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STUDY ON ANCIENT MARBLES FROM IASOS OF CARIA

KARYA IASOS'UNDA BULUNAN ANTİK MERMERLER ÜZERİNE İNCELEME

Halit CANOL – Asuman BALDIRAN*

Abstract

The scope of research is taking samples which are pieces of marble at Ancient City of Iasos in this article. Samples from the various monuments of the ancient city of Iasos were analysed to determine the mineralogical-petrographic characteristics and the chemical composition. The main objectives of this research are the investigation of the basic chemical characteristics of these marbles. This article contained part of mineralogical and petrographic study and quantitative chemical composition analysis of these kind of marble. All these data compared information marble cave that territory of an Ancient City of Iasos such as Kalınağıl marbles. Considering results of analysis; marbles of Ancient City of Iasos had low metaformism, granoblastic texture and % 99 Calcite ($CaCO_3$) chemical compositions. According to the results, marbles properties of Ancient City of Iasos are close to in the quarries in the Muğla region. It has been determined that it is suitable sampling to be taken for conservation test in laboratory from these sources.

Keywords: Conservation of Archaeological Stone, Petrography, Optical Microsopy, SEM-EDX, XRD

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Öz

Bu makaledeki araştırmanın amacı, İasos Antik Kenti'ndeki mermer parçalarından örnekler alınarak, bu örneklerin mineralojik-petrografik özelliklerini ve kimyasal bileşimini belirlemek için analizler yapılmıştır. Bu araştırmanın temel amacı, bu mermerlerin temel kimyasal özelliklerinin araştırılmasıdır. Bu makale, mineralojik ve petrografik çalışmanın bir bölümünü ve bu tür mermerlerin kantitatif kimyasal bileşim analizini içermektedir. Tüm bu veriler, İasos Antik Kenti'ne yakın bölgelerde bulunan mermer ocaklarındaki (Kalınağıl gibi) çıkarılan mermerlerin verileri ile karşılaştırılmıştır. Analiz sonuçlarına bakıldığında; İasos Antik Kenti mermerleri düşük metaformizma, granoblastik doku ve %99 Kalsit (CaCO3) kimyasal bileşimine sahiptir. Elde edilen sonuçlara göre Muğla yöresindeki ocaklarda İasos Antik Kenti mermer özellikleri birbirine yakındır. Bu kaynaklardan laboratuvarda konservasyon testleri için numune alınmasının uygun olduğu belirlenmiştir.

Anahtar Kelimeler: Arkeolojik Taş Konservasyonu, Petrografi, Optik Mikroskop, SEM-EDX, XRD

1. Introduction

Iasos was established on a peninsula surrounded on three sides by the sea within Kıyıkışlacık neighbourhood that is 26 kilometers away from Milas (Fig. 1). It is an important coastal city that provides maritime communication with south of Valley of Meander. The city that was defined by Straboas 'an island next to the land' establishes the current peninsula by uniting with the mainland by a narrow isthmus.[1]

Fig. 1: Location of Iasos of Caria in Turkey

The ancient city of Iasos was constructed close to sea (Fig. 2). Antique marbles have remained on the ground without protection after all excavations. Fundamentally the causes of decay are the presence of soluble salts in water, the recrystallization of salts to increase humidity and the air pollution.[2] In the open air, due to its proximity to the sea, the sources of the salts in the ancient city were marine aerosols. Other sources of salts are the ground and the presence of animals and their waste. Natural stones have contained water-soluble salts in the bodies.[3]

[1] Baldıran-Pehlivan 2021, 19.
[2] Canol 2012, 9.
[3] Canol 2012, 1.

Fig. 2: Agora of Iasos, View from north

In archaeology is essential to develop the scientific research in material properties, prerequisite for restoration and conservation work. It is indispensable evaluating the deterioration rate and developing procedures for stopping or slowing down further degradation. All this has stimulated an enormous amount of detailed research on the use of marble in ancient times Iasos of Caria. The complexity is also related to the fact that the study of marbles from Iasos of Caria is a multidisciplinary activity involving the fields of archeology, history, art as well as the scientific disciplines of geology, chemistry, and physics [2]. This kind of multidisciplinary works occurred with permission of mission of Iasos of Caria and Milas Museum from 2010 to 2011. All analysis occurred in Laboratories of University of Bologna from 2011 to 2012.

2. Materials and Methods

2.1. Description of samples

Samples were taken from two different parts of Ancient City of Iasos: Agora and Balık Pazarı Museum. Piece of marble samples (there were element part an architectural monuments) came from surface of Agora of Iasos (AI). Samples codes were AI 1, AI 2, AI 3, AI 4, AI5, AI 6, AI 7, AI 8, AI 9, AI 10, and AI 11 (Fig. 3). Powder samples taken from calcareous surfaces of columns of Balık Pazarı’s mausoleum (I) (Fig. 4). Marble samples performed for mineralogical and petrographic study and powder samples performed for the quantitative chemical analyses.

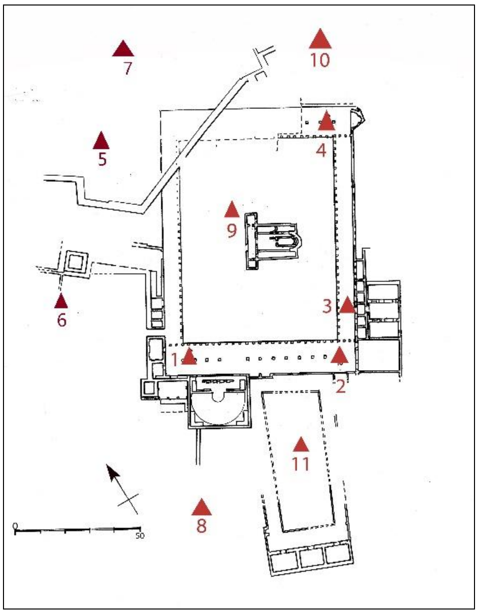


Fig. 3: Sampling in Agora

Fig. 4: Sampling place of powder samples

2.2. Microscopy

2.2.1. Optical Microscopy

The study in transmitted polarized light microscopy has provided information on the structure and crystal size and on the mineralogical composition. These characteristics have led to the classification of the examined rocks. In addition, micro photographic images were taken to document these features.

2.3. Scanning Electron Microscopy and Coupled with Energy Dispersive X-ray Spectroscopy (SEM-EDS) Analysis

Petrographic analysis of samples was completed with the study by SEM. The Scanning Electron Microscope is an electro-optical instrument that allows, following the emission of an electron beam, to analyze the various signals produced by the interaction of the electrons of the beam with the sample under examination. As regards the present study the analysis was carried out using a scanning electron microscope coupled with an Energy Dispersive spectrometer at X-ray (EDS). The polished sections of the samples to be analyzed were previously embedded in epoxy resin Araldite BY 158 with hardener Aradur 21. All observations were performed at the laboratory SEM / EDS of the Department of Earth and Geo-environmental Sciences of the University of Bologna with a scanning electron microscope equipped with a Philips 515b microprobe EDAX DX4; working conditions are: 15 kV, beam current 1 nA.

3.4. X-Ray Diffraction (XRD) Analysis

Samples I1, I2 I3, I4, I5, I6, I7, I8, I9 are from Fish Bazaar Museum's mausoleum. The samples consist of a coarse powder taken from the stone calcareous surface of the monuments. The samples were refined in an agate mortar, reducing them to very fine powder. The refined powders were placed on glass slides with the help of a spatula and deionized water, to obtain preparations semi-oriented. The glasses were then inserted into the sample holder chamber where, for analysis started, have received the incident beam of X-rays.

The XRD analysis was conducted using an automatic scanning diffractometer Philips PW 1710, equipped with an X-ray tube which emits monochromatic radiation (wavelength λ = 1.542 mm). The system is interfaced to a computer which, using special software Philips, calculates interplanar distances corresponding to each angle 2θ and the relative intensity of each detected signal.

4. Results

4.1. Results of Optical Microscopy

According to optical microscopy investigation of eleven samples showed that five different kinds of detected in general. Samples of AI1, AI2, AI3 and AI4 are low grade metamorphic rocks and AI1, AI2 and AI4 have consisted of a granoblastic polygonal calcite ($CaCO_3$) crystals with sizes of 50 μ; sample of AI3 have consisted of a mosaic of heteroblastic calcite ($CaCO_3$) crystal is 100 μ (Image 1,2). Samples of AI5 and AI8 are consists of a homogeneous mosaic of granoblastic, polygonal calcite ($CaCO_3$) crystals with sizes of more than 400 μ. It's coarse grained, high-grade metamorphic, marbles with isotropic textures (Fig. 5,6).[4]

Samples of AI 6 has a texture that markedly iso-oriented and consists of lenticular levels of marble derived by cataclastic fracturing (Fig. 5,6). The levels are composed by a mosaic of granoblastic, polygonal calcite crystals ($CaCO_3$) with sizes of less than 100 μ associated with quartz crystals (SiO_2). The surface of the lenticels of marble is coated with a film of iron oxides, which gives a red color to the whole marble. It's high-grade metamorphic, medium-fine grained, impure marble with cataclastic iso-oriented texture (Fig. 5,6). AI 7 is typical cell texture is due to the metamorphic crystallization of serpentine. Partially oxidized opaque minerals, sometimes in large aggregates, are arranged around the serpentine cells. Veins of talc with a thickness exceeding 100 μ cut the rock. It's low-grade metamorphic serpentinite with cell texture (Fig. 5,6). AI 9 is Xenoblastic texture is due to the association of calcite with quartz and white mica (muscovite), crystals with sizes of more than 400 μ. It's high-grade metamorphic pink, impure marble (Fig. 5,6). Samples of AI10 has granoblastic texture is due to the association of quartz crystals, partially oriented, with some white mica (muscovite) and rare opaque minerals (Fig. 5,6). Heterogranular texture, the crystal size varies from 500 to 200 μ. It has metamorphic quartzite. AI11 has texture is considerably laminated (Fig. 5,6). Are present layers consisting of a mosaic of heterogranular calcite crystals ($CaCO_3$) with dimensions below 50 μ, associated with rare quartz and opaque (Fig. 5,6). These levels are alternated with portions with the same mineralogical composition but with a crystal size greater than 50 μ [2].

[4] Canol 2012, 28-34.

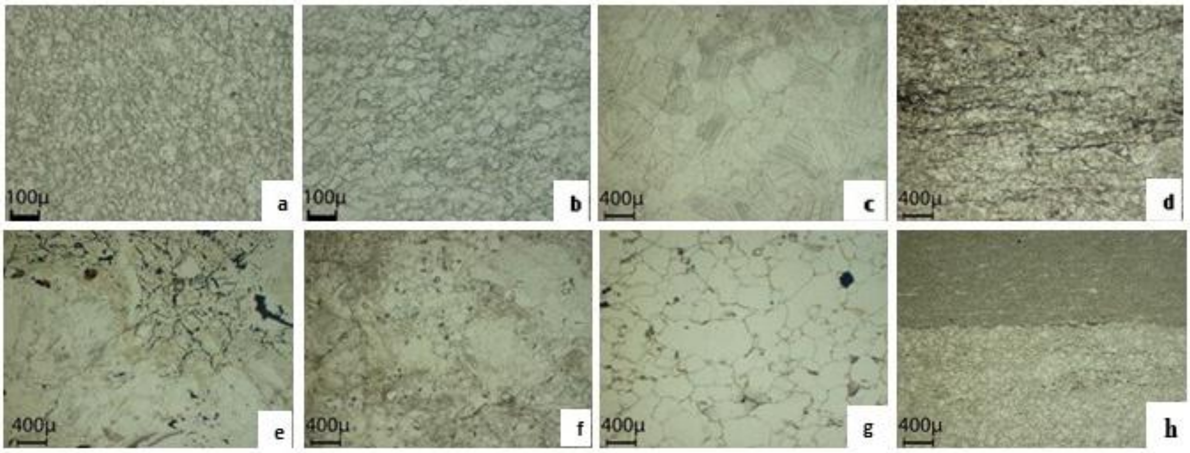


Fig. 5: Plane polarized light (PPL) visions; AI1 granoblastic, polygonal calcite ($CaCO_3$) crystals (a), AI3 mosaic of heteroblastic, polygonal calcite ($CaCO_3$) (b), AI5 coarse grained homogeneous mosaic of granoblastic, polygonal calcite ($CaCO_3$) (c), AI6 iso-oriented and consists of lenticular levels of marble derived by cataclastic fracturing with quartz crystals (SiO_2) (d), AI7 metamorphic crystallization of serpentine (e), AI9 xenoblastic texture is due to the association of calcite with quartz and white mica (muscovite) (f), AI10 granoblastic texture is due to the association of quartz crystals, partially oriented, with some white mica (muscovite) and rare opaque minerals (g), AI11 heterogranular calcite crystals ($CaCO_3$) (h).

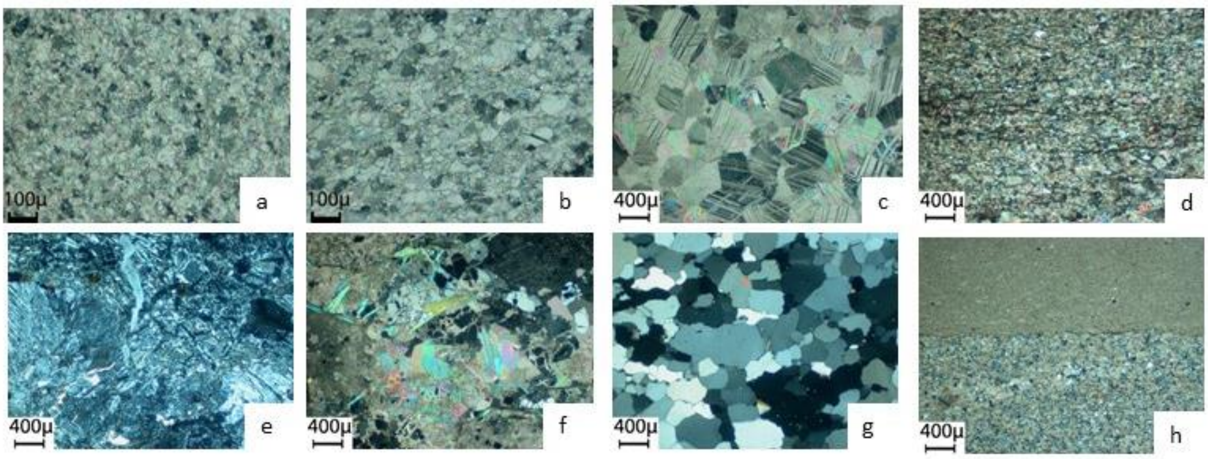


Fig. 6: Cross polarized light (CPL); AI1 granoblastic, polygonal calcite ($CaCO_3$) crystals (a), AI3 mosaic of heteroblastic, polygonal calcite ($CaCO_3$) (b), AI5 coarse grained homogeneous mosaic of granoblastic, polygonal calcite ($CaCO_3$) (c), AI6 iso-oriented and consists of lenticular levels of marble derived by cataclastic fracturing with quartz crystals (SiO_2) (d), AI7 metamorphic crystallization of serpentine (e), AI9 xenoblastic texture is due to the association of calcite with quartz and white mica (muscovite) (f), AI10 granoblastic texture is due to the association of quartz crystals, partially oriented, with some white mica (muscovite) and rare opaque minerals (g), AI11 heterogranular calcite crystals ($CaCO_3$) (h).

4.2. Results of Scanning Electron Microscopy (SEM-EDS) Analysis

A total of 11 particles were analyzed by SEM-EDX. According to their chemical composition, morphologies are variety (Table 1). The first mineral assemblage calcite + quartz + dolomite was observed in nine representative samples. In addition to this assemblage, all contained one or more of following minerals, apatite, pyrite, hematite. In here is not visibly hematite or iron oxide in AI10 with SEM imaging defined in EDS spectrography. In seven samples main component of texture is calcite and quartz. The second mineral assemblage titanite was observed in AI6 and AI7.In here following minerals; albite, allanite, amphibole, chlorite, magnetite, pekoite, serpentine.[5]

Dolomite and pyrite to occur in two samples (AI5 and AI8) and exhibits distinctive size in the texture; sizes dolomites are respectively 0.074 mm, 0.072 mm in AI5 and AI8. Pyrites dimensions are smallest than dolomites; pyrites sizes are respectively 0.0062 mm, 0.0024 mm in AI5 and AI8 (Fig 6,7) High pressure minerals assemblages found in marbles of Iasos region are unusual in that they are not typically observed at all. Minerals are assemblages such as calcite + quartz + dolomite and secondary minerals are hematite (some iron oxide?), muscovite-mica used to help constrain the temperature and pressure conditions to which the marbles were subjected. The assemblages, which are dependent on both bulk composition of the protolith (parent rock) and fluid composition, seem to indicate a water-rich, CO_2^- poor environment. The presence of titanite instead of futile, coexisting with calcite and quartz indicates a low value of CO_2^-.[6]

	Minerals														
Sp.Nº	*Ab*	*Aln*	*Amp*	*Ap*	*Cal*	*Chl**	*Dol*	*Hem*	*Mgt*	*Ms*	*Pek*	*Py*	*Qtz*	*Srp*	*Ttn*
AI1				▲	▲										
AI2					▲								▲		
AI3					▲										
AI4				▲	▲										
AI5				▲	▲		▲					▲			
AI6	▲	▲				▲		▲		▲					▲
AI7			▲						▲		▲			▲	▲
AI8					▲		▲					▲	▲		
AI9										▲					
AI10										●			▲		
AI11					▲			▲					▲		

● Smallest granular size

Ab- albite *Chl*- chlorite* *Ms* – muscovite *Srp*- serpentine*

Aln –allanite *Dol*- dolomite *Pek* –pekoite *Ttn*- titanite

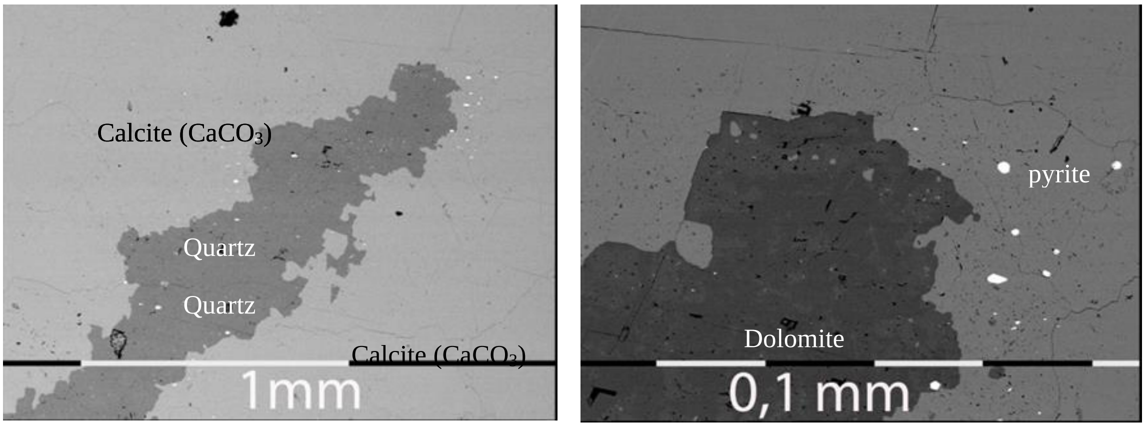
Ap- apatite* *Hem* –hematite *Py*- pyrite *Amp* -amphibole*

Cal – calcite *Mgt*- magnetite *Qtz* -quartz

Chlorite* *Names shown with an asterisk refer to series of minerals, i.e., they are not names of a single mineral species. This list is a compilation heavily inspired by the original listing of Kretz[7] (1983); it incorporates selected symbols from the listing of Spear (1993),[8] and includes symbols used by authors of papers published in The Canadian Mineralogist.*

[5] Canol 2012, 37-47.
[6] Ballevre-Micheal-Yves Lagabrielle 1994, 203-212.
[7] Kretz 1983, 277-279.

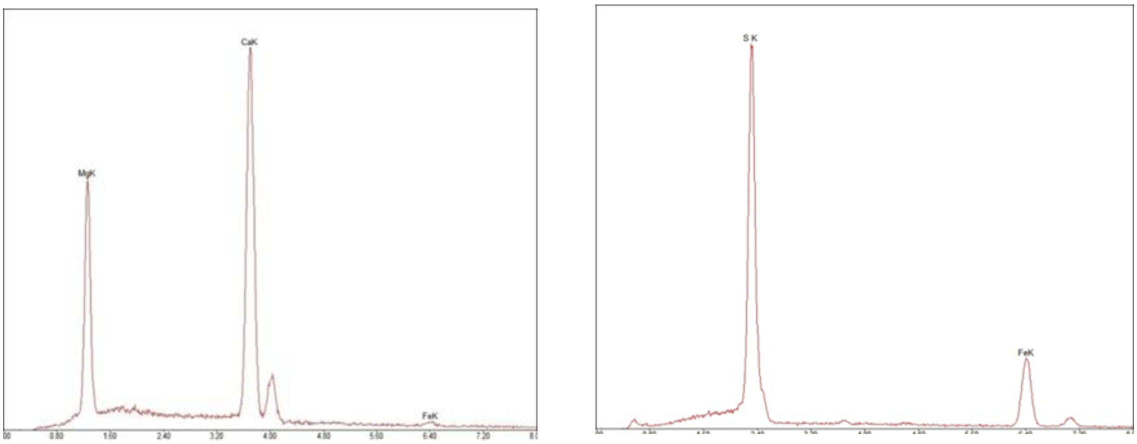
Tab. 1. Chemical compositions of all samples



a

b

Fig. 6. SEM vision of AI8; (a) Calcite ($CaCO_3$) and quartz (SiO_2) (b). SEM vision of AI8, annotated with minerals; pyrite (FeS_2) and dolomite [($CaMg(CO_3)$)]



a b

Fig. 7: EDX spectra of AI5; (a) dolomite [$CaMg(CO_3)$)] and (b) pyrite (FeS_2)

4.3. Results of X-Ray Diffraction (XRD)

Analyze assets have given information about powder samples which are collected from mausoleum of Fish Market Museum. Each sample clearly identified large proportion of calcite, minor dolomite, and quartz. As the investigated, samples were composed primarily either of calcite (Tab. 2). Although from this point of view all analyzed cold be divided into three categories: calcite dominant, calcite with small amount of quartz and calcite with small quantities of dolomite. Also excluding of main contest, another minor mineral contained; whewellite in I6. Three samples show the same evolution of calcite / quartz ratio (I7, I8, and I9).

The mineralogical composition of each sample described in XRD spectra and parameters,

These are:

$2\theta= 29.04$ and Å=3.04 nm (average peak of calcite)

[8] Spear 1993.

Sample	Calcite %	Dolomite %	Quartz %	Minor min.%	Whewellite %
I 1	98	-	2		
I 2	100	-	-		
I 3	100	-	-		
I 4	99	-	-	≤ 1	
I 5	98	2	-		
I 6	91	7	-		≤ 2
I 7	96	-	3	≤ 1	
I 8	99	-	1		
I 9	99.5	-	0.5		

Tab: 2. Quantitative XRD results of powder samples from Fish Market Museum

The investigation of I6 was composed primarily either of calcite, either of dolomite or whewellite. Whewellite is a mineral with hydrated calcium oxalate and formula has $CaC_2O_4.H_2O$. Because of its organic content it is thought to have an indirect biological origin, and this is supported by it being found in coal and sedimentary nodules. However, it has also been found in hydrothermal deposits where a biological source appears improbable. For this reason, it can be classed as true mineral. Whewellite, or at least crystalline calcium oxalate, does also arise from natural sources. [6] XRD data have been useful compare the other results of petrographic analysis. As a matter of average assets are %99 calcite ($CaCO_3$) (Figure 2) and these compounds generally finds in the lime stones and their metamorphism is marbles.[9]

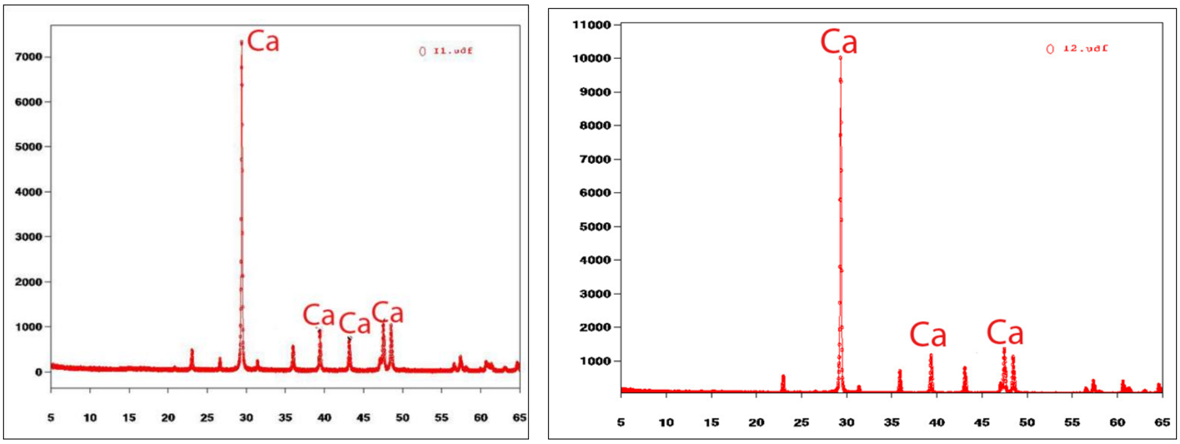


Fig. 2: $CaCO_3$ peaks of spectra (max. 2θ=29.3 and λ= 3.04)

[9] Canol 2012, 48-53.

5. Conclusions

Most of the samples analyzed, from the Agora of the antique city of Iasos of result to be marbles with one serpentinite and one quartzite (Tab. 3).

Marbles			Low Grade Serpentite	Quartzite
Low Grade		**High Grade**	AI 7	AI 10
AI 1	AI 6°	AI 5		
AI 2	AI11	AI8*		
AI 3	AI 4	AI9•		

° Red color, * White color, • pink color

Tab. 3: Classification for Rock samples of Agora of Iasos of Caria

These are high-grade metamorphic marble, pure, white, coarse grained, consisting exclusively of calcite. Others are impure and contain quartz and muscovite. Other marbles are of low or very low metamorphic grade, are very fine-grained, sometimes laminated and sometimes impure for the presence of quartz, with variable color from white to pink. There are also high-grade marbles intensely fragmented (cataclastic) and permeated by iron oxides (hematite) that give red color. Chemical analyses with XRD have been demonstrated for all samples have including calcite ($CaCO_3$). The XRD spectra of all analyzed samples show only the presence of calcite.[10]

Calcite ($CaCO_3$) basement stones have a hydrophilic surface, because it contains the formation of CO_3, aluminates, or oxides crystals are presences in samples of Agora of Iasos of Caria and Mausoleum of Balık Pazar Museum. The chemical structure of the stone ($CaCO_3$) is weighted; the surfaces are prone to break into acids react. Marbles have a different kind of degradations.[11]

Heterogeneity, many stones are not homogeneous, these causes of different hardness in the stone. This phenomenon is called differential weathering, in this case, the effect of wind and rain or the crystallization of salts cause the stones to deteriorate easily.[12]

In addition, main stones constituent of architectural monuments came from ancient marble caves (territory of Muğla) to the Ancient city of Iasos. Main of these ancient caves were Ioniapolis (Miletus and Heraclea), Kuşini.[13] Principally, rock units of these region rock units are divided into three parts; according to stratification are recognized a gneiss grain with a Paleozoic cover of schist. Last cover is coating on these two layers (is the layer of the old marble with increasing metamorphism with the direction of grain).[14] Grain is composed of meta-granites, gneisses, paragneiss, and meta-gabbros. Cover level includes schists and

[10] Canol 2012, 78-79.
[11] Canol 2012, 78-79.
[12] Canol 2012, 78-79.
[13] Monna 1977; Peschlow-Bindokat 1981, 157-235; Bingöl 2004.
[14] Durr 1975, 106; Ketin 1966; Şengör-Akkök 1984b, 693-707.

marbles. Marble cover is composed of schists alternating with the marbles of Triassic-Liassic age and shades in low grade marble levels.[15]

One of the characteristics of the marble of Muğla is the red color due to the presence of hematite. Marbles have been separated into four groups: emery-bearing marbles, yellow, grey, and white marbles. Marbles are composed mainly of medium to coarse grained calcite crystal, and display granoblastic texture. In emery-bearing marbles, tourmaline, hematite, chloritoid crystals, have been observed. Kalınağıl marbles consisting mainly of calcite include quartz, feldspar, muscovite, piemontite, chlorite and opaque minerals. The marbles and overlying lime stones have been analyzed for their major, trace and rare earth element contents. SiO_2, Fe_2O_3, Al_2O_3 contents of the rocks occurring in Kalınağıl region increase, CaO contents decrease.[16]

All data in this study are evaluated; it can be said that the marbles in the Ancient City of Iasos will light on the methods and materials to be selected for conservation studies (the physical properties of these marbles continue to be investigated). According to characteristics of the marble of Muğla region (e.g. Kalınağıl) is similar to some marbles of Ancient City of Iasos. In this case, new marble samples will take from these kind of marble quarries and it can be investigated convenient conservation methods in the laboratory environment for marbles of the ancient city of Iasos.

Yazarların Katkısı / *Author Contributions:* Çalışmaya; Yazar 1: % 95, Yazar 2: % 5 oranında katkı sağlamıştır. / To work; Author 1: 95%, Author 2: 5% contributed to the study.

Çıkar Çatışması / *Conflicts of Interest:* Yazarlar, herhangi bir çıkar çatışması olmadığını beyan eder. / The authors declare no conflict of interest.

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