.p.f.u). In dolomitic marbles, calcite is either pure or slightly Mg-rich (< 0.100 a.p.f.u). In Pont Canavese marble, calcite is Sr-rich with a Sr/Ca ratio around 0.07.

In dolomitic marbles, dolomite shows an homogeneous composition with Mg/Ca ratio ranging from 0.93 to 0.95 for most data. Minor amounts of dolomite are also present in calcitic marbles where it seems to occur as a late phase. It is worth to note the presence of Fe and Mn in Ca site determining an Mg/Ca ratio higher than unity (around 1.2), in Garessio marble dolomite. Finally, dolomite of Prali marble resulted Fe-rich (Mg site contains up to 8% Fe^{2+}).

Further comparisons between Piedmont white marbles can be made using the abundance and the chemical composition of subordinate and accessory minerals (Table 4b-d). At first, it has to point out that Prali marble is characterised by a significant abundance (up to 5 wt.%) of white silicate minerals (quartz, muscovite, Mg-chlorite, Mg-amphibole, feldspar and talc) which makes it clearly distinguishable from all other Piedmont white marbles.

In all other white marbles, the most common silicate minerals present as subordinate phases are quartz, micas and chlorite. Quartz is an ubiquitous mineral phase occurring as rare and small grains in all calcitic marbles, and as rare and variable grain-sized grains (from few tens to several hundreds micrometer) in dolomitic marbles.

As the chemical composition of muscovite is sensitive to pressure (Massonne and Schreyer, 1987), it can be used for discriminative purposes. In calcitic marbles, muscovite is ubiquitous; in Brossasco and Prali it has a strongly phengite composition, according to Capedri et al., (2004), with respect to Ornavasso and Garessio where it corresponds to a normal phengite (Fig. 4). Ornavasso white mica is also characterised by a Ba-rich core (Table 4b). In dolomitic marbles, muscovite occurs in Foresto and Chianocco where it shows a wide and overlapping compositional range spreading from the phengite to the strongly phengite field. These comparable values are well-explained by the similar metamorphic evolution suffered by Foresto and Chianocco coming from the same geological unit (Dora-Maira massif).

Chlorite is present in the same marbles where muscovite occurs (Table 3). Its chemical composition is shown in Fig. 5 and Table 4c. In calcitic marbles, chlorite is rare and occurs as isolated rose-shaped grains (pennina-talc-chlorite) or in aggregates with epidote, in Ornavasso and Brossasco marbles, respectively. In Prali marble, chlorite is more widespread occurring either as Fe-poor colourless variety (clynoclore-pennina) or as green coloured variety (pycnochlorite). In Foresto and Chianocco dolomitic marbles, chlorite is present as Mg-chlorite ranging from clinoclore to pennina.

As regards to silicate accessory minerals characteristic of single marbles it has to stress the occurrence of aggregates of paragonite in Pont Canavese, Mg-amphibole + epidote in Foresto, and zoned epidote crystals in Brossasco marbles. In Crevola marble, phlogopite is a peculiar accessory phase responsible for the honey coloured levels. Moreover, its composition allows to distinct the Crevola marble varieties. Indeed, pholopite has a homogeneous composition in Crevola Grey marble and a zoned composition with Ba enrichment in the Light Grey variety.

Feldspars typically occur in calcitic marbles of high metamorphic grade as Ornavasso and Brossasco. In the latter, both orthoclase and albite occur (Table 4d). In Ornavasso alkali feldspars are more widespread and represented by ialofane $[(K,Na,Ba)[(Al,Si)_4O_8]$ as celsiane molecule ranges from 5 to 30%. In Crevola marble, rare orthoclase was found in both varieties, and very rare plagioclase (oligoclase) in the Grey variety.

The occurrence of barite crystals in Ornavasso marble and aggregates of apatite + dolomite + phengite in Garessio and Prali marbles has to be pointed out, too. Finally, Fe-sulphides and/or Fe-oxides are widespread in both dolomitic and calcitic marbles as accessory phases.

C-O stable isotope analysis

The approach based on measurement of the isotopic ratios of carbon and oxygen resulted interesting and promising ever since its first appearance (Craig and Craig, 1972; Manfra et al., 1975). In the last decade, isotopic data sets were significantly implemented by Moens et al. (1988; 1992) and Gorgoni et al. (2002), producing excellent reference diagrams for marbles coming from the main quarries active in the Greek and Roman times. These diagrams according to the compilation of Lazzarini (2004) were widely used by archaeometrists, also for other marbles belonging to the Mediterranean basin. However, no isotopic analyses are up to now reported for the white marbles of the Western Alps. The data here performed represent the first stable isotope characterization of Piedmont alpine marbles for archaeometric purposes.

$\delta^{18}O$ and $\delta^{13}C$ have been determined on both calcite and dolomite. The results referred to the PDB standard are reported in Table 5. The first striking feature is the compositional homogeneity of $\delta^{13}C$ with respect to $\delta^{18}O$ (Fig. 6). In particular, $\delta^{13}C$ is positive ranging from 0.9 to 2.8 with a mean value around 1, whereas $\delta^{18}O$ is negative ranging from -14.0 to -1.0. Therefore, the Piedmont white marbles show a spread distribution along x-axis. Moreover, in any marble sample, calcite and dolomite $\delta^{18}O$ as well as $\delta^{13}C$ do not differ much.

The $\delta^{13}C$ homogeneity and $\delta^{18}O$ variability can be correlated with the different provenance and metamorphic-structural evolution of the studied marbles (Fig.7a). Indeed, the most negative $\delta^{18}O$ values (lower than -8) are found in Ornavasso which is characterised by a high temperature metamorphic imprinting (granulite facies). The marbles equilibrated under eclogite facies conditions spread in a relatively short range (from -10 to -4; Fig. 7a) which also includes Crevola and Brossasco marbles equilibrated under amphibolite and UHP facies, respectively. Finally Garessio marble, which suffered a low grade metamorphic evolution under green schists facies shows the less negative $\delta^{18}O$ values (around -1.4 / -1.2).

A further division may be done between the Palaeozoic marbles, coming from the polymetamorphic basement of the Western Alps, and the Triassic marbles belonging to meta-sedimentary covers which only suffered the Alpine metamorphic imprinting (Fig. 7b). The Triassic marble (Crevola, Foresto, Chianocco and Garessio) plot in the right side of the diagram showing a $\delta^{18}O$ ratio ranging between –5.55 and –1.20. On the contrary, the Palaeozoic marbles are marked by lower $\delta^{18}O$ values (- 13.22 / - 6.00). This isotope distribution, related to the age of the marbles, probably depends by the different metamorphic evolution suffered by the two groups of marbles. Indeed, the Triassic marbles were only metamorphosed during the Alpine orogeny when temperature lower than 550 °C were reached, whereas the older marbles underwent a higher temperature metamorphic evolution.

In particular, they previously suffered the Variscan orogeny that, in the Alpine region, developed under the amphibolite and granulite facies conditions (i.e. higher temperature). Therefore, the isotope discrimination between marbles of different age is probably due to the suffered thermal evolution.

The analysed marbles were finally compared with the white marbles of the Antiquity, plotting the performed isotope data on the diagrams compiled by Lazzarini (2004) where the fields of the main Greek and Roman white marbles are drawn (Fig. 8). The studied marbles spread along several Greek-Roman marble fields. In particular, in the diagram for marbles with MGS< 2 mm (Fig 8a), most eclogitic marbles overlap the field of Docimium and partly Penteli marbles, whereas the green schists Garessio marble plots in Carrara-Hymettian fields. In the diagram for marble with the MGS> 2 mm, Ornavasso and Brossasco marbles plot along the lower boundary of Naxos field (Fig. 8b).

Flow chart discriminative diagram

The fabric and textural parameters, the C-O stable isotope data, and the minero-chemical features of the main carbonate phase and of subordinate or accessory silicate minerals have provided the parameters necessary for the Piedmont white marble distinction, as reported on the flow-chart of Fig. 9. This scheme, which summarises the main minero-petrographic features, is obtained taking into account the following sequence of parameters. The first major parameter is the nature of the main carbonate phase which can be calcite or dolomite (i.e. calcitic or dolomitic marbles). The second one is the textural feature regarding the presence or absence of an oriented foliation which discriminates between isotrope and anisotrope marbles. They can be further divided according to the MGS value, the occurrence of index accessory minerals and finally the chemical composition of peculiar minerals depending on the different metamorphic conditions suffered by the geological unit of provenance. The inferred flow-chart coupled with stable isotope data can be applied for archaeometric purposes as the attribution of an ancient artefact to its provenance site. A further implementation to this proposed discriminative scheme may derive from cathodoluminescence studies and cathodomicrofacies determination on a complete set of marbles as otherwise proposed by Barbin et al., 1992.

CONCLUSION

In this paper, the Piedmont white marbles have been described according to their petrographic, minero-chemical and isotopic features by means of classical techniques, as optical microscope and stable isotope analysis, as well as of unusual techniques for archaeometric studies of marbles as the electron scanning microscope and electron microprobe.

In the studied marbles, MGS and the $\delta^{18}O$ values are the more variable parameters depending on the recorded metamorphic temperature. These parameters allow discriminating marbles crystallised under quite distinct T conditions as Ornavasso and Brossasco (high T marbles) with respect to Garessio (low T marble). On the contrary, for marbles which suffered a similar metamorphic history, the classical petrographic analysis coupled with C-O isotope data may not be sufficient for their discrimination. Therefore, the careful determination of paragenetic and textural features by SEM-EDS system becomes fundamental for their distinction. In particular, the identification of peculiar accessory minerals and their micro-chemical composition allow discriminating the white marbles up to their almost univocal classification.

In conclusions, it was observed that the petrographic, isotopic and paragenetic features of white marble depend on the metamorphic conditions, ages and geological pertinence. Therefore, it is possible to assert that the key to discriminate white marbles is represented by their different geological history. As a consequence a preliminary geologic study of possible host units (i.e. historical and contemporary quarry sites) is recommended any time the provenance of a marble variety is requested.

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FIGURE CAPTIONS

Fig. 1 - Tectonic sketch map of the Western Alps. The locations of the historical quarries are reported.
Legend: 1 = Post-orogenic continental and marine terrigenous deposits of Plio-Pleistocene to Quaternary age. 2 = Late orogeny fore-deep turbiditic deposits of Oligo-Miocene age 3 = Apennine chain; 4 = South-Alpine Domain; 5 = Canavese Zone; 6 = Austroalpine Domain; 7-11 = Penninic Domain: 7 = Upper Piedmont Unit; 8 = Lower Piedmont Unit; 9 = Internal Crystalline Massifs; 10 = Briançonnais Zone; 11 = Lower Penninic Unit; 12 = Helvetic Domain. 13 = Tectonic Lines: FP = Penninic Front, LS = Simplon Line; LCE = External Canavese Line; LCI = Internal Canavese Line; SV = Sestri Voltaggio Line, RF = Rio Freddo Line, VV = Villalvernia Varzi Line.

Fig. 2 Back-scattered electron images showing the typical aspect of the investigated marbles: (A) Ornavasso; (B) Crevola; (C) Pont Canavese; (D) Foresto; (E) Chianocco; (F) Prali; (G) Brossasco; (H) Garessio. For details see Table 2. Bar = 1 mm for all samples except Garessio. Mineral symbol according to Kretz, 1983.

Fig. 3 – MGS parameter for the investigated marbles.

Fig. 4 – Si- Al_{Tot} classification diagram for muscovite. The field of high phengite (high-phe), phengite (phe) and muscovite (ms) are reported according to Capedri et al. (2004).

Fig.5 – Classification diagram for chlorites after Hey (1954).

Fig. 6 - $\delta^{18}O$ versus $\delta^{13}C$ diagram for the investigated marbles.

Fig. 7 - $\delta^{18}O$ versus $\delta^{13}C$ diagram according to the metamorphic grade (a) and age (b) of the investigated marbles.

Fig. 8 – Position of the Piedmont white marbles (Western Alps) in the $\delta^{18}O$ versus $\delta^{13}C$ reference diagram. C-O isotope fields for the main Mediterranean marbles are from Lazzarini, 2004 and references therein.
a) MGS< 2mm. C = Carrara; D = Docimaenian; Hy = Hymettian; Pa-1 = Parian lychnites; Pa-3 = Parian from Karavos; Pe-1 and 2 = Pentelic.
b) MGS > 2 mm. Aph= Aphrodisian; N = Naxian; Pa- 2 = Parian from Lefkes; Pr-1 = Prokonnesian from Salaylar; Pr-2 = Prokonnesian from Çamlik ; T-1 = Taxian from Phanari; T-2 = Taxian from Alikì; T-3 = dolomitic Taxian from Vathy Saliara.

Fig. 9 – Discriminative flow chart of the Piedmont white marbles.