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Physico-Chemical, Petrographical and Mechanical Properties of Mortars used in an Ancient Roman Basilica in Amasra/Turkey

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ABSTRACT

In this study, physicochemical, petrographic and mechanical properties of original lime mortars from a Roman Period basilica in Amasra dated to the 1st or 2nd centuries were examined. The study of mortar samples has been performed through polarising and stereo microscope observations, mineralogical (XRD) analyses, chemical (ICP-ES), physical and mechanical analyses. The micro structure and semi-quantitative chemical composition of the binding media and aggregate phases were determined by Scanning Electron Microscope with Energy Dispersive (SEM-EDS) analysis. This mortar possessed its hydraulic characteristic is due to the addition of volcanic rock with pozzolanic nature. Mechanical tests revealed that the average compressive strength of mortars is 6.1 N/mm^2 and flexural strength is about 3.3 N/mm^2. Pull-off tests for determining the adhesion properties proved that a good adhesion is established between mortar and andesite stone owing to its pozzolanic nature.

Keywords: Basilica, lime mortar, physicochemical- mechanical characterization, andesite, adherence of mortars

1. INTRODUCTION

Amasra is located on a small peninsula at the western part of Black Sea coast of Anatolia. The history of Amasra, the name of which was derived from the name of the Persian Princess Amastris, is dated to 3000 years back. The region has undergone the Ionian, Pontus, Roman, Byzantine, Genoa and Ottoman periods. The Romans, who dominated the region more than 450 years (70 BC-395 AD), left numerous edifices some of that perished away but many others are partially standing. Built by Gaius Julius Aquilla who was the major of the Emperor Tiberius Claudius Germanicus (41-54 AD), the monument is considered as "unique" by the historians of art and architecture since there is no other one alike in Anatolia. It has two inscription panels written in Greek and Latin languages.

It is thought that the Emperor Traianus (98-117 AD) made the greatest contribution to the development of Amastris and its expansion in a very wide area. The forum (administrative zone), the basilica (state parliament palace), amphitheatre (with 5000 audience capacity), protocol artery between the southern side of the basilica and amphitheatre, streets with regular pattern from acropolis towards the peninsula, potable water and sewage disposal networks are all believed to be constructed during Traianus Period. In addition to the basilica, the entrance of the amphitheatre, a certain portion of the fortress walls of acropolis, a few of the columns from protocol artery and the remnants from

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water distribution and sewage disposal systems once formed the infrastructure of the town can be seen on the site at present. As the pieces of some sculptures found during the recent excavations for the foundations of new constructions revealed, the *palaestra* (gymnasium) and the ruins of *agora* remained under the thick layers of alluvium accumulated throughout centuries (Fig. 1) [1].

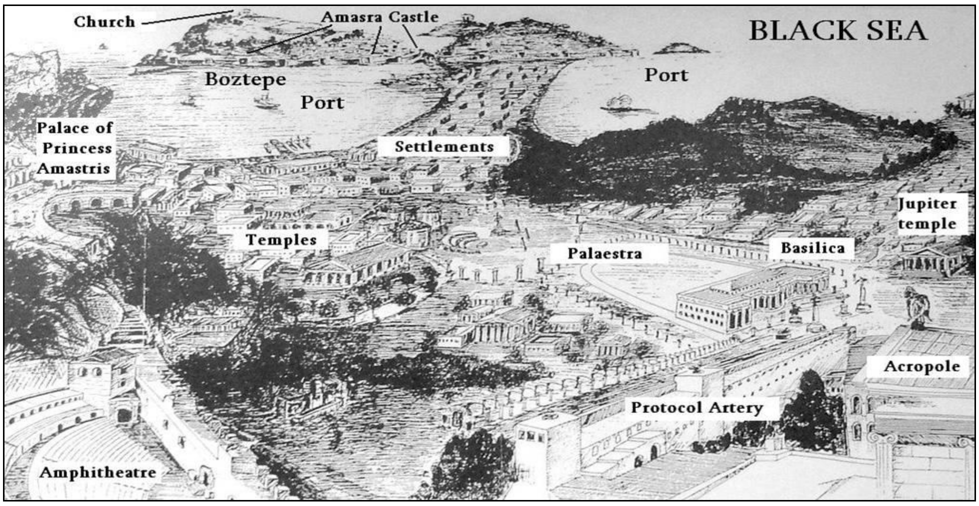


Fig. 1 Amastris in Roman Era (Ist century BC and IInd century AD) (Modified from the restitution drawings of Archeologist Özkan Beceren and Necdet Sakaoğlu in 1965 based on the remains on the site and plans of other antique towns) [1]

The building, being the subject of the study, was assumed to be a bath or a gymnasium previously, but defined as basilica at present is dated to the end of the 1st or the beginning of the 2nd century AD. It is also likely that it was one of the greatest monuments built by Romans in Anatolia [1] (Fig. 2a). The structure exhibits typical characteristics of Roman basilicas. As well as its external dimensions (45 x 118m), planning features and construction techniques employed are also conforming to the basilica definition addressed by Vitruvius [2]. The structure is named as *Bedesten* – covered bazaar in Ottoman Period – by local people today, likely due to that those buildings of the period also served for commercial activities as well as administration [1].

The basilica was built with the combined use of *opus reticulatum* and brick masonry techniques (Fig. 2b). It is identical to Roman architecture, but particularly to the period of Hadrianus (2nd century AD). *Opus reticulatum* is composed of cubic stones each side of which measures approximately 10-15cm and forming a grid with 45^{o} angles on the surface [3]. The southern facade of the building was completely collapsed and its features are unknown. Except its exterior decoration, the longer northern façade, however, is relatively well preserved. The major portion of the remaining walls is 1-1.5m in thickness, and built of andesite stone of volcanic origin and brick.

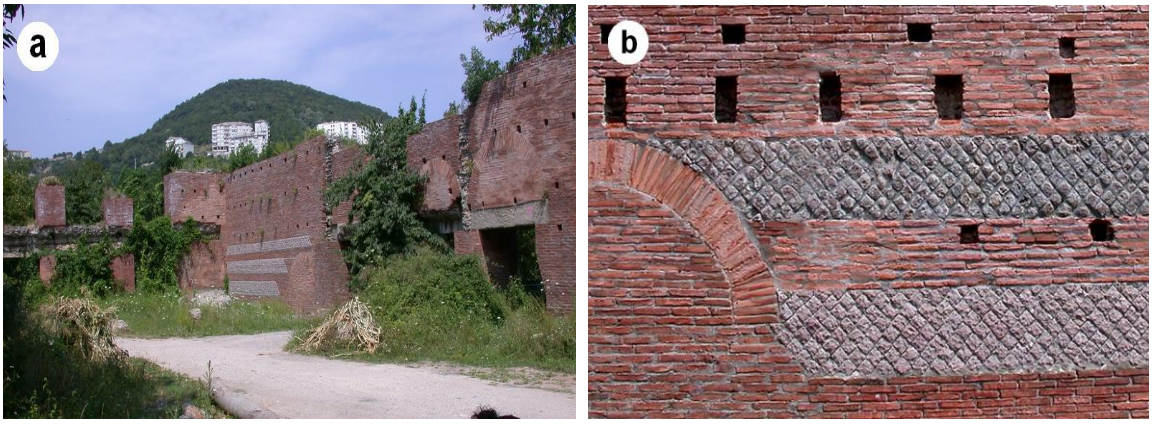


Fig. 2 [a] Basilica remains, [b] Opus reticulatum masonry

Efflorescence, observed on the bricks and joints in some parts of the basilica is the indicative of use of portland cement binder in the restoration mortars during a recent repair, which is deemed as an extremely harmful intervention material to be used in historic buildings (Fig. 3). While determining the raw material composition of the original mortars used in the building, the aim of this study is therefore to provide useful information for the production of intervention mortars free from soluble salts and compatible with the structure of the basilica in terms of physical and mechanical properties.

Fig. 3 Efflorescence on wall surface due to use of mortar containing portland cement binder during the previous restoration

The binding mediums of white-grey colored hydraulic mortars are lime, and, the aggregates of mortars are quartz and volcanic sand in different sizes as well as pebbles and rock fragments. The presence of volcanic stone particles in aggregates of cast mortars may indicate that this was a concrete production technique specific to Roman period. This volcanic material product of mortar is referred as *Opus Caementicium* and was used in many famous Roman monuments, such as the Pantheon, Roman Baths and Colloseum of Rome in the first century AD. [2,4,5]. Apart from this type, no other lime binding mortar was observed in Basilica. Therefore it can be deduced that the structure was not subjected to any repair work in later periods.

2. EXPERIMENTAL STUDY AND METHODS

2.1 Samples

Nine infill mortar samples in sufficient amounts and sizes were collected from four different spots (MA, MB, MC and MD) of stone masonry parts of the basilica for analyses (Fig. 4). The surfaces of the samples were cleaned with a soft plastic brush and properly cut according to the standard dimensions (40x40x160 ± 3mm). While they were subjected to the mechanical tests, the fragments of the same samples left after cutting process were utilized in physical, chemical, petrographic and SEM-EDS analyses. In addition, the tensile and the adhesion properties of another 4 mortar (MP) and 4 stone/mortar composite (MS) samples were determined by the specifically designed test method.

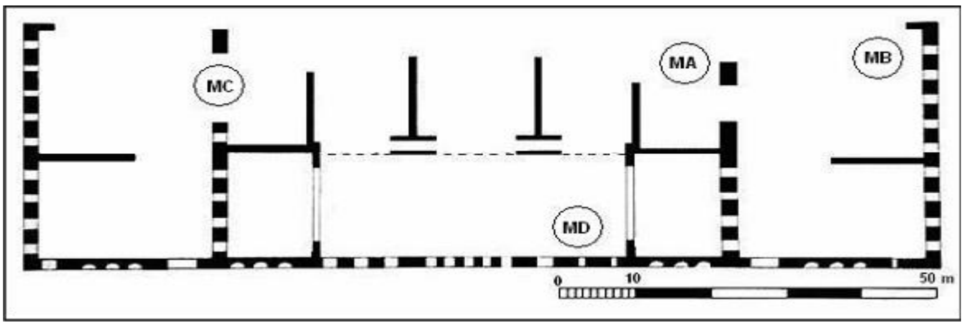


Fig. 4 Plan of Basilica remains [1] and locations of samples

2.2 Chemical analysis of mortars

For determining the chemical composition of mortars, and andesite rock, that is used as aggregate in mortars, 125 µm on sized 0,200 gr samples were dissolved with lithium metaborate ($LiBO_2$) fusion and analyzed using ICP-ES (*Inductively Coupled Plasma Emission Spectroscopy,* (ACME Labs)). As the deterioration agent or by products, the qualitative analysis of the water soluble salts as chlorine ($C\ell^-$), sulphate ($SO_4^=$), nitrate (NO_3^-) and carbonate ($CO_3^=$) were conducted by spot tests. For this analysis a stock solution (1.00 g sample in 100 ml pure water) was prepared and qualitative water soluble salts were determined. The presence of organic additives as proteins or oils was also analyzed by spot tests.

2.3 Analysis of samples by X-ray diffraction (XRD)

The semi-quantitative mineral composition of the lime mortars and andesite rock was determined through X-ray diffraction (XRD) analysis using a Philips X-Pert Pro X-Ray diffractometer with CuKα radiation. Patterns were obtained by step scanning from 5° to 70°, 2θ with exploration speed of 2.12°/min at 45KV and 40mA.

2.4 Physical, petrographic and microscopic analysis

European EN 1936 [6] and Turkish TS EN 13755 [7] standards were used for the determination of basic physical properties, such as bulk density, apparent porosity and water absorption capacity of mortars. Stereo microscope (MBC 10, with single nicol) was used in for determining the morphological features on cross sections of the samples. For the polished thin section analyses of mortars to examine their mineral contents and textural features, mortar samples were molded in epoxy (Raku-tool EL-2200 and hardener EH-2900), were cut, stuck on lam, thinned to the 30 µm and examined through polarizing microscope (Soif, with double nicols).

2.5 Scanning Electron Microscope with Energy Dispersive (SEM-EDS) analysis

The micro structure and semi-quantitative chemical composition of the binding media and aggregate phases were determined by EVO/LS10 ZEIZZ SEM and BRUKER EDS. For this analysis the samples were cut and polished by STRUERS Discoplan-TS diamond saw.

2.6 Mechanical analysis and pulse velocity measurements

Before mechanical tests, the dynamic modulus of elasticity of 9 mortar samples (40x40x160±3mm) was calculated by the measurements of ultrasonic pulse velocity (UPV) was performed with PUNDIT (CNS Electronics Ltd.) equipment with the transmitter and receiver probes of 54 kHz [8]. Flexural strength was determined based on the Turkish standard TS EN 1015-11 [9], with a universal hydraulic press. The compressive and tensile splitting tests were carried on half of the samples, remained from the flexural test. Compressive strength test was based on TS EN 1015-11 and tensile splitting test on TS EN 12390-6 [10]. Mechanical tests were carried out by using an AMSLER Type 6DB7F120 universal hydraulic press, 60-600 N capacity device with ¼ loading speed.

3. RESULTS AND DISCUSSION

3.1. Chemical properties of mortars

The general chemical composition of mortars and andesite stone used as aggregate were determined as oxides in percentages and trace elements remained were determined as ppm by ICP-ES analyses. The hydraulic index (HI) and cementation index (CI) were also calculated using SiO_2, Al_2O_3, Fe_2O_3, MgO and CaO values. The results are shown in Table 1 and Table 2.

Table 1 Chemical composition of mortars and andesite rock (HI: Hydraulic Index; CI: cementation index of mortars)

oxides / sample	SiO_2	Al_2O_3 (%)	Fe_2O_3 (%)	MgO (%)	CaO	Na_2O (%)	K_2O	TiO_2	P_2O_5 (%)	MnO (%)	Cr_2O_3 (%)	HI	CI
MA	17.	3.69	2.74	1.16	39.	0.44	0.7	0.2	0.1	0.08	0.008	0.6	1.36
MB	29.	6.92	2.27	1.35	28.	1.09	1.6	0.1	0.11	0.07	0.004	1.2	3.01
MC	34.	7.51	3.09	1.93	25.	1.23	1.9	0.2	0.13	0.09	0.01	1.6	3.87
MD	34,	7.57	3.16	1.96	27.	1.34	1.9	0.2	0.11	0.09	0.01	1.5	3.57
Andesit	59.	17.76	4.33	1.36	3.7	4.42	5.7	0.4	0.25	0.13	0.004	-	-

Table 2 Trace element contents of mortars and andesite, (C) and (S) ratio in total content and ignition loss

elements / samples	Cu ppm	Ba ppm	Zn ppm	Ni ppm	Co ppm	Sr ppm	Zr ppm	Ce ppm	Y ppm	Nb ppm	Sc Ppm	Ta ppm	LOI %	TOT %	TOT/C %	TOT/S %
MA	120	326	33	<20	<20	295	113	<30	10	<5	6	<20	33.9	99.94	8.97	<0.02
MB	457	475	73	32	<20	267	63	<30	13	7	7	<20	28.1	99.97	5.59	0.06
MC	554	465	90	38	<20	315	70	<30	14	10	11	<20	24.2	99.98	3.92	<0.02
MD	277	436	59	29	<20	348	58	<30	14	9	11	<20	21.3	99.95	5.08	<0.02
Andesite	175	886	66	52	39	423	107	<30	21	10	7	<20	1.82	99.91	0.14	<0.02

As the results of ICP revealed, CaO ratio in mortars varied between 25.0 - 39.1%. These results imply that the mortar contains high ratio of $CaCO_3$ (Table 1). On the contrary of very low amount of magnesium oxide and high amount calcium oxide contents showed that the binder of the mortars is essentially calcitic lime with low dolomitic property. The results of ICP-ES performed on andesite stone, high content of SiO_2, Al_2O_3 Fe_2O_3, and Na_2O together with the same compounds found in mortars proved that the dust and fragments of andesite stone was used as aggregate in mortars. The comparison of the ratios of these compounds in mortars with the andesite rock sample, were also indicated that the aggregates are based on the andesite.

In addition, oxide compound concentrations obtained through chemical analyses give important information about the hydraulic properties of mortars. According to the ICP results, hydraulic (HI) and cement (CI) indexes were calculated with Boynton formula (Eq. 1 and 2) [11] and shown in Table 1. All *Hl* (<0.4) and *Cl* (0.7-1.1) indexes of mortars are mostly parallel to each other and present high values (except MA). It is well known that, if these indexes of binder are high, its hydraulic property is high as well [12, 13]. According to the *CI* and *HI* values of binder, all mortars can be classified as moderately or highly hydraulic binder.

$$Hl = \frac{\%Al_2O_3 + \%Fe_2O_3 + \%SiO_2}{\%CaO + \%MgO} \quad [1]$$

$$Cl = \frac{1.1\%Al_2O_3 + 0.7\%Fe_2O_3 + 2.8\%SiO_2}{\%CaO + 1.4\%MgO} \quad [2]$$

Besides, the parallelism between trace element concentrations such as barium (Ba) and strontium (Sr) indicates that the source of these elements is also those aggregates (Table 2).

3.2 Water soluble salts and analysis of protein and oil

In literature, it is mentioned that the additives such as casein, urea, albumin, oil etc., were used in Roman period mortars [14, 15]. Qualitative determination of water soluble salts (chlorine, sulphate, carbonate and nitrate salts) and possible additives such as saponifiable oil, protein, were done by simple spot tests and the results were shown in Table 3.

Table 3. The results of analysis for salt and organic materials in mortars

samples	salts				organic materials	
	Cl^-	NO_3^-	SO_4^{2-}	CO_3^{2-}	protein	oil
MA	-	+	±	-	-	-
MB	±	-	±	-	-	-
MC	±	±	+	-	-	-
MD	+	-	±	-	-	-

- = none, ± = available-none, + = very few available

It is assumed that the presence of low amount of chlorine salt [Cl^-] is provided by soil or the material itself. The presence of nitrate salt [NO_3^-] however is probably due to birds droppings and residues of the insects, microorganisms etc. and sulphate [$SO_4^=$] is the result of air pollution. The absence of oil and protein in the analyses revealed that organic additives were not used in the production of mortars of the basilica.

3.3 Physical properties of mortars

According to physical test results results, the unit weight of mortars varied between 1.64-1.83g/cm^3, apparent porosity 26.7-33.1% and water absorption ratio between 14.9-19.9% respectively (Table 4).

Table 4 Physical and mechanical properties of mortars

Group	samples	bulk density (g/cm^3)	apparent porosity (%)	water absorption (%)	pulse velocity (km/sn)	E_d (kN/mm^2)	compressive strength (N/mm^2)	flexure strength (N/mm^2)	splitting strength (N/mm^2)
MA	M1	1.70	32.6	19.2	2.5	10.5	6.6	4.7	4.6
	M2	1.69	30.5	17.0	2,1	8.3	5.0	3.2	3.3
	M3	1.79	26.7	14.9	2.7	13.5	8.9	4.3	6.8
MB	M4	1.82	29.2	16.0	2.5	11.9	6.5	3.1	3.9
	M5	1.79	29.7	16.6	2.5	11.5	5.5	3.2	3.6
MC	M6	1.67	33.1	19.9	2.6	11.3	5.0	3.4	3.5
	M7	1.64	32.3	19.7	2.4	9.9	4.5	2.8	3.1
MD	M8	1.72	31.6	18.4	2.3	9.5	5.7	2.5	3.2
	M9	1.83	28.5	15.6	2.7	13.1	6.8	4.6	5.0
	average	1.74	30.5	17.5	2.5	11.1	6.1	3.5	4.1

Depending on the types of pozzolans and aggregates, the unit weight of various lime mortars generally used in historical buildings is about 1.5-2.1 g/cm^3 and their porosity ratio changes between 18-45% [16]. The results obtained from the mortars of the basilica above are also similar to the values reported in the literature.

3.4. Microscopic analysis, binder and aggregates

The texture, pore structure and other morphological characteristics of mortars were examined through stereo microscope on polished cross sections. As well as the pores in different sizes, voids, likely occurred following the shrinkage occurred in hydrated lime lumps were also detected (Fig. 5a). These voids were present in all mortars, but in some of the samples voids are relatively more in number, thereby their porosity, and respectively, water absorption ratios are higher than the rest. Although rarely, but some cracks were also observed in some of the samples. It is noticed that the surfaces of both cracks and voids covered or filled with the secondary calcite layer formed by the re-carbonation of precipitated lime binder.

By the same observations, it is estimated that the volume of binding part is around 35-45%, and that, the aggregate phase is composed of andesite and other siliceous aggregates in different sizes in addition to 5-10% calcite particles. The shells with flat shapes found in aggregate parts imply that those aggregates may be originated from marine environment. If the calcite particles, reacted with hydrochloric acid are taken into account, aggregate/binder ratio might change between 2:1 and 1:1.

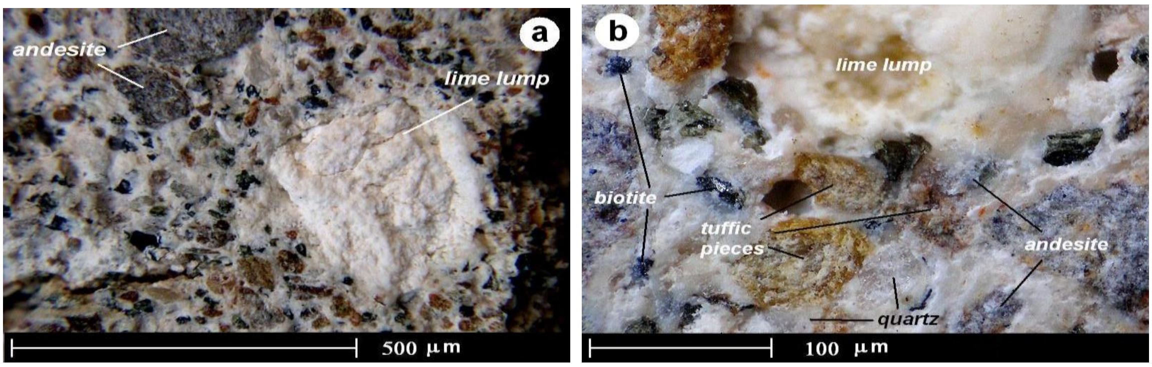


Fig. 5 [a] The textural view by stereo microscope, [b] Minerals and volcanic rock fragments

Calcite minerals detected in mortars are natural and due to carbonized lime, and, feldspars and biotite are originated from volcanic aggregates (Fig. 5b). Binder and aggregate phases in different size and types in the texture of mortars are in good condition. In all samples generally rounded lime lumps in various dimensions which occupied large volume of spaces were observed. However, relatively smaller, 2-10 mm sized shrunk and cracked lime lumps were also noticed as unstable spots. These two cases can be explained in two groups:

- white colored, homogeneous and hard lumps (Fig. 6a),

- orange-red colored, shrunk and cracked lumps (Fig. 6b).

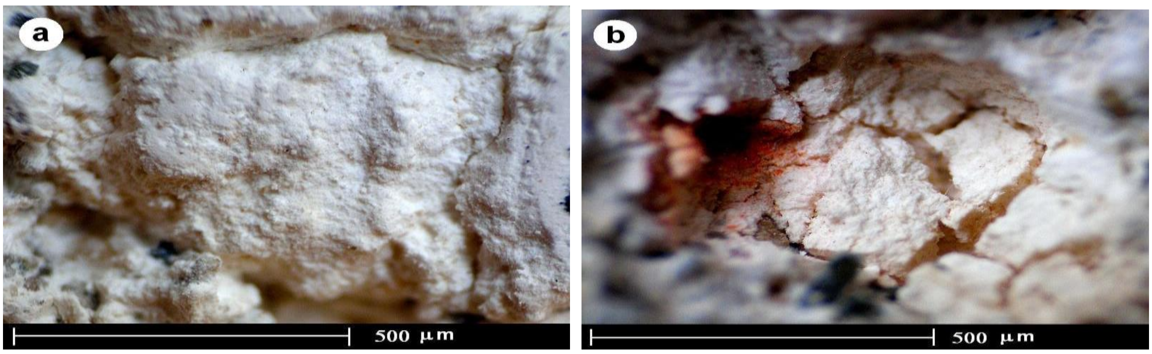


Fig. 6 Lime lumps in the mortar, [a] pure $CaCO_3$, [b] lime lump shrinkage cracks

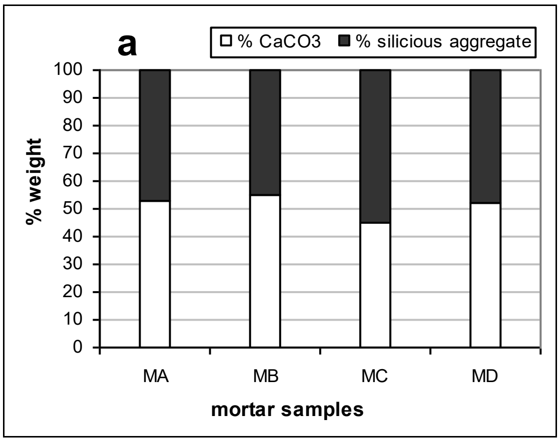
White colored homogeneous lumps, being pure $CaCO_3$, can be considered as those completed their carbonization in lime pits, therefore not mixed with the aggregates and other dust sized particles, hence, functioned as calcitic aggregates in mortars. On the other hand, colored lumps with cracks are those remained without mixing with the aggregates, carbonated in the mortar, shrunk and cracked by volume change highly likely due to over hydration and/or fast drying. Their colored appearance is probably due to volcanic and dust sized materials mixed during lime slaking.

The presence of such lumps was also observed by some authors mentioning that the lumps would have indicated the type of the lime. For instance, an examination on 12-13th century mortars, these were determined as high calcium-concentrated limes containing less than 5 % SiO_2 and MgO components [17]. This result is very common in historical lime mortar researches [18, 19]. Hughes and Leslie demonstrated that these lumps, found abundantly in historic mortars, may have a variety of textures that reflect the nature of the original limestone source rock [20]. According to results of analysis of 1st century mortars, these are the indicatives of hydraulic and non-hydraulic limes. While the non hydraulic group contains high amount of calcite and trace amount of quartz, the hydraulic group is composed of CaO (quick lime) and activated SiO_2-Al_2O_3 (marn-clay) minerals [18]. Despite the similarities between basilica mortars and those mentioned in the literature above, the lime used as binder in the mortars of the basilica are non hydraulic as it was proved by XRD analysis.

3.5 Particle size distribution of mortars

Except the little amount of brick particles and pebbles, all big sized aggregate components generally composed of volcanic rock fragments as andesite (mainly) and tufa (little amount), while the smaller aggregate part is composed of quartz sand, volcanic rock particles and dusts. Aggregates, usually angular in shape, have strong adherence to the binder and stabilize the mortars. Besides, the angular shape of andesite aggregates indicate that those were added in mortars as broken pieces of this stone.

The analysis of acidic treatment shows that 45-55% of mortars were dissolved (Fig. 7a). As microscopic observations showed above, the binding media of samples were estimated to be varied between 35-45 %. Therefore it can be deduced that the aggregates composed of carbonate particles, which are also dissolved in acid were 5-10%. If these carbonated aggregates are added to those siliceous aggregates, aggregate/binder ratio is 2:1 to 1:1. While Vitrivius, Ancient Roman architect, states that the aggregate/lime ratio for pozzolanic mortars is 3:1 [2], Plinius recorded this ratio as 4:1 [4, 21]. Some researchers express that the aggregate/binder ratio in Roman structures is identical with those above; however, they claim that the ratio is usually 2:1 [22, 23].



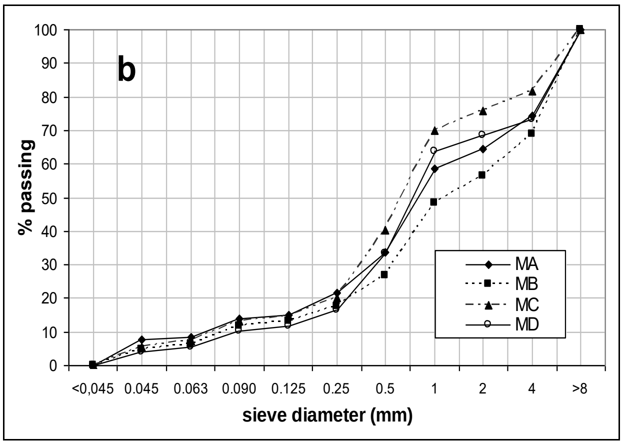


Fig. 7 [a] Aggregate/binder ratio of mortars, [b] grain size distributions of the aggregates

As the granulometric curves confirm collected mortars are mostly composed of similar grain sizes and distribution (Fig. 7b). The grains of each sieve sizes were also examined for their mineral contents under stereo microscope. The results are given in percentages in Table 5. As the table shows, 5-10% of 45 mm sized aggregates and those below are quartz, and the rest of the aggregates are composed of feldspars. This composition of the minerals observed by this analysis, also indicate that most of the siliceous aggregates were andesite.

While using mostly local material sources, it seems that Roman master builders also preferred volcanic stones as pozzolanic aggregates. In literature, it is mentioned that grinded volcanic tuff and pyroclastic rocks which contained pozzolanic components such as volcanic glass, sanidine, and pyroxene [4], were used as natural pozzolan in hydraulic mortars of Roman monuments [24]. Basilica mortars are typical examples of Roman mortar production and prove that the identical production technology was also applied in Northern Anatolia.

3.6 Petrographic Properties of Mortars

By the petrographic analyses, it is determined that apart from the quartz, mortars are composed of volcanic rock aggregates (Fig. 8 a,b,c,d). Mostly found feldspars, biotite, tuffic pieces, are the minerals of broken pieces of the andesite rock. The angular shapes of those pieces indicate that andesite rock were crushed and might be grinded during the production. Feldspar minerals detected in mortar textures (sanidine, pyroxene) are also the minerals of andesite. The hydraulic properties of mortars are provided by these dust sized pozzolanic materials.

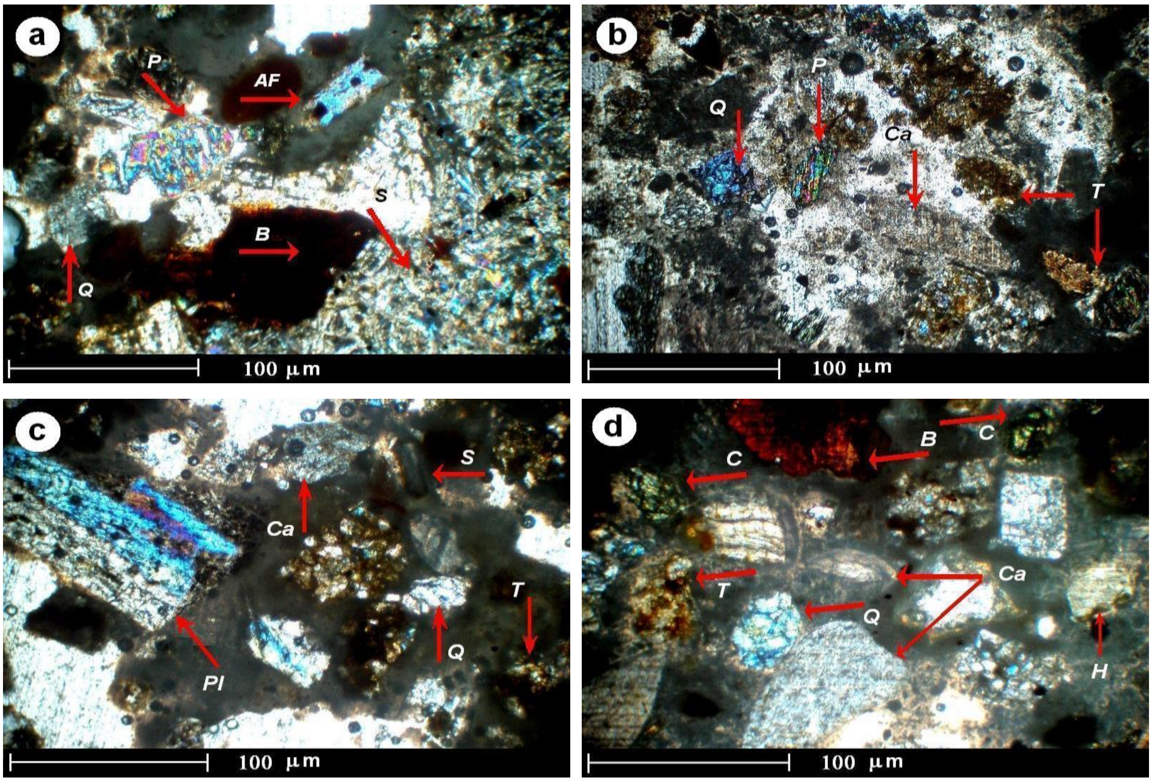
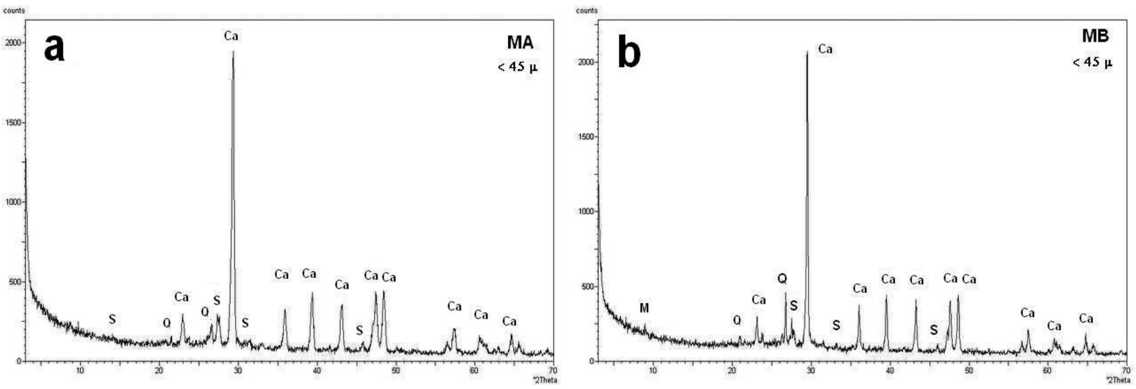


Fig. 8 [a,b,c,d] Thin section view (double nicols) of mortars and their minerals (Ca: calcite, B: biotite, Q: quartz, Pl: plagioclase, P: pyroxene, AF: alkaline feldispar, S: sanidine, C: chlorite, H: hornblend, T: tuffic pieces.

3.7 XRD analysis of mortars and andesite rock

In order to determine the mineralogical composition of binder of the mortars, the samples were taken from binder parts between aggregates. They were sieved with 45 µm sized sieve and subjected to the X-Ray diffraction analysis. The results are shown in Table 5.



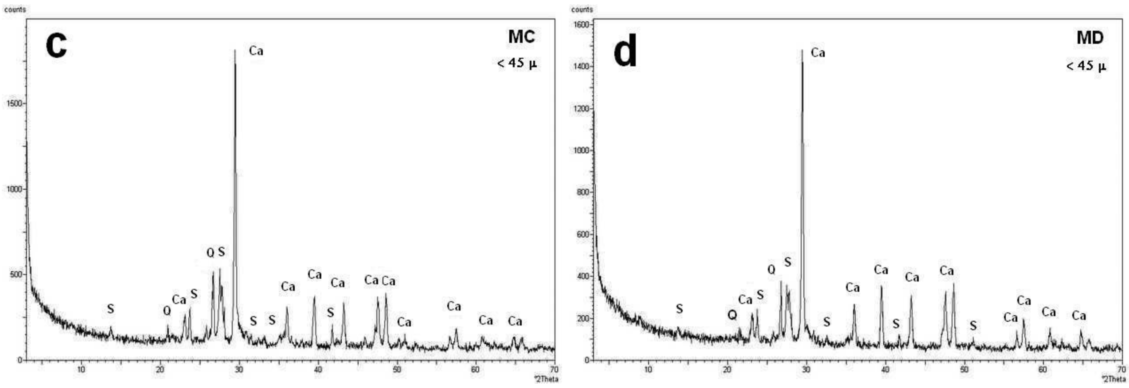


Fig. 9 [a,b,c,d] XRD pattern of mortars (Ca: Calcite, Q: Quartz, S: Sanidine, M : Muscovite)

Table 5 Mineralogical composition of the mortars and andesite

minerals / samples	quartz (Q)	sanidine (S)	muscovite (M)	illit (I)	orthoclase (O)	calcite (Ca)
MA	±	+	-	-	-	+++
MB	+	+	±	-	-	+++
MC	+	++	-	-	-	+++
MD	+	++	-	-	-	+++
Andesite	++	+++	-	+	++	-

+++ : abundant, ++: present +: small amount, ± : minimal, - : undetected

The X-Ray diagrams of samples (Fig. 9 a,b,c,d) are generally identical and their binders are mostly composed of calcium carbonate but also contained sanidine minerals likely used as pozzolanic additive. Sanidine is alkali feldspar (K,Na)(Si,Al) $4O_8$) mineral and it is common in andesite rock. Large amount of sanidine minerals which were detected by XRD analysis of andesite rock sample (Fig. 10 a, b) proved that they were originated from andesite. As reported in the literature, when they react with slaked lime, $Ca(OH)_2$, they yield tetracalcium aluminum hydrate which imports hydraulic properties to the mortar [25, 26]. The existence of quartz peaks likely originated from volcanic rock may imply that the binder is slightly hydraulic, in other words, the limestone used in the production of lime contained small amount of clay. The SEM-EDS analyses are also support this phenomenon. A research on mortars of a Roman period temple, Serapis Temple in Pergamum (7th century AD), reveals that the Roman mortars are composed of pure lime, coarse aggregates with particle sizes greater than nearly 1 mm and pozzolanic fine dust [27]. Thus, it can be concluded that, although the non hydraulic lime used as binder, due to the use of pozzolanic additives and aggregates, the mortars used in the basilica are hydraulic.

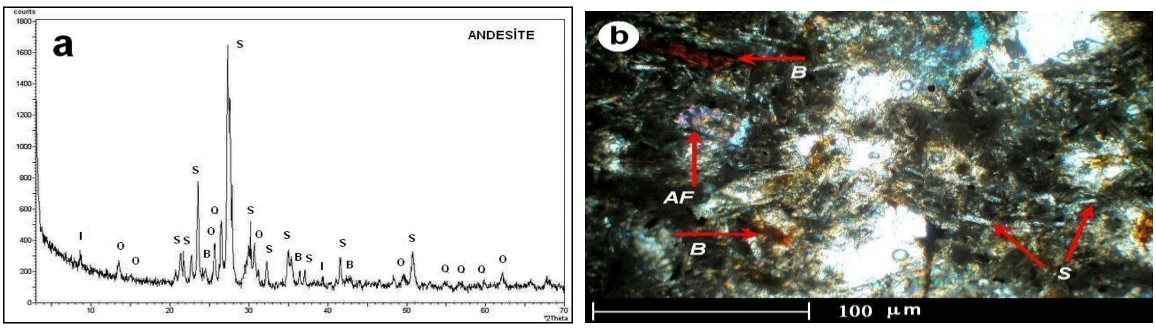


Fig. 10 [a] XRD pattern (S: sanidine, Q: quvartz, :I: illite, O: Orthoclase) and [b] thin section view (double nicol) of andesite rock

3.8 Mechanical properties of mortars

The results of mechanical tests showed that, compressive strength of mortars are about 4.5-8.9 N/mm², flexural strength 2.5-4.7 N/mm^2 and tensile splitting strength is between 3.1-6.8 N/mm^2 (Table 4) (Fig 11). Generally, compressive strength of mortars produced by non hydraulic lime and hydraulic lime, are in between 0.5-2.0 N/mm^2 and 2.0-1.5 N/mm^2 respectively [28, 29]. Regarding to this generalization, it can be seen that the mechanical properties of basilica mortars are close to the mechanical properties of hydraulic lime mortars. Undoubtedly, the type of aggregate has an important role in determining the mechanical properties. Together with the tuffic particles, the presence of pores and calcite lumps in binder phase cause relatively low mechanical values in some samples (MA2, MC6, MC7). Dynamic modulus of elasticity (E_d) values, which are evaluated from ultrasound pulse velocity (UPV) of these samples, are low and porosity ratios are higher than the other samples. As acknowledged, porosity has a considerable effect on both the durability and mechanical properties of the material [30]. When the crack surfaces were observed after flexural and tensile splitting tests, it is determined that the cracks were occurred in binder phase and andesite aggregates remained stable. This indicates that the high content of binders and weak calcite (lime) lumps in different dimensions in binder phase, have usually negative effect on mechanical properties.

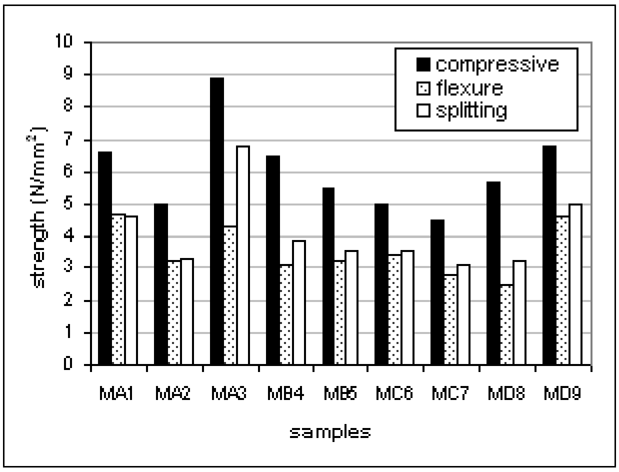


Fig. 11 Mechanical properties of the mortars

3.9 Adherence of mortars

Pulling tests were made in order to determine the adherence of mortars with stones. The results and test apparatus are shown in Fig.12 Intermediate surfaces of broken pieces were measured and splitting regions were determined in mm^2.

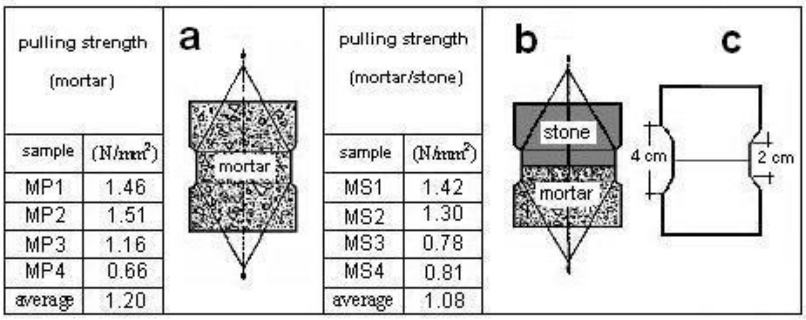


Fig. 12 [a] Mortar, [b,c] mortar/stone pulling test apparatus, and results

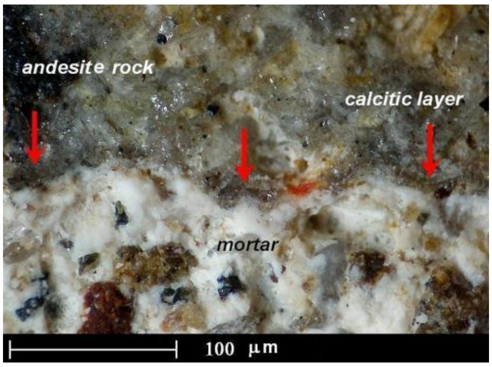


Fig. 13. Secondary calcite layer formation between andesite aggregate and binder

The adherence tests provided that the pulling strength of mortar/mortar system and mortar/stone system are in between 0.66-1.46 N/mm^2 and 0.78-1.42 N/mm^2 respectively. These values can also be accepted as the tension value of mortar. Because, splits did not occur on phase (inter surface) of mortar/stone but on mortar/mortar phase. Namely breakings are not due to adhesion of mortars with stone at the inter surface, but the cohesion strength of mortar itself. This indicates that the binder has strong adhesion with andesite stone surfaces and volcanic originated aggregates functioning as pozzolan have positive effect on mortar adhesion (Fig 13). Moreover, the pozzolanic effects of crushed andesite dusts on lime mortars were determined through Standard ASTM C 593-95 pozzolanic activity test [31]. and it is found in between 3.9-4.6 N/mm^2. This property provides cohesion features to the mortars and increase the adherence in consequence of chemical reaction in binder/aggregate phase caused hydraulic reaction and produced CSH (calcium silicate) and CAH (calcium aluminate) interphases. As a result, in mechanical terms, binder/aggregate phases (adhesion) are stronger than binder/binder (cohesion) phase. If large calcite areas and lumps are not considered, it can be said that binder and andesite aggregate surfaces combine well and diffusion of binder is homogenous on surfaces as observed by stereo microscopic examinations and SEM-EDS analysis.

Volcanic aggregates contain considerable amount of vitreous matrix and unstable minerals, such as sanidine feldspar, favoring chemical reaction with the lime, in the interfaces [4]. Therefore it both facilitates the chemical reaction with binder and provides hydraulic property to mortar.

3.8 SEM-EDS analysis of Mortars

The comparison of EDS analysis result of andesite stone (A4, N1 and N2) with the mortars indicated that the binding area A1, A2, A3 and binding points N3, N4, N5 have both lime and andesitic material. (Fig.14 a,b), (Fig. 15 a,b). According to the EDS results, while the point N3 is rich in lime, the points N4 and N5 are rich in andesine particles. These results also indicated that the pozzolanic properties are gained by these dusted andesitic particles. The high amount magnesium oxide (MgO) of the binding media (A2, A3 and N3) indicated that the lime used in the mortars has dolomitic properties. The EDS results of the area (A1) and points (N4 and N5), which have quite high amount MgO, also support this thesis, that they should be dolomite rich parts of the binding media (Table 6). The high content of iron oxide (Fe_2O_3) at A1, N3, N4 and N5 should be caused by the iron minerals of the andesite rock particles.

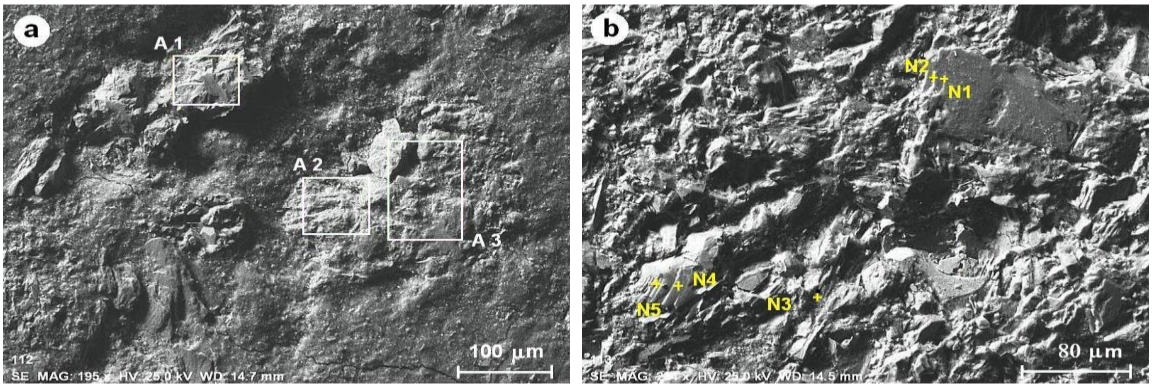


Fig.14 [a] SEM photos and [b] EDS (area and points) analysis of mortar samples.

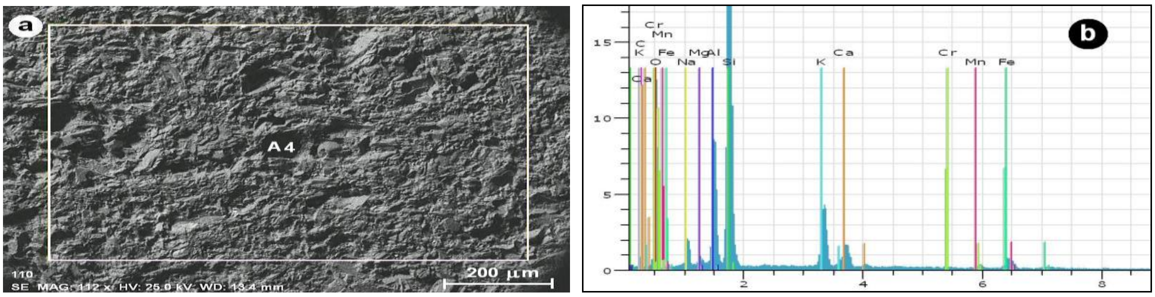


Fig.15 [a,b] SEM photo and graphic of the semi-quantitative EDS analysis of andesite stone, Area 4.

Table 6 Semi quantitative EDS analysis of andesite stone and mortar samples.

points / elements	A4(An)	A1	A2	A3	N1	N2	N3	N4	N5
CaO	4.83	22.96	77.19	75.97	1.42	2.96	57.27	21.03	21.34
SiO_2	55.25	44.20	8.96	10.14	56.54	58.30	27.37	45.81	47.69
Al_2O_3	20.07	8.90	5.29	6.26	21.70	20.80	2.44	3.26	2.95
Fe_2O_3	3.07	7.30	1.72	2.19	1.86	-	6.28	6.10	5.69
K_2O	8.26	-	0.09	0.23	8.86	12.81	-	-	-
Na_2O	5.79	1.89	1.13	-	8.07	5.12	-	1.70	-
MgO	1.56	14.75	5.62	5.22	1.55	-	4.32	21.16	20.50
MnO	1.12	-	-	-	-	-	1.20	0.94	1.83

A: Area; N: Point; An: Andesite stone

4. CONCLUSIONS

The white colored masonry mortars and fillings are generally identical, thus, indicating that produced by the same techniques. Slaked non hydraulic lime with low dolomitic properties was used as binder in all mortars. The aggregates of mortars are usually composed of crushed andesite fragments. Except few calcite particles, the coarse aggregate components of all mortars contain angular volcanic particles. Angular shape indicates that these are broken pieces of stone and increases the surface areas and more actively react with lime as owing to their pozzolanic nature. Therefore, the adherence of aggregates with binder also increases. Smaller aggregate part is composed of quartz and andesite originated feldspar minerals which provide hydraulic property of mortars. Aggregate/binder ratio is usually varying from 1:1 to 2:1 indicating high content of lime use. It is also confirmed by the large calcite lumps on mortar surfaces. The presence of lime lumps indicated that the carbonation process of slaked lime is partly carried in lime pit, after slaking of quick lime in the waiting period. These groups of lumps have air slaked lime features. In mechanical terms, mortars have medium strength that is identical with hydraulic mortars. Based on the mechanical tests, despite the long time passed since the first building phase of the basilica, the mortars are still stable and un-decomposed which indicates that they were produced through proper techniques.

CONFLICT OF INTEREST

No conflict of interest was declared by the authors.

REFERENCES

[1] Sakaoğlu, N., Çeşm-i Cihan AMASRA. Kültür Bakanlığı, Tarih Vakfı Yay., (Turkish book), İstanbul; 1999.

[2] Vitruvius, Mimarlık Üzerine On Kitap. Şevki Vanlı Mimarlık Vakfı Yay., (Turkish book) İstanbul; 2005.

[3] Ünsal, B., Mimari Tarihi. İDMMA, Yay, (Turkish book) İstanbul; p.541, 1973.

[4] Sanchez-Moral, S., Lugue, J., Canaveras, J.C., Soler, V., Garcia-Guinera, J., Aparicio, A., "Lime pozzolana mortars in Roman catacombs: composition, structures and restoration", Cement and concrete Research, 35, 1555-1565, 2005.

[5] Moropoulou, A., Polikreti, K., Bakolas, A., and Michailidis, P., "Correlation of physicochemical and mechanical properties of historical mortars and

classification by multivariate statistics",. Cem. Con.Research, 33, 891–898, 2003.

[6] Turkish Standard TS EN 14579. Natural Stone Test Methods - Determination of sound speed propagation. TSE, Ankara, October 2006.

[7] European Standard EN 1936, Natural Stone Test Methods. Determination of Real Density and Apparent Density, and of total and open Porosity. Brussels, December 2006.

[8] Turkish Standard TS EN 13755, Natural stone test methods - Determination of water absorption at atmospheric pressure. Ankara, January 2003.

[9] Turkish Standard TS EN 1015-11 Methods of Test Mortar for Masonry – Part 11 Determination of compressive and flexure strength of hardened mortars. TSE, Ankara, October 2000.

[10] Turkish Standard TS EN 12390-6. Testing hardened concrete - Part 6: Tensile splitting strength of test specimens. TSE, Ankara, April 2002.

[11] Böke, H., Çizer, Ö., İpekoğu, B., Uğurlu, E., Şerifaki, K., Toprak, G., "Characteristics of lime produced from limestone containing diatoms", Construction and Building Materials, 22, 866–874, 2008.

[12] Banfill, P.F.G., Forrester, A.M., "A relationship between hydraulicity and permeability of hydraulic lime" Proceedings of the International RILEM workshop: Historic mortars: characteristics and tests", 173–83, Paisley, 2000.

[13] Callebaut, K., Elsen, J., Van Balen, K., and Viaene, W., "Nineteenth century hydraulic restoration mortars in the Saint Michael's Church (Leuven. Belgium) Natural hydraulic lime or cement?", Cement and Concrete Research, 397-403, 2001.

[14] Davidovits,. Les Mortiers de pouzzolanes artificielles chez Vitruve evolution et historique architecturale. These de D.E.A Les Cultures de L'Antiquite Classique. Universite Paris X-Nanterre France, 1995.

[15] Pavia, S. and Caro, S., "An investigation of Roman mortar technology through the spectrographic analysis of archeological material", Construction and Building Materials, 22, 1807–1811, 2008.

[16] Moropoulou, A., Bakolas, A. and Anagnostopoulou, S., "Composite Materials in Ancient Structures", Cement and Concrete Composites, 27, 295-300; 2005..

[17] Tunçoku, S.S. and Caner-Saltık, E.N., "Opal-A rich additives used in ancient lime mortars", Cement and Concrete Research, 36, 1886–1893, 2006.

[18] Zamba, L.C., Stamatakis, M.G., Cooper, F.A., Themelis, P.G. and Zambas, C.G.,. "Characterization of mortars used for the construction of Saithidai Heroon Podium (1st century AD) in ancient Messene, Peloponnesus, Greece", Materials Characterization, 58, 1229–1239, 2007.

[19] Elsen, J., Brutsaert, A., Deckers, M. and Brulet, R., "Microscopical study of ancient mortars from Tournai (Belgium)", Materials Characterization, 53, 289-294, 2004.

[20] Hughes, J. and Leslie, A.B., "The petrography of lime inclusions in historic lime based mortars", Proc 8th Euroseminar on Microscopy Applied to Building Materials. September 4–7 Athens, 359–364, 2001.

[21] Plinius, M.A., Historia Natural, Facsimile, ITGE, 2 vols, (1629), 1998.

[22] Güleç, A. And Tulun, T., "Physico-chemical and petrographical studies of old mortars and plasters of Anatolia", Cem. Concr. Res, 27, 227-234, 1997.

[23] Riccardi, M.P., Duminuco, P., Tomasi, C. and Ferloni, P., "Thermal, microscopic and X-Ray diffraction studies on some ancient mortars", Therm. Acta, 321, 207-214, 1998.

[24] Elsen, J., "Microscopy of historic mortars-A review, "Cement and Concrete Research, 36, 1416–1424 2006.

[25] Aardt, J.H.P. and Visser, S., "Calcium Hydroxide Attack on Feldspars and Clays: Possible Relevance to Cement-Aggregate Reactions", Cement and Concrete Research, 7, 643-648, 1977.

[26] Baronio, G. and Binda, L., "Study of the pozzolanicity of some bricks and clays" Construction and Building Materials, 11(1), 70-78, 1997.

[27] Özkaya, Ö.A. and Böke, H., "Properties of Roman bricks and mortars used in Serapis temple in the city of Pergamon", Materials Characterizations, 60, 995–1000, 2009.

[28] Lawrence, R.M.M., A Study of Carbonation in Non-Hydraulic Lime Mortars. A thesis submitted for the degree of Doctor of Philosophy University Bath, Faculty of Engineering and Design Department of Architecture and Civil Engineering, UK, October 2006.

[29] Stewart, J., Glover, R., Houston, J., Seeley, N. and Proudfoot, T., "Field and laboratory assessment of lime-based mortars", Journal of Architectural Conservation , 7 (1), 7-41, 2001.

[30] Thomson, M., Lindqvish, E.J., Elsen, J. and. Groot, C.J.W.P., "Porosity of historic mortars", Proceedings of the 13 th Int. Brick and Block Masonry Conference, Amsterdam, 1-10, 2004.

[31] ASTM C 593-95. Standard Specification for Fly Ash and Other Pozzolans for Use with Lime (Reapproved 2000).